Introduction to Process Engineering and Design

Second Edition

About the Authors

Shuchen B Thakore graduated in Chemical Engineering from Gujarat University and obtained his ME in Chemical Engineering from M S University, Vadodara. He is currently Associate Professor of Chemical Engineering at L D College of Engineering, Ahmedabad, Gujarat. He has been teaching undergraduate and postgraduate students in Chemical Engineering, for more than 18 years. Besides teaching, he also undertakes consulting assignments in process and mechanical designs of heat exchangers, distillation columns, storage tanks, pressure vessels, etc., and environmental audit for various chemical industries.

Prior to joining the academic institute, he worked in the industry for about five years. He served Alembic Chemical Works, Naveen Heat Exchangers Ltd., Gujarat Alcohol and Allied Chemicals Ltd. and Indian Engineering Software Co.

Mr. Thakore has earlier authored a book on Process Equipment Design.

Bharat I Bhatt graduated in Chemical Engineering from the Institute of Chemical Technology, Mumbai. He is a Consulting Engineer to Chemical Industries for the last ten years in Ahmedabad. His firm, Aavishkar Consultancy Services, provides consulting services in debottlenecking in chemical industries, energy conservation, effluent treatment, etc. He earlier served chemical industries for nearly 34 years. His industrial experience covered the fields of project execution, process engineering, debottlenecking, troubleshooting, production management, management reporting, etc. He spent nearly 18 years with Indian Farmers Fertiliser Cooperative Limited (IFFCO), Kalol Unit in various capacities.

Prior to joining the industry, he served at the reputed textile research institute (ATIRA) for nearly five years where he was involved in industrial research and consultation to member textile units. He also worked in many fine chemical and pharmaceutical industries as Vice President (Technical).

Mr. Bhatt has also authored a book titled *Stoichiometry*, published by McGraw-Hill Education (India). Till date, five editions are published of which the last three editions were in SI Units. While first four editions were co-authored by Dr. S. M. Vora, fifth edition was co-authored by Prof. S. B. Thakore. In addition, Mr. Bhatt carried out units conversion work from FPS units to SI units of the book *Introduction to Chemical Engineering Thermodynamics,* 7th Edition, by Smith, Van Ness and Abbott, a publication of McGraw-Hill, USA. The other book was, *Design Data Book: Properties of Steam, Selected Refrigerants, n-Hexane and Brines (SI units)*.

In 2011, Mr. Bhatt was awarded the UDCT Distinguished Alumnus Award.

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Second Edition

Shuchen B Thakore
Associate Professor
Department of Chemical Engineering
L D College of Engineering
Ahmedabad, Gujarat

Bharat I Bhatt
Consulting Engineer
Aavishkar Consultancy Services
Ahmedabad, Gujarat

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Contents

Foreword *ix*
Preface *xi*

1. Dimensions and Units **1**

1.1 Introduction *1*
1.2 Fundamental Quantities or Dimensions *1*
1.3 Derived Units *2*
1.4 Recommended Rules for Writing Unit Symbols *8*
1.5 Conversion Factors *9*
References *9*

2. Stoichiometry **11**

2.1 Introduction *11*
2.2 Basic Chemical Calculations *11*
2.3 Material Balances *16*
2.4 Energy Balances *20*
2.5 Enthalpy Changes Accompanying Chemical Reactions *22*
2.6 Heat Changes in Mixing Processes *31*
Exercises *36*
References *42*

3. Process Engineering **43**

3.1 Introduction *43*
3.2 Examples of Alternative Routes in Process Engineering *43*
3.3 Generalized Approach to the Chemical Plant Design *44*
3.4 Preparation of Operating Instructions Manual *51*
3.5 Conductance of Performance Test Runs *52*
3.6 Troubleshooting *52*
3.7 Cost Cutting Measures *53*
3.8 Preparation of Data Sheets of Equipments *53*
3.9 Green Engineering *53*
3.10 Process Intensification *55*

3.11 Conclusion *57*
Exercises *57*
References *58*

4. Importance of Process Diagrams in Process Engineering 60

4.1 Introduction *60*
4.2 Need for PFD and P&ID *60*
4.3 Block Diagrams *61*
4.4 Development and Utility of Process Flow Diagrams *62*
4.5 Development of Piping and Instrumentation Diagrams *66*
4.6 Conclusion *78*
Exercises *78*
References *79*

5. Process Design of Piping, Fluid Moving Devices and Flow Meters 80

5.1 Introduction *80*
5.2 Process Design of Piping *80*
5.3 Process Design of Fluid Moving Devices *93*
5.4 Evaluation of Centrifugal Pump Performance When Handling Viscous Liquids *111*
5.5 Power Required in Fan, Blower and in Adiabatic Compressor *115*
5.6 Flow Meters *120*
5.7 Process Design of Orifice Meter *125*
5.8 Process Design of Rotameter *130*
5.9 Two-Phase Flow *138*
5.10 Troubleshooting of Fluid Flow Systems *144*
Exercises *145*
References *147*

6. Process Design of Heat Exchangers 149

6.1 Introduction *149*
6.2 Shell and Tube Heat Exchangers *149*
6.3 Various Parts of Shell and Tube Heat Exchangers *152*
6.4 Advantages and Disadvantages of Different Types of Shell and Tube Heat Exchangers Over Each Other *168*
6.5 Different Types of TEMA Designations *169*
6.6 General Design Method for Shell and Tube Heat Exchangers *169*
6.7 Clasical Method of Heat Transfer and Pressure Drop Calculations for Shell and Tube Heat Exchangers *178*
6.8 Criteria of Selection between Horizontal Condenser and Vertical Condenser *197*
6.9 Condensation with Non-Condensables in Shell and Tube Heat Exchangers *206*
6.10 Multicomponent Condensation *214*
6.11 Tinker's Flow Model *232*
6.12 Delaware Method for Design of Baffled Shell and Tube Heat Exchangers *234*

6.13 Process Design of Reboilers and Vaporizers *254*
6.14 Air Cooled Heat Exchangers and Air Heaters *283*
6.15 Plate Heat Exchangers *305*
6.16 Spiral Flow Heat Exchangers *317*
6.17 Brazed Aluminum Plate-Fin Heat Exchanger *329*
6.18 Other Types of Heat Exchange Equipments *331*
6.19 Heat Exchanger Networking for Energy Integration *331*
6.20 Heat Transfer in Solids *347*
Exercises 358
References 363

7. Process Design of Liquid-Liquid Extractors 365

7.1 Introduction *365*
7.2 Important Industrial Applications of Liquid-Liquid Extraction *366*
7.3 Theory of Liquid-Liquid Extraction Operations *366*
7.4 Desirable Solvent Properties or Choice of Solvent *369*
7.5 Design of Counter Current Multistage Extractor *371*
7.6 Industrially Important Extractors *384*
7.7 Supercritical Extraction (SCE) *416*
Exercises 427
References 431

8. Process Design of Distillation Columns 432

8.1 Introduction *432*
8.2 Criteria of Selection *432*
8.3 Selection of Equipment for Distillation *433*
8.4 Distillation Column Design *434*
8.5 Batch Distillation *540*
8.6 Short Path Distillation *569*
8.7 Reactive and Catalytic Distillation *576*
8.8 Azeotropes and Separation Thereof *589*
8.9 Energy Conservation in Distillation *620*
Exercises 638
References 649

9. Process Design of Absorbers 652

9.1 Introduction *652*
9.2 Criteria of Selection *653*
9.3 Design of Packed Tower-Type Absorber *656*
9.4 Absorption with Chemical Reaction *694*
9.5 Process Design of Spray Chamber or Spray Tower-Type Absorber *703*
9.6 Venturi Scrubber *709*
9.7 Process Design of Falling Film Absorber *712*
9.8 Process Design of Sieve Plate Absorber *734*
Exercises 755
References 757

10. Process Design of Reactors **758**

10.1 Introduction *758*
10.2 Different Types of Reactors *758*
10.3 Batch Reactors *759*
10.4 Continuous Flow Reactors *772*
10.5 Degree of Completion of Reaction *783*
10.6 Mixing for Different Type of Reaction Systems *784*
10.7 Heat Transfer in Batch Reactors *800*
10.8 Bubble Column Reactor *802*
10.9 Thin Film Absorber Cum Reactor *817*
10.10 Catalysis *824*
10.11 Design of Fixed Catalyst Bed Reactors for Gaseous Reactions *829*
Exercises *839*
References *843*

11. Process Design of Utility Systems **845**

11.1 Preamble *845*
11.2 Process Heating Systems *845*
11.3 Process Cooling Systems *861*
11.4 Compressed Air Systems *872*
11.5 Inert Gas Systems *876*
11.6 Other Utilities *879*
Exercises *880*
References *881*

Appendix 1 ***882***

Appendix 2 ***888***

Index ***889***

Foreword

Dr. Jack Welch, CEO of General Electric, built its market cap by more than $450 billion and established himself as the most admired business leader in the world. He is Doctorate in Chemical Engineering and is proud to be a Chemical Engineer. In his business autobiography he wrote, "I have always felt that chemical engineering was one of the best backgrounds for a business career because both the class-work and required thesis teach you one very important lesson: There are no finite answers to many questions."

A chemical process engineer's job is, therefore, tough. It is hard because he has to find not only a finite answer but to select the best answer for the situation from a variety of options. That is why process engineering is interesting and at the same time intriguing.

A process engineer has to design a plant or a part of it. He may have to design a unit operation or a unit process or a system. The important aspect is that it must work and operate reliably and smoothly. The unit is to be designed for a commercial purpose and, hence, it has to be economical and cost effective. A good process engineer has to have plant experience and, in addition, a good understanding of process economics and knowledge of current costs and trends.

Chemical process industry covers a very wide spectrum of industrial products. Apart from the chemicals they handle, the quantities and sizes also vary. This requires the process engineer to have flexibility of mind and vision to adopt itself to any scale. Selection and design of the equipment should therefore be capable to handle the capacities and instruments to measure the parameters for large chemical plants to micro units to manufacture enzymes.

Till 1973, fuel and energy costs were a small component of the manufacturing costs of a chemical product. Since then, the rising fuel prices have made it mandatory that the plant design and its operation be energy efficient. The approach to process design has undergone a paradigm change.

Bhopal gas leakage disaster and Chernobyl catastrophe made another impact on the minds of process engineers and changed the practices followed till then. Greater responsibility now rests on the process engineers for safer designs of the plants. HAZAN (Hazard Analysis) and HAZOP (Hazard and Operatability) studies have become a part of process studies, attaching one more design aspect in the minds of

process engineers. Several lessons regarding storage and handling were learnt by the process engineer. He has to now respect environmental and ecological sustainability of the process and its design.

Software for the design of equipment such as heat exchangers, distillation columns, etc. are available. It has simplified piping design. Drawing and drafting has become easier, error free and faster. But without understanding the design principles, explained in this book, one cannot use them efficiently.

Yet, the process flow sheeting or the development of a process scheme still remains a brain teasing function. It can be explained and perhaps taught but cannot be standardized. Process engineer has to be innovative and creative. Preparation of a Process Flow Diagram is still a prominent and challenging domain of a process engineer.

It is more admirable that Mr. Bharat I. Bhatt and Prof. Shuchen B. Thakore decided to go deeper into some of these topics and write a book on Process Engineering and Design. They have done it with success. I had the opportunity of working with Mr. Bhatt. He is one of the best process engineers I have come across. The long industrial experience of Mr. Bhatt and extensive teaching experience of Prof. Thakore have made them eminently suitable to offer their knowledge to student fraternity and practicing engineers. I have no doubt that the book will find its place with most academicians involved in teaching the subject.

The book describes systematic methods for the process design with many illustrations, drawn from various sections of the industry. They have included several solved and unsolved problems with sufficient details. Authors' approach is user friendly and result oriented. Indepth study of these examples will boost confidence of the students in solving real life design and operating problems.

Large number of references, cited at the end of each chapter, indicates the extensive literature study made by the authors. Further, study of these references will provide more insight to process design aspects.

An exclusive chapter is devoted on development of Process Flow Diagrams (PFD) and Piping and Instrumentation Diagrams (P&ID) which is justified by my earlier comments. Sufficient emphasis is placed on 'green' engineering. These aspects have made this book unique.

Many recent topics like pinch technology, energy conservation in distillation, two-phase flow of fluids, design of loop reactors and many others are to be appreciated which are hard to find in other books on process design.

I have no doubt that the book will make the library of a student and practicing engineer richer.

Suhas M Mokashi
Ex-Director of Technology & Marketing
Kaevernar Powergas Pvt. Ltd.

Preface

Introduction

Chemical process industry is expanding globally to meet new challenges. In this environment, cost reduction has assumed great importance for the process engineers. New processes and technologies are knocking the doors of the process industry to achieve results with stricter environmental regulations. Process engineers have been engaged in meeting these tasks. With these requirements in mind, the present edition has been brought out.

New to this Edition

- New chapter on process design of utility systems
- 30 new process flow-charts are added
- 5 new process and instrumentation diagrams are added
- Two-phase flow in pipelines
- Delaware method of design of heat exchanger
- Design of reactive distillation columns
- Short-path distillation units
- Design of catalysis and catalytic reactors
- Excellent pedagogy that includes
  98 Examples
  82 Exercise Questions
  242 Figures
  208 Tables

Salient Features of the Book

- Focus on Process Design leading to understanding of the design of process plant equipment and systems
- Unit operations to highlight the process design aspects. In each of these operations, current developments are included which provide energy efficient, more reliable and safer designs
- Industrial examples for each concept/principle and a large number of solved problems drawn from the industry to make the book practical and to give students

a good understanding of the design steps and the confidence to solve problems themselves in real life
- State-of-the-art techniques of supercritical extraction, reactive distillation, shore path distillation, etc.

About the Book

The text is thoroughly revised and each chapter is refreshed with newer developments and their design aspects, such as development of piping and instrumentation diagrams, which will be a welcome feature for newer process engineers. Design of shell and tube heat exchangers, Delaware method is considered to offer economic design over the conventional design methods. Its inclusion in this revised edition will surely make the book richer and will be helpful to process engineers. Inclusion of new industrial design problems is an added feature in the book. *Chapter 7* on liquid-liquid extractor is rewritten to include description of various extractors available for selection. In *Chapter 8* on design of distillation column, design of multicomponent batch distillation and example on extractive distillation are included. Absorption with chemical reaction is a new topic in *Chapter 9*. Design of packed tower-type absorber involving chemical reaction is included with industrial example. Use of falling film absorber as a reactor is gaining popularity in the industry. Design of such equipments is covered in *Chapter 10*.

A new chapter on process design of utility systems (Chapter 4) makes this book different from other books on the subject. In this chapter, stress is given on role of a process engineer in selecting and specifying utility requirements correctly rather than carrying out detailed design of the equipment used in delivering the utility. Typical P&I diagrams of the utility systems would help process engineer to develop his own diagrams with confidence.

Green engineering and process intensification have assumed great importance in process industry. A sincere attempt is made to make aware of these requirements to the process engineer in his design.

Use of SI units is the highlight of the book. Current developments by International Bureau of Weights and Measures are well covered in this edition. Many problems in the book are taken from the industry and have been discussed from alternative approaches. Advantages and limitations of newer technologies are spelled out at every stage so that process engineer will use the new ideas judiciously.

In addition, exercises and references at the end of each chapter, and Appendices at the end of the book are added advantages to the book.

Web Supplements

For Instructors
- Solutions Manual
- PowerPoint Slides

For Students
- Objective-type Questions with answers

Acknowledgements

We acknowledge the patronage of the faculty of Chemical Engineering, Industrial Chemistry, Process Engineering, Environmental Engineering, Biochemical Engineering and others. It was an inspiration for us to write this revised edition.

We are thankful to the Principal of L D College of Engineering, Ahmedabad for granting us the permission to revise and publish this edition. We also appreciate the continued support of our spouses; Dimple Thakore and Minaxi Bhatt without whom the task of preparing the manuscript could not have been possible.

We are also thankful to the following reviewers for going through our manuscript and offering valuable suggestions:

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We thank Ms Vibha Mahajan and her editorial team at McGraw-Hill Education (India) for their sincere efforts in bringing out this edition in an attractive format.

Shuchen B Thakore
Bharat I Bhatt

Feedback

Syllabi of various reputed chemical engineering institutes were studied which are being revised from time to time. Every attempt has been made to address their valued comments in this edition. Addition of newer topics is commensurate with revised syllabi.

We are positive that our efforts in making this edition more useful will be appreciated at large. Nevertheless suggestions and constructive criticism are most welcome. Readers can write to us at *b_bhatt26@hotmail.com* and *sbthakore@yahoo.co.in*

Shuchen B Thakore
Bharat I Bhatt

Publisher's Note

We look forward to receiving valuable views, comments and suggestions for improvements from teachers and students, all of which can be sent to *info.india@mheducation.com*, mentioning the title and author's name on the subject line.

Piracy related issues can also be reported.

Dimensions and Units 1

1.1 INTRODUCTION

Knowledge of dimensions and units is imperative in day-to-day dealings. While dimensions are recognized by our sensory perceptions, their use requires definite scales of measurement known as *units*. When a solid is observed, one thinks about its volumetric and mass dimensions. Similarly, a liquid is recognized by its volume and density measurements. A gas exerts definite pressure and temperature which are important for defining its state.

Various quantities, required to identify a substance and its state can be classified in two categories; namely fundamental and derived. Every quantity can be expressed as a product of a dimensionless number and a unit where the unit is a selected reference quantity in terms of which all quantities of the same kind can be expressed.

1.2 FUNDAMENTAL QUANTITIES OR DIMENSIONS

Fundamental quantities or dimensions are length, mass, time, thermodynamic temperature, electric current, amount of substance and luminous intensity. Units, representing these quantities are known as *base units*.

There are basically two systems of units; FPS and metric. The former is widely used in the USA and is also known as *US Customary Units*. It is based on foot, pound and second as standard measurements of length, mass and time, respectively.

Metric (mks) system is based on the unit of length known as metre. The units of mass and time in this system are kilogram and second. Its important feature is the decimal expression.

International system of units (or *Le Système International d'Unités* in French or simply SI Units) was adopted by the 11th General Conference of Weights and Measures in 1960. Base units of the MKS system for length, mass and time are retained in SI. Base units of the other four fundamental quantities are defined in SI. Table 1.1 lists the fundamental quantities in different units.

The thermodynamic temperature (Kelvin), defined in SI, is accepted as the absolute temperature in the metric system. For definitions of all the fundamental quantities, references 1 to 4 may be consulted.

The table of elements based on carbon-12 is updated from time to time by the International Union of Pure and Applied Chemistry (IUPAC), USA which gives atomic masses (m) and atomic numbers of the elements. Current listing[5] is available as Atomic Weights of Elements, 2007.

Table 1.1 Fundamental Quantities

Symbol	Fundamental quantity	System of units with symbolic abbreviation			Dimension
		SI	Metric	fps	
l	Length	Metre (m)	Metre (m)	Foot (ft)	L
m	Mass	Kilogram (kg)	Kilogram (kg)	Pound (lb)	M
θ	Time	Second (s)	Second (s)	Second (s)	θ
I	Electric current	Ampere (A)	Ampere (A)	Ampere (A)	I
T, t or $\mathcal{T}$	Temperature	Kelvin (K)	Celsius (°C)	Fahrenheit (°F)	T
n	Amount of substance	Mole (mol)			n
I_v	Luminous intensity	Candela (cd)			

In 1971, the concept of the amount of a substance was introduced as a fundamental quantity. It is defined as the amount of substance of system containing as many elementary entities as the number of atoms in 0.012 kilogram of carbon-12 element. This unit of quantity was called a *mole* (abbreviated as mol). Thus unified scale of mole gives value of the relative atomic mass (m). When a compound is represented by a chemical formula, then the total mass computed based on the formula is called **molar mass** (popularly known as molecular weight) and is represented by the symbol M.

1.3 DERIVED UNITS

Derived units (including supplementary units) are expressed algebraically and in terms of base units by means of mathematical signs of multiplication and division. For example, the SI unit of work or energy is Joule which is defined as work done when one Newton (N) force acting on a body moves it by a distance of one metre (see Sec. 1.3.4).

Table 1.2 lists some of the important SI derived units used in Chemical Engineering with their special names while Table 1.3 lists frequently used derived units.

Table 1.2 SI Derived Units with Special Names, Including SI Supplementary Units

SI derived unit (Derived quantity)	Special name	Symbol	Expressed in terms of SI base units and SI derived units
Plane angle	radian	α, β, γ	1 rad = 1 m/m = 1
Solid angle	steradian	Ω, ω	1 sr = 1 m^2/m^2 = 1
Frequency	hertz	f	1 Hz =1 s^{-1}

(*Contd.*)

Table 1.2 (*Contd.*)

SI derived unit (Derived quantity)	Special name	Symbol	Expressed in terms of SI base units and SI derived units
Force	newton	F	1 N = 1 kg · m/s^2
Pressure or stress	pascal	p, σ	1 Pa = 1 N/m^2
Energy or Work or Quantity of heat	joule	E, H	1 J = 1 N · m
Power Radiant flux	watt	P	1 W = 1 J/s
Electric charge or Quantity of electricity	coulomb	Q	1 C = 1 A · s
Electric potential or Potential difference, or Tension or Electromotive force	volt	V	1 V = 1 W/A
Electric resistance	ohm	R	1 Ω = 1 V/A
Electric conductance	siemens	$G = 1/R$	1 S = 1 Ω^{-1}
Celsius temperature	degree Celsius[1]	t	1°C = 1 K

[1]Degree Celsius is a special name for the unit Kelvin. For use in stating value of Celsius temperature. t/°C = T/K – 273.15.

Table 1.3 Additional Frequently Used SI Derived Units

Quantity	Symbol	SI unit	Practical units	Notes
Acceleration	a	m/s^2	m/s^2, cm/s^2	
Acceleration of free fall	g	m/s^2	m/s^2	
Isentropic exponent	k			$k = C_p/C_v$ (for ideal gas)
Ambient Pressure	p_{amb}	N/m^2, Pa	mbar, bar, Torr	
Angular acceleration	α	rad/s^2	rad/s^2	
Angular velocity	ω	rad/s	rad/s	
Area	A, A_s, S, a	m^2	m^2, cm^2	
Concentration of substance B	C_B	mol/m^3	mol/m^3, mol/L	
Current density,	j	A/m^2	A/m^2, A/cm^2	
Density (mass concentration)	ρ	kg/m^3	kg/m^3, g/cm^3, kg/L	
Relative density/Specific gravity	d	ρ_{t_1}/ρ_{t_2}	—	Non-dimensional quantity
Diametre	d, D	m	cm, mm	
Diffusion coefficient	D	m^2/s	m^2/s, cm^2/s	

(*Contd.*)

Table 1.3 (*Contd.*)

Quantity	Symbol	SI unit	Practical units	Notes
Efficiency	η	—	—	Non-dimensional quantity
Heat (enthalpy) of evaporation	λ	J/g, J/mol	kJ/kg, kJ/kmol	
Volumetric flow rate	q_v	m^3/h	m^3/h, m^3/s, L/min	
Molar flow rate*	$\dot{n}$	mol/s	mol/s, kmol/h	
Gas constant (molar)	R	J/(mol · K)	(mbar · L)/(mol · K), (m^3 · bar)/(kmol · K)	
Gas constant of substance B (specific)	R_B	J/(kg · K)	(mbar · L)/(kg · K), (m^3 · bar)/(kg · K)	
Heat capacity of a given mass	C	J /K	J/K, kJ/K	
Heat capacity, isobaric, molar	C_{mp}	J/(mol · K)	kJ/(kmol · K)	
Heat capacity, isobaric, specific	C_p, C_l	J/(g · K)	kJ/(kg · K)	
Heat capacity, isochoric: molar	C_{mv}	J/(mol · K)	kJ/(kmol · K)	
Heat flow rate or heat flux	ϕ	W, J/s	kW, kJ/s, kJ/h	
Heat transfer coefficient	h, U	W/(K · m^2)	W/(K · m^2), kcal/(h · m^2 · °C)	
Height	h	m	m, cm, mm	
Kinematic viscosity	γ	m^2/s	m^2/s, mm^2/s, cm^2/s, cSt	
Kinetic energy	E_k	J	J, kJ	
Leak rate (in a vacuum system)	q_L	N · m/s	mbar · L/s	
Length	L	m	m, cm, mm	
Liner expansion coefficient	α_l	m/(m · K),	m/(m · K), 1/K	
Mass	m	kg	kg, g	
Mass flow rate	q_m . $\dot{m}$	kg/s	kg/s, kg/h, g/s	
Mass fraction	w, X, Y	kg/kg	%, ppm	ppm = parts per mega parts
Molality	$m^{\ominus}$	mol/kg	mol /kg	—
Molarity	M	mol/dm^3	mol/m^3, mol/L,	—

*In the book, alphabets in italic are also used for molar flow rates. (*Contd.*)

Table 1.3 (*Contd.*)

Quantity	Symbol	SI unit	Practical units	Notes
Molar mass	M	kg/kmol	kg/kmol, g/mol	
Molar volume	V_m	m^3/mol	m^3/kmol, L/mol	
Mole fraction	x (in liquid) y (in vapor/ gas)	–	–	
Number of revolutions	n	1/s	1/s, 1/min (or rpm)	
Partial pressure of component B	p_B	N/m^2, Pa	mbar, kPa, Torr	
Period (periodic time)	θ	s	s, ms, µs	
Permeability (for gases)	μ	Nm^3/ ($m^2 \cdot s \cdot Pa$)	$Nm^3/(m^2 \cdot d \cdot bar)$	
Pumping speed (rate)/ volumetric flow rate	$q_v, \dot{V}$	m^3/s	L/s, m^3/h, m^3/s	
Quantity (volume) of gas	V	Nm^3	L, Nm^3, Sm^3	
Quantity of heat	Q, H, i	J	J, kJ, kWh, W · s	
Radius (also of molecule)	r	m	cm, m, µm	
Vapour pressure	p_v	N/m^2, Pa	mbar, kPa, Pa, Torr	
Saturation vapour pressure	p_s	N/m^2, Pa	mbar, kPa, Pa, Torr	
Temperature difference	$\Delta\mathcal{T}, \Delta t$	K	K, °C	
Thermal conductivity	k	W/(K · m)	W/(K · m), kW/(m · K), kcal/(h · m · °C)	
Thermal diffusivity	a	m^2/s	m^2/s	
Velocity	v	m/s	m/s, mm/s, km/h	
Viscosity (dynamic)	μ	Pa · s	mPa · s, cP	
Volume	V	m^3	m^3, L, cm^3	
Volume (specific)	v	m^3/kg	m^3/kg, cm^3/g, m^3/kmol	
Volume fraction of substance B	ϕ_B	m^3/m^3	L/L, %, ppm	ppm = parts per mega parts
Wavelength	λ	m	nm, Å	
Weight (force)	G	N	N, kgf	
Specific/absolute humidity	H	kg /kg	kg/kg, g/g	
Relative humidity	RH	—	—	No dimensions
Saturation ratio	φ	—	—	- do -

It will now be appropriate to elaborate a few of the important SI derived units for better understanding.

1.3.1 Force

According to the Newton's second law of motion,

$$F \propto m \times a \tag{1.1}$$

or

$$F = K\, m\, a \tag{1.2}$$

where, K = proportionality constant

Selecting K as unity, the unit newton is defined. Thus, when a force of one newton (N) is applied to a body having a mass of one kilogram it gives an acceleration of one m/s^2.

Definition of technical unit of force is obtained by selecting K equivalent to $1/g_c$ where g_c is called *Newton's law conversion factor*. Its value is 9.806 65 $(kg \cdot m)/(kgf \cdot s^2)$ and is equal to the acceleration due to gravity (g) at mean sea level. It may be noted that g_c is not equal to g at all locations on the earth.

Kilogram-force (kgf) is the force which when applied to a body having a mass of one kilogram, gives it an acceleration of 9.806 65 m/s^2. It is the base unit in metric system of units.

$$1 \text{ N} = 0.101\,972 \text{ kgf}$$

or

$$1 \text{ kgf} = 9.806\,65 \text{ N}$$

Although mass is the base unit, it *nearly* equals weight under gravitational acceleration (g). This is why 'mass' and 'weight' terms are commonly used without differentiation in general practice.

1.3.2 Pressure

Pressure is the force acting on a unit area, exposed to it.

$$p = \frac{F}{A} \tag{1.3}$$

Common units of pressure in SI and metric systems are N/m^2 (known as Pascal; symbol Pa) and kgf/cm^2, respectively. Pressure, measured with the help of a gauge, is called *gauge pressure* or over pressure and the letter 'g' follows the unit. When (local) atmospheric pressure is added to the gauge pressure, it is called absolute pressure and the letter 'a' follows the unit. Standard atmospheric pressure (symbol atm) is measured at mean sea level and is assigned the value 101 325 N/m^2 or Pa or 1.033 kgf/cm^2. Since Pascal is a very small unit of pressure, bar is chosen as a practical pressure unit in the SI system.

$$1 \text{ bar} = 10^5 \text{ Pa} = 1.019\,716 \text{ kgf/cm}^2 = 0.986\,923 \text{ atm}$$

$$1 \text{ atm} = 1.013\,25 \text{ bar}$$

Pressure is also expressed in terms of fluid heads, e.g., water column (WC), liquid column (LC), mercury column (Hg), etc.

$$\text{Fluid head} = \text{Pressure /density} = p/\rho \tag{1.4}$$

$$1 \text{ atm} = 760 \text{ mm Hg at } 0°\text{C or Torr} = 10.33 \text{ m WC at } 4°\text{C}$$

Vacuum refers to pressure below atmospheric pressure and hence absolute pressure of a vacuum system is derived by substracting vacuum from atmospheric pressure.

1.3.3 Volume

Volume is the space occupied by a substance. It is measured in cubic metres and liters in SI and metric units, respectively. Approximately, 1 $m^3 \approx 1000$ L $\approx$ 1 kL.

1.3.4 Work, Energy, Power and Heat

Work is defined as the product of force acting on a body and the distance traveled by the body.

$$W = F \times d \tag{1.5}$$

Units of work $(kg \cdot m^2)/s^2$ or N · m or joule (symbol J) and m · kgf in SI and metric systems, respectively.

Internal energy (Symbol U or E) is a physical entity which is present in a system in different forms; mechanical, chemical, thermal, etc. Heat is the form of energy in transit that flows from higher temperature to lower temperature. Total **enthalpy** (symbol H) of a substance is the total energy possessed by it under the given conditions.

$$H = U + pV \tag{1.6}$$

Unit of energy, heat and enthalpy is joule and calorie (symbol cal) in SI and metric systems, respectively.

1 calorie (International Steam Tables) = 4.1868 J

It may be noted that unit of work, energy, heat and enthalpy in SI is same, i.e., J (unlike metric system).

Power P is defined as the work done per unit time

$$P = \frac{W}{\theta} \tag{1.7}$$

Unit of power is Watt (i.e., J/s) and m · kgf/s in SI and metric systems, respectively. Practical unit of power in metric system is horsepower.

1 metric system horsepower = 75 (m · kgf)/s = 0.7355 kW

1.3.5 Derived Electrical Units

Electric potential (V) is defined as

$$V = \frac{W}{I} \tag{1.8}$$

Its unit is volts.

Resistance of the conductor is defined as

$$R = \frac{V}{I} \tag{1.9}$$

Its unit is ohms (Ω). Reciprocal of resistance is called *conductance*; symbol (G) and has units siemens (S). Quantity of electricity (symbol Q) is defined as the product of current and time.

$$Q = I \cdot \theta \tag{1.10}$$

Unit of quantity of electricity is coulomb (C).

1 Faraday (F) = 96 485.3415 C/mol

1.4 RECOMMENDED RULES FOR WRITING UNIT SYMBOLS

Following guidelines may be followed while using SI unit symbols:

1. SI prefixes are given in Table 1.4.

Table 1.4 SI Prefixes

Factor	Prefix	Symbol	Factor	Prefix	Symbol
10^{24}	yotta	Y	10^{-1}	deci	d
10^{21}	zetta	Z	10^{-2}	centi	c
10^{18}	exa	E	10^{-3}	milli	m
10^{15}	peta	P	10^{-6}	micro	μ
10^{12}	tera	T	10^{-9}	nano	n
10^{9}	giga	G	10^{-12}	pico	p
10^{6}	mega	M	10^{-15}	femto	f
10^{3}	kilo	k	10^{-18}	atto	a
10^{2}	hecto	h	10^{-21}	zepto	z
10^{1}	deca	da	10^{-24}	yocto	y

2. An exponent attached to compound prefix-unit implies that the exponent refers to the entire compound unit and not just to the base symbol. 1 cm^3 means volume of a cube having one cm side.
3. The product of two or more units may be indicated in any one of the following ways:
   Correct: N m, or N · m Incorrect: Nm
4. A solidus (oblique stroke), a horizontal line or negative powers may be used to express a derived unit, formed from two others by division, e.g., linear velocity can be expressed in one of following four ways:

$$\text{m/s},\ \frac{\text{m}}{\text{s}},\ \text{m} \cdot \text{s}^{-1} \text{ or m s}^{-1}$$

5. A solidus must not be repeated on the same line unless ambiguity is avoided by a parenthesis.

Correct	Incorrect
m/s^2 or $m \cdot s^{-2}$ or $m\ s^{-2}$	m/s/s
$W/(m^2 \cdot K)$ or $W\ m^{-2}\ K^{-1}$	$W/m^2 \cdot K$ or $W\ m^{-2}/K$

6. Unit symbols do not change in the plural. For example, 5 metres should be abbreviated as 5 m and not as 5 ms.
7. Unit symbols are not followed by a full stop (period) except at the end of a sentence.
   Correct: 8 kg Incorrect: 8 kg.
8. When numerical values fall outside the range of 0.1 to 1000, it is recommended that the numerals be separated into groups of three with a space replacing the traditional comma.

Recommended	Not recommended
3 600 or 3600	3,600
19 625 725	19,625,725
0.001 625	0.001,625
0.046 89	0.046,89

In this book, four digits or decimals are grouped.

9. Prefix symbols are printed without any space between the prefix symbol and the unit symbol.
   Correct: 10 kW Incorrect: 10.5 kW
10. Compound prefixed formed by the juxtaposition of two or more SI prefixes are not to be used.
    Correct: 6 GJ Incorrect: 6 MkJ
11. A prefix should never be used alone.
    Correct: $10^6/m^3$ Incorrect: M/m^3
12. Although kilogram is the base unit in SI units, names of decimal multiples and submultiples of mass are formed by attaching prefixes to the word 'gram'.
    Correct: 1 mg Incorrect: 1 μkg
13. Good practice recommends selection of a prefix which, whenever possible, provides a numerical value between 0.1 and 1000. Prefer expression 10.0 kPa over 0.01 MPa. However, when values are tabulated, they should be expressed in the same unit multiple even though their numerical value lies outside 0.1 to 1000 range.
    12×10^4 J can be written as: 120 kJ.
    1632 W can be written as 1.632 kW.
14. If the magnitude of the number is less than unity, the decimal sign should preferably be preceded by zero, e.g., .072 should be written as 0.072.
15. The SI prefixed are not to be used with °C or K.
16. In the expression of a quantity, the unit symbol is placed after the numerical number and a space is left between the two.
    Examples 52 kg or 25.4 cm

As an exception, in this book, a space is not left between the numerical value and the symbol for the value of celsius temperature. Also while expressing pressure in terms of fluid heads, unit and fluid name are separated.

1.5 CONVERSION FACTORS

Appendix 1 lists the conversion factors in a direct usable form for conversion from one system of units to another system of unit.

REFERENCES

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4. Bhatt, B. I., and S. B. Thakore, *Stoichiometry*, 5th Ed., McGraw-Hill Education, New Delhi, 2010.
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Stoichiometry 2

2.1 INTRODUCTION

Stoichiometry is a branch of chemistry which deals with combining the proportions of the elements or compounds involved in chemical reactions. It is based on three concepts; conservation of mass, the relative masses of atoms and definition of a mole. While the last two concepts were introduced in Chapter 1, the law of conservation of mass is dealt in this chapter.

In any chemical reaction or in a unit operation, exchange of thermal energy also takes place. Therefore, heat balance is also important in stoichiometric calculations. Similar to mass balance, heat balance calculations are based on the law of conservation of energy (the first law of thermodynamics).

2.2 BASIC CHEMICAL CALCULATIONS

Before attempting mass and energy balance problems, knowledge of basic principles of chemical engineering calculations is necessary. A brief review of these calculations is presented as follows.

2.2.1 Atomic Mass and Molar Mass

Elements are basic entities which are combined (or reacted) to form a compound. As seen in Chapter 1, relative atomic masses for each element are presented on a unified scale which is known as *Atomic Masses (Weights) of Elements*[1].

For chemical compounds, a *mole* is defined as the amount of substance equal to its formula weight. Molar mass (M) is thus the total mass of elements, contained in the compound. Thus it is easy to calculate molar mass of a compound, knowing its chemical formula.

Example 2.1 Calculate molar mass of the following:

(a) Sulphurous chloride ($SOCl_2$) and
(b) 2,4,6 – trinitrophenol (also known as picric acid)

Solution

(a) Molar mass of $SOCl_2$ = Atomic mass of sulphur + Atomic mass of oxygen + 2 × Atomic mass of chlorine

$$= 32.065 + 15.9994 + (2 \times 35.453)$$
$$= 118.9704 \text{ kg/kmol (exact)}$$

With rounded off values of atomic masses,

molar mass of $SOCl_2 = 32 + 16 + 2 \times 35.5$
$= 119$

(b) Chemical formula of picric acid = $C_6H_2(NO_2)_3OH$

Molar mass of picric acid $= 6 \times 12.0107 + 3 \times 1.007\,94 + 3 \times 14.0067 + 7 \times 15.9994$
$= 72.0642 + 3.023\,82 + 42.0201 + 111.9958$
$= 229.103\,92$ kg/kmol (exact)
≈ 229 (rounded off value)

In this book, rounded off values of atomic masses will be used for convenience.

2.2.2 Mole

In Chapter 1, mole is defined as a base unit in SI. It can be further defined as the amount of substance containing $6.022\,142 \times 10^{23}$ molecules (also known as Avogadro's number, N_A). In simple terms, number of moles can be found by dividing mass of the compound by its molar mass.

Example 2.2 Convert the following into moles:

(a) 1000 kg boron trichloride (BCl_3)
(b) 1500 kg phenol (C_6H_5OH)

Solution

(a) Molar mass of $BCl_3 = 11 + 3 \times 35.5 = 117.5$ kg/kmol

$$1000 \text{ kg } BCl_3 \equiv \frac{1000}{117.5} = 8.5106 \text{ kmol}$$

(b) Molar mass of $C_6H_5OH = 6 \times 12 + 6 \times 1 + 16$
$= 94$ kg/kmol

$$1500 \text{ kg } C_6H_5OH \equiv \frac{1500}{94} \equiv 15.9574 \text{ kmol.}$$

2.2.3 Mass Fraction and Mole Fraction

In a mixture, mass fractions (w) or mole fractions (x, y) are expressed to represent its composition. *Mass fraction* is the ratio of the mass of a particular substance divided by total mass of all the substances. Similarly, mole fraction is the ratio of the moles of a particular substance divided by total moles of all the substances. One can be calculated knowing the other.

Example 2.3 Aqueous calcium chloride brine contains 35% $CaCl_2$ by mass. Convert the same in mole %.

Solution

Basis: 100 kg brine

Table 2.1 Composition of Brine

Component	Mass kg	Molar mass kg/kmol	moles kmol	mole % (x_i)
$CaCl_2$	35	111	0.3153	8.03
H_2O	65	18	3.6111	91.97
Total	100		3.9264	100.00

$$\text{Average molar mass } (M) \text{ of brine} = \frac{100}{3.9264} = 25.47 \text{ kg/kmol}$$

Example 2.4 Feed gas to ammonia synthesis loop has molar composition 24.75% N_2, 74.25% H_2, 0.7% CH_4 and 0.3% Ar. Calculate its mass composition, average molar mass and specific gravity (with respect to air).

Solution

Basis: 100 kmol feed gas

Table 2.2 Composition of Feed Gas to Ammonia Synthesis Loop

Component	kmol	Molar mass kg/kmol	Mass kg	mass %
N_2	24.75	28	693.0	80.14
H_2	74.25	2	148.5	17.17
CH_4	0.70	16	11.2	1.30
Ar	0.30	40	12.0	1.39
Total	100.00		864.7	100.00

$$\text{Average molar mass } (M) \text{ of feed gas} = \frac{864.7}{100} = 8.647 \text{ kg/kmol}$$

Similarly, average molar mass of air is be calculated to be 28.9697 in Example 2.10 (rounded off value to be 29).

$$\text{Specific gravity of feed gas} = \frac{8.647}{28.9697} = 0.2985$$

While mass fractions are useful in establishing material balances, mole fractions are logical expressions in chemical reactions.

2.2.4 Density and Specific Gravity

Density of a compound in any state (solid, liquid or gas) is expressed as the ratio of mass per unit volume. It is normally expressed in g/cm^3, kg/L or kg/m^3. Density of iron is 7.75 g/cm^3 at 20°C while that of aqueous solution of 24% $BaCl_2$ at 40°C is 1.243 g/cm^3.

Specific gravity (d) of a liquid or solid is a dimensionless number and it is defined as the ratio of density of solid or liquid at a given temperature to the density of water at 4°C (which is nearly equal to 1.000 g/cm^3). For liquids, specific gravities are often expressed in terms of certain hydrometers. Among these, °Twaddell (°Tw) and °Baumé (°Be′) scales are well known. In petroleum industry, hydrometer scale called °API is used which is very useful in finding certain properties like molar mass, viscosity, heating value, etc. Specific gravity of 24% aqueous $BaCl_2$ solution at 40°C is 48.6°Tw. Strength of certain important inorganic solutions such as that of caustic soda solutions, sulphuric acid solutions, etc., are tabulated on °Tw or °Be′ scale as hydrometers are convenient for use in industry.

Specific gravity of a gas is the parameter which indicates the heaviness of a gas as compared to that of air. One definition is the ratio of molar mass of gas to the molar mass of air. In Example 2.4, specific gravity of feed gas to ammonia synthesis loop is determined to be 0.2985, indicating that it is much lighter than air (both at same operating conditions).

Another definition of specific gravity of a gas is the ratio of the density of the gas at given conditions of pressure and temperature to the density of air at reference conditions. Although this is a more logical expression, molar mass ratio is chosen in this book to express heaviness of a gas as compared to the air.

2.2.5 Vapour Pressure and Boiling Point of Liquid

When a liquid is heated, it gets converted to vapour at a given temperature and pressure. For a pure liquid, since degree of freedom is one, it boils at a constant temperature for a given pressure. Conversely, for a pure liquid at a given temperature, its equilibrium pressure is fixed which is known as *vapour pressure*. This implies that when both phases exist in equilibrium, there is only one definite pressure (called vapour pressure) at a given temperature for a single component.

Boiling point (t_B) of a pure liquid at standard atmospheric pressure (1 atm or 101.325 kPa) is called *normal boiling point* (NBP). At 1 atm, water boils at 100°C, ethyl alcohol boils at 78.4°C, methylene chloride boils at 40.1°C and refrigerant 134a (tetrafluoroethane) boils at –26.1°C. All are called normal boiling points.

Relationship of vapour pressure with its saturation temperature is expressed either empirically or in tabulated form. Variety of equations are known for calculating vapour pressure of a liquid. Among these, Antoine equation is well known and is expressed as follows:

$$\log p_v = A - \frac{B}{(T + C)} \tag{2.1}$$

where p_v is vapour pressure at temperature T (in K) while A, B and C are constants. Ref. 1 lists values of A, B and C for several liquids. Validity of equations for a given

liquid is limited in a definite temperature range. Number of other equations are reported in literature for accurate prediction of vapour pressure.

Some of the fluids are extensively used in industry over a wide range of pressure and temperature. Their applications could vary from heating, cooling or processing. Steam and certain vapour phase heating fluids are used for heating. Similarly, a variety of refrigerants are used for cooling. *n*–Hexane is used for extraction of oil from seeds. Nitrogen, oxygen, etc., are also used as feed in various chemical reactions. Carbon dioxide, water, propane and butanes are used in their supercritical states for extraction and for carrying out certain chemical reactions. Thus, thermo-physical properties of all such fluids are required at different pressures and temperatures. For this reason, properties of these fluids are available in tabular form. These tables list temperature and its corresponding vapour pressure as one property. For example, steam tables (Ref. 2) are widely used in industry. Data, read from the table, permit quick calculations.

Several other properties of liquids and solutions are of importance in chemical engineering calculations. References 1 and 3 may be referred for the discussions on these properties.

2.2.6 Gases

Gaseous state of a pure component is defined by its pressure and temperature. However, when a mixture of gases is present, its composition is also required to be specified. In a conventional practice, mole % composition of a gaseous mixture is specified (as seen in Example 2.4).

Gases behave as ideal in a definite range of pressure and temperature such that they obey the well-known ideal gas law.

$$pV = RT \tag{2.2}$$

where, p = absolute pressure of the gas, kPa
V = volume occupied by the gas, m^3/kmol
R = Universal Gas Constant
= 8.314 472 J/(mol·K) or m^3·kPa/(kmol·K)
T = absolute temperature of the gas, K

When p is chosen as one standard atmosphere (101.325 kPa) and T is chosen as 273.15 K (i.e., 0°C), these are called normal conditions (i.e., NTP).

Molar volume at NTP of an ideal gas is 22.414 m^3/kmol.

Conversely an ideal gas is one which follows Eq. (2.2). Most gases under ordinary circumstances (such as ambient conditions) obey ideal gas law. However, real gases deviate significantly at high pressures and high temperatures. Real gas behaviors are described by equations of state (also called *p-V-T* relationships). Reference 4 may be referred for more information on various equations of state. *p-V-T* relations can be represented on phase diagrams. *p–t* diagrams for water and carbon dioxide are given in Figs. 7.28 and 7.29.

Critical conditions of a gas are those beyond which a gas cannot be liquefied. These conditions are defined by critical temperature, critical pressure and critical volume. By definition, critical temperature is the maximum temperature at which a gas can be liquefied. The critical pressure is the saturation (vapour) pressure corresponding to the critical temperature. The volume, occupied by the fluid at critical point is the

critical volume. At the critical point, densities (or specific volumes) of gas and liquid (in equilibrium) are equal while latent heat of evaporation (λ) is zero. Reference 1 lists the critical properties of number of compounds.

Physical state of a fluid above its critical conditions is known as *supercritical state*. In Chapter 7, extraction in supercritical fluids is discussed. Table 7.8 lists the critical properties of commonly used supercritical fluids.

When a mixture of gases is handled, its composition is specified in mole %. For an ideal gas mixture,

mole % = volume % = pressure %

Partial pressure (or pure component pressure) of a component in the ideal gas mixture is defined as total pressure of the system multiplied by the mole fraction of the component.

$$p_i = p \cdot y_i \tag{2.3}$$

where, p_i = partial pressure, kPa
p = total pressure, kPa
y_i = mole fraction of the component in gas phase

For an ideal liquid–vapour mixture, equilibrium at a given temperature is represented by Raoult's law.

$$p_i = p \cdot y_i = p_{vi} \cdot x_i \tag{2.4}$$

where, p_{vi} = vapour pressure of ith component at the given temperature, kPa
x_i = mole fraction of ith component in liquid phase

Conversely, an ideal liquid mixture is one which follows Raoult's law. Non-ideal behavior of the liquid mixture is expressed in different ways for which a standard text book on thermodynamics may be referred (Ref. 4).

For certain liquid mixtures at a definite composition, liquid and vapour compositions are same at a given pressure. Composition of such a mixture is known as *azeotropic composition*. Ethyl alcohol-water, toluene-water and many multicomponent mixtures exhibit this property which is advantageously used in the industry to remove water from organic reaction mass.

2.3 MATERIAL BALANCES

A process represents a series of actions or operations or treatments that result in an end product. In chemical engineering, physical and/or chemical changes take place during the operations and the material flows and its inventory in a system are quantified during these operations which is known as *material balance*.

A system can be defined as the body or assemblage in which the process is set out for analysis. When a system is isolated from the surroundings, it is called a *batch system*. In other words, there is no transfer of materials from a batch during the time interval of interest. Contrary to this, in an open system, material is transferred across the boundaries, i.e., from and to the surroundings. Thus it is important to define the system being analyzed.

Having defined the system, it is important to fix a basis, e.g., 100 kg feed, 100 kg reactant A, 100 kmol gas mixture, 1000 L liquid, etc. All computations are performed considering this basis. In a prolonged analysis of the system, it may become necessary to shift bases but interrelation of all the bases should be clear.

It is thus clear that material balance is a quantitative expression of law of conservation of matter. It can be expressed as

$$\text{Input} = \text{Output} + \text{Accumulation} \tag{2.5}$$

Equation (2.5) is always true for elements but often not true for compounds participating in chemical reactions. Because of its fundamental nature, material balance is one of the most useful tools in system analysis and therefore in process design.

2.3.1 Material Balances Involving No Chemical Reactions

In most unit operations, reactions do not take place. Material balances of such systems are worked out by making balances of a tie material or inert material. When two or more compounds in the system are simultaneously affected, material balance equations are solved by satisfying simultaneous equations.

Example 2.5 Soybean seeds are batch extracted with *n*–hexane. Flaked seeds containing 19% oil, 69% solids and 12% moisture (by mass) are fed to the batch extractor. At the end of the extraction process, deoiled cake (DOC) is found to contain 1% oil, 88% solids and rest moisture (by mass). Find the percentage recovery of oil.

Solution

Basis: 100 kg soybean seeds

Since solids do not take part in the extraction process, its balance can be made.

Solids in the feed = 100 × 0.69 = 69 kg

DOC contains 88% solids.

$$\text{Quantity of DOC} = \frac{69}{0.88} = 78.41 \text{ kg}$$

$$\text{Oil in seeds} = 100 \times 0.19 = 19 \text{ kg}$$

$$\text{Oil in DOC} = 78.41 \times 0.01 = 0.784 \text{ kg}$$

$$\text{Oil recovery} = \frac{(19 - 0.784)}{19} \times 100$$

$$= 95.87\%$$

Example 2.6 *n*–Butanol-water forms an azeotrope having composition of 57.5% *n*–butanol (by mass) at 93°C and 1 atm. When azeotropic vapour is condensed and cooled to 20°C, the mixture separates in two layers. Top layer and bottom layer consist of 79.93% and 7.81% *n*–butanol, respectively (by mass) at 20°C. Calculate the proportions of top and bottom layers.

Solution

Basis: 100 kg azeotropic vapour to condenser

n–Butanol in vapour = 57.5 kg

Water in vapour = 100 – 57.5 = 42.5 kg

In the liquid phase, the mixture separates in two layers. Since both components are affected, simultaneous equations need to be solved.

Let m_1 kg be top layer and m_2 kg be the bottom layer.
Overall balance: $m_1 + m_2 = 100$
n–butanol balance : $0.7993\ m_1 + 0.0781\ m_2 = 57.5$
Solving the equations, $m_1 = 68.9$ kg, $m_2 = 31.1$ kg
Proportion of top layer/bottom layer = 68.9/31.1 = 2.215 (mass)

Solving simultaneous equations for a binary system could be easily done manually. However, when many components are present, use of a mathematical software such as Mathcad®, will be advantageous.

2.3.2 Material Balances Involving Chemical Reactions

Material balance calculations of processes involving chemical reactions follow the same law of conservation of mass. However, moles are important rather than mass in chemical reactions. For material balance calculations, conversion and yield are required.

Conversion is defined as the fraction of the reactant in the feed which is converted to product(s). Conversion is normally defined on the basis of the limiting reactant which is present in the smallest stoichiometric amount. Yield is defined by the following equation:

$$\text{Yield} = \frac{\text{Moles of desired product} \times \text{Stoichiometric factor } (v_i)}{\text{Moles of specific reactant consumed}} \tag{2.6}$$

where, stoichiometric factor (v_i) = stoichiometric requirement of the specific reactant (in moles) per mole of the desired product

Selectivity is the ratio of amount of limiting reactant that reacts to give the desired product to the amount that reacts to give undesirable product(s).

One more term used in reaction engineering is the excess reactant which is defined as the reactant present in excess of stoichiometric (theoretical) requirement of the limiting reactant on mole basis.

While conversion is related to the degree of completion of reaction, yield and selectivity are related to the degree to which the desired reaction proceeds.

Study of oxidation reaction to produce nitrogen monoxide can be useful to understand various terms.

Following are the chief competing reactions taking place in the reactor in a nitric acid plant:

$$4\ NH_3 + 5\ O_2 \rightarrow 4\ NO + 6\ H_2O \tag{a}$$
$$4\ NH_3 + 3\ O_2 \rightarrow 2\ N_2 + 6\ H_2O \tag{b}$$
$$4\ NH_3 + 4\ O_2 \rightarrow 2\ N_2O + 6\ H_2O \tag{c}$$

Above reactions are carried out over a precious metal catalyst at high temperature. Aim is to maximize production of NO. Ammonia and oxygen (in the form of air) are the reactants. At the exit of the reactor, ammonia is completely consumed and hence it is the *limiting reactant*. Oxygen is fed in excess over the stoichiometric requirement (5/4 times ammonia feed rate) and is termed as *excess reactant*. In this case, conversion is 100% based on ammonia. Stoichiometric factor (v_i) for reaction (a) is one, i.e., 1 mole ammonia is required per mole of NO. Typical yield of NO is found to be

0.93–0.98 depending on operating temperature and catalyst activity. Take for example, yields of NO, N_2 and N_2O as 0.95, 0.02 and 0.03, respectively.

NH_3 consumed for desired product = 0.95 kmol

NH_3 consumed for undesired products = $0.02 \times 2 + 0.03 \times 2$
= 0.1 kmol

Selectivity of the catalyst = 0.95/0.1 = 9.5

Combustion of a fuel is another oxidation reaction in which oxygen (i.e., air) is fed in excess to achieve complete combustion. Fuel is thus the limiting reactant. Oxygen is supplied in excess over the stoichiometric requirement in the form of air. Knowing the flue gas and fuel analyses, it is possible to calculate the excess air.

Example 2.7 Flue gas mixture from a waste incinerator is analysed and found to contain 12% CO_2, 5.5% O_2 and rest N_2 (by mole) on dry basis. Assuming complete combustion, calculate the excess air and C : H mass ratio in fuel.

Solution

Basis: 100 kmol dry flue gases

Table 2.3 Flue Gas Analysis on Dry Basis

Component	kmol	O_2 accounted kmol
CO_2	12	12
O_2	5.5	5.5
N_2	82.5	–
Total	100.0	17.5

Assume negligible nitrogen in the fuel (i.e., waste).

O_2 supplied in the form of air = $\frac{21}{79} \times 82.5 = 21.93$ kmol

O_2 consumed in reaction with hydrogen = 21.93 – 17.5
= 4.43 kmol

Hydrogen reacted = 4.43×2
= 8.86 kmol

Carbon in waste = $12 \times 12 = 144$ kg

Hydrogen in waste = $8.86 \times 2 = 17.72$ kg

C : H ratio in waste = $\frac{144}{17.72}$
= 8.13:1 by mass

Total oxygen consumed for combustion (or stoichiometric oxygen requirement)
= 12 + 4.43
= 16.43 kmol

Excess oxygen or air = (5.5/16.43) 100 = 33.48%

2.3.3 Recycling and Bypassing Operations

Recycle stream is a process stream from exit or downstream from a unit back to the inlet or upstream of the same unit. Nature is a classical example in which nitrogen and oxygen are recycled. Animals inhale oxygen and exhale carbon dioxide. The latter is used by plants for manufacture of food in presence of sunlight. In this photosynthesis reaction, oxygen is released. In unit operations and in chemical reactions, recycling operations are performed for a variety of reasons. One of the chief reasons of recycling is to maximize the utilization of reactant(s) in the reaction. Hydrogen is used in excess in many reactions. After the reaction, excess hydrogen is separated and recycled from the separation unit back to the reaction unit. For recovery of volatile organic compounds (VOC) from vent gases, carbon adsorption units are commonly employed. Nitrogen is used for regeneration of the carbon bed from which VOC is separated. After separation of most VOC, nitrogen is recycled.

Other reasons for recycle are utilization of heat from the outgoing stream, to improve the performance of an equipment, to improve selectivity of a product, to improve safety of the process, to minimize waste generation, etc.

Chemical engineers are familiar with reflux in a distillation column. It is basically a recycle stream to maintain specific quality of the top product. In Chapter 8, number of examples are given for calculating reflux ratio which represent material balance in a unit operation.

In a loop reactor, fraction of product stream (after cooling) is recycled back to the reactor to control the exhothermicity of reaction.

2.4 ENERGY BALANCES

It is known that most physical and chemical changes are accompanied by enthalpy changes. It is therefore necessary to study the enthalpy balances.

Energy exists in different forms such as potential energy, kinetic energy, thermal energy, electrical energy, etc. In SI units, the unit of all types of energy is Joule (represented as J). Conversion of one form of energy into another is feasible. However, total energy of the universe is constant.

In this section, discussions will be limited to thermal energy and the terms energy, enthaply and heat are essentially used for thermal energy. While enthalpy of a substance is associated with its state (i.e., pressure and temperature), the term heat is used for the enthalpy in transit. In physical and chemical changes, flow rate of heat (in W in SI units) is of interest to process engineers.

2.4.1 Sensible Heat Changes

Sensible heat changes in solid, liquid or gas means change in temperature of the substance without phase change. Increase in temperature is termed as *heating* while a decrease is termed as *cooling*. For evaluation of the heat change, mass, temperatures and heat capacity are required.

For any substance, heat capacity is defined as the heat required to effect the temperature change by 1°C of 1 kg substance. Thus, units of heat capacity are kJ/(kg · °C). If the heat capacity of one kmol substance is specified, it is known as *molar heat capacity* and its units are kJ/(kmol · °C).

For gases, heat capacity depends on the route, followed for changing the temperature. One route is known as *constant pressure path* while another route is *constant volume path*. Majority of the processes follow constant pressure route and the heat capacity for the route is denoted by C_{mp} and has units kJ/(kmol · °C). This quantity is dependent on pressure and temperature. At 1 atm pressure, it is denoted by symbol C°_{mp}. A number of equations are developed, offering a relationship of C°_{mp} with temperature at 101.325 kPa. Among these, polynomial forms are used extensively.

$$C^{\circ}_{mp} = a + bT + cT^2 \tag{2.7}$$

or

$$C^{\circ}_{mp} = a + bT + cT^2 + dT^3 \tag{2.8}$$

where, a, b, c and d are constants (separate values for both equations) and T is temperature in K.

Values of C°_{mp}, derived from the equations, are accurate at 1 atm but they can be safely used for subatmospheric pressure and up to about 10 bar a. Correlations, graph and tables are available in literature for prediction of C_{mp} at high pressures (Ref. 1, 3, 4).

For a mixture of ideal gases, sensible heat changes are calculated by calculating heat capacity of the mixture, using the following equation:

$$C^{\circ}_{mp\ \mathrm{mix}} = \Sigma\, y_i \cdot C^{\circ}_{mpi}$$

where, $C^{\circ}_{mp\ \mathrm{mix}}$ = heat capacity of gas mixture, kJ/(kmol · °C)
y_i = mole fraction of ith component
C°_{mpi} = heat capacity of ith component of gas mixture, kJ/(kmol · °C)

This equation is strictly valid for an ideal gas mixture but can be used for most practical purpose up to moderate pressures.

In case of liquids, heat capacities at constant pressure and at constant volume are nearly equal. Also variation of the heat capacity with pressure is insignificant for most liquids. Symbol for heat capacity of liquid is C_l. Polynomial equations are also developed for correlating C_l with T. For aqueous solutions, liquid mixtures, petroleum fluids, etc., graphical and tabulated data are available in literature. Heat capacity of an immiscible liquid mixture is given by

$$C_{lm\ \mathrm{mix}} = \Sigma\, x_i \cdot C_{lm\,i} \tag{2.9}$$

where, $C_{lm\ \mathrm{mix}}$ = heat capacity of liquid mixture, kJ/(kmol · °C)
x_i = mole fraction of ith component
$C_{lm\,i}$ = heat capacity of ith component of liquid mixture, kJ/(kmol · °C)

Heat capacity of solids (C_s) are lower than that of liquids and increase with the increase in the temperature. However, variation of C_s with respect to T is generally insignificant.

2.4.2 Heat Changes During Phase Change

A study of p–t diagram (Fig. 7.28 and Fig. 7.29) reveals interesting facts about phase changes. Above triple point, there are two phase changes. When a solid is melted above its freezing point, latent enthalpy (or heat) of fusion is to be supplied. Ice melts at 0.01°C at standard atmospheric pressure (101.325 kPa). Latent enthalpy of fusion at this temperature is 339.4 kJ/kg. In general, melting point of a pure fluid increases with increase in pressure while latent enthalpy of fusion decreases.

When heat is supplied to convert liquid to vapour at a constant temperature, it is called that *latent enthalpy or latent* (*heat*) *of vaporization* (λ). Water evaporates at

100°C at 101.325 kPa when heat equivalent to 2256.9 kJ/kg is supplied. Saturation temperature of a pure fluid increases with increase in pressure while latent enthalpy of vaporization decreases and becomes zero at critical point.

Below triple point, a fluid sublimes from solid to vapour. Heat, supplied to change the solid phase to vapour phase of a pure substance, is called the *latent enthalpy* (*heat*) *of sublimation*. Sublimation temperature and latent enthalpy of sublimation decreases with decrease in pressure below triple point. Carbon dioxide sublimes at –78.46°C at 101.325 kPa and latent heat of sublimation is 571.0 kJ/kg. At triple point, latent heat of sublimation can be considered a total of latent heat of fusion and latent heat of vaporization. Triple point of carbon dioxide is –56.56°C at 5.18 bar. At this temperature, latent heat of fusion is 197.22 kJ/kg and latent heat of vaporization is 350.38 kJ/kg. Thus latent heat of sublimation at this temperature is 547.60 kJ/kg.

Above critical point, again sublimation of solid to vapour is experienced as there is no liquid phase. Critical temperature and critical pressure of water are 374.15°C and 221.2 bar, respectively. Water sublimes at –5°C at 600 bar and latent heat of sublimation is 319.92 kJ/kg at this temperature.

Various correlations are available in literature for prediction of latent heat of fusion and vaporization. However, latent heat of vaporization (λ) is most useful to a process engineer. Tables of thermodynamic properties (Ref. 2) are available in literature for large number of compounds. At standard atmospheric pressure, Riedel equation is found to give latent heat of vaporization within 5% accuracy:

$$\frac{\lambda}{RT_B} = \frac{1.092\,(\ln p_c - 5.6182)}{0.930 - T_{Br}} \tag{2.10}$$

where, λ = latent heat of vaporization at T_B, kJ/kmol
p_c = critical pressure, kPa
T_B = normal boiling point, K
T_{Br} = reduced temperature at $T_B = T_B/T_c$
R = 8.314 472 kJ/(kmol · K)

Knowing latent heat of vaporization at one temperature, it can be calculated at another temperature, using the following Watson equation:

$$\frac{\lambda_1}{\lambda_2} = \left[\frac{T_c - T_1}{T_c - T_2}\right]^n \tag{2.11}$$

where, λ_1 and λ_2 refer to latent heat of vaporization at temperatures T_1 and T_2 (in K) respectively, T_c is critical temperature (in K) and n is characteristic constant, having a value of 0.38. Watson equation is simple and reliable in most cases.

For heat changes calculations during phase change, latent heat of appropriate phase change is to be taken into account.

2.5 ENTHALPY CHANGES ACCOMPANYING CHEMICAL REACTIONS

When any chemical reaction takes place, enthalpy changes are encountered. This is known as *heat of reaction*. For combustion reactions, it is termed as *heat of combustion*. For calculation of heat of reaction, heat of formation data are required.

Standard heat of formation is defined as the isothermal enthalpy change in a synthesis reaction from the elements in their standard states. Standard heat of formation, denoted by ΔH_f° of large number of compounds, is available in literature[1] in tabular form. Standard states of compounds are pure crystalline solid at 25°C (298.15 K) and for pure liquid and pure gas are 1 bar a and 25°C (298.15 K). Since combustion reactions are easy for experiments, standard heat of combustion (ΔH_c°) data so obtained are used to calculate the standard heat of formation.

Standard heat of formation data are obtained for isothermal conditions. It varies with temperature. Empirical relation of ΔH_f with T is a polynominal equation, similar to that for heat capacity of gas [Eq. (2.7) or Eq. (2.8)].

Consider a chemical reaction between A and B to produce C and D. Stoichiometric relation can be written as

$$n_A\,\mathrm{A} + n_B\,\mathrm{B} = n_C\,\mathrm{C} + n_D\,\mathrm{D}$$

where, n_i = number of moles of ith component. Standard heat of reaction (ΔH_r°) for the above reaction can be calculated by the following equation:

$$\Delta H_r^\circ = \Sigma\,(n_i \cdot \Delta H_{fi}^\circ)_{\text{Products}} - \Sigma\,(n_i \cdot \Delta H_{fi}^\circ)_{\text{Reactants}}$$

where, ΔH_{fi}° = Standard heat of formation of ith component

Since ΔH_{fi}° will be at 25°C, ΔH_r° will be also at 25°C. However, if ΔH_{fi} at some another temperature is taken (but all at one specific temperature), ΔH_r will correspond to that temperature.

Enthalpy is known to be a state function. Hence, change in enthalpy taking place while going from one state to another state (either in physical operations or in chemical reactions) is independent of the pathway. This principal is known as *Hess's Law*.

If heat of reaction is positive, the reaction is said to be endothermic while if the heat of reaction is negative, the reaction is termed exothermic.

Example 2.8 Standard heat of formation of liquid oleic acid ($C_{18}H_{34}O_2$) is –802.9 kJ/mol at 25°C. Calculate its gross calorific value at 25°C.

Solution

Basis: 1 mole liquid oleic acid

Gross calorific value or higher heating value is the heat liberated when a mole of the combustible compound is burnt with stoichiometric amount of pure oxygen at 25°C, product carbon dioxide is in gaseous form while product water is in liquid form. Combustion reaction of oleic acid can be written as

	$C_{18}H_{34}O_2(l)$	$+\ 25.5\ O_2(g)$	=	$18\ CO_2(g)$	+	$17\ H_2O(l)$
ΔH_{fi}°	–802.9	0		$18 \times (-393.51)$		$17 \times (-285.83)$

Standard heat of formation data for CO_2 and H_2O are taken from Ref. 1.

$$\Delta H_r^\circ = 18\,(-393.51) + 17\,(-285.83) - (-802.9)$$
$$= -11\,139.4 \text{ kJ/mol of liquid oleic acid}$$

Literature reports ΔH_r at 20°C for liquid oleic acid as –11 154 kJ/mol.

Example 2.9 Natural gas having following molar composition is considered as a raw material for manufacture of ammonia.

CH_4: 78.5%, C_2H_6: 8.2%, C_3H_8: 4.1%, i-C_4H_{10}: 0.6%, n-C_4H_{10}: 0.4% and CO_2: 8.2%

Hydrogen is produced by reforming of hydrocarbons with steam as shown below.

$$C_nH_{2n+2} + 2n\ H_2O = n\,CO_2 + (3n + 1)\ H_2$$

Dry air, having molar composition 78% N_2, 21% O_2 and 1% Ar, is used for supplying nitrogen. Oxygen of air will react with H_2 as under.

$$2\ H_2 + O_2 = 2\ H_2O$$

(a) Assume 100% conversions of all reactions, 100% removal of ammonia from the reaction mass and venting of Ar to atmosphere. Calculate stoichiometric requirement of natural gas per tonne of ammonia.

(b) Considering net calorific values, of hydrocarbons, calculate specific energy requirement per tonne of ammonia.

Solution

Basis: 1000 kg ammonia production

Molar mass of ammonia = 17.0305

Molar production of ammonia = 1000/17.0305

= 58.7182 kmol

Reaction of ammonia manufacture:

$$3\ H_2 + N_2 = 2\ NH_3$$

Stoichiometric H_2 requirement = (3/2) 58.7182

= 88.0773 kmol

Stoichiometric N_2 requirement = (1/2) 58.7182

= 29.3591 kmol

Supply of air brings 21 O_2 kmol of 78 kmol N_2.

O_2 equivalent to N_2 requirement = (21/78) 29.3591

= 7.9044 kmol

H_2 consumed by O_2 = 2×7.9044

= 15.8088 kmol

Total H_2 required = 88.0773 + 15.8088

= 103.8861 kmol

H_2 produced by reforming reaction of natural gas

$$= 0.785 \times 4 + 0.082 \times 7 + 0.041 \times 10 + 0.01 \times 13$$

= 4.254 kmol/kmol

Natural gas requirement for 1000 kg ammonia

= 103.8861/4.254

= 24.4208 kmol

$\equiv$ 547.37 Nm^3 Ans. (a)

Table 2.4 Composition of Natural Gas

Component	Molar mass (M)	kmol n_i	NCV* (ΔH_c°) kJ/mol	$n_i \cdot \text{NCV}_i$ kJ
CH_4	16.0425	0.785	802.62	630.057
C_2H_6	30.0690	0.082	1428.64	117.148
C_3H_8	44.0956	0.041	2043.11	83.768
i-C_4H_{10}	58.1222	0.006	2648.12	15.889
n-C_4H_{10}	58.1222	0.004	2657.32	10.629
CO_2	44.0095	0.082	–	–
Total		1.000		857.491

*Ref. 1.

$$\text{NCV of natural gas} = 857.491 \text{ kJ/mol}$$

$$\text{Specific energy requirement} = 857.491 \times 1000 \times 24.4208/10^6$$

$$= 20.941 \text{ GJ/t ammonia} \qquad \text{Ans. (b)}$$

Example 2.10 In the manufacture of nitric acid, ammonia is oxidized to nitric oxide on a Pt/Rh gauge (as catalyst) as per the following reactions:

$$4\ NH_3(g) + 5\ O_2(g) = 4\ NO(g) + 6\ H_2O(g) \qquad (a)$$

$$4\ NH_3(g) + 3\ O_2(g) = 2\ N_2(g) + 6\ H_2O(g) \qquad (b)$$

$$4\ NH_3(g) + 4\ O_2(g) = 2\ N_2O(g) + 6\ H_2O(g) \qquad (c)$$

Liquid ammonia at atmospheric pressure and –33.33°C is pumped to 9.0 bar a. It is first vaporized with cooling water and subsequently superheated to desired temperature using low pressure saturated steam at 5 bar a. Oxygen in the form of air is supplied. Air is compressed to 9.3 bar a and 130°C in a two–stage turbo compressor, driven by a steam turbine. Further it is heated to 260°C with the reactor exit stream. Superheated ammonia at the rate of 3266 Nm^3/h and hot air at the rate of 29 394 Nm^3/h are fed to the reactor. Ambient air has 0.016 kg moisture/kg dry air. In the reactor, ammonia is completely consumed. Yields of NO, N_2 and N_2O are 95%, 2% and 3%, based on ammonia conversion, respectively. Reactor exit gas stream temperature is observed to be 915°C at 7.5 bar g. Carry out the material and energy balances of the reactor system and preheating equipments.

The process flow diagram is given as Fig. 2.1.

Solution

Basis: Ammonia flow rate to reactor, q_{am} = 3266 Nm^3/h

Molar flow rate of ammonia, q_{am} = 3266/22.414

= 145.71 kmol/h

Volumetric flow rate of moist air, q_a = 29 394 Nm^3/h

Molar flow rate of moist air, q_a = 29 394/22.414

= 1311.44 kmol/h

Assume dry air molar composition to be 21% O_2, 1% Ar and 78% N_2.

Fig. 2.1 *Manufacture of Nitric Acid*

Average molar mass of dry air $= 0.21 \times 31.9988 + 0.01 \times 39.948 + 0.78 \times 28.0134$
$= 28.9697$ (referred in Example 2.4)
Moisture in dry air $= 0.016$ kg/kg dry air
$= 0.016 \times 28.9697/18.0153$
$= 0.0257$ kmol/kmol dry air
Moist air $= 1.0257$ kmol/kmol dry air
Moisture, entering with air $= 1311.44 \times 0.0257/1.0257$
$= 32.86$ kmol/h
Dry air flow rate to reactor $= 1311.44 - 32.86$
$= 1278.58$ kmol/h
O_2 supply to reactor $= 1278.58 \times 0.21 = 268.50$ kmol/h
Argon supply to reactor $= 1278.58 \times 0.01 = 12.79$ kmol/h
N_2 supply to reactor $= 1278.58 - 268.50 - 12.79$
$= 997.29$ kmol/h

Table 2.5 Thermodynamic Data[1]

Component	State	Molar mass kg/kmol	ΔH_f° kJ/mol	Heat capacity equation constants			
				a_i	$b_i \times 10^3$	$c_i \times 10^6$	$d_i \times 10^9$
NH_3	gas	17.0305	–45.94	25.6503	33.4806	0.3518	–3.0832
O_2	gas	31.9988	0	26.0257	11.7551	–2.3426	–0.5623
N_2	gas	28.0134	0	29.5909	–5.141	13.1829	–4.968
Ar	gas	39.948	0	20.7723	–	–	–
NO	gas	30.0061	90.25	29.4867	–2.0524	11.3379	–4.8195
N_2O	gas	44.0128	82.05	23.2082	63.6695	–42.3402	10.4061
NO_2	gas	46.0055	33.18	23.5804	53.5944	–31.4901	6.5394
H_2O	gas	18.0153	–241.82	32.4921	0.0796	13.2107	–4.5474
H_2O	liquid	18.0153	–285.83				
HNO_3	liquid	63.0128	–174.10				

Conversion = 100% of NH_3 and Yield of NO = 95%
NH_3 converted to NO $= 145.71 \times 0.95$
$= 138.42$ kmol/h
NO produced $= 138.42$ kmol/h
NH_3 converted N_2 $= 145.71 \times 0.02$
$= 2.91$ kmol/h
N_2 produced $= 2.91 \times 2/4$
$= 1.457$ kmol/h
NH_3 converted to N_2O $= 145.71 - 138.42 - 2.91$
$= 4.38$ kmol/h
N_2O produced $= 4.38 \times 2/4$
$= 2.19$ kmol/h
Total O_2 consumed $= 138.42\ (5/4) + 2.91\ (3/4) + 4.38\ (4/4)$
$= 173.025 + 2.183 + 4.38$
$= 179.588$ kmol/h

O_2 in reactor exit stream = 268.50 – 179.588
= 88.912 kmol/h
Total H_2O produced = 145.71 × 6/4
= 218.565 kmol/h
Total H_2O in reactor exit stream = 218.565 + 32.86
= 251.425 kmol/h
Total N_2 in reactor exit stream = 1.457 + 997.29
= 998.747 kmol/h

Table 2.6 Reactor Ingoing Stream

Component	kmol/h $\dot{n}_i$	mole % (wet)	kg/h $\dot{m}_i$	Heat capacity equation constants			
				$\dot{n}_i \cdot a_i$	$\dot{n}_i \cdot b_i \times 10^3$	$\dot{n}_i \cdot c_i \times 10^6$	$\dot{n}_i \cdot d_i \times 10^9$
NH_3	145.71	10.00	2 481.51	3 737.5	4878.5	51.3	–449.3
N_2	997.29	68.44	27 937.75	29 510.7	–5127.1	13 147.2	–4954.5
O_2	268.50	18.43	8 591.68	6 987.9	3156.2	–629.0	–151.0
Ar	12.79	0.87	510.93	265.7	–	–	–
H_2O	32.86	2.26	591.98	1 067.7	2.6	434.1	–149.4
Total	1457.15	100.00	40 113.85	41 569.5	2910.2	13 003.6	–5704.2

Average molar mass of reactor ingoing stream
= 40 113.85//1457.15 = 27.53 kg/kmol

Table 2.7 Reactor Exit Stream

Component	kmol/h $\dot{n}_i$	mole % (wet)	kg/h $\dot{m}_i$	Heat capacity equation constants			
				$\dot{n}_i \cdot a_i$	$\dot{n}_i \cdot b_i \times 10^3$	$\dot{n}_i \cdot c_i \times \times 10^6$	$\dot{n}_i \cdot d_i \times 10^9$
N_2	998.747	66.92	27 978.30	29 553.8	–5134.6	13 166.4	–4961.8
O_2	88.912	5.96	2 845.08	2 314.0	1045.2	–208.3	–50.0
Ar	12.790	0.85	510.93	265.7	–	–	–
NO	138.420	9.27	4 153.44	4 081.5	–284.1	1569.4	–667.1
N_2O	2.190	0.15	96.39	50.8	139.4	–92.7	22.8
H_2O	251.425	16.85	4 529.50	8 169.3	20.0	3321.5	–1143.4
Total	1492.484	100.00	40 113.64	44 435.1	–4214.1	17 756.3	–6799.5

Average molar mass of reactor exit stream
= 40 113.64/1492.484 = 26.88 kg/kmol

It may be noted that mass flow rates of ingoing and outcoming streams are *equal* which should be checked during every material balance calculations. However, molar flow rates are different.

Energy Balance

For carrying out energy balance calculations, reference (or base) temperature need to be fixed. Since standard heat of formation data are based on 25°C, selection of 25°C as the reference temperature is logical.

$T_0 = 25 + 273.15 = 298.15$ K

Reactor exit gas temperature is desired at 915°C (1188.15 K).

Heat flow rate of reactor exit stream

$$\phi_8 = \int_{298.15}^{1188.15} (44\,435{\cdot}1 - 4214{\cdot}1 \times 10^{-3}\,T + 17\,756{\cdot}3 \times 10^{-6}\,T^2 - 6799.5 \times 10^{-9}\,T^3)\,\mathrm{d}T$$

$= 43\,156\,523$ kJ/h $\equiv 11\,987.92$ kW

Standard Heats of Reactions

For reaction (a):

$\Delta H^\circ_{r1} = [4(90.25) + 6(-241.82)] - [4(-45.94) + 5(0)]$

$= 361 - 1450.92 + 183.76$

$= -906.16$ kJ/4 mol NH_3 reacted

$\equiv -226.54$ kJ/mol NH_3 reacted (exothermic)

For reaction (b):

$\Delta H^\circ_{r2} = [2(0) + 6\,(-241.82)] - [4\,(-45.94) + 3(0)]$

$= -1450.92 + 183.76$

$= -1267.16$ kJ/4 mol NH_3 reacted

$\equiv -316.79$ kJ/mol NH_3 reacted (exothermic)

For reaction (c):

$\Delta H^\circ_{r3} = [2\,(82.05) + 6\,(-241.82)] - [4\,(-45.94) + 4(0)]$

$= 164.1 - 1450.92 + 183.76$

$= -1103.06$ kJ/4 mol NH_3 reacted

$\equiv -275.765$ kJ/mol NH_3 reacted (exothermic)

Total heat liberated during reaction,

$\phi_7 = (226.54 \times 138.42 + 316.79 \times 2.91 + 275.765 \times 4.38)\,1000$

$= 33\,487\,377$ kJ/h $\equiv 9302.05$ kW

Heat input to reactor (i.e., of the reactants' stream)

$\phi_6 = 43\,156\,523 - 33\,487\,377$

$= 9\,669\,146$ kJ/h

$\equiv 2685.87$ kW

Let temperature of reactants' stream be T_4.

$$\phi_6 = \int_{298.15}^{T_4} (41\,569.5 + 2910.2 \times 10^{-3}\,T + 13\,003.6 \times 10^{-6}\,T^2 - 5704.2 \times 10^{-9}\,T^3)\,\mathrm{d}T$$

$= 9669\,146$ kJ/h

Solving by trial and error or by Mathcad, $T_4 = 515.22$ K or $t_4 = 242.07$°C

Among the two reactants, air is introduced at 260°C or 533.15 K (T_3).

Heat input of air,

Heat capacity equation constants for air are drawn from Table 2.6.

$$\phi_5 = \int_{298.15}^{533.15} (37\,832 - 1968.3 \times 10^{-3}\,T + 12\,952.3 \times 10^{-6}\,T^2 - 5254.9 \times 10^{-9}\,T^3)\,\mathrm{d}T$$

$= 9142\,364$ kJ/h $\equiv 2539.55$ kW

Heat input by superheated ammonia,

$\phi_4 = 9669\,146 - 9142\,364$

$= 526\,782$ kJ/h $\equiv 146.33$ kW

If temperature of superheated ammonia is T_2,

$$\phi_4 = \int_{298.15}^{T_2} (3737.5 + 4878.5 \times 10^{-3}\,T + 51.3 \times 10^{-6}\,T^2 - 449.3 \times 10^{-9}\,T^3)\,\mathrm{d}T$$

$= 526\,782$

Solving by Mathcad,

$T_2 = 395.41$ K or $t_2 = 122.26$°C

Ammonia is fed at –33.33°C (saturation temperature at 101.325 kPa) as liquid. Assume that in the evaporator, liquid ammonia is vaporized and heated at 9.0 bar a to its saturation temperature of 21.5°C with cooling water.

$T_1 = 21.5 + 273.15 = 294.65$ K

From ammonia property tables (Ref. 2):

Enthalpy of liquid ammonia, $h = 49.08$ kJ/kg at –33.33°C

Enthalpy of vapour ammonia at 9 bar a and 21.5°C, $H_1 = 1480.78$ kJ/kg

Note that the values of both the above enthalpies are based on reference temperature of 0°C in ammonia tables which is different from original reference temperature of 25°C. Nevertheless, since difference in two enthalpy values only is required, it does not matter.

Heat duty of ammonia evaporator,

$\phi_1 = 145.71 \times 17.0305\,(1480.78 - 49.08)$

$= 3552\,784$ kJ/h

$\equiv 986.88$ kW

Cooling water at 32°C is used to evaporate liquid ammonia. Assume a drop in cooling water temperature by 5°C.

Cooling water flow, $\dot{m}_{CW} = \dfrac{3552\,784}{5 \times 4.1868}$

$= 169\,714$ kg/h

$\equiv 169.7$ m^3/h

Enthalpy of ammonia gas at 9.0 bar a and 122.26°C, $H_2 = 1739.55$ kJ/kg

Heat duty of superheater, $\phi_2 = 145.71 \times 17.0305\,(1739.55 - 1480.18)$

$= 643\,630$ kJ/h

$\equiv 178.79$ kW

If heat capacity data (at 1 atm) for ammonia, are used for calculations of ϕ_2,

$$\phi_2 = \int_{294.65}^{395.41} (3737.5 + 4878.5 \times 10^{-3}\,T + 51.3 \times 10^{-6}\,T^2 - 449.3 \times 10^{-9}\,T^3)\,\mathrm{d}T$$

$= 544\,914$ kJ/h

$\equiv 151.37$ kW

$$\text{Error} = \frac{(643\,630 - 544\,914)}{643\,630} \times 100$$

$= 15.34\%$ – significant

Error indicates justification of tabular data, particularly at high pressures.

Saturated steam at 5 bar a is used for superheating ammonia vapour from 21.5°C to 122.26°C.

Latent heat of vaporization of steam at 5 bar a, $\lambda = 2107.4$ kJ/kg (Ref. 2)

Steam consumption in superheater,

$$\dot{m}_s = \frac{643\,630}{2107.4} = 305.4 \text{ kg/h}$$

Heat duty of air preheater,

$$\phi_3 = \int_{403.15}^{533.15} (37\,832 - 1968.3 \times 10^{-3}\,T + 12\,952.3 \times 10^{-6}\,T^2 - 5254.9 \times 10^{-9}\,T^3)\,\mathrm{d}T$$

$= 5098\,328$ kJ/h $\equiv 1366.16$ kW

2.6 HEAT CHANGES IN MIXING PROCESSES

When a solid or gas is dissolved in a solvent, heat change takes place. This is known as *heat of solution*. Also when two liquids are mixed, a heat change takes place which is termed as *heat of mixing*. These heat changes are measured at constant temperature, say at 25°C (298.15 K) and at standard atmospheric pressure. Units of the heat changes are kJ/mol solute, kJ/kg solution, etc., depending on user's convenience.

When solids like caustic soda, caustic potash, sodium carbonate, etc., are dissolved in water, heat is evolved. Similarly, when acids are mixed (e.g., a mixture of $HNO_3 + H_2SO_4 + H_2O$), heat is released. Dissolution of ammonia or formaldehyde in water is exothermic. These heat changes should be considered while making heat balance calculations.

Heat of mixing data are extensively covered in many handbooks. Heat of formation data for large number of compounds and their solutions are published by National Institute of Standards and Technology, USA which permits heat of mixing calculations. Reference 1 may be consulted for more discussions on the subject.

Following example will illustrate the method of calculations relating to heat of absorption in gas–liquid system.

Example 2.11 Refer to Example 2.10. Reactor exit stream is cooled in various heat exchangers to 40°C and introduced in an absorber along with the condensate (formed in the coolers), operating at 1.5 bar g. Secondary air at the rate of 8145 Nm^3/h is also introduced at the bottom of the absorber at 40°C which converts NO to NO_2. Fresh demineralized water is introduced from the top at 25°C which scrubs NO_2 to produce HNO_3. Absorption of NO_2 in water is exothermic and therefore cooling coils are provided in the absorber. Tail gas, leaving the absorber at

0.1 bar g and 50°C, is found to contain 0.2% NO (on dry basis). Aqueous 58% acid (by mass) leaves absorber at 50°C from the bottom. Make material and energy balance of the absorber. Find cooling water requirement for the absorption system assuming availability of cooling water at 32°C and a rise of 5°C.

Solution

Basis: Same as that of Example 2.10.

Pressure of gas at the inlet of absorber, = 1.5 bar g
= 251.325 kPa a

Vapour pressure of water at 40°C = 7.375 kPa

Refer to Table 2.6.

Moisture in ingoing gas mixture $= \dfrac{7.375(1492.484 - 251.425)}{(251.325 - 7.375)}$

= 37.519 kmol/h at 40°C

≡ 675.92 kg/h

Liquid water entering absorber = 251.425 – 37.519

= 213.906 kmol/h at 40°C

≡ 3853.58 kg/h

Pressure of outgoing mixture = 0.1 bar g

= 111.325 kPa a

Vapour pressure of water at 50°C = 12.335 kPa

Water content of tail gas $= \dfrac{12 \cdot 335}{(111 \cdot 325 - 12 \cdot 335)}$

$= 0.1246 \dfrac{\text{kmol}}{\text{kmol dry gas}}$

Secondary air flow = 8145 Nm^3/h

Molar flow rate of wet air = 8145/22.414

= 363.4 kmol/h

Moisture, entering with air = 363.4 × 0.0257/1.0257

= 9.11 kmol/h

Dry secondary air flow rate = 363.4 – 9.11

= 354.29 kmol/h

O_2 entering the absorber = 354.29 × 0.21

= 74.40 kmol/h

Ar entering the absorber = 354.29 × 0.01

= 3.54 kmol/h

N_2 entering the absorber = 354.29 – 74.40 – 3.54

= 276.35 kmol/h

Table 2.8 Gas Stream Entering Absorber

Component	kmol/h $\dot{n}_i$	mole % (wet)	kg/h $\dot{m}_i$	Heat capacity equation constants			
				$\dot{n}_i \cdot a_i$	$\dot{n}_i \cdot b_i \times 10^3$	$\dot{n}_i \cdot c_i \times 10^6$	$\dot{n}_i \cdot d_i \times 10^9$
N_2	1275.097	77.66	35 719.80	37 731.3	–6555.3	16 809.5	–6334.7
O_2	163.312	9.95	5 225.79	4 250.3	1919.7	–382.6	–91.8
Ar	16.330	0.99	652.35	339.2			
NO	138.420	8.43	4 153.44	4 081.5	–284.1	1569.4	–667.1
N_2O	2.190	0.13	96.39	50.8	139.4	–92.7	22.8
H_2O	46.629	2.84	840.04	1515.1	3.7	616.0	–212.0
Total	1641.978	100.00	46 687.81	47 968.2	–4776.6	18 519.61	–7282.8

Average molar mass of gas mixture, entering absorber = $\dfrac{46\,687.81}{1641.978} = 28.43$

Reaction

$$2\ NO(g) + 1.5\ O_2(g) + H_2O(l) = 2\ HNO_3(l) \qquad \text{(d)}$$

Let $\dot{n}_1$ kmol/h NO is reacted by the above reaction.

$$\begin{aligned} O_2 \text{ consumed} &= 1.5\ \dot{n}_1/2 \\ &= 0.75\ \dot{n}_1 \text{ kmol/h} \\ \text{NO in tail gas} &= 138.42 - \dot{n}_1 \text{ kmol/h} \\ \text{Dry tail gas} &= 1641.978 - \dot{n}_1 - 0.75\ \dot{n}_1 - 46.629 \\ &= 1595.349 - 1.75\ \dot{n}_1 \text{ kmol/h} \end{aligned}$$

Tail gas contains 0.2% NO on dry basis.

$$\frac{(138 \cdot 42 - \dot{n}_1)\ 100}{(1595 \cdot 349 - 1.75\ \dot{n}_1)} = 0.2$$

$$\begin{aligned} \dot{n}_1 &= 135.70 \text{ kmol/h} \\ \text{Dry tail gas} &= 1595.349 - 1.75 \times 135.70 \\ &= 1357.874 \text{ kmol/h} \\ \text{Moisture in tail gas} &= 0.1246 \times 1357.874 \\ &= 169.191 \text{ kmol/h} \end{aligned}$$

Table 2.9 Absorber Exit (Tail Gas) Stream

Component	kmol/h $\dot{n}_i$	mole % (dry)	kg/h $\dot{m}_i$	Heat capacity equation constants			
				$\dot{n}_i \cdot a_i$	$\dot{n}_i \cdot b_i \times 10^3$	$\dot{n}_i \cdot c_i \times 10^6$	$\dot{n}_i \cdot d_i \times 10^9$
N_2	1275.097	93.90	35 719.80	37 731.3	–6555.3	16 809.5	–6334.7
O_2	61.537	4.54	1 969.11	1 602.9	724.0	–144.3	–34.6
Ar	16.330	1.20	652.35	339.2	—	—	—
NO	2.720	0.20	81.62	82.3	–5.7	31.6	–13.4
N_2O	2.190	0.16	96.39	50.8	139.4	–92.7	22.8
H_2O	169.191	—	3 048.03	5647.3	13.8	2296.1	–790.4
Total	1527.065	100.00	41 567.30	45 453.8	–5683.8	18 900.2	–7150.3

Average molar mass of exit gas stream = 41 567.3/1527.065 = 27.22 kg/kmol
Reference temperature, T_0 = 298.15 K
Heat input to absorber (of gas stream)

$$\phi_9 = \int_{298.15}^{313.15} (47\,968.2 - 4776.6 \times 10^{-3}\,T + 18\,519.6 \times 10^{-6}\,T^2 - 7282.8 \times 10^{-9}\,T^3)\,\mathrm{d}T$$

= 720 459 kJ/h
≡ 200.13 kW

Heat in tail gas stream

$$\phi_{10} = \int_{298.15}^{323.15} (45\,453.8 - 5683.8 \times 10^{-3}\,T + 18\,900.2 \times 10^{-6}\,T^2 - 7150.3 \times 10^{-9}\,T^3)\,\mathrm{d}T$$

= 1132 458 kJ/h
≡ 314.57 kW

Heat of reaction of Reaction (d):

ΔH_r° = 2 (–174.10) – [2 (90.25) + 1.5(0) + (–285.83)]
= –348.2 – 180.5 + 285.83
= –242.87 kJ/2 mol NO reacted
≡ –121.435 kJ/mol NO reacted (exothermic)

Total heat generated during reaction:

ϕ_{11} = 121.435 × 1000 × 135.7
= 16 478 730 kJ/h
≡ 4577.42 kW

Water evaporated in absorber = 169.191 – 46.629
= 122.562 kmol/h
≡ 2208 kg/h

Assume average temperature of 45°C in the absorber.
λ at 45°C = 2394.9 kJ/kg (Ref. 2)
Heat absorbed during evaporation,

ϕ_{12} = 2208 × 2394.9
= 5287 939 kJ/h ≡ 1468.9 kW

Total HNO_3 produced = 135.7 kmol/h
= 8549.1 kg/h (100%)

Strength of aqueous acid at the bottom of the absorber = 58%

Aqueous acid produced $= \dfrac{8549.1}{0.58}$
= 14 739.8 kg/h

Water in the acid = 14 739.8 – 8549.1
= 6190.7 kg/h

Fresh demineralized water input
= 6190.7 + (135.7/2) 18.0153 – (3853.58 + 840.04 – 3048.03)
= 5767.4 kg/h
≡ 5.76 m^3/h

Since demineralized water is added at 25°C, its heat input is zero.

$$\phi_{13} = 0$$

When HNO_3 dissolves in water, heat change takes place.

ΔH_f° of 58% HNO_3 solution = –197.0 kJ/mol HNO_3

ΔH_f° of 100% $HNO_3(l)$ = –174.2 kJ/mol HNO_3

Heat change = –197.0 – (–174.2)

= –22.8 kJ/mol HNO_3 (exothermic)

Heat evolved by dissolution of HNO_3 in water at 25°C:

$\phi_{14} = 22.8 \times 1000 \times 135.7$

= 3093 960 kJ/h

≡ 859.43 kW

Heat capacity of 58% HNO_3 = 2.64 kJ/(kg · K)

Enthalpy of liquid 58% HNO_3 over 298.15 K

= 2.64 (313.15 – 298.15)

= 39.6 kJ/kg at 313.15 K

Heat of 58% acid at 40°C over 25°C

$\phi_{15} = 8549.1 \times 39.6$

= 338 544 kJ/h

≡ 94.04 kW

Heat of secondary air at 40°C

$$\phi_{16} = \left(\frac{8145}{29\,394}\right)\int_{298.15}^{313.15} (37\,832 - 1968.3 \times 10^{-3}\,T + 129\,523 \times 10^{-6}\,T^2 - 5254.9 \times 10^{-9}\,T^3)\,dT$$

$$= \frac{8145 \times 574\,358}{29\,394} = 159\,153 \text{ kJ/h}$$

≡ 44.21 kW

Enthalpy of condensate, entering absorber:

$\phi_{17} = 3853.58\ (40 - 25)\ 4.1868$

= 242 013 kJ/h

≡ 67.23 kW

Heat to be removed by cooling water

$= \phi_9 + \phi_{11} + \phi_{13} + \phi_{14} + \phi_{16} + \phi_{17} - \phi_{10} - \phi_{12} - \phi_{15}$

= 200.13 + 4577.42 + 0 + 859.43 + 44.21 + 67.23 – 314.57 – 1468.9 – 94.04

= 3964.95 kW

Cooling water is supplied to internal coils at 32°C and leaves it at 37°C.

Required cooling water flow rate, $\dot{m}_{CW} = \dfrac{3964.95 \times 3600}{5 \times 4.1868}$

= 681 849 kg/h

≡ 681.8 m^3/h

An example relating to absorption of formaldehyde in water to produce 37% aqueous solution is solved as Example 9.6 in Chapter 9. Both these examples refer to absorption with heat of absorption.

EXERCISES

2.1 A cylinder is filled with 6 Nm^3 hydrogen. Its pressure and temperature are 150 bar g and 35°C. If the actual specific volume of hydrogen at these conditions is 0.1847 m^3/kmol, what is the geometric capacity of the cylinder? Instead of using actual specific volume data, if ideal gas law is used for calculations, what will be the % error?

2.2 The chloride of some element contains 90.8% chlorine (by mass). What is the formula of the chloride compound?

2.3 A mixture, amounting to 18.3 kg of a trivalent metal and its oxide, is dissolved in 32.0 kg sulphuric acid having 98% strength. The mixture reacts with stoichiometric amount of sulphuric acid and forms metal sulphate. There is no excess sulphuric acid leftover. Calculate the composition of the mixture.

2.4 Complete combustion of an alkane produces water vapour, equivalent to the oxygen consumed (on mole basis). Find the alkane.

2.5 Nitration product of a hydroxybenzene [$C_6H_{(6-a)}(OH)_a$] contains 49% oxygen by mass. Determine the structure of the compound.

2.6 Reduction of nitrobenzene is to be carried out in a batch reactor. At the end of the reaction, aniline is steam distilled and collected in a receiver at 40°C. Distilled mass is separated in two layers. Top layer is decanted in another vessel and measured to be 910.2 kg. Bottom layer is measured to be 1089.8 kg. It is known from the literature[5] that top aniline rich layer contains 94.3% aniline while the bottom aqueous layer contains 3.83% aniline by mass at 40°C.

(a) Calculate total aniline distilled.

(b) Assume that all aniline, produced during reaction, is distilled out. If 1200 kg nitrobenzene was charged to the reactor and its conversion is 100%, what is the yield of aniline?

2.7 A wet scrubber is deployed to capture suspended particles from a gas stream. For this purpose water is circulated in the scrubber in countercurrent direction to gas stream which scrubs the suspended particles. A finite purge stream is maintained to control accumulation of solids in the scrubber.

A wet scrubber is to be designed for treating 50 000 Am^3*/h at near room temperature with a particulate loading of 1830 mg/Am^3. Assume that the scrubber particulate removal efficiency of 95% and the maximum suspended solids level, desirable in the scrubber, is 2% by mass. Use a liquid to gas ratio of 1.1 L/Am^3. Calculate the liquid purge rate and required recirculation pump flow rate for the scrubber system. Assume specific gravity of the circulating solution to be 1.02.

2.8 Natural gas, specified in Example 2.9, is considered as a raw material for the manufacture of methanol. H_2 and CO_2 produced by reforming with steam are reacted at specified conditions in a converter.

$$CO_2 + 3\,H_2 = CH_3OH + H_2O$$

(a) Consider 100% conversion of all reactions and calculate theoretical requirement of natural gas per tonne methanol.

(b) Considering net calorific values of hydrocarbons, calculate specific energy requirement per tonne of methanol.

2.9 Refined soybean oil is split with water at 30 bar g and 240°C in a high pressure autoclave. Composition of the soybean oil is given in Table 2.10.

*Am^3 means volume of gas at actual flowing conditions.

Table 2.10 Composition of Soybean Oil

Fatty acid	Formula	mass %	Molar mass
Palmitic acid	$C_{16}H_{32}O_2$	11.1	256.42
Stearic acid	$C_{18}H_{36}O_2$	3.8	284.48
Oleic acid	$C_{18}H_{34}O_2$	27.3	282.46
Linoleic acid	$C_{18}H_{32}O_2$	51.1	280.45
Linolenic acid	$C_{18}H_{30}O_2$	6.7	278.43

Assume that all fatty acids are present as triglycerides and refined oil does not have any other impurity.

(a) Calculate theoretical requirement of soybean oil for production of 100 kg fatty acids.
(b) Calculate theoretical water requirement for splitting.
(c) If the mixed feed to the autoclave contains 0.35 kg water per kg soybean oil, what will be the excess water fed (as percentage of theoretical required).
(d) Splitted dry mass contains fatty acids and glycerine. It is analysed by testing with aqueous KOH solution. Acid value of the dry mass is defined as mg KOH required for neutralization of 1 g mass product. Calculate theoretical acid value of the dry product, assuming 100% splitting.

2.10 A medium oil length alkyd resin is prepared by processing a mixture of 300 kg soybean oil, 200 kg phthalic anhydride [$C_6H_4(CO)_2O$] and glycerol [$(CH_2OH)_2CHOH$] together. Basically these are estrification reactions. Assume soybean oil composition as that given in Exercise 2.9. The process stipulates 10% excess of total hydroxyl (–OH) equivalents (e_B) over total acid (–COOH) equivalents (e_A). Calculate the required quantity of glycerol to be used in the mixture.

2.11 Ethylene chlorohydrin and sodium bicarbonate are reacted in a continuous stirred tank reactor. Reaction takes place as given in the following equation at 110 kPa a and 82°C:

$$\begin{array}{l} CH_2OH \\ | \\ CH_2Cl \end{array} + NaHCO_3 = \begin{array}{l} CH_2OH \\ | \\ CH_2OH \end{array} + NaCl + CO_2$$

Aqueous solution of 30% of ethylene chlorohydrin and aqueous solution of 15% $NaHCO_3$ (both by mass) are fed to the reactor in equimolar proportion. Conversion of the reactants is 95% while the yield of ethylene glycol is 100%. Calculate the mass % and mole % compositions of the product stream.

Carbon dioxide, produced during the reaction, is vented out at 110 kPa a and 82°C. Calculate the concentrations of carbon dioxide, water vapour, ethylene chlorohydrine and ethylene glycol in the vent gas stream. Assume ideal behavior of the liquid mixture.

Data:

(a) Vapour pressures of water, ethylene chlorohydrine and ethylene glycol at 82°C are 51.329 kPa, 17.065 kPa and 0.79 kPa, respectively.
(b) Lowering of vapour pressure of water due to the presence of inorganic salts ($NaCl + NaHCO_3$) having less than 10% concentration (by mass) in water at 82°C is 2.62 kPa.

2.12 Effluents generate at various locations in a composite textile unit. Reject water from reverse osmosis plant for production of water for boiler feed (BF) make-up purpose,

boiler and cooling tower blowdowns, dye-house effluents, fabric washing liquor, etc., collect in a pit. Raw water from an underground source and combined effluent analyses are given in Table 2.11.

Table 2.11 Analysis of Raw Water and Combined Effluent

Parameter	Raw water	Combined effluent
pH	7.8–7.9	7.5–7.9
Dissolved solids (DS), mg/L	1250	12 600
Total hardness as $CaCO_3$, mg/L	230	500
Permanent hardness as $CaCO_3$, mg/L	Nil	Nil
Calcium hardness as $CaCO_3$, mg/L	170	375
Total alkalinity as $CaCO_3$, mg/L	310	1190
Chlorides as Cl, mg/L	320	6260
Sulphates as SO_4, mg/L	156	210
Silica as SiO_2, mg/L (max.)	3	30
Suspended solids, mg/L (max.)	Nil	100
Chemical oxygen demand (COD), mg/L (max.)-dichromate value	Nil	800
Biological oxygen demand (BOD), mg/L (max.)	Nil	75
Oil and grease, mg/L (max.)	Nil	3
Colour	None	Dark

At first, the textile unit adopted primary and secondary treatments for removal of oil, grease, suspended matter (< 30 mg/L), COD (< 250 mg/L), BOD (< 30 mg/L) and colour. Effluents with nearly 13 000 mg/L DS after these treatments used to go to solar evaporation lagoons. Capacity of the plant was 100 m^3/d.

Shortage of water of underground source over a period forced the textile unit to consider integrated additional treatments for recovery of water for reuse. The unit requires about 75 m^3/d of raw water for BF Make-up purpose for passing through the reverse osmosis (RO) plant.

In the newly designed treatment facility (see Fig. 2.2) effluent from the existing treatment plant is first passed through an ultrafilter in which multicapillary hollow fiber membrane is used. This filter acts as a polishing filter in which suspended solids, organic impurities, colloidal silica, etc., are removed. Clear effluents with nearly same composition (in terms of inorganic compounds) and 12 250 mg/L DS is passed through two stage RO units. In the first stage, spirally wound membranes for brackish water are housed. Effluents enter at 25 bar g to this unit. Recovery of 62% is achieved as permeate which contains 520 mg/L DS and 275 mg/L chlorides (as Cl). Reject from the first unit is pumped to 50 bar g and passed through the second stage of the RO unit. This unit houses spirally wound membranes, designed for high solids brackish water desalination. Recovery of 28% is achieved from the second stage as permeate which contains 960 mg/L DS and 486 mg/L Cl. Permeates from both the stages are collected, mixed and passed through the existing reverse osmosis plant at 10 bar g to produce BF make-up water.

Fig. 2.2 *Proposed Effluent Treatment Facility for a Composite Textile Unit*

Reject from the second stage RO plant is pumped again to 7.5 bar g and passed through a nanofilter. In this unit, special spiral wound membranes are housed which selectively removes chlorides in the permeate. Recovery from the nanofilter is 90% in which 95% of the chlorides, present in the feed, are dissolved. Reject from the nanofilter is sent to solar evaporation lagoons.

(a) Calculate the total permeates, available from two stages of RO system. Check whether this quantity is adequate for production of BF make-up water.
(b) Brine (i.e., permeate with high chlorides from the nanofilter) is reused for production of 12% NaCl solution for dyehouse. Calculate the flow rate and concentration of brine.
(c) Calculate the reduction in final effluent, discharged to solar evaporation lagoons.

Hint : Assume chlorides as NaCl at all points in the treatment plant.

2.13 Ethyl benzene is an important intermediate for the manufacture of styrene. This intermediate can be manufactured by reacting benzene with ethylene over a catalyst in vapour phase. Patented Mobil–Badger process uses Zeolite as catalyst. Chief reaction can be written as under.

$$\underset{\text{Benzene}}{C_6H_6} + \underset{\text{Ethylene}}{CH_2 = CH_2} = \underset{\text{Ethyl benzene}}{C_6H_5CH_2CH_3}$$

Alkylation reaction does not stop at monoethyl benzene but a part of ethyl benzene is further alkylated to diethyl benzene, triethyl benzene, etc., collectively referred to as polyethyl benzenes.

$$\underset{\text{Ethyl benzene}}{C_6H_5CH_2CH_3} + \underset{\text{Ethylene}}{CH_2 = CH_2} = \underset{\text{Diethyl benzene}}{CH_3CH_2C_6H_4CH_2CH_3}$$

A high benzene to ethylene ratio in the feed gives a low polyethyl benzenes to ethyl benzene ratio in the product stream. Excess benzene is recovered from the product gas stream and recycled to the feed.

While pure ethylene stream can be used as a feed, dilute gas stream, such as treated off gas mixture from Fluid Catalytic Cracking (FCC) unit of a petroleum refinery with low ethylene concentration, can also be used as the feed.

Figure 2.3 is a simplified flow diagram of the process. Fresh benzene is vaporized and mixed with recycled benzene vapour. Ethylene in the form of offgas mixture from an FCC unit of refinery, having molar composition of CH_4: 37%, C_2H_6: 19%, C_2H_4: 19%, H_2: 9%, N_2: 13% and CO: 3%, is compressed to 12 bar g and fed to the reactor after heating in a heater, using molten salt.

Reactor has three 3 catalyst beds and operates at 12 bar a. Offgases are heated and sent to first bed. An overall molar ratio of 10 : 1 of benzene to ethylene is used for the reaction. Of the total benzene, 40% quantity is fed to the first bed at 350°C. Assume 50% conversion of ethylene in the first bed with 90% yield to ethyl benzene. Offgas temperature is so controlled that the product gas mixture from the first bed is maintained at 420°C. A definite quantity of benzene at 300°C is mixed into the production gas mixture from the first bed. In the second bed, additional 25% conversion of ethylene (on total feed basis) is achieved with 90% yield to ethyl benzene. Temperature of product gas from the second bed is maintained at 400°C. The product gas mixture from the second bed is quenced with balance quantity of benzene at 300°C. In the third bed, rest 25% conversion takes place with 90% yield to ethyl benzene. All polybenzenes may be assumed as diethyl benzene for calculation purpose.

Fig. 2.3 *Manufacture of Ethyl Benzene*

Table 2.12 Data on Gaseous Components[1]

Component	Formula	ΔH_f°, (gas) kJ/mol at 1 bar & 25°C	Gaseous heat capacity equation constants at 1 bar			
			a_i	$b_i \times 10^3$	$c_i \times 10^6$	$d_i \times 10^9$
Benzene	C_6H_6	82.93	–37.99	490.42	–321.39	79.36
Methane	CH_4	–74.52	19.25	52.11	11.97	–11.32
Ethane	C_2H_6	–83.82	5.41	178.09	–67.37	8.71
Ethylene	C_2H_4	52.50	4.13	155.02	–81.55	16.98
Hydrogen	H_2	0	28.61	1.02	–0.15	0.77

(Contd.)

Table 2.12 (*Contd.*)

Component	Formula	ΔH_f°, (gas) kJ/mol at 1 bar & 25°C	Gaseous heat capacity equation constants at 1 bar			
			a_i	$b_i \times 10^3$	$c_i \times 10^6$	$d_i \times 10^9$
Nitrogen	N_2	0	29.59	–5.14	13.18	–4.97
Carbon monoxide	CO	–110.53	29.12	0.25	–2.07	5.90
Ethyl benzene	C_8H_{10}	29.92	–36.72	671.12	–422.02	101.15
Diethyl benzene	$C_{10}H_{14}$	22.27	–37.41	867.09	–556.0	141.10

Calculate: (a) Required temperature of the offgas mixture, entering the first bed. (b) Benzene fed (in % of total) to the second bed as quench, and (c) Temperature of product gas stream, leaving third bed.

Note: Assume that enthalpy of formation and heat capacity data of 1 bar are valid at 12 bar a.

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Process Engineering 3

3.1 INTRODUCTION

A process is simply a method by which products can be manufactured from the raw materials. Selection of process steps for the manufacture of the product to the required specifications is the job of a process engineer. There could be number of methods to manufacture the same product. The best method for manufacturing a product has neither been discovered nor can it be said that a job can be performed in only one way. The process engineer faces alternative solutions to manufacturing problems. This possibility comes out of a combination of factors such as information gained by him from the past processes, imagination, his own knowledge of known processes, etc. However, economy of manufacture is a factor that frequently decides the best route to be followed. Thus, it is safe to say that innovation, imagination and economy are the key factors in process engineering.

3.2 EXAMPLES OF ALTERNATIVE ROUTES IN PROCESS ENGINEERING

Process engineering and design can be explained best with the following illustrations:

1. Consider treatment of water for various end uses. End use of treated water is a chief governing factor in deciding the process steps to be selected. Production of drinking water demands different steps as compared to production of high quality water for boiler feed (BF) make-up purpose.

   Drinking water requirements can be met by different water treatment steps. If water contains less dissolved solids, say less than 500 mg/L, clarification and disinfection by chlorination could be satisfactory. However, if raw water has high dissolved solids, dual filtration followed by desalination by reverse osmosis and *p*H adjustment would be necessary. These are relatively simple process steps.

   In case of BF make-up water requirements for high pressure boilers, number of routes of the process are possible. Clarification, activated carbon treatment and demineralization by ion exchange is one route. However, this route may not be economical if raw water has high dissolved solids (say more than 1500 mg/L).

In the latter case pre-treatment, reverse osmosis treatment, followed by ion exchange treatment would become necessary. The final process route to be followed will depend on raw water quality, operating pressure of the boiler, effluent disposal problem, etc.

2. In another case, consider the classic example of production of anhydrous (100%) ethanol. Ethanol produced by fermentation of molasses can be first distilled to produce azeotropic mixture (containing 95.6% alcohol by mass) which can be further dehydrated to 100% by use of an entrainer such as benzene in azeotropic distillation. New development of pervaporation process can be used in place of azeotropic distillation. Similarly, drying of azeotropic mixture by using molecular sieve beds can be also employed (Refer to Table 8.65). Innovations thus have a strong impact on process design alternatives.
3. Partially hydrogenated edible oils are extensively used for variety of applications. It is normally manufactured by hydrogenation of an edible oil in presence of a nickel catalyst under pressure (say at 2 bar g) and at 130 to 160°C in an autoclave. In a batch process, the reactor can have an anchor agitator with convolute at the top of the liquid level for proper mixing of hydrogen with the oil. This age old technology is replaced by installing a gas induced agitator (Refer to Exercise 10.2) which can result in a better product in a shorter period. This innovation has several advantages over the conventional design. Further research has demonstrated that use of carbon dioxide along with hydrogen at high pressure results in a product with stringent specifications (e.g., with less *trans* fatty acids).
4. Modern ammonia plants are large size plants (1800 t/d or more) which are continuous and reliable. These are quite compact than those originally developed to operate at very high pressures. Also, newer plants are safe and highly energy efficient, proving the economy of the scale.

Several examples can be cited where technological developments have totally changed the manufacturing methods. This can be attributed to newer technologies, efficient equipment designs, use of alternate raw materials, etc. Process engineer's job is, therefore, to evaluate various alternatives available to him and decide over the best method to be adopted. Batch type hydrogenation plants are still preferred over continuous one due to many reasons such as relatively small capacities, different feedstocks for different batches, safety in handling, etc. Design of ammonia plants are currently more guided by available raw material and specific energy consumption. Thus it cannot be said that the best method for manufacturing a product can be performed in only one way. The possibility of alternate process operations comes as combination of information gained from the past, imagination, and the process engineer's knowledge of the known processes.

3.3 GENERALIZED APPROACH TO THE CHEMICAL PLANT DESIGN

Design of chemical plant involves chemical technology, unit operations, instrumentation/controls, use of specialized equipments, safety considerations, materials of construction, etc. Chemical technology is the branch of chemical engineering dealing

with chemical changes and physical methods involved in industrial process. It is necessary to supplement the knowledge of unit operations with considerations of chemical technology in order to obtain a concept of the complete plant.

From the above discussions, it is clear that before attempting process engineering design it is necessary to fix the product specifications. Capacity of the plant can then be decided based on demand and/or investment. Evaluation of available technologies will have a say on investment. In order to arrive at consumption figures of raw materials and energy, preliminary stoichiometric calculations would be necessary. This would give some rough idea of cost of production due to inputs which normally accounts for higher proportion in the cost.

Following sequence might be followed in implementation of the project right from conception of the idea to construction and operation of the plant:

1. Process Research
2. Preliminary Process Design
3. Preliminary Process Evaluation
4. Process Development
5. Final Process Design
6. Final Process Evaluation

3.3.1 Process Research and Pilot Studies

Chemically feasible process is fundamentally characterized by process research. It is an accepted fact that process research is a basic necessity of the process industry. Orderly research and development has become an integral part of all leading industrial organizations and is synonymous with the progress. Basic operating conditions are explored and sufficient information is obtained from process research for preparing a preliminary material and energy balance flow sheet.

In many instances, development of a pilot plant is a necessity, particularly where laboratory scale data are not sufficient to scale-up or safety aspects are not well known. It is better to commit blunders on a small scale and make profits on a large scale. Such pilot plants can result in eliminating production of the off-spec product, good yields, reduced cost of manufacturing, etc., by fixing best operating conditions, installation of corrosion resistant plants, deployment of best equipment, reduced recycles due to better yields and so on.

Need for a pilot plant is justified in any manufacturing (Refs 1, 2, 3 and 4). Take for example, manufacture of bulk drugs. Manufacture of antibiotics involve reactions (of exothermic nature) at very low temperatures; at times at cryogenic temperatures. Use of ice or dry ice suffice at laboratory scale but at production level it is necessary to deploy industrial refrigeration which can be simulated in a pilot reactor. Design of a reactor changes significantly with industrial refrigeration in which possibly use of liquid nitrogen may be necessary. Similarly, solvent to reactant ratio used at laboratory scale may not be economically viable at the plant scale and it may be necessary to reduce the ratio to an optimum by experiments on a pilot scale.

As seen in the earlier section, hydrogenation of edible oil is carried out in a slurry comprising a catalyst. Selection of agitator is crucial. Pilot studies are valuable in the development of an agitator ideally suited for the reaction. Similarly, ozonation is a dangerous reaction and is very much dependent on intensive diffusion of gas in the liquid. Use of a static mixer with liquid circulation and simultaneous injection of ozone in it could prove to be advantageous. However, such designs should be supported by pilot studies for its efficacy and control of reaction. Design of a seal for agitator can

become an important design factor when dealing with high pressure gas reactions or with dangerous gases such as hydrogen, chlorine, ozone, etc. Hydrogenation of glucose to sorbitol is carried out in presence of slurry catalyst at quite high pressure (> 50 bar). Design of an agitator for such an autoclave and its sealing arrangement are challenges for the design engineers. Viscosity of reactant mass, operating pressure and temperature, suspended solids concentration, heterogeneous reaction mixture, etc., can have a strong bearing on reactor design and process research with a pilot plant could therefore be fully justified.

Pilot scale studies may be warranted for other unit operations. Its requirement can be decided based on the data available in literature or from the lab scale experiments. For example, if sufficient vapour–liquid equilibrium data are available for separation of a liquid mixture, a distillation equipment can be designed with a degree of certainty and pilot study may not be required. Similarly, mutual solubility data, if available, extraction equipment design can be carried out with known principles of unit operations. In the event of a specific design of the equipment, pilot studies are recommended. For example, for carrying out high vacuum distillation of a heat sensitive product, specialized equipment such as short path distillation or a wiped film evapourator is recommended. Manufacturers of such equipments provide pilot study on a small scale equipment with reasonable scale up confidence.

Pilot scale study can provide more reliable flow sheet than the one obtained from lab scale study. Versatile equipments which can be procured off the shelf are available for pilot studies. Alternately process engineers can design a tailor made pilot scale equipment for the study.

Pilot plant can be used for ongoing improvements of the existing process or development of a next generation process. One of the objectives of such a study is substitution of a raw material. Improvement in product quality, reduction in process costs, safety and minimizing the pollution effects are other objectives of the pilot study.

Introduction of a new product adds a dimension of risk relative to the existing product. Manufacturing a small volume of the new product in a pilot plant can be valuable in supplying it for commercial testing to a customer whose evaluation is crucial before introducing the product on a commercial scale. In many instances, when the customer demands some product specifications change, it could be achieved by process research on a pilot plant.

It can, therefore, be said that a pilot plant is an intermediate sized plant, modeling the process of interest, constructed and operated so as to test the model in a convenient, less expensive and safe manner.

Results obtained in the pilot plant builds confidence in management and help them in taking commercial, technical and financial decisions, thereby focusing their attention and allocating the resources accordingly.

For the design of pilot plants, useful guidelines can be obtained from the book by Jorden[4]. Several examples are drawn from industry in the book to develop the tailor-made pilot plants.

Cost of a pilot plant could be significant but risks are relatively low and payoffs could be substantial.

Another important consideration in pilot plant operation is the selection of manpower. A project leader (a chemical engineer), familiar with the chemical process and the equipments, is chosen for the job. He should be assisted by one or more chemists, depending on the size and complexicity of the pilot plant. Careful observations during the operation and interpretation of data are the skills desired from the personnel. Effective communication among the staff is crucial. Cost of such talented staff would be high but is rewarding. Motivation to the staff enhances self satisfaction and increases dedication towards the work.

Logging of data can range from visual examinations to instrument readings. Relatively inexpensive data acquisition system based on electronic instruments is available which can be installed and connected to a computer to record the data, generate trend analysis, know critical events, find temperature or pressure spikes, etc.

Instrumentation needs for the pilot plant depends on its complexity. A reasonably straight forward process may not justify complex instrumentation/control system but a high pressure and hazardous pilot plant will call for an expensive instrumentation package. Take, for example, a pilot plant for the manufacture of alkyd resins or antibiotics require normal instrumentation. However pilot plants for hydrogenation, ozonation, supercritical extraction with carbon dioxide, carbonylation with carbon monoxide, etc., may call for full automatic operation with data logging systems.

Pilot plant can be used as a training tool to plant operating personnel. It is well known that aeroplane pilots are trained on a simulator. Pilot plant can be considered as a simple version of 'simulator' of the main plant.

Project leader will be able to prepare a preliminary operating manual of the main plant based on his study of the pilot plant. Pre-startup testing, startup, normal operation and shutdown, emergency shutdowns, etc., can be well visualized by the project leader. The manual can be subsequently updated by the plant personnel based on their operating experience.

A process flow diagram can be offered by a team leader for further process design based on pilot plant studies.

3.3.2 Process Design and Development

After carrying out process research and pilot plant study, the process details are now available to the process engineer which can be scaled up with sufficient confidence level to a commercial plant. Scale-up from a pilot plant to manufacturing scale plant cannot be simply carried out by geometric scaling. Use of dimensionless numbers is recommended in scale-up for process designs. Zlokarnik[5] has given large number of interesting design problems of stirrers, absorber, solids processing, etc., in his book on scale-up. The book primarily deals with the scale-up methods involving geometric similarity principles and use of various dimensional numbers. In addition, it also describes the selection of process parameters that are to be critically studied during the experiments, decision on size of model experiment, model to be adopted for scale-up, etc. The book will generate a confidence to a process engineer in his design. Immediately the process engineer is not concerned with detailed design of

pieces of equipment but is concerned with the overall process covering following major items:

1. Preparation of a block diagram for presentation to the management for preliminary feasibility of the project.
2. Carryout overall and individual material balances showing the yield and quality of all materials to and from all pieces of equipment.
3. Heat balances over each piece of equipment.
4. Prepare a Process Flow Diagram (PFD) based on data in Steps (2) and (3).
5. Establish flow quantities and suction and discharge conditions of all rotating equipments.
6. Size reactors and storage tanks.
7. Separation process calculations with optimum operating conditions to effect the desired separation.
8. Estimate utility requirements such as steam, cooling water, refrigeration, electricity, thermic fluid, fuel, etc.
9. Prepare a Piping and Instrumentation Diagram (P&ID) so that requirement of instrumentation/control system is assessed.
10. Develop a tentative layout of the plant that should take into considerations of safety requirements.
11. Estimate preliminary capital investment and production cost.

Preliminary process design is necessary to find economic viability of the project and focus on important practical aspects. Take for example carbonylation of ethanol to propionic acid. This is high pressure and high temperature catalytic reaction of ethanol and carbon monoxide. Toxic nature of carbon monoxide dictates prominently in deciding over the safety requirements and practical operational aspects over the economic viability of the project.

Preliminary design will show the preferred process and whether further research and development (R&D) work is necessary. This additional R&D efforts will be specifically targetted to obtain missing information. Many a times physical properties of intermediates, corrosion data, heat of reaction, etc., are not available in literature and may have to be obtained by R&D efforts.

Process engineering calculations do not end with stoichiometric calculations. Process design of equipments for unit operations and unit processes require detailed literature survey to select best equipment suited for the service. While design and selection methods for many such equipments are discussed in Chapters 5 to 11, property determination of pure fluids and their mixtures is a task by itself. It is recommended that serious attempts be made to find properties of pure fluids that are reported in literature. Only when measured values could not be found in literature, estimation methods may be considered. Reed *et al*[7] has given extensive treatment to the subject of estimation of properties. For example, for pure fluids, group contribution methods are developed for a number of property parameters. Take a case of heat of formation data. While accurate data are published for large number of compounds by NIST, AIChE, etc., there are many compounds for which data are not available. Group contribution data by Reed *et al*. are quite useful for such compounds. Properties of mixture of fluids (such as density, viscosity, surface tension, etc.) are needed in most process design calculations. While some data are available for specific mixtures in literature,

estimation of these properties by various correlations is carried out. Process engineer must ensure that correlations, selected for calculations of the mixture properties, are reliable. In chapters to follow on process design of unit operations and reactors in the book, number of such correlations are used for calculation of properties of mixture of fluids.

Duration of completion of project can be predicted based on the preliminary design. Based on this information, marketing strategy of the product can be developed.

Development of PFD and P&ID are dealt in detail in Chapter 4 with specific examples.

After completion of the preliminary process design and its evaluation, detailed process design work is taken up. For the detailed design, variety of 'tools' are used in calculations. Estimation of properties by group contribution methods, literature search for different types of reactors, correlations for heat transfer, pressure drop and mass transfer calculations, mixing, etc., are the tools available to a process engineer. Many a times empirical sizing of equipment is carried out based on experience or literature survey. Numerous process design examples are worked out in Chapters 5 to 10 relating to fluid flow, heat transfer, separation processes and reaction kinetics.

Instrument engineer is simultaneously consulted in selecting the final control strategy. Suggestions/comments of the project leader of the pilot plant are quite valuable on the preliminary PFD and are incorporated as far as possible.

On completion of all the above steps, final PFD and P&ID are prepared by the process engineer and handed over to the project manager with equipment datasheets (containing process specifications) for further execution of the project.

During project execution stage also, role of the process engineer cannot be undermined. He will have an advisory role to ensure that process will function smoothly as anticipated during design stage.

3.3.3 Batch vs Continuous Operations

When designing processes or facilities for some applications, process engineer must approach the task differently than he would for a project in other chemical process industries. Bulk drugs, pharmaceutical intermediates, dyes intermediates, fine chemicals, etc., are the products in which batch operations dominate. Take the pharmaceutical industry sector. In this sector, lots of manufactured product are required to be segregated. In this way waste is minimized and regulations could be easily conformed to. Secondly, most pharmaceutical processes deal with relatively smaller amounts of material than with typical chemical processing operations. For this reason, batch processing equipment is often the preferred choice. Many a times, an equivalent continuous based unit operation that is small enough to accommodate the volume of material to be processed is not available. Further, most pharmaceutical processes come out of the lab where operations are predominately in batches. These batch-type processing techniques migrate to the larger production scale.

Although one would not propose changing batch processes to continuous process for small volumes, many operations could be made continuous. A continuous unit operation can be used to process a tank, full of material. For example, a continuous filtration method, such as a rotary drum filter can be used instead of batch method such as a plate and frame filter.

Another example of making continuous unit operations work involves two-phase extraction process. The traditional batch approach is to use a simple mixer-settler that mixes two phases together in a tank before they are allowed to separate. In a continuous analog, a small extraction column could be used in which two phases flow past one another continuously to facilitate phase contact. This continuous operation can be performed in a much smaller equipment. It also provides numerous 'stages' of phase contact resulting in more efficient mass transfer operation. Similar examples can be cited for distillation, fermentation, precipitation, crystallization, drying, etc.

Conversion from batch processing to continuous processes would thus allow smaller equipment with lower capital cost. Continuous equipment could be designed to be enclosed and cleaned easily, allowing the use of less costly production space. Many of these continuous equipment would operate at 'steady state' and it would be easy to automate them, reducing labour cost.

Batch operations and the equipment that facilitate them are by no means obsolete. Continuous equipment by its nature must be designed around a standard process that will be repeated for a significant period. For products that are made infrequently it is impractical to design continuous operations to support them. Processes where the input material varies over time also exist. These applications require an adjustment to the processing techniques to accommodate new variables. In many such cases only batch operations are flexible enough to handle these situations. Further some processes handle materials that are so valuable or hazardous that the transients that might occur in a continuous process are unacceptable. In these cases, only batch processes offer the control and containment, required to make them successful.

Continuous extraction of oil from oil-rich cake with *n*-hexane is practiced in industries on a large scale. However, when residual oil is to be extracted from spent bleaching earth before its disposal, a batch operation is normally performed as its quantity is small.

Bleaching of edible and non-edible oils was performed traditionally in batch equipment. Development of continuous bleachers give better quality of bleached oil with less bleaching earth/activated carbon and eliminate need for intermediate storage of bleached oil before its passage to continuous deodorizer. Also, the size of a continuous bleacher is much smaller than a batch bleacher.

Many a times, solvent to reactants ratio is kept high (may be in excess of 15) in a batch reactor based on lab studies. Many of such reactions could be relatively fast and endothermic. At the end of the reaction, solvent is required to be distilled out. Such a process lends itself for conversion to continuous operation. A mixed stream of solvent and reactants (possibly with much lower ratio) can be heated in a standard shell and tube heat exchanger, passed through a mixing equipment (such as a static mixer) and sent to a reactive distillation column where sufficient reaction time can be provided and simultaneously solvent is distilled out. Such a conversion can result in substantial energy savings apart from reduced time cycle of production.

Large capacity chemical plants are continuous in nature. Manufacture of ammonia, methanol, refining of petroleum products, acetic acid, carbon black, formaldehyde and many others are the examples where continuous plants are designed and operated. Consistent quality, optimum energy input, lower cost of installation of plant per unit (say kg) of product, etc., are inherent benefits of continuous plants.

The process engineer must evaluate both the operations before deciding the best operation, suited for the product. As explained in previous paragraphs, batch operations should be restricted to a minimum so that economy of scale, a feature of continuous operations, can be availed to maximum degree.

Reference 6 deals exclusively on batch process design.

3.4 PREPARATION OF OPERATING INSTRUCTIONS MANUAL

One of the less appreciated tasks of a process engineer is preparation of an Operating Instructions Manual (also known as Operation Manual) — OIM of the plant. When a calculator is purchased, a leaflet is provided along with the calculator which describes its functioning. In a similar fashion, OIM is prepared for safe, smooth and efficient operation of the plant. Well-prepared OIM serves as a tutor to the operating personnel. It is also a valuable reference tool in day-to-day operation.

Safe operation of the plant is the prime objective of preparation of the manual. Therefore, safe operating limits are prescribed clearly for each equipment and process. Dangers, if any, of crossing the critical parameters must be underlined.

Start-up and shut-down procedures are outlined in the manual. These include purging, slow pressure build-up, well-defined heating procedure, etc.

Normal operating procedures are covered for achieving optimum performance of the plant. These procedures cover desired operating parameters' range, generation of minimum effluent quantity, optimum utility consumptions, etc. Emergency handling procedures are well covered in the manual. Power failure, leakages, utility failure, fire, etc., are the emergencies which must be well thought of and a plan of each emergency must be described.

PFDs and P&IDs are included in OIM. Wherever required, additional sketches are included to clear the operational aspects. Calibration charts of tanks, reactors, dosing systems, etc., are included in OIM. For example, capacity of a screw feeder with its speed is an essential requirement of an operator.

Process datasheets and manufacturers' datasheets are normally compiled as Equipment Datasheets and it is a separate manual by itself. Instrument specifications are also included in this manual.

Useful guidelines are drawn from the pilot plant operation for the preparation of OIM. Equipment and instrument supplier catalogues provide their own operating instructions. OIM should cover comprehensively operating instructions of the manufacturers.

In many instances, standby pumps, filters, etc., are provided. Periodic operation of standby equipments is to be included in OIM. Similarly, periodic calibration of instruments is needed to ensure that they are functioning in order. For example, in case of pressure gauges, zero error is checked periodically. In case of temperature measuring instruments, calibration with a standard instrument is recommended periodically.

Preparation of OIM is normally undertaken by the process engineer during project execution. Draft copy of OIM is circulated to operating personnel and project leader of pilot plant. Suggestions received from them should be critically reviewed and if found useful should be included in the final OIM.

Over a period of time, OIM needs upgradation as more experience is gained in actual operation. Also the changes in OIM should cover the modifications that are carried out over the years. Thus upgradation is a never-ending process.

Preparation of log sheets for manual logging of data is normally the job of operating personnel. Nevertheless, the process engineer can review the draft of the log sheets and may offer suggestions. For medium scale and small scale plants, these are also known as *batch sheets*.

The process engineer should study the log sheets periodically and offer suggestions to the operating personnel. If necessary, OIM may require upgradation on account of review of the log sheets.

3.5 CONDUCTANCE OF PERFORMANCE TEST RUNS

On installation of the plant, process engineer is required to conduct a performance test of an equipment or entire plant. When a centrifugal pump is commissioned, its capacity, head and power consumption are checked with the datasheet. When a water treatment plant; say a reverse osmosis plant, is commissioned, its performance is checked for permeate quality (*vis-a-vis* feed water quality), permeate/feed ratio and power consumption. In the same manner, performance of cooling tower is checked for achieving anticipated cooling water temperature for the prevalent wet bulb temperature of ambient air and for its power consumption.

In large chemical plants, performance guarantees are well specified in the contract. These include product quality specifications, raw materials consumptions and utility consumptions. All these parameters are averaged over a specified continuous run of the plant, say for 120 hours or so. Average values are compared with those guaranteed.

Conductance of performance test run requires specialized skills. It is carried out by the process engineer in coordination with the plant personnel and contractor personnel. Before starting the test run, instruments are calibrated. Any specific deviation from normality is documented in the log sheet.

Test run report comprises log sheets, observation sheets, analytical reports and summary of findings. It is used as Acceptance Report for the equipment or the plant.

3.6 TROUBLESHOOTING

Troubleshooting is yet another task of the process engineer. During commissioning or normal operation, many operational problems are experienced. Some of them are related to the process. Process engineer is required to study the problem, discuss with operating personnel and present a solution.

Take an example in which cooling water cools a fluid. If cooling is less effective than design, reasons for the same are to be determined. Less flow of cooling water or high supply cooling water temperature or inadequate heat transfer surface could be the problem for a new system. Material and energy balance across the cooler will reveal the problem and corrective action can be taken. During the course of operation, if the cooler performance deteriorates, fouling of cooling surface could be anticipated.

Take another example of a separator in which organic liquid is separated from water. While studying the performance of the separator, check moisture content of the

organic layer and chemical oxygen demand (COD) of the water layer. If the results are not satisfactory in terms of separation, check operating temperature of the separator or its design.

In a distillation column, if the distillate quality is not maintained, one can check the reflux ratio and other parameters.

3.7 COST CUTTING MEASURES

In a normal running plant, process engineer has to consider cost cutting measures in the plant. This is a never ending exercise as there exists an opportunity for improvement in each plant. For finding the opportunity, indepth study of the plant is recommended.

Consider a chilling plant in which brine at –10°C is produced using refrigerant R-22. It is a known fact that in this temperature range Coefficient of Performance (COP) of ammonia refrigeration cycle is better than that of R-22 cycle. Thus replacement of the refrigeration skid of R-22 system by ammonia system will consume less power. However, fresh investment for the ammonia refrigeration system will be needed. A cost benefit analysis will have to be carried out by process engineer.

Take another case of a plant in which hot oil at 150–175°C is used to heat reaction mass and carry out the reaction at around 105°C. Alternative system with saturated steam at 3.5 bar g could be equally or more effective. Both economical and practical aspects need be examined before any change in the plant is considered.

In large chemical plants such as ammonia plants many opportunities exist for cost reduction. With methane and ambient air as feedstocks at 25°C, theoretical energy consumption is 20.618 GJ/t ammonia. Actual ammonia plants consume energy equivalent to 27 to 42 GJ/t ammonia, depending on the feedstock and technology. This indicates a potential for reduction in energy consumption and thereby in cost of production. Similar opportunities can be found in each plant.

Newer technologies and innovations can contribute significantly to cost reduction. Process engineering plays a vital role in their implementation.

3.8 PREPARATION OF DATA SHEETS OF EQUIPMENTS

Ordering of equipments starts as soon as the process design of equipments is completed. Inquiries are sent to prospective vendors for getting their technical and commercial offers. For this reason, it is necessary that adequate technical data are furnished to the vendors in the technical data sheets. Most engineering firms have their own standard format for data sheets which are filled by process engineers. This important task is not to be undermined as incomplete data may invite incomplete or at times wrong offer from vendors. As an example, specifications of a heat exchanger can be filled in TEMA data sheet. Figures 10.7 and 10.8 are sample TEMA data sheets. It will be easy for vendors to submit their correct technical offer based on such data sheets.

3.9 GREEN ENGINEERING

A set of 12 guiding principles of 'green chemistry' were established by Dr. P. Anastas and J. C. Warner in 1998 for creating safer chemicals and designing for degradation to save the environment. Further to these guidelines, a multidisciplinary group of

engineering disciplines met in May 2003 in Florida, USA to draft the principle of Green Engineering. The conference focused on the definition of the scope and tone of green engineering relative to all engineering disciplines; including chemical, mechanical, civil, electrical and environmental.

The objective of green engineering is to transform existing engineering disciplines and practices to those that promote sustainability. It incorporates the development and implementation of technologically and economically viable products, processes and systems to promote human welfare while protecting human health and elevating the protection of the biosphere as a criterion in engineering solutions. To implement these solutions, engineers are advised to use the following principles[8]:

1. Engineer processes and products holistically, use systems analysis and integrate environmental impact assessment tools.
2. Conserve and improve natural ecosystems while protecting human health and well-being.
3. Use life-cycle thinking in all engineering activities.
4. Ensure that all material and energy inputs and outputs are as inherently safe and benign as possible.
5. Minimize depletion of natural resources.
6. Strive to prevent waste.
7. Develop and apply engineering solutions while being cognizant of local geography, aspirations and cultures.
8. Create engineering solutions beyond current or dominant technologies; improve, innovate and invent (technologies) to achieve sustainability.
9. Actively engage communities and stakeholders in development of engineering solutions.

Process engineer can play a vital role in achieving the objectives of green engineering. At every stage of process engineering, the engineer must consider and review whether the design adheres to the principles of green engineering. A few examples will demonstrate implementation of principles of green engineering in practice.

1. In solvent extraction plants, *n*-hexane is used to extract oil from oil-rich cake. It is known that *n*-hexane is not totally safe for human beings. New supercritical extraction process based on carbon dioxide as an extracting agent is found to be eco-friendly, safe for human beings and also gives good quality oil.
   Water can also be used as supercritical fluid to produce biocrude from organic waste. In this application, water is a safe extracting agent while it helps in safe 'disposal' of organic (including hazardous) waste.
2. Hydrogenated edible oils are used extensively in bakery and other food products. Presence of trans-fatty acids in the hydrogenated fat is known to be unfit for human consumption. Technological developments should be targetted to produce the fat with low trans-fatty acids. One such development is hydrogenation in presence of carbon dioxide at low temperature. Another development relates to the use of noble metal catalyst (instead of nickel) for hydrogenation.
3. Methyl tertiary butyl ether (MTBE) is known an oxygenating product for improvement of octane number of the gasoline. Research has revealed the health hazards of MTBE when autovehicles emit gases to atmosphere. Addition

of anhydrous ethanol (a renewable product) is said to improve octane number without creating health hazards.

4. Trans esterification of fatty acids with methanol can produce biodiesel which is considered an eco-friendly fuel for DG sets or for autovehicles. Fatty acids can be obtained from edible and non-edible oils which are renewable resources.
5. Ozone depleting substances are threats to human beings. Montreal protocol, 1987 bans the use of certain chlorofluoro hydrocarbons which are established to have very high ozone depleting potential, apart from their low degradability. Classical example is dichlorodifluoromethane (CFC-12) which is banned for use as a refrigerant, aerosole agent, etc. Engineers are advised to use ammonia (R-717) or chlorodifluoromethane (CFC-22) in place of CFC-12. Eventually CFC-22 is also to be phased out.
6. In a classical Bechamp process, nitro-compound is converted to a corresponding amine by using iron power and water mixture. Reaction takes long time. At the end of the reaction, total mass is required to be filtered out of the iron sludge (fine in nature). Later the mass is separated in two layers. Water, so separated, has high Chemical Oxygen Demand (COD) and pose an effluent problem.
   In new developed processes, hydrogen is used in presence of a catalyst under pressure. Use of gas-induced agitator, nickel or noble metal catalysts, etc., are the developments which result in reduced time of reaction and also do not generate effluents. Thus, it is an eco-friendly process.
7. While methanol is used as a solvent in many chemical plants, it is toxic to human beings. Its use is considered unsafe in pharmaceutical and food processing. Its higher homolog ethyl alcohol is safe for use in the industry as a solvent.

3.10 PROCESS INTENSIFICATION

Process intensification can be defined as improving productivity of a system by changing operational parameters and/or by carrying out desirable modifications. In a process plant, improvement in productivity means product quality improvement, yield/production rate increase, reduction in energy consumption, reduction in other inputs (such as raw materials), safer operation, or reduction in pollution load of the plant. Process intensification differs from troubleshooting (Sec. 3.6) as the latter mainly focuses on solving the problems, faced by operating personnel. Cost cutting measures (Sec. 3.7) can be considered as one of the objectives of process intensification.

There is a caveat to the definition of process intensification. Development of a new chemical route or a change in composition of a catalyst, however, do not quality as process intensification.

Process engineer can contribute significantly in process intensification; either at the process design stage or in the running plant. Scientific approach to the design and critical analysis and review of the operating data are the keys to process intensification.

At the process design stage, a number of options are available to the process engineer. Consider design of a multiple-effect evaporator system. Apart from the process parameters; such as concentration of ingoing/outcoming solution, heating medium and its conditions (e.g., pressure and temperature), etc., process engineer

can design the evaporators with equal or unequal heat transfer area (HTA) in the stages. Optimization techniques such as Evolutionary Operation (EVOP) or dynamic programming can be used to find HTA for both the alternatives, thereby cost of the installed systems for both the alternatives can be worked out. Optimum design of the system can then be selected.

A cascading type steam balance offers an interesting example of utility system optimization as a part of process intensification. Modern heavy chemical plants such as ammonia plant, methanol plant, ethylene manufacturing, petroleum refinery, etc., have cascading steam systems which can be optimized to consume minimum fuel in the boiler and also reduce load on cooling tower.

In a simpler case, selection of an agitator in a reactor can result in substantial energy saving-with improved agitation.

Linear programming is another tool which helps in optimizing the proportions of components in a reaction mixture. Consider manufacture of alkyd resins by reaction of polyols (such as glycerol) with fatty acids by azeotropic distillation method. Fatty acids could be either distilled (pure) fatty acid or in the form of an oil. Similarly, a variety of alcohols such as glycerol, sorbitol, etc., are used. Theoretical calculations can be made with the help of linear programming to achieve desired properties of the product with best possible yield which can later be confirmed in laboratory trials.

Innovations and adoption of newer technologies are integral to process intensification. Adoption of a standard operating practice in one industry into another industry should be considered for a similar situation. A fluid bed cooler is used in large urea plant to produce urea of uniform (narrow range particle size distribution) size prills at low temperature so that urea prills do not agglomerate to lumps on storage in a silo which produce dust on breaking them. Similar problems are also experienced in a carbon black plant. Adoption of the fluid bed cooler should be considered in a carbon black plant.

Absorption of hydrogen chloride in water is highly exothermic and is efficiently carried out in a Falling Film Absorber, made from graphite blocks. Such an absorption system is found more effective than a packed bed absorber. Such type of an absorber can well be considered for absorption cum reaction of sulphur trioxide (gas) in organic compounds such as linear alkyl benzene, methyl ester of saturated fatty acids, etc.

Use of a static mixer in mixing an organic compound with a mixed acid (HNO_3 + H_2SO_4) to form nitro compound is found to be very effective in controlling side reactions with efficient temperature control in a batch reactor.

Several examples can thus be cited to demonstrate that process intensification at the design stage or in an operating plant can result in a plant with lower operating cost, possibly lower initial cost, ease of operation and safer operation.

In an operating plant, critical review of operational parameters can result in substantial savings. Use of statistical methods are invaluable in this exercise. In fact, ISO 9001 gives stress on statistical methods for improvement in quality control which is advised as a continuous improvement process. An article by Bader[9] gives a systematic method for quality assurance by statistical means.

In a similar manner, it is advised that variation in process parameters and its effect on plant performance be studied which can reflect the best and safer operating conditions.

Modifications in the plant can be aimed at reduction in energy consumption or reduction in raw materials consumptions. In ammonia plants, purge gas from synthesis loop was being vented or used a fuel in the furnace in many grass root plants. Adoption of purge gas recovery plant to recover and recycle hydrogen from the purge gas could result in increase in production by about 5% and simultaneously reduce energy consumption of the entire production by about 2%.

In a bulk drug plant, some reactions are carried out at very low temperatures (around –80°C). Injection of liquid nitrogen is quite common for achieving such low temperature in the industry. This method is rather inefficient and expensive. Instead, a cascading type mechanical refrigeration system with R-23 and R-22/R-717 in tandem can be considered for adoption to achieve the same temperature at much lower investment and operating cost. Also the space requirement is less for the refrigeration system. Adoption of cascading refrigeration system may bring down the refrigeration cost by about 50% as compared to liquid nitrogen injection system for a given plant.

Use of aqueous calcium chloride brine was common in refrigeration. However, numerous operating problems were experienced with it such as slimy spills, chocking of pipelines due to freezing, difficulty in maintaining brine concentration, etc. Aqueous monoethylene glycol (MEG) is found safe and convenient substitute to aqueous calcium chloride brine, thereby design of brine circulation system is simplified. Use of aqueous methanol brine is made for low temperature applications (–20 to –50°C) but the brine solution is flammable in nature and requires specific care in design. Thus adoption of new systems is a part of process intensification.

In large chemical plants, the network of heat exchangers can be reworked in line with pinch technology (Sec. 6.19). Such an exercise can result in substantial energy savings.

3.11 CONCLUSION

Process engineering is the heart of any process design. While adequate attention at the design stage can pay rich dividends, adoption of process engineering principles in a running plant is always rewarding.

EXERCISES

3.1 Give brief answers of the following:

(a) Name ten biofuels that are used for steam generation.

(b) Differentiate petroleum based lube oils and eco-friendly lube oils.

(c) An innovative hydrogenation process for edible oil is to be developed which is based on supercritical principle. Carbon dioxide and propane are considered as alternative supercritical fluids (SCFs) for the purpose. Study the advantages and disadvantages of each for the purpose.

(d) Name five 'green' solvents for use in chemical synthesis.

(e) An aqueous *iso*–propyl alcohol mixture, containing 50% alcohol (by mass), is to be separated to a stream of 100% alcohol. Suggest process steps with reasonings.

(f) Nitrogen stream at atmospheric pressure and dew point of –60°C is used for deriming a cold box of air liquefaction plant. If argon is used instead of nitrogen, what should be the dew point on equivalent moisture content basis?

(g) For relatively small capacity storage of liquefied gases (such as ammonia, LPG, etc.), a sphere construction is selected. Why?

(h) As a rule of thumb for most of the distillation cases, an increase in column pressure means: (a) decrease or (b) increase in the column capacity and (i) better or (ii) work fractionation, given other parameters.

(i) Opening stock of a fuel oil took was calculated as 600 kL × SG of 0.94 = 564 t, and the closing stock as 550 kL × SG of 0.94 = 517 t in a day, i.e., transfer of 47 t to the boiler. Temperature variations were ignored thinking that the variation in SG is about 1% and should, for all practical purposes, be acceptable. In reality, the temperatures of the fuel oil at the time of measurements of opening and closing stocks were such that corresponding SG were 0.94 and 0.95, respectively. It really meant an error of about as much as (i) 5.5, (ii) 3.5, (iii) 1.5, (iv) 0.15 or (v) 0.05 t.

(j) Name 2 alternative refrigerants which can be used in place of R-12 (an ozone depleting compound).

3.2 Chlorination of an organic compound is to be carried out with chlorine. Hydrogen chloride gas with some chlorine are vented out from a batch reactor at near atmospheric pressure. Consider the various options, available for trapping hydrogen chloride gas from the reactor and develop process flow diagrams for each of them.

3.3 Study a batch reactor operation. Write start-up, normal operating and shutdown procedures for the reactor, including safety requirements. Also prepare a log sheet for the batch operation with a view to collect consolidated information for review and comments.

3.4 In a chemical plant, a cooling tower of 1000 kW capacity is operated. An operator reports in summer that the production is limited due to cooling tower. How will you check the efficiency of the cooling tower?

3.5 In a batch reactor, reaction is carried out under reflux conditions for long hours with a condenser using cooling water at 32°C. Solvent used in the vessel is methanol. Plant data indicate substantial loss of methanol to atmosphere during the reaction. Analyze the situation and suggest remedial measures for loss prevention with reasonings.

3.6 A pharmaceutical plant, manufacturing bulk drugs, uses acetone for final purification of a product. After filtration, the wet cake is dried in a fluid bed drier in which nitrogen is used as a drying medium. The offgases from the dryer is saturated nitrogen with vapour of water and acetone at near atmospheric pressure and 75 to 90°C. Local pollution control authority requires that the emissions from the drier must contain acetone below 200 ppm. Suggest a process scheme with a diagram such that the final gas mixture meets the local pollution authority's requirement with reasons for selection of the scheme.

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4 Importance of Process Diagrams in Process Engineering

4.1 INTRODUCTION

Engineering Diagrams are vital for understanding of a process to chemical engineers, of equipments to mechanical engineers, of electrical circuits to electrical engineers, and so on. These diagrams are prepared in many different ways to suit the specific requirements. Process Flow Diagrams (PFD) and Piping and Instrumentation Diagrams (P&ID) are basic requirements of process engineering and are prepared before attempting any detailing. Steps followed in preparation of these diagrams and interpretation of the same are covered in this chapter.

4.2 NEED FOR PFD AND P&ID

When any chemical manufacturing project is conceived, immediate information is sought on the requirements of raw materials and utilities. This information can be calculated using the Laws of Conservation of Mass and Energy, covered in the previous chapter on Stoichiometry. When these data are presented in a sequential manner with symbolic representation of equipments, it is known as PROCESS FLOW DIAGRAM (PFD) or PROCESS FLOW SHEET. It can be said that PFD is a schematic illustration of the system. It shows relationships between the major components in the system. It also tabulates the process design values for the components.

PIPING & INSTRUMENTATION DIAGRAM (P&ID) is different from PFD. While PFD is a quantitative representation of the process, sequential presentation of flow, piping with instrumentation and controls that are required for smooth operation of the process are given on a P&I diagram. It can therefore be said that P&ID is a schematic illustration of functional relationship of piping, instrumentation, and system equipment components. It shows all the piping including the physical sequence of branches, fittings, equipments, instrumentation and control interlocks.

PFD is a useful diagram in effective communication of design information. It helps an operator in adjusting his operating parameters, a shift supervisor in checking the plant operation, management in discussions across the table and project engineers in comparison and evaluation of different processes and their quantitative requirements.

Modern plants have extensive instrumentation and controls such that a minimum attention is required of an operator. Apart from controlling the plant variables, these

plants give better productivity in terms of yield and quality. Also, less manpower is required to operate the plant with increased safety.

Process engineers are involved in preparation of both these types of diagrams.

4.3 BLOCK DIAGRAMS

Simplest flowsheets are block diagrams. Process engineer begins the process design with a block flow diagram in which only the feed and product streams are identified. Such input-output diagrams are not very detailed and as such are most useful in early stages of process development.

Figure 4.1 is a block diagram of the process to manufacture carbon dioxide from flue gas of power generation plant using electrochemical membrane separation technique.

Fig. 4.1 *Block Diagram for Manufacture of 200 t/d CO_2 by Electrochemical Membrane Process*[1] *(Reproduced with the Permission of American Institute of Chemical Engineers, USA)*

Absorption of carbon dioxide from the flue gas mixture using an aqueous amine solution or caustic potash solution is difficult because of presence of oxygen. Electrodialysis cell has an edge over the absorption technology in which presence of oxygen is not a hindrance to the recovery of carbon dioxide. The block diagram is useful in understanding overall process and also gives rough quantitative picture of raw materials and utility consumptions. These data can help in working out preliminary variable cost.

The processes, described in the block diagram, are then broken down into basic functional elements such as reaction and separation sections. Process engineer can also identify the recycle streams and additional unit operations to achieve the desired operating conditions. Identification of basic elements help in performing material and energy balance calculations which in turn help in preparation of the PFD.

4.4 DEVELOPMENT AND UTILITY OF PROCESS FLOW DIAGRAMS

Any product or process development starts with stoichiometric calculations. At first, required capacity of the final product is fixed. The product can normally be manufactured by inputting different raw materials and following different process routes. For example, manufacture of ammonia is possible with different hydrocarbon feed stocks such as natural gas, naphtha, fuel oil, coal, etc. Process route for each feed stock is different. For example, reforming is a preferred route for natural gas or naphtha while partial oxidation is the route followed for fuel oil or coal. Nitrogen for the ammonia manufacture is invariably obtained from air but it may be fed as air or nitrogen is separated from air by liquefaction and it is fed to the process. PFD (Process Flow Diagram) for each continuous process is different.

Another example is reduction of nitro aromatic compounds to corresponding amines. In a classical Bechamp process, nitro compound is mixed with water and fine iron powder in a batch reactor. The reaction mixture is moderately heated in presence of a catalyst to initiate the reaction (basically in an acidic medium). Hydrogen is generated in situ and reduces the nitro compound to corresponding amine. Reaction is exothermic and temperature is controlled by cooling. Reaction takes long time to complete. Product mixture is separated in organic and aqueous layers. While aqueous layer is drained out, organic layer is siphoned and purified by filtration and distillation under vacuum.

In newer version of the process, hydrogen gas is bubbled in the organic liquid under pressure. Nickel or noble metal-based catalyst is used. Gas dispersion through the liquid is improved by different methods such as use of static mixer, use of gas induced agitator, liquid jet ejector system, etc. Such a system is quite efficient and the reaction can be completed in relatively short period. Both the process routes are used in industry. PFDs for both the routes are different.

Above examples involve chemical reactions. However, there are PFDs which involve unit operations but no chemical reactions. For example, separation of components of natural gas in a distillation column by cryogenic means, removal of acid gas by absorption–desorption, removal of Volatile Organic Compounds (VOC)

by adsorption–desorption, etc., do not involve a chemical reaction. Each PFD is, therefore, to be treated on its own requirements.

A Process Flow Diagram generally includes following information:

1. Flow rate of each stream in case of continuous process or quantity of each reactant in case of a batch process.
2. Composition of streams.
3. Operating conditions of each stream such as pressure, temperature, concentrations, etc.
4. Heat added or removed in a particular equipment.
5. Flows and design operating conditions of utilities such as steam, cooling water, brine, hot oil, chilled water, thermic fluid, etc.
6. Major equipment symbols, names and identification.
7. Any specific information which is useful in understanding the process. For example, symbolic presentation of a hazard, safety precautions, sequence of flow, etc.

PFD of a laboratory scale process could be somewhat different from that of plant scale PFD. In laboratory, more emphasis is given on safety aspects. Also some utilities could be different from those used on a plant scale. For example, in laboratory use of ice and dry ice are common for achieving required chilling. On a plant scale, chilled water or chilled brine at different temperatures are used. Similarly, once through raw water system is common for cooling in condensers in the laboratory while closed circuit of cooling water is a practice in the industry. Batch distillation is commonly employed in the laboratory while in plants, batch or continuous distillation columns are employed with specific controls to achieve desired purity of the distilled materials.

Process Flow Diagram of a laboratory experimental unit could be simple and different from that of a pilot plant. Thodos *et al*[2] have described a PFD for the study of reaction kinetics to synthesize ethyl chloride from ethylene and hydrogen chloride gas. This is a gas phase endothermic catalytic reaction, studied in the pressure range of 8–11.5 bar a and temperature range of 150°C–300°C. A 100 mm steel pipe reactor with electric heating element, wrapped over it, was used. Dry ice trap, following the reactor, removed ethyl chloride while excess HCl was scrubbed in a water trap. Thus a continuous experimental set-up provided useful data for determining reaction rate equation.

When the above process is to be studied on a pilot plant, the reactor could still be a pipe (bigger in size) with jacket in which thermic fluid (hot oil) circulation is provided. For separation of ethyl chloride, heat exchanger with chilled brine can be considered. Scrubbing of unreacted HCl can be performed in an efficient packed tower or in a falling film absorber.

Prugh[3] has developed a laboratory scale PFD for the manufacture of phenylethyl alcohol by reacting ethylene oxide and benzene in presence of aluminum chloride catalyst. Such a PFD is helpful in transferring information from the research chemist to the process engineer, engaged in designing a pilot plant or in developing a PFD for a commercial plant. Sufficient stress is laid on safe scale-up. Effectiveness of the research efforts is thereby improved in designing a plant.

In the laboratory, many hazardous reactions are carried out such as chlorination, hydrogenation, ozonation, carbonylation, etc. Some of them are not only explosive but

also prone to health hazards. During scale-up of such processes, these aspects need special attention so that the plant is safe in operation and environmental pollution problem is not created. For example, venting of hydrogen containing gas mixture must ensure that the gas mixture is outside flammability limits. Also a right type of flame arrestor is to be installed on the vent. Similarly chlorine to be vented is to be scrubbed in aqueous caustic solution. These details form a part of process (although not a part of reaction) and therefore PFD must include these equipments.

PFD of a commercial plant covers material and energy balance details extensively. In PFDs of batch processes, quantities of inputs and outputs are specified with normal operating conditions. For a continuous process plant, a good practice is to give process stream flow rates and compositions in a tabular form at the bottom of PFD. Reference 4 gives several PFDs for batch as well as continuous processes.

Development of PFD of a process plant starts with conceptual design. At first the process flow sequence is established with the help of symbols of equipments or blocks. While symbols of equipments give better idea of the type of equipment to be deployed, block diagrams are satisfactory to start with. End product and capacity of production are defined which are entered at the end of PFD. Then an overall theoretical material balance is established to find the theoretical raw materials' requirements. These quantities are entered at appropriate places in PFD where the specific raw materials are to be introduced. Thus a preliminary PFD is prepared. Now start establishing material balance and energy balance across each equipment taking into conversion limitations, yield, selectivity, catalyst activity, equipment limitations, etc. It is well known that conversions are dictated by kinetic considerations and hence, it is not possible to achieve 100% conversion every time. These calculations are performed for each equipment with actual conversions and results are entered in the PFD. With these revised data, PFD becomes more like a working PFD. To this, add utilities and safety aspects. Final PFD is now ready for presentation.

Material and energy balance calculations for the manufacture of formaldehyde from methanol based on Formox process are well covered in Ref. 4. Using this information, its PFD is developed for a plant having methanol feed rate of 4000 kg/h (Fig. 4.2). In this diagram, process flow rates and operating conditions are given along the process lines. Heat duties of various equipments are also specified near the respective equipment. Stream compositions are given in tabular form at the bottom of the PFD.

The above PFD is quite useful to a process engineer in detail design of the equipments, selection of right equipment for the duty, pipe sizing, selection of right instrumentation and control system, etc. Further, PFDs, submitted by different vendors at the quotation stage, help the project engineer in comparison and evaluation of alternative process routes. Competitive processes can be evaluated from different angles such as ease of operation, minimum energy requirement, less hazardous operation, turndown capability of the plant, etc. Such an evaluation becomes useful in discussions with the management across the table thereby the technologically best plant can be selected.

PFD of an operating plant provides design data at 100% plant load. Such a diagram helps the operator in adjusting his process parameters and the shift supervisor in checking/controlling the plant operation to optimize the production.

Manufacture of formaldehyde is a catalytic process. Comparison of actual operating data with the design conditions (such as those given is Fig. 4.2) can provide a trend analysis thereby a decision can be taken on when to change the catalyst. However, if the supervisor finds that productivity of the plant is not commensurate with the anticipated catalyst life, he would look for other factors like poisoning of catalyst, fouling of coolers, etc., responsible for the same and corrective actions can be taken.

In Fig. 4.3, PFD for the production of liquid oxygen from air at low pressure (10 bar g) is given. In this process, only physical separation takes place unlike chemical reaction, described in Fig. 4.2. This PFD was developed to compare the low pressure process with the high pressure (135–140 bar g) process. It does not give the process flows and stream compositions and hence, it is not directly useful to plant operating personnel. Nevertheless it is useful to the project engineer in preliminary evaluation.

Figure 4.4 is the PFD for separation of ethane and heavier components from natural gas. In this process also only physical separation takes place but operating conditions of all streams are specified. Flow rates of major streams and their compositions are given in a tabular form at the bottom of the PFD. An interesting feature of the PFD is incorporation of two heat pumps which will result in substantial energy saving. Also an expander is provided to generate power when gas pressure is reduced. External refrigeration is provided at sufficiently high temperature level so that a standard refrigeration plant with R-22 can be used and cryogenic liquids are not required. Development of PFD thus provides an opportunity to a process engineer for energy conservation.

PFD of a utility system is somewhat different from the PFD of the process plant. Steam generation, steam distribution and utilization, cooling water circuit, thermic fluid circuit, instrument/plant air system, nitrogen (inert) gas system, refrigeration system, etc., fall under this category. Figure 4.5 is a typical cascade steam balance of a large chemical plant. This is also a PFD of the steam system. Development of an efficient steam system can pay rich dividends in terms of optimum energy input.

In Chapter 11, detailed discussion on utilities is given with different P&I diagrams and flow diagrams.

In Ref. 4, several steam balances are worked out for the same plant and corresponding PFDs are presented to exemplify the alternatives available to a process engineer during the design stage.

Similar PFDs can be developed for other utility systems. Liu *et al*[8] have presented PFDs for raw water in a chemical plant. Reduction in raw water consumption, as much as 50%, could be achieved by optimizing such a PFD.

Cooling water is yet another utility for which an efficient distribution network can be developed. In Fig. 9.24, cooling water network is demonstrated.

Process flowsheeting can be carried out with the help of a computer. Steady state material and energy balance calculations can be performed on a spreadsheet or with the help of a mathematical software which also can generate block diagrams. Specialized software such as Microsoft Visio, Aspen Plus, etc., are available for the preparations of PFDs. References 9 to 11 are useful in developing computerized flow sheets.

Note: All flows are in kg/h.

Fig. 4.5 *Steam Balance in a Large Chemical Plant*[7] *(Reproduced with the Permission from Chemical Engineering by Access Intelligence, USA)*

4.5 DEVELOPMENT OF PIPING AND INSTRUMENTATION DIAGRAMS

Having understood the requirements of PFD, it is rather easy to understand the requirements of P&ID. Emphasis is changed from process flows to process control. Controls are not possible without measurements of variables; such as pressure, temperature, concentration, etc., for which instruments are required. A diagram incorporating piping, instruments and controls is known as P&ID.

In simple plants minimum instruments are installed. They are just adequate for manual control. However, it is needless to emphasize that installation of adequate instruments justify the cost in terms of productivity and safety. Many batch processes are being controlled in manual manner. But as the scale of plant is getting bigger and bigger, role of instrumentation and control (I&C) is becoming important. This I&C hardware not only results in smoother and efficient plant operation but also gives safer operation and high onstream factor. Modern chemical plants incorporate sophisticated I&C, thereby centralized operation from a control room is possible. PLC (Programmable Logic Control) and DCS (Distributed Control System) have become common terminology in I&C. Touch screen operation of the panel gives a wealth of information to the operator at his finger tips.

In addition to I&C, P&ID also incorporates other details such as pipeline details, location of valves and fittings, safety valves' locations, etc. In major chemical engineering projects, piping and fittings are classified to cover specific range of pressure and temperature for each fluid. This classification lists material of construction, thickness, types of fittings required, etc., for piping. This classification is mentioned on each pipe symbol on P&ID in addition to the size of the pipe. Many times, fluid flowing in the pipe, is indicated by symbolic letters, e.g., NG for natural gas, S for steam, etc. This helps project engineer to specify the piping material with fittings for procurement with ease.

Apart from instrumentation and control details, many other details are also covered on P&ID. Cold and hot insulations are marked. At times elevation is mentioned when relative height of a specific equipment is important. For example, barometric condenser is installed at 10.5 m height from hotwell water level as standard atmospheric pressure equals 10.33 meters of water column. Similarly, when a pump takes suction from a vacuum system, NPSH requirements are to be met which means positive suction is required depending on the vacuum. Deaerator for boiler feed water (BFW) is installed at a height to provide sufficient NPSH to the BFW pump. In many instances, minimum height of the stack, is specified by the local pollution control authority.

When complete draining of a vessel is desired, slope of specific pipeline is desired which can be indicated on P&ID in a symbolic manner. Man-entry and maintenance requirements are kept in mind while preparing P&ID. For this purpose, purging of vessels with inert gas/compressed air is necessary although this requirement is not indicated on PFD. Location of sample points and their arrangements are also shown on P&ID.

Symbolic presentation on the diagram should be easily understood by the engineer. There are standard symbols for such presentation. All engineering companies have their own standard symbols which are supplied along with P&ID.

However, standards are prevalent for the graphical symbols. Among them, following are prominently followed by many:

Indian Standards:

IS: 3232	Graphical Symbols for Process Flow Diagrams
IS: 9446	Graphical Symbols for Pipelines and Instrumentation Diagrams

US Standards:

ANSI Y32.2.3	Graphical Symbols for Pipe Fittings, Valves and Piping
ANSI Y32.2.11	Graphical Symbols for Process Flow Diagrams
ISA 5.5	Graphical Symbols for Process Displays

British Standards:

BS: 1646	1 to 4	Symbolic Representation for Process Measurement, Control Functions and Instrumentation

German Standards:

DIN 19227	P1 to P3	Graphical Symbols and Identifying Letters for Process Measurement and Control Functions

Figures 4.6(a) and (b) give recommended symbols for equipments while Figs 4.7(a) and (b) give recommended symbols for piping, instruments and controls which will be generally followed in this book.

Fig. 4.6(a) *Symbols for Equipments*

Fig. 4.6(b) *Symbols for Equipments*

	Gate Valve Butt Welded Gate Valve Flanged		Diaphragm Operated Butterfly Valve
	Globe Valve Butt Welded	M	Electric Motor Operated Valve
	Plug Valve		Flush Bottom Tank Valve
	Three Way Plug Valve		Needdle Valve
	Ball Valve		Direct Float Operated Valve
	Angle Valve	P	Piston Operated Valve (Air or Oil)
	Damper Valve	HP LP	Pressure Reducing Valve
	Diaphragm Valve		Slide Valve
	Butterfly Valve	(a) (b)	Rupture Disc (a) In Line (b) On Line
	Air Motor Operated Valve		Hose Connection
Or	Bellows Seal Valve		Vent or Drain with Blind
	Combination of Safety Relief Valve and Rupture Disc Assy		Y-Type Strainer
	Safety Relief Valve with Hand Lever		Valve Closed
	Control Valve		Vent or Drain Plugged
	Diaphragm Operated Control Valve	OR	Check (Non-Return) Valve
	Diaphragm Operated Valve (Angle Type)	S	Solenoid Operated Gate Valve
	Diaphragm Operated Control Valve (Three Way Type)	S	Solenoid Operated Gate Valve with Manual Reset
	Main Line		Spectacle Blind (Normally Closed)
	Auxiliary Line		Spectacle Blind (Normally Open)
	Piping Underground		Drain
	Insulated Piping	T	Trap Assembly
	Jacketed Piping		Restriction Orifice and Flange
	Tracing (Process)		Union

Fig. 4.7(a) *Symbols for Piping*

	Electrically Traced Piping		Reducer (concentric)
OR	Piping Crossover		Reducer (eccentric)
	Piping Junctions (distinguished from Crossover by Displacement)		Elbow 90° Short Radius (R = 1 × D)
	Flow Direction (Arrows at Change at Routing)	(a) (b)	Elbow 90° Long Radius (R = 1.5 × D) (a) Butt Weld (b) Screwed/sw
	Piping Flange		Elbow 45° Screwed or Socket Welded
	Piping Flanged with Blind		Alteration in Pipe Size (only Shown where Essential to Understand Process Requirement)
	Pipe Termination with Cap/BW		Self Actuated Control Valve
	Pipe Termination with Screwed/SW	Piping Identification (Typical) Example: S-3-100-CS1 Steam, Number, Pipe Size, Piping Specifications	
* OR	Pipe Specification Change		
Owner Contractor	Boundary by Indication (suitably Closed)	*Note:* Process engineer can develop his own piping identifications.	

Fig. 4.7(a) *Symbols for Piping (Contd.)*

I. Instruments

Orifice Meter

Venturi Meter

Gauge (Pressure or Temperature)

Rotameter

Bypass Rotameter

Level Gauge

Turbine Flow Meter

II. Instruments Signal Lines

Pneumatic (Air) Signal

Electric Signal

Instrument Impulse Line

Instrument Capillary Tube

Radioactive, Sonic or Light Signal

E/P Potential to Pneumatic Converter
I/P Current to Pneumatic Converter
E/I Potential to Current Converter

Fig. 4.7(b) *Symbols for Instrumentation and Controls*

Fig. 4.7(b) *Symbols for Instrumentation and Controls (Contd.)*

Bartow *et al.*[12] have presented a P&ID for a pilot plant to carry out research and development work relating to manufacture active pharmaceutical ingredients (API) in a bulk drug plant. Different types of reactions can be carried out in such a pilot plant with heating and cooling, varying form cryogenic conditions (up to –100°C) to hot conditions (up to +250°C). Process engineer can develop P&ID for such an universal facility.

4.5.1 Development of Simplified P&I Diagrams

With the use of guidelines and symbols, given in previous paragraphs, P&I diagrams for various systems can be developed with ease.

Consider the unit operation of distillation. Prime objectives of instrumentation and control (I&C) system of this unit operation are to maintain purities of top and bottom products. There could be variety of disturbances in the system and I&C should be developed to take care of these disturbances. Change in feed flow rate and/or its composition, change in thermal condition of the feed, changes in heating/cooling medium conditions, changes in ambient conditions, etc., are some disturbances that are to be taken care and purities must be maintained.

For simplicity, a binary distillation system will be considered. Cooling water is considered as a cooling medium while steam at suitable pressure is considered for heating in the reboiler. Purity of the top product is generally controlled by controlling the top temperature and varying the reflux ratio while purity of the bottom product is chiefly controlled by maintaining a specified temperature of the solution in the reboiler. Figure 4.8(a) gives a typical P&I diagram for a system in which distillation is carried under vacuum in a packed column. In this case, top product purity is of prime importance. Temperature of vapour, leaving the column, is of prime importance. Reflux ratio is adjusted to control this temperature. While temperature of liquid in the reboiler dictates the quality of the bottom product, differential pressure across the column is controlled near design value by energy input to the reboiler.

Where bottom product quality is of significant importance, a scheme shown in Fig. 4.8(b) could be considered. It may be noted that the temperature of liquid in the reboiler is of prime consideration. Hence, a casade controller with reboiler liquid temperature as a master parameter and differential pressure across the column as the slave parameter could prove useful. In this case, distillation is carried out under pressure in a plate column. In a binary distillation, temperatures of vapour, leaving top or liquid leaving bottom are nearly constant for specified purities. In addition to the temperature control, it is desired that pressure drop across the column is also controlled to avoid excessive boiling in the reboiler. Selection of plate or packed column is process designer engineer's choice but control philosophy remains nearly unaffected by the column internals. However, it is possible that a sharp (or significant) change in temperature at a particular plate in plate column or at a particular point in packed column can take place with change in the feed composition or its thermal condition. This location can be adjudged by tray to tray calculations and additional measurement control of temperature at this location can help in controlling the purities of both products during changes in feed conditions. In Fig. 4.8(a), it is considered that sharp change in temperature is expected in rectification section while in Fig. 4.8(b), the sharp change in temperature in the stripping section is visualized. P&I diagram, shown in Fig. 4.8(a) relates to a vacuum system and hence, elevation marking was considered necessary on it to ensure that equipments' placement and piping details can be worked out accordingly.

Considered another system in which a continuous stirred tank reactor (CSTR) is used for carrying out an exothermic reaction. For initiation of the reaction, initially heat transfer fluid (hot oil) from a thermo pack is used to supply heat to the reactor.

Once the reaction has picked up and exothermic heat of reaction is to be removed, cooled thermic fluid can be circulated in the jacket. This scheme is shown in Fig. 4.9(a). In an alternate arrangement, a CSTR with limpet coils can be considered. One coil can be used for heating with hot thermic fluid while another coil can be used for cooling with cooling water. Note that curved baffles are provided in the jacket so that thermic fluid flows in a guided manner and channeling of it can be avoided.

Fig. 4.8(a) *P&I Diagram for High Purity Distillate*

Figure 4.9(b) is the P&I diagram for this arrangement. Latter case can be considered when the thermic fluid is simultaneously used in different equipments for heating and cooling of thermic fluid. However, it may be a waste of energy in the thermo pack. If CSTR has only one jacket or one limpet coil, heat removal can be considered by circulation of liquid in an external heat exchanger with the help of a pump and providing sufficient liquid head for hot reactants to overcome required net positive suction head $(NPSH)_R$. Figure 4.9(c) gives the details of such a system. In these three P&I diagrams, different type of agitators are shown but the control philosophy is unaffected by these internals.

Fig. 4.8(b) *P&I Diagram for High Purity Bottom Product*

Fig. 4.9(a) *Use of Thermic Fluid for Heating/Cooling in CSTR with Jacket*

Fig. 4.9(b) *Use of Thermic Fluid for Heating and of Cooling Water for Cooling in CSTR with Limpet Coils*

Fig. 4.9(c) *Use of Thermic Fluid for Heating/Cooling in CSTR with Thermopack and External Cooler*

Fig. 4.9 *P&I Diagrams for Temperature Control in CSTR*

Development of the above simplified P&I diagrams with reasoning may prove useful in learning development of P&I diagrams for entire plants involving several equipments and having number of utilities.

Figure 4.10 is P&ID for formaldehyde manufacturing, described in Fig. 4.2 with recycle option. It is developed to ensure safe and smooth operation of the plant in an efficient manner.

Types of control system, shown in Fig. 4.10 can vary from one process engineer to another. For example, air flow to the reactor can be controlled by a control valve on feed air line (A-3-750-CS1). However, in Fig. 4.10, vent controls the feed air flow. Such a control system is found more reliable and accurate in practice. Also, discharge pressure of the blower could be lower in the suggested scheme. Flow of demineralized (DM) water to the absorber may not be a critical requirement. Manual control could therefore be adequate and costly I&C system for its flow control may not be warranted. Thus, preparation of P&ID provides an opportunity to the process engineer to demonstrate his skills. Further, the diagram has to take care of start-up requirements, emergency response, purging, etc.

In certain plants, universal facilities are required for the manufacture or processing of similar products. For example, manufacture of different amino and alkyd resins call for such a universal facility. Lanson[13] has presented a P&ID for the resin manufacturing either by azeotropic distillation process or by vacuum process. An edible oil refinery in which edible oils of different seeds can be refined also represents a similar case.

Several softwares are available for preparation of P&ID also. Microsoft Visio, Aspen Plus, Smart Sketch, etc. can be used to reduce the time in such preparation.

4.6 CONCLUSION

PFD and P&ID are indispensable as transmitters of information, essential in the design stages. They can also be considered as the instruments of communication. In the final analysis, process diagrams communicate an astonishing amount of engineering information about many things to many groups for a variety of uses.

It is not surprising that preparations of PFD and P&ID have assumed great importance in chemical process industry. Process engineers can produce innovative designs which can be well presented by these diagrams. Time spent on preparation can easily result in cost savings, efficient plant layout, and safer plant operation.

EXERCISES

4.1 Prepare P&I diagrams for following cases with safety measures:

(a) Hydrogen from a bullet at 10 bar g and 40°C is let down to 2 bar g for feeding to edible hydrogenation reactor.

(b) Carbon dioxide is to be cooled from 110°C to 40°C using cooling water in an intercooler at 68 bar a. Cooling water is supplied at 32°C. Critical pressure and critical temperature of carbon dioxide are 73.77 bar and 31°C, respectively.

(c) A heat sensitive product is concentrated in a wiped film evaporator (refer to Chapter 8) at absolute pressure of 1×10^{-3} Pa and 180°C. Ultra high vacuum is achieved with the help of dry vacuum pump and three-stage ejector system (with barometric condenser) in series. A cold trap is provided with brine/steam utilities for knocking off organic vapours, being carried to the vacuum system. Temperature in the evaporator is controlled by thermic fluid, entering at 240°C in the jacket and leaving at 220°C from the jacket. Adequate instrumentation and controls are to be provided for smooth operation.

(d) Visit a nearby liquefied gas (such as ammonia, carbon dioxide, LPG, etc.) storage facility, study the same and prepare a P&I diagram of the system, including storage tank and holding refrigeration system.

4.2 A pilot plant reactor (SS 316L) of 250 L geometric capacity is to be designed for conducting sponsored research at any temperature, ranging from –80°C to +220°C. For this purpose, it is desired to use a specialty synthetic (silicone polymer based) heat transfer medium, such as Syltherm XLT® of Dow Chemical Co., USA. Typical properties of Syltherm XLT are as under.

Chemical: Dimethyl polysiloxane
Average molar mass: 317 kg/kmol
Flash point: 54°C
Auto ignition temperature: 350°C
Freezing point: Less than –110°C
Density @ 25°C: 0.852 kg/L
Gross heat of combustion at 25°C = 32 800 kJ/kg

The reactor will be suitable for maximum operating pressure of 25 bar *g*. It will be provided with a turbine agitator with a variable frequency (speed) drive for speed

control in the range, 80–200 rpm. A flameproof electric motor of 3 kW capacity, operating on 415 ± 10% V, 3 phase, 50 ± 2 Hz power will be used as a drive. Agitator will be sealed with a mechanical seal with cooling arrangement with cooling water circulation.

The reactor will also be suitable for operation under vacuum up to 700 Torr. Reflux arrangement with two condensers; one using cooling water at 32°C and another vent condenser, using brine at –10°C, will be provided.

Heat transfer medium (HTM) will be circulated in the jacket with the help of a gear pump. A degassing tank of 100 L capacity will be provided at 2 m elevation relative to the outlet nozzle of the reactor.

(a) HTM will be cooled from –40°C to –100°C with liquid nitrogen in a welded plate heat exchanger (PHE).
(b) HTM will be cooled with the help of refrigerant R–22 in the temperature range –40°C and +40°C in the welded PHE.
(c) HTM will be cooled with cooling water at 32°C in the temperature range +40°C and +80°C in a shell and tube heat exchanger.
(d) HTM will be heated up to +160°C with saturated steam at 7 bar *g* in a shell and tube heat exchanger.
(e) HTM will be heated beyond +160°C up to +220°C with the help of thermic fluid, supplied at +280°C in the shell and tube heat exchanger.

Heat transfer duty will be 5 kW for each case.

Based on above information, develop a P&I diagram of the above system.

4.3 Prepare a PFD for the manufacture of nitric acid based on stoichiometric calculations, given in Examples 2.10 and 2.11.

4.4 Prepare a P&ID for VOC emission control scheme, developed for Exercise 3.6.

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5 Process Design of Piping, Fluid Moving Devices and Flow Meters

5.1 INTRODUCTION

A fluid is a substance which when in static equilibrium cannot sustain tangential or shear forces. This property of action under stress distinguishes it from the 'solid' state of the matter. Fluids are commonly divided into two substances; liquids and gases. A liquid occupies a definite volume, independent of the dimensions of its container, while a gas tends to expand to fill any container in which it is contained. A vapour differs from a gas by being readily condensable to a liquid on cooling.

Strictly speaking all fluids are compressible. However, gases are frequently regarded as compressible while liquids are as incompressible as the latter undergoes small changes in the properties under compression. Ambient air is treated as incompressible fluid as its density changes insignificantly with minor changes in atmospheric pressure and temperature. However, compressed air is treated as a compressible fluid.

In a pump, a fan or a compressor, mechanical work is transformed into fluid energy. A turbine or an expander is a machine for transforming fluid energy into mechanical work at the rotating shaft. The word 'pump' is normally used for a machine handling liquid. In a fan, the density change is so small, due to relatively low pressure difference, that the gas is regarded as incompressible for evaluation. In a compressor, the density change is appreciable.

This chapter deals with fluids, its transportation through various moving devices and various flow measuring instruments.

5.2 PROCESS DESIGN OF PIPING

Design of piping system incorporates all major disciplines of engineering like chemical engineering, mechanical engineering, civil engineering, instrumentation and control engineering, electrical engineering, etc.

Process design of pipe is a balance between size or diameter of pipe and pressure drop in the pipe. For a given flow rate of fluid if larger size of pipe is selected then it gives lesser pressure drop. Larger size of pipe increases the fixed cost of pipe and

lesser pressure drop means lesser power consumption or lesser operating cost of pipe. Lesser pressure drop in pipe may reduce the size of flow moving device like pump and thereby it may reduce the fixed cost of pump. Thus capital cost of pipe increases with diameter, whereas pumping or compression cost decrease with increasing diameter. Ideally one should find the pipe size which gives the sum of amortized capital cost plus operating cost a minimum or one should find the optimum pipe size.

5.2.1 Optimum Pipe Size

Several equations and nomographs are available in literature to estimate the optimum pipe diameter. But these equations or nomographs are not reliable as optimum pipe diameter depends on the current cost of material, cost of power, rate of interest at particular place and at particular time. It changes with place and time. For example, optimum pipe diameter equation, derived in USA, cannot be used to find the same in India. One such equation[1] derived for carbon steel pipe and turbulent flow of an incompressible fluid is

$$d_{opt} = 293\, \dot{m}^{0.53}\, \rho^{-0.37} \tag{5.1}$$

where, d_{opt} = Optimum pipe size, mm
$\dot{m}$ = Flow rate, kg/s
ρ = Density of fluid, kg/m^3

This equation was developed by the Institution of Chemical Engineers, UK based on cost data of 1987, updated to mid 1992.

5.2.2 Recommended Fluid Velocities in Pipe

The velocities, recommended in Table 5.1, are only for approximate calculations of pipe diameter which can be used for the quick calculation of short distance pipe line or for estimating pipe size as a starting point for pressure drop calculations.

Table 5.1 Recommended Fluid Velocities

Fluid		Recommended velocity, m/s
Water	Pump suction line	0.3 to 1.5
	Pump discharge line	2 to 3
	Average service	1 to 2.5
	Gravity flow	0.5 to 1
Steam	0 to 2 atm g, saturated	20 to 30
	2 to 10 atm g, saturated	30 to 50
	Superheated below 10 atm g	20 to 50
	Superheated above 10 atm g	30 to 75
	Vacuum lines	100 to 125
Air	0 to 2 atm g	20
	> 2 atm g	30
Ammonia/ refrigerant	Liquid	1.8
	Gas	30

(*Contd.*)

Table 5.1 (*Contd.*)

Fluid		Recommended velocity, m/s
Organic liquids and oils		1.8 to 2
Natural gas		25 to 35
Chlorine	Liquid	1.5
	Gas	10 to 25
Hydrochloric acid	Liquid (aqueous)	1.5
	Gas	10
Inorganic liquids		1.2 to 1.8
Gas and vapours		15 to 30

5.2.3 Pressure Drop in Pipe

Relation between pressure drop and pipe diameter is given by Fanning or Darcy equation. It is derived for steady flow in uniform circular pipes running full of liquid under isothermal conditions.

$$\frac{\Delta p}{L} = \frac{2f\ v^2 \rho}{g_c \cdot D_i} = \frac{32f\ \dot{m}^2}{\pi^2 \rho\ g_c D_i^5} \tag{5.2}$$

For SI units, g_c = Newton's law conversion factor = 1

where, Δp = Pressure drop, Pa

L = Length of pipe, m

$\dot{m}$ = Mass flow rate of fluid, kg/s

ρ = Density of fluid, kg/m^3

D_i = Pipe inside diameter, m

v = Velocity of fluid, m/s

f = Fanning friction factor

The Fanning friction factor is a function of the Reynolds number (*Re*) and the roughness of inside surface (ε).

Values of ε for various materials are given in Table 5.2.

Table 5.2 Values of Surface Roughness for Various Materials

Material	Surface roughness ε, mm
Commercial steel or Wrought iron	0.045 72
Galvanized iron	0.152
Cast iron	0.259
Concrete	0.305 – 3.05
Riveted steel	0.914 – 9.14
Brass, Lead, Glass, Cement, and Bituminus Linings	0.001 524

A plot of Fanning friction factor as a function of Reynolds number (*Re*) and relative roughness, ε/D, is given as Fig. 5.1.

Fig. 5.1 *Fanning Friction Factors*[2]
(Reproduced with the Permission of McGraw-Hill Book Co., USA)

For turbulent flow in commercial steel pipe, $f = 0.0394\ Re^{-0.16}$ (5.3a)
Above equation gives the value f on higher (safer) side. One of the more accurate relationships between f and Re for turbulent flow (Ref.: 3)

$$\frac{1}{\sqrt{f}} = -1.7372 \ln\left(\frac{Re}{1.964 \ln(Re) - 3.8215}\right) \tag{5.3b}$$

where, $Re = \dfrac{D_i v \rho}{\mu} = \dfrac{4\dot{m}}{\pi D_i \mu}$ (Reynolds number) (5.4)

μ = Viscosity of fluid, Pa·s or kg/(m·s) or N·s/m^2

From Eqs. (5.2), (5.3a) and (5.4),

$$\frac{\Delta p}{L} = \frac{32}{\pi^2} \times \frac{0.0394\ Re^{-0.16} \dot{m}^2}{\rho\ g_c D_i^5}$$

$$\frac{\Delta p}{L} = 0.127\ 7457 \left(\frac{4\dot{m}}{\pi D_i \mu}\right)^{-0.16} \left(\frac{\dot{m}^2}{\rho\ D_i^5}\right)$$

$$\frac{\Delta p}{L} = 0.122\ 9024\ \dot{m}^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1}$$

If G in kg/s, μ in kg/(m·s) or in N·s/m^2, D_i in m, ρ in kg/m^3, L in m, then Δp is in Pa or in N/m^2.
Normally pipe diameters are expressed in mm. If D_i is in mm then Δp in Pa is given by

$$\frac{\Delta p}{L} = 0.122\ 9024 \times 1000^{-4.84} \dot{m}^{1.84} D_i^{-4.84} \rho^{-1} \cdot \mu^{0.16}$$

$$\frac{\Delta p}{L} = 4.07 \times 10^{13} \dot{m}^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \tag{5.5a}$$

If Δp is in kPa or in kN/m^2, then

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} \dot{m}^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \tag{5.5b}$$

Example 5.1 Estimate the optimum pipe diameter for a flow of dry chlorine gas of 10 000 kg/h at 6 atm a and 20°C through carbon steel pipe using Eq. (5.1).

Solution

For carbon steel pipe, optimum pipe diameter

$$d_{opt} = 293\ \dot{m}^{0.53}\ \rho^{-0.37} \tag{5.1}$$

$$\dot{m} = 10\ 000 \text{ kg/h} = 2.7778 \text{ kg/s}$$

$$\rho = \frac{pM}{RT} = \frac{pM}{T} \times \frac{T_s}{p_s V_s} = \frac{6 \times 71}{(273 + 20)} \times \frac{273}{(1 \times 22.414)}$$

$$\rho = 17.71 \text{ kg/m}^3$$

$$d_{opt} = 293(2.7778)^{0.53}(17.71)^{-0.37}$$
$$= 173.855 \text{ mm}$$

Use next heigher size of standard pipe, i.e., 200 mm (8 in) NB standard pipe.

5.2.4 Standard Pipes

Standard pipes are available from 3 mm (1/8 in) to 600 mm (24 in) size. Standard pipes are specified with three different diameters, namely inside diameter, outside diameter and nominal diameter. For standard pipes having diameter more than 300 mm (12 in), nominal diameters are equal to the actual outside diameter but for smaller pipe there is no relation between nominal diameter and inside diameter or outside diameter.

The wall thickness of standard pipe is indicated by the schedule number as per the US standards.

$$\text{Schedule Number, SCH} = \frac{p_{sf} \times 1000}{\sigma_s} \tag{5.6}$$

where, p_{sf} = Safe working pressure, N/mm^2
σ_s = Safe allowable stress, N/mm^2

Standard pipes are available with variety of schedule numbers; 5, 10, 20, 30, 40, 60, 80, 100, 120, 140, 160. With carbon steel pipe, schedule 40 up to 150 mm (6 in) and with stainless steel pipe, schedule 10 are used for general purposes. Thickness of standard pipe increases with increases in schedule number. Seamless standard pipes do not have welding joint. Fabricated pipes are fabricated by rolling the plates followed by joining the ends of plate by welding which are also known as Electric Resistance Welded (ERW) pipes.

In matric system (including those specified in Indian Standards), thickness of a pipe is specified in mm.

Thickness of pipe, subjected to internal pressure, is determined by equation.

$$t = \frac{p\, r_i}{\sigma E - 0.6p} + \text{CA} = \frac{p\, r_o}{\sigma E + 0.4p} + \text{CA} \tag{5.7}$$

where, t = Thickness of pipe, mm
p = Internal design pressure, N/m^2
r_i = Inside radius of pipe, mm
r_o = Outside radius of pipe, mm
σ = Allowable stress of pipe material at design temperature, N/m^2
E = Joint efficiency; for seamless standard pipe, $E = 1$
CA = Corrosion allowance, mm

5.2.5 Pressure Drop in Fittings and Valves

In addition to pipes, piping system contains fittings and valves. These fittings and valves offer the additional frictional loss or additional pressure drop. This additional frictional loss of a fitting or of a valve is expressed either as an equivalent straight pipe length (L_e) or as a number of velocity heads (K), lost in pipe of same size and of same material.

Equivalent Length of Pipe (L_e) for Fittings and Valves Equivalent length of a valve or of a fitting is the length of straight pipe of same size creating the same friction loss as the fitting or the valve in consideration. Nomographs for finding equivalent length of pipe for various fittings and valves are given in various books (Ref. 4, 5 and 6).

Often, L_e is expressed in terms of inside diameter of pipe. Then $L_e = \frac{L_e}{D_i} D_i$ where, D_i = Inside diameter of pipe.

Values of L_e/D_i for few valves and fittings are given in Table 5.3.

Table 5.3 Equivalent Lengths of Fittings and Valves

Valve or fitting	$\frac{L_e}{D_i} = \frac{\text{Equivalent length}}{\text{Inside diameter}}$
Gate valve (fully open)	7 to 10
Gate valve (3/4 closed)	800 to 1100
Gate valve (1/2 closed)	190 to 290
Globe valve (fully open)	330 to 480
Angle valve (fully open)	165 to 220
Plug valve (fully open)	18
90° elbows (standard radius)	30
45° elbows (long radius)	5.8
45° elbows (short radius)	8.0
Return bend (medium radius)	39 to 56
Coupling or union	Negligible
Tee, straight through	22

Another way of calculating pressure drop through the fittings and valves is the use of factor K.

"Number of velocity heads (K) lost in pipe" for fittings or valves is defined by equation

$$\frac{\Delta F}{v^2/2g_c} = \frac{\Delta p/\rho}{v^2/2g_c} = K \tag{5.8}$$

or

$$\Delta p = \frac{K\rho v^2}{2g_c}, \text{ in SI system, } g_c = 1 \tag{5.9}$$

where, ΔF = Additional frictional loss, J/kg or N·m/kg
Δp = Additional pressure drop, N/m^2
v = Average fluid velocity through the pipe of same size as valve or fitting, m/s

Values of K for normally used fittings and valves are given in Table 5.4.

Table 5.4 Equivalent Velocity Heads of Fittings and Valves

Type of fitting or valve	Equivalent number of velocity heads (K) (applicable only for turbulent flow)
Gate valve (open)	0.17
Globle valve, bevel seat (open)	6.0
Plug valve (open)	0.4
Diaphragm valve (open)	2.3
Angle valve (open)	2.0
Check valve (swing)	2.0
Foot valve	15
Coupling	0.04
90° elbows (standard)	0.75
90° elbows (long radius)	0.45
45° elbows (long radius)	0.2
45° elbows standard	0.35
90° bend	0.75
180° bend (closed return)	1.50
Tee straight through	0.4
Butterfly valve ($\alpha = 5°$)	0.24
Check valve (swing type)	2.0

Simpson[7] has given values of K for variety of other fittings, such as miter bends, different types of reducers, different bends, etc.

Example 5.2 Carbon dioxide is to be conveyed from the top of the stripper of ammonia plant to urea plant. Calculate the pipe size required based on following data:

Flow rate of CO_2 = 1000 t/day
Total length of pipe = 800 m
Available pressure at inlet of pipe = 24 kPa g
Discharge pressure of CO_2 from pipe required = atmospheric
No. of 90° elbows in pipe line = 8
No. of butterfly valve = 1
No. of flow nozzle = 1
Temperature of gas = 60°C
Viscosity of CO_2 gas = 0.016 mPa · s or cP

Solution

Density of carbon dioxide (CO_2) gas at the discharge of pipe:

$$\rho = \frac{pM}{RT}$$

p = 1 atm, Molar mass of CO_2, M = 44

$$R = 0.0821 \frac{\text{atm} \cdot \text{m}^3}{(\text{kmol} \cdot \text{K})}, \quad T = 273 + 60 = 333 \text{ K}$$

$$\rho = \frac{1 \times 44}{0.0821 \times 333} = 1.6094 \text{ kg/m}^3$$

Mass flow rate of CO_2, $\dot{m} = \dfrac{1000 \times 10^3}{24 \times 3600} = 11.574$ kg/s

Let the velocity of gas through pipe, $v = 20$ m/s

$$\frac{\pi}{4} D_i^2 = \frac{\dot{m}/\rho}{v}$$

$$= \frac{11.574/1.6094}{20} = 0.3596 \text{ m}^2$$

$$D_i = 0.677 \text{ m}$$

Selecting fabricated pipe having inside diameter, $D_i = 677$ mm

Reynolds number

$$Re = \frac{D_i v \rho}{\mu} = \frac{0.677 \times 20 \times 1.6094}{0.016 \times 10^{-3}}$$

$$= 1361\,955 > 10\,000 \quad \text{(i.e., turbulent flow)}$$

Pressure drop in straight steel pipe for turbulent flow

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} \dot{m}^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \tag{5.5}$$

$$= 4.07 \times 10^{10} (11.574)^{1.84} (0.016 \times 10^{-3})^{0.16} (677)^{-4.84} (1.6094)^{-1}$$

$$= 7.8046 \times 10^{-3} \text{ kPa/m} \equiv 7.8046 \text{ Pa/m}$$

Pressure drop in butterfly valve:

$$\Delta p_{E1} = \frac{K_i \rho v^2}{2} = \frac{0.24 \times 1.6094 \times 20^2}{2}$$

$$= 77.25 \text{ Pa}$$

Pressure drop in 90° elbow (standard):

$$\Delta p_{E2} = \frac{K_i \rho v^2}{2} = \frac{0.75 \times 1.6094 \times 20^2}{2}$$

$$= 241.41 \text{ Pa}$$

Pressure drop in the flow nozzle is considered negligible.

Total pressure drop in pipe line

$$\Delta p_t = 7.8046 \times 800 + 77.25 + 8 \times 241.41 = 8252 \text{ Pa} \equiv 8.25 \text{ kPa}$$

Available pressure is 24 kPa. Hence, to decrease the fixed cost, smaller pipe can be considered. Similar calculations for smaller pipe sizes give the following results:

Table 5.5 Comparison of Pressure Drop Calculations

Nominal size	Schedule No.	ID of pipe, mm	Δp, kPa
677 mm	—	677	8.25
600 mm (24 in)	20	590.55	15.558
500 mm (20 in)	20	488.95	37.525

Select 600 mm, SCH-20 pipe size for the given duty.

Example 5.3 Calculate the pipe size based on following data. Fluid flowing through pipe is carbon monoxide. Discharge pressure of carbon monoxide required from the pipe is atmospheric.

Available pressure at inlet of pipe = 50 kPa g
Length of pipe = 4 km
Flow rate of CO = 1500 kg/h
Temperature of gas = 50°C
No. of gate valves in pipeline = 2
No. of 45° elbows = 3
No. of 90° elbows = 6
Viscosity of CO = 0.018 mPa · s or cP

Solution

Density of carbon monoxide (CO) gas:
Molar mass of CO, $M = 28$

$$\rho = \frac{pM}{RT}$$

$$= \frac{pM}{T} \times \frac{T_s}{p_s V_s} = \frac{1 \times 28}{(273 + 50)} \times \frac{273}{(1 \times 22.414)}$$

$$= 1.0558 \text{ kg/m}^3$$

Pressure drop in straight pipe for steel material and for turbulent region:

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} \dot{m}^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \tag{5.5}$$

Let $v = 5$ m/s which implies $D_i = 0.317$ m.
For the first trial calculations let the nominal size be 12 in NB (300 mm), SCH-40 pipe having inside $D_i = 304.8$ mm.

$$\frac{\Delta p_1}{L} = 4.07 \times 10^{10} \left(\frac{1500}{3600}\right)^{1.84} (0.018 \times 10^{-3})^{0.16} \times (304.8)^{-4.84} \times (1.0558)^{-1}$$

$$= 1.2724 \times 10^{-3} \text{ kPa/m} \equiv 1.2724 \text{ Pa/m}$$

Reynolds numbers, $Re = \dfrac{4\dot{m}}{\pi D_i \mu} = \dfrac{4 \times \left(\dfrac{1500}{3600}\right)}{\pi \times 0.3048 \times 0.018 \times 10^{-3}}$

= 96 697 > 10 000, i.e., CO is flowing in turbulent region

$$\text{Velocity of gas, } v = \frac{\dot{m}}{\left(\rho \times \frac{\pi}{4} D_i^2\right)} = \frac{1500/3600}{[1.0558 \times (\pi/4)(0.3048)^2]} = 5.4 \text{ m/s}$$

For gate valve,

$$\Delta p_{E1} = \frac{K_i \rho v^2}{2} = \frac{0.17 \times 1.0558 \times 5.4^2}{2} = 2.617 \text{ N/m}^2 \text{ per gate valve}$$

$$\text{Similarly for 45° elbows, } \Delta p_{E2} = \frac{K_i \rho v^2}{2} = \frac{0.35 \times 1.0558 \times 5.4^2}{2}$$

$$= 5.388 \text{ N/m}^2 \text{ per 45° elbow}$$

$$\text{For 90° elbows, } \Delta p_{E3} = \frac{K_i \rho v^2}{2} = \frac{0.75 \times 1.0558 \times 5.4^2}{2}$$

$$= 11.545 \text{ N/m}^2 \text{ per 90° elbow}$$

Total pressure drop in pipe line

$$\Delta p_t = 1.2724 \times 4000 + 2.617 \times 2 + 5.388 \times 3 + 11.545 \times 6$$

$$\Delta p_t = 5180.27 \text{ Pa} \equiv 5.18 \text{ kPa} \equiv 0.0511 \text{ atm}$$

For 300 mm NB SCH-40 pipe, pressure drop in piping is very low. Hence, to reduce the fixed capital investment, smaller pipe diameter can be considered. Similar calculations of smaller pipe diameters gives the following results:

Table 5.6 Comparison of Pressure Drop Calculations

Nominal size	Schedule No.	ID of pipe, mm	Δp_t, kPa	Remarks
100 mm (4 in)	SCH-40	102.26	1012.38	Very high
150 mm (6 in)	SCH-40	154.05	139.75	High
200 mm (8 in)	SCH-40	202.72	37.10	Reasonable
300 mm (12 in)	SCH-40	304.80	5.18	Very low

Considering fixed capital investment, operating cost and safety margin in design, 200 mm (8 in) SCH-40 pipe can be selected for the given application. For this size of pipe, velocity of gas through pipe is equal to 12.23 m/s which is acceptable (refer Table 5.1).

Example 5.4 100 000 kg/h of water is to be transferred from canal to reservoir by gravity. Maximum height of water level above the discharge of pipe in reservoir will be 2 m. Difference between minimum level of water in canal and maximum level of water in reservoir is 6 m.

Length of pipe = 3000 m

Equivalent length of pipe for fittings and valves = 200 m

Maximum temperature of water = 40°C

Density of water at 40°C = 993 kg/m^3

Viscosity of water at 40°C = 0.67 mPa · s or cP

Surface roughness for carbon steel, $\varepsilon = 0.0457$ mm
Surface roughness for concrete, $\varepsilon = 1.2$ mm
Material of pipe is carbon steel. Determine the suitable pipe size. If material of pipe is concrete, will there be any change in pipe size required?

Solution

Maximum allowable pressure drop in pipe = 6 m WC
Δp_{max} = 6 m WC = 58.84 kPa = 0.58 atm
Let the velocity of water through pipe $v = 0.5$ m/s (For first trial calculations)

$$v = 0.5 \text{ m/s} = \frac{\dot{m}/\rho}{(\pi/4)\, D_i^2} = \frac{(100\,000)/3600}{993 \times (\pi/4)\, D_i^2}$$

$$D_i = 0.2669 \text{ m} \equiv 266.9 \text{ mm}$$

Select 300 mm NB, SCH-40 carbon steel pipe.
ID of pipe, $D_i = 304.8$ mm, $\mu = 0.67 \times 10^{-3}$ kg/(m · s)

Reynolds number, $$Re = \frac{4\dot{m}}{\pi D_i \mu} = \frac{4 \times (100\,000/3600)}{\pi \times 0.3048 \times 0.67 \times 10^{-3}} = 173\,188$$

$$\frac{\varepsilon}{D_i} = \frac{0.0457}{304.8} = 1.499 \times 10^{-4}$$

From Fig. 5.1, $f = 0.0042$

Pressure drop in pipe, $$\Delta p = \frac{32 f\, \dot{m}^2}{\pi^2 \rho g_c D_i^5} \times L \qquad (5.2)$$

$$\Delta p = \frac{32 \times 0.0042 \times (100\,000/3600)^2 \times (3000 + 200)}{\pi^2 \times 993 \times 1 \times 0.3048^5}$$

$$\Delta p = 12\,871.2 \text{ Pa} \equiv 12.871 \text{ kPa} < \Delta p_{max}$$

For the next smaller size of pipe, i.e., for 250 mm NB, SCH-40 pipe, $D_i = 254.5$ mm,

$$Re = 207\,417.3$$

$$\frac{\varepsilon}{D_i} = 1.7956 \times 10^{-4}, \quad f = 0.0041 \text{ and } \Delta p = 30.959 \text{ kPa} < \Delta p_{max}$$

Select 250 mm NB SCH-40 carbon steel pipe.
If pipe material is concrete, then for the same size

$$\frac{\varepsilon}{D_i} = \frac{1.2}{254.5} = 4.715 \times 10^{-3} \text{ and } Re = 207\,417.3$$

From Fig. 5.1, $f = 0.0077$
Pressure drop $\Delta p = 58.142$ kPa $\approx \Delta p_{max}$
Thus calculated pressure drop is nearly equal to the maximum allowable pressure drop. For safer design, next higher size of concrete pipe, i.e., 300 mm (12 in) pipe, should be used.

Example 5.5 An organic liquid is discharged at the rate of 5000 kg/h from a reactor to a storage tank at 50°C as shown in Fig. 5.2. Reactor is under pressure at 600 kPa g. Density of the organic liquid is 930 kg/m^3 and viscosity is 0.91 mPa · s or cP. Assume negligible flashing of the organic liquid across the control valve.

Fig. 5.2 *Liquid Flow System*

Piping system details:

Linear length of straight pipe = 50 m
No. of 90° elbows of standard radius = 6 (All are not shown in Fig. 5.2.)
No. of Tees = 2
Pressure drop in orifice meter = 40 kPa
No. of gate valves = 4
No. of globe valve = 1
No. of flow control valve = 1

Determine the pipe size. Assume it to be uniform throughout. Also find the residual pressure drop that must be taken by the flow control valve.

Solution

Flow rate of liquid, $\dot{m}$ = 5000 kg/h = 1.4 kg/s
Volumetric flow rate of liquid:

$$q_V = \frac{\dot{m}}{\rho} = \frac{5000/3600}{930}$$

$$= 1.4934 \times 10^{-3}\ \text{m}^3/\text{s}$$

Based on Table 5.1, select velocity of liquid to be 1.8 m/s.
Inside diameter of pipe required based on selected velocity

$$D_i = \sqrt{\frac{4 \times q_v}{\pi \times v}}$$

$$= \sqrt{\frac{4 \times 1.4934 \times 10^{-3}}{\pi \times 1.8}} = 0.0325\ \text{m} \equiv 32.5\ \text{mm}$$

Selected 40 mm (1.5 in) NB, SCH-40 pipe having D_i = 40.894 mm

Reynolds number, $Re = \dfrac{4\dot{m}}{\pi D_i \mu} = \dfrac{4 \times 1.4}{\pi \times 0.040\,894 \times 0.91 \times 10^{-3}} = 47\,900$

(i.e., turbulent flow)

For the turbulent flow in commercial steel pipe

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} \dot{m}^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \tag{5.5}$$

$$= 4.07 \times 10^{10} \left(\frac{5000}{3600}\right)^{1.84} (0.91 \times 10^{-3})^{0.16} \times (40.894)^{-4.84} \times (930)^{-1}$$

$$= 0.413\,65 \text{ kPa/m}$$

Linear length of pipe = 50 m

Table 5.7 Equivalent Lengths of Fittings and Valves

Type	No.	Pipe ID, mm	L_e, m (From Table 5.3)
90° Elbows	6	40.894	$6(30D_i)$ = 7.36
Tees	2	40.894	$2(22D_i)$ = 1.8
Gate valves	4	40.894	$4(10D_i)$ = 1.636
Total			10.796

Total pressure drop

$$\Delta p_t = 0.413\,65\,(50 + 10.796) + \text{Pressure drop in orifice meter}$$

$$= 0.413\,65\,(50 + 10.796) + 40 = 65.15 \text{ kPa}$$

Pressure balance:

Pressure in reactor $- \Delta p_t - \Delta p_{\text{control valve}}$ = Pressure in storage tank

$$(600 + 101.325) - 65.15 - \Delta p_c = 101.325$$

$$\Delta p_c = 534.85 \text{ kPa} \approx 5.35 \text{ bar}$$

This residual pressure drop should be offered by the control valve. Specifications of the control valve should be prepared accordingly.

5.3 PROCESS DESIGN OF FLUID MOVING DEVICES

It includes process design of pump, fan, blower, and compressor.

5.3.1 Process Design of Pump

Pump is a device used in flow system of liquid to increase the mechanical energy of the flowing liquid. Pumps are broadly divided into two types; (i) centrifugal pumps and (ii) positive displacement pumps. The following terminologies are used with pump:

1. Capacity of pump: Flow rate (q_v) of fluid created by the pump is known as capacity of pump. In SI units, capacity is expressed in m^3/h or L/s.

2. Total Dynamic Head: Total dynamic head H of a pump is the difference between total discharge head h_d and total suction head h_s.

$$H = h_d - h_s \tag{5.10}$$

Before installation of pump it is possible to estimate the total discharge head h_d by using following equation:

$$h_d = h_{sd} + h_{fd} \tag{5.11}$$

where, h_{sd} = Static discharge head = $p' \pm Z'$ (5.12)

p' = Absolute pressure over free surface of liquid in receiver

Z' = Vertical distance between free surface of liquid in receiver and centerline of pump placed horizontally (For vertical pump Z' is distance between free surface of liquid and eye of suction of impeller).

$h_{sd} = p' + Z'$, if pump is below the level of free surface of liquid in receiver and

$h_{sd} = p' - Z'$, if it is placed above the free surface of liquid in receiver.

h_{fd} = Discharge friction head = frictional loss in discharge line.

During operation, total discharge head h_d can be determined by following equation:

$$h_d = h_{gd} + \text{atm pressure} + h_{vd} \tag{5.13}$$

where, h_{gd} = Discharge gauge pressure measured by pressure gauge. If pressure is below atmospheric, vacuum gauge reading is used for h_{gd} in Eq. (5.13) but with negative sign.

h_{vd} = Velocity head at the point of gauge attachment in discharge line

Before installation of pump, total suction head can be estimated by following equation:

$$h_s = h_{ss} - h_{fs} \tag{5.14}$$

where, h_{ss} = Static suction head = $p \pm Z$

p = Absolute pressure over free surface of liquid at source

Z = Vertical distance between free surface of liquid at source and centerline of horizontal pump (or suction eye impeller for vertical pump)

$h_{ss} = p + Z$, if pump is going to be installed below the free surface of liquid

$h_{ss} = p - Z$, if pump is going to be installed above the free surface of liquid

h_{fs} = Friction loss in suction line

In existing installation, total suction head h_s can be determined by following equation:

$$h_s = \text{atm pressure} + h_{gs} + h_{vs} \tag{5.15}$$

where, h_{gs} = suction gauge pressure,

h_{vs} = velocity head at the point of gage attachment.

In the above equations, all pressure heads are in terms of liquid heads (LC), flowing in the system.

3. Net Positive Suction Head (NPSH): Net positive suction head is the excess of sum of velocity head and pressure head in suction line over the vapour pressure head of liquid. When pump installation is designed, available net positive suction head $(NPSH)_A$ must be equal to or greater than net positive suction head required; $(NPSH)_R$.

$(NPSH)_R$ is normally specified by the pump supplier, while based on the installation of pump, $(NPSH)_A$ should be calculated and specified by the process engineer. Theoretically, $(NPSH)_A$ should be greater than zero to avoid cavitation.

$(NPSH)_R$ depends on properties of liquid, the total liquid head, pump speed, capacity and impeller design. Practical curves of $(NPSH)_R$ vs capacity and speed of pump are supplied by the pump manufacturer. Figures 5.3 and 5.4 can be used as a guideline to find the value of $(NPSH)_R$ for centrifugal pump handling water at temperatures below 100°C and above 100°C, respectively.

Fig. 5.3 *Net Positive Suction Head for High Pressure Centrifugal Hot-Water Pumps*[8] *(Reproduced with the Permission of the Hydraulic Institute (USA)*

When $(NPSH)_A$ is less than $(NPSH)_R$, cavitation can take place and bubbles of vapour form in the suction line. Eventually these bubbles collapse inside the casing of the pump when pressure is exerted on them by the impeller of the pump. Such collapse of bubbles can cause severe erosion and damage to the pump. It may form minor cavities on the inside surface of the casing and of the impeller. Hence, this phenomenon is called *cavitation*.

When a pump installation is being designed, the available net positive suction head $(NPSH)_A$ can be calculated by the following equation:

$$(NPSH)_A = h_{ss} - h_{fs} - p_v \tag{5.16}$$

where, h_{ss} = Static suction head, m LC = $p \pm Z$

h_{fs} = Friction loss in suction line, m of liquid column (LC)

p_v = Vapour pressure of liquid at suction temperature expressed in m of liquid column (LC)

For existing installation, $(NPSH)_A$ can be determined by following equation:

$$(NPSH)_A = \text{atm pressure} + h_{gs} - p_v + h_{vs} \tag{5.17}$$

where, h_{gs} = suction gauge pressure, m of liquid column (LC)

p_v = vapour pressure of liquid at suction temperature expressed in m of liquid column (LC)

Fig. 5.4 *Temperature Correction Chart for Net Positive Suction Head Requirement for Centrifugal Hot Water Pumps*[8]
(Reproduced with the Permission of the Hydraulic Institute, USA)

h_{vs} = Velocity head at the point of gauge attachment, m of liquid column (LC)

As a general guide $(NPSH)_A$ should preferably be above 3 m for pump capacities up to a flow rate of 100 m^3/h, and 6 m above this capacity.

For a given system, if $(NPSH)_A$ is less than $(NPSH)_R$, following remedial measures are recommended:

(a) Change the location of the pump to improve $(NPSH)_A$. In other words, positive suction head (h_{ss}) may be increased.
(b) Provide jacketed cooling in the suction line to decrease the vapour pressure (p_v) of the liquid.
(c) Reduce operating speed of the pump; thereby specific speed of the pump is reduced and its $(NPSH)_R$ is less.

5.3.2 NPSH Requirement for Liquids Saturated with Dissolved Gases

In many situations, the liquid to be pumped is saturated with gases, which have definite solubilities in the liquid. When a suction system for such a liquid is to be designed for a centrifugal pump, $(NPSH)_A$ calculations differ from Eqs. (5.16) or (5.17).

Pumping of cooling water (saturated with air), pumping of condensate from a knock-out drum of a compressor, pumping of solution from an absorber, etc., can be cited as examples of such situations where the liquids are saturated with gases.

Dissolved gases start desorbing when the pump is started and suction is generated at the pump eyes. Normally, a pump can tolerate 2 to 3% flashed gases at the pump eye without encountering cavitations. If the design of the suction system is made to restrict about 2.5% flash, it is considered safe for the pump operation.

In Eq. (5.16) suction source pressure is the system pressure (p) minus the vapour pressure (p_v) for a normal liquid having practically no dissolved gases. For a liquid saturated with dissolved gases, p_v is replaced by p_{va} which is called *artificial liquid vapour pressure*. For evaluation of p_{va}, following procedure is recommended:

1. Calculate molar mass of the gas mixture, dissolved in the liquid.
2. Calculate mass fraction (w_o) of the dissolved gas mixture.
3. Calculate psuedo-critical properties of the dissolved gas mixture, if system pressure is high.
4. Calculate specific volume of the dissolved gas mixture (V_{Ga}) at the operating conditions.

Steps (1) to (4) can be avoided if the solubility of the gas mixture in the liquid (such as air in water) is known from the literature.

5. Calculate volume fraction of the dissolved gas (GVP) in a hypotheoretical gas-liquid mixture. Consider one unit mass of the liquid in which the gas mixture is dissolved. If GVP is less than or equal to 2.5%, Eq. (5.16) can be safely used to calculate $(\text{NPSH})_\text{A}$ using vapour pressure (p_v) of the liquid at the operating temperature. If higher than calculate volume fraction (a) of flashed gas mixture (as pressure is lowered) over the liquid, saturated with the dissolved gas mixture, using the following equation[9]:

$$a = 1 \Bigg/ \left[\frac{\left(\dfrac{p}{p_0} - \dfrac{p_v}{p_0}\right)^2 \left(1 - \dfrac{p_v}{p_0}\right)}{\left(\dfrac{V_{Ga}}{V_L}\right)\left(\dfrac{p}{p_0}\right)\left(1 - \dfrac{p}{p_0}\right)} + 1 \right] \tag{5.18}$$

where, p = liquid pressure at pump eye, kPa
p_v = vapour pressure of liquid at the operating temperature, kPa
p_0 = system pressure, kPa
V_{Ga} = specific volume of the dissolved gas mixture, m^3/kg [see Step (4) above]
V_L = specific volume of the liquid at the operating conditions, m^3/kg

Equation (5.18) assumes that the dissolved gas mixture follows ideal gas law, Dalton's law and Henry's law.

6. Calculate a for different value of p. Draw a graph of a vs p. Read p corresponding to $a = 0.025$ which is called p_{va}. Alternately by trial and error, calculate p_{va}, for $a = 0.025$.
7. Use Eq. (5.16) and insert p_{va} in place of p_v and calculate $(\text{NPSH})_\text{A}$. The above procedure is well exemplified in Example 5.10.

5.3.3 Power Required for Pumping

Power required for pumping an incompressible fluid is given by equation:

$$P = \frac{H q_v \rho}{3.67 \times 10^5 \times \eta} \tag{5.19}$$

where, P = Power required, kW
H = Total dynamic head, m of liquid column
q_v = Capacity, m^3/h
ρ = Fluid density, kg/m^3
η = Efficiency of pump

Example 5.6 A centrifugal pump draws benzene from an overhead tank. Operating vacuum in the tank is 700 Torr. Vertical distance between free surface of liquid in tank and centerline of pump is 12 m. Maximum operating temperature is 50°C. Vapour pressure of benzene at 50°C is 280 Torr. Density of benzene at 50°C is 870 kg/m^3. Frictional loss in suction line of pump is 1 m of benzene column. Calculate the $(NPSH)_A$ of centrifugal pump.

Solution

$$(NPSH)_A = h_{ss} - h_{fs} - p_v \tag{5.16}$$

$$h_{ss} = p + Z$$

$$p = 760 - 700 = 60 \text{ Torr a}$$

$$1 \text{ atm} = 10.33 \text{ m WC}$$

$$p = \frac{60}{760} \times 10.33 \times \frac{\text{Density of water}}{\text{Density of benzene at 50°C}} = \frac{60}{760} \times 10.33 \times \frac{1000}{870}$$

$$= 0.937 \text{ m LC}$$

$$p_v = \frac{280}{760} \times 10.33 \times \frac{1000}{870} = 4.37 \text{ m LC}$$

$$(NPSH)_A = p + Z - h_{fs} - p_v = 0.937 + 12 - 1 - 4.37 = 7.567 \text{ m LC}$$

Example 5.7 A centrifugal pump is drawing methanol from an underground tank. Operating pressure in the underground tank is 1 atm. Vertical distance between centreline of pump and free surface of liquid in the tank is 3 m. Maximum operating temperature is 50° C. Vapour pressure and density of methanol at 50° C are 400 Torr and 785 kg/m^3, respectively. Friction loss in suction line is 1 m of liquid column. Calculate the $(NPSH)_A$ of centrifugal pump.

Solution

$$(NPSH)_A = h_{ss} - h_{fs} - p_v = p - Z - h_{fs} - p_v \tag{5.16}$$

$$p = \frac{101.325 \times 10^3}{(785 \times 9.81)} = 13.16 \text{ m LC}$$

$$p_v = \frac{400}{760} \times \frac{101.325 \times 10^3}{(785 \times 9.81)} = 6.925 \text{ m LC}$$

$$(\text{NPSH})_A = 13.16 - 3 - 1 - 6.925 = 2.235 \text{ m LC}$$

Note: Effect of vapour pressure (p_v) of the fluid is significant in calculation of $(\text{NPSH})_A$. Selection of the pump is critical. As a general guideline $(\text{NPSH})_A$ should be greater than 3 m for a centrifugal pump.

Example 5.8 Installation of pump is to be decided for the a pump which will handle hot water at a temperature of 176.7°C and a suction pressure of 1.034 MPa g. Capacity of pump will be 10 m^3/h. It is planned to use a centrifugal pump with single suction for this applications. Speed of its impeller will be 2900 rpm.

Find the minimum distance that must be kept between free surface of liquid at source and centreline of horizontal centrifugal pump.

Solution

$$(\text{NPSH})_A \geq (\text{NPSH})_R$$

From Figs 5.3 and 5.4, data for centrifugal pump having a speed of 3550 rpm are given. Assume these data to be applicable for 2900 rpm (a safer design). For 3550 rpm, 10 m^3/h, 176.7°C and $p < 35$ bar:

$$(\text{NPSH})_R = 3.048 + 3.6576 \text{ m (additional)}$$
$$= 6.7 \text{ m}$$
$$(\text{NPSH})_A \geq 6.7 \text{ m}$$
$$p \pm Z - h_{fs} - p_v \geq 6.7 \text{ m}$$
$$p = 1.034 \text{ MPa g} = 1.034 \times 10^6 \text{ Pa g}$$
$$1 \text{ atm} = 101.325 \text{ kPa}$$
$$p = (1.034 \times 10^6 + 101.325 \times 10^3) \text{ Pa a}$$
$$p = 1.135\,325 \times 10^6 \text{ Pa a}$$

Vapour pressure of hot water at 176.7°C = 0.928 MPa

$$p_v = 0.928 \times 10^6 \text{ Pa}$$

Density of water at 176.7°C, $\rho = 890$ kg/m^3

$$p - p_v = 0.207\,325 \times 10^6 = \frac{0.207\,325 \times 10^6}{\rho \times g}$$

$$= \frac{0.207\,325 \times 10^6}{890 \times 9.81} = 23.746 \text{ m WC}$$

Select the pipe size and piping system such that $h_{fs} \leq 1$ m.

$$-Z - 1 + 23.746 \geq 6.7 \text{ m}$$

(if pump is installed above the free surface of liquid at source)

or $\quad Z \leq 16.046$ m

Pump can be installed maximum up to an elevation 16.046 m above the free surface of liquid at source to avoid cavitation. Pump can be installed at any distance, if it is to be installed below the free surface of liquid at source.

Example 5.9 In the previous example, if hot water is saturated at 176.7°C temperature at source, what will be the minimum height that must be kept between free surface of liquid at source and centreline of selected pump? Type, speed and capacity of pump are same as that for previous example.

Solution

For the single suction centrifugal pump having speed 2900 rpm and capacity 10 m^3/h,

$$(NPSH)_R = 6.7 \text{ m}$$

$$(NPSH)_A \geq (NPSH)_R \text{ or } (NPSH)_A \geq 6.7 \text{ m}$$

Here hot water is saturated at 176.7° C at source.

$$p = p_v$$

In this case pump must be installed below the free surface of liquid at source.

$$(NPSH)_A = p + Z - h_{fs} - p_v = Z - h_{fs} \geq 6.7 \text{ m}$$

Let the velocity of water in suction line = 1 m/s

$$q_v = v \times (\pi/4)D_i^2 = 10 \text{ m}^3/\text{h} \equiv 2.7778 \times 10^{-3} \text{ m}^3/\text{s}$$

$$D_i = \sqrt{\frac{4 \times q_v}{\pi \times v}} = \sqrt{\frac{4 \times 2.7778 \times 10^{-3}}{\pi \times 1}} = 0.059\,47 \text{ m}$$

From the table of standard pipe select 62.5 mm NB SCH-40 steel pipe having $D_i = 62.71$ mm

$$Re = \frac{D_i v \rho}{\mu}$$

ρ of water at 176.7°C = 890 kg/m^3

Velocity based on $D_i = 62.71$ mm, $v = 0.9$ m/s

Viscosity of water at 176.7°C and at saturation pressure, 9.08 atm a must be determined.

Effect of pressure on viscosity of liquid is insignificant up to about 50 atm.

For reduced temperature $T_r < 0.75$, relation between viscosity of liquid and temperature is given by equation

$$\ln \mu_w = A + \frac{B}{(T + C)}, \quad T \text{ in } K \tag{5.20}$$

where, $C = 17.71 - 0.19\, T_B$, T_B is normal boiling point (i.e., at 1 atm) in K

For water, $C = 17.71 - 0.19\,(100 + 273) = -53.16$

From Appendix 14 of Ref. 10:

μ_w at 149°C = 0.185 cP, μ_w at 115.6°C = 0.242 cP or mPa · s

$$\ln (0.185) = A + \frac{B}{[(149 + 273) - 53.16]},$$

$$\ln (0.242) = A + \frac{B}{[(115.6 + 273) - 53.16]}$$

$A + 0.0027\,112\,B = -1.6874, \quad A + 0.002\,981\,B = -1.4188$

Solving simultaneous equations,

$A = -4.3865, \quad B = 995.55$

$$\ln \mu_w = \frac{995.55}{[(176.7 + 273) - 53.16]} - 4.3865$$

$\mu_w = 0.1532$ cP or mPa · s at 176.7°C

$\mu_w = 0.1493$ mPa · s at 180°C from JSME Steam Tables – a check

$$Re = \frac{0.062\,71 \times 0.9 \times 890}{0.1532 \times 10^{-3}} = 327\,876.7 \text{ (i.e. turbulent)}$$

For turbulent flow through commercial steel pipe

$$\frac{\Delta p}{L} = 4.07 \times 10^{10}\, \dot{m}^{1.84}\, \mu^{0.16}\, D_i^{-4.84}\, \rho^{-1} \tag{5.5}$$

$q_v = 10\ \text{m}^3/h = 2.7778 \times 10^{-3}\ \text{m}^3/\text{s}$

$\dot{m}$ = mass flow rate = $q_v \times \rho = 2.7778 \times 10^{-3} \times 890 = 2.4722$ kg/s

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} \times (2.4722)^{1.84} \times (0.1532 \times 10^{-3})^{0.16} \times (62.71)^{-4.84} \times (890)^{-1}$$

$$\frac{\Delta p}{L} = 0.1186 \text{ kPa/m}$$

For 6.7 m elevation,

Let L = actual length of pipe = 7 m, including horizontal length for pump suction. Assuming three 90° elbows and one globe valve in the suction line.

Table 5.8 Equivalent Lengths of Fitting and Valve

Valve or Fitting	No.	L_e
90° elbows (standard)	3	3 (30 D_i) = 5.64 m
Globe valve (open)	1	350 D_i = 21.95 m

$L \cong 7 + 5.64 + 21.95 \cong 34.59$ m

$$\Delta p = 4.102 \text{ kPa} = \frac{4.102 \times 10^3}{\rho \times g} \text{m Liquid Column (i.e., WC at 176.7°C)}$$

$$h_{fs} = \Delta p = \frac{4.102 \times 10^3}{890 \times 9.81} = 0.47 \text{ m of WC}$$

$Z \geq (6.7 + 0.47)$ m or $Z \geq 7.17$ m

Minimum elevation required between free surface of water at source and centreline of pump = 7.17 m

Example 5.10 Carbon dioxide gas (> 99.5% pure) from ammonia plant at 1.01 atm a and 40°C is washed with cooling water in a packed column before compression in urea plant. Cooling water from bottom of the column is pumped back to cooling tower. Solubility of carbon dioxide in water is

0.0973 kg/100 kg water at 1 atm and 40°C. Find the volume fraction of carbon dioxide over cooling water. Also calculate artificial liquid vapour pressure.

Solution

Molar mass of carbon dioxide, $M = 44.0095$

From JSME Stream Tables, vapour pressure of water at 40°C,

$$p_v = 0.073\,75 \text{ bar}$$
$$= 0.0728 \text{ atm}$$

Mass fraction of carbon dioxide in the liquid

$$w_0 = \frac{0.0973 \times 1.01}{(100 + 0.0973)}$$
$$= 9.8177 \times 10^{-4}$$

$$V_{\text{Ga}} = \frac{9.8177 \times 10^{-4} \times 0.082\,06 \times 313.15}{44.0095 \times 1.01} \tag{5.18}$$
$$= 5.676 \times 10^{-4} \text{ m}^3/\text{kg H}_2\text{O}$$

From JSME Steam Tables

$$V_L = 1.0078 \text{ m}^3/\text{kg H}_2\text{O at } 40°\text{C}$$

$$\text{GVP} = \frac{5.676 \times 10^{-4} \times 100}{(1.0078 + 5.676 \times 10^{-4})}$$
$$= 0.0563\%$$

Thus, volume per cent of carbon dioxide is only 0.0563% (< 2.5%) over cooling water and there is no danger of pump cavitation.

$$p_o = 1.01 \text{ atm}$$

$$a = 1 \Bigg/ \left[\frac{\left(\frac{p}{1.01} - \frac{0.0728}{1.01}\right)^2 \left(1 - \frac{0.0728}{1.01}\right)}{\left(\frac{5.676 \times 10^{-4}}{1.0078}\right)\left(\frac{p}{1.01}\right)\left(1 - \frac{p}{1.01}\right)} + 1 \right] \tag{5.18}$$

By trial and error,

For $p = 0.145$ atm, $a = 0.0247$

Hence, $p_{va} = 0.145$ atm

Thus, if vapour pressure of water (0.0728 atm) is used instead of p_{va} (i.e., 0.145 atm), $(\text{NPSH})_\text{A}$ would have been erroneous by 0.75 m H_2O.

Note: Above calculations indicate that at higher temperature and lower suction pressure, p_{va} could have significantly affected $(\text{NPSH})_\text{A}$.

Example 5.11 Benzene at 37.8°C is pumped through the system at a rate of 9.09 m^3/h with the help of a centrifugal pump. The reservoir is at atmospheric pressure. Pressure at the end of discharge line is 345 kPa g. The discharge head is 3.05 m and the pump suction head is 1.22 m above the level of liquid in reservoir. The friction loss in suction line is 3.45 kPa and that in the discharge line

is 37.9 kPa. The mechanical efficiency of the pump (η) is 0.6. The density of benzene is 865 kg/m^3 and its vapour pressure at 37.8°C is 26.2 kPa.
Calculate (a) $(\text{NPSH})_A$ and (b) power required by centrifugal pump.

Solution

(a) $(\text{NPSH})_A = h_{ss} - h_{fs} - p_v = p - Z - h_{fs} - p_v$

$$p = 1 \text{ atm} = \frac{101.325 \times 10^3}{865 \times 9.81} = 11.94 \text{ m LC}$$

$Z = 1.22$ m (suction)

$$h_{fs} = 3.45 \text{ kPa} = \frac{3.45 \times 10^3}{865 \times 9.81} = 0.4066 \text{ m LC}$$

$p_v = 26.2 \text{ kPa} \equiv 3.088 \text{ m LC}$

$(\text{NPSH})_A = 11.94 - 1.22 - 0.4066 - 3.088 = 7.2254$ m of LC

(b) Power required by centrifugal pump

$$P = \frac{Hq_v\rho}{3.67 \times 10^5 \times \eta} \tag{5.19}$$

p' = Pressure at discharge = 345 + 101.325 = 446.325 kPa a

$$\equiv \frac{446.325 \times 10^3}{865 \times 9.81} \equiv 52.6 \text{ m LC}$$

$Z' = 3.05 - 1.22 = 1.83$ m
$h_{sd} = 52.6 + 1.83 = 54.43$ m LC

$$h_{fd} = 37.9 \text{ kPa} = \frac{37.9 \times 10^3}{865 \times 9.81} = 4.47 \text{ m LC}$$

$h_d = 58.9$ m LC
$h_s = h_{ss} - h_{fs} = p - Z - h_{fs} = 11.94 - 1.22 - 0.4066 = 10.31$ m
$H = 58.9 - 10.31 = 48.59$ m

$$P = \frac{48.59 \times 9.09 \times 865}{3.67 \times 10^5 \times 0.6} = 1.735 \text{ kW}$$

Example 5.12 It is proposed to pump 10 000 kg/h of saturated toluene at 114°C and 1.1 atm a from the reboiler of a distillation tower to a second distillation unit without cooling. If the friction loss in the line between the reboiler and pump is 7 kPa and the density of toluene is 866 kg/m^3, how much liquid level in the reboiler is to be maintained to give a net positive suction head of 2.5 m? Calculate the power required to drive the pump if the pump is to elevate the toluene to 10 m to a second unit at atmospheric pressure. Assume friction loss in the discharge line to be 35 kPa. Pump efficiency is 62%.

Solution

In this case, the pump is installed below the liquid level.

$$(\text{NPSH})_A = p + Z - h_{fs} - p_v = 2.5 \text{ m}$$

Liquid in reboiler is saturated liquid at reboiler conditions. Therefore, $p = p_v$

$$Z - h_{fs} = 2.5 \text{ m}$$

$$h_{fs} = 7 \text{ kPa} = \frac{7 \times 10^3}{\rho \times g} = \frac{7000}{866 \times 9.81}$$

$$= 0.824 \text{ m LC}$$

$$Z = 2.5 + 0.824 = 3.324 \text{ m LC}$$

Pump must be installed at a level 3.324 m below the free surface of liquid in reboiler.

Power required to drive the pump

$$P = \frac{Hq_v\rho}{3.67 \times 10^5 \times \eta} \tag{5.19}$$

$$p' = 1 \text{ atm} = 101.325 \text{ kPa} = \frac{101325}{\rho \times g} \text{ m LC}$$

$$= \frac{101325}{866 \times 9.81} = 11.927 \text{ m LC}$$

$$Z' = 10 \text{ m}$$

$$h_{sd} = 10 + 11.927 = 21.927 \text{ m}$$

$$h_{fd} = 35 \text{ kPa} = \frac{35\,000}{866 \times 9.81} = 4.12 \text{ m LC}$$

$$h_d = 21.927 + 4.12 = 26.047 \text{ m}$$

Total suction head $h_s = h_{ss} - h_{fs} = p + Z - h_{fs}$ (5.14)

$$p = 1.1 \text{ atm} = \frac{111.4575 \times 10^3}{866 \times 9.81} = 13.12 \text{ m LC}$$

$$h_s = 13.12 + 3.324 - 0.824 = 15.62 \text{ m LC}$$

$$H = h_d - h_s = 26.047 - 15.62 = 10.427 \text{ m LC}$$

$$q_v = \frac{10\,000}{866} = 11.5473 \text{ m}^3/\text{h}$$

$$P = \frac{10.427 \times 11.5473 \times 866}{3.67 \times 10^5 \times 0.62} = 0.4582 \text{ kW}$$

Example 5.13 A centrifugal pump is drawing water from an overhead tank, exposed to atmosphere. Vertical distance between free surface of liquid in the tank and centerline of pump is 10 m. Capacity of centrifugal pump is 10 000 kg/h. Maximum operating temperature is 50°C. Vapour pressure of water at 50°C is 92.51 Torr. Total length of suction pipe 10.5 m, having two 90° elbows.

Determine the size of suction pipe and $(NPSH)_A$ of centrifugal pump. Material of pipe is carbon steel. Density of water = 1000 kg/m^3, Viscosity of water = 0.558 mPa · s (or cP).

Solution

In Table 5.1, velocity of water in suction line (v) is recommended in the range of 0.3 to 1.5 m/s.

Let velocity of water in suction line, v = 1 m/s

$$\frac{\pi}{4}D_i^2 = \frac{q_v}{v} = \frac{10 \times (1/3600)}{1}$$

$$D_i = 0.0595 \text{ m}$$

Select 62.5 mm NB SCH-40 pipe, D_i = 62.7 mm

$$v = \frac{q_v}{\frac{\pi}{4}D_i^2} = \frac{(10\,000/3600)(1/1000)}{\frac{\pi}{4} \times (0.0627)^2} = \frac{2.778 \times 10^{-3}}{\frac{\pi}{4} \times (0.0627)^2}$$

Actual velocity, v = 0.9 m/s through the selected pipe.

$$Re = \frac{D_i v \rho}{\mu} = \frac{0.0627 \times 0.9 \times 1000}{0.558 \times 10^{-3}}$$

$$= 101\,129 \quad \text{(i.e., turbulent flow)}$$

Surface roughness of carbon steel, ε = 0.0457 mm

$$\frac{\varepsilon}{D_i} = \frac{0.0457}{62.7} = 0.000\,729$$

From Fig. 5.1, f = 0.005

$$\frac{\Delta p}{L} = \frac{32 f\, \dot{m}^2}{\pi^2 \rho\, g_c D_i^5} = \frac{32 \times 0.005 \times (10\,000/3600)^2}{\pi^2 \times 1000 \times 1 \times 0.0627^5} \tag{5.2}$$

$$= 129 \text{ Pa/m}$$

For 90° elbows, additional pressure drop

$$\Delta p_E = \frac{K \rho v^2}{2 g_c} \tag{5.9}$$

$$\Delta p_E = \frac{0.75 \times 1000 \times (0.9)^2}{2} = 303.75 \text{ Pa per elbow}$$

Total pressure drop in suction line

$$\Delta p_t = 129 \times 10.5 + 303.75 \times 2$$

$$\Delta p_t = 1962 \text{ Pa} \equiv 1.962 \text{ kPa} \equiv \frac{1.962 \times 10^3}{1000 \times 9.81} \equiv 0.2 \text{ m WC}$$

For 62.5 mm NB SCH-40 pipe,

$$(NPSH)_A = h_{ss} - h_{fs} - p_v = p + Z - h_{fs} - p_v \tag{5.16}$$

$$p = 1 \text{ atm} = \frac{101.325 \times 10^3}{1000 \times 9.81} = 10.33 \text{ m WC}$$

$$p_v = \frac{92.51}{760} = 0.1217 \text{ atm} \equiv 0.1217 \times 10.33 \equiv 1.26 \text{ m WC}$$

$$(\text{NPSH})_A = 10.33 + 10 - 0.2 - 1.26 = 18.87 \text{ m WC}$$

Example 5.14 A centrifugal pump is used to transfer hot water at 110°C from a deaerator to boiler as shown in Fig. 5.5. Hot water is saturated liquid in the deaerator at 110°C. Capacity of pump is 16.5 m^3/h and its efficiency is 60%. Speed of the impeller is 2900 rpm. Pipe line to the boiler contains one control valve and five 90° bends. Its straight length is 25 m.

Calculate:

(a) Recommended size of suction line
(b) Minimum vertical distance Z required between lowest liquid level in the deaerator and centerline of pump and
(c) Recommended size of pipe line to boiler.

Fig. 5.5 *Typical Boiler Feed Water System*

Solution

$$(\text{NPSH})_A \geq (\text{NPSH})_R$$

For the single suction centrifugal pump having speed 2900 rpm and capacity 16.5 m^3/h,

$$\text{rpm} \times \sqrt{q_v} = \text{constant}$$

$$2900 \times \sqrt{16.5} = 3550 \times \sqrt{q_{v1}}$$

$$q_{v1} = 11 \text{ m}^3/\text{h}$$

Operating pressure, $p = 0.147$ MPa g (From JSME Steam Tables corresponding to the 110°C saturation temperature.)

$$p = 0.147 \text{ MPa g} = 248.325 \text{ kPa} = 2.48 \text{ bar} < 35 \text{ bar}$$

From Figs 5.3 and 5.4, $(\text{NPSH})_\text{R} = 3.048 + 0.4572 = 3.5052$ m, say 3.5 m WC

$$(\text{NPSH})_\text{A} = p + Z - h_{fs} - p_\text{v} \geq 3.5 \text{ m}$$

In the deaerator, water is saturated, hence $p = p_v$

$$Z - h_{fs} \geq 3.5 \text{ m}$$

Let $h_{fs} = 1$ m (assumed) $\quad Z \geq 4.5$ m

$Z_{\min} = 4.5$ m, Suggested $Z = 5$ m Ans. (b)

Size of suction line

Let velocity of hot water in suction line = 1 m/s (Table 5.1)

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{v} = \frac{16.5/3600}{1} = 4.5833 \times 10^{-3} \text{ m}^2$$

$$D_i = 0.0764 \text{ m}$$

Select 75 mm NB, SCH-40 MS pipe

$$D_i = 77.92 \text{ mm}$$

Reynolds number, $Re = \dfrac{D_i v \rho}{\mu}$

Velocity of water based on 77.92 mm diameter, $v = 0.961$ m/s

Viscosity water at 110°C, $\mu = 0.256$ mPa · s (or cP), ρ at 110°C = 950.12 kg/m^3

$$Re = \frac{0.077\,92 \times 0.961 \times 950.12}{0.256 \times 10^{-3}} = 277\,914.3$$

Surface roughness of steel pipe = 0.045 72 mm (Table 5.2)

$$\frac{\varepsilon}{D_i} = \frac{0.045\,72}{77.92} = 0.000\,587$$

From Fig. 5.1, $f = 0.0045$, $\dot{m} = 16.5 \times \dfrac{1}{3600} \times 950.12 = 4.3547$ kg/s

$$\frac{\Delta p}{L} = \frac{32 f\, \dot{m}^2}{\pi^2 \rho g_c D_i^5} \tag{5.2}$$

$$= \frac{32 \times 0.0045 \times 4.3547^2}{\pi^2 \times 950.12 \times 1 \times 0.077\,92^5}$$

$$= 101.38 \text{ Pa/m}$$

For 90° elbows, additional pressure drop

$$\Delta p_E = \frac{K \rho v^2}{2 g_c}$$

$$= \frac{0.75 \times 950.12 \times 0.961^2}{2} = 329.05 \text{ Pa per elbow}$$

Total pressure drop in suction line:

$$\Delta p_t = 101.38 \times 5 + 1 \times 329.05 = 835.95 \text{ Pa}$$

$$= \frac{835.95}{950.12 \times 9.81} = 0.09 \text{ m WC}$$

Corrected value of $Z_{\min} = 3.59$ m, say 3.6 m

The recommended size of suction line is 75 mm NB, SCH-40 pipe and minimum vertical distance $Z_{\min}$ required between lowest liquid level in deaerator and centerline of pump is 3.6 m. **Ans.** (a) and (b)

Size of discharge line to boiler:

Let the velocity of water in discharge line, $v = 2$ m/s

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{v} = \frac{16.5/3600}{2}$$

$$D_i = 0.054 \text{ m}$$

Select 50 mm (2 in) NB, SCH-40 MS pipe for which $D_i = 52.5$ mm

Velocity of water based on 52.5 mm inside diameter $v = 2.12$ m/s

$$Re = \frac{D_i v \rho}{\mu} = \frac{0.0525 \times 2.12 \times 950.12}{0.256 \times 10^{-3}}$$

$$= 413\,079.5$$

Surface roughness of steel pipe $\varepsilon = 0.045\,72$ mm

$$\frac{\varepsilon}{D} = \frac{0.045\,72}{52.5} = 8.7 \times 10^{-4}$$

From Fig. 5.1, $f = 0.0047$

$$\dot{m} = 4.3547 \text{ kg/s}$$

For the turbulent flow through steel pipe,

$$\frac{\Delta p}{L} = \frac{32 f \dot{m}^2}{\pi^2 \rho g_c D_i^5} = \frac{32 \times 0.0047 \times 4.3547^2}{\pi^2 \times 950.12 \times 1 \times (0.0525)^5}$$

$$= 762.6 \text{ Pa/m}$$

Table 5.9 Equivalent Velocity Heads of Fittings and Valves

Fittings/valves	No. (n)	K
Gate valve	1	0.17
Control valve	1	6
90° Bends	5	0.75
Check valve	1	2

Total pressure drop in discharge line

$$\Delta p_t = \frac{\Delta p}{L} \times \text{straight length} + (\sum K_i n_i)\frac{\rho v^2}{2g_c}$$

$$= 762.6 \times 25 + \frac{(0.17 + 6 + 0.75 \times 5 + 2) \times 950.12 \times 2.12^2}{2 \times 1}$$

$$= 19\,065 + 25\,450.5 = 44\,515.5 \text{ Pa} \equiv 44.515 \text{ kPa}$$

This value of pressure drop is within permissible limit.
Hence size of discharge line to boiler is recommended to be 50 mm NB, SCH-40.

Example 5.15 Crude dichlorobenzene is pumped from a storage tank to a distillation column. The tank is blanketed with nitrogen and the pressure above the liquid surface is held constant at 0.1 bar g. The minimum depth of liquid in the tank is 1 m.

The distillation column operates at a pressure of 500 Torr a. The feed point to the column is 12 m above the base of the tank. The tank and column are connected by a 50 mm internal diameter commercial steel pipe, 200 m long. The pipe, running from the tank to the column, contains the following valves and fittings: 20 No. of standard radius 90° elbows, two gate valves (fully open), an orifice plate and a flow control valve.

If the maximum flow rate required is 20 000 kg/h, calculate the power required by pump. Pump efficiency is 70%. Pressure drops across the control valve is 0.5 bar. Density of the dichlorobenzene is 1300 kg/m^3 and viscosity is 1.4 m Pa · s (or cP).

Flow meter/Valve/Elbows	Equivalent number of velocity heads, *K*
(i) Standard radius 90° Elbows	0.75
(ii) Gate valve (Fully open)	0.17
(iii) Orifice meter	10

Solution

Power required by pump is calculated by following equation:

$$P = \frac{H q_v \rho}{3.67 \times 10^5 \times \eta} \tag{5.19}$$

$$q_v = \frac{20\,000}{1300} = 15.3846 \text{ m}^3/\text{h}$$

$$\eta = 0.7$$

$$H = h_d - h_s = (p' + Z' + h_{fd}) - (p + Z - h_{fs})$$

p' = Operating pressure in distillation column
= 500 Torr a = 66 661.184 Pa

To convert p' in m of liquid column,

$$p' = \frac{66\,661.184}{\rho \times g} = \frac{66\,661.184}{1300 \times 9.81} = 5.227 \text{ m LC}$$

$$Z' = 12 \text{ m}$$

$$p = 0.1 \text{ bar g}$$

or $$p = 10\,000 + 101\,325 = 111\,325 \text{ N/m}^2 \text{ a}$$

$$= \frac{111\,325}{1300 \times 9.81} = 8.7293 \text{ m LC}$$

$$Z = 1 \text{ m}$$

h_{fs} = Friction loss in suction line ≅ 0 m LC (i.e., negligible)

h_{fd} = Total friction loss in discharge line in m of liquid column

$$h_{fd} = \frac{\Delta p}{\rho \times g}$$

where, Δp = Total pressure drop in discharge line

Velocity of dichlorobenzene in discharge line

$$v = \frac{q_v}{\frac{\pi}{4} D_i^2} = \frac{15.3846 \times (1/3600)}{\frac{\pi}{4} \times (0.05)^2} = 2.1765 \text{ m/s}$$

$$Re = \frac{D_i v \rho}{\mu} = \frac{0.05 \times 2.1765 \times 1300}{1.4 \times 10^{-3}} = 101\,052 > 4000 \text{ (i.e., turbulent)}$$

For the turbulent flow of fluid through commercial steel pipe

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} \dot{m}^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1}$$

$$= 4.07 \times 10^{10} \left(\frac{20\,000}{3600}\right)^{1.84} (1.4 \times 10^{-3})^{0.16} \times (50)^{-4.84} \times (1300)^{-1}$$

$$= 1.535\,73 \text{ kPa/m}$$

$$\Delta p_t = \left(\frac{\Delta p}{L}\right) \times L + \Delta p \text{ of control valve}$$

$$+ \Delta p \text{ across } 90° \text{ elbows, gate valves and orifice meter}$$

$$= 1.535\,73 \times 10^3 \times 200 + 0.5 \times 10^5 + (20 \times 0.75 + 2 \times 0.17 + 1 \times 10) \times \frac{\rho v^2}{2}$$

$$= 307\,146 + 50\,000 + \frac{25.34 \times 1300 \times 2.1765^2}{2}$$

$$= 435\,171.63 \text{ Pa} \equiv 435.172 \text{ kPa}$$

$$h_{fd} = \frac{435\,171.63}{1300 \times 9.81} = 34.123 \text{ m LC}$$

$$H = (5.227 + 12 + 34.123) - (8.7293 + 1 - 0)$$
$$= 41.62 \text{ m LC}$$

$$P = \frac{41.62 \times 15.3846 \times 1300}{3.67 \times 10^5 \times 0.7}$$

$$= 3.24 \text{ kW}$$

5.4 EVALUATION OF CENTRIFUGAL PUMP PERFORMANCE WHEN HANDLING VISCOUS LIQUIDS

When centrifugal pumps, designed and tested for handling water are used for viscous liquids, significant increase in power consumption and decrease in head and capacity occur.

Figures 5.6 and 5.7 provide means of determining the performance of a conventional centrifugal pump handling a viscous liquid when its performance on water is known. The same can also be used as an aid in selecting a pump for a given application.

Fig. 5.6 *Viscosity Correction Chart*[8]

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Fig. 5.7 *Performance Correction Chart*[8]
(Reproduced with the Permission of the Hydraulic Institute, USA)

Limitations on use of viscous liquid performance correction chart (Figs 5.6 and 5.7):

1. Extrapolation is not recommended.
2. Use only for pumps of conventional hydraulic design in the normal operating range with open or closed impellers. Do not use for mixed flow or axial flow pumps.
3. Use only for Newtonian liquids. Do not use for gels or slurries or other non-Newtonian liquids.

Example 5.16 Performance of a centrifugal pump on water is known. It can deliver 9.5 m^3/h of water at 50 m total head of water with 80% efficiency. By using Figs 5.6 and 5.7 predict the performance of this pump if it is used to deliver lubricating oil with 43×10^{-3} m^2/s (or 43 cSt) viscosity at 40°C. Determine the percentage change in Head, Capacity and Power required by the pump. Density of lubricating oil at 40°C is 869 kg/m^3 at the operating temperature.

Solution

Power required by centrifugal pump for delivering 9.5 m^3/h of water at 50 m total head with 80% efficiency

$$P = \frac{Hq_v\rho}{3.67 \times 10^5 \times \eta} \tag{5.19}$$

(Density of water at 40°C, $\rho = 992.215$ kg/m^3 from Table 3.28 of Ref. 2)

$$P = \frac{50 \times 9.5 \times 992.215}{3.67 \times 10^5 \times 0.8} = 1.6053 \text{ kW}$$

Enter the graph (Fig. 5.6, viscosity correction chart) with 9.5 m^3/h capacity, go up to 50 m head, over to 43 cSt viscosity, and then up to the correction factors.

$C_Q = 0.9$
$C_H = 1$
$C_E = 0.71$

Capacity of centrifugal pump when it handles the lubricating oil

$Q_{\text{oil}} = C_Q Q_W = 0.9 \times 9.5 = 8.55$ m^3/h

Head developed by pump

$H_{\text{oil}} = C_H \times H_W = 1 \times 50 = 50$ m

Efficiency of pump $E_{\text{oil}} = C_E \times E_W = 0.71 \times 0.8 = 0.568$

Note that there is a significant drop (29%) in the efficiency.

Power required by pump when it handles the lubricating oil,

$$P' = \frac{H_{\text{oil}} \times Q_{\text{oil}} \times \rho_{\text{oil}}}{3.67 \times 10^5 \times E_{\text{oil}}} = \frac{50 \times 8.55 \times 869}{3.67 \times 10^5 \times 0.568} = 1.7821 \text{ kW}$$

% decrease in capacity of pump = $\frac{(9.5 - 8.55)}{9.5} \times 100 = 10\%$

% decrease in head developed of pump = 0% (i.e., no change)

$$\% \text{ increase in power required by pump} = \frac{(1.7821 - 1.605\,25)}{1.605\,25} \times 100 = 11.02\%$$

Example 5.17 Figure 5.8 shows the performance curve of a centrifugal pump operated on water. If the same pump is used for handling oil with a specific gravity of 0.9 and a viscosity of 1000 SSU (220 cSt) at the pumping temperature, develop the performance curve of the same pump for oil.

Fig. 5.8 *Sample Performance Chart*

Solution

On the performance curve for water, locate the best efficiency point. In this example, from Fig. 5.8 (from the efficiency *vs* capacity curve for water), it is 170 m^3/h. For water with 170 m^3/h capacity, efficiency of pump is maximum (86%). Tabulate capacity, head and efficiency for 0.6×170, 0.8×170 and 1.2×170 m^3/h flow rates. By using Fig. 5.8, for 170 m^3/h, 31 m of head is developed by the pump when it handles the water (from head *vs* capacity curve for water).

Enter the graph (Fig. 5.7, viscosity correction chart) with 170 m^3/h capacity, go up to 31 m head, over to 1000 SSU (220 cSt) viscosity and then up to correction factors.

$C_E = 0.645$
$C_Q = 0.95$
$C_H = 0.895$ (for $1.2 \times Q_N$)
$C_H = 0.92$ (for $1 \times Q_N$)
$C_H = 0.94$ (for $0.8 \times Q_N$)
$C_H = 0.96$ (for $0.6 \times Q_N$)

Sample calculations for $0.6 \times Q_{NW}$ capacity:

$Q_{NW} = 170\ \text{m}^3/\text{h}$

$0.6 \times Q_{NW} = 102\ \text{m}^3/\text{h} = Q_W$

$H_W = 34.62$ m (from head *vs* capacity curve for water in Fig. 5.8)

$\eta_W = 0.76$ (from efficiency *vs* capacity curve for water in Fig. 5.8)

Viscosity of oil, $\mu = 220$ cSt

$C_Q = 0.95\ Q_{\text{oil}} = C_Q \cdot Q_W = 0.95 \times 102 = 96.9\ \text{m}^3/\text{h}$

$C_H = 0.96\ H_{\text{oil}} = C_H \cdot H_W = 0.96 \times 34.62 = 33.235$ m

$C_E = 0.645\ \eta_{\text{oil}} = C_E \cdot \eta_W = 0.645 \times 0.76 = 0.4902$

Here again, note the drop (24%) in efficiency.

Power required by pump when it handles the oil

$$P = \frac{H_{\text{oil}} \times Q_{\text{oil}} \times \rho_{\text{oil}}}{3.67 \times 10^5 \eta_{\text{oil}}} = \frac{33.235 \times 96.9 \times 900}{3.67 \times 10^5 \times 0.4902} = 16.11\ \text{kW}$$

Calculations for obtaining the performance curve for oil given in Table 5.10. Same data are presented in Fig. 5.8.

Table 5.10 Revised Performance of Centrifugal Pump for Handling Oil

Capacity	$0.6 \times Q_{NW}$	$0.8 \times Q_{NW}$	$1.0 \times Q_{NW}$	$1.2 \times Q_{NW}$
Q_w m^3/h	102	136	170	204
H_W m	34.62	33.21	21	27.3
η_W	0.76	0.84	0.86	0.85
C_Q	0.95	0.95	0.95	0.95
C_H	0.96	0.94	0.92	0.895
C_E	0.645	0.645	0.645	0.645
$Q_{\text{oil}} = C_Q \times Q_W$	96.9	129.2	161.5	193.8
$H_{\text{oil}} = C_H \times H_W$	33.325	31.217	28.52	24.434
$\eta_{\text{oil}} = \eta_W \times C_E$	0.4902	0.5418	0.5547	0.5483
P, kW	16.11	18.26	20.363	21.18

5.5 POWER REQUIRED IN FAN, BLOWER AND IN ADIABATIC COMPRESSOR

Fans, blowers and compressors are used to increase the mechanical energy of gases. Discharge pressure of a fan is less than 3.45 kPa g (350 mm WC). If discharge pressure required is higher than 3.45 kPa g then a blower is used. Maximum discharge pressure of blower is 1 atm g. If the discharge pressure required is more than 2 atm a then a compressor is used. Discharge pressure of compressor ranges from 2 atm a to thousands of atmospheres.

5.5.1 Power Required by Fan

Fans are of two types: (i) centrifugal fans and (ii) axial flow fans. Efficiency of a fan ranges from 40 to 70%, depending on its specific speed. Power input to a fan is given by the equation

$$P_0 = 2.72 \times 10^{-5}\, q_v\, p_d / \eta \tag{5.21}$$

where, P_0 = Power required by fan, kW
q_v = Capacity of fan, m^3/h
p_d = Discharge pressure of fan, cm WC (gage pressure)
= Sum of pressure and velocity head, developed by fan, cm WC
η = Efficiency of fan

5.5.2 Power Required in Blower and in Adiabatic Compressor

Blowers are always operated in an adiabatic manner. Many compressors are also operated in adiabatic manner (Example: air compressors up to 10 bar g). In adiabatic compressor, jacketed cooling is not provided. While in other types; polytropic and isothermal compressors, jacketed cooling is provided around the compressor section. In isothermal compressor, inlet temperature of gas is equal to outlet temperature of gas. If both temperatures are not same even after providing the jacket cooling, it is called polytropic compressor.

For adiabatic compression

$$\frac{p_2}{p_1} = \left(\frac{V_1}{V_2}\right)^k, \quad \frac{T_1}{T_2} = \left(\frac{V_1}{V_2}\right)^{k-1}, \quad \frac{p_2}{p_1} = \left(\frac{T_2}{T_1}\right)^{\frac{k}{k-1}} \tag{5.22}$$

where, $k = C_p/C_v$
= ratio of specific heat at constant pressure to specific heat at constant volume
p_2, p_1 = outlet/inlet pressure of gas, kPa
V_2, V_1 = outlet/inlet volume of gas, m^3/kmol
T_2, T_1 = outlet/inlet temperatures of gas, K

Power required in single stage blowers or in single stage adiabatic compressor,

$$P_0 = \frac{2.78 \times 10^{-4}}{\eta}\left(\frac{k}{k-1}\right) q_{v1} p_1 \left[\left(\frac{p_2}{p_1}\right)^{\frac{k-1}{k}} - 1\right] \tag{5.23}$$

where, P_0 = Power required, kW
p_1, p_2 = Absolute inlet pressure, kPa
q_{v1} = Volumetric flow rate of gas based on inlet condition, m^3/h
η = Efficiency of blower

Discharge temperature of gas from the blower or single stage adiabatic compressor,

$$T_2 = T_1\left(\frac{p_2}{p_1}\right)^{\frac{k-1}{k}} \tag{5.24}$$

Value of k ranges from 1.39 to 1.41 for air and perfect diatomic gases. For $k = 1.395$, value of bracketted expression in Eq. (5.23) is known as X factor

$$X = \left[\left(\frac{p_2}{p_1}\right)^{\frac{k-1}{k}} - 1\right] = r^{\frac{k-1}{k}} - 1 \tag{5.25}$$

where, $k = 1.395$ for air or a diatomic gas
r = Compression ratio

Power required for blower and adiabatic compressor

$$P_0 = 9.81 \times 10^{-4} q_{v1}\, p_1\, X/\eta \text{ kW} \tag{5.26}$$

Other gases are having value of k in the range of 1 to 1.4. Power required by blower or adiabatic compressor, for the gases having value of k other than $k = 1.395$, is given by the following equation:

$$P_0 = 6.37 \times 10^{-4}\, q_{v1} p_1 \frac{X_G}{d\eta} \text{ kW} \tag{5.27}$$

where, $d = 2.292\ [(k-1)/k]$

Values of $\left(\frac{X_G}{X}\right)$ and d can be obtained from Fig. 5.9 for the different values of k and for different values of r.

For a multistage compressor with N stages of compression in which

1. adiabatic compression takes place in each stage,
2. near equal work done in each stage (i.e., equal compression ratio in each stage; r) and
3. intercooling is provided between each two stages of compression to same inlet gas temperature (T_1),

Power requirement can be calculated by the following equation:

$$P_0 = \frac{6.37 \times 10^{-4} N\, q_{v1} p_1}{\eta \cdot d}\left(\sqrt[N]{X_G + 1} - 1\right) \text{ kW} \tag{5.28}$$

For the multistage compressor, discharge temperature from the last stage can be predicted by the following equation:

$$T_2 = T_1\left(\sqrt[N]{X_G + 1}\right) \text{ K} \tag{5.29}$$

where, N = number of stages

Fig. 5.9 *(X_G/X) vs k and d vs k (Ref. 2)*
(Reproduced with the Permission of McGraw-Hill Education, USA)

Example 5.18 Find out the power required for a turbo blower for the following duty:

Fluid = Atmospheric air
Capacity = 1000 Nm^3/h
Discharge pressure = 2 atm a
Efficiency of turbo blower, $\eta = 0.7$
Also find the discharge temperature of air.

Solution

Power required for turbo blower

$$P_0 = \frac{2.78 \times 10^{-4}}{\eta} \frac{k}{k-1} q_{v1} p_1 \left[\left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}} - 1 \right] \tag{5.23}$$

For air, $k = 1.395$
q_{v1} = Volumetric flow rate based on inlet condition in m^3/h

$$\frac{p_1 q_{v1}}{T_1} = \frac{1 \times 1000}{(273 + 25)}$$

$$q_{v1} = \frac{1000 \times 1}{298} \times (273 + 50) = 1083.9 \text{ m}^3/\text{h}$$

$$P_0 = \frac{2.78 \times 10^{-4}}{0.7} \times \frac{1.395}{(1.395 - 1)} \times 1083.9 \times 101.325 \times (2^{[(1.395-1)/1.395]} - 1)$$
$$= 33.4 \text{ kW}$$

Discharge temperature of air

$$T_2 = T_1 \left(\frac{p_2}{p_1}\right)^{\left(\frac{k-1}{k}\right)} \tag{5.24}$$

$$T_2 = 323(2)^{\left(\frac{0.395}{1.395}\right)} = 393.04 \text{ K}$$

Discharge temperature of air, $t_2 = 120°C$

Example 5.19 A three stage reciprocating compressor is used to compress 306 Sm^3/h of methane from 0.95 atm a to 61.3 atm a. The inlet temperature is 26.7° C.
Specific heat ratio of methane, $k = 1.31$

Calculate (a) Power required for compression, if mechanical efficiency is 80 percent and
(b) Discharge temperature of gas after 1st stage

Solution

For a multistage compressor, total power required is minimum if each stage consumes the same power or same compression ratio in each stage.

For same compression ratio in each stage,

Compression ratio in each stage $r = \left(\frac{61.3}{0.95}\right)^{1/3} = 4.01$

Power required for each stage

$$P_o = \frac{2.78 \times 10^{-4}}{\eta} \frac{k}{k-1} q_{v1} p_1 \left[\left(\frac{p_2}{p_1}\right)^{\frac{k-1}{k}} - 1\right] \tag{5.23}$$

$k = 1.31, \quad n = 0.8$
q_{v1} = Volumetric flow rate based on inlet condition m^3/h

$$\frac{p_1 q_{v1}}{T_1} = \frac{p_2 q_{vs}}{T_s} = \frac{1 \times 306}{273}$$

$$q_{v1} = \frac{306}{273} \times \frac{(273 + 26.7)}{0.95} = 353.6 \text{ m}^3/\text{h}$$

$$P_0 = \frac{2.78 \times 10^{-4}}{0.8} \frac{1.31}{1.31 - 1} \times 353.6 \times 0.95 \times 101.325 \left((4.01)^{\frac{0.31}{1.31}} - 1 \right)$$

$$= 19.45 \text{ kW}$$

If power loss in allied piping and intercoolers is neglected, then total power required for three stages,

$$P_0' = 3 \times 19.45 = 58.35 \text{ kW}$$

(b) Discharge temperature after first stage

$$T_2 = T_1 \left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}}$$

$$T_2 = (273 + 26.7)(4.01)^{(0.31/1.31)}$$

$$T_2 = 416.3 \text{ K}, \; t_2 = 143.3°\text{C}$$

or

for $k = 1.31$ and $r = 4.01$, from Fig. 5.9:
$d = 0.54$, $(X_G/X) = 0.8$

$$X = r^{\frac{0.395}{1.395}} - 1 = 4.01^{\left(\frac{0.395}{1.395}\right)} - 1 = 0.4818$$

$$X_G = 0.8 \times 0.4818 = 0.3854$$

Power required for each stage

$$P_0 = 6.37 \times 10^{-4} \frac{q_{v1} p_1}{\eta} \frac{X_G}{d} \tag{5.27}$$

$$P_0 = \frac{6.37 \times 10^{-4} \times 353.6 \times 96.2587}{0.8} \times \frac{0.3854}{0.54} = 19.34 \text{ kW}$$

Total power required $P_0' = 3 \times 19.34 = 58.03$ kW
(Neglecting pressure loss in the intercoolers and allied piping)

5.6 FLOW METERS

A variety of flow meters are in use in chemical process industry. One category is the traditional differential pressure (DP) type volumetric flow meters. In many applications, the volumetric flow rate is of direct interest to the user. Because of its accuracy, simplicity and relatively lower cost, these flow meters are popular in the chemical industry. By multiplying the flow rate with the actual density, mass flow rate can be obtained. Pitot tube, orifice meter, venturi meter and flow nozzles fall under this category. Among these, orifice meter is by far the most popular in the industry. Figure 5.10 shows these meters schematically.

Less commonly used flow meter is vortex meter which works on Von Karman effect. Flowing fluid separates on two sides of shedder bar face. Vortices form behind the face and cause alternating differential pressures (DP) around the back of the shedder.

The frequency of the alternating vortex development is linearly proportional to the volumetric flow rate. Although vortex meters are as reliable and as accurate as other DP instruments, they can be used for only clean liquids and are recommended for turbulent flows.

(a) Various Tap Locations for Orifice Meter

Pipe Diameters from Inlet Face of Orifice Plate
(*Extracted from the ASME Flow Meter Computation Handbook, 1961*)

(b)

Orifice Meter with Vena Contracta Formation

(c)

Fig. 5.10 *Orifice Meter*

Another popular category is of variable area flow meters which are also known as rotameters. Figure 5.11 shows two different types of rotameters. While DP type instruments can be installed in horizontal position, rotameters are installed in vertical position. Bypass rotameters are installed in horizontal position which is a combination of an orifice and a rotameter. Bypass rotameters are installed on larger size pipelines and occupy quite less space. Near linear scale of the meter is an advantage over logarithmic scale for the DP meters. However, rotameters with glass tubes are relatively less accurate and are mainly used for low pressure applications.

Disadvantage of low pressure applications due to glass tubes has led to the development of magnetic rotameters in which a magnetic float is used in a metallic tapered tube and its accurate position is monitored outside the tube. Float's movement is calibrated on a dial-type indicator. Both DP type and magnetic rotameter can be connected to a microprocessor-based control unit for totalizing and/or controlling purpose.

Positive displacement-type flow meters incorporate a pair of gears or a turbine which rotate on their axes and the revolutions are measured. Since volume displaced per revolution is nearly constant, volumetric flow is calculated by multiplying two parameters. Traditional water meters are of gear type. Turbine flow meters are available for number of liquids such as ammonia, chlorine, etc., which flow under relatively high pressure. These instruments are sufficiently accurate and are used for custody transfer.

Weirs are popularly used for volumetric flow measurements in open canals and alike situations. Figure 5.12 shows different types of weirs in use.

Fig. 5.11 *Rotameters*

Fig. 5.12 *Flow Measurement with Weirs*

Another category relates to ultrasonic flow meters. Its underlying principle is measurement of the effect a flowing fluid has on an ultrasound signal either transmitted through or reflected from the fluid. These instruments are largely of clamp-on designs and offer simplicity of installation. These meters suffer from a reputation for unreliability and poor long-term stability, compared with DP type meters. However, newer designs are being developed for high pressure and temperature fluids, including gases. Designs are available when a single instrument can be clamped on a range (though limited) of pipes and volumetric flows can be checked periodically. This type of meter is quite handy to a process engineer in checking plant performance from time to time.

Electromagnetic flow meters (also known as magmeters) work on Faraday's law of conductance which states that when a conductive fluid flows through a magnetic field, generated perpendicular to the flow, the voltage induced in the fluid is linearly proportional to the volumetric flow rate. Thus, this type of instrument operates only with electrically conductive liquid. Non-invasive nature of ultrasonic flow meter is also the feature of magmeter. Both permit unobstructed flow through the measuring element. Both are clamp on meters whose transducers are strapped to the pipeline. Unlike ultrasonic meters, magmeters have a reputation of accuracy.

True mass flow meter based on Coriolis principle was introduced in 1977 by Mirco Motion, Inc., USA, a division of Emerson Process Management. The Coriolis principle, discovered by Gustave Coriolis in 1935 states that fluid flowing through a tube, vibrating at its natural frequency, generates a force that slightly distorts the tube. This extremely small distortion, picked up by sensors, is directly proportional to the mass flow rate (Newton's second law of motion) and is independent of any other property of the fluid; say pressure, temperature, density or viscosity. Figure 5.13 schematically represents the mass flow meter.

Fig. 5.13 *Coriolis Mass Flow Meter (Courtesy: Micro Motion, Inc., USA)*

In a single mass flow meter, four parameters of the fluid can be measured at a time; mass flow, volumetric flow, temperature, and density. This instrument offers high accuracy and can be used for any fluid; clean or dirty, transparent or opaque,

conductive or non-conductive. Originally developed instrument required extra space for installation but recently developed meter has a straight tube configuration. Among different types of flow meters, Coriolis device is the costliest.

In the forthcoming sections, process designs of orifice meter and rotameter are discussed. Reader is advised to refer standard handbooks on instrumentation/flow meters for design of other flow meters.

Flow Nozzle with Differential Gauge

(a)

(b) Venturi Meter

Fig. 5.14

5.7 PROCESS DESIGN OF ORIFICE METER

Orifice meter is a widely used flow meter in chemical industry, compared to venturi meter and rotameter.

Advantages of orifice meter are:

1. Fixed cost is less
2. Easy to fabricate and install
3. Occupies less space as compared to the venturi meter
4. Provides more flexibility. Orifice plate can be easily replaced.

Disadvantages of orifice meter is higher power consumption and hence operating cost of orifice meter is higher than the same of venturi meter and of rotameter.

In orifice meter, as shown in Fig. 5.10, a square edged or sharp edged orifice plate is mounted between two flanges at the flanged joint. When fluid flows through the orifice it forms free flowing jet. This free flowing jet first contracts and then expands. Minimum flow area achieved by free flowing jet is known as Vena Contracta.
Mass flow rate through orifice is given by the following equation:

$$\dot{m} = C_o Y A_o \sqrt{\frac{2g_c(p_1 - p_2)\rho}{1 - \beta^4}} \tag{5.30}$$

where, $\dot{m}$ = Mass flow rate of fluid, kg/s

A_0 = Area of orifice = $(\pi/4)d_0^2$, m^2

p_1, p_2 = Pressure at upstream and downstream static pressure taps, respectively, Pa

ρ = Density of fluid, kg/m^3

$$\beta = \frac{\text{Diameter of orifice}}{\text{Inside diameter of pipe}}$$

Y = Expansion factor
= 1 for liquids
= $1 - [((1 - r)/k)(0.41 + 0.35\,\beta^4)]$ for gases (5.31)

where, $r = \dfrac{p_2}{p_1}$, ratio of downstream to upstream pressure

$k = \dfrac{C_p}{C_v}$, specific heat ratio

C_o = Coefficient of orifice = $f\,(Re_o, \beta$, Location of taps)

where, Re_o = Reynold's number for the fluid flowing through orifice

$$Re_o = \frac{d_o u_o \rho}{\mu} = \frac{4\dot{m}}{\pi d_o \mu} \tag{5.32}$$

where, u_o = Velocity of fluid through orifice, m/s

μ = Viscosity of fluid, kg/(m · s)

Equation of orifice meter (Eq. 5.30) is empirical in nature and it requires experimental support.

In addition to Re_o and β, C_o also depends on the location of pressure taps. Location of pressure taps on upstream side and down stream side are also standardized as discussed in Fig. 5.10 (a).

There are total five standard locations of pressure taps:

1. **Corner taps:** Static holes made in upstream and downstream flange. They are very close to the orifice plate. With corner taps, it is possible to drill both static holes in the orifice plate itself. Then entire orifice meter can be easily inserted in any flanged joint without drilling the holes in pipe or flanges.
2. **Flange taps:** Static holes made at a distance 25.4 mm on upstream side and 25.4 mm on downstream side.

3. **Radius taps:** Static holes located at a distance one pipe diameter on upstream side and 1/2 pipe diameter on downstream side. Radius taps are the best from practical standpoint of view as it gives reasonably good pressure difference, compared to other taps except vena contracta taps. Higher pressure difference means more accurate measurement of flow rate.
4. **Vena contracta taps:** Upstream static hole is 1/2 to 2 times pipe diameter from the plate. Downstream tap is located at the position of minimum pressure. Vena contracta taps give the maximum pressure difference for a given flow rate. But it is not suitable, if orifice size is changed from time to time.
5. **Pipe taps:** Static holes are located at 2.5 times pipe diameter upstream side and 8 times pipe diameter on down stream side. This means fluid is flowing normally on both sides without being affected by turbulence, created by the orifice plate.

   For $Re_o > 30\,000$,

   C_o = Between 0.595 to 0.62 for vena contracta taps

   C_o = Between 0.595 to 0.8 for radius taps

   C_o = 0.62 for corner taps

   Relation between discharge coefficient C_o, β and Re_D is given by Stolz equation (ISO: 5167).

$$C_o = 0.5959 + 0.0312\,\beta^{2.1} - 0.184\,\beta^8 + 0.0029\,\beta^{2.5}\,(10^6/Re_D)^{0.75} + 0.09\,L_1\,\beta^4\,(1-\beta^4)^{-1} - 0.0337\,L_2\,\beta^3 \quad (5.33)$$

   where, Re_D = Reynolds number based on internal diameter of pipe D

$$L_1 = \left(\frac{l_1}{D}\right), \quad L_2 = \left(\frac{l_2}{d_o}\right)$$

   where, d_o = diameter of orifice

   $\beta = d_o/D$

   l_1 = Distance of the upstream tapping from the upstream face of orifice plate, mm

   l_2 = Distance of the downstream tapping from the downstream face of the orifice plate, mm

Example 5.20 Design an orifice meter based on the following data:
Name of fluid = water
Flow rate = 100 000 kg/h
Inside diameter of pipe = 154 mm (6 in, SCH-40 pipe)
Operating temperature = 32°C
Density of water at 32°C = 995.026 kg/m^3
Viscosity of water at 32°C = 0.765 mPa · s or cP
Manometer fluid = Mercury
Density of Mercury at 32°C = 13 516.47 kg/m^3

Solution

Inside diameter of pipe D = 154 mm
Let $\beta = 0.5$, i.e., $d_o/D = 0.5$

Diameter of orifice, $d_o = 77$ mm

Re_D = Reynolds number based on inside diameter of pipe

$$= \frac{4\dot{m}}{\pi D\mu} = \frac{4 \times (100\ 000/3600)}{\pi \times 0.154 \times 0.765 \times 10^{-3}}$$

$$= 300\ 210.2$$

Use square edged circular orifice plate with radius taps.

Radius taps are best from practical stand point. In radius taps, downstream tap is located approximately at the mean location of vena contracta (1/2 times pipe diameter) and also upstream tap is sufficiently far so that it is not affected by distortion of flow in the vicinity of orifice.

Based on Stolz's equation,

$$C_o = 0.5959 + 0.0312\beta^{2.1} - 0.184\beta^8 + 0.0029\ \beta^{2.5}\left(\frac{10^6}{Re_D}\right)^{0.75}$$

$$+ 0.09L_1\beta^4(1-\beta^4)^{-1} - 0.0337\ L_2\beta^3 \tag{5.33}$$

$$L_1 = \frac{l_1}{D} = 1,\ L_2 = \frac{l_2}{d_o} = 1$$

$$C_o = 0.5959 + 7.2776 \times 10^{-3} - 7.1875 \times 10^{-4} + 1.264 \times 10^{-3} + 6 \times 10^{-3}$$
$$- 4.2125 \times 10^{-3}$$
$$= 0.6055$$

Mass flow rate of water

$$\dot{m} = C_o Y A_o \sqrt{\frac{2g_c \Delta p \rho}{1-\beta^4}} \tag{5.30}$$

Y = expansion factor for liquid = 1

$$\frac{100\ 000}{3600} = 0.6055 \times 1 \times \frac{\pi}{4}(0.077)^2 \times \sqrt{\frac{2 \times 1 \times \Delta p \times 995.026}{1-0.5^4}}$$

$$\Delta p = 45\ 722.597 \text{ Pa} \equiv 45.722 \text{ kPa} \equiv 4.661 \text{ m WC}$$

$$\Delta p = R_m \frac{g}{g_C}(\rho_m - \rho_f)$$

$$45\ 722.597 = R_m \times \frac{9.81}{1}(13\ 516.47 - 995.026)$$

R_m = Manometer reading = 0.3722 m Hg

$\equiv$ 372.2 mm Hg

In actual industrial practice, DP transmitter is used. Select the transmitter with 550 mm WC range so that the normal reading is at about 85% of the range.

Similarly, one can find different values of R_m for the different variation in flow rate or in other words calibration of orifice meter can be carried out.

Example 5.21 Design an orifice meter based on the following data:
Name of fluid = Chlorine gas
Flow rate = 1500 Nm^3/h
Operating pressure = 1.2 atm a
Operating temperature = 30°C
Viscosity of chlorine gas at 30°C = 0.0145 mPa · s or cP
Inside diameter of pipe = 154 mm (6 in. SCH-40)
Specific heat ratio for Cl_2 gas, $k = 1.355$

Solution

Let $\beta = 0.5$

$$\frac{d_o}{D} = 0.5 \quad \text{or} \quad d_o = 77 \text{ mm}$$

$$Re_D = \frac{4\dot{m}}{\pi D \mu}$$

Density of Cl_2 gas at normal condition

$$\rho = \frac{1 \times 71}{(273 + 25)} \times \frac{273}{1 \times 22.414} = 2.902 \text{ kg/m}^3$$

$$\dot{m} = 1500 \times 2.902 = 4353 \text{ kg/h}$$

$$Re_D = \frac{4 \times (4353/3600)}{\pi \times 0.154 \times 0.0145 \times 10^{-3}}$$

$$= 689\,457.6$$

If corner taps are made then $l_1 = l_2 = 0$
Coefficient of orifice meter can be determined by Stolz's equation.

$$C_o = 0.5959 + 0.0312\,\beta^{2.1} - 0.184\,\beta^8 + 0.0029\,\beta^{2.5}\left(\frac{10^6}{Re_D}\right)^{0.75}$$

$$+ 0.09 L_1 \beta^4 (1 - \beta^4)^{-1} - 0.0337 L_2 \beta^3 \tag{5.33}$$

$$= 0.5959 + 7.2776 \times 10^{-3} - 7.1875 \times 10^{-4} + 6.7755 \times 10^{-4}$$

$$= 0.6031$$

Expansion factor

$$Y = 1 - [((1 - r)/k)(0.41 + 0.35\beta^4)]$$

$$= 1 - [((1 - r)/1.355) \times 0.4319]$$

For the first trial calculation let $r = 0.8$, $Y = 0.9362$
Mass flow rate of chlorine gas

$$\dot{m} = C_o Y \frac{\pi}{4} d_o^2 \sqrt{\frac{2 g_c \Delta p \rho}{1 - \beta^4}} \tag{5.30}$$

Density of chlorine gas at operating condition:

$$\rho = \frac{1.2 \times 71}{(273 + 30)} \times \frac{273}{1 \times 22.414} = 3.425 \text{ kg/m}^3$$

$$\frac{4353}{3600} = 0.6031 \times 0.9362 \times \frac{\pi}{4}(0.077)^2 \times \sqrt{\frac{2 \times 1 \times \Delta p \times 3.425}{1 - 0.5^4}}$$

$$\Delta p = 28\,946.4 \text{ Pa}$$
$$\equiv 28.946 \text{ kPa}$$
$$p_1 = 1.2 \text{ atm}, p_2 = p_1 - \Delta p = 1.2 \times 101.325 - 28.946$$
$$p_2 = 92.644 \text{ kPa}$$
$$r = \frac{p_2}{p_1} = \frac{92.644}{121.59} = 0.762, \quad Y = 0.9241$$
$$\Delta p \propto \frac{1}{Y^2} \quad \text{or} \quad \Delta p_1 Y_1^2 = \Delta p_2 Y_2^2$$
$$\Delta p_2 = 28.946 \times \left(\frac{0.9362}{0.9241}\right)^2 = 29.7 \text{ kPa}$$
$$r = \frac{(121.59 - 29.7)}{121.59} = 0.7557 \equiv 0.762 \text{ (close enough to the previous value of } r\text{)}$$
$$\Delta p_2 = 29.7 \text{ kPa} \equiv 3.028 \text{ m WC (final)}$$

Similarly, for the different variation in the flow rate Δp can be determined. In this case also, a DP transmitter having 3500 mm WC range can be selected. Normal reading will be at about 87% of the range.

5.8 PROCESS DESIGN OF ROTAMETER

Rotameter is used for the flow measurement of liquids and gases. It is a variable area flow meter in which pressure drop is nearly constant and the area through which the fluid flows varies with the flow rate. It consists of tapered tube with the smallest diameter at the bottom. The tube contains a freely moving float which rests on a stop at the base of the tube. When the fluid is flowing the float rises until its weight is balanced by the upthrust of the fluid, its position then indicates the rate of flow. Float is **not** floating but is completely submerged in the fluid and its density is greater than the density of fluid. The tube is marked in divisions and the reading of the meter is obtained from the scale reading at the reading edge of the float which is taken at the largest cross section of the float. It is always mounted in the perfect vertical position. Rotameters are designed to measure the flow rate of fluid in certain range like 5 m^3/h to 50 m^3/h, 10 m^3/h to 100 m^3/h, 3 Lpm to 30 Lpm, etc.

For a linearly tapered tube with a diameter at the bottom about the float diameter, flow is almost a linear function of the height or position of float. As a tube material glass is the first choice. For high temperatures or pressures or for other conditions where glass is impracticable, metal tubes are used. In this case since, in metal tube, the float is invisible, means must be provided for either indicating or transmitting the meter reading.

Mass flow rate through rotameter is given by the equation

$$q_m = C_D A_2 \sqrt{\frac{2gV_f(\rho_f - \rho)\rho}{A_f[1 - (A_2/A_1)^2]}} \tag{5.34}$$

where, q_m = Mass flow rate, kg/s

C_D = Discharge coefficient depends on the shape of the float and the Reynolds number though annulus. It can be obtained from literature such as Fig. 6.22 of Ref. 11. For the float having the shape same as shown in Fig. 5.15, value of C_D is almost constant for the value of *Re* above 500 and it is 0.775.

A_2 = Cross-sectional area of annulus between float and tube, m^2

$A_2 = A_1 - A_f$

A_1 = Cross-sectional area of tube at the float position, m^2

A_f = Maximum cross-sectional area of the float in a horizontal plane, m^2

g = Acceleration of gravity = 9.81 m/s^2 at mean sea level

V_f = Volume of float, m^3

ρ_f = Density of float, kg/m^3

ρ = Density of fluid, kg/m^3

Example 5.22 Determine the minimum and maximum flow rates of water at 40°C which can be measured by rotameter of the given dimensions. Dimensions of tube and float of rotameter are given in Fig. 5.15.

Viscosity of water of 40°C = 0.7 cP or mPa · s

Density of water at 40°C = 992.2 kg/m^3

Density of stainless steel = 8000 kg/m^3

Float is made from stainless steel plate of 2 mm thickness. Calibrate the rotameter or find the different values of flow rates for the different float positions.

Fig. 5.15 *Typical Details of Float and Tube*

Solution

Volume of float

$$V_f = \left(2 \times \frac{\pi}{4}(15)^2 \times 5 + \frac{\pi}{4}\left(\frac{25+15}{2}\right)^2 \times 5\right) \times 10^{-9}$$

$$V_f = 3.338 \times 10^{-6}\ \text{m}^3$$

Mass of float

$$m_f = \left(2 \times \frac{\pi}{4}(15)^2 + \pi(15)(10) + \frac{\pi}{4}(25^2 - 15^2) + \pi\left(\frac{15+25}{2}\right) \times 5\right) \times 10^{-6} \times 0.002 \times 8000$$

$$= 0.023\ 25\ \text{kg}$$

Density of float, $\rho_f = \dfrac{m_f}{V_f} = \dfrac{0.023\ 25}{3.338 \times 10^{-6}} = 6965\ \text{kg/m}^3 < 8000\ \text{kg/m}^3$

Maximum flow rate at top:
Mass flow rate through Rotameter is given by the equation

$$q_m = C_D A_2 \sqrt{\frac{2gV_f(\rho_f - \rho)\rho}{A_f\left[1 - \left(\frac{A_2}{A_1}\right)^2\right]}} \tag{5.34}$$

For the first trial calculation, let discharge coefficient, $C_D = 0.75$.

A_1 = Cross-sectional area of tube

$$= \frac{\pi}{4}(40)^2 = 1256.6\ \text{mm}^2 \equiv 1256.6 \times 10^{-6}\ \text{m}^2$$

A_f = Maximum cross-sectional area of the float in a horizontal plane, m^2

$$= \frac{\pi}{4}(25)^2 = 490.87\ \text{mm}^2 \equiv 490.87 \times 10^{-6}\ \text{m}^2$$

A_2 = Cross-sectional area of annulus between float and tube, m^2

$$A_2 = A_1 - A_f = 765.767 \times 10^{-6}\ \text{m}^2,\ \frac{A_2}{A_1} = 0.6094$$

$$q_m = 0.75 \times 0.765\ 767 \times 10^{-3} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 992.2 \times (6965 - 992.2)}{490.87 \times 10^{-6}[1 - 0.6094^2]}}$$

$$= 0.6441\ \text{kg/s}$$

$$\equiv 2318.76\ \text{kg/h} \equiv 2.337\ \text{m}^3/\text{h} \quad (\text{taking } \rho = 0.9922\ \text{kg/L for water})$$

Mass velocity through annulus

$$G = \frac{q_m}{A_2} = \frac{0.6441}{0.765\,767 \times 10^{-3}} = 841.12 \text{ kg/(m}^2 \cdot \text{s)}$$

Equivalent diameter

$$d_e = \frac{4 \times \text{Cross-sectional area}}{\text{Wetted Parameter}}$$

$$= \frac{4 \times 765.767 \times 10^{-6}}{\pi(40 + 25) \times 10^{-3}} = 0.015 \text{ m}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.015 \times 841.12}{0.7 \times 10^{-3}} = 18\,024 > 500$$

From Fig. 6.22 of Ref. 11:

$$C_D = 0.775$$

$$q_m = \frac{0.775}{0.75} \times 0.644 = 0.6655 \text{ kg/s}$$

$$\equiv 2395.7 \text{ kg/h} \equiv 2.414 \text{ m}^3\text{/h (corrected)}$$

First mark for indicating flow rate is at 27 mm diameter of tube.

$$A_1 = \frac{\pi}{4}(27)^2 = 572.55 \text{ mm}^2 \equiv 572.55 \times 10^{-6} \text{ m}^2$$

$$A_2 = A_1 - A_f = 81.685 \times 10^{-6} \text{ m}^2$$

$$\frac{A_2}{A_1} = 0.142\,67$$

Let $C_D = 0.775$ (for first trial)

$$q_m = 0.775 \times 81.685 \times 10^{-6}\sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 992.2 \times (6965 - 992.2)}{490.87 \times 10^{-6}[1 - 0.142\,67^2]}}$$

$$= 0.056\,873 \text{ kg/s} \equiv 204.7 \text{ kg/h} \equiv 0.2063 \text{ m}^3\text{/h}$$

$$G = \frac{0.056\,873}{81.685 \times 10^{-6}} = 696.25 \text{ kg/(m}^2 \cdot \text{s)}$$

$$d_e = \frac{4 \times 81.685 \times 10^{-6}}{\pi(27 + 25) \times 10^{-3}} = 0.002 \text{ m}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.002 \times 696.25}{0.7 \times 10^{-3}} = 1989.3$$

For $Re = 1989.3$, $C_D = 0.775$ (from Fig. 6.22 of Ref. 11). Hence, no change in q_m or q_v. Based on similar calculations, Table 5.11 is prepared.

Table 5.11 Flow Rate Calculation for Rotameter

Tapered tube diameter, mm	A_1 mm^2	A_2 mm^2	A_2/A_1	q_v m^3/h
25	490.87	—	—	0
27	572.55	81.685	0.142 67	0.2063
30	706.85	215.98	0.3056	0.5672
32	804.25	313.38	0.3897	0.85
34	907.92	417.05	0.4593	1.174
36	1017.876	527	0.5178	1.54
38	1134.11	643.24	0.5672	1.9528
40	1256.6	765.73	0.6094	2.4148

Let angle made by tapered tube with vertical plane is 2 degrees.

Let h = Float position in mm from the bottom-most point (i.e., from the point where diameter of tube is 25 mm)

For q_v = 2.4146 m^3/h, inside diameter of tapered tube = 40 mm

Value of h at 40 mm diameter

$$h = \frac{(40 - 25)/2}{\tan 2} = 214.77 \text{ mm}$$

Similarly float position h can be determined for the different flow rates.

Table 5.12 Float Position Calculations for Rotameter

Diameter of tapered tube, mm	q_v, m^3/h	h, float position from bottom, mm
25	0	0
27	0.2063	28.64
30	0.5672	71.59
32	0.85	100.23
34	1.174	128.86
36	1.54	157.5
38	1.9528	186.135
40	2.4148	214.77

Draw the graph of h vs q_v.
Refer Fig. 5.16.

Fig. 5.16 *Calibration of Rotameter*

Example 5.23 In Example 5.22, a rotameter is calibrated to measure the flow rate of water at 40°C. If the same rotameter is used for measuring the flow rates of the following fluids then what is the change in calibration?

(a) Monoethylene glycol (MEG) brine containing 60% MEG by mass[12]
Operating temperature: –25°C
Operating pressure: 3 bar g
Density: 1098 kg/m^3
Viscosity: 40 mPa · s (or cP)

(b) Ethyl alcohol
Operating temperature: 35°C
Pressure: 3 bar g
Density: 781 kg/m^3
Viscosity: 0.9 mPa · s (or cP)

Solution

At the topmost position of the float,
h = 214.77 mm, diameter of tapered tube = 40 mm,

$$A_1 = 1256.6 \text{ mm}^2, \quad A_2 = 765.73 \text{ mm}^2, \quad \frac{A_2}{A_1} = 0.6094$$

For this position of float, flow rate of MEG:

$$q_m = C_D A_2 \sqrt{\frac{2gV_f(\rho_f - \rho)\rho}{A_f\left[1 - \left(\frac{A_2}{A_1}\right)^2\right]}} \tag{5.34}$$

For the first trial calculations, let $C_D = 0.775$

$$q_m = 0.775 \times 765.73 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 1098 \times (6965 - 1098)}{490.87 \times 10^{-6}[1 - 0.6094^2]}}$$

$$= 0.6939 \text{ kg/s}$$

or $$q_v = \frac{0.6939}{1098} \times 3600 = 2.275 \text{ m}^3/\text{h}$$

Mass velocity, $$G = \frac{q_m}{A_2} = \frac{0.6939}{765.73 \times 10^{-6}} = 906.194 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.015 \times 906.194}{40 \times 10^{-3}} = 339.82$$

Value of $C_D = 0.77$ (Fig. 6.22 of Ref. 11)

$$q_m = \frac{0.77}{0.775} \times 0.6939 = 0.6894 \text{ kg/s}$$

or $q_v = 2.26 \text{ m}^3/\text{h}$

For the same position of float, flow rate of ethyl alcohol:

Let $C_D = 0.775$ (for the first trial calculations)

$$q_m = 0.775 \times 765.73 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 781 \times (6965 - 781)}{490.87 \times 10^{-6}\left[1 - 0.6094^2\right]}}$$

$$= 0.6 \text{ kg/s}$$

$$q_v = \frac{0.6}{781} \times 3600 = 2.77 \text{ m}^3/\text{h}$$

Mass velocity, $$G = \frac{q_m}{A_2} = \frac{0.6}{765.73 \times 10^{-6}} = 783.57 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.015 \times 783.57}{0.9 \times 10^{-3}} = 13\,059.5$$

$C_D = 0.775$ (From Fig. 6.22 of Ref.: 11)

No change in the values of q_m and q_v.

At diameter of tapered tube = 27 mm,

Float position, $h = 28.64$ mm,

$A_1 = 572.55 \text{ mm}^2$, $A_2 = 81.685 \text{ mm}^2$, $A_2/A_1 = 0.142\,67$

Flow rate of MEG: For the first trial, let $C_D = 0.77$

$$q_m = 0.77 \times 81.685 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 1098(6965 - 1098)}{490.87 \times 10^{-6}[1 - 0.142\,67^2]}}$$

$$q_m = 0.059 \text{ kg/s}$$

$$q_v = \left(\frac{0.059}{1098}\right) \times 3600 = 0.193\,44 \text{ m}^3\text{/h}$$

$$G = \frac{q_m}{A_2} = \frac{0.059}{81.685 \times 10^{-6}} = 722.287 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.002 \times 722.287}{40 \times 10^{-3}} = 36.114$$

$C_D = 0.7$ (from Fig. 6.22, Ref. 11)

$$q_m = \frac{0.7}{0.77} \times 0.059 = 0.0536 \text{ kg/s}$$

$$q_v = 0.1757 \text{ m}^3\text{/h}$$

For the same position of float, flow rate of ethyl alcohol:
Let $C_D = 0.775$ (for the first trial calculation)

$$q_m = 0.775 \times 81.685 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 781 \times (6965 - 781)}{490.87 \times 10^{-6}(1 - 0.142\,67^2)}}$$

$$q_m = 0.051\,34 \text{ kg/s}$$
$$q_v = 0.2367 \text{ m}^3\text{/h}$$

$$G = \frac{q_m}{A_2} = \frac{0.051\,34}{81.685 \times 10^{-6}} = 628.5 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.002 \times 628.5}{0.9 \times 10^{-3}} = 1396.67$$

$C_D = 0.775$ (From Fig. 6.22 of Ref. 11)

Hence q_m and q_v remain unchanged.
By similar calculations, following table is prepared.

Table 5.13 Flow Rates of Different Fluids for the Same Position of Float

Float position h, mm	Diameter of tapered tube D, mm	q_v m³/h		
		Water	MEG solution	Ethyl alcohol
0	25	0	0	0
28.64	27	0.2063	0.1757	0.2367
71.59	30	0.5672	0.5274	0.65
100.23	32	0.85	0.8	0.976
128.86	34	1.174	1.106	1.348
157.5	36	1.54	1.45	1.766
186.135	38	1.9528	1.84	2.24
214.77	40	2.4148	2.26	2.77

Following table is made from the graph (Fig. 5.16).

Table 5.14 Calibration for Rotameter

q_v, m^3/h	Float Position, h mm		
	Water	MEG Solution	Ethyl Alcohol
0.1	14.31	15.94	11.828
0.2	27.8	31.02	23.66
0.4	52.67	55.8	45.224
0.6	75.06	79.22	66.28
0.8	95.21	100.00	84.77
1.0	113.81	118.94	102.08
1.2	131.0	137.37	117.47
1.4	146.93	155.47	132.73
1.6	161.89	170.05	147.63
1.8	175.96	183.46	161.88
2.0	189.19	196.67	172.9
2.2	201.81	209.83	183.93
2.4	213.91	—	194.78

Comments: It can be seen from Table 5.13 that rotameter calibration is essentially immune from the viscosity effect (say up to about 100 mPa·s or cP). However, calibration is significantly affected by the density of the fluid which changes drag force.

In actual practice, rotameters are tested for actual water capacities on a test bench to confirm the calibration.

5.9 TWO-PHASE FLOW

In many applications, two-phase flow is experienced in industry. If pipe sizing is not done properly, it can result in serious problems. A few such problems are illustrated as follows:

1. When a saturated liquid under pressure is flashed through a control valve, two-phase flow will be realized in the downstream piping. Pressure drop in the two-phase flow could be much higher (several fold in some cases) in the pipe. This will result in much higher back pressure on the control valve, restricting its capacity. Apart from excessive pressure drop, there could be erosion in the pipeline.
2. When vapour and liquid flow in upward direction and the line is sized for low pressure drop, it may cause slug flow, i.e., slugs of liquid flow in vapour. This can result in pressure pulsation and severe vibration. This phenomenon is commonly observed and is known as *water hammer* (refer to Sec. 5.9.2) in steam lines, particularly during cold start-up when steam condenses during travel and water slugs flow with steam. Water hammers can break open the flanges or damage

the supports. Adequate draining facility is provided on long steam pipelines to avoid such water hammers.

3. If cooling water is flowing downward at temperature in excess of 40°C from a height of 10.5 m or more to an atmospheric pond, vibrations could result due to vapour formation at the top and two phase formation in downflow pipe. A provision of appropriate size orifice at the base of the pipe can solve the problem.

A process engineer should visualize such a problem and design the piping suitably so that both the phases flow smoothly without creating any problem.

Estimation of pressure drop in an isothermal two-phase system can be done with empirical relations, developed by Martinelli's and Dukler's correlations. Simpson[7] has dealt at a length on both the correlations (refer to Sec. 5.9.1)

A common problem of two-phase flow is experienced in steam condensate lines. When steam is used for heating in a heat exchanger, its condensate is discharged through a steam trap. While the heat exchanger operates under pressure, the trap discharges condensate to low pressure. In most cases, the condensate line opens into an atmospheric storage tank while in a few instances, condensate is conveyed to a deaerator, operating under positive pressure. In both cases, flashing of condensate takes place which calls for larger size pipe. If not designed properly, water hammer could be anticipated and also the back pressure on the trap could be much higher than anticipated. Steam traps are sized and rated for a specific pressure drop and condensate flow discharge rate. If the pressure drop is less than design, trap's capacity will be reduced and condensate accumulation is expected in the heat exchanger, thereby heat transfer will be affected.

Empirical correlations could be used for calculating pressure drop in steam condensate pipes. However, pipe sizing could be done with the help of Fig. 5. 17 which is based on approximate pressure drop of 0.1 kPa/m (or 1 mbar/m) length of pipe. This represents only friction loss. To this, head loss and equipment pressure (such as that of deaerator) are to be added to arrive at a back pressure of the trap.

Example 5.24 Saturated steam at 10 bar g is used in a heat exchanger at the rate of 2000 kg/h. Condensate is discharged through a trap and taken to a deaerator, located at 10 m above the heat exchanger. Pipe length, including equivalent length of fittings, is calculated to be 60 m. Deaerator operates at 0.4 bar g. Size the condensate pipe and calculate the back pressure on the trap.

Solution

Assume back pressure of 2 bar on the trap.

Steam at 10 bar g has saturation temperature of 184.13°C and condensate has specific enthalpy of 781.6 kJ/kg (from Steam Tables). At 2 bar g, specific enthalpy of condensate and flash vapours will be 562.2 kJ/kg and 2725.5 kJ/kg, respectively. If $\dot{m}$ is the flash steam rate after the trap, enthalpy balance can be written as

$$2000 \times 781.6 = \dot{m} \times 2725.5 + (2000 - \dot{m})\ 562.2$$

$$\dot{m} = 202.8 \text{ kg/h}$$

This represents 10.14% flashing. Specific volume of flash steam at 2 bar g and 133.7°C is 0.603 m^3/kg.

Thus volumetric flow rate of flash steam will be 122.29 m^3/h.

From Fig. 5.17, pipe size for 25 m/s flash steam velocity is read between 40 and 50 mm NB. Select 50 mm NB condensate pipe, downstream of trap which could be of 25 mm size.

Fig. 5.17 *Steam Condensate Line Sizing Chart*
(Reproduced with the Permission of Spirax-Sarco Ltd., UK)

Friction loss in pipe = 0.1 × 60 = 6 kPa ≡ 0.06 bar
Deaerator pressure = 0.4 bar
Elevation of deaerator = 10 m = 0.98 bar
Total back pressure = 0.06 + 0.4 + 0.98 = 1.44 bar
Calculated back pressure is less than the assumed back pressure. Condensate pipe design should therefore be satisfactory.

Refrigerants are another class of fluids which produce flash vapours on pressure reduction of saturated liquids. Quantity of flash vapour normally varies from 15 to 40%, depending on the refrigerant and the pressure difference across the expansion valve. To avoid any problem of high pressure drop, the expansion valve is normally situated close to the evapourator.

Charts, such as Fig. 5.17, are not readily available in literature for other fluids. Hence, empirical equations for two phase flow are to be used for pressure drop estimation.

5.9.1 Generalized Approach to Two-Phase Flow

For the cocurrent two-phase flow in a horizontal pipe, Lockhart and Martinelli correlations[13] are used for pressure drop calculations. These can be written as shown below.

$$\left(\frac{\Delta p}{L}\right)_{TP} = Y_l \left(\frac{\Delta p}{L}\right)_l \tag{5.35a}$$

$$\left(\frac{\Delta p}{L}\right)_{TP} = Y_g \left(\frac{\Delta p}{L}\right)_g \tag{5.35b}$$

where $(\Delta p/L)_l$ or $(\Delta p/L)_g$ can be calculated using the Fanning equation [Eq. (5.2)] by assuming that each phase is flowing alone in the pipe. Raynolds number (Re) and pressure drop (Δp) are determined based on superficial velocity of either phase.

$$Y_l = f_1(X) \text{ and } Y_g = f_2(X) \tag{5.36}$$

$$\text{where, } X = \left[\frac{(\Delta p/L)_l}{(\Delta p/L)_g}\right]^{1/2} \tag{5.37}$$

Based on foregoing equations, it can be deduced that

$$Y_g = X^2\, Y_l \tag{5.38}$$

Y_g and Y_l can be obtained from Fig. 5.18 in which 3 separate curves are shown for Y_g vs X and Y_l vs X. These three curves represent the following cases:

1. Flows of liquid and gas are in turbulent regions.
2. Flow of one phase is turbulent while another phase is laminar, i.e., viscous.
3. Flows of both phases are in laminar regions.

If $Re \leq 2000$, flow is considered laminar and if $Re > 2000$, flow is considered turbulent. These correlations can be applied to pipe diameters up to 100 mm (4 in) NB. Based on actual measurements, pressure drops, predicted by the above correlations are high for stratified, wavy and slug flows and low for laminar flow.

Fig. 5.18 *Parameters for Pressure Drop in liquid-gas flow through horizontal pipes. [Reproduced with the permission of McGraw-Hill Book Company, [USA]*

5.9.2 Water Hammer

It is a hydraulic shock which occurs in pipe whenever there is a sudden change in the velocity of liquid, flowing in the pipe. Conditions that can result in water hammer can be as given below.

1. Rapid closing of a valve. A control valve may shut instantly (may be in 1 or 2 seconds).
2. Start/stop or an abrupt change in pump speed.

In water hammer, kinetic energy of moving liquid is transformed into pressure energy due to sudden stoppage or abrupt change in velocity. Such a pressure rise in piping often results in severe mechanical damage.

If water is flowing through a pipeline then maximum pressure developed by water hammer is given by the following equation:

$$h_{wh} = \frac{a_w V_w}{g} \tag{5.39}$$

where,

h_{wh} = Maximum pressure developed by water hammer, m LC

V_w = Reduction in velocity, m/s

g = Gravitation acceleration = 9.81 m/s^2

a_w = Velocity of propagation of elastic vibration in the discharge pipe, m/s

$$= \frac{1420.37}{(1 + K_a R)^{1/2}} \tag{5.40}$$

R = Ratio of inside pipe diameter-to-wall thickness

K_a = Ratio of elastic modulus of water to the pipe material

Table 5.15 Values of K_a

Pipe material	K_a
Steel	0.01
Copper	0.017
Alluminum	0.03

Time interval (θ_s) required for the pressure wave to travel back and forth in the pipe is

$$\theta_s = \frac{2L}{a_w} \tag{5.41}$$

where, θ_s = time, s

L = Length of pipe, m

When the actual shut-off time for the valve (or pump stoppage) is shorter than θ_s, then maximum pressure equal to h_{wh} will be exerted on the closed system or pipeline which can cause water hammer.

Example 5.25 A 200 mm (8 in) NB SCH-40 carbon steel pipe is used for transferring water from reservoir to a user plant. Capacity of pump is 250 m^3/h. Length of discharge pipe from pump to control valve (including equivalent length of fittings) is 800 m. Calculate the maximum pressure, developed in pipe, when the control valve is suddenly closed.

Solution

Thickness of 200 mm NB SCH-40 pipe, t = 8.18 mm

Inside diameter of pipe = 202.72 mm

$$R = \frac{202.72}{8.18} = 24.78$$

$$V_w = \frac{q_v}{\frac{\pi}{4}(D_i^2)} = \frac{(250/3600)}{(\pi/4)(0.202\,72)^2} = 2.153 \text{ m/s}$$

$$a_w = \frac{1420.37}{(1 + 0.01 \times 24.78)^2} = 912.245 \text{ m/s}$$

$$h_{ws} = \frac{912.245 \times 2.153}{9.81} = 200.12 \text{ m WC}$$

$$\equiv 19.3757 \text{ atm}$$

Total pressure in piping:

$$p = 19.3757 \text{ atm} + \text{discharge pressure of pump}$$

Time interval for pressure wave travel:

$$\theta_s = \frac{2 \times 800}{912.245} = 1.75 \text{ s}$$

If actual shutoff time of the control valve is less than 1.75 s then water hammer pressure can be equal to p.

5.10 TROUBLESHOOTING OF FLUID FLOW SYSTEMS

Utilities such as cooling water, steam, refrigeration, etc., are important to process industry. Design of utility network (Chapter 11) should therefore be carried out carefully so that no user equipment starves of any utility. This can also result in energy saving.

Apart from utilities, piping of process fluids also require careful design. Piping should neither offer excessive pressure drop nor should be overdesigned as initial cost could be a deterrent.

In many instances, problems might arise in actual operation due to various reasons. Plant might be operated at a higher capacity thereby pumping capacity might become limiting, higher pressure drop might be experienced in pipe line, etc. In another example, fluid distribution in manifold piping might not be satisfactory. In yet another instance, a new boiler of higher capacity is added and steam from the new boiler is carried in the existing pipelines. In all these situations, process engineer is required to evaluate the bottlenecks and carry out troubleshooting exercise.

Take the instance of installation of new higher capacity boiler. If pressure drop calculations reveal high pressure drop in the existing steam pipeline, process engineer can consider laying of another parallel pipeline from the boiler to nearest steam consuming equipment. Alternately, steam at a higher pressure is conveyed from the boiler if the existing pipeline rating permits its operation at higher pressure.

Instrument air manifold becomes a bottleneck in many situations as more instruments are added over a period of operation. Also, fluctuations in supply instrument air pressure could be a problem. After careful evaluation, installation of an air receiver of adequate size near the plant may prove valuable.

For pumping system, upgradation of existing centrifugal pumps offers many options. While evaluating the options, safety, reliability, ease of maintenance and cost benefit analysis must be considered. Chaware *et al.*[14] have dealt in detail the upgrading options of a centrifugal pump. Impeller replacement, operation of two pumps in parallel, installation of a bigger pump, change of a type of a pump, etc., are the options that can be considered. In small and medium scale industry, suction pipe of the centrifugal pump is observed smaller or equal to suction nozzle size. If suction pipe size is selected one size higher than the nozzle size, pump capacity is observed to be in the design range. Process engineer can contribute significantly in troubleshooting.

EXERCISES

5.1 Answer the following:

(a) At 7 bar g, the leakage of steam through 3 mm, 6 mm and 12.5 mm diameter holes amounts to 34, 230 and 540 kg/h, respectively. One of the leakage figures is a typographical error. Which figure? Why?

(b) A two-stage reciprocating compressor is operating at 0.7 bar a inlet and 6.2 bar a outlet pressure. If the pressure drop across the intercooler is 0.14 bar, the pressure at the inlet of second stage will be approximately; (a) 3.38 bar a, (b) 2.69 bar a, or (c) 2.0 bar a.

(c) If volumetric flow rate and head of a centrifugal pump are proportional to speed raised to power 0.8 and 1.5, respectively (for a given specific speed), then power would be proportional to speed raised to power: (a) 3, (b) 2.3, (c) 2.0, (d) 1.5 or (e) 1.7.

(d) Pressure drop in a 150 mm NB diameter pipe in a well-designed distribution system is 0.07 bar due to water flowing at a certain rate. What will be the pressure drop in 80 mm NB pipe for the same length and same flow rate? (a) 4.4 bar, (b) 1.62 bar, (c) 1.1 bar, (d) 0.55 bar, (e) 0.276 bar or (e) 0.138 bar.

(e) An orifice type flow meter, installed on a liquid stream is coupled to a differential pressure (DP) transmitter and a flow recorder. Normal reading is 5 on a 0–10 square-root scale and 25 on a 0–100 linear scale. If the range of the DP transmitter is doubled, the reading will be

(i) 2.5 on square-root scale and 6.3 on linear scale.
(ii) 2.25 on square-root scale and 12.5 on linear scale or
(iii) 7.1 on square-root scale and 50 on linear scale.

(f) For a centrifugal pump, power formula can be written as $P = 80\ K\ (d)^3$

where, P = power in hp
K = dimensionless pump constant
d = impeller diameter, in

To write this formula in metric units, P should be in kW and d should be in cm. What will be the constant (instead of 80)?

5.2 A saturated gas mixture, obtained from a coal liquefaction plant after the acid removal section, has composition of 38.2% H_2, 12.3% CO, 18% CO_2, 31.0% CH_4 and 0.5% N_2 (by mole) on dry basis. It is available at 120 kPa a and 60°C. It is cooled to 40°C in a heat exchanger and sent to suction drum of a compressor. Condensate from the drum is to be continuously pumped out with the help of a centrifugal pump. Calculate the volume fraction of the gas mixture over the condensate and the artificial vapour pressure using ideal gas low.

Table 5.16 Solubility of Gases[9] in Water at 1 atm and 40°C

Gas	Solubility, kg/100 kg water
H_2	0.000 1384
CO	0.002 075
CO_2	0.0973
CH_4	0.001 586
N_2	0.001 391

5.3 Natural gas is to be delivered through 0.6 m diameter steel pipe. Straight length of pipe is 3 km. Equivalent length of fittings and valves of this pipe line is 300 m. Desired flow rate of natural gas is 180 000 Nm^3/h. For the purpose, a centrifugal compressor is installed at the Gas Gathering Station (GGS). Natural gas is to be delivered at the plant at 313 K and 170 kPa a and it leaves the compressor at 313 K. What pressure must be developed at the compressor discharge in order to achieve this flow rate? Average molar mass of natural gas is 17.4 and its viscosity at 313 K is 0.01 cP.

5.4 It is proposed to pump 5000 kg/h of acetic acid at 129.5°C and 1.4 atm a pressure from the reboiler of a distillation column to a hold up tank without cooling. If the friction loss in the line between the reboiler and pump is 8 kPa and the density of acetic acid is 1048 kg/m^3, how far above the pump must the liquid level in the reboiler be maintained to give a net positive suction head of 2.5 m? Friction loss in discharge line is 40 kPa. Maximum liquid level of acetic acid above the discharge point is 5 m. Pump efficiency is 60%. Calculate the power required to drive the pump.

5.5 Performance of centrifugal pump on water is known. It can deliver 100 m^3/h of water at 80 m total head of water with 65% efficiency. By using Fig. 5.7, predict the performance of this pump, if it is used to deliver the oil of viscosity 176 cSt (or 800 SSU) at the pumping temperature. Determine the head, capacity and power required by this pump when it is used to deliver oil. Density of oil at pumping temperature is 800 kg/m^3.

5.6 Hexane at 37.8°C is pumped through the system at a rate of 9.09 m^3/h. The tank is at atmospheric pressure. Pressure at the end of discharge line is 345 kPa g. The discharge head is 3.05 m and the suction lift is 1.22 m above the level of liquid in the tank. The friction loss in suction line is 3.45 kPa and that in the discharge line is 37.9 kPa. The mechanical efficiency of the pump is 0.6. The density of hexane is 659 kg/m^3 and its vapour pressure at 37.8°C is 33.71 kPa.
Calculate (a) $(NPSH)_A$, and (b) power required by centrifugal pump.

5.7 In a carbon dioxide liquefaction facility to liquefy 25 000 kg/day, it is condensed at –20°C and stored in a bullet. Before liquefaction, gas is compressed from atmospheric pressure and 35°C to 20 bar a, dried and sent to a chiller. Reciprocating compressors are selected for the duty. One engineer decides to use a two stage compressor while another decides to use a three stage compressor. Considering 75% overall mechanical efficiency of either compressor and equal compression ratio in each stage, calculate the power requirement in each case. Also calculate temperature of the gas at the discharge of each stage.
Data: Specific heat ratio, k for CO_2 = 1.3

5.8 Design an orifice meter based on the following data:
Name of fluid = Natural gas
Flow rate = 1000 Nm^3/h
Operating pressure = 1.35 atm a
Operating temperature = 38°C
Average molar mass of natural gas = 17.2
Viscosity of natural gas = 0.01 cP at 38°C
Inside diameter of pipe = 128.2 mm (5 in, SCH-40)
Specific heat ratio for natural gas (k) = 1.3

5.9 Design a venturi meter based on the data given in Example 5.21.
Mass flow rate through venturi meter is calculated by the following equation:

$$\dot{m} = \frac{C_v A_t}{\sqrt{1-\beta^4}}\sqrt{2 g_c \Delta p \rho} \tag{5.42}$$

where, C_v = Venturi coefficient = 0.98

A_t = Throat area of venturi = $\frac{\pi}{4} D_t^2$

D_t = Diameter of throat, m

β = Ratio of diameter of throat to diameter of pipe

g_c = 1

Δp = Pressure drop measured by manometer, Pa

ρ = Density of flowing fluid, kg/m^3

Angle of upstream cone of venturi meter is 30° and the same of downstream cone is 10°. Take β = 0.5. Draw a sketch of suggested venturi.
Determine: (a) Throat diameter of venturi, (b) Pressure drop and manometer reading, and (c) Lengths of upstream and downstream cones.

5.10 Determine the minimum and maximum flow rates of air at 40°C and at 1.5 atm a, which can be measured by the rotameter of the dimension of tube and float of rotameter, given in Fig. 5.15, except material of float is polypropylene.
Viscosity of air at 40°C = 1850×10^{-5} cP
Density of polypropylene = 905 kg/m^3
Thickness of float = 2 mm
Calibrate the rotameter or find the different values of flow rates for the different float positions.

5.11 In Example 5.22, a rotameter is calibrated to measure the flow rate of water at 40°C. If the same rotameter is used for measuring flow rates of glycerine at 30°C then what is the change in calibration?
Viscosity of glycerine at 30°C = 400 cP
Density of glycerine at 30°C = 1.25 kg/L

5.12 In a single stage ammonia-water absorption refrigeration plant, pure saturated liquid ammonia is flashed from 15.55 bar a to 2.90 bar a through a throttling valve. On flashing, 18.3% of liquid ammonia is converted to vapour. If the distance between the throttling valve and evapourator (chiller) is 2 m, determine the pressure drop for two-phase flow in the horizontal line.
Flow of saturated liquid ammonia (at upstream) = 0.1935 kg/s
Inside diameter of the pipe = 100 mm
Temperature of liquid-vapour mixture after flashing = –10°C.
At –10°C, density of liquid ammonia is 639 kg/m^3 and viscosity values of liquid and vapour ammonia are 0.15 cP and 0.0095 cP, respectively.

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Process Design of Heat Exchangers

6

6.1 INTRODUCTION

Second Law of Thermodynamics states that heat flows from higher temperature to lower temperature. All chemical reactions and unit operations involve heat transfer. Solids, liquids and gases require heating or cooling during various operations that are performed to get the desired product. This transfer can be performed in a variety of equipments called *heat exchangers*. Each of the equipments offer advantages and have limitations for a given service. This chapter is devoted to selection and process design of various types of heat exchangers and their integration in the process.

Shell- and tube-type heat exchangers are most common in the process industry. Plate heat exchangers, spiral heat exchangers, brazed finned-type heat exchangers, etc., are also used for specific applications. It is intended to cover all these type of heat exchangers in this chapter.

6.2 SHELL AND TUBE HEAT EXCHANGERS

Shell and tube heat exchangers are the most widely used equipments in chemical industry. This is because segmented baffles make such a heat exchanger sturdy enough for industrial use as longitudinal tube bundle when inserted in the shell gets support. They are mostly used as heat transfer equipments but in a few cases they are also used as Reactors, Falling Film Absorbers, etc. Sizes of various parts of shell and tube heat exchangers like shell, tubes, tierods are standardized. Standards developed by Tubular Exchanger Manufacturers Association, USA (TEMA) are universally used for design of shell and tube heat exchangers. Equivalent Indian Standard is IS:4503. TEMA or IS:4503 specify standard sizes of shell, tubes, tierods, etc., and also maximum allowable baffle spacing, minimum tube sheet thickness, baffle thickness, numbers of tierods required, etc. For mechanical design and fabrication, in addition to TEMA standard, ASME Code Section VIII DIV. **I** or IS:2825 is used. In TEMA standard, shell and tube heat exchangers are classified in three categories:

1. Class R covers heat exchangers which are used for severe duties in petroleum and related industries. Also it covers heat exchangers that are going to handle toxic gas, highly flammable fluid or hazardous fluid.
2. Class B covers the heat exchangers which are used in chemical process industries not involving severe duties.

(a) Internal Floating Head Heat Exchanger with Backing Ring, Type AES

(b) Fixed Tube Sheet Heat Exchanger, Type BEM

(c) Outside-Packed Floating Head Heat Exchanger, Type AEP

(d) U-Tube Heat Exchanger, Type CFU

(e) Kettle-type Floating Head Reboiler, Type AKT

(f) Exchanger with Packed Floating Tube sheet and Lantern Ring, Type AJW

Nomenclature of Parts of Various Shell and Tube Heat Exchangers

1. Stationary Head—Channel
2. Stationary Head—Bonnet
3. Stationary Head Flange—Channel or Bonnet
4. Channel Cover
5. Stationary Head Nozzle
6. Stationary Tube sheet
7. Tubes
8. Shell
9. Shell Cover
10. Shell Flange—Stationary Head End
11. Shell Flange—Rear Head End
12. Shell Nozzle
13. Shell Cover Flange
14. Expansion Joint
15. Floating Tube sheet
16. Floating Head Cover
17. Floating Head Flange
18. Floating Head Split Backing Ring
19. Split Shear Ring
20. Slip-on Backing Flange
21. Floating Head Cover—External
22. Floating Tube sheet Skirt
23. Packing Box Flange
24. Packing
25. Packing Gland
26. Lantern Ring
27. Tie rods and Spacers
28. Transverse Baffles or Support Plates
29. Impingement Plate
30. Longitudinal Baffle
31. Pass Partition
32. Vent Connection
33. Drain Connection
34. Instrument Connection
35. Support Saddle
36. Lifting Lug
37. Support Bracket
38. Weir
39. Liquid Level Connection

Fig. 6.1 *Different Types of Shell and Tube Heat Exchangers*[1] *(Fig. N-2 of TEMA 8th Edition), Reproduced with the Permission of the Tubular Exchanger Manufacturers Association Inc., USA*

3. Class C covers the heat exchangers which are used in commercial and in less important process applications. Example: Heat exchangers used for recovery of energy from an effluent stream.

Most popular and reliable softwares used for the design of shell and tube heat exchangers are as follows:

1. **HTRI:** Heat Transfer Research Inc., USA
2. **HTFS:** Heat Transfer and Fluid Flow Services, UK
3. **BJAC:** USA based company
4. **HEI:** Heat Exchange Institute, USA.

Design methods and equations used by these softwares are not available in open literature. For the design of shell and tube heat exchanger involving fluid without phase change, methods used by these softwares are based on Tinker's flow model (Sec. 6.11).

6.3 VARIOUS PARTS OF SHELL AND TUBE HEAT EXCHANGERS

6.3.1 Shell

Shell is the costliest part of the heat exchanger. Cost of shell and tube heat exchanger sensitively changes with change in the diameter of shell. As per the TEMA standard, shell size ranges from 6 in (152 mm) to 60 in (1520 mm). Standard pipes are available up to 24 in size (600 mm NB). If shell size is greater than 24 in, it is fabricated by rolling a plate.

Shell diameter depends on tube bundle diameter. For fixed tube sheet shell and tube heat exchanger, the gap between shell and tube bundle is minimum, ranging from 10 to 20 mm. For pull through floating head heat exchanger, it is maximum, ranging from 90 to 100 mm.

Required shell diameter is determined by one of the following three methods:

1. Based on the actual tube sheet layout drawing: All latest computer softwares available for the design of heat exchangers use this method to find shell inside diameter. Popular computer program, written in Autolips, is used to draw the tube sheet layout. To draw the tube sheet layout or to find the tube bundle diameter, total six parameters are fixed: (i) Outside diameter (OD) of tube, (ii) Numbers of tube side passes, (iii) Tube pitch (center-to-center distance between adjacent tubes), (iv) Tube arrangement (triangular, square, etc.), (v) Total number of tubes, and (vi) Type of shell and tube heat exchanger (fixed tube sheet, U-tube or floating head).

2. Use of standard tables: Based on actual tube sheet layout, standard tables are prepared. These tables give the maximum number of tubes that can be accommodated in various standard sizes of shell ranging from 6 in (150 mm NB) to 120 in (3000 mm NB), for the different numbers of tube side passes, different values of tube pitch and size and for the different arrangements of tubes. Table 6.1 gives the data on tube sheet layouts.

Table 6.1 Tube Sheet Tube Hole Count[2,3]

Table 6.1 (a) $^{5}/_{8}$ in OD tubes on $^{13}/_{16}$ in square pitch

Shell ID		TEMA P or S Number of passes				TEMA U Number of passes		
mm	**in**	**1**	**2**	**4**	**6**	**2**	**4**	**6**
203	8	55	48	34	24	52	40	32
254	10	88	78	62	56	90	80	74
305	12	140	138	112	100	140	128	108
337	13 ¼	178	172	146	136	180	164	148
387	15 ¼	245	232	208	192	246	232	216
438	17 ¼	320	308	274	260	330	312	292
489	19 ¼	405	392	352	336	420	388	368
540	21 ¼	502	484	442	424	510	488	460
591	23 ¼	610	584	536	508	626	596	562
635	25	700	676	618	600	728	692	644
686	27	843	812	742	716	856	816	780
737	29	970	942	868	840	998	956	920
787	31	1127	1096	1014	984	1148	1108	1060
838	33	1288	1250	1172	1148	1318	1268	1222
889	35	1479	1438	1330	1308	1492	1436	1388
940	37	1647	1604	1520	1480	1684	1620	1568
991	39	1840	1794	1700	1664	1882	1816	1754
1067	42	2157	2112	2004	1968	2196	2136	2068
1143	45	2511	2458	2326	2288	2530	2464	2402
1219	48	2865	2808	2686	2656	2908	2832	2764
1372	54	3656	3600	3462	3404	3712	3624	3556
1524	60	4538	4472	4310	4256	4608	4508	4426

Table 6.1 (b) $^{3}/_{4}$ in OD tubes on $^{15}/_{16}$-in triangular pitch

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	**in**	**1**	**2**	**4**	**6**	**1**	**2**	**4**	**6**	**2**	**4**	**6**
114	4.5	14	14	—	—	—	—	—	—	—	—	—
152	6	30	30	24	22	—	—	—	—	—	—	—
203	8	64	48	34	24	34	32	16	18	32	24	24
254	10	85	72	52	50	60	62	52	44	64	52	52

(*Contd.*)

Tube Sheet Tube Hole Count (*Contd.*)

Table 6.1 (b) (*Contd.*)

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	**in**	**1**	**2**	**4**	**6**	**1**	**2**	**4**	**6**	**2**	**4**	**6**
305	12	122	114	94	96	109	98	78	68	98	88	78
337	13 ¼	151	142	124	112	126	120	106	100	126	116	108
387	15 ¼	204	192	166	168	183	168	146	136	180	160	148
438	17 ¼	264	254	228	220	237	228	202	192	238	224	204
489	19 ¼	332	326	290	280	297	286	258	248	298	280	262
540	21 ¼	417	396	364	348	372	356	324	316	370	352	334
591	23 ¼	495	478	430	420	450	430	392	376	456	428	408
635	25	579	554	512	488	518	498	456	444	534	500	474
686	27	676	648	602	584	618	602	548	532	628	600	570
737	29	785	762	704	688	729	708	650	624	736	696	668
787	31	909	878	814	792	843	812	744	732	846	812	780
838	33	1035	1002	944	920	962	934	868	840	978	928	904
889	35	1164	1132	1062	1036	1090	1064	990	972	1100	1060	1008
940	37	1304	1270	1200	1168	1233	1196	1132	1100	1238	1200	1152
991	39	1460	1422	1338	1320	1365	1346	1266	1244	1390	1336	1290
1067	42	1703	1664	1578	1552	1611	1580	1498	1464	1632	1568	1524
1143	45	1960	1918	1830	1800	1875	1834	1736	1708	1882	1820	1770
1219	48	2242	2196	2106	2060	2132	2100	1998	1964	2152	2092	2044
1372	54	2861	2804	2682	2660	2730	2684	2574	2536	2748	2680	2628
1524	60	3527	3476	3360	3300	3395	3346	3228	3196	3420	3340	3286
1676	66	4292	4228	4088	4044							
1829	72	5116	5044	4902	4868							
1981	78	6034	5964	5786	5740							
2134	84	7005	6934	6766	6680							
2286	90	8093	7998	7832	7708							
2438	96	9203	9114	8896	8844							
2743	108	11696	11618	11336	11268							
3048	120	14459	14378	14080	13984							

Table 6.1 (c) 3/4 in OD tubes on 1 in square pitch

Shell ID		TEMA P or S Number of passes				TEMA U Number of passes		
mm	in	1	2	4	6	2	4	6
152	6	12	12	—	—	14	—	—
203	8	28	26	16	12	28	24	12
254	10	52	48	44	24	52	44	32
305	12	80	76	66	56	78	72	70
337	13 ¼	104	90	70	80	96	92	90
387	15 ¼	136	128	128	114	136	132	120
438	17 ¼	181	174	154	160	176	176	160
489	19 ¼	222	220	204	198	224	224	224
540	21 ¼	289	272	262	260	284	280	274
591	23 ¼	345	332	310	308	348	336	328
635	25	398	386	366	344	408	392	378
686	27	477	456	432	424	480	468	460
737	29	554	532	510	496	562	548	530
787	31	637	624	588	576	648	636	620
838	33	730	712	682	668	748	728	718
889	35	828	812	780	760	848	820	816
940	37	937	918	882	872	952	932	918
991	39	1048	1028	996	972	1056	1044	1020
1067	42	1224	1200	1170	1140	1244	1224	1212
1143	45	1421	1394	1350	1336	1436	1408	1398
1219	48	1628	1598	1548	1536	1640	1628	1602
1372	54	2096	2048	2010	1992	2108	2084	2068
1524	60	2585	2552	2512	2476	2614	2584	2558

Table 6.1 (d) 3/4 in OD tubes on 1 in triangular pitch (most popular arrangement)

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	in	1	2	4	6	1	2	4	6	2	4	6
114	4.5	14	14	—	—	—	—	—	—	—	—	—
152	6	26	26	24	22	14	14	—	—	14	12	10
203	8	42	40	26	24	31	26	16	12	32	24	24
254	10	73	66	52	44	56	48	42	40	52	48	40

(*Contd.*)

Tube Sheet Tube Hole Count (*Contd.*)

Table 6.1 (d) (*Contd.*)

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	**in**	**1**	**2**	**4**	**6**	**1**	**2**	**4**	**6**	**2**	**4**	**6**
305	12	109	102	88	80	88	78	62	68	84	76	74
337	13 ¼	136	128	112	102	121	106	94	88	110	100	98
387	15 ¼	183	172	146	148	159	148	132	132	152	140	136
438	17 ¼	237	228	208	192	208	198	182	180	206	188	182
489	19 ¼	295	282	258	248	258	250	228	220	226	248	234
540	21 ¼	361	346	318	320	320	314	290	276	330	316	296
591	23 ¼	438	416	382	372	400	384	352	336	400	384	356
635	25	507	486	448	440	450	442	400	392	472	440	424
686	27	592	574	536	516	543	530	488	468	554	528	502
737	29	692	668	632	604	645	618	574	556	648	616	588
787	31	796	774	732	708	741	716	666	648	744	716	688
838	33	909	886	836	812	843	826	760	740	852	816	788
889	35	1023	1002	942	920	950	930	878	856	974	932	908
940	37	1155	1124	1058	1032	1070	1052	992	968	1092	1056	1008
991	39	1277	1254	1194	1164	1209	1184	1122	1096	1224	1180	1146
1067	42	1503	1466	1404	1372	1409	1378	1314	1296	1434	1388	1350
1143	45	1726	1690	1622	1588	1635	1608	1536	1504	1652	1604	1560
1219	48	1964	1936	1870	1828	1887	1842	1768	1740	1894	1844	1794
1372	54	2519	2466	2380	2352	2399	2366	2270	2244	2426	2368	2326
1524	60	3095	3058	2954	2928	2981	2940	2932	2800	3006	2944	2884
1676	66	3769	3722	3618	3576							
1829	72	4502	4448	4324	4280							
1981	78	5309	5252	5126	5068							
2134	84	6162	6108	5954	5900							
2286	90	7103	7040	6898	6800							
2438	96	8093	8026	7848	7796							
2743	108	10260	10206	9992	9940							
3048	120	12731	12648	12450	12336							

Table 6.1 (e) 1 in OD tubes on 1¼ in square pitch

Shell ID		TEMA P or S Number of passes				TEMA U Number of passes		
mm	in	1	2	4	6	2	4	6
203	8	17	12	8	12	14	8	6
254	10	30	30	16	18	30	24	12
305	12	52	48	42	24	44	40	32
337	13 ¼	61	56	52	50	60	48	44
387	15 ¼	85	78	62	64	80	72	74
438	17 ¼	108	108	104	96	104	100	100
489	19 ¼	144	136	130	114	132	132	120
540	21 ¼	173	166	154	156	172	168	148
591	23 ¼	217	208	194	192	212	204	198
635	25	252	240	230	212	244	240	230
686	27	296	280	270	260	290	284	274
737	29	345	336	310	314	340	336	328
787	31	402	390	366	368	400	384	372
838	33	461	452	432	420	456	444	440
889	35	520	514	494	484	518	504	502
940	37	588	572	562	548	584	576	566
991	39	661	640	624	620	664	644	640
1067	42	776	756	738	724	764	748	750
1143	45	900	882	862	844	902	880	862
1219	48	1029	1016	984	972	1028	1008	1004
1372	54	1310	1296	1268	1256	1320	1296	1284
1524	60	1641	1624	1598	1576	1634	1616	1614

Table 6.1 (f) 1 in OD tubes on 1¼ in triangular pitch

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	in	1	2	4	6	1	2	4	6	2	4	6
203	8	27	26	8	12	18	14	8	12	14	12	6
254	10	42	40	34	24	33	28	16	18	28	24	24
305	12	64	66	52	44	51	48	42	44	52	40	40
337	13 ¼	81	74	62	56	73	68	52	44	64	56	52
387	15 ¼	106	106	88	92	93	90	78	76	90	80	78

(*Contd.*)

Tube Sheet Tube Hole Count (*Contd.*)

Table 6.1 (f) (*Contd.*)

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	**in**	**1**	**2**	**4**	**6**	**1**	**2**	**4**	**6**	**2**	**4**	**6**
438	17 ¼	147	134	124	114	126	122	112	102	122	112	102
489	19 ¼	183	176	150	152	159	152	132	136	152	140	136
540	21 ¼	226	220	204	186	202	192	182	172	196	180	176
591	23 ¼	268	262	236	228	249	238	216	212	242	224	216
635	25	316	302	274	272	291	278	250	240	286	264	246
686	27	375	360	336	324	345	330	298	288	340	320	300
737	29	430	416	390	380	400	388	356	348	400	380	352
787	31	495	482	452	448	459	450	414	400	456	436	414
838	33	579	554	520	504	526	514	584	464	526	504	486
889	35	645	622	586	576	596	584	548	536	596	572	548
940	37	729	712	662	648	672	668	626	608	668	636	614
991	39	808	792	744	732	756	736	704	692	748	728	700
1067	42	947	918	874	868	890	878	834	808	890	856	830
1143	45	1095	1068	1022	1000	1035	1008	966	948	1028	992	972
1219	48	1241	1220	1176	1148	1181	1162	1118	1092	1180	1136	1100
1372	54	1577	1572	1510	1480	1520	1492	1436	1416	1508	1468	1442
1524	60	1964	1940	1882	1832	1884	1858	1800	1764	1886	1840	1794
1676	66	2390	2362	2282	2260							
1829	72	2861	2828	2746	2708							
1981	78	3368	3324	3236	3216							
2134	84	3920	3882	3784	3736							
2286	90	4499	4456	4370	4328							
2438	96	5144	5104	4986	4936							
2743	108	6546	6494	6360	6300							
3048	120	8117	8038	7870	7812							

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3. Use of approximate equations: Approximate equations are available in literature to find the shell diameter or to find the tube bundle diameter. One of such equations[3] is given as follows:

$$D_b = d_o \left(\frac{N_t}{k_1} \right)^{\frac{1}{n_1}} \tag{6.1}$$

where, D_B = Tube bundle diameter, mm

d_o = Tube OD, mm

N_t = Total number of tubes

k_1 and n_1 are constants, values of which depend on ratio $\frac{P_t}{d_o}\left(=\frac{\text{tube pitch}}{\text{tube OD}}\right)$, arrangement of tubes and number of tube side passes.

Use Tables 6.2 and 6.3 for finding constants of Eq. (6.1).

Table 6.2 Constants for Eq. (6.1)

A. For P_t/d_o = 1.25, Triangular Pitch

No. of tube side passes	1	2	4	6	8
k_1	0.319	0.249	0.175	0.0743	0.0365
n_1	2.142	2.207	2.285	2.499	2.675

Table 6.3 Constants for Eq. (6.1)

B. For P_t/d_o = 1.25, Square Pitch

No. of tube side passes	1	2	4	6	8
k_1	0.215	0.156	0.158	0.0402	0.0331
n_1	2.207	2.291	2.263	2.617	2.643

Equation (6.1) can be used for fixed tube sheet type and floating head shell and tube-type heat exchangers. For U-tube heat exchangers, following equation can be used:

$$D_b = d_o\left(\frac{N_t'}{k_1}\right)^{\frac{1}{n_1}} \tag{6.2}$$

where, $$N_t' = N_t + \frac{D_b}{P_t} \tag{6.3}$$

N_t = Number of tube holes on tube sheet

After finding tube bundle diameter D_b, shell ID (Inside diameter); D_i can be determined by following equation:

$$D_i = D_b + C \tag{6.4}$$

where, C = Diametric clearance between shell ID and D_b

C = 10 to 20 mm for fixed tube sheet and U-tube

C = 50 to 80 mm for split-ring floating head

C = 90 to 100 mm for pull through floating head

6.3.2 Shell Side Pass Partition Plate

Single pass shell is used in the most of the cases. Two pass shell is rarely used and is recommended where shell and tube temperature difference is unfavourable for the single shell side pass. For such cases, normally two or more smaller size 1-1 heat exchangers, connected in series, are recommended. Shell side pass partition plate is not provided to improve shell side heat transfer coefficient but it is provided to avoid the unfavourable temperature difference or to avoid the cross of temperatures between hot fluid and cold fluid.

Example 6.1 Hot oil is to be cooled from 45°C to 37°C by cooling water. Cooling water will enter at 32°C and leave at 40°C. Select the suitable shell and tube heat exchanger.

Solution

Minimum driving force required for heat transfer in shell and tube heat exchanger is 3 to 5°C. Hence, for this case 1-1 shell and tube heat exchanger can be selected. But it will provide poor heat transfer coefficient and may require large heat transfer area. 1-2 shell and tube heat exchanger cannot be selected for this case because of the occurrence of hypothetical temperature cross between hot and cold fluids. 2-2 shell and tube heat exchanger can be selected. 2-2 heat exchanger will provide higher heat transfer coefficient than 1-1 heat exchanger. Hence, either select 2-2 heat exchanger or provide two identical heat exchangers with smaller diameters, connected in series.

6.3.3 Baffles

There are two functions of baffles:

1. Baffles are used in the shell to direct the fluid stream across the tubes to increase the velocity of shell side flow and thereby to improve the shell side heat transfer coefficient. In other words, baffles are used in shell to increase the turbulence in shell side fluid.

Note: This function is useful only if there is no phase change in shell side fluid.

2. Baffles indirectly support the tubes and thereby reduce the vibrations in tubes. If shell side liquid velocity is higher; say more than 3 m/s, vibration analysis calculations should be carried out to check whether baffle spacing is sufficient or not. Similarly, for very high velocity of gas or vapour and also for the higher baffle spacing (higher than shell ID), vibration analysis calculation must be carried out to check the baffle spacing. Vibration analysis calculations are given in TEMA standard.

Different types of baffles are used in shell and tube heat exchangers; (i) Segmental baffle, (ii) Nest baffle, (iii) Segmental and Strip baffle, (iv) Disk and Doughnut baffle, (v) Orifice baffle, (vi) Dam baffle, etc. Figure 6.2 shows various types of baffles.

Most widely used type of baffle is segmental baffle. Other types of baffles like nest baffle, segmental and strip baffle and disk and doughnut baffle provide less pressure drop for the same baffle spacing but provide lower heat transfer coefficients as compared to segmental baffle. Situations when other types of baffles might be used could be as given below.

1. For shell and tube heat exchangers shell side pressure drop controls the overall design. For example, intercoolers of compressors and heat exchangers used in very high vacuum system. In intercoolers of compressors maximum allowable shell side pressure drop may be as low as 3.45 kPa. For such cases different types of shells are also used. Split flow (G shell) or divided flow (J shell) designs provide low shell side pressure drop compared to commonly used single pass (E shell) as shown in Fig. 6.3.

Fig. 6.2 *Different Types of Baffles used in Shell and Tube Heat Exchangers*[2] *(Reproduced with the Permission of McGraw-Hill Education, USA)*

2. For shell and tube heat exchanger in which boiling or condensation is carried out on shell side. In such a case baffles are required only to reduce the vibrations in tubes.

For other type of baffles (other than segmental baffle) correlations for finding heat transfer coefficient and pressure drop are not easily available in open literature.

Helical baffles are also developed by some heat exchanger manufacturers. These are claimed to substantially alter the shell side flow pattern by inducing a swirling pattern with a velocity component parallel to the tubes. Optimum helix angle of 40° is recommended. This arrangement is expected to improve the heat transfer and reduce the pressure drop. However, its design procedure is proprietary.

Fig. 6.3 *(a) One Pass Shell (E Shall) (b) Split Flow (G Shell) (c) Divided Flow (J Shell)*

Segmental baffle is made by cutting the circular metal disk. Segmental baffles are specified in terms of % baffle cut. Fractional baffle cut is denoted by (***x***).

$$\% \text{ Baffle cut} = (h/D_B) \times 100 = \boldsymbol{x} \times 100 \tag{6.5}$$

where, h = Height of segment removed

D_B = Diameter of circular metal disk from which segment is removed

= ID of shell – Diametric clearance (***C***)

% Baffle cut ranges from 15 to 45%. If there is no phase change on shell side fluid, then decrease in % baffle cut increases shell side heat transfer coefficient. Decrease in window area results in increase in window velocity but at the expense of pressure drop. Baffle cut equal to 20 to 25% is found to be common in use.

All calculations in the book cover only segmental baffles.

Baffle outside diameter is always less than shell inside diameter. Certain clearance between baffle OD and shell ID is provided to facilitate removal and insertion of tube bundle for maintenance. Recommended diametric clearance between shell ID and baffle OD is as follows.

Table 6.4 Clearance Between Shell and Segmental Baffle[1]

Shell ID	**Diametric clearance, *C*** **Shell ID – Baffle OD = ($D_i - D_B$)**
Standard pipe shell 6 in to 24 in (150 mm to 600 mm)	1.6 mm
Plate shell 6 in to 25 in (150 mm to 625 mm)	3.2 mm
Plate shell 27 in to 42 in (675 mm to 1050 mm)	4.8 mm

If shell is fabricated from a plate then it may not be a perfect cylinder. Hence, for plate shell, more clearance is a permitted. For heat exchanger without phase change, increase in clearance between shell ID and baffle OD could mean decrease in shell side heat transfer coefficient due to leakage. Fraction of shell side fluid flowing through this clearance is not utilized for heat transfer. Hence this clearance, if it is more than recommended value, is undesirable.

In kettle-type reboiler, full baffle (with 0% baffle cut) is used, as in this type of heat exchanger extra space is available for the flow of shell side liquid.

In case of horizontal inclined condenser, segmental baffles with horizontal cut should not be used as they create resistance to the flow of condensate. Hence, in this case there are two options; (i) Use segmental baffles with vertical cut (Fig. 6.2 (h)) or (ii) Use segmental baffles with horizontal cut but their base must be trimmed (Fig. 6.2 (g)).

Tube holes are made in baffle. Diameter of tube hole in baffle is kept higher than tube OD. Normally this diametric clearance is less than 0.8 mm.

Thickness of baffle depends on size of shell and spacing between baffles. For the given size of shell, thickness of baffle increases with increase in baffle spacing. It can be determined from the table given in TEMA standard. As per Kern[5], baffle spacing B_s ranges from 0.2 to 1 times shell ID. Lesser baffle spacing gives higher heat transfer coefficient but at the expense of higher pressure drop. Optimum baffle spacing is in between 0.3 to 0.5 times shell inside diameter.

6.3.4 Tube

Tube size range from 1/4 in (6.35 mm) to 2.5 in (63.5 mm) in shell and tube heat exchanger. Data for standard tubes are given in TEMA standard and in Ref. 2. For the standard tubes, its size is equal to outer diameter of tube. Thickness of standard tubes are expressed in BWG (Birmingham Wire Gauge). Increase in the value of BWG means decrease in tube thickness. For no phase change heat exchangers and for condensers, 3/4 in (19.05 mm) OD tube is widely used in practice. While for reboiler 1 in (25.4 mm) OD tube size is common. Tubes are available in standard lengths like 6 ft (1.83 m), 8 ft (2.44 m), 12 ft (3.66 m), 16 ft (4.88 m) and 6 m.

6.3.5 Tube Side Pass Partition Plate

Tube side passes are provided to decrease the tube side flow area and to increase tube side fluid velocity thereby to improve the tube side heat transfer coefficient at the expense of pressure drop. This is true only if there is no phase change on tube side. Hence, more number of tube side passes are recommended only if there is no change in the phase of tube side fluid.

For example, at the design stage, if the number of tube side passes are increased from one to two, then for the given volumetric flow rate, flow area becomes half and velocity becomes double. Since, tube side heat transfer coefficient, $h_i \propto u_t^{0.8}$ (where u_t is tube side fluid velocity), on increasing number of tube side passes from 1 to 2, h_i nearly becomes 1.74 times. But, $\Delta p_t \propto u_t^{2.8}$, so the pressure drop increases by 6.96 times. Increase in h_i means decrease in heat transfer area required and decrease in fixed cost. Increase in Δp_t means increase in power required for pumping the tube

side fluid and increase in operating cost. Hence, ideally optimum number of tube side passes must be decided. Many a times, tube side velocity in a single pass could be calculated very low (say 0.3 to 0.5 m/s). Under such circumstances, two passes or four passes could be beneficial. It is recommended that $u_t > 1$ m/s. This is essential if tubeside fluid is cooling water; otherwise rate of fouling will be higher.

Tube side passes are very common and are advantageously used for improving tube side heat transfer coefficient. These passes can be achieved in many ways by locating partition plates in channel covers. Figure 6.4 gives different designs for achieving desired tube passes.

Fig. 6.4 *Nozzle Orientation Designs for Tube Side Passes*[4]

6.3.6 Tie Rods

Baffles are supported by tie rods (Fig. 6.5). Tie rods are made from solid metal bar. Normally four or more tie rods are required to support the baffles. Diameter of tie rod is less than the diameter of tube. Diameter and number of tie rods required for given shell diameter are specified by TEMA standard and IS:4503.

Fig. 6.5 *Baffle Spacer Detail*

6.3.7 Spacers

Spacers (Fig. 6.5) are used to maintain the space between baffles. Spacers are the pieces of pipes or in the most of the cases they are the pieces of extra available tubes. Spacers are passed over the tie rods and because of them baffles do not slide over tie rods under the effect of the force of fluid. Hence, spacers fix the location of baffles and maintain the space between them. Length of spacer is equal to space between the baffles.

6.3.8 Tube Sheet

Tubes and one end of tierods are attached to tube sheet (also called tube plate). Hence, entire load of tube bundle is transferred to one or two tube sheets. In U-tube shell and tube heat exchanger (as shown in Fig. 6.1(d), type CFU or Fig. 6.1(e), type AKT) only one tube sheet is used. While in fixed tube sheet shell and tube heat exchanger, two tube sheets are used. One surface of tube sheet is exposed to tube side fluid and other surface is exposed to shell side fluid. This point is very important in the selection of material for tube sheet and also in determining tube sheet thickness.

In majority cases tube to tube sheet joints are two types; (a) Expanded joint, and (b) Welded joint as shown in Fig. 6.6.

Fig. 6.6 *Tube to Tube Sheet Joint*[5]

In expanded type joint, tube holes are drilled in a tube sheet with a slightly greater diameter than the tube OD. Two or more grooves are cut in the wall of each hole. The tube is placed inside the tube hole and a tube roller is inserted into the end of the tube. Roller is slightly tapered. On application of the roller, tube expands and tube material flows into grooves and forms an extremely tight seal. Welding joint is used only for the cases where leakage of fluid can be disastrous.

Detailed method for calculating tube sheet thickness is given in TEMA and in IS: 4503. Minimum required tube sheet thickness is also specified in the same. Simplified equation is also available for calculating tube sheet thickness.

$$t = FG\left(\frac{0.25\,p}{f}\right)^{\frac{1}{2}} \tag{6.6}$$

where, t = Effective tube sheet thickness (minimum), mm

F = constant (1 for fixed tube sheet and 1.25 for U-tube)
p = Design pressure, kPa
f = Allowable stress of tube sheet material at design temperature, kPa
G = Mean diameter of gasket, mm

Then thickness of tube sheet T is given by equation

$T \geq$ (t + depth of pass partition groove + tube side corrosion allowance + shell side corrosion allowance)

Also $T \geq$ Minimum thickness specified in the standard

6.3.9 Sealing Strip

It is a shell side component. Sealing strips are attached on the inside surface of shell as shown in Fig. 6.7 throughout the length of shell.

Fig. 6.7 *Sealing Strips*

There are two functions of sealing strips:

1. Sealing strips reduce the amount of bypass stream of shell side fluid flowing through the clearance between shell inside diameter and tube bundle diameter and thereby improve the shell side heat transfer coefficient. (This is valid only if there is no phase change of shell side fluid).
2. Sealing strips also make the removal of tube bundle from the shell easy. Hence, they are also known as *sliding strips*.

In Kern's method (Ref. 5), it is assumed that entire shell side fluid is flowing across the tube bundle and between the baffles. Actually, shell side fluid is flowing in various ways. In all latest methods of process design, shell side fluid flow is divided in five streams. Among these streams, one of the streams is flowing through the clearance between shell ID and tube bundle. This clearance is significant in case of pull through floating head heat exchanger (about 90 to 100 mm). In addition, this clearance provides low pressure drop path for shell side fluid. Hence, actual fraction of shell side fluid bypassed through this clearance is considerably higher. Sealing strip partially blocks the gap between bundle and shell and thereby reduces the amount of this bypass stream considerably. In case of pull through floating head heat exchanger, all latest computer programs, like HTRI, suggest the use of sealing strip. Also in such programs shell side heat transfer coefficient is a function of dimension and numbers of sealing strips provided. Kern's method is not recommended for use to predict heat transfer coefficient and pressure drop for pull through floating head heat exchanger.

6.3.10 Expansion Joint

Expansion joint is attached to shell wall. In this case, shell is made from two pipe pieces. Two pipe pieces are joined together by an expansion joint as shown in Fig. 6.8.

Fig. 6.8 *Expansion Joints*[2]
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Expansion joint is used in fixed tube heat exchanger to permit the differential thermal expansion or contraction between shell and tubes which otherwise is not permitted by fixed tube sheet heat exchanger. Differential thermal expansion between shell and tube is significant if there is a large temperature difference between temperature of shell material and temperature of tube material during operation or if tube material and shell material are different. For the given case of fixed tube sheet heat exchanger, whether an expansion joint is required or not can be determined by calculations given in TEMA. If expansion joint is not provided in fixed tube sheet heat exchanger, then fixed tube sheet does not permit the unequal expansion or contraction between shell and tube and it can result in the development of thermal stress across the tube sheet. If this thermal stress is higher than permissible value, then it may develop a crack in tube sheet, and can result in leakage at tube to tube sheet joint. Other options, available to avoid the development of thermal stress in the tube sheet, are use of either U-tube heat exchanger or floating head heat exchanger.

6.4 ADVANTAGES AND DISADVANTAGES OF DIFFERENT TYPES OF SHELL AND TUBE HEAT EXCHANGERS OVER EACH OTHER

6.4.1 Advantages of U-tube and Floating Head Heat Exchangers (Figs. 6.1(a) and 6.1(d)) Over Fixed Tube Sheet Heat Exchangers

1. Maintenance and cleaning of tube bundle is easier. In U-tube and floating head heat exchanger, entire tube bundle can be easily taken out for cleaning and maintenance. While the same is not possible in fixed tube sheet heat exchanger.
2. U-tube and floating head heat exchangers permit the differential thermal expansion or contraction between shell and tube. Therefore, use of expansion joint is not required in U-tube and floating head heat exchangers.

6.4.2 Disadvantages of U-tube and Floating Head Heat Exchangers Over Fixed Tube Sheet

1. For a given heat duty, U-tube and floating head heat exchangers are costlier than fixed tube sheet heat exchangers because of the following reasons:
   (a) For the same number of tubes, tube arrangement and tube pitch, shell sizes required by U-tube and floating head heat exchangers are higher than the same required by fixed tube sheet heat exchangers. In U-tube heat exchanger, minimum bend radius of U-tube is $1.5d_o$. Hence, in central portion of tube bundle, tubes cannot be provided. In floating head heat exchanger more clearance between shell ID and tube bundle diameter (50 to 100 mm) is required.
   (b) In case of cooling or heating of shell side fluid (no phase change) heat transfer coefficients obtained in U-tube and floating head heat exchangers for the given heat duty are less than the same obtained in fixed tube sheet heat exchangers as bypassing area is higher in case of U-tube or floating head as compared to fixed tube sheet heat exchanger.

6.4.3 Advantages and Disadvantages of U-tube Heat Exchanger Over Floating Head Heat Exchanger

Advantage Fixed cost of U-tube heat exchanger is always less than the same of floating head heat exchanger for the given duty.

Disadvantages

1. In U-tube, tube material becomes weak in bending portion, hence use of U-tube heat exchanger is not recommended for severe conditions. While for the same conditions floating head heat exchanger can be used.
2. In U-tube heat exchanger number of tube side passes are fixed (two). It reduces the flexibility in design calculations.

3. U-tube heat exchanger is not recommended for tube side dirty fluid. As it is difficult to remove dirt from U-tube. While floating head heat exchanger can be used where both; shell side fluid and tube side fluid are relatively dirty.

6.4.4 Floating Head Heat Exchangers

There are two types of floating head heat exchangers.

1. Split-ring floating head heat exchanger
2. Pull through floating head heat exchanger

Split-ring floating head heat exchanger is widely used in the chemical process industries (Fig. 6.1 (a) with designation S of Fig. 6.9). With this type, split backing ring is provided. Advantages and disadvantages of this heat exchanger over pull through floating head (see designation T of Fig. 6.9) heat exchanger are as follows:

Advantage Compared to pull through floating head heat exchanger, less diameter is required for the shell to accommodate the same number of tubes.

Disadvantages More maintenance time is required. In maintenance, shell cover is removed first, then the split backing ring, then floating head cover and finally tube bundle is removed from the stationary end.

Pull through floating head heat exchanger is the costliest type of shell and tube heat exchanger because it requires largest shell diameter for the given numbers of tubes. With this heat exchanger, it is possible to remove the entire tube bundle including floating head assembly from the stationary end.

6.5 DIFFERENT TYPES OF TEMA DESIGNATIONS

In TEMA, standard different types of shell and tube heat exchangers are designated by three letters. First letter indicates type of front head, second letter indicates type of shell and third indicates type of rear head. Different types of TEMA designations are shown in Fig. 6.9.

For example, BEM shell and tube type heat exchanger means bonnet type (B type) front head, single pass (E type) shell and stationary head with fixed tube sheet (M type) rear head. BFL type shell and tube heat exchanger means bonnet-type front head, two pass shell with longitudinal baffle (F type shell) and stationary head with fixed tube sheet and removable flat cover (L type rear head).

6.6 GENERAL DESIGN METHOD FOR SHELL AND TUBE HEAT EXCHANGERS

6.6.1 Calculation of Heat Duty

1. For cooling or heating or for no phase change, heat duty is calculated by the equation

$$\phi = \dot{m} C_p \, \Delta t \tag{6.7}$$

where, ϕ = Heat duty required, kW

$\dot{m}$ = Mass flow rate of fluid, kg/s

C_p = Specific heat of fluid, kJ/(kg · °C)

Δt = Temperature difference to be effected, °C

Fig. 6.9 *TEMA-type Designations for Shell and Tube Heat Exchangers*[1]*. (Fig. No. N-1.2 of TEMA 8th Edition)*
(Reproduced with the Permission of Tubular Exchanger Manufacturers' Association, Inc. USA.)

2. For condensation with subcooling

$$\phi = \dot{m}\lambda + \dot{m}C_L\,\Delta t \tag{6.8}$$

where, $\dot{m}$ = Mass flow rate of vapour, kg/s

C_L = Specific heat of condensate, kJ/(kg · °C)

λ = Latent heat of vaporization, kJ/kg

3. For reboilers

$$\phi_B = \dot{m}_v\lambda \times 1.05 \qquad \text{(considering 5\% heat loss)} \tag{6.9}$$

where, $\dot{m}_v$ = Vaporization rate, kg/s

λ = Latent heat of vaporization, kJ/kg

Actually heat duty of the reboiler is determined based on energy balance around distillation column.

6.6.2 Selection of Cooling Medium or Heating Medium

Selection of cooling or heating medium mainly depends on the temperature (inlet temperature) at which cooling medium or heating medium is required. Other factors like heat transfer coefficient provided by cooling medium or heating medium, cost of the medium, etc., are also considered in the final selection. Tables 6.5 and 6.6 can be used as a guideline for the selection of heating medium and cooling medium respectively.

Table 6.5 Commonly Used Heating Fluids

Heating medium	Recommended temperature range
Saturated steam	100 to 180°C
Oil (Thermic Fluid)	180 to 300°C
Dowtherm E	180 to 260°C
Dowtherm A	300 to 400°C
Molten salt	400 to 590°C
Na-K alloys	590 to 750°C
Flue gas or Hot air	750 to 1100°C
Electric heating	> 1100°C

Table 6.6 Commonly Used Cooling Medium

Cooling medium	Operating range, °C	Recommended to use for temperature
1. Brines	–68 to 5	< 8°C
Examples:		
Ethanol water solution	–5 to 5	< 8°C
Methanol water solution	–40 to –1	< 8°C
Ethylene glycol solution	–34 to 5	< 8°C
Aqueous solution of calcium chloride	–20 to 0	< 8°C

(*Contd.*)

Table 6.6 (*Contd.*)

	Cooling medium	Operating range, °C	Recommended to use for temperature
	Aqueous solution of sodium chloride	–9 to 5	< 8°C
	Methylene chloride	–68 to –37	< –33°C
	Trichloroethylene	–68 to –37	< –33°C
	Trichlorofluoromethane	–68 to –37	< –33°C
2.	Chilled water	5 to 12	Below ambient
3.	Cooling water	32 to 60	ambient to 100°C
4.	Oil	–1 to 300	< 300°C
5.	Air	Atmospheric temperature	> 60°C

Heating mediums If temperature of heating medium is required in between 100° to 180°C, saturated steam is preferably used as heating medium. Saturated steam condenses in dropwise manner, hence it provides very high heat transfer coefficient [about 6000 W/(m^2 · °C)]. Superheated steam is not generally selected as heating medium for indirect (through the wall) heat transfer because desuperheating zone provides very low heat transfer coefficient [about 100-150 W/(m^2 · °C)].

If the temperature of heating medium required is greater than 180°C, then use of saturated steam as a heating medium is not economical. From 180°C to 300°C, hot oil (thermic fluid) is preferred as heating medium. Hot oil system is easily available at comparatively low cost. Hot oil provides low heat transfer coefficient and also properties of oil are uncertain. Dowtherm A® is an organic fluid of high heat stability, an eutectic mixture containing 73.5 percent diphenyl oxide and 26.5 per cent diphenyl by mass. Dowtherm A is used as a heating medium in both phases; as a liquid as well as saturated vapour. Dowtherm E is specially processed orthodichlorobenzene. For the precise application on a large scale, Dowtherm E can be considered as an alternate to hot oil. Molten salts are a molten mixtures of inorganic salts; one of which is an eutectic, consisting 40% $NaNO_2$, 7% $NaNO_3$ and 53% KNO_3. Sodium potassium alloys; 56% Na–44% K and 22% Na–78% K, are also used as heating mediums.

Cooling mediums In refrigeration terminology a brine is any liquid cooled by a refrigerant and circulated as a heat transfer fluid. Operating temperature range is –68°C to 5°C. For any fluid to be cooled below 8°C (minimum possible temperature of chilled water, 5°C + minimum driving force required for heat transfer in shell and tube heat exchanger, 3°C), brines are used as cooling medium. Brine may be

1. an aqueous solutions of inorganic salts, e.g., sodium chloride solution, calcium chloride solution, etc.
2. aqueous solutions of organic compounds such as alcohols or glycols, e.g., ethanol, methanol, ethylene glycol, propylene glycol, etc.
3. chlorinated or fluorinated hydrocarbons, e.g., methylene chloride, trichloroethylene, trichlorofluoromethane, halogenated hydrocarbons as refrigerants, etc.

Outlet temperature of chilled water from chilled water plant ranges from 5 to 15°C. While its return temperature is kept from 7°C to 12°C higher than inlet temperature. When any fluid is to be cooled below the temperature equal to design temperature of cooling water from cooling tower + minimum driving force required for heat transfer in shell and tube heat exchanger (3 to 5°C), chilled water is used as cooling medium.

Cooling water is the most widely used cooling medium at near ambient temperature.

Design temperature of cooling water from cooling tower = Design wet bulb temperature + Approach to the wet bulb temperature (ranges from 4 to 6°C)

Cooling water can be used as cooling medium even though inlet temperature of hot fluid is more than 100°C. But in this case extra care must be taken as at any time cooling water can be converted into steam. In case of exothermic reaction, oil (thermic fluid) is used for dual purpose. Initially to achieve the reaction temperature, oil is used as heating medium. Thereafter as the reaction proceeds, the same oil is used as cooling medium. For this, one heater and one cooling water cooler (or economizer) are provided in oil cycle.

Fig. 6.10 *Hot Oil (Thermic Fluid) Cycle*

For more information on design of process utility systems, refer to Chapter 11.

Air cooling Air cooled heat exchanger has certain advantages as well as disadvantages over cooling water cooled heat exchanger using cooling water.

Advantages

1. Generally operating cost of Air Cooled Heat Exchanger (ACHE) is less than the same of Water Cooled Heat Exchanger (WCHE). Operating costs of WCHE includes cost of make-up water for the cooling tower, power required for the cooling tower fans and for the circulating pump, cooling tower maintenance, etc. While operating cost of ACHE includes cost of power consumed by fans. Day by day cost of water increases and hence, the difference in operating cost between WCHE and ACHE increases.

2. Inlet temperature of water to cooling tower (or outlet temperature of cooling water from heat exchanger circuit) should not be greater than 60°C otherwise it creates the scale formation and also affects the material of construction of cooling tower. While for air practically there is no limit for temperature.
3. Fouling or scale formation does not take place with aircooling.

Disadvantages

1. Fixed cost of ACHE is higher than the same of WCHE. Heat transfer coefficient of air is quiet less than heat transfer coefficient provided by cooling water and hence, heat transfer area required by ACHE is more. Also design inlet temperature of cooling water from cooling tower is always less than design ambient temperature. It gives lesser Mean Temperature Difference (MTD) for ACHE compared to the same for WCHE and hence it increases heat transfer area required for ACHE.
2. Minimum driving force required for heat transfer, ΔT_{min}, is 10° to 15°C for ACHE and 3 to 5°C for WCHE. Hence, minimum temperature that can be achieved with ACHE is ambient temperature plus 10°C, while with WCHE it is design temperature of cooling water from cooling tower plus 3°C. Hence, if the fluid is to be cooled or condensed below 55°C in a tropical location, ACHE cannot be used.

Based on atmospheric conditions, cost of heat exchangers and cost of water, one can find out the temperature above which ACHE becomes cheaper than WCHE.

Fluid allocation

1. **Fluid allocation in case of phase change:** Condensing or vaporizing fluid is normally taken on shell side as in the shell, liquid and vapour phases are easily separated. In case of a condenser, if condensation is carried out in tube side, then the entire liquid condensate forms a film on heat transfer surface which provides additional resistance to heat transfer. In case of reboilers normally on both sides phase change take place and hence, fluid allocation depends on other factors.
2. **Fluid allocation in case of no phase change:** If there is no phase change in shell side and tube side fluid, then fluid allocation depends on the following factors:
   (a) *Corrosion:* Corrosive fluid is allocated tube side as the cheaper material like mild steel can be used for costly shell, baffles, etc. Also, other costlier parts like tubes and tube sheets require the use of non-corrosive or costlier material of construction in case of corrosive fluid, whether it is on tube side or in shell side. For example, in cooling of ethyliodide, it should be taken on tube side as it requires the use of special material (Hastelloy-B).
   (b) *Fouling:* In fixed tube sheet heat exchanger inside surface of tubes can be easily cleaned and hence, the fluid which has the greatest tendency to foul on heat transfer surface should be placed in the tubes. For example, cooling water has a tendency to foul. Hence, normally it is passed through tubes.
   (c) *Fluid temperature:* At very high temperature as well as at very low temperature use of special alloy is required. Hence, very hot or very cold fluid is placed on tube side to avoid the use of costlier material for the shell.

At moderate temperature, hotter fluid is better passed through tubes. If it is placed on shell side more insulation is required not only to reduce the heat loss but also for safety purpose.

(d) *Operating pressure:* High pressure stream should preferably be placed on tube side as high pressure tubes are cheaper than high pressure shell. Diameter of shell is very much greater than diameter of tubes hence, its thickness is more sensitively changed with change in pressure as compared to that of the tubes.

(e) *Fluid flow rates and viscosity:* Very low value of flow rate of fluid and high value of viscosity of fluid give low value of Reynolds number. Fluid which provides very low value of Reynolds number should be placed on shell side as the dependency of shell side heat transfer coefficient on Reynolds number is less as compared to the same of tube side heat transfer coefficient. Hence, for the lower value of Reynolds number shell side heat transfer coefficient is higher than tube side heat transfer coefficient. For example, in cooling of viscous oil, it is allocated shell side as it provides poor Reynolds number.

6.6.3 Energy Balance and Heat Transfer Calculations

Establish energy balance or heat duty balance and based on that find the mass flow rate of heating medium or cooling medium.

For example in case of condenser

$$\phi_t = \dot{m}\lambda = \dot{m}_w C_{Lw}(t_2' - t_1') \text{ kW} \tag{6.10}$$

where, $\dot{m}$ = Mass flow rate of vapour condensed, kg/s

λ = Latent heat of condensation kJ/kg

$\dot{m}_w$ = Mass flow rate of cooling medium required kg/s

C_{Lw} = Specific heat of cooling medium kJ/(kg · °C)

t_2', t_1' = Outlet/inlet temperatures of cooling medium, °C

6.6.4 Mean Temperature Difference

Mean temperature difference (MTD) is calculated from equation

$$\Delta\mathcal{T}_m = F_t \cdot \Delta\mathcal{T}_{lm} \tag{6.11}$$

where, $\Delta\mathcal{T}_{lm}$ = Logarithmic mean temperature difference, °C

$$\Delta\mathcal{T}_{lm} = \frac{\Delta\mathcal{T}_2 - \Delta\mathcal{T}_1}{\ln\left(\dfrac{\Delta\mathcal{T}_2}{\Delta\mathcal{T}_1}\right)} \tag{6.12}$$

where, $\Delta\mathcal{T}_2$ and $\Delta\mathcal{T}_1$ are terminal temperature differences.

For 1-1 heat exchanger (one shell side pass and one tube side pass), $F_t = 1$

When shell side passes and/or tube side passes are more than one, F_t must be determined.

$$F_t = f(R, S) \text{ where } R \text{ and } S \text{ are temperature ratios} \tag{6.13}$$

$$R = \frac{t_1 - t_2}{t_2' - t_1'} \tag{6.14}$$

$$S = \frac{t_2' - t_1'}{t_1 - t_1'} \tag{6.15}$$

where, t_1 = Hot fluid inlet temperature, °C
t_2 = Hot fluid outlet temperature, °C
t_1' = Cold fluid inlet temperature, °C
t_2' = Cold fluid outlet temperature, °C

Graphs of F_t vs S for the different values of R, for the different cases are given in Ref. 1. Figure 6.11 gives values of F_t for even number of tube passes. If $F_t < 0.75$, economic design of shell and tube heat exchanger cannot be achieved. For such a case, alternate type of heat exchanger design should be considered. In the cases where temperature cross is likely to occur, multiple shell pass heat exchanger or more numbers of heat exchangers in series, should be considered.

Fig. 6.11 *Temperature Correction Factor: One Shell Pass; Even Number of Tube Passes*[1] *(Fig. T-3.2A of TEMA, 8th Edition)*
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Empirical correlations[6] are reported by Wales in literature for calculating F_t for different passes on shell and tube sides.

6.6.5 Estimation of Overall Heat Transfer Coefficient

Assume the value of U, overall heat transfer coefficient. For this, use the standard tables, in which the range of the values of U for the different cases are given. Refer to Table 6.7 for the recommended values.

Table 6.7 Typical Overcell Coefficients for Shell and Tube Heat Exchangers

	Shell side	Tube side	U, W/(m$^2 \cdot$ °C)
		[A] Heat exchangers	
1.	Water	Water	800–1400
2.	Organic solvent	Water	300–850
3.	Organic solvent	Brine	200–500
4.	Light oil	Water	350–900
5.	Heavy oil	Water	60–300
6.	Water	Brine	600–1200
7.	Gas	Brine	15–250
8.	Gas	Gas	10–50
9.	Gas	Water	20–300
10.	Naphtha	Water	300–400
11.	Lube oil	Water	150–500
12.	Gasoline	Water	350–550
13.	Kerosene	Water	150–300
14.	Water	10 to 30% Caustic soda solution	500–1250
		[B] Condensers	
1.	Organic vapour	Water	700–1000
2.	Organic vapour + Gas	Water or Brine	100–300
3.	Low boiling hydrocarbons vapour mixture	Water	450–1100
4.	Naphtha vapour	Water	300–425
5.	Steam	Water	2000–5000
6.	Alcohol vapour	Water	500–1000
7.	Saturated Organic vapour	Water or Brine	280–680
		[C] Vaporizers/Reboilers	
1.	Light organics	Steam	800–1200
2.	Heavy organics	Steam	600–900
3.	Aqueous solution	Steam	1000–1500
4.	Chlorine	Steam	700–1500
5.	Ammonia	Steam	700–1500
6.	Water	Steam	1250–2000
7.	Refrigerants	Water	425–850

Find out anticipated heat transfer area based on this selected or assumed value of U.

$$A = \frac{\phi_t}{U \times \Delta T_m} \quad (\phi_t \text{ to be in W}) \tag{6.16}$$

For the first trial calculation, this A is the heat transfer area provided or actual heat transfer area of available or selected heat exchanger.

$$A = A_{\text{pro}} = N_t \pi\, d_o L \text{ m}^2 \tag{6.17}$$

where, d_o = Outside diameter of tube, m

L = Tube length, m

N_t = Total number of tubes

Values of d_o and L are decided by designer and from Eq. (6.17), value of N_t is determined. Then for the first trial, number of tube side passes and shell side passes are decided. Tube arrangement and type of heat exchanger are now selected.

6.6.6 Finding Shell Diameter

Based on all these informations find the inside diameter of shell. Select the type of baffle. First choice is 25% cut segmental baffle. Fix the value of baffle spacing. If there is cooling or heating on shell side then for the first trial baffle spacing B_s could be 0.3 to 0.5 times shell ID and if there is condensation or boiling on shell side, B_s could be equal to shell ID.

6.7 CLASICAL METHOD OF HEAT TRANSFER AND PRESSURE DROP CALCULATIONS FOR SHELL AND TUBE HEAT EXCHANGERS

6.7.1 Calculations of Tube Side Heat Transfer Coefficient

1. For heating or cooling on tube side (no phase change), tube side heat transfer coefficient is determined by Sieder–Tate equation. If Reynolds number; Re is less than 2000.

$$Nu = \frac{h_i d_i}{k_f} = 1.86\left(Re \cdot Pr \cdot \frac{d_i}{L}\right)^{0.33}\left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.18}$$

where, Nu = Nusselt number

In this equation

If $Nu \leq 3.5$ then Nu is taken 3.5.

If $Re > 4000$, tube side heat transfer coefficient is determined by Dittus–Bolter equation:

$$Nu = \frac{h_i d_i}{k} = C\, Re^{0.8} Pr^{0.33}\left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.19}$$

Re = Reynolds number = $\dfrac{d_i u_t \rho}{\mu} = \dfrac{d_i G_t}{\mu}$

Pr = Prandtl number = $\dfrac{C_p \mu}{k}$

h_i = Tube side heat transfer coefficient, W/(m^2 · °C)
d_i = Tube ID, m
L = Length of tube, m
k = Thermal conductivity of fluid, W/(m · °C)
C_p = Specific heat of fluid, kJ/(kg · °C)
μ = Viscosity of fluid at the bulk fluid temperature, (N · s)/m^2 or Pa · s
μ_w = Viscosity of fluid at tube wall temperature, (N · s)/m^2 or Pa · s
C = 0.021 for gases
= 0.023 for non-viscous liquid
= 0.027 for viscous liquid
G_t = Tubeside mass velocity, kg/(m^2 · s)
$= \dfrac{\dot{m}}{a_t}$

where, a_t = Tube side flow area = $\dfrac{N_t}{N_p} \times \dfrac{\pi}{4} d_i^2$ (6.20)

$\dot{m}$ = Tube side mass flow rate of fluid, kg/s
N_t = Number of tubes
N_p = Number of tube side passes
u_t = Tube side fluid velocity = G_t/ρ, m/s
ρ = Density of fluid, kg/m^3

To calculate tube side heat transfer coefficient, first calculate tube side flow area (a_t), next tube side mass velocity (G_t), then tube side Reynolds number (Re) and finally tube side Prandtl number (Pr). Depending on the value of Re, use Eq. (6.18) or Eq. (6.19) to calculate h_i.

For the transition region or for $2000 < Re < 4000$, no reliable equation is available, Hence, for this region h_i is found by using both Eqs. (6.18) and (6.19) and the lesser of two values is considered.

Alternatively, tube side heat transfer coefficient h_i can be calculated (by Kern's equation) from the value of "heat transfer factor; J_h", for the entire range of Reynolds number (from Re = 10 to 10^6). Equation relating h_i and J_h is

$$N_u = h_i \frac{d_i}{k} = J_h Re \cdot Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \tag{6.21}$$

Graph of J_h versus Re is given in Fig. 6.12 for the different values of L/d_i.

Fig. 6.12 *Tube Side Heat-Transfer Factor*[3]
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2. For condensation inside the tubes: Condensation coefficient depends on the position of condenser. For condensation, shell side condensation with horizontal position is the best which gives the maximum value of the coefficient. But if condensation is carried out in tube side then for horizontal position, h_i is calculated by following two equations and higher of the two values is considered:

(a) $$h_{ci} = 0.76\, k_L \left(\frac{\rho_L(\rho_L - \rho_v)g}{\mu_L \tau_h} \right)^{1/3} \tag{6.22}$$

This equation is called Nusselt equation, valid for stratified flow.

where, h_{ci} = Condensation coefficient, W/(m^2 · °C)
k_L = Thermal conductivity of liquid condensate, W/(m · °C)
ρ_L = Density of liquid condensate, kg/m^3
ρ_V = Density of vapour, kg/m^3
g = Acceleration of gravity = 9.81 m/s^2
μ_L = Viscosity of liquid condensate, (N · s)/m^2 or kg/(m · s) or Pa · s
τ_h = Horizontal tube loading or flow of condensate per unit length of tube, kg/(m · s)

All liquid condensate properties k_L, ρ_L and μ_L must be determined at mean temperature of condensate film.

$$\tau_h = \frac{\dot{m}_c}{L \times N_t} \tag{6.23}$$

where, L = Tube length, m
$\dot{m}_c$ = Total condensate flow, kg/s
N_t = Total number of tubes

(b) $$h_{ci} = h_i' \left[\frac{1 + \sqrt{\rho_L/\rho_V}}{2} \right] \tag{6.24}$$

where, $$h_i' = 0.021 \left(\frac{k_L}{d_i} \right) Re_c^{0.8} Pr_c^{0.43} \tag{6.25}$$

This equation is known as Boyko–Kruzhilin equation, valid for annular flow.

$$Re_c = \text{Reynolds number for the condensate film} = \frac{4\tau_h}{\mu_L} \tag{6.26}$$

$$Pr_c = \text{Prandtl number of liquid condensate} = \frac{C_{pL}\mu_L}{k_L} \tag{6.27}$$

For vertical position, $$h_{ci} = 0.926\, k_L \left(\frac{\rho_L(\rho_L - \rho_v)g}{\mu_L \tau_v} \right)^{1/3} \tag{6.28}$$

where, $$\tau_v = \frac{\dot{m}_c}{N_t \pi d_i} \tag{6.29}$$

$\dot{m}_c$ = Mass flow rate of liquid condensate, kg/s

$$Re_c = \frac{4\tau_v}{\mu_L} \tag{6.30}$$

This equation is also called Nusselt equation, valid for $Re_c \leq 2000$. For $Re_c > 2000$, use Boyko–Kruzhilin equation, i.e., Eq. (6.24).

6.7.2 Calculation of Tube Side Pressure Drop

1. If their is no phase change in tube side fluid, then tube side pressure drop can be calculated by following equation:

$$\Delta p_t = N_p\left(8J_f\left(\frac{L}{d_i}\right)\left(\frac{\mu}{\mu_w}\right)^{-m} + 2.5\right)\frac{\rho u_t^2}{2} \tag{6.31}$$

$$m = 0.25 \text{ for } Re \leq 2100$$
$$= 0.14 \text{ for } Re > 2100$$

where, Δp_t = Tube side friction pressure drop, N/m^2 or Pa
N_p = Number of tube side passes
J_f = Tube side friction factor can be obtained from Fig. (6.13)
ρ = Density of tube side fluid, kg/m^3
u_t = Tube side fluid velocity, m/s

Δp_t calculated by this equation is actually permanent pressure loss. Calculated pressure drop (loss) should be less than maximum allowable pressure drop. In some applications, maximum allowable pressure drop is decided by process conditions. While in other applications, maximum allowable pressure drop is actually optimum pressure drop (loss). Heat exchanger design means the balance between two opposite factors, heat transfer coefficients related to fixed cost and pressure drop related to operating cost. Increase in heat transfer coefficient by modifying the heat exchanger design also increases pressure drop. Hence, ideally actual pressure drop should be equal to optimum pressure drop which gives the total minimum cost of heat exchanger (i.e., fixed cost + operating cost). As a general guideline (Table 6.8) can be used to decide the value of maximum or optimum pressure drop. As per Kern, optimum pressure drop for gases is 2 psi (13.8 kPa) and for liquids it is 10 psi (69 kPa) (Ref. 5).

2. If condensation takes place inside the tubes then it is difficult to predict the pressure drop as vapour mass velocity is changing throughout the condenser. A common practice is to calculate $\Delta p_t'$ by Eq. (6.31) for inlet vapour flow rate and conditions, and multiply it by factor 0.5.

$$\Delta p_t = 0.5\ \Delta p_t' \tag{6.32}$$

6.7.3 Calculation of Shell Side Heat Transfer Coefficient

1. For heating or cooling on shell side or for no phase change on shell side, heat transfer coefficient h_o can be calculated by following procedure:

Fig. 6.13 *Tube Side Friction Factors*[3]
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(a) Calculate shell side flow area by following equation:

$$A_s = \frac{(P_t - d_o)D_s B_s}{P_t} \tag{6.33}$$

where, A_s = Shell side flow area, m^2
P_t = Tube pitch, m
d_o = Outside diameter of tube, m
B_s = Baffle spacing, m
D_s = Shell inside diameter, m

(b) Calculate shell side mass velocity G_s and linear velocity (u_s).

$$G_s = \frac{\dot{m}_s}{A_s} \tag{6.34}$$

$$u_S = \frac{G_s}{\rho_s} \tag{6.35}$$

where, G_s = Shell side mass velocity, $kg/(m^2 \cdot s)$
$\dot{m}_s$ = Shell side mass flow rate of fluid, kg/s
A_s = Shell side flow area
ρ_s = Density of shell side fluid, kg/m^3

(c) Calculate the shell side equivalent diameter (d_e).
For square pitch arrangement.

$$d_e = \frac{4 \times \text{cross-sectional area}}{\text{wetted perimeter}}$$

$$= \frac{4 \times \left(P_t^2 - \frac{\pi}{4} d_o^2\right)}{\pi d_o}$$

$$= \frac{1.27}{d_o}(P_t^2 - 0.785 d_o^2) \tag{6.36}$$

For triangular pitch arrangement,

$$d_e = \frac{4 \times \left(\frac{1}{2} P_t \times P_t \sin 60 - \frac{\pi}{8} d_o^2\right)}{(\pi d_o/2)}$$

$$= \frac{1.1}{d_o}(P_t^2 - 0.907 d_o^2) \tag{6.37}$$

(d) Calculate shell side Reynolds number.

$$Re_o = \frac{d_e G_s}{\mu} \tag{6.38}$$

where, μ = Viscosity of shell side fluid at average temperature, $kg/(m \cdot s)$

Also calculate shell side Prandtl number

$$Pr_o = \frac{C_p \mu}{k}$$

C_p, μ and k are the properties of shell side fluid at average temperature.

(e) Calculate the shell side heat transfer coefficient by the following correlation (Ref. 5):

$$Nu_o = \frac{h_o d_e}{k} = 0.36\, Re^{0.55} Pr^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.39}$$

This correlation is valid for the range of Reynolds number from 2000 to 1000 000. Alternately, shell side heat transfer, coefficient can also be found out from "shell side heat transfer factor; J_h" (by Kern's method).

$$Nu = \frac{h_o d_e}{k} = J_h \cdot Re \cdot Pr^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.40}$$

Graph of J_h versus Re_s given in Fig. 6.14.

2. If condensation takes place on shell side then its coefficient depends on the position of condenser.

Shell side condensation coefficient for horizontal position can be determined by following modified Nusselt equation:

$$h_{co} = 0.95\, k_L \left[\frac{\rho_L(\rho_L - \rho_V)g}{\mu_L \tau_h}\right]^{1/3} N_r^{-1/6} \tag{6.41}$$

where, h_{co} = Shell side condensation coefficient, W/(m^2 · °C)

k_L = Thermal conductivity of liquid condensate, W/(m · °C)

ρ_L = Density of liquid condensate, kg/m^3

ρ_V = Vapour density, kg/m^3

g = Acceleration of gravity = 9.81 m/s^2

μ_L = Viscosity of liquid condensate, N · s/m^2 or Pa · s

τ_h = Horizontal tube loading or flow of condensate per unit length of tube

$$= \frac{\dot{m}_c}{LN_t} \tag{6.23}$$

N_r = Average number of tubes in vertical tube row

$$N_r \cong \frac{2}{3} N_r' \tag{6.42}$$

N_r' = Number of tubes in central row

$$= \frac{D_b}{P_t} \tag{6.43}$$

D_b = Tube bundle diameter, m

P_t = Tube pitch, m

Fig. 6.14 *Shell Side Heat-transfer Factors for Segmental Baffles*[3]
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For condensation outside the vertical tube bundle, shell side condensation coefficient is given by Nusselt equation.

$$h_{co} = 0.926\, k_L \left(\frac{\rho_L(\rho_L - \rho_V)g}{\mu_L \tau_v} \right)^{1/3} \tag{6.28}$$

where, $$\tau_v = \frac{\dot{m}_c}{N_\tau \pi d_o} \tag{6.29}$$

In Nusselt equation; Eq. (6.22) and Eq. (6.28), suffixes i and o to h_c denote inside and outside of tubes, respectively.

6.7.4 Calculation of Shell Side Pressure Drop

1. For no phase change on shell side or for heating or cooling on shell side, shell side pressure drop Δp_s can be calculated by the following equation:

$$\Delta p_s = 8J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \left(\frac{\rho_s u_s^2}{2} \right) \left(\frac{\mu}{\mu_w} \right)^{-0.14} \tag{6.44}$$

where, Δp_s = Shell side pressure drop, N/m^2 or Pa
D_s = Shell inside diameter, m
d_e = Shell side equivalent diameter, m
L = Tube length, m
B_s = Baffle spacing, m
ρ_s = Density of shell fluid, kg/m^3
u_s = Shell side linear velocity, m/s
μ = Viscosity of fluid at the bulk fluid temperature, (N · s)/m^2
μ_w = Viscosity of fluid at tube wall temperature, (N · s)/m^2
J_f = Shell side friction factor for the different values of % baffle cut of segmental baffle and for the different values of *Re*. It can be obtained from Fig. 6.15.

Shell side pressure drop Δp_s should be less than maximum allowable pressure drop. Maximum allowable pressure drop for shell side is also decided either based on process requirement or based on economic considerations.

Table 6.8 can be used to judge optimum pressure drop.

Table 6.8 Optimum Pressure Drop (Maximum Allowable Pressure Drop) Based on Economic Considerations (Ref. 3)

Fluid	Optimum, Δp_t or Δp_s, kPa
Liquids of $\mu_L < 1$ cP or mPa · s	35
Liquids of $\mu_L = 1$ to 10 cP or mPa · s	50 to 70
Gas or vapour at 1 to 2 atm	13.8
Gas or vapour at high vacuum (up to 60 Torr absolute pressure)	0.4 to 0.8 (3 to 6 Torr)
Gas or vapour at high pressure > 10 atm	0.1 times operating pressure

Fig. 6.15 *Shell Side Friction Factors for Segmental Baffles*[3] *(Reproduced with the Permission of Elsevier, UK)*

2. In the case of shell side condensation, no reliable correlation is available for predicting Δp_s. Hence, Δp_s can be approximately calculated by following equation:

$$\Delta p_s = 0.5\,\Delta p_s' \tag{6.45}$$

where, $\Delta p_s'$ = shell side pressure drop calculated by Eq. (6.44) based on inlet vapour flow rate and inlet conditions.

This also should be less than maximum allowable pressure drop.

Calculated pressure drops on tube side and shell side do not account for nozzle losses.

6.7.5 Calculation of the Overall Heat Transfer Coefficients

$$U_o = \frac{1}{\dfrac{1}{h_o} + \dfrac{1}{h_{od}} + \dfrac{d_o \ln(d_o/d_i)}{2k_w} + \dfrac{d_o}{d_i} \times \dfrac{1}{h_{id}} + \dfrac{d_o}{d_i} \times \dfrac{1}{h_i}} \tag{6.46}$$

where, U_o = Overall heat transfer coefficient based on outside area of tubes, W/(m^2 · °C)

h_o = Shell side heat transfer coefficient, W/(m^2 · °C)

d_o = OD of tube, m

d_i = ID of tube, m

k_w = Thermal conductivity of tube wall material, W/(m · °C)

h_{id} = Fouling coefficient for tube side fluid, W/(m^2 · °C)

h_{od} = Fouling coefficient for shell side fluid, W/(m^2 · °C)

Fouling refers to any unwanted deposit of material on a heat transfer surface, or by extension, to any deposit that alters the desired fluid flow pattern. Fouling creates an additional resistance to heat transfer and increases pressure drop by narrowing flow passages.

For the various types of fluids, recommended fouling coefficients are given in Table 6.9.

Table 6.9 Recommended Fouling Coefficients (Ref. 3, 6 & 7)

Fluid	Fouling coefficient W/(m^2 · °C)	Fluid	Fouling coefficient W/(m^2 · °C)
Cooling water	3000–6000	Light Hydrocarbon	5000
Demineralized water	8000–10 000	Heavy Hydrocarbon	3000
Soft water	5000–8000	Aqueous Organic Solution	5000
Steam	4000–10 000	Vegetable oils	3000
Steam condensate	1500–5000	Caustic Solutions	5000
Brine	3000–5000	Gasoline	5000
Air	5000–10 000	Kerosene	3000–5000
Organic vapours	10 000	Light gas oil	3000–5000
Organic liquids	5000	Heavy gas oil	2000–3000
Heavy hydrocarbons	2000	Naphthas	5000
Aqueous salt solutions	3000–5000	Coal flue gas	1000

6.7.6 Calculating the Heat Transfer Area Required

$$A_r = \frac{\phi_t}{U_o \Delta \mathcal{T}_m} \quad (\phi_t \text{ to be in W}) \tag{6.47}$$

Also calculate actual heat transfer area A_o of selected heat exchanger.

$$A_o = N_t \pi d_o L \tag{6.17}$$

$$\frac{A_o}{A_{\text{req}}} \approx \{1.1 \text{ to } 1.2\}$$

$$\frac{\Delta p_t}{\Delta p_{t\,\max}} < \{0.8 \text{ to } 1\}, \quad \frac{\Delta p_s}{\Delta p_{s\,\max}} < \{0.8 \text{ to } 1\}$$

Thus an optimum design of shell and tube heat exchanger can be obtained.

Example 6.2 A lube-oil cooler for 2 MW steam turbine is to be designed for following operating conditions: Lube-oil, conforming to ISO VG-46 grade, flows at the rate of 450 L/min and is to be cooled from 65°C to 45°C with cooling water.

Kinematic viscosity of lube oil = 45.5 cSt at 40°C
Density of lube oil = 869 kg/m^3
Specific heat of lube oil = 2.1413 kJ/(kg · °C)
Thermal conductivity of lube oil at 55°C = 0.13 W/ (m · K)
Viscosity of lube oil at 55°C = 15 cP or mPa · s
Cooling water inlet/outlet temperatures = 35°C/39°C

Solution

Heat duty, $\phi_t = \dot{m}_o C_{Lo} \Delta t$

Volumetric flow rate of oil = 450 L/min = $450 \times 60 \times 10^{-3}$ m^3/h = 27 m^3/h

$$\dot{m} = 27 \times 869 = 23\,463 \text{ kg/h} \equiv 6.5175 \text{ kg/s}$$
$$\phi_t = 23\,463 \times 2.1413(65 - 45)$$
$$\phi_t = 1004\,826 \text{ kJ/h} \equiv 279.118 \text{ kW}$$
$$\equiv 279.118 \times 10^3 \text{ W}$$

Heat balance

$$\phi_t = \dot{m}_w C_{Lw} \Delta t'$$
$$279.118 = \dot{m}_w \times 4.1868(39 - 35)$$
$$\dot{m}_w = 16.667 \text{ kg/s } (\equiv 60 \text{ m}^3\text{/h})$$
$$\Delta \mathcal{T}_1 = 65 - 39 = 26°\text{C}$$
$$\Delta \mathcal{T}_2 = 45 - 35 = 10°\text{C}$$

Fig. 6.16

$$\Delta \mathcal{T}_{lm} = \frac{\Delta \mathcal{T}_1 - \Delta \mathcal{T}_2}{\ln\left(\frac{\Delta \mathcal{T}_1}{\Delta \mathcal{T}_2}\right)} = \frac{26 - 10}{\ln\left(\frac{26}{10}\right)} = 16.745°\text{C} \tag{6.12}$$

Allocate lube oil on tube side and cooling water on shell side.

For the first trial calculations, let the number of tube side passes $N_p = 4$

$$R = \frac{t_1 - t_2}{t_2' - t_1'} \qquad S = \frac{t_2' - t_1'}{t_1 - t_1'}$$

$$t_1 = 65°\text{ C}, \quad t_2 = 45°\text{ C}, \quad t_1' = 35°\text{ C}, \quad t_2' = 39°\text{ C}$$

$$R = \frac{65 - 45}{39 - 35} = 5 \qquad S = \frac{39 - 35}{65 - 35} = 0.1333$$

From Fig. 6.11, $F_t = 0.95$

Mean temperature difference, $\Delta T_m = F_t\, \Delta T_{lm}$

$$\Delta T_m = 0.95 \times 16.745 = 15.908°\text{C}$$

Assume overall heat transfer coefficient $U = 400$ W/(m^2·°C). (Ref.: Table 6.7)

Provisional heat transfer area for the first trial calculation.

$$A = \frac{\phi_t}{U \Delta T_m} = \frac{279.118 \times 10^3}{400 \times 15.908}$$

$$A = 43.8644 \text{ m}^2 = N_t \pi d_o L$$

$$d_o = 5/8 \text{ in} = 15.875 \text{ mm}, \quad L = 6 \text{ ft} = 1.8288 \text{ m}$$

$$N_t = \frac{43.8644}{\pi \times (0.015\,875 \times 1.8288)} = 480.93 \cong 480$$

Select BEM type heat exchanger with tube pitch $P_t = 1.25\ d_o = 19.8437$ mm and triangular pitch arrangement.

Tube bundle diameter $D_b = d_o \left(\frac{N_t}{k_1} \right)^{1/n_1}$ (6.1)

From Table 6.2 for $N_p = 4$, $k_1 = 0.175$, $n_1 = 2.285$

$$D_b = 15.875 \left(\frac{480}{0.175} \right)^{1/2.285} = 507.5 \text{ mm}$$

Let diametric clearance between shell ID and D_b, $C = 12.5$ mm

Shell ID, $D_i = D_b + 12.5$

$= 507.5 + 12.5 = 520$ mm

Evaluation of h_i:

Tube side flow area:

$d_i = 13.3858$ mm (for 18 BWG tube)

$$a_t = \frac{480}{4} \times \frac{\pi}{4} d_i^2 \qquad (6.20)$$

$$= \frac{480}{4} \times \frac{\pi}{4} (0.013\,3858)^2 = 0.016\,887 \text{ m}^2$$

Tube side mass velocity $G_t = \frac{\dot{m}}{a_t}$

$$G_t = \frac{(23\,463/3600)}{0.016\,687} = 390.57 \text{ kg/(m}^2 \cdot \text{s)}$$

Viscosity of oil:

$$\mu = 15 \text{ cP} = 15 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$Re_t = \frac{d_i G_t}{\mu} = \frac{0.013\,3858 \times 390.57}{15 \times 10^{-3}} = 348.54 \text{ which is low.}$$

To increase the Reynold number, let the number of tube side passes $N_p = 8$
$k_1 = 0.0365$, $n_1 = 2.675$ from Table 6.2.

$$D_b = 15.875\left(\frac{480}{0.0365}\right)^{\frac{1}{2.675}} = 550 \text{ mm}$$

Shell ID, $D_i = D_b + 12 = 562$ mm
Evaluation of h_i:

$$a_t = \frac{480}{8} \times \frac{\pi}{4}(0.013\,3858)^2 = 8.4436 \times 10^{-3} \text{ m}^2$$

$$G_t = 390.57 \times \frac{8}{4} = 781.14 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_t = \frac{G_t}{\rho} = \frac{781.14}{869} = 0.9 \text{ m/s}$$

$$Re_t = 348.54 \times 2 = 697 \quad \text{low but can be acceptable.}$$

$$Pr = \frac{C_p \mu}{k} = \frac{2.1413 \times 15 \times 10^{-3} \times 10^3}{0.13}$$
$$= 247$$

Using Sieder–Tate equation ($Re_t < 2000$)

$$Nu = \frac{h_i d_i}{k} = 1.86(Re \cdot Pr \cdot d_i/L)^{0.33}\left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.18}$$

$$= 1.86\left(697 \times 247 \times \frac{0.013\,3858}{1.8288}\right)^{0.33} \times 1$$

$$= 19.618 > 3.5$$

$$h_i = 19.62 \times \frac{k}{d_i} = \frac{19.618 \times 0.13}{0.013\,3858} = 190.53 \text{ W/(m}^2 \cdot {}^\circ\text{C)} \quad \text{(too low)}$$

To improve oil side heat transfer coefficient, allocate lube oil on shell side and water on tube side. Increase the tube length from 6 ft to 10 ft for the same value of heat transfer area provided. It will decrease the shell side flow area and will improve the shell side heat transfer coefficient.

$$L = 10 \times 0.3048 = 3.048 \text{ m}$$
$$A_{pro} = 43.8644 \text{ m}^2 = N_t \pi d_o L = N_t \times \pi \times 0.015\,875 \times 3.048$$
$$N_t = 288.56 \cong 288$$

Let number of tube side passes $N_p = 4$

$$k_1 = 0.175,\ n_1 = 2.285 \qquad \text{(Ref.: Table 6.2)}$$

$$D_b = 15.875\left(\frac{288}{0.175}\right)^{\frac{1}{2.285}} = 406 \text{ mm}$$

Shell ID, $D_i = 406 + 12 = 418$ mm

Evaluation of h_i:

$$a_t = \frac{288}{4} \times \frac{\pi}{4}(0.013\,3858)^2 = 0.01 \text{ m}^2$$

Tube side mass velocity, $G_t = \dfrac{16.667}{0.01} = 1666.7 \text{ kg/(m}^2 \cdot \text{s)}$

Density of water at 37°C, $\rho = 993.328$ kg/m^3 (Table 3.28 of Ref. 2)

$$u_t = \frac{G_t}{\rho} = \frac{1666.7}{993.328} = 1.678 \text{ m/s} \quad \text{(good velocity)}$$

$$Re = \frac{d_i G_t}{\mu}$$

Viscosity of water at 37°C, $\mu = 0.73$ cP $= 0.73 \times 10^{-3}$ kg/(m · s)

$$Re = \frac{0.013\,3858 \times 1666.7}{0.73 \times 10^{-3}} = 30\,561.8 > 4000$$

Thermal conductivity of water at 37°C, $k = 0.628$ W/(m · °C)

Prandtl number of water, $Pr = \dfrac{C_p \mu}{k}$

$$Pr = \frac{4.1868 \times 0.73 \times 10^{-3} \times 10^3}{0.628} = 4.867$$

Using Dittus–Bolter equation

$$Nu = \frac{h_i d_i}{k_f} = 0.023\, Re^{0.8}\, Pr^{0.33}\left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.19}$$

$$h_i = 0.023 \times \frac{0.628}{0.013\,3858} \times (30\,561.8)^{0.8} \times 4.867^{0.33}$$

$$= 7046.6 \text{ W/(m}^2 \cdot \text{°C)}$$

Evaluation of oil side heat transfer coefficient (h_o):

Shell side flow area

$$A_s = \frac{(P_t - d_o)}{P_t} \times D_i \times B_S \tag{6.33}$$

$$P_t/d_o = 1.25,\ D_i = 418 \text{ mm}, \quad B_s = \frac{D_i}{5} = \frac{418}{5} = 83.6 \text{ mm}$$

$$A_s = \frac{1.25d_o - d_o}{(1.25d_o)} \times 0.418 \times 0.0836 = 6.989 \times 10^{-3} \text{ m}^2$$

Shell side mass velocity, $G_s = \dfrac{\dot{m}}{A_s}$ (6.34)

$$G_s = \frac{23\,463/3600}{6.989 \times 10^{-3}} = 932.537 \text{ kg/m}^2 \cdot \text{s}$$

$$u_s = \frac{G_s}{\rho_s} = \frac{932.537}{869} = 1.073 \text{ m/s acceptable velocity} \qquad (6.35)$$

Shell side equivalent diameter for triangular pitch

$$d_e = \frac{1.1}{d_o}(P_t^2 - 0.907 d_o^2) \qquad (6.37)$$

$$d_e = \frac{1.1}{15.875}[19.8437^2 - 0.907 \times (15.875)^2]$$

$$= 11.4465 \text{ mm}$$

Shell side Reynolds number $Re_s = \dfrac{d_e G_s}{\mu}$

$$= \frac{0.011\,4465 \times 932.537}{15 \times 10^{-3}} = 711.62 \quad \text{(rather poor)}$$

$$Pr = \frac{C_p \mu}{k} = \frac{2.1413 \times (15 \times 10^{-3}) \times 10^3}{0.13}$$

$$= 247$$

For $Re = 711.62$, $J_h = 2 \times 10^{-2}$ (From Fig. 6.14 for 25% baffle cut)

$$Nu = \frac{h_s d_e}{k_f} = J_h \, Re \, Pr^{1/3} \left(\frac{\mu}{\mu_w}\right)^{0.14} \qquad (6.40)$$

$$h_s = 2 \times 10^{-2} \times \frac{0.13}{0.011\,4465} \times 711.62 \times (247)^{1/3} \times 1$$

$$= 1014.18 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Overall heat transfer coefficient by Eq. (6.46):

From Table 6.9,

$h_{od} = 3000$ W/(m^2 · °C), $h_{id} = 5000$ W/(m^2 · °C)

Tube material: steel or cupronickel

Thermal conductivity of tube material $k_w = 50$ W/(m · °C)

$$\frac{1}{U_o} = \frac{1}{1014.18} + \frac{1}{3000} + \frac{0.015\,875 \ln\left(\dfrac{15.875}{13.3858}\right)}{2 \times 50} + \frac{15.875}{13.3858} \times \frac{1}{5000} + \frac{15.875}{13.3858} \times \frac{1}{7046.6}$$

$$U_o = 570.8 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area required, $A_r = \dfrac{\phi_t}{U_o \Delta \mathcal{T}_m}$ (6.47)

$$= \frac{279.118 \times 10^3}{570.8 \times 15.908} = 30.739 \text{ m}^2$$

% Excess heat transfer area $= \left(\dfrac{A_{\text{pro}}}{A_r} - 1\right) \times 100$

$$= \left(\frac{43.8644}{30.739} - 1\right) \times 100 = 42.7\% \text{ (sufficiently high)}$$

Shell side pressure drop:

$$\Delta p_s = 8 J_f \left(\frac{D_s}{d_e}\right)\left(\frac{L}{B_s}\right)\frac{\rho_s u_s^2}{2}\left(\frac{\mu}{\mu_w}\right)^{-0.14} \quad (6.44)$$

$D_s = 0.418$ m, $d_e = 0.011\,4465$ m, $L = 3.048$ m

$B_s = 0.0836$ m, $u_s = 1.073$ m/s, $\rho_s = 869$ kg/m^3

For $Re_s = 711.62$, $J_f = 7.7 \times 10^{-2}$ (From Fig. 6.15)

$$\Delta p_s = 8 \times 7.7 \times 10^{-2}\left(\frac{0.418}{0.011\,4465}\right)\left(\frac{3.048}{0.0836}\right) \times \frac{869 \times 1.073^2}{2}$$

$$= 410\,281.7 \text{ Pa} \equiv 410.282 \text{ kPa} \quad \text{(too high)}$$

To decrease the shell side pressure drop, baffles spacing can be increased from 83.6 mm to 160 mm.

Revised value of $B_s = 160$ mm $= 0.16$ m

Shell side flow area, $A_s = 6.989 \times 10^{-3} \times \dfrac{0.16}{0.0836} = 0.013\,376 \text{ m}^2$

Shell side mass velocity, $G_s = 932.537 \times \dfrac{6.989 \times 10^{-3}}{0.013\,376} = 487.25 \text{ kg/(m}^2 \cdot \text{s)}$

$$u_s = \frac{G_s}{\rho_s} = \frac{487.25}{869} = 0.56 \text{ m/s} \quad \text{(rather poor)}$$

$$Re = \frac{0.011\,4465 \times 487.25}{15 \times 10^{-3}} = 371.82$$

for $Re = 371.82$, $J_h = 2.7 \times 10^{-2}$ (from Fig. 6.14)

$$Nu = \frac{h_s d_e}{k_f} = 2.7 \times 10^{-2} \times 371.82 \times (247)^{1/3} = 62.989$$

$$h_s = 62.989 \times \frac{0.13}{0.011\,4465} = 715.37 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$\frac{1}{U_o} = \frac{1}{570.8} - \frac{1}{1014.18} + \frac{1}{715.37}$$

$$U_o = 462.15 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$A_r = \frac{\phi_t}{U_o \Delta T_m} = \frac{279.118 \times 10^3}{462.15 \times 15.908} = 37.96 \text{ m}^2$$

$$\%\text{ Excess heat transfer area} = \left(\frac{43.8644}{37.96} - 1\right) \times 100 = 15.55\% \text{ (adequate)}$$

Revised shell side pressure drop:
For $D_s = 0.418$ m, $B_s = 0.16$ m, $d_e = 0.011\,4465$ m, $L = 3.048$ m, $u_s = 0.56$ m/s, $\rho_s = 869$ kg/m^3

$$\Delta p_s = 8J_f\left(\frac{D_s}{d_e}\right)\left(\frac{L}{B_s}\right)\frac{\rho_s u_s^2}{2}\left(\frac{\mu}{\mu_w}\right)^{-0.14} \tag{6.44}$$

For $Re_s = 371.82$, $J_f = 9.26 \times 10^{-2}$ (From Fig. 6.15)

$$\Delta p_s = 8 \times 9.26 \times 10^{-2}\left(\frac{0.418}{0.011\,4465}\right)\left(\frac{3.048}{0.16}\right) \times \frac{869 \times 0.56^2 \times 1}{2}$$

$$\Delta p_s = 70\,220.7 \text{ Pa} \equiv 70.22 \text{ kPa} \quad \text{(acceptable)}$$

Tube side pressure drop:

$$\Delta p_t = N_p\left(8\,J_f\left(\frac{L}{d_i}\right)\left(\frac{\mu}{\mu_w}\right)^{-m} + 2.5\right) \times \frac{\rho u_t^2}{2} \tag{6.31}$$

$N_p = 4$, $L = 3.048$ m, $d_i = 0.013\,3858$ m, $u_t = 1.678$ m/s
$\rho = 993.328$ kg/m^3, $\mu/\mu_w \cong 1$

For $Re_t = 30\,561.8$ $J_f = 3.6 \times 10^{-3}$ (From Fig. 6.13)

$$\Delta p_t = 4\left(8 \times 3.6 \times 10^{-3} \times \left(\frac{3.048}{0.013\,3858}\right) \times 1 + 2.5\right) \times \frac{993.328 \times 1.678^2}{2}$$

$$\Delta p_t = 50\,668 \text{ Pa} \equiv 50.668 \text{ kPa} \quad \text{(acceptable)}$$

Comment: In this case, viscosity of lube oil is very high. Hence, it gives very poor Reynolds number. Fluid, which provides poor Reynolds number must be allocated shell side to get the higher heat transfer coefficient. Here also, by changing the allocation of lube oil from tube side to shell side, lube oil side coefficient increased considerably. In any (without phase change) shell and tube heat exchanger for the fixed value of heat transfer area, increase in tube length (correspondingly decrease in number of tubes and shell inside diameter) increases the overall heat transfer coefficient but at the expense of pressure drop or in other words increase in tube length for the fixed value of heat transfer area decreases the fixed cost of heat exchanger but increases its operating cost.

6.8 CRITERIA OF SELECTION BETWEEN HORIZONTAL CONDENSER AND VERTICAL CONDENSER

1. For only filmwise condensation, horizontal position gives higher condensation coefficient than vertical position. In filmwise condensation, thickness of condensate film over heat transfer surface decides the value of condensation coefficient. With horizontal position and shellside condensation, the condensate travels less distance over heat transfer surface before falling down by gravity compared to vertical position. With vertical position, condensate travels over the entire tube length before falling down by gravity. Hence, average condensate film thickness obtained with horizontal position is less than the same with vertical position and hence, it can provide higher condensation coefficient.
2. In case of condensation with subcooling in a shell and tube heat exchanger, condensation coefficient is higher with horizontal position while subcooling coefficient is higher with vertical position. In case of subcooling with horizontal position, small fraction of gravitational force is acting on the pool of condensate and hence, condensate is flowing with almost no turbulence. Hence, subcooling with horizontal position is natural convection heat transfer. While in subcooling with vertical position, entire gravitational force is acting on pool of condensate. Hence, it creates turbulence in pool of condensate and provides higher subcooling coefficient. Subcooling with vertical position can be considered as forced convection heat transfer.

   Therefore, in the case of condensation with subcooling, shell and tube heat exchanger is designed for both positions (horizontal and vertical) and the position that gives higher value of overall coefficient is selected.
3. In case of condensation with non-condensables, selection of position depends on the percentage of non-condensables present in inlet vapour for which guidelines are given by Frank[5].
   (a) If non-condensables are < 0.5% (by mass), then the presence of non-condensables is ignored in design calculations. Heat exchanger is designed as a total condenser. For this case, horizontal position should be selected as it provides the higher condensation coefficient.
   (b) If non-condensables are > 70% (by mass), then for the entire flow rate, without phase change correlation is applied to calculate the heat transfer coefficient. However, in calculation of heat duty (ϕ_t), condensation is considered. For this case, selection of the position does not depend on heat transfer coefficient, as without phase change coefficient does not depend on position of heat exchanger so in this case position is decided by other factors like available area or available height.
   (c) Between 0.5 to 70% non-condensables, heat transfer coefficient is determined by considering both condensation as well as cooling of non-condensables and vapours. For this case horizontal position should be selected as it gives higher condensation coefficient, if condensation with cooling is carried out on shell side. If the same is carried out on tube side, then vertical position is better, because dry gas film over the heat transfer surface is continuously swept away by incoming stream.

Example 6.3 10 900 kg/h of nearly pure saturated methyl ethyl ketone vapour at 13.73 kPa g is to be condensed and cooled to 60°C by cooling water which is available in plant at 32°C. Consider pressure drops of 13.7 kPa for vapour and 68.7 kPa for the water as permissible. Design the suitable shell and tube heat exchanger for the heat duty.

Solution

Heat duty calculations

$$\phi_t = \dot{m}\lambda + \dot{m}C_L\Delta t = \phi_c + \phi_{\text{sub}}$$

where, $\phi_c = \dot{m}\lambda$ = heat duty for condensation

$\phi_{\text{sub}} = \dot{m}C_L\Delta t$ = heat duty for subcooling

$\dot{m}$ = 10 900 kg/h = 3.0278 kg/s

λ = Latent heat of vaporization of methyl ethyl ketone (MEK) at condensation temperature

At condensation temperature of pure MEK vapour

$$p_v = p_t = 13.73 \text{ kPa g} = 115.055 \text{ kPa a} = 863 \text{ Torr a}$$

Antoine equation for methyl ethyl ketone

$$\ln p_v = 16.5986 - \frac{3150.42}{T - 36.65}$$

where, p_v is in Torr and T in K

$$p_v = p_t = 863 \text{ Torr}, \quad T = 356.87 \text{ K} \quad t = 83.87°\text{C}$$

From Table 3-179 of Ref. 2,

λ_{MEK} at 78.2°C = 105.93 kcal/kg = 443.5 kJ/kg

Value of λ_{MEK} is required at 83.87°C.

Using Watson relation,

$$\lambda_2 = \lambda_1 \left(\frac{1 - T_{r2}}{1 - T_{r1}} \right)^{0.38} \tag{2.11}$$

T_r = reduced temperature = T/T_c

T_c of MEK = 535.6 K

$$\lambda \text{ at } 83.87°\text{C} = \lambda_{78.2°\text{C}} \left(\frac{1 - \dfrac{(83.87 + 273.15)}{535.6}}{1 - \dfrac{(78.2 + 273.15)}{535.6}} \right)^{0.38}$$

$$\lambda_{83.87°\text{C}} = 443.5 \times 0.9882 = 438.27 \text{ kJ/kg}$$

$$\phi_c = \dot{m}\lambda = 3.0278 \times 438.27 = 1327 \text{ kW}$$

Specific heat of MEK liquid at $\dfrac{(83.87 + 60)}{2} = 72°\text{C}$:

$$C_L = 2.298 \text{ kJ/(kg} \cdot °\text{C)} \qquad \text{(From Table 3-184 of Ref. 2)}$$

$$\phi_{\text{sub}} = 3.0278 \times 2.298 \times (83.87 - 60) = 166 \text{ kW}$$

$$\phi_t = \phi_c + \phi_{\text{sub}} = 1327 + 166 = 1493 \text{ kW}$$

Energy Balance

$$\phi = \dot{m}_w C_{pw} (t'_o - 32)$$

Fixing the outlet temperature of cooling water (t'_o) as 40°C,

mass flow rate of cooling water required

$$\dot{m}_w = \frac{\phi_t}{(C_{pw} \cdot \Delta t')} = \frac{1493}{(4.1868 \times 8)} = 44.5746 \text{ kg/s} \equiv 160.5 \text{ m}^3\text{/h}$$

First trial calculations:

Using Table 6.7, let overall heat transfer coefficients be

for condensation $U_c = 800$ W/(m^2 · °C)

for subcooling $U_{\text{sub}} = 200$ W/(m^2 · °C)

Assume that entire flow of cooling water is first utilized for subcooling and then for condensation.

$$\phi_{\text{sub}} = \dot{m}_w C_{pw}(t'_1 - 32)$$

where t'_1 = intermediate temperature of cooling water

$$166 = 44.5746 \times 4.1868 \times (t'_1 - 32)$$

$$t'_1 = 32.89°\text{C}$$

LMTD for condensation:

$$\Delta\mathcal{T}_1 = 83.87 - 32.89 = 50.98°\text{C}$$

$$\Delta\mathcal{T}_2 = 83.87 - 40 = 43.87°\text{C}$$

$$\Delta\mathcal{T}_{lm} = \frac{50.98 - 43.87}{\ln\left(\frac{50.98}{43.87}\right)} = 47.336°\text{C}$$

$$= \Delta\mathcal{T}_{mc}$$

Fig. 6.17(a) Condensation

For condensation of pure component $R = 0$ and $F_t = 1$, hence LMTD = MTD

LMTD for subcooling:

$$\Delta\mathcal{T}_1 = 83.87 - 32.89 = 50.98°\text{C}$$

$$\Delta\mathcal{T}_2 = 60 - 32 = 28°\text{C}$$

$$\Delta\mathcal{T}_{lm\,\text{sub}} = \frac{50.98 - 28}{\ln\left(\frac{50.98}{28}\right)} = 38.349°\text{C}$$

$$= \Delta\mathcal{T}_{m\,\text{sub}}$$

Fig. 6.17(b) Subcooling

Assume that subcooling will take place in the perfect counter current manner.

$F_t = 1$, hence LMTD = MTD

Area based on assumed values of U:

For condensation:

$$A_c = \frac{\phi_c}{U_c \Delta\mathcal{T}_{mc}} = \frac{1327 \times 1000}{800 \times 47.336} = 35.042 \text{ m}^2$$

For subcooling:

$$A_{\text{sub}} = \frac{\phi_{\text{sub}}}{U_{\text{sub}} \Delta\mathcal{T}_{m\text{sub}}} = \frac{166 \times 1000}{200 \times 38.349} = 21.643 \text{ m}^2$$

Total area $A = A_c + A_{\text{sub}} = 56.685$ m^2

$$\frac{A_{\text{sub}}}{A} = \frac{21.643}{56.685} = 0.3818 \text{ and } A_{\text{sub}} < A_c$$

Based on the selected values of overall coefficients, area required for subcooling (A_{sub}) is less than area required for condensation (A_c). Hence, to get the smaller size of heat exchanger horizontal position is selected. Ideally in such a case, heat exchanger should be designed for both positions and the position which requires lesser heat transfer area should be selected.

Area provided for the first trial calculation

$$A = 56.685 \text{ m}^2 = N_t \pi d_o L$$

Choose 3/4 in OD (19.05 mm OD), 6 ft (1.83 m) long tubes.

$$N_t = \frac{A}{\pi d_o L} = \frac{56.685}{(\pi \times 0.019\,05 \times 1.83)} = 518$$

Shell side fluid is a clean fluid, hence, select triangular pitch arrangement.

$$P_t = 1.25\, d_o = 23.8125 \text{ mm}$$

Number of tube side passes = 2 (for first trial calculations)

Tube bundle diameter:

$$D_b = d_o \left(\frac{N_t}{k_1} \right)^{\frac{1}{n_1}} \tag{6.1}$$

$$= 19.05 \left(\frac{518}{0.249} \right)^{\frac{1}{2.207}} \quad \text{(Using Table 6.2)}$$

$$= 607 \text{ mm}$$

Select a fixed tube sheet heat exchanger.

Let diametric clearance between shell ID and D_b, $C = 13$ mm

Shell inside diameter, $D_i = 607 + 13 = 620$ mm

Calculation of tube side heat transfer coefficient:

$$a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2 = \frac{518}{2} \times \frac{\pi}{4} (0.015\,748)^2 = 0.050\,45 \text{ m}^2$$

(From Table 11.2, of Ref. 2 for 16 BWG tube $d_i = 15.748$ mm)

Tube side mass velocity,

$$G_t = \frac{\dot{m}}{a_t} = \frac{44.5746}{0.050\,45} = 883.54 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_t = \frac{G_t}{\rho}$$

$$= \frac{883.54}{992.9} = 0.89 \text{ m/s} < 1 \text{ m/s}$$

To avoid the excessive fouling it is recommended to keep the velocity of water greater than 1 m/s.

Increase the number of tube side passes from 2 to 4.

for $N_p = 4$, $a_t = 0.025\ 22\ \text{m}^2$

$$D_b = 19.05\left(\frac{518}{0.175}\right)^{\frac{1}{2.285}} \qquad \text{(Table 6.2)}$$

$$= 630\ \text{mm}$$

Let diametric clearance between shell ID and D_b, $C = 15$ mm
Shell inside diameter = 630 + 15 = 645 mm (revised)

$$G_t = \frac{44.5746}{0.025\ 22} = 1767.43\ \text{kg/(m}^2 \cdot \text{s)}$$

$u_t = 1.78$ m/s acceptable

Viscosity of water at 36°C, $\mu = 0.72$ cP $= 0.72 \times 10^{-3}$ kg/(m · s)

$$Re_t = \frac{0.015\ 748 \times 1767.43}{0.72 \times 10^{-3}} = 38\ 657.6$$

Prandtl number (Pr):
Thermal conductivity of water at 36°C, $k = 0.6228$ W/(m · °C)

$$Pr = \frac{C_p \mu}{k} = \frac{4.1868 \times (0.72 \times 10^{-3})}{0.6228} \times \frac{10^3}{1}$$

$$= 4.84$$

Using Dittus–Bolter equation

$$\frac{h_i d_i}{k} = 0.023\ Re^{0.8}\ Pr^{0.33}\left(\frac{\mu}{\mu_w}\right)^{0.14} \qquad (6.19)$$

Considering $\left(\frac{\mu}{\mu_w}\right)^{0.14} = 1$

$$h_i = \frac{0.023 \times 0.6228}{0.015\ 748} \times (38\ 657.6)^{0.8} \times (4.84)^{0.33}$$

$$= 7155.5\ \text{W(m}^2 \cdot \text{°C)}$$

Shell side coefficient:
(a) For condensation zone, h_{co}:
Calculation for mean temperature of condensate film:
Let t_w = Tube wall temperature, °C
$h_{co} = 1500$ W/(m^2 · °C) (assumed for first trial calculation)
At steady state
Heat transfer rate through condensate film = overall rate of heat transfer

$$h_{co} A_c (t_c - t_w) = U_c A_c (t_c - t_{av})$$

where, t_c = Condensation temperature of vapour on shell side, °C
t_{av} = Average tube side fluid temperature for condensation zone, °C

$$1500(83.87 - t_w) = 800\left(83.87 - \frac{(32.89 + 40)}{2}\right)$$

$$t_w = 58.577°\text{C}$$

Mean temperature of condensate film

$$t_{av} = \frac{t_c + t_w}{2} = \frac{(83.87 + 58.577)}{2} = 71.22°\text{C}$$

Physical properties of liquid condensate at 71.22°C:
Viscosity of liquid MEK at 71.22°C, $\mu_L = 0.32$ cP $= 0.32 \times 10^{-3}$ kg/(m · s)
Density of liquid MEK at 71.22°C, $\rho_L = 805$ kg/m^3
Thermal conductivity of liquid MEK at 71.22°C, $k_L = 0.173$ W/(m · °C)
Shell side condensation coefficient with horizontal position

$$h_{co} = 0.95\, k_L \left(\frac{\rho_L(\rho_L - \rho_V)g}{\mu_L \tau_h} \right)^{1/3} \cdot N_r^{-1/6} \tag{6.41}$$

Acceleration of gravity $g = 9.81$ m/s^2

Density of vapour $\rho_v = \dfrac{PM}{RT} = \dfrac{PM}{T} \times \dfrac{T_s}{P_s V_s}$ (s for standard conditions)

$$\rho_v = \frac{863 \times 72}{(273.15 + 83.87)} \times \frac{273.15}{760 \times 22.414}$$

$$= 2.79 \text{ kg/m}^3$$

$$\tau_h = \frac{\dot{m}_c}{LN_t} = \frac{(10\,900/3600)}{(1.83 \times 518)} = 3.194 \times 10^{-3} \text{ kg/(m · s)} \tag{6.23}$$

Average number of tubes in vertical rows (6.42)

$$N_r = 2/3\, N_r' = \frac{2}{3}\left(\frac{D_b}{P_t}\right)$$

$$= \frac{2}{3} \times \frac{630}{23.8125} = 17.64 \cong 18$$

$$h_{co} = 0.95 \times 0.173 \left[\frac{805 \times (805 - 2.79) \times 9.81}{0.32 \times 10^{-3} \times 3.194 \times 10^{-3}} \right]^{1/3} \times (18)^{-1/6}$$

$$= 1864.86 \text{ W/(m}^2 \cdot °\text{C)}$$

h_{co} is close enough to assumed value of h_{co}. Hence, correction in the value of t_w is not required.

(b) For subcooling zone, $h_{o\text{sub}}$:

With horizontal position, subcooling is taking place via natural convection only for which reliable correlations are not available. Based on Kern's recommendation (Ref. 5), $h_{o\text{sub}}$ for horizontal position.

$$h_{o\text{sub}} = 50 \text{ Btu/(h · ft}^2 \cdot °\text{F)}$$
$$= 283.77 \text{ W/(m}^2 \cdot °\text{C)}$$

Overall Heat Transfer Coefficient for Condensation Eq. (8.46):
From Table 6.7,
For organic vapours, $h_{od} = 10\,000$ W/(m^2 · °C)

For cooling water, $h_{id} = 4000\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$

With methyl ethyl ketone and cooling water, stainless steel-304 is a suitable material for the tube.

Thermal conductivity of SS-304 material, $k_w = 16.3\ \text{W/(m} \cdot {}^\circ\text{C)}$

$$\frac{1}{U_{oc}} = \frac{1}{1864.86} + \frac{1}{10\,000} + \frac{0.019\,05 \ln\left(\frac{19.05}{15.748}\right)}{2 \times 16.3} + \frac{19.05}{15.748} \times \frac{1}{7155.5} + \frac{19.05}{15.748} \times \frac{1}{4000}$$

$$U_{oc} = 820.38\ \text{W/m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area required for condensation

$$A_{cr} = \frac{\phi_{\text{cond}}}{U_{oc} \times \Delta T_{mc}} = \frac{1327 \times 1000}{820.38 \times 47.336}$$

$$A_{cr} = 34.17\ \text{m}^2$$

Overall heat transfer coefficient for subcooling:

$$U_{o\text{sub}} = \frac{1}{\left[\frac{1}{283.77} + \frac{1}{10\,000} + \frac{0.019\,05 \ln(19.05/15.748)}{2 \times 16.3} + \frac{19.05}{15.748} \times \frac{1}{4000} + \frac{19.05}{15.748 \times 7155.5}\right]}$$

$$U_{o\text{sub}} = 237.72\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area required for subcooling,

$$A_{\text{subr}} = \frac{\phi_{\text{sub}}}{U_{o\text{sub}} \times \Delta T_{m\text{sub}}} = \frac{166 \times 1000}{237.72 \times 38.349} = 18.21\ \text{m}^2$$

Total heat transfer area required

$$A_{tr} = A_{cr} + A_{\text{subr}} = 34.17 + 18.21 = 52.38\ \text{m}^2$$

$$\frac{A_{t\text{pro}}}{A_{t\text{req}}} = \frac{56.685}{52.38} = 1.082$$

or Excess heat transfer area = (1.082 – 1) × 100 = 8.2%

% Excess heat transfer area should be at least 10%. Hence, to increase the value of heat transfer area, increase the tube length. New value of tube length is

$$L \cong 1.83 \times \frac{1.15}{1.082} = 1.95\ \text{m}$$

Let new or revised tube length $L = 2$ m with same number of tubes

Heat transfer area provided $A_{\text{pro}} = \frac{2}{1.83} \times 56.685 = 61.95\ \text{m}^2$

Revised value of shell side condensation coefficient

$$h_{oc} \propto L^{1/3}$$

$$h_{oc} = \left(\frac{2}{1.83}\right)^{1/3} \times 1864.86 = 1920.9 \text{ W/(m}^2 \cdot \text{°C)}$$

New value of U_{oc}

$$\frac{1}{U_{oc}} = \frac{1}{820.38} - \frac{1}{1864.86} + \frac{1}{1920.9}$$

$$U_{oc} = 831 \text{ W/(m}^2 \cdot \text{°C)}$$

$$A_{oc} = \frac{1327 \times 1000}{831 \times 47.336} = 33.73 \text{ m}^2$$

$$A_{\text{sub}} = 18.21 \text{ m}^2$$

$$A_{\text{req}} = 33.73 + 18.21 = 51.94 \text{ m}^2$$

$$\frac{A_{t\text{pro}}}{A_{t\text{req}}} = \frac{61.95}{51.94} = 1.1927$$

Excess heat transfer area = (1.1927 – 1) × 100 = 19.27%

If the equal percentage of excess heat transfer area (19.27%) in both zones are provided then,

Heat transfer area provided for condensation:

$$A_{c\text{pro}} = 33.73 \times 1.1927 = 40.23 \text{ m}^2$$

Heat transfer area provided for subcooling:

$$A_{\text{subpro}} = 18.21 \times 1.1927 = 21.72 \text{ m}^2$$

$$\frac{A_{\text{sub}}}{A_{t\text{pro}}} = \frac{21.72}{61.95} = 0.3506$$

Hence, 35.06% of total heat transfer area should be provided for subcooling.

Assuming that tube will be uniformly distributed in the cross section of shell.

$$\frac{A_{\text{subpro}}}{A_{t\text{pro}}} = \frac{\text{Area of segment of cross section utilized for subcooling}}{\text{Total cross-sectional area of shell}}$$

$$0.3506 = \frac{x\, D_i^2}{\frac{\pi}{4} D_i^2}$$

$$x = \frac{\pi}{4} \times 0.3506 = 0.2754$$

From Table 1.19b of Ref. 2, for $x = 0.2754$, $h/D_i = 0.382$

Hence, 38.2% of shell inside diameter should be submerged in the pool of condensate to facilitate subcooling.

This can be achieved by providing inverted U-seal (Fig. 6.18) height of inverted U-seal from the base of shell ID.

$$h = 0.382 \times 645 = 246.4 \text{ mm}$$

Fig. 6.18 *Detail of U-Seal*

Shell side pressure drop Δp_s:
Shell side flow area

$$A'_s = \frac{(P_t - d_o)B_s D_s}{P_t} \times x'$$

where $x' = 1 - \dfrac{h}{d_i} = 1 - 0.382 = 0.618$

Let baffle spacing B_s = Shell ID = 645 mm

$$A_s = \frac{(23.8125 - 19.05)}{23.8125} \times 0.645 \times 0.645 \times 0.618 = 0.051\,42 \text{ m}^2$$

Shell side mass velocity, $G_s = \dfrac{\dot{m}}{A_s} = \dfrac{3.0278}{0.051\,42}$

$$G_s = 58.884 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_s = (G_s/\rho_v) = \frac{58.884}{2.79} = 21.105 \text{ m/s}$$

Equivalent diameter for triangular pitch arrangement

$$d_e = \frac{1.1}{d_o}(P_t^2 - 0.907 d_o^2) \tag{6.37}$$

$$= \frac{1.1}{19.05}(23.8125^2 - 0.907 \times 19.05^2) = 13.736 \text{ mm}$$

$$Re_s = \frac{0.013\,736 \times 58.884}{1100 \times 10^{-7} \times 10^{-1}} = 73\,530$$

(Viscosity of MEK vapour at 83.87°C = 1100×10^{-7} Poise)
From Fig. 6.15, for 25% cut segmental baffle
Shell side friction factor, $J_f = 0.0358$ for $Re = 73\,530$

$$\Delta p_s = 0.5 \times 8 \times J_f \left(\frac{D_S}{d_e}\right)\left(\frac{L}{B_S}\right)\frac{\rho_v u_s^2}{2}\left(\frac{\mu}{\mu_w}\right)^{-0.14} \tag{6.44}$$

$$= 0.5 \times 8 \times 0.0358\left(\frac{645}{13.736}\right)\left(\frac{2000}{645}\right) \times \frac{2.79 \times 21.105^2}{2}$$

$$= 129\ 56 \text{ Pa} \equiv 12.956 \text{ kPa} < \Delta p_{s\max} \ (13.73 \text{ kPa})$$

To decrease the shell side pressure drop further increase the % baffle cut from 25% to 35%, then for 35% cut segmental baffle

$$J_f = 0.03$$

$$\Delta p_s = \frac{0.03}{0.0358} \times 12.956 = 10.86 \text{ kPa} < \Delta p_{s\max} \ (13.73 \text{ kPa})$$

Tube side pressure drop, Δp_t:

$$\Delta p_t = N_p \left(8 J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \times \frac{\rho u_t^2}{2} \tag{6.31}$$

$$L = 2000 \text{ mm}, \quad N_p = 4, \quad d_i = 15.748 \text{ mm}, \quad \rho = 993.684 \text{ kg/m}^3,$$

$$u_t = 1.78 \text{ m/s}$$

$$Re_s = 38\ 657.6, J_f = 3.41 \times 10^{-3} \text{ (from Fig. 6.13)}$$

$$\Delta p_t = 4 \times \left(8 \times 3.41 \times 10^{-3} \times \left(\frac{2000}{15.748} \right) \times 1 + 2.5 \right) \times \frac{993.684 \times 1.78^2}{2}$$

$$\Delta p_t = 37\ 558 \text{ Pa} \equiv 37.558 \text{ kPa} < \Delta p_{t\max} \ (68.6 \text{ kPa})$$

Resulting Heat Exchanger Specifications:

1-4, fixed tube sheet shell and tube heat exchanger (BEM type),
Shell ID = 645 mm, Baffles: 35% cut segmental,
Baffle spacing = 645 mm,
Tube OD = 19.05 mm, 16 BWG, Tube length = 2000 mm,
Δ Pitch arrangement, $P_t = 1.25\ d_o$, Number of tubes, $N_t = 518$

Note: As shown in this example, in case of condensation with subcooling, inverted U-seal or dam baffle is required to accumulate condensate within the condenser.

6.9 CONDENSATION WITH NON-CONDENSABLES IN SHELL AND TUBE HEAT EXCHANGERS

For the design of this type of heat exchanger, methods available are: (i) Colburn and Hougen method, and (ii) Porter and Jaffreys method. But these methods are complex, requiring trial and error calculations. These methods consider both resistances; resistance provided by heat transfer and diffusional resistance provided by mass transfer.

Approximate methods are also available for the same. In approximate methods, only the resistance, offered by heat transfer is considered while mass transfer resistance is neglected. Hence, in these methods it is assumed that overall rate of heat transfer in condensation with cooling of non-condensables is totally controlled by heat transfer resistance. One of the approximate methods is Gilmore's equation[5].

$$\frac{1}{h_{cg}} = \frac{1}{h_c} + \frac{\phi_g}{\phi_t} \frac{1}{h_g} \tag{6.48}$$

where, h_{cg} = Heat transfer coefficient for condensation with cooling of non-condensables, W/(m^2 · °C)

h_c = Average condensate film coefficient, determined by using single component condensation correlations, at the average condensate composition and total condensate loading, W/(m^2 · °C)

h_g = Average gas film coefficient determined for the average vapour gas mixture flow rate, W/(m^2 · °C)

ϕ_g = Total sensible heat transfer from vapour-gas mixture, kW

ϕ_t = Total heat transfer, kW

ϕ_t = Latent heat of vaporization + sensible heat for cooling the gas-vapour mixture.

For non-condensables, present in vapour, guidelines given by Frank (Sec. 6.8), may be followed.

Example 6.4 Air saturated with *n*-butanol vapour at 49 kPa g pressure and at 100°C temperature is to be sent to a shell and tube heat exchanger for recovering butanol. Air-butanol vapour mixture is to be cooled to 50°C by cooling water which enters at 32°C and leaves the heat exchanger at 40°C.

Air flow rate (butanol free) = 500 Nm^3/h

Check that whether the shell and tube heat exchanger with following specifications is suitable for the given duty earlier:

(i) Type of heat exchanger: BEM as per TEMA

(ii) Material of Construction of heat exchanger: SS-304

(iii) Shell ID = 387 mm
Tube ID/OD = 15.748/19.05 mm

(v) Tube length = 1500 mm

(vi) Tube Pitch = 25.4 mm

(vii) Number of tubes = 134

(viii) Number of tube side passes = 4

(ix) Number of shell side passes = one

(x) Baffle type = 25% cut segmental

(xi) Baffle spacing = 120 mm

Properties of fluids and material

(a) Thermal conductivity of SS-304, k_w = 16 W/(m · °C)

(b) Antoine equation for butanol

$$\ln p_v = 17.2160 - \frac{3137.02}{T - 94.43}$$ where p_v is in Torr and T is in K.

(c) Latent heat of vaporization of butanol at 75°C, λ = 642 kJ/kg

(d) Physical properties of butanol condensate at average condensate film temperature

Specific heat, C_L = 2.8763 kJ/(kg · °C)

Viscosity, μ_L = 0.95 cP or mPa · s

Thermal conductivity, k_L = 0.168 W/(m · °C)

Density, $\rho_L = 810$ kg/m^3

(e) Physical properties of air-butanol vapour mixture of average composition at 75°C and at the operating pressure:

Specific heat, $C_p = 1.3043$ kJ/(kg · °C)

Viscosity, $\mu = 0.0174$ cP or mPa · s

Thermal conductivity, $k = 0.03$ W/(m · K)

Density, $\rho = 2.012$ kg/m^3

(f) Physical properties of cooling water

$C_{Lw} = 4.1868$ kJ/(kg · °C), $\mu_w = 0.72$ cP

$k_w = 0.63$ W/(m · K), $\rho_w = 1000$ kg/m^3

(g) Fouling coefficients $h_{od} = h_{id} = 5000$ W/(m^2 · °C)

Solution

Heat duty equation for the given case

$$\phi_t = \dot{m}_a C_{pa} \Delta t + \dot{m}_v C_{pv} \Delta t + \dot{m}_{BC} \lambda = (\dot{m}_a + \dot{m}_v)\, C_{p\text{av}}\, \Delta t + \dot{m}_{BC} \lambda$$

where, ϕ_t = Heat duty, kW

$\dot{m}_a$ = Mass flow rate of air, kg/s

$\dot{m}_v$ = Average mass flow rate of butanol vapour, kg/s

$\dot{m}_{BC}$ = Condensation rate of butanol, kg/s

C_{pa} = Specific heat of air, kJ/(kg · °C)

C_{pv} = Specific heat of butanol vapour, kJ/(kg · °C)

λ = Latent heat of vaporization of butanol, kJ/kg

$C_{p\text{av}}$ = Specific heat of air-butanol vapour mixture, kJ/(kg · °C)

Density of air at normal condition

$$\rho_{\text{air}} = \frac{pM}{RT} = \frac{pM}{T} \times \frac{T_s}{p_s V_s} \qquad (s \text{ for standard conditions})$$

$$\rho_{\text{air}} = \frac{1 \times 29}{(273.15 + 25)} \times \frac{273.15}{1 \times 22.414} = 1.1853 \text{ kg/m}^3$$

$\dot{m}_a = 500 \times 1.1853 = 592.65$ kg/h $\equiv 0.1646$ kg/s

$\dot{m}_v$ = Average flow rate of butanol vapour

$$\dot{m}_v = \frac{\dot{m}_{vi} + \dot{m}_{vo}}{2}$$

where, $\dot{m}_{vi}$ = Mass flow rate of butanol vapour at inlet, kg/s

$\dot{m}_{vo}$ = Mass flow rate of butanol vapour at outlet, kg/s

At inlet, air is saturated with butanol vapour at 100°C and at 49 kPa g.

Vapour pressure of butanol at 100°C = $p_v = \overline{p}_v$

where $\overline{p}_v$ = partial pressure of butanol vapour at inlet

Using Antoine equation, for $T = 100 + 273.15 = 373.15$ K, $p_v = 385.617$ Torr
Mole fraction of butanol vapour in incoming air-butanol mixture:

$$y_i = \frac{\overline{p}_v}{p_t} = \frac{385.617}{p_t}$$

$$p_t = 49 \text{ kPa g} = 49 + 101.325 = 150.325 \text{ kPa a} \equiv 1127.53 \text{ Torr a}$$

$$y_i = \frac{385.617}{1127.53} = 0.342$$

If $\dot{n}_a$ = molar flow rate of air, kmol/s

$$\dot{m}_{vi} = y_i \times \left(\frac{\dot{n}_a}{1 - y_i}\right) \times \text{molar mass of butanol } (C_4H_9OH)$$

$$= 0.342 \times \frac{(0.1646/29)}{(1 - 0.342)} \times 74 = 0.2183 \text{ kg/s}$$

$$\dot{m}_{vo} = y_o \left(\frac{\dot{n}_a}{1 - y_o}\right) \times \text{molar mass of butanol}$$

y_o = Mole fraction of butanol vapour in air-butanol vapour mixture at outlet

$$= \frac{\overline{p}'_v}{p'_t} = \frac{p'_v}{p'_t}$$

At the outlet also air is saturated with butanol vapour.

$\overline{p}'_v = p'_v$ = vapour pressure of butanol at 50°C temperature

Using Antoine equation, for $T = 273.15 + 50 = 323.15$ K, $p'_v = 32.8336$ Torr

p'_t = Operating pressure of air – Vapour mixture of butanol at outlet = $p_t - \Delta p_s$

where, Δp_s = Shell side pressure drop.

Assume shell side pressure drop $\Delta p_s = 13.8$ kPa = 2 psi

$$p'_t = 150.325 - 13.8 = 136.525 \text{ kPa a} \equiv 1024 \text{ Torr a}$$

$$y_o = \frac{32.8336}{1024} = 0.032\,06$$

$$\dot{m}_{vo} = 0.032\,06 \times \frac{(0.1646/29)}{(1 - 0.032\,06)} \times 74 = 0.0139 \text{ kg/s}$$

$$\dot{m}_v = \left(\frac{0.2183 + 0.0139}{2}\right) = 0.1161 \text{ kg/s} \quad \text{(average)}$$

$\dot{m}_{Bc}$ = Condensation rate of butanol = $\dot{m}_{vi} - \dot{m}_{vo}$

$= 0.2183 - 0.0139 = 0.2044$ kg/s

$$\phi_t = (0.1646 + 0.1161) \times 1.3043\,(100 - 50) + 0.2044 \times 642$$

$$= 149.53 \text{ kW}$$

Heat balance:

$$\phi_t = \dot{m}_w C_{pw}\, \Delta t'$$

$$149.53 = \dot{m}_w \times 4.1868 \times (40 - 32)$$
$$\dot{m}_w = 4.4643 \text{ kg/s} = 16.07 \text{ m}^3\text{/h}$$

Fig. 6.19

MTD: $\Delta\mathcal{T}_m = F_t \times \Delta\mathcal{T}_{lm}$ (6.11)

$\Delta\mathcal{T}_{\ln}$

$$\Delta\mathcal{T}_{lm} = \frac{\Delta\mathcal{T}_1 - \Delta\mathcal{T}_2}{\ln\left(\dfrac{\Delta\mathcal{T}_1}{\Delta\mathcal{T}_2}\right)}, \quad \Delta\mathcal{T}_1 = 100 - 40 = 60°\text{C}, \quad \Delta\mathcal{T}_2 = 50 - 32 = 18°\text{C}$$

$$\Delta\mathcal{T}_{lm} = \frac{60 - 18}{\ln\left(\dfrac{60}{18}\right)} = 34.88°\text{C}$$

F_t = LMTD correction factor

$$R = \frac{t_1 - t_2}{t_2' - t_1'} \quad \text{and} \quad S = \frac{t_2' - t_1'}{t_1 - t_1'}$$

$$R = \frac{100 - 50}{40 - 32} = \frac{50}{8} = 6.25, \quad S = \frac{40 - 32}{100 - 32} = \frac{8}{68} = 0.1176$$

From Fig. 6.11, for 1–4 shell and side tube heat exchanger,

$$F_t = 0.96$$
$$\Delta\mathcal{T}_m = 0.96 \times 34.88°\text{C} = 33.485°\text{C}$$

Tube side heat transfer coefficient, h_i:

Tube side flow area, $a_t = \dfrac{N_t}{N_p} \times \dfrac{\pi}{4} d_i^2$ (6.20)

$$= \frac{134}{4} \times \frac{\pi}{4} (0.015\,748)^2 = 6.525 \times 10^{-3} \text{ m}^2$$

Tube side mass velocity, $G_t = \dfrac{\dot{m}_w}{a_t} = \dfrac{4.4643}{6.525 \times 10^{-3}}$

$$= 648.2 \text{ kg/(m}^2 \cdot \text{s)}$$

Tube side linear velocity $u_t = \dfrac{G_t}{\rho} = \dfrac{684.2}{1000} = 0.6842$ m/s (low)

$$Re_t = \frac{d_i G_t}{\mu} = \frac{0.015\,748 \times 684.2}{0.72 \times 10^{-3}}$$

$$= 14\,965$$

$$Pr = \frac{C_p \mu}{k} = \frac{4.1868 \times (0.72 \times 10^{-3}) \times 10^3}{0.63}$$

$$Pr = 4.7849$$

$$\frac{h_i d_i}{k} = 0.023\, Re_t^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.19}$$

$$h_i = 0.023 \times \frac{0.63}{0.015\,748} \times (14\,965)^{0.8} \times (4.7849)^{0.33} \times 1$$

$$h_i = 3374.9 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Shell side heat transfer coefficient, h_o:

$$\frac{1}{h_o} = \frac{1}{h_{cg}} = \frac{1}{h_c} + \frac{\phi_g}{\phi_t}\frac{1}{h_g} \tag{6.48}$$

Mean condensate film coefficient, h_c:

For horizontal position, modified Nusselt's equation is applicable.

$$h_c = 0.95\, k_L \left(\frac{\rho_L(\rho_L - \rho_V)g}{\mu_L \tau_h}\right)^{1/3} \times N_r^{-1/6} \tag{6.41}$$

$$\tau_h = \frac{\dot{m}_{BC}}{LN_t} = \frac{0.2044}{1.5 \times 134} = 1.017 \times 10^{-3} \text{ kg/(m} \cdot \text{s)} \tag{6.23}$$

$$N_r = \frac{2}{3} N_r', \quad N_r' = \frac{D_s}{P_t} = \frac{387}{25.4} \cong 15 \tag{6.42}$$

$$N_r = 10$$

Density of vapour, $\rho_v = \dfrac{pM}{RT} = \left(\dfrac{\left(\dfrac{p_t + p_t'}{2}\right)M}{RT}\right)$

$$= \frac{\left(\dfrac{150.325 + 136.525}{2}\right) \times 74}{(273 + 75)} \times \frac{273}{101.325 \times 22.414}$$

$$\rho_v = 3.666 \text{ kg/m}^3$$

$$h_c = 0.95 \times 0.168 \left(\frac{810(810 - 3.666) \times 9.81}{0.95 \times 10^{-3} \times 1.017 \times 10^{-3}}\right)^{1/3} \times (10)^{-1/6}$$

$$h_c = 2042.9 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Average gas film coefficient, h_g:

Shell side flow area, $A_s = \dfrac{(P_t - d_o)B_s D_s}{P_t}$ (6.33)

$$= \left(\frac{25.4 - 19.05}{25.4}\right) \times 0.12 \times 0.387 = 0.011\ 61\ \text{m}^2$$

Equivalent diameter for triangular pitch arrangement

$$d_e = \frac{1.1}{d_o}(P_t^2 - 0.907 d_o^2) = \frac{1.1}{0.019\ 05}(0.0254^2 - 0.907 \times 0.019\ 05^2)$$

$$= 0.018\ 247\ \text{m}$$

Shell side mass velocity G_s:

$$G_S = \frac{\dot{m}_s}{A_s} = \frac{(0.1646 + 0.1161)}{0.011\ 61} = 24.18\ \text{kg/(m}^2 \cdot \text{s)}$$

Reynolds number $Re_s = \dfrac{d_e G_s}{\mu} = \dfrac{0.018\ 247 \times 24.18}{0.0174 \times 10^{-3}}$

$$= 25\ 357$$

$$Pr = \frac{c_p \mu}{k} = \frac{1.3043 \times 0.0174 \times 10^{-3} \times 10^3}{0.03}$$

$$= 0.7565$$

Gas film coefficient can be determined by following correlation:

$$h_g \frac{d_e}{k} = 0.36\, Re_s^{0.55}\, Pr^{1/3} \left(\frac{\mu}{\mu_w}\right)^{0.14} \quad (6.39)$$

$$h_g = 0.36 \times \frac{0.03}{0.018\ 247} \times (25\ 357)^{0.55} \times (0.7565)^{1/3} \times 1 \quad \left(\frac{\mu}{\mu_w} \cong 1\right)$$

$$= 142.59\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

$$\frac{1}{h_o} = \frac{1}{h_c} + \frac{\phi_g}{\phi_t}\frac{1}{h_g} = \frac{1}{2042.9} + \frac{\phi_g}{\phi_t} \times \frac{1}{142.59} \quad (6.48)$$

where, $\phi_g = (\dot{m}_a + \dot{m}_v) C_{p\ \text{av}}\, \Delta t$

$$= (0.1646 + 0.1161) \times 1.3043\ (100 - 50)$$

$$= 18.306\ \text{kW}$$

$$\phi_t = 149.53\ \text{kW} \equiv 149.53 \times 10^3\ \text{W}$$

$$\frac{1}{h_o} = \frac{1}{2042.9} + \frac{18.306}{149.53} \times \frac{1}{142.59}$$

$$h_o = 741.8\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

Overall heat transfer coefficient U_o by Eq. (6.46).

Thermal conductivity of tube material (SS–304), $k_m = 16\ \text{W/(m} \cdot {}^\circ\text{C)}$

$$\frac{1}{U_o} = \frac{1}{741.8} + \frac{1}{5000} + \frac{0.019\,05 \times \ln\left(\frac{19.05}{15.748}\right)}{2 \times 16} + \frac{19.05}{15.748} \times \frac{1}{3374.9} + \frac{19.05}{15.748} \times \frac{1}{5000}$$

$$U_o = 442.13 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area required, A_{or}:

$$A_{or} = \frac{\phi_t}{U_o \Delta T_m} = \frac{149.53 \times 10^3}{442.13 \times 33.485}$$

$$= 10.1 \text{ m}^2$$

Heat transfer area provided,

$$A_{o\text{pro}} = N_t \pi d_o L = 134 \times \pi \times 0.019\,05 \times 1.5 = 12.0293 \text{ m}^2$$

$$\text{Excess heat transfer area} = \left(\frac{A_{o\text{pro}}}{A_{or}} - 1\right) \times 100 = \left(\frac{12.0293}{10.1} - 1\right) \times 100 = 19.1\%$$

Hence, heat transfer area provided by heat exchanger is sufficient for the given duty.

Shell side pressure drop Δp_s:

$$\Delta p_S = 8 J_f \left(\frac{D_s}{d_e}\right)\left(\frac{L}{B_s}\right)\frac{\rho_s u_s^2}{2}\left(\frac{\mu}{\mu_w}\right)^{-0.14} \tag{6.44}$$

$Re_s = 253\,57$, $J_f = 4.2 \times 10^{-2}$ (From Fig. 6.15)

$L = 1500$ mm, $B_s = 120$ mm, $D_s = 387$ mm, $d_e = 18.247$ mm

ρ_s = Density of shell side fluid (air–vapour mixture)

Average mole fraction of butanol vapour, $y_{\text{av}} = \dfrac{y_i + y_o}{2}$

$$y_{\text{av}} = \frac{0.342 + 0.032\,06}{2} = 0.187\,03$$

$M_{\text{av}} = y_{\text{av}} \times$ molar mass of butanol + $(1 - y_{\text{av}}) \times$ molar mass of air

$M_{\text{av}} = 0.187\,03 \times 74 + (1 - 0.187\,03) \times 29 = 37.4163$ kg/kmol

$$\rho_s = \frac{pM_{\text{av}}}{RT} = \frac{\left(\frac{150.325 + 136.525}{2}\right) \times 37.4163}{(273 + 75)} \times \frac{273}{101.325 \times 22.414}$$

$\rho_s = 1.8537$ kg/m^3

u_s = shell side linear velocity

$$= \frac{G_s}{\rho_s} = \frac{24.18}{1.8537} = 13.04 \text{ m/s} \quad \text{(acceptable)}$$

$$\Delta p_s = 8 \times 4.2 \times 10^{-}\left(\frac{387}{18.247}\right)\left(\frac{1500}{120}\right) \times \frac{1.8537 \times 13.04^2}{2} \times 1$$

$$= 14\,039 \text{ Pa} \equiv 14.04 \text{ kPa} < 49 \text{ kPa (Available pressure drop)}$$

Calculated shell side pressure drop ($\Delta p_s = 14.04$ kPa) is also close to optimum pressure drop ($\Delta p_{opt} = 13.8$ kPa). Hence, it is satisfactory.

Tube side pressure drop, Δp_t:

$$\Delta p_t = N_p\left(8J_f\left(\frac{L}{d_i}\right)\left(\frac{\mu}{\mu_w}\right)^{-m} + 2.5\right)\frac{\rho u_t^2}{2} \tag{6.31}$$

$N_p = 4$, $L = 1500$ mm, $d_i = 15.748$ mm, $\rho = 1000$ kg/m^3, $u_t = 0.6842$ m/s

for $Re = 14\,965$ from Fig. 6.13 $J_f = 4.3 \times 10^{-3}$

$$\Delta p_t = \left(8 \times 4.3 \times 10^{-3}\left(\frac{1500}{15.748}\right) \times 1 + 2.5\right) \times \frac{1000 \times 0.6842^2}{2}$$

$$\Delta p_t = 5408.4 \text{ Pa} \equiv 5.408 \text{ kPa} << \Delta p_{t\,\max} \text{ (68 kPa)}$$

Hence, tube side pressure drop is very much less than maximum or optimum pressure drop.

Note: Considering % excess heat transfer area, Δp_s and Δp_t, given shell and tube heat exchanger is suitable for the required heat transfer duty. If the given shell and tube heat exchanger is at design stage (not fabricated), then number of tube side passes could be increased from 4 to 8 because of the following reasons:

(i) Cooling water has a tendency to foul. To avoid the excessive fouling it is better to keep the velocity of cooling water more than 1 m/s, preferably near 2 m/s.

(ii) Increase in tube side passes will increase the tube side heat transfer coefficient and overall coefficient. Consequently, it will decrease the required heat transfer area and hence the fixed cost. However, it will increase the operating cost for pumping the tube side fluid. Also in the given case, overall heat transfer coefficient is controlled by shell side heat transfer coefficient and hence, increase in number of tube side passes will have little effect on the value of overall heat transfer coefficient.

6.10 MULTICOMPONENT CONDENSATION

In condensation of pure component (Example: condensation of pure ethanol vapour), condensation is taking place in isothermal manner or at constant temperature (i.e., degree of freedom = 1). While in case of multicomponent condensation, during the condensation, temperature decreases from dew point to bubble point temperature, except in the following cases:

1. Condensation of the vapour mixture of two immiscible components (immiscible in liquid phases). Example: Vapour mixture of toluene and water vapour.
2. Condensation of the azeotropic vapour mixture.
   Example, condensation of vapour mixture containing 89.4% (by mole) ethanol vapour and 10.6% (by mole) steam at 1 atm pressure.

In condensation of vapour mixture of miscible components (miscible in liquid phase), condensation starts from dew point temperature and terminates at bubble

point temperature. For the calculations of dew point, bubble point and compositions of vapour phase and liquid phase in between dew point and bubble point, vapour–liquid equilibrium data at operating pressure for the given system is required.

For finding the vapour-liquid equilibrium data from dew point to bubble point at operating pressure, there are three options.

(i) Find the VLE data by conducting the actual experiment. Particularly if the operating pressure is atmospheric or vacuum, then determination of VLE data by actual experiment is easier.

(ii) Find the VLE data from literature.

(iii) Find the VLE data by theoretical equation.

$$y_i = K_i x_i \tag{6.49}$$

where, y_i = Mole fraction of component i in vapour phase
x_i = Mole fraction of component i in liquid phase
K_i = Equilibrium constant for component i

K_i is a function of operating pressure, temperature as well as of composition.
For an ideal solution (i.e., the solution following Raoult's law),

$$K_i = \frac{p_{vi}}{p_t} = f(T, p, x_i, y_i) \tag{6.50}$$

where, p_{vi} = Vapour pressure of pure component i at the operating temperature
p_t = Operating pressure

For non-ideal vapour–liquid equilibrium, provide

$$K_i = \frac{p_{vi}\gamma_i}{p_t\varphi_i} = f(T, p, x_i, y_i) \tag{6.51}$$

where, γ_i = Activity coefficient of component i in liquid phase
φ_i = Fugacity coefficient of component i in vapour phase

If the solution can be considered as ideal solution then $\gamma_i = 1$. (Example: for the liquid mixture of benzene and toluene $\gamma_i = 1$). If vapour mixture can be considered as ideal gas mixture then $\varphi_i = 1$ (Example: vapour mixture of benzene and hexane at 1 atm pressure).

In the design of multi-component condenser calculations, weighted temperature difference is important instead of mean temperature difference.

To calculate weighted temperature difference, entire condensing range is divided in different small intervals. The weighted temperature difference is calculated by the following equation:

$$\text{Weighted } \Delta\mathcal{T} = \frac{\Sigma\phi_i}{\Sigma\dfrac{\phi_i}{\Delta\mathcal{T}_{avi}}} \tag{6.52}$$

where, ϕ_i = Heat load for an interval, kW
$\Delta\mathcal{T}_{avi}$ = Average temperature difference for the interval, °C

In condensing multicomponent mixture from dew point to bubble point, vapour and condensate are also cooled by sensible heat transfer. Vapour is cooled simultaneously during condensation but cooling of condensate should be facilitated by providing the

inverted U-seal or by using a dam baffle (Fig. 6.2(f)). Cooling of condensate formed at or near the dew point, heat the condensate formed at or near the bubble point in condensate collecting vessel and regenerate the vapour. Hence, it is necessary to cool the condensate simultaneously with vapour mixture from the dew point to bubble point in the condenser to avoid the regeneration of vapour from condensate and for the cooling of condensate certain number of tubes must be submerged in the pool of condensate which is assured by using inverted U-seal or dam baffle.

Design of multicomponent condenser is illustrated by the following example.

Example 6.5 Overhead vapour flows at the rate of 500 kmol/h from a distillation column at 4 atm a. It contains saturated hydrocarbons and has the composition; 30% n–C_3H_8, 25% n–C_4H_{10}, 20% n–C_5H_{12} and 25% n–C_6H_{14} (mole basis). It is to be condensed in a condenser. Permissible pressure drop in the condenser is 13.8 kPa.

(a) Determine the condensing range.
(b) Compute the condensing curve.
(c) Compute the weighted average temperature difference, and
(d) Design and specify a suitable condenser.

Table 6.10 Composition of Overhead Vapour

Component	mole % ($z_i \times 100$)	kmol/h $\dot{n}_i$	Molar mass	kg/h $\dot{m}_i$	mass %
n–C_3H_8	30	150	44	6 600	20.75
n–C_4H_{10}	25	125	58	7 250	22.80
n–C_5H_{12}	20	100	72	7 200	22.64
n–C_6H_{14}	25	125	86	10 750	33.81
Total	100	500		31 800	100.00

Solution

(a) Condensing range: Condensation starts from dew point and terminates at bubble point.

Dew point:

At dew point, $$\Sigma x_i = \sum \frac{y_i}{K_i} = 1 \text{ and } z_i = y_i \tag{6.49}$$

Value of equilibrium constants K_i for hydrocarbons can be obtained from Fig. 6.20. This calculation requires trial and error.

Assume, $t = 81°C$ and $p = 4$ atm a,

$K_{C_3H_8} = 5.7$, $K_{C_4H_{10}} = 2.3$, $K_{C_5H_{12}} = 0.89$, $K_{C_6H_{14}} = 0.4$

$$\sum \frac{y_i}{K_i} = \frac{0.3}{5.7} + \frac{0.25}{2.3} + \frac{0.2}{0.89} + \frac{0.25}{0.4} = 1.01 \cong 1$$

Dew point temperature of vapour mixture = 81°C at 4 atm a pressure

At bubble point, $\Sigma y_i = \Sigma K_i x_i = 1$, where $x_i = z_i$ (6.49)

Fig. 6.20 *K_i Values for Hydrocarbons at High Temperatures*[9]
(Reproduced with the Permission of the American Institute of Chemical Engineers, USA.)

Assume $t = 31°C$

$$K_{C_3H_8} = 2.5, \quad K_{C_4H_{10}} = 0.74, \quad K_{C_5H_{12}} = 0.24, \quad K_{C_6H_{14}} = 0.085$$

$$\Sigma K_i x_i = (0.3 \times 2.5 + 0.25 \times 0.74 + 0.20 \times 0.24 + 0.25 \times 0.085) = 1.00$$

Bubble point of vapour mixture = 31°C at 4 atm a

Therefore, the condensation range is from 81°C to 31°C.

(b) Condensing curve:

Divide the condensation range in five intervals; say 81°C to 70°C, 70°C to 60°C, 60°C to 50°C, 50°C to 40°C and 40°C to 31°C.

Equilibrium constant K_i values (From Fig. 6.20):

Table 6.11 Equilibrium Constants K_i Values (from Fig. 6.20)

Component	K_i					
	81°C	70°C	60°C	50°C	40°C	31°C
C_3H_8	5.7	4.9	4.2	3.6	3.0	2.5
C_4H_{10}	2.3	1.75	1.5	1.2	0.95	0.74
C_5H_{14}	0.89	0.7	0.56	0.42	0.32	0.24
C_6H_{14}	0.4	0.3	0.23	0.17	0.12	0.085

Let F = Total molar flow rate of saturated vapour mixture entering to condenser = 500 kmol/h

First interval:

Let, L_1 = Molar flow rate of condensate at the exist of 1st interval (at 70°C)

V_1 = Molar flow rate of vapour at the exist of 1st interval

$F = L_1 + V_1$

Let C_1 = Molar flow rate of condensate formed in the interval

for first interval $C_1 = L_1$

Component balance $F z_i = L_1 x_i + V_1 y_i$ (6.53)

$$L_1 x_i = \frac{F z_i}{1 + \frac{V_1}{L_1} K_{i\,(t\,=\,70°C)}} \tag{6.54}$$

Assume the value of V_1/L_1 and find the value of $L_1 x_i$ by Eq. (6.54). Then $\Sigma L_1 x_i = L_1$, $F - L_1 = V_1$, V_1/L_1 = ? Value of V_1/L_1 obtained at the end of calculations should be same as the assumed one. So it requires trial and error calculations.

Table 6.12 Flash Calculations for Range 81°C to 70°C

Component	Fz_i	$K_{i\,(t\,=\,70°C)}$	$L_1 x_i$	$C_1 x_i$	λ_i, kJ/kg	C_{mpi} kJ/(kmol · K)	C_{Li} kJ/(kg · K)
C_3H_8	150	4.90	10.917	10.917	230.27	84.253	3.12
C_4H_{10}	125	1.75	22.522	22.522	301.450	111.08	2.70
C_5H_{12}	100	0.70	35.460	35.460	303.540	137.08	2.51
C_6H_{14}	125	0.30	70.225	70.225	309.820	163.17	2.43
Total	500		139.124	139.124			

Assume $\dfrac{V_1}{L_1} = 2.6$

$$\frac{150}{1+2.6\times 4.9}+\frac{125}{1+2.6\times 1.75}+\frac{100}{1+2.6\times 0.7}+\frac{125}{1+2.6\times 0.3} = 139.124$$

$$L_1 = 139.124 \text{ kmol/h}$$
$$V_1 = F - L_1 = 500 - 139.124 = 360.876 \text{ kmol/h}$$
$$\frac{V_1}{L_1} = \frac{360.876}{139.124} = 2.5939 \approx 2.6$$

Values of λ_i at average temperature (75.5°C) are obtained from Fig. 3.9 of Ref. 2. Specific heat of vapour are determined by using following equation:

C_3H_8 : $C_{mpi} = -5.338 + 310.24\times 10^{-3}T - 164.64\times 10^{-6}T^2 + 34.691\times 10^{-9}T^3$

C_4H_{10} : $C_{mpi} = -1.779 + 386.96\times 10^{-3}T - 193.25\times 10^{-6}T^2 + 34.833\times 10^{-9}T^3$

C_5H_{12} : $C_{mpi} = -3.411 + 485.01\times 10^{-3}T - 251.94\times 10^{-6}T^2 + 48.677\times 10^{-9}T^3$

C_6H_{14} : $C_{mpi} = -4.738 + 582.41\times 10^{-3}T - 310.64\times 10^{-6}T^2 + 62.923\times 10^{-9}T^3$

where C_{mpi} are in kJ/(kmol · K) and T is in K (Table 5.1 of Ref. 10)

Specific heat of liquids are obtained from Fig. 4 of Ref. 5 at average temperature (75.5°C).

Values of C_{mp} and C_L are at 1 bar a in ideal state. However, these are not significantly affected under pressure up to 10 bar.

For the interval 81°C to 70°C,

$$\phi_1 = \Sigma C_1 x_i \lambda_i + \left(\frac{Fz_i + V_1 y_i}{2}\right) C_{mpi}\,\Delta t + \left(\frac{0 + L_1 x_i}{2}\right) C_{Li}\,\Delta t$$

$$\begin{aligned}\phi_1 = {} & (10.917\times 230.27\times 44 + 22.522\times 301.45\times 58 + 35.46\times 303.54\times 72 \\ & + 70.225\times 309.82\times 86) + \left(\frac{150+(150-10.917)}{2}\right)\times 84.253\times 11 \\ & + \left(\frac{125+(125-22.522)}{2}\right)\times 111.08\times 11 \\ & + \left(\frac{100+(100-35.46)}{2}\right)\times 137.08\times 11 \\ & + \left(\frac{125+(125-70.225)}{2}\right)\times 163.17\times 11 + \frac{10.917}{2}(3.12\times 44)\times 11 \\ & + \frac{22.522}{2}\times(2.7\times 58)\times 11 + \frac{35.46}{2}(2.51\times 72)\times 11 \\ & + \frac{70.225}{2}\times(2.43\times 86)\times 11\end{aligned}$$

$= 3150\,472.1 + 558\,323.68 + 143\,602.7$

$= 3852\,398.5 \text{ kJ/h} \equiv 1070.1 \text{ kW}$

Second interval:

Table 6.13 Flash Calculations for Range 70°C to 60°C

Component	$V_1y_i\,(= Fz_i - L_1x_i)$	$K_{i(t=60°C)}$	L_2x_i	$V_2y_i\,(= Fz_i - L_2x_i)$	$C_2x_i'^{*}$
C_3H_8	139.083	4.2	24.834	125.166	13.917
C_4H_{10}	102.478	1.5	44.643	80.357	22.121
C_5H_{12}	64.540	0.56	59.810	40.190	24.350
C_6H_{14}	54.775	0.23	97.962	27.038	27.737
Total	360.876		227.25	272.75	88.125

$^{*}x_i'$ represents mole fraction of liquid component, condensed in the specified interval.

For the second interval let L_2 is total moles of condensate leaving at 60°C and C_2 is moles of condensate formed in second interval.

$$L_2 = \Sigma L_2x_i = \sum\left(\frac{Fz_i}{1+\dfrac{V_2}{L_2}K_{i(t=60°C)}}\right)$$

where, V_2 = Total moles of vapour at 60°C, leaving the second interval

$$L_2 = \Sigma L_2x_i = \frac{150}{1+\dfrac{V_2}{L_2}\times 4.2} + \frac{125}{1+\dfrac{V_2}{L_2}\times 1.5} + \frac{100}{1+\dfrac{V_2}{L_2}\times 0.56} + \frac{125}{1+\dfrac{V_2}{L_2}\times 0.23}$$

Assume $\dfrac{V_2}{L_2} = 1.2$

$L_2 = \Sigma L_2x_i = 24.834 + 44.643 + 59.81 + 97.962 = 227.25$ kmol/h

$F = V_2 + L_2$

$V_2 = F - L_2 = 500 - 227.25 = 272.75$ kmol/h

$\dfrac{V_2}{L_2} = \dfrac{272.75}{227.25} = 1.2$ (Check)

$V_2y_i = Fz_i - L_2x_i, \qquad C_2x_i' = V_1y_i - V_2y_i$

Table 6.14 Calculated Properties at 65°C

Component	λ_i, kJ/mol	C_{mpi}, kJ/(kmol · K)	C_{Li}, kJ/(kmol · K)
C_3H_8	11 605.81	82.053	132.64
C_4H_{10}	18 212.58	108.28	150.56
C_5H_{12}	22 608.72	133.62	177.86
C_6H_{14}	28 445.12	159.06	205.24

For the interval 70 to 60°C,

$$\phi_2 = \Sigma C_2 x_i' \lambda_i + \sum\left(\frac{V_1 y_i + V_2 y_i}{2}\right) C_{mpi}\,\Delta t + \sum\left(\frac{L_1 x_i + L_2 x_i}{2}\right) C_{Li}\,\Delta t$$

$$\begin{aligned}\phi_2 = {} & (13.917 \times 11\,605.81 + 22.121 \times 18\,212.58 + 24.35 \times 22\,608.72 \\ & + 27.737 \times 28\,445.12) \\ & + \left(\frac{139.083 + 125.166}{2}\right) \times 82.053 \times 10 + \left(\frac{102.478 + 80.357}{2}\right) \times 108.28 \times 10 \\ & + \left(\frac{64.54 + 40.19}{2}\right) \times 133.62 \times 10 + \left(\frac{54.775 + 27.038}{2}\right) \times 159.06 \times 10 \\ & + \left(\frac{10.917 + 24.834}{2}\right) \times 132.64 \times 10 + \left(\frac{22.522 + 44.643}{2}\right) \times 150.56 \times 10 \\ & + \left(\frac{35.46 + 59.81}{2}\right) \times 177.86 \times 10 + \left(\frac{70.225 + 97.962}{2}\right) \times 205.24 \times 10 \\ = {} & 1903\,903.2 + 342\,435 + 331\,589 \\ = {} & 2577\,927.2 \text{ kJ/h} \equiv 716.1 \text{ kW}\end{aligned}$$

Table 6.15 Flash Calculations for Range 60°C to 50°C

Component	$V_2 y_i$	$K_{i\,(t = 50°C)}$	$L_2 x_i$	$L_3 x_i$	$V_3 y_i$	$C_3 x_i'$
C_3H_8	125.166	3.60	24.834	48.5751	101.425	23.741
C_4H_{10}	80.357	1.20	44.643	73.7028	51.297	29.060
C_5H_{12}	40.190	0.42	59.810	80.4117	19.588	20.600
C_6H_{14}	27.038	0.17	97.962	113.7812	11.220	15.820
Total	272.750		227.250	316.4708	183.530	89.220

Value of L_3x_i, V_3y_i and C_3x_i' of above table are obtained via following calculation: L_3 is total mole of condensate at 50°C.

$$L_3 = \Sigma L_3 x_i = \sum\left(\frac{F z_i}{1 + \frac{V_3}{L_3} K_{i(t = 50°C)}}\right)$$

$$L_3 = \Sigma L_3 x_i = \frac{150}{1 + \frac{V_3}{L_3} \times 3.6} + \frac{125}{1 + \frac{V_3}{L_3} \times 1.2} + \frac{100}{1 + \frac{V_3}{L_3} \times 0.42} + \frac{125}{1 + \frac{V_3}{L_3} \times 0.17}$$

Trial: Assume $\frac{V_3}{L_3} = 0.58$

$$L_3 = \Sigma L_3 x_i = \frac{150}{1 + 0.58 \times 3.6} + \frac{125}{1 + 0.58 \times 1.2} + \frac{100}{1 + 0.58 \times 0.42} + \frac{125}{1 + 0.58 \times 0.17}$$

$$L_3 = \Sigma L_3 x_i = 48.5751 + 73.7028 + 80.4117 + 113.7812 = 316.4708 \text{ kmol/h}$$

$$V_3 = 500 - 316.4708 = 183.5292 \text{ kmol/h}$$

$$\frac{V_3}{L_3} = 0.5799 \cong 0.58 \quad \text{(Check)}$$

$$C_3 x_i' = (L_3 x_i)_3 - (L_2 x_i)_2 = (V_2 y_i)_2 - (V_3 y_i)_3$$

Table 6.16 Calculated Properties at 55°C

Component	λ_i, kJ/kmol	C_{mpi}, kJ/(kmol·K)	C_{Li}, kJ/(kmol·K)
C_3H_8	12 895.3	79.932	128.95
C_4H_{10}	19 183.9	105.58	148.13
C_5H_{12}	23 513.1	150.29	173.33
C_6H_{14}	28 805.2	155.09	201.64

$$\phi_3 = \Sigma C_3 x_i' \lambda_i + \sum\left(\frac{V_2 y_i + V_3 y_i}{2}\right) C_{mpi}\Delta t + \sum\left(\frac{L_2 x_i + L_3 x_i}{2}\right) C_{Li}\Delta t$$

$$\begin{aligned}\phi_3 &= (23.741 \times 12\,895.3 + 29.06 \times 19\,183.9 + 20.6 \times 23\,513.1 + 15.82 \times 28\,805.2) \\ &\quad + \left(\frac{125.166 + 101.425}{2}\right) \times 79.932 \times 10 + \left(\frac{80.357 + 51.297}{2}\right) \times 105.58 \times 10 \\ &\quad + \left(\frac{40.19 + 19.588}{2}\right) \times 150.29 \times 10 + \left(\frac{27.038 + 11.22}{2}\right) \times 155.09 \times 10 \\ &\quad + \left(\frac{24.834 + 48.575}{2}\right) \times 128.95 \times 10 + \left(\frac{44.643 + 73.7028}{2}\right) \times 148.13 \times 10 \\ &\quad + \left(\frac{59.81 + 80.4117}{2}\right) \times 173.33 \times 10 + \left(\frac{97.962 + 113.7812}{2}\right) \times 201.64 \times 10 \\ &= 1803\,699.6 + 234\,646.85 + 469\,986 = 2508\,332.5 \text{ kJ/h} \equiv 696.76 \text{ kW}\end{aligned}$$

Fourth interval:

Table 6.17 Flash Calculation for Range 50°C to 40°C

Component	$V_3 y_i$	$K_{i(t = 40°C)}$	$L_3 x_i$	$L_4 x_i$	$V_4 y_i$	$C_4 x_i'$
C_3H_8	101.425	3.00	48.5751	85.714	64.286	37.14
C_4H_{10}	51.297	0.95	73.7028	101.01	23.990	27.31
C_5H_{12}	19.588	0.32	80.4117	92.593	7.407	12.18
C_6H_{14}	11.220	0.12	113.7812	121.36	3.640	7.58
Total	183.53		316.4708	400.677	99.323	84.21

Value of $L_4 x_i$, $V_4 y_i$ and $C_4 x_i'$ of above table are obtained by the following calculations:

$$L_4 = \Sigma L_4 x_i = \sum\left(\frac{Fz_i}{1+\frac{V_4}{L_4}K_{i(t=40°C)}}\right)$$

$$\therefore \quad L_4 = \Sigma L_4 x_i = \frac{150}{1+\frac{V_4}{L_4}\times 3} + \frac{125}{1+\frac{V_4}{L_4}\times 0.95} + \frac{100}{1+\frac{V_4}{L_4}\times 0.32} + \frac{125}{1+\frac{V_4}{L_4}\times 0.12}$$

Trial: Assume $\frac{V_4}{L_4} = 0.25$ (Check)

$$L_4 = 85.714 + 101.01 + 92.593 + 121.36 = 400.677 \text{ kmol/h}$$

$$V_4 = 99.323, \frac{V_4}{L_4} = 0.248 \cong 0.25$$

$$V_4 y_i = Fz_i - L_4 x_i,\ C_4 x_i' = (L_4 x_i)_4 - (L_3 x_i)_3$$

$$\phi_4 = \Sigma C_4 x_i' \lambda_i + \Sigma\left(\frac{V_3 y_i + V_4 y_i}{2}\right) C_{mpi}\,\Delta t + \Sigma\left(\frac{L_3 x_i + L_4 x_i}{2}\right) C_{Li}\,\Delta t$$

Table 6.18 Calculated Properties at 45°C

Component	λ_i, kJ/kmol	C_{mpi}, kJ/(kmol·K)	C_{Li}, kJ/(kmol·K)
C_3H_8	13 448.0	77.785	125.27
C_4H_{10}	19 912.4	102.850	143.27
C_5H_{12}	24 116.0	126.910	168.81
C_6H_{14}	29 525.3	151.440	198.04

$$\phi_4 = \Sigma C_4 x_i' \lambda_i + \left(\frac{V_3 y_i + V_4 y_i}{2}\right) C_{mpi}\Delta t + \left(\frac{L_3 x_i + L_4 x_i}{2}\right) C_{Li}\Delta t$$

$$= (37.14 \times 13\,448 + 27.31 \times 19\,912.4 + 12.18 \times 24\,116 + 7.58 \times 29\,525.3)$$

$$+\left(\frac{101.425 + 64.286}{2}\right) \times 77.785 \times 10$$

$$+\left(\frac{51.297 + 23.99}{2}\right) \times 102.85 \times 10 + \left(\frac{19.588 + 7.407}{2}\right) \times 126.91 \times 10$$

$$+\left(\frac{11.22 + 3.64}{2}\right) \times 151.44 \times 10 + \left(\frac{48.5751 + 85.714}{2}\right) \times 125.27 \times 10$$

$$+\left(\frac{73.7028 + 101.01}{2}\right) \times 143.27 \times 10 + \left(\frac{80.4117 + 92.593}{2}\right) \times 168.81 \times 10$$

$$+\left(\frac{113.7812 + 121.36}{2}\right) \times 198.04 \times 10$$

ϕ_4 = (156 0801 + 131 547.2 + 588 128.9) = 2280 477.1 kJ/h ≡ 633.46 kW

Fifth (final) Interval:

Table 6.19 Flash Calculations for Range 40°C to 31°C

Component	V_4y_i	V_5y_i	L_4x_i	C_5x_i	$L_5x_i = Fz_i$
C_3H_8	64.286	0	85.714	64.286	150
C_4H_{10}	23.990	0	101.010	23.990	125
C_5H_{12}	7.407	0	92.593	7.407	100
C_6H_{14}	3.640	0	121.360	3.640	125
Total	99.323		400.677	99.323	500

Table 6.20 Calculated Properties at 35.5°C

Component	λ_i, kJ/kmol	C_{mpi}, kJ/(kmol·K)	C_{Li}, kJ/(kmol·K)
C_3H_8	14 553.3	75.72	123.43
C_4H_{10}	20 155.3	100.23	140.84
C_5H_{12}	24 718.9	123.67	165.8
C_6H_{14}	30 245.4	147.22	194.4

$$\phi_5 = \Sigma C_5 x_i' \lambda_i + \left(\frac{V_4 y_i + 0}{2}\right) C_{mpi}\Delta t + \left(\frac{L_4 x_i + L_5 x_i}{2}\right) C_{Li}\Delta t$$

$$\phi_5 = (64.286 \times 14\,553.3 + 23.99 \times 20\,155.3 + 7.407 \times 24\,718.9 + 3.64 \times 30\,245.4)$$

$$+\left(\frac{64.286}{2} \times 75.72 \times 11 + \frac{23.99}{2} \times 100.23 \times 11 + \frac{7.407}{2} \times 123.67 \times 11 + \frac{3.64}{2} \times 147.22 \times 11\right)$$

$$+\left(\frac{85.714 + 150}{2}\right) \times 123.43 \times 11 + \left(\frac{101.01 + 125}{2}\right) \times 140.84 \times 11$$

$$+\left(\frac{92.593 + 100}{2}\right) \times 165.8 \times 11 + \left(\frac{121.36 + 125}{2}\right) \times 194.4 \times 11$$

$$= 17\,12285.2 + 47\,806.1 + 77\,4123.5 = 25\,34214.8 \text{ kJ/h} \equiv 703.95 \text{ kW}$$

Total heat duty $\phi_t = \phi_1 + \phi_2 + \phi_3 + \phi_4 + \phi_5$

$= 1070.1 + 716.1 + 696.76 + 633.46 + 703.95 = 3820.37$ kW

Assume chilled water as a cooling medium.

Let inlet temperature of chilled water, $t_i' = 6°C$

Outlet temperature of chilled water, $t_o' = 9°C$

Energy balance:

$$\phi_t = \sum_{1}^{5} \phi_i = 3820.37 \text{ kW} = \dot{m}_w \times 4.1868 \times (9 - 6)$$

Mass flow rate of chilled water required

$$\dot{m}_w = \frac{3820.37}{4.1868 \times 3} = 304.16 \text{ kg/s}$$

$$\equiv 1094\ 976 \text{ kg/h} \equiv 1095 \text{ m}^3\text{/h}$$

$$\Delta\mathcal{T}_{avi} \text{ (LMTD) for 1st interval} = \frac{(81 - 9) - (70 - 8.159)}{\ln\left(\dfrac{81 - 9}{70 - 8.159}\right)} = 66.79°\text{C}$$

$$F_t = 1$$

Table 6.21 Summary of Heat Balance Calculations

Interval (Based on vapour temperature)	Temperatures of chilled water		Heat duty ϕ, kW	$\Delta\mathcal{T}_{avi}$, (LMTD)
81°C to 70°C	8.16°C	to 9°C	1070.1	66.79°C
70°C to 60°C	7.6°C	to 8.16°C	716.1	56.99°C
60°C to 50°C	7.05°C	to 7.6°C	696.76	47.52°C
50°C to 40°C	6.553°C	to 7.05°C	633.46	38°C
40°C to 31°C	6°C	to 6.553°C	703.95	29.02°C
Total			3820.37	

Figure 6.21 gives the results in diagrammatic form.

Fig. 6.21 *Condenser for Condensation of Hydrocarbon Vapours*

$$\text{Weighted } \Delta\mathcal{T} = \frac{\Sigma\phi_i}{\Sigma\left(\dfrac{\phi_i}{\Delta\mathcal{T}_{avi}}\right)} \tag{6.52}$$

$$\text{Weighted } \Delta\mathcal{T} = \frac{3820.37}{\left(\dfrac{1070.1}{66.79} + \dfrac{716.1}{56.99} + \dfrac{696.76}{47.52} + \dfrac{633.46}{38} + \dfrac{703.95}{29.02}\right)}$$

$$= 45.385°\text{C}$$

Heat duty for subcooling:

$$\phi_{\text{sub}} = 143\ 602.7 + 331\ 589 + 469\ 986 + 588\ 128.9 + 774\ 123.5$$

$$= 2307\ 430.1 \text{ kJ/h} \equiv 640.95 \text{ kW} \equiv 640.95 \times 10^3 \text{ W}$$

Heat duty for condensation:

$$\phi_c = \phi_t - \phi_{\text{sub}} = 3820.37 - 640.95 = 3179.42 \text{ kW} \equiv 3179.42 \times 10^3 \text{ W}$$

This value includes heat load required for cooling of vapour. Heat transfer coefficient for the cooling of vapour in presence of condensation is quite high. Also, cooling of vapour and condensation of vapour, both are taking place simultaneously. Hence, as per the Kern's suggestion, separate calculation for determining heat transfer coefficient for cooling of vapour is not required.

Assume values of overall coefficients for the first trial calculation.

Overall coefficient for condensation $U_{\text{oc}} = 800 \text{ W/(m}^2 \cdot °\text{C)}$

Overall coefficient for subcooling $U_{o\text{sub}} = 200 \text{ W/(m}^2 \cdot °\text{C)}$

Provisional heat transfer area:

$$\text{For condensation, } A_{\text{cpro}} = \frac{\phi_c}{U_{oc} \times \text{weighted } \Delta\mathcal{T}}$$

$$= \frac{3179.42 \times 10^3}{800 \times 45.385} = 87.568 \text{ m}^2$$

$$\text{For subcooling, } A_{\text{sub, pro}} = \frac{\phi_{\text{sub}}}{U_{\text{osub}} \times \text{weighted } \Delta\mathcal{T}} = \frac{640.95 \times 10^3}{200 \times 45.386} = 70.61 \text{ m}^2$$

Total heat transfer area $A_{\text{pro}} = 87.568 + 70.61 = 158.18 \text{ m}^2$

Let tube length $L = 8$ ft $= 2.4384$ m

Tube outside diameter $d_o = 3/4$ in $= 0.019\ 05$ m

$$\text{Number of tubes,} \quad N_t = \frac{A_{\text{pro}}}{\pi d_o L} = \frac{158.18}{\pi \times 2.4384 \times 0.019\ 05}$$

$$N_t = 1084$$

Let tube pitch $P_t = 1.25\ d_o = 0.023\ 81$ m, triangular pitch arrangement, number of tube side passes = 4 (for first trial calculations)

Tube bundle diameter

$$D_b = d_o\left(\frac{N_t}{k_1}\right)^{\frac{1}{n_1}} \tag{6.1}$$

for $N_p = 4$, $k_1 = 0.175$, $n_1 = 2.285$ (Table 6.2)

$$D_b = 19.05\left(\frac{1084}{0.175}\right)^{\frac{1}{2.285}} = 870 \text{ mm}$$

Let the clearance between shell inside diameter and tube bundle diameter = 16 mm.
Shell ID $D_s = 870 + 16 = 886$ mm
Tube side heat transfer coefficient h_i:
Allocate chilled water on tube side

Tube side flow area, $a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2$ (6.20)

Tube inside diameter, $d_i = 15.748$ mm (For 16 BWG tube)

$$a_t = \frac{1084}{4} \times \frac{\pi}{4}(0.015\,748)^2 = 0.052\,785 \text{ m}^2$$

Tube side mass velocity $G_t = \frac{\dot{m}}{a_t} = \frac{304.16}{0.052\,785} = 5762.24$ kg/(m^2 · s)

Density of water at 7.5°C, $\rho = 999.877$ kg/m^3 (From Table 3.28 of Ref. 2)

Tube side velocity, $u_t = \frac{G_t}{\rho} = \frac{5762.24}{999.877} = 5.76$ m/s (very high)

Velocity of water in tubes should preferably be in between 1 to 3 m/s.
Let number of tube side passes = 1
For $N_p = 1$, $k_1 = 0.319$, $n_1 = 2.142$ (From Table 6.2)

$$D_b = 19.0\left(\frac{1084}{0.319}\right)^{\frac{1}{2.142}} = 848 \text{ mm}$$

Shell ID, $D_s = 848 + 16 = 864$ mm (Revised)

Tube side flow area, $a_t = \frac{1084}{1} \times \frac{\pi}{4}(0.015748)^2 = 0.2111$ m^2

$$G_t = \frac{304.16}{0.2111} = 1440.83 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_t = \frac{G_t}{\rho} = \frac{1440.83}{999.877} = 1.44 \text{ m/s which is within acceptable range}$$

Tube side Reynold's number, $Re_t = \frac{d_i G_t}{\mu}$

Viscosity of water at 7.5°C, $\mu = 1.3 \times 10^{-3}$ kg/(m · s)

$$Re_t = \frac{0.015\,748 \times 1440.83}{1.3 \times 10^{-3}} = 17\,454$$

Prandtl number, $Pr = \dfrac{C_p \mu}{k}$

Thermal conductivity of water at 7.5°C, $k = 0.596\,85$ W/(m · °C)

$$Pr = \frac{4.1868 \times 1.3 \times 10^{-3} \times 10^{3}}{0.596\,85} = 9.12$$

Dittus–Bolter equation

$$\frac{h_i d_i}{k} = 0.023\, Re^{0.8}\, Pr^{0.3} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.19}$$

$$h_i = 0.023 \times \frac{0.596\,85}{0.0157\,48} \times (17\,454)^{0.8} \times (9.12)^{0.33} \times 1$$

$$h_i = 4473.85 \text{ W/(m}^2 \cdot \text{°C)}$$

h_o: Heat transfer coefficients for both condensation and subcooling must be determined separately.

For condensation $\bar{h}_{oc}$

For the first trial calculation let $\bar{h}_{oc} = 1500$ W/(m^2 · °C)

$$\bar{h}_{oc}\,(t_s - t_w) = U_{oc}(t_s - t_t)$$

where, $U_{oc} = 800$ W/(m^2 · °C)

t_w = Tube wall temperature, °C

t_s = shell side average temperature = $\dfrac{81 + 31}{2} = 56$°C

t_t = Tube side average temperature = $\dfrac{6 + 9}{2} = 7.5$°C

$$1500(56 - t_w) = 800(56 - 7.5)$$

$$t_w = 30.13\text{°C}$$

Mean temperature of condensate film, $t_f = \dfrac{t_s + t_w}{2} = \dfrac{56 + 30.13}{2} = 43$°C

Physical properties of liquid condensate should be determined for the average condensate composition. Here, properties are determined based on the composition of condensate in the intermediate (third) interval.

Table 6.22 Calculated Properties of Liquid Condensate in Third Interval

Component	$C_3 x_i'$	Mole fraction	Mass fraction	ρ_L, kg/m^3	μ^*, cP	k, W/(m · °C)
C_3H_8	23.741	0.2660	0.1874	582	0.10	0.1211
C_4H_{10}	29.060	0.3257	0.3024	579	0.15	0.1263
C_5H_{12}	20.600	0.2309	0.2661	626	0.20	0.1305
C_6H_{14}	15.820	0.1773	0.2441	659	0.27	0.1367
Total	89.221	1.0000	1.0000			

* For viscosity, Fig. 14 of Ref. 5

$$\rho_L = \sum \frac{1}{\left[w_i/\rho_{Li}\right]} \tag{6.55}$$

$$= \frac{1}{\left(\frac{0.1874}{582} + \frac{0.3024}{579} + \frac{0.2661}{626} + \frac{0.2441}{659}\right)} = 609.84 \text{ kg/m}^3$$

Viscosity of nonpolar liquid mixture by Irving's equation (Eq. (3.107) of Ref. 2)

$$\ln \mu_{L\text{mix}} = \Sigma w_i \ln \mu_i \tag{6.56}$$

$$= [0.1874 \ln (0.1) + 0.3024 \ln (0.15) + 0.2661 \ln (0.2) + 0.2441 \ln (0.27)]$$

$$\mu_{L\text{mix}} = 0.1732 \text{ cP} \equiv 0.1732 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$k_{L\text{mix}} = \Sigma k_i x_i = 0.266 \times 0.1211 + 0.3257 \times 0.1263 + 0.2309 \times 0.1305 + 0.1773 \times 0.1367$$

$$= 0.1277 \text{ W/(m} \cdot \text{K)}$$

Average molar mass of vapour mixture,

$$M_{\text{av}} = \Sigma M_i y_i = 0.3 \times 44 + 0.25 \times 58 + 0.2 \times 72 + 0.25 \times 86 = 63.6 \text{ kg/kmol}$$

Density of vapour mixture, $\rho_v = \dfrac{pM_{\text{av}}}{RT}$

$$= \frac{4 \times 63.6}{(273 + 56)} \times \frac{273}{1 \times 22.414} = 9.418 \text{ kg/m}^3$$

Position of condenser: Horizontal

Shell side condensation coefficient

$$\bar{h}_{oc} = 0.95\, k \left[\frac{\rho_L(\rho_L - \rho_v)g}{\mu_L \tau_h}\right]^{1/3} \cdot N_r^{-1/6} \tag{6.41}$$

$$\tau_h = \frac{\dot{m}_c}{LN_t},\ N_r = \frac{2}{3} N_r' = \frac{2}{3}\left(\frac{D_b}{p_t}\right) = \frac{2}{3} \times \left(\frac{848}{23.81}\right) \cong 24$$

$$\dot{m}_c = (23.741 \times 44 + 29.06 \times 58 + 20.6 \times 72 + 15.82 \times 86)/3600$$

$$= 1.548 \text{ kg/s}$$

$$\tau_h = \frac{1.548}{(1084 \times 2.4384)} = 5.856 \times 10^{-4} \text{ kg/(m} \cdot \text{s)}$$

$$\bar{h}_{oc} = 0.95 \times 0.127 \left[\frac{609.84(609.84 - 9.418) \times 9.81}{0.1732 \times 10^{-3} \times 5.856 \times 10^{-4}}\right]^{1/3} \times (24)^{-1/6}$$

$$= 0.95 \times 0.1277 \times 32\,839.57 \times 0.5888 = 2345.7 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

For the second trial calculation if it is assumed $\bar{h}_{oc} = 2345.7$ W/(m^2 · °C) (resulting value of first trial calculation) condensate film temperature, $t_f = 47.73$°C (revised). Hence, minor change in the value of film temperature is noted. Also, precise temperature wise physical properties of these components (particularly density and thermal conductivity) are not available. So, second trial calculation will not make any significant difference in the final value.

$$\bar{h}_{oc} = 2345.7 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

As stated earlier with horizontal position, subcooling of condensate is taking place by natural convection only. No reliable correlation is available for natural convection. Hence, based on Kern's recommendation, assume

$$h_{o\text{sub}} = 50 \text{ Btu/(h} \cdot \text{ft}^2 \cdot {}^\circ\text{F)} = 283.77 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Overall heat transfer coefficient for condensation, Eq. (6.46):
For light hydrocarbon mixture $h_{od} = 5000$ W/(m^2 · °C) and
For cooling water $h_{id} = 4000$ W/(m^2 · °C) (From Table 6.9)
Thermal conductivity of tube material

$$k_w = 50 \text{ W/(m} \cdot {}^\circ\text{C) (Steel or cupronickel tube)}$$

$$\frac{1}{U_{oc}} = \frac{1}{2345.7} + \frac{1}{5000} + \frac{0.019\,05 \ln\left(\frac{19.05}{15.748}\right)}{2 \times 50} + \frac{19.05}{15.748} \times \frac{1}{4473.85} + \frac{19.05}{15.748} \times \frac{1}{4000}$$

$$U_{oc} = 809.47 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area required for condensation

$$A_{\text{creq}} = \frac{\phi_c}{U_{oc} \times \text{weighted } \Delta\mathcal{T}} = \frac{3179.42 \times 10^3}{809.47 \times 45.385}$$

$$A_{\text{creq}} = 86.54 \text{ m}^2$$

Overall heat transfer coefficient for subcooling

$$\frac{1}{U_{o\text{sub}}} = \frac{1}{283.77} + \frac{1}{5000} + \frac{0.019\,05 \ln\left(\frac{19.05}{15.748}\right)}{2 \times 50} + \frac{19.05}{15.748} \times \frac{1}{4473.85} + \frac{19.05}{15.748} \times \frac{1}{4000}$$

$$U_{o\text{sub}} = 230.78 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$A_{\text{sub}} = \frac{\phi_{\text{sub}}}{U_{o\text{sub}} \times \text{weighted } \Delta\mathcal{T}} = \frac{640.95 \times 10^3}{230.78 \times 45.385}$$

$$A_{\text{subreq}} = 61.19 \text{ m}^2$$

Total heat transfer area required,

$$A_{\text{req}} = 86.54 + 61.19 = 147.73 \text{ m}^2$$

Total heat transfer area provided,

$$A_{\text{pro}} = 158.16 \text{ m}^2$$

$$\text{Excess heat transfer area} = \left(\frac{158.16}{147.73} - 1\right) \times 100 = 7.06\ \%$$

It is less than 10%.

If number of tubes is increased to 1200,

$$D_b = 889,\ \text{shell ID} = 905\ \text{mm},\ N_t = 1200$$

$$a_t = \frac{1200}{1084} \times 0.2111 = 0.2337\ \text{m}^2$$

$$G_t = \frac{304.16}{0.2337} = 1301.5\ \text{kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{1301.5}{1440.83} \times 17\,454 = 15\,766.18$$

$$u_t = G_t/\rho = \frac{1301.5}{999.877} = 1.3016\ \text{m/s}$$

$$h_i = 4473.85 \times \left(\frac{15\,766.18}{17\,454}\right)^{0.8}$$

$$h_i = 4124.26\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

$$\bar{h}_{oc} \propto N_t^{1/3} \cdot N_r^{-1/6}$$

$$N_r = (2/3)\, N_r' = (2/3)\frac{D_b}{P_t} = \frac{2}{3}\left(\frac{889}{23.81}\right) = 24.9 \cong 25$$

$$\bar{h}_{oc} = 2345.7 \times \left(\frac{1200}{1084}\right)^{1/3} \times \left(\frac{25}{24}\right)^{-1/6} = 2410\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

$$U_{oc} = 801.97\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area required for condensation

$$A_{\text{creq}} = \frac{\phi_c}{U_{oc} \times \text{weighted } \Delta T} = \frac{3179.42 \times 10^3}{801.97 \times 45.385}$$

$$A_{\text{creq}} = 87.35\ \text{m}^2$$

$$U_{o\text{sub}} = 229.57\ \text{W/(m}^2 \cdot {}^\circ\text{C)} \quad \text{(based on new value of } h_i\text{)}$$

$$A_{\text{subreq}} = \frac{640.95 \times 10^3}{229.57 \times 45.385} = 61.52\ \text{m}^2$$

Total area required, $A_{\text{req}} = 87.35 + 61.52 = 148.87\ \text{m}^2$

$$A_{\text{pro}} = N_t \pi\, d_o L = 1200 \times \pi \times 0.019\,05 \times 2.4384\ \text{m} = 175.12\ \text{m}^2$$

$$\text{Excess heat transfer area} = \left(\frac{175.12}{148.87} - 1\right) \times 100 = 17.63\%\ \text{(adequate)}$$

If equal % excess heat transfer area for condensation and subcooling are considered then,

Area provided for condensation $A_{c\,\text{pro}} = 1.1763 \times A_{c\,\text{req}}$

$$A_{c\text{pro}} = 102.75\ \text{m}^2$$

$$A_{\text{sub pro}} = 175.12 - 102.75 = 72.37\ \text{m}^2$$

$$\frac{A_{\text{sub pro}}}{A_{t\,\text{pro}}} = \frac{72.37}{175.12} = 0.4133$$

Thus, 41.33% of total heat transfer area is available for subcooling and the same area should be submerged in the pool of condensate. Assuming that tubes will be uniformly distributed in the cross section of shell.

$$\frac{A_{\text{subpro}}}{A_{t\,\text{pro}}} = 0.4133 = \frac{\text{Area of segment of cross section utilised for subcooling}}{\text{Total cross section area of shell}}$$

$$\frac{xD_i^2}{\frac{\pi}{4}D_i^2} = 0.4133 \text{ or } x = 0.3246$$

From Table 1.19 b of Ref. 2:

for $x = 0.3246$, $\dfrac{h}{D_i} = 0.432$

To facilitate the subcooling of condensate or to submerge 41.33% of total heat transfer area in the pool of condensate, height of inverted U-seal (h) required from the bottom most point of shell inside diameter.

$$h = 0.432 \times 905 = 390.96 \text{ mm} \simeq 391 \text{ mm (as shown in Fig. 6.21)}$$

6.11 TINKER'S FLOW MODEL

All the latest methods (Delaware method; Sec. 6.12), for the process design of without phase change heat exchangers, are based on Tinker's flow model. If there is no phase change in shell side fluid then shell side heat transfer coefficient and shell side pressure drop in all recent methods are calculated based on this model. In old methods, like Kern's method, it is assumed that entire shell side fluid is flowing across the tube bundle and between the baffles. But actually shell side fluid is flowing in various ways. In Tinker's flow model shell side flow is divided in five different steams.

Fig. 6.22 *Tinker's Flow Model*[11]

Stream *A* Stream *A* is the tube-to-baffle leakage stream or it is the fraction of shell side fluid flowing through the clearance between tube hole in baffle and tube outside diameter. This stream does not bypass the heat transfer area (outside area of tubes) and hence, it does not create any adverse effect on the value of heat transfer coefficient. However, it makes the significant difference in pressure drop (loss). When stream *A* leaves this clearance it forms free flowing jet. Hence, boundary layer separation occurs and considerable friction loss or pressure drop takes place. Effect of stream *A* must be considered in the calculation of pressure drop.

Stream *B* Stream *B* is the actual cross flow stream or it is the fraction of shell side fluid which is flowing across the tube bundle and between the baffles. In Kern's method and other old methods it is assumed that entire shell side fluid is flowing like stream *B*.

Stream *C* Stream *C* is bundle to shell bypass stream or it is the fraction of shell side fluid flowing through the clearance area between shell inside diameter and tube bundle. Stream *C* is the main bypass stream. (bypassing the heat transfer area). This clearance area provides low pressure drop path for the shell side fluid. Hence, % of shell side fluid bypassed through this clearance area is quite significant. It is maximum with pull through floating head heat exchanger and minimum with fixed tube sheet heat exchanger. Amount of stream *C* can be reduced considerably by using sealing strips which are attached on inside surface of shell (See Fig. 6.7). They provide the partial blockage or the additional resistance in the path of stream *C*.

There is no stream designated as stream "*D*".

Stream *E* Stream *E* is the baffle to shell leakage stream. It is a part of shell side fluid flowing through the clearance between the edge of baffle and shell wall. Like stream *C*, this stream is also bypassing the heat transfer area and hence, reduces shell side heat transfer coefficient. But amount of stream *E* is lesser than amount of stream *C*. Normally, the clearance between baffle outside diameter and shell inside diameter is in the range of 1.6 to 4.8 mm while clearance between tube bundle and shell inside diameter is in the range of 10 to 100 mm.

Stream *F* Stream *F* is the pass-partition bypass stream. In tube sheets where pass partition plates are attached (for forming tube side passes), in that portion tubes cannot be provided. In multipass heat exchangers, one can find more number of gaps in tube bundle. Stream *F* is the fraction of shell side fluid flowing through these gaps. If the gap is vertical it provides low pressure drop path for fluid flow. Just like stream *C* and stream *E*, this stream is also bypassing the heat transfer area and reduces shell side heat transfer coefficient. Amount of stream *F* and its adverse effects are significant in multipass heat exchanger. To reduce the amount of this stream sometimes dummy tubes are used.

6.12 DELAWARE METHOD FOR DESIGN OF BAFFLED SHELL AND TUBE HEAT EXCHANGERS

It was seen in Sec. 6.11 that flow path across the tubes and various bypasses on shell side of a shell and tube heat exchanger are far more complicated than assumed in Kern's method. Provision of excess heat transfer area in Kern's method was just to account for the correction in the shell side heat transfer coefficient (h_o) due to the disturbed fluid flow path. In a competitive environment, it became necessary to design a baffled shell and tube heat exchanger with reasonable accuracy.

Prof. A. P. Colburn, Prof. O. P. Bergelin and their other colleagues undertook a collaborative efforts at the University of Delaware, USA to define various corrections for the fluid path B and other bypasses on shell side. Dr. K. J. Bell, a Research Fellow of the program, compiled the finding and prepared a report[15] on the subject. These findings and correlations are updated from time to time[16]. The method, developed on the basis of the report, is known as *Delaware method* (also popularly known as Bell's method). This method gives more satisfactory predictions of shell side heat transfer coefficient ($\boldsymbol{h_o}$) and its pressure drop ($\Delta \boldsymbol{p_s}$) for cooling and heating of shell side fluid. An important change in definition of Reynolds number ($\boldsymbol{Re_o}$) is based on mass velocity through the minimum cross-sectional area in tube banks.

$$\boldsymbol{Re_o} = \frac{d_o \boldsymbol{G_s}}{\mu} = \frac{d_o \boldsymbol{u_o} \rho}{\mu} \tag{6.57}$$

where, d_o = Outside diameter of tube, m

$\boldsymbol{G_s}$ = Mass velocity on shell side, kg/(m^2 · s)

$= \dot{m}/\boldsymbol{A_s}$

μ = Viscosity of shell side fluid at average temperature, kg/(m · s)

$$\boldsymbol{u_s} = \boldsymbol{G_s}/\rho \quad \text{m/s} \tag{6.58}$$

$\boldsymbol{A_s}$ is shell side flow area which is determined by following correlations:
For triangular layout of tube bundle,

$$\boldsymbol{A_s} = B_s \left[D_i - D_b + \left(\frac{D_b - d_o}{P_t} \right) \left(P_t - d_o \right) \right] \tag{6.59a}$$

and for linear and rotated square tube layouts,

$$\boldsymbol{A_s} = B_s \left[D_i - D_b + \left(\frac{D_b - d_o}{P_n} \right) \left(P_t - d_o \right) \right] \tag{6.59b}$$

where, B_s = Baffle spacing,

D_i = Inside diameter of shell, m

D_b = Tube bundle diameter, m

d_o = Outside diameter of tube, m

P_t = Tube pitch, m

P_n = Tube pitch normal to flow or distance between centres of adjacent tubes normal to flows, m

Note that correlation, (Eq. 6.58) for $\boldsymbol{A_s}$ is different from A_s in Eq. (6.33) of Kern's method.

Based on $\boldsymbol{Re_o}$, defined in Eq. (6.57), flow regimes are defined as under which are different from Re_o defined in Eq. (6.38).

0	$< \boldsymbol{Re_o} < 100$	Laminar
100	$< \boldsymbol{Re_o} < 4000$	Transition
4000	$< \boldsymbol{Re_o}$	Turbulent

In this book, correlations for $\boldsymbol{Re_o} > 100$ only are considered.

6.12.1 Shell Side Heat Transfer Coefficient

Heat transfer coefficient ($\boldsymbol{h_o}$) for no phase change (of fluid) is now defined as

$$\boldsymbol{h_o} = h_{oc}\, J_c\, J_b\, J_l\, J_s \tag{6.60}$$

where, h_{oc} = Shell side heat transfer coefficient, calculated for cross flow (stream *B* of (Fig. 6.22) over ideal tube bank with no leakage or bypassing, W/(m^2.°C).

J_c = Correction factor to consider the effect of baffle configuration

J_b = Bypass stream correction factor

J_l = Baffle leakage correction factor

J_s = Correction factor for unequal baffle spacing at inlet/outlet

Heat transfer coefficient (h_{oc}) can be calculated using following the correlation:

$$\frac{h_{oc}\, d_o}{k} = \boldsymbol{J_h}\, \boldsymbol{Re_o}\, Pr^{1/3} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.61}$$

where, $\boldsymbol{J_h}$ = Colburn *J*-factor for an ideal tube bank

Values of $\boldsymbol{J_h}$ can be read from Fig. 6.23.

To consider the effect of window zone on shell side heat transfer coefficient, fraction (F_c) of total tubes that are in cross flow is found from Fig. 6.24. Correction factor J_c can then be read from Fig. 6.25 as a function of F_c. For no tubes in the window zone, $J_c = 1.0$.

To consider the effect of bypass streams *C* and *F* (see Fig. 6.22), fraction of cross-flow area, available for bypass flow, is calculated by the following correlation:

$$F_{sbp} = \frac{\left[D_i - D_b + 1/2\,(n_{pv}\, W_p)\right] B_s}{\boldsymbol{A_s}} \tag{6.62}$$

where, n_{pv} = Number of vertical gaps in tube bundle created by vertical pass partition plates or number of pass partition lanes, through the tube field, parallel to the direction of cross-flow stream

W_p = Width of above gaps or lanes, m

= Nominal pass partition plate thickness + 0.006 35 m

B_s = Central baffle spacing, m

Fig. 6.23 *Correlation of Colburn J_h for Ideal Tube Banks with Plain Tubes*[16]

Fig. 6.24 *Estimation of Fraction of Tubes in Cross Flow*[16]

Table 6.23 Nominal Pass Partition Plate Thickness

Nominal size of shell, mm	Plate thickness, mm	
	Carbon steel	Alloy material
610	9.5	6.35
610 – 1524	12.7	9.5
1549 – 2540	15.9	12.7

Fig. 6.25 *Correction Factor for Baffle Configuration Effects*[16]

Fig. 6.26 *Correction Factor for Bypass Stream*[16]

Adverse effect of bypass stream on heat transfer coefficient can be significantly reduced by providing sealing strips (see Fig. 6.7).

Let N_{ss} = Number of pairs of sealing strips, encountered by bypass stream (C in Fig. 6.22) in cross-flow zone

N_c = Number of tube rows crossed during flow through one cross-flow section

$$= \frac{(D_b - 2\,\boldsymbol{x}\,D_b)}{P_t \sin 60} \text{ (for } \triangle \text{ pitch arrangement)} \tag{6.63a}$$

$$= \frac{(D_b - 2\,\boldsymbol{x}\,D_b)}{P_t} \text{ (for } \square \text{ pitch arrangement)} \tag{6.63b}$$

$$= \frac{(D_b - 2\,\boldsymbol{x}\,D_b)}{P_n} \text{ (for } \diamond \text{ pitch arrangement)} \tag{6.63c}$$

$\boldsymbol{x}$ = Fraction of baffle cut (see Fig. 6.2)

Bypass stream correction factor J_b can be read from Fig. 6.26, knowing F_{sbp} and N_{ss}/N_c ratio.

To consider the effect of leakage streams A and E on $\boldsymbol{h_o}$, tube to baffle leakage area can be determined by the following correlation:

$$A_{tb} = \left(\frac{\pi}{4}\right) d_o\, C_{tb}\, (1 + F_c)\, N_t \tag{6.64}$$

where, C_{tb} = Diametrical clearance between tube hole in baffle and OD of tube

= 0.8 mm or 0.0008 m (normal value)

F_c = Fraction of total tubes in cross flow

Shell to baffle leakage area for one baffle, A_{sb}, can be determined by the following correlation:

$$A_{sb} = \frac{\pi\, D_i\, C_{sb}}{2}\left[1 - \frac{\alpha}{2\pi}\right] \tag{6.65}$$

where, C_{sb} = Diametrical clearance between shell and baffle, m

α = Baffle cut angle or angle subtended by intersection of the cut edge of the baffle with inside surface of shell, radians

$$= 2\cos^{-1}(1 - 2\,\boldsymbol{x}) \text{ radians} \tag{6.66}$$

Calculations of A_{tb}, A_{sb} and $\boldsymbol{A_s}$ yield baffle leakage correction factor J_l from Fig. 6.27.

Correction factor, J_s, for unequal baffle spacing at inlet and/or outlet can be determined by the following correlation:

$$J_s = \frac{(N_b - 1) + R_{si}^{(1-n)} + R_{so}^{(1-n)}}{(N_b - 1) + R_{si} + R_{so}} \tag{6.67}$$

where, N_b = Number of baffles
$R_{si} = B_{si}/B_s$
$R_{so} = B_{so}/B_s$
B_s = Internal (central) baffle spacing, m
B_{si} = Entrance baffle spacing, m
B_{so} = Exit baffle spacing, m
n = 0.6 for $\boldsymbol{Re_o} > 100$; i.e. for transition and turbulent flows

$$N_b = \frac{L - B_{si} - B_{so}}{B_s} + 1 \tag{6.68}$$

Substitution of various parameters in Eq. (6.60) will yield $\boldsymbol{h_o}$.

Fig. 6.27 *Correction Factor for Baffle Leakage Effects*[16]

6.12.2 Shell Side Pressure Drop

$$\Delta p_s = [(N_b - 1)\,(\Delta p_{bi})\,R_b + N_b\,\Delta p_{wi}]R_l + 2\,\Delta p_{bi}\,R_b\left(1 + \frac{N_{cw}}{N_c}\right)R_s \tag{6.69}$$

where, N_{cw} = Number of effective cross-flow rows in each window section

$$= \frac{x\,D_b}{p_t \sin 60} \text{ for } \triangle \text{ pitch arrangement} \tag{6.70a}$$

$$= \frac{x\,D_b}{p_t} \text{ for } \square \text{ pitch arrangement} \tag{6.70b}$$

$$= \frac{x\,D_b}{p_n} \text{ for } \diamond \text{ pitch arrangement} \tag{6.70c}$$

Δp_{bi} = Pressure drop during flow across one ideal cross-flow section, Pa
Δp_{wi} = Pressure drop through one ideal window section, Pa
R_b = Correction factor for effect of bundle bypass
R_l = Correction factor for baffle leakage effect
R_s = Correction factor for unequal baffle spacing

Pressure drop during flow across an ideal cross-flow section, Δp_{bi}, can be determined using the following correlation:

$$\Delta p_{bi} = 8\,J_f\,N_c\left(\frac{\rho_s\,u_o^2}{2}\right)\left(\frac{\mu}{\mu_w}\right)^{-0.14} \tag{6.71}$$

where, J_f = Friction factor for ideal tube bank. It can be obtained from Fig. 6.28.
N_c = Number of tube rows crossed during one flow through one cross-flow section
ρ_s = Density of shell side fluid, kg/m^3

Equation (6.71) is similar to Eq. (6.44) with a few changes.

Pressure drop through one ideal window section, Δp_{wi}, can be determined using the following correlation:

$$\Delta p_{wi} = \frac{\dot{m}^2\,(2 + 0.6\,N_{cw})}{2\,A_s\,A_w\,\rho_s} \tag{6.72}$$

where,

N_{cw} = Number of effective cross-flow rows in each window section
A_w = Flow area of window, m^2

$$= A_{wg} - A_{wt} \tag{6.73}$$

A_{wg} = Gross window area, m^2

$$= \frac{D_i^2}{4}\left[\frac{\alpha}{2} - (1 - 2x)\sin\left(\frac{\alpha}{2}\right)\right] \tag{6.74}$$

Fig. 6.28 *Correlation of Friction Factors for Ideal Tube Banks*[16]

Fig. 6.29 *Correction Factor for Pressure Drop for Tube Bundle Bypass Flow*[16]

A_{wt} = Window area occupied by tubes, m^2

$$= \frac{N_t}{8}(1 - F_c)\,\pi\, d_o^2 \tag{6.75}$$

Correction factor, R_b, for the effect of tube bundle bypass can be read from Fig. 6.29.

Correction factor, R_l, for the effect of baffle leakage can be read from Fig. 6.30.

Fig. 6.30 *Correction Factor for Baffle Effect on Pressure Drop*[16]

Correction factor, R_s, for unequal baffle spacing can be determined using the following correlation:

$$R_s = \frac{1}{2}\left(R_{si}^{-n'} + R_{so}^{-n'}\right) \tag{6.76}$$

where, $n' = 1.6$ for turbulent flow ($\boldsymbol{Re_o} > 100$)

Substitution of various parameters in Eq. (6.69) yields predicted pressure drop on shell side. In actual practice, it is observed[15] that $\Delta \boldsymbol{p_s}$, calculated by correlation Eq. (6.69) ranged from 0.75 to 1.05 times the measured overall pressure drop with an average value of 0.9. This pressure drop does not account for nozzle (pressure) losses.

Delaware method is much more detailed than given in this Sec. 6.12 and contains variety of correlations for different tube pitches, types of tubes (such as finned) and other configurations. Reader is advised to refer Ref. 15 and 16 for other correlations.

Example 6.6 Synthesis make-up gas of ammonia plant is to be cooled in a shell and tube heat exchanger from 320°C to 155°C by exchanging heat with boiler feed water (BFW) entering at 125°C in countercurrent direction. Total (wet) volumetric gas flow rate is 140 000 Nm^3/h which flows through the tubes at 27.35 bar a while BFW flows at the rate of 41 500 kg/h in the shell at 125 bar a. Dry gas composition is 74% H_2, 24.7% N_2, 0.3% Ar and 1% CH_4 by mole. Stoichiometric calculations reveal that presence of water in the gas mixture is 0.0205 kmol/kmol dry gas. Following heat exchanger is available for the cooling service. Check whether the heat exchanger will be adequately rated for the service by Delaware method.

Type of heat exchanger: 1 – 1 shell and tube BEM type
Shell diameters = 964.4 mm (outside), 939 mm (inside)
No. of tubes = 1150
Details of tubes: MS, OD = 19.05 mm, ID 14.8336 mm (14 BWG)
Tube length = 9140 mm
Tube layout: Triangular pitch (Δ) = 25.4 mm
Tube bundle diameter (D_b) = 925 mm
Baffles: 25% cut segmental
Baffle spacings: 300 mm (central), 370 mm (at inlet and outlet)
Diametrical clearance between tube and baffle = 0.8 mm
Thermodynamic and transport properties of gas mixture components and water are listed in Table 6.24.

Solution

Basis: Total volumetric gas flow rate = 140 000 Nm^3/h (wet)

$$\text{Molar gas flow rate,} \qquad \dot{n} = \frac{140\,000}{22.414} = 6246.096 \text{ kmol/h (wet)}$$

Moisture content of gas, H_m = 0.0205 kmol/kmol dry gas
Thus 1.0205 kmol wet gas mixture contains 0.74 kmol H_2, 0.247 kmol N_2, 0.003 kmol Ar, 0.01 kmol CH_4 and 0.0205 kmol H_2O.
On this basis, mole fractions of the components of the wet gas mixture are calculated to be 0.7251 H_2, 0.242 N_2, 0.0029 Ar, 0.0098 CH_4 and 0.0202 H_2O.

Nitrogen in the gas mixture, $\dot{n}_{N_2} = 6246.096 \times 0.242$
$= 1511.555$ kmol/h

Similarly,

$$\dot{n}_{H_2} = 6246.096 \times 0.725 = 4529.044 \text{ kmol/h}$$

$$\dot{n}_{Ar} = 6246.096 \times 0.0029 = 18.114 \text{ kmol/h}$$

$$\dot{n}_{CH_4} = 6246.096 \times 0.0098 = 61.212 \text{ kmol/h}$$

$$\dot{n}_{H_2O} = 6246.096 \times 0.0202 = 126.17 \text{ kmol/h}$$

Table 6.24 Thermodynamic and Transport Properties

Component	Phase	Pres., bar a	Temp., °C	Property value*				
				Enthalpy** (H), kJ/kg	Viscosity (μ), μPa · s	Thermal cond. (k), mW/(m · K)	Density (ρ), kg/m^3	Heat capacity (C_p), kJ/(kg · °C)
Hydrogen	Gas	atm	155	5807.8	11.407	246.71	0.0574	14.492
M = 2.0159			320	8203.1	14.277	324.81	0.0414	14.546
	Gas	19.8315	155	5920.4	11.438	247.85	1.1131	14.514
			320	8218.1	14.250	325.70	0.8053	14.555
Nitrogen	Gas	atm	155	566.92	23.334	34.615	0.7971	1.0473
M = 28.0134			320	741.64	29.345	44.455	0.5763	1.0737
	Gas	6.6187	155	560.86	23.381	34.747	5.1977	1.0513
			320	741.62	29.370	44.549	3.7495	1.0755
Methane	Gas	atm	155	1224.3	14.982	54.767	0.4568	2.6340
M = 16.0425			320	1709.5	19.245	87.356	0.3296	3.2479
	Gas	0.2680	155	1224.7	14.975	54.733	0.1208	2.6323
			320	1709.7	19.239	87.332	0.0872	3.2471
Argon	Gas	atm	155	340.20	30.289	23.743	1.1370	0.5209
M = 39.948			320	426.11	38.672	30.320	0.8207	0.5206
	Gas	0.0793	155	340.28	30.259	23.721	0.0902	0.5204
			320	426.14	30.665	30.303	0.0651	0.5204
Water	Gas	atm	155	2367.4	14.379	29.262	0.5169	1.9827
M = 18.0153			320	2695.8	21.122	45.610	0.3709	2.0232
	Gas	0.5525	155	2370.5	14.401	28.990	0.2808	1.9513
			320	2696.8	21.126	45.544	0.2021	2.0181
Water	Satd. liquid	2.322	125	106.02	222.080	683.56	939.02	4.2521
M = 18.0153		64.166	280	817.83	93.506	581.15	750.28	5.2889
	Compr. liquid	125	125	114.53	225.270	690.96	945.16	4.2210
			280	814.88	95.472	591.64	759.90	5.1385

* Ref.: REFPROP Ver. 8.0, NIST Standard Reference Data Base 23, 2007

** Reference enthalpy of saturated liquid (h) = 0 kJ/kg at NBP

Average molar mass of the wet gas mixture,

$$M_{\text{mix}} = 0.7251 \times 2.0159 + 0.242 \times 28.0134 + 0.0029 \times 39.948 + 0.0098 \times 16.0425 + 0.0202 \times 18.0153$$
$$= 8.8778 \text{ kg/kmol}$$

Properties of each component at respective partial pressure (p_i) are required.

$$p_i = y_i\, p$$

where, p = total pressure = 27.35 bar

In Table 6.24, properties are listed at atmospheric pressure as well as at respective partial pressure for each component. Study of the property data reveal that except for the density (ρ), other properties are close enough at two pressures. For heat transfer rate, evaluations are made at both pressures.

At atmospheric pressure,

$$\phi = (8203.1 - 5807.8)\, 2.0159 \times 4529.044 + (741.64 - 566.92)\, 28.0134 \times 1511.555 + (1709.5 - 1224.3)\, 16.0425 \times 61.212 + (426.11 - 340.20)\, 39.948 \times 18.114 + (2695.8 - 2367.4)\, 18.0153 \times 126.171$$
$$= 30\,552\,721 \text{ kJ/h} \equiv 8486.867 \text{ kW}$$

At partial pressures,

$$\phi' = (8218.1 - 5920.4)\, 2.0159 \times 4529.044 + (741.62 - 560.86)\, 28.0134 \times 1511.555 + (1709.7 - 1224.7)\, 16.0425 \times 61.212 + (426.14 - 340.28)\, 39.948 \times 18.114 + (2696.8 - 2370.5)\, 18.0153 \times 126.171$$
$$= 29\,912\,374 \text{ kJ/h} \equiv 8038.993 \text{ kW} \equiv 8038.993 \times 10^3 \text{ W}$$

Gas mixture's heat transfer rate can also be calculated with the use of ideal gas heat capacity data and considering the mixture to be an ideal one (Ref. 10).

$$C^{\circ}_{p\,\text{mix}} = \sum \left(\dot{n}_i\, a_i + \dot{n}_i\, b_i \times 10^{-3} T + \dot{n}_i\, c_i + 10^{-6}\, T^2 + \dot{n}_i\, d_i \times 10^{-9}\, T^2 \right)$$
$$= 28.8715 - 0.338 \times 10^{-3}\, T + 4.0491 \times 10^{-6}\, T^2 - 1.1367 \times 10^{-9}\, T^3$$
$$\phi'' = \dot{n} \int_{(273.15+155)}^{(273.15+320)} C^{\circ}_{p\,\text{mix}}\, dT$$
$$= 30\,514\,829 \text{ kJ/h} \equiv 8476.341 \text{ kW}$$

ϕ' differ by –2.14% from ϕ and –2.01% from ϕ''. Since actual enthalpy values could be determined at respective partial pressures, heat transfer rate at partial pressures (ϕ') will be considered for the heat exchanger design.

For the first estimation of BFW outlet temperature (t'), consider C_p = 4.1868 kJ/(kg · °C) for water.

$$t' = \frac{29\,912\,374}{(41\,500 \times 4.1868)} + 125$$
$$= 297.16°\text{C}$$

However, it can be seen from Table 6.24 that overage C_p of compressed water (at 125 bar a) is 4.6798 kJ/(kg · °C).

With this value,

$$t' = \frac{29\,912\,374}{(41\,500 \times 4.6798)} + 125$$
$$= 279.02°\text{C, say } 279°\text{C}$$

If average C_p value for saturated liquid water would have been considered, t' would be calculated as 276.09°C.

Above calculations indicate importance of correct thermodynamic and transport properties in process design.

Following average values of properties will be considered for the heat exchanger design:

Table 6.25 Average Thermodynamic and Transport Properties

Component	Phase	Average property value			
		Viscosity (μ), μPa · s	Thermal cond. (k), mW/(m · K)	Density (ρ) kg/m^3	Heat capacity, (C_p) kJ/(kg · °C)
Hydrogen	Gas	12.844	286.780	0.9592	14.5345
Nitrogen	Gas	26.376	39.648	4.4736	1.0634
Methane	Gas	17.107	71.033	0.1040	2.9397
Argon	Gas	30.462	27.012	0.0777	0.5204
Water	Gas	17.764	37.267	0.2415	1.9847
Water	Compr. liquid	160.371	641.300	852.53	4.6798

Mass flow rate of gas mixture, $\dot{m}_1 = 6246.096 \times 8.8778$ (in tubes)

$= 55\,451.6$ kg/h

Based on Eq. (6.20), $$a_t = \frac{1150}{1} \times \frac{\pi}{4}(0.014\,8336)^2 = 0.198\,74 \text{ m}^2$$

Tube side mass velocity, $$G_t = \frac{(55\,451.6/3600)}{0.198\,74}$$

$$= 77.5044 \text{ kg/(m}^2 \cdot \text{s)}$$

Viscosity of gas mixture is calculated using Eq. (3.87) of Perry's Handbook (Ref. 2)

$$\mu_{\text{mix}} = \sum_{i=1}^{n} \frac{y_i\ \mu_i}{\sum_{j=1} y_j\ \Theta_{ij}} \tag{6.77}$$

Selecting simpler form of Θ_{ij},

$$\Theta_{ij} = (M_j/M_i)^{1/2} \tag{6.78}$$

i represents any component while j represents component, other than i.

$$\mu_{\text{mix}} = \frac{(0.242 \times 26.376)}{(0.7251 \times 0.2683 + 0.0029 \times 1.194 + 0.0098 \times 0.757 + 0.0202 \times 0.802)}$$

$$+ \frac{(0.7251 \times 12.844)}{(0.242 \times 3.7278 + 0.0029 \times 4.4516 + 0.0098 \times 2.821 + 0.0202 \times 2.9894)}$$

$$+ \frac{(0.0029 \times 30.462)}{(0.7251 \times 0.2246 + 0.242 \times 0.8374 + 0.0098 \times 0.6337 + 0.0202 \times 0.6715)}$$

$$+ \frac{(0.0098 \times 17.107)}{(0.7251 \times 0.3545 + 0.242 \times 1.3214 + 0.0029 \times 1.578 + 0.0202 \times 1.0597)}$$

$$+ \frac{(0.0202 \times 17.764)}{(0.7251 \times 0.3345 + 0.242 \times 1.247 + 0.0098 \times 0.9436 + 0.0027 \times 1.489)}$$

$$= 39.2353 \ \mu\text{Pa} \cdot \text{s}$$

If $\mu'_{\text{mix}} = \sum y_i \, \mu_i$ form would have been selected,

$\mu'_{\text{mix}} = 16.3107 \ \mu\text{Pa} \cdot \text{s}$ [41.6% of μ_{mix} computed by Eq. (6.77)]

Reynolds number,

$$Re_t = \frac{d_i \, G_t}{\mu_{\text{mix}}} = \frac{0.014\,8336 \times 77.5044}{39.2353 \times 10^{-6}}$$

$$= 29\,302$$

Average heat capacity of gas mixture,

$$C_{p\text{mix}} = \frac{\phi'}{\dot{m}_i \, \Delta t'} = \frac{29\,912\,374}{55\,451.6\,(320 - 155)}$$

$$= 3.269 \ \text{kJ/(kg} \cdot {}^\circ\text{C)}$$

If $C_{p\,\text{mix}}$ would have been calculated by $C_{p\,\text{mix}} = \sum \dot{n}_i \, C_{pi}$, it would have yielded same answer.

Average thermal conductivity of gas mixture (Ref. 2),

$$k_{\text{mix}} = \sum k_i \, y_i \tag{6.79}$$

$$= (286.78 \times 0.7251 + 39.684 \times 0.242$$

$$+ 71.033 \times 0.0098 + 27.012 \times 0.0029 + 37.267 \times 0.0202)$$

$$= 219.066 \ \text{mW/(m} \cdot \text{K)}$$

$$\equiv 0.219 \ \text{W/(m} \cdot \text{K)}$$

Prandtl number, $Pr = \dfrac{3.269 \times 10^3 \times 39.2353 \times 10^{-6}}{0.219}$

$$= 0.585\,66$$

Using Dittus–Bolter equation [Eq. (6.19)],

$$\frac{h_i \, d_i}{k_{\text{mix}}} = 0.021 \times Re_t^{0.8} \quad Pr^{0.33} \left(\frac{\mu}{\mu_\omega} \right)^{0.14}$$

$$h_i = 0.021 \times \frac{0.219}{0.014\,8336} \times (29\,302)^{0.8} \times (0.585\,56)^{0.33}$$

$$= 973.3 \ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

Shell side coefficient by Delaware method:

BFW flows in the shell at 125 bar a. This will require much thicker shell.

$$\boldsymbol{h_o} = h_{oc} \, J_c \, J_b \, J_l \, J_s \tag{6.60}$$

For triangular pitch, referring Eq. (6.59a),

$$\boldsymbol{A_s} = 300\left[939 - 925 + \left(\frac{925 - 19.05}{25.4}\right)(25.4 - 19.05)\right]$$

$$= 72\,146.25 \text{ mm}^2$$

$$\equiv 0.072\,146 \text{ m}^2$$

$$\boldsymbol{G_s} = \dot{m}_2/\boldsymbol{A_s}$$

$$= \frac{41\,500}{(3600 \times 0.072\,146)}$$

$$= 159.784 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\boldsymbol{Re_o} = \frac{0.019\,05 \times 159.784}{0.160\,371 \times 10^{-3}} = 18\,980.3$$

From Fig. 6.23, for $\boldsymbol{Re_o} = 18\,980.3$,

$$\boldsymbol{J_h} = 0.008$$

$$Pr = \frac{4.6789 \times 10^3 \times 0.160\,371 \times 10^{-3}}{0.6413}$$

$$= 1.1703$$

Referring Eq. (6.61)

$$h_{oc} = 0.008 \times \frac{0.6413}{0.019\,05} \times 18\,980.3 \times (1.1703)^{1/3}$$

$$= 5386.7 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

For 25% cut baffles, referring Fig. 6.24, $F_c = 0.647$
Referring Fig. 6.25, $J_c = 1.018$
Effect of bypass streams C and F:
For 1–1, heat exchanger, $n_{pv} = 0$
Referring Eq. (6.62),

$$F_{sbp} = \frac{(939 - 925)\,300}{72\,146.25} = 0.0582$$

No. of sealing strips, $N_{ss} = 0$ or $N_{ss}/N_c = 0$

$$\mathbf{x} = 0.25$$

Referring Fig. 6.26, $J_b = 0.931$
Effect of leakage streams A and E:
Referring Eq. (6.64),

$$A_{tb} = \left(\frac{\pi}{4}\right) 19.05 \times 0.8\,(1 + 0.647)\,1150$$

$$= 22\,670.8 \text{ mm}^2$$

Baffle

Eq. (6.66); $\alpha = 2\cos^{-1}(1 - 2 \times 0.25)$

$$= 120^\circ \equiv 2.0944 \text{ rad}$$

Eq. (6.65); $$A_{sb} = \left(\frac{\pi}{2}\right) 939 \times 4.8 \left[1 - \frac{2.0944}{2\pi}\right]$$

$$= 4719.9 \text{ mm}^2$$

$$\frac{A_{sb} + A_{tb}}{\boldsymbol{A_s}} = \frac{(4719.9 + 22\,670.8)}{72\,146.25} = 0.3796$$

$$\frac{A_{sb}}{A_{sb} + A_{tb}} = \frac{4719.9}{(4719.9 + 22\,670.8)} = 0.1723$$

From Fig. 6.27, $J_l = 0.685$

No. of baffles, $N_b = 28$

$$R_{si} = 370/300 = 1.2333$$

$$R_{so} = 370/300 = 1.2333$$

$$n = 0.6$$

Referring Eq. (6.67)

$$J_s = \frac{(28 - 1) + 1.2333^{(1-0.6)} \times 2}{(28 - 1) + 2 \times 1.2333}$$

$$= 0.99$$

Substitute various parameters in Eq. (6.60).

$$\boldsymbol{h_o} = 5386.7 \times 1.018 \times 0.931 \times 0.685 \times 0.99$$

$$= 3462.15 \text{ W/(m}^2 \cdot \text{°C)}$$

For calculating overall heat transfer coefficient, U_o, use Eq. (6.46).

$$h_{od} = 10\,000 \text{ W/(m}^2 \cdot \text{°C) for BFW (i.e. for clean water)}$$

$$h_{id} = 10\,000 \text{ W/(m}^2 \cdot \text{°C) for gas mixture (clean fluid)}$$

$$\frac{1}{U_o} = \frac{1}{3462.15} + \frac{1}{10\,000} + \frac{0.019\,05 \ln (19.05/14.8336)}{2 \times 16} + \frac{19.05}{14.8336}\left(\frac{1}{973.3} + \frac{1}{10\,000}\right)$$

$$U_o = 503.61 \text{ W/(m}^2 \cdot \text{°C)}$$

$$\Delta T_{lm} = \frac{(320 - 279.02) - (155 - 125)}{\ln\left(\frac{320 - 279.02}{155 - 125}\right)}$$

$$= 35.205\text{°C} = \Delta T_m \text{ for 1-1 pass HE}$$

Using Eq. (6.16),

$$A_{\text{req}} = \left(\frac{29\,912\,374 \times 1000}{3600}\right) \times \frac{1}{(503.61 \times 35.205)}$$

$$= 468.65 \text{ m}^2$$

Actual provided HTA,

$$A_o = \pi \times 1150 \times 0.019\,05 \times 9.14$$
$$= 629.055 \text{ m}^2$$

$$\text{Excess HTA} = \frac{(629.055 - 468.65)}{468.65} \times 100$$
$$= 34.23\% \qquad \text{(highly adequate)}$$

Tube side pressure drop:

For $Re_t = 29\,302.4$,

$J_f = 0.0038$ (Fig. 6.13)

Density of gas mixture,

$$\rho = \frac{27.35 \times 8.8778}{[273.15 + [(320 + 155)/2]} \times \frac{273.15}{1.013\,25 \times 22.414}$$
$$= 5.7173 \text{ kg/m}^3$$
$$u_t = 77.5056/5.7173 = 13.556 \text{ m/s}$$

Using Eq. (6.31),

$$\Delta p_t = \left[8 \times 0.0038 \times \left(\frac{9140}{14.8336}\right) \times 1 + 2.5\right]\left(\frac{5.7173 \times 13.556^2}{2}\right)$$
$$= 11\,154 \text{ Pa}$$
$$\equiv 0.111 \text{ bar (quite low)}$$

Shell side pressure drop:

Pressure drop for an ideal cross-flow section can be calculated using Eq. (6.48).

For $\boldsymbol{Re_o} = 18\,980.27$, $J_f = 0.153$ (Fig. 6.15)

$$\Delta p_{bi} = 8 \times 0.153 \times 21 \times \frac{(852.53 \times 0.1874^2)}{2}$$
$$= 348.8 \text{ Pa}$$

For calculating shell side pressure drop by Delaware method, Eq. (6.69) is to be used.

$$\dot{m}_2 = (41\,500/3600) = 11.5278 \text{ kg/s}$$

$$N_{cw} = \frac{\boldsymbol{x}\, D_b}{P_t \sin 60}$$
$$= \frac{0.25 \times 925}{25.4 \times \sin 60} = 10.513$$
$$\boldsymbol{A_s} = 0.072\,146 \text{ m}^2$$

Using Eq. (6.74),

$$A_{wg} = \frac{939^2}{4}\left[\frac{2.0944}{2} - (1 - 2 \times 0.25) \sin 60\right]$$
$$= 135\,385.46 \text{ mm}^2$$

Using Eq. (6.75),

$$A_{wt} = \frac{1150}{8}(1 - 0.647)\,\pi \times (19.05)^2$$
$$= 65\,555.3 \text{ mm}^2$$

Referring Eq. (6.73),

$$A_w = 135\,358.46 - 65\,555.3$$
$$= 69\,830.16 \text{ mm}^2$$

Referring Eq. (6.71),

$$\Delta p_{wi} = \frac{11.5278^2\,(2 + 0.6 \times 10.513)}{2 \times 0.072\,146 \times 0.069\,830 \times 852.53}$$
$$= 128.404 \text{ Pa}$$

Correction factors R_b and R_t can be obtained from Figs. 6.29 and 6.30, respectively.

For $F_{sbp} = 0.0582$ and $N_{ss}/N_c = 0$,

$R_b = 0.792$ (Fig. 6.29)

For $\dfrac{A_{sb}}{(A_{sb} + A_{tb})} = 0.1723$ and $\dfrac{A_{sb} + A_{tb}}{\boldsymbol{A_s}} = 0.3796$,

$R_l = 0.434$ (Fig. 6.30)

Using Eq. (6.76),

$$R_s = \frac{1}{2}\left[1.2333^{-1.6} \times 2\right]$$
$$= 0.715$$

Using Eq. (6.69),

$$\Delta \boldsymbol{p_s} = [(28 - 1) \times 384.8 \times 0.792 + 28 \times 128.404]$$
$$\times 0.434 + 2 \times 384.8 \times 0.792 \times \left(1 + \frac{10.5}{21}\right) 0.715$$
$$= 5785.3 \text{ Pa}$$
$$\equiv 5.785 \text{ kPa (acceptable)}$$

Notes: (i) Temperature ranges on shell and tube sides are high and hence, thermal expansion analysis as per TEMA is required. A suitable expansion joint may be necessary on the shell.

(ii) The example was also solved with Kern's method and comparison of the results are given below.

	Delaware (Bell's) Method	Kern's method
h_0	3462.15 W/(m^2 · °C)	3102.5 W/(m^2 · °C)
Δp_s	5.785 kPa	8.867 kPa
U_o	503.61 W/(m^2 · °C)	495.26 W/(m^2 · C)
% Excess HTA	34.23	32

It may be noted that there is not much difference in the results. This is because it is BEM type (i.e., fixed tube sheet type) 1-1 heat exchanger. Contributions of bypass streams (C and *F)* are not significant to show any difference in the results. However, if the type of heat exchanger is AET (i.e., pull through floating head) with more number of tube side passes, contributions of bypass streams; C and *F* would have been significant. In Exercise 6.5, the heat exchanger is AET with 8 number of tube side passes is designed. For such a case, h_0, given by Kern's method, could be 1.5 to 2 times the $\boldsymbol{h_o}$, calculated by Delaware method. Also shell side pressure drop (Δp_s), given by Kern's method could have been 3 to 5 times the value given by Delaware method. Thus Delaware method gives correct design for AET type heat exchanger with more number of tube passes. Hence, for such heat exchangers, Kern's method gives lesser HTA than actually required.

6.13 PROCESS DESIGN OF REBOILERS AND VAPORIZERS

Design correlations for reboilers and vaporizers are same. Reboilers are used with distillation columns. In a reboiler, part of liquid feed is vaporized while in a vaporizer the entire feed of liquid is vaporized.

Following types of reboilers are used in chemical industries:

1. Kettle-type Reboiler
2. Bundle-in-column Reboiler
3. Horizontal Thermosyphon Reboiler
4. Vertical Thermosyphon Reboiler
5. Forced Circulation Reboiler.

Process designs of Kettle-type Reboiler and Vertical Thermosyphon Reboiler are given in subsequent sections.

6.13.1 Process Design of Kettle-type Reboiler

Kettle-type reboiler (see Fig. 6.1(e)) is normally used with a distillation column and also as a vaporizer. Vapour leaving the kettle-type reboiler is always in equilibrium with the liquid. In this type of reboiler, heating surface is immersed in the pool of liquid. Hence, perfect pool boiling is taking place. In pool boiling, maximum value of heat transfer coefficient is achieved in nucleate boiling region. To achieve the nucleate boiling region, $\Delta\mathcal{T}$ (Temperature difference between temperature of heating surface and boiling temperature of liquid) should be less than and close to critical temperature drop. Critical temperature drop for water is 20 to 30°C and for light organics is 20 to 50°C at atmospheric pressure. In kettle-type reboiler boiling fluid side pressure drop is negligible, hence, it is preferred with high vacuum distillation column.

Process design steps

1. **Calculation of heat duty** In actual design calculation, heat duty of reboiler is calculated based on energy balance equation around distillation column.

$$\phi_B = H_D\dot{n}_D + H_W\dot{n}_W - H_F\dot{n}_F + \phi_C + \phi_L \tag{6.79}$$

where, ϕ_B = Heat duty of reboiler, kW

H_D = Enthalpy of distillate, kJ/kmol
$\dot{n}_D$ = Molar flow rate of distillate, kmol/s
H_w = Enthalpy of residue, kJ/kmol
$\dot{n}_W$ = Molar flow rate of residue, kmol/s
H_F = Enthalpy of feed, kJ/kmol
$\dot{n}_F$ = Molar flow rate of feed, kmol/s
ϕ_C = Heat duty of condenser, kW
ϕ_L = Heat loss of distillation system, kW

If the vaporization rate of reboiler is given then heat duty can be determined by the following equation:

$$\phi_B = \dot{m}_v \lambda \times 1.05 \text{ kW} \quad \text{(considering 5\% heat loss to surrounding)} \tag{6.79a}$$

$\dot{m}_v$ = Vaporization rate, kg/s

2. Fix the value of mean temperature difference $\Delta\mathcal{T}_m$. It should be less than and close to critical temperature drop.
   Average temperature of heating medium $t_h = \Delta\mathcal{T}_m + t_B$ (6.80)
   where, t_B = boiling temperature of liquid, °C
3. Select the suitable heating medium (Sec. 6.6.2).
4. Based on energy balance, find the mass flow rate of heating medium. If the temperature of heating medium required is less than 180°C, then saturated steam can be selected as heating medium. In this case,

$$\phi_B = \dot{m}_s \lambda_s \tag{6.81}$$

   where, λ_s = Latent heat of vaporization of steam, kJ/kg (to be obtained from Steam Tables)
   $\dot{m}_s$ = Mass flow rate of steam required, kg/s
5. To get the first estimate of heat transfer area, assume the value of overall heat transfer coefficient (Table 6.7). Find the heat transfer area based on the assumed value of U_o.

$$A = \frac{\phi_B \times 10^3}{U_o \Delta\mathcal{T}_m} = A_{\text{provided}} = N_t \, \pi \, d_o \, L \tag{6.16 and 6.17}$$

6. Fix the value of tube outside diameter d_o and tube length. Find the number of tubes N_t. Decide the tube arrangement. In reboiler, normally 1 in (25.4 mm OD) tubes are used when tube side fluid is saturated steam. Square pitch arrangement with U-tube-bundle is preferred. In kettle-type reboiler, higher value of tube pitch ($P_t = 1.5d_o$ to $2d_o$) is preferred to avoid the "vapour blanketting" of heat transfer surface inside the pool of liquid. With kettle-type reboiler full baffles (% baffle cut = 0) are used as extra space is available for the flow of liquid. Nucleate boiling coefficient or any phase change coefficient does not depend on type of baffle or baffle spacing. Here, baffles are provided only to reduce the vibration in tubes. Here, baffle spacing can be kept equal to tube bundle diameter or even higher than that but within permissible limit, specified in TEMA standard.
7. Calculate shell side boiling coefficient by Mostinski equation.

$$h_o = h_{nB} = 0.104\, p_c^{0.69}\left(\frac{\phi_B \times 10^3}{A}\right)^{0.7}\left[1.8\left(\frac{p}{p_c}\right)^{0.17} + 4\left(\frac{p}{p_c}\right)^{1.2} + 10\left(\frac{p}{p_c}\right)^{10}\right] \tag{6.82}$$

where, $h_o = h_{nB}$ = Shell side nucleate boiling heat transfer coefficient in W/(m^2 · °C)

$\frac{\phi_B \times 10^3}{A}$ = Heat flux of reboiler, W/m^2

p_c = Critical pressure of component or psuedo-critical pressure of the mixture of components vaporized, bar

p = Operating pressure in reboiler, bar

This equation is reliable for single component boiling. For, a mixture, actual coefficient is lesser than the same predicted by this equation.

8. Calculate tube side heat transfer coefficient, h_i:
   If saturated steam is used as heating medium, then calculation for h_i is not required. Value of h_i = 6000 W/(m^2·°C) can be safely used. This value also includes fouling resistance.

   or $$\frac{1}{h_1} = \frac{1}{h_i'} + \frac{1}{h_{id}} \tag{6.83}$$

   If hot oil is used as heating medium, then h'_i is calculated by forced convection correlation; Eq. (6.18) or (6.19), depending on the value of Reynolds number.
9. Calculate the overall heat transfer coefficient U_o, by Eq. (6.46).
10. Calculate the heat transfer area required using Eq. (6.47).
11. Calculate % excess heat transfer area. It should be in between 10 to 20%. If not, then change the number of tubes or tube length. Repeat the calculations until % excess area is in the desired range.
12. Based on the final or last value of heat transfer area provided, calculate the actual heat flux.

    $$\text{Actual heat flux, } \phi_a = \frac{\phi_B \times 10^3}{A_{\text{provided}}} \text{ W/m}^2 \tag{6.84}$$

13. Actual heat flux, $\phi_a \le 0.7\,(\phi_B \times 10^3/A)_c$
    where $(\phi_B \times 10^3/A)_c$ is the critical heat flux. Critical heat flux is the heat flux corresponding to critical temperature drop. This condition must be satisfied. Critical heat flux can be estimated by modified Zuber's equation[3] which can be written as

$$\left(\frac{\phi_B \times 10^3}{A}\right)_c = K_b\left(\frac{P_t}{d_o}\right)\left(\frac{\lambda}{\sqrt{N_t}}\right)(\sigma g(\rho_L - \rho_V)\rho_V^2)^{0.25} \text{ W/m}^2 \tag{6.85}$$

K_b = 0.44 for square pitch arrangement
0.41 for triangular pitch arrangement
P_t = Tube pitch, mm
d_o = Tube outside diameter, mm
N_t = Total number of tubes in bundle
λ = Latent heat of vaporization, J/kg
ρ_v = Vapour density, kg/m^3
σ = Liquid surface tension, N/m
ρ_L = Liquid density, kg/m^3
g = Acceleration due to gravity = 9.81 m/s^2

14. Find the shell inside diameter. Shell inside diameter of kettle-type reboiler should be greater of following two values.
    (a) Shell ID = Liquid level + 0.15 to 0.25 m
    Liquid level in kettle-type reboiler = Tube bundle diameter (D_b) + 50 to 100 mm
    To find the tube bundle diameter for U-tube bundle, Eq. (6.2) and for tube bundle with floating head, Eq. (6.1), can be used.
    (b) Minimum Shell ID can be obtained from the following table:

Table 6.26 Minimum Shell Diameter of Kettle-type Reboiler

Heat flux, W/m^2	Shell ID/Tube bundle diameter (D_i/D_b)
< 25 000	1.2 to 1.5
25 000 to 40 000	1.4 to 1.8
> 40 000	1.7 to 2.0

15. Check the liquid entrainment. To avoid the excessive entrainment, vapour velocity at liquid surface should be less than v_{max}.

$$v_{max} = 0.2\left(\frac{\rho_L - \rho_v}{\rho_v}\right)^{1/2} \tag{6.86}$$

where, v_{max} = Maximum permissible vapour velocity at liquid surface, m/s
Actual vapour velocity at liquid surface.

$$v = \frac{\dot{m}_v/\rho_v}{\text{Liquid surface area}} \tag{6.87}$$

$$\text{Liquid surface area} \cong L \times W \tag{6.88}$$

where, L = Tube length, m
W = width of liquid level, m

Boiling of Liquid Mixtures For the close boiling liquid mixture (boiling range is less than 5°C), Eq. (6.82) can be used to find the nucleate boiling coefficient. If for the given liquid mixture boiling range is wide then, Palen and Small equation can be used.

$$(h_{nb})_{\text{mixture}} = f_m \times (h_{nb})_{\text{single component}} \tag{6.89}$$

where, $$f_m = \exp\left[-0.0083\,(t_{bo} - t_{bi})\right] \tag{6.90}$$

t_{bo} = Temperature of vapour mixture leaving the reboiler, °C
t_{bi} = Temperature of liquid entering the reboiler, °C

Kettle-type reboiler can be considered as one equilibrium stage. Hence, the vapour leaving the reboiler is in equilibrium with the residue. Dew point temperature of vapour leaving the reboiler (t_{bo}) is equal to bubble point temperature (t_{bp}) of residue. To find t_{bi}, composition of liquid entering to reboiler must be determined. Material balance around reboiler gives composition of liquid (x_{1i}) entering to reboiler.

$$\bar{L} = \dot{n}_L = \dot{n}_V + \dot{n}_W \tag{6.91}$$

$$\bar{L}x_{1i} = \dot{n}_L x_{1i} = \dot{n}_V \cdot y_i + \dot{n}_W \cdot x_i \tag{6.92}$$

where, $\dot{n}_W$ = Molar flow rate of residue, kmol/s
x_i = Mole fraction of component i in residue
$y_i = K_i\, x_i$ = Mole fraction of component i in vapour leaving the reboiler
x_{1i} = Mole fraction of component i in liquid entering to reboiler.
$\bar{V} = \dot{n}_V$ = Molar flow rate of vapour leaving the reboiler, kmol/s
$\bar{L} = \dot{n}_L$ = Molar flow rate of liquid entering the reboiler, kmol/s

Bubble point of liquid entering to reboiler, gives the value of t_{bi}.

Example 6.7 A packed distillation tower with overhead condenser and kettle-type reboiler is used for aromatics separation. Design conditions of the column are as follows.

Table 6.27 Composition of Distillation Column Streams

Component	Composition, mole %		
	Feed	Distillate	Bottoms
Benzene	2.2	22.8	0
Toulene	7.4	72.2	0.5
Ethyl benzene	43.4	5.0	47.5
Styrene	47.0	0	52.0

Feed flow rate to distillation tower is 100 kmol/h.

A reflux ratio of 6.0 is selected for the design. Operating pressure at the base of column is fixed at 21.3 kPa a (160 Torr). All the streams may be taken as saturated liquids. Also assume that the mixtures are ideal mixtures. Saturated steam at 0.2 MPa a pressure is used as heating medium in kettle-type reboiler.

Design the kettle-type reboiler for this distillation column.

Solution

Heat duty of kettle-type reboiler

$$\phi_B = \phi_C + H_D\dot{n}_D + H_W\dot{n}_W - H_F\dot{n}_F + \phi_L \tag{6.79}$$

To calculate the heat duty of reboiler ϕ_B by using above equation flow rates ($\dot{n}_F$, $\dot{n}_D$ and $\dot{n}_W$), latent heats at bubble point temperatures, specific heats of liquid streams, bubble point temperatures of streams, dew point temperature of overhead vapour, etc., must be determined.

$\dot{n}_F$ = Feed flow rate = 100 kmol/h

$\dot{n}_F = \dot{n}_D + \dot{n}_W = 100$

Benzene balance:

$0.228\ \dot{n}_D = 2.2$

or $\dot{n}_D = 9.65$ kmol/h

$\dot{n}_W = 100 - 9.65 = 90.35$ kmol/h

ϕ_C = Heat duty of condenser = $(R + 1)\ \dot{n}_D\lambda$

$R = 6$

Latent heat of vaporization must be determined at the average temperature of the dew point and bubble point of distillate product. Assuming negligible pressure drop in the tower, operating pressure is constant throughout the distillation column and is equal to operating pressure at the base of column for heat duty calculation, i.e., at 21.3 kPa a.

Assumption of constant pressure throughout the column is not true in real case but for simplicity this assumption is made. In actual case, top pressure is to be found by distillation calculations and dew point at the real pressure at the top is to be calculated.

Bubble point of Distillate:

For ideal vapour liquid equilibrium,

at bubble point $\Sigma x_i p_v = p_t$ and $z_i = x_i$.

Vapour Pressure Data:

Table 6.28 Antoine Constants[10]

Component	*A*	*B*	*C*
Benzene	6.0306	1211.0	–52.35
Toluene	6.0795	1344.8	–53.65
Ethyl benzene	6.0821	1424.3	–59.95
Styrene	6.1911	1507.4	–58.15

Antoine equation $\log_{10} p_v = A - \dfrac{B}{T + C}$ (2.1)

where, p_v is in kPa and T is in K.

At bubble point of distillate,

$0.228\ p_{vB} + 0.722\ p_{vT} + 0.05\ p_{vEB} = p_t$

Trial for $T = 328.3$ K

$p_{vB} = 43.865$ kPa, $p_{vT} = 15.244$ kPa and $p_{vEB} = 5.95$ kPa

$0.228 \times 43.865 + 0.722 \times 15.244 + 0.05 \times 5.95 = 21.3$ kPa $= p_t$

Bubble point of distillate, $T_{BP} = 328.3$ K (55.3°C)

At dew point of distillate

$$\sum \frac{y_i p_t}{p_v} = 1 \quad \text{and} \quad z_i = y_i$$

Try $T = 334.7$ K

$p_{vB} = 55.1565$ kPa, $p_{vT} = 19.7054$ kPa, $p_{vEB} = 7.909$ kPa

$$\left(\frac{0.228}{55.1565} + \frac{0.722}{19.7054} + \frac{0.05}{7.909}\right) \times 21.3 = 1.003 \quad \text{(Check)}$$

Dew point temperature of distillate, $T_{DP} = 334.7$ K (61.7°C)
Latent heat of vaporization must be determined at average temperature.

$$T_{\text{av}} = \frac{328.3 + 334.7}{2} = 331.5 \text{ K}$$

Table 6.29 Properties of Components of Distillate

Component	y_i	Critical temperature T_c, K	T_1, K	λ at T_1, kJ/kmol
Benzene	0.228	562.16	353.3	30 761
Toluene	0.722	591.79	383.7	33 179
Ethyl benzene	0.05	617.2	409.3	35 564

$$\frac{\lambda_2}{\lambda_1} = \left[\frac{T_C - T}{T_C - T_1}\right]^{0.38} \quad \text{(Watson equation)}$$

For Benzene

$$\frac{\lambda_B}{30\,761} = \left[\frac{562.16 - 331.5}{562.16 - 353.3}\right]^{0.38}$$

$$\lambda_B = 31\,943.68 \text{ kJ/kmol at } 331.5 \text{ K}$$

Similarly,

$$\lambda_T = 36\,124.5 \text{ kJ/kmol at } 331.5 \text{ K}$$
$$\lambda_{EB} = 40\,130.2 \text{ kJ/kmol at } 331.5 \text{ K}$$
$$\lambda_{\text{av}} = \Sigma\lambda_{vi}\, y_i$$
$$= 0.228 \times 31\,943.68 + 0.722 \times 36\,124.5 + 0.05 \times 40\,130.2$$
$$= 35\,371.56 \text{ kJ/kmol}$$

Heat duty of condensation

$$\phi_C = (R + 1)\dot{n}_D\, \lambda_{\text{av}}$$
$$\phi_C = (6 + 1) \times 9.65 \times 35\,371.56 = 238\,9348.9 \text{ kJ/h} \equiv 663.7 \text{ kW}$$

Let 331.5 K be the reference temperature for enthalpy calculations, i.e., $T_0 = 331.5$ K

$$H_D = 0, \quad H_D\dot{n}_D = \phi_D = 0$$

Residue is bottom product of distillation and is a saturated liquid or at its bubble point. To find the enthalpy of residue, first the bubble point of residue must be determined.

At bubble point of liquid: $\Sigma x_i p_v = p_t$ and $z_i = x_i$

Trial: At $T = 363.55$ K (90.55°C), $\sum \frac{x_i p_v}{p_t} = \Sigma y_i = 1$

Vapour pressures: $p_{vT} = 54.959$ kPa, $p_{vEB} = 24.588$ kPa

$p_{vS} = 18$ Pka, $p_t = 21.3$ kPa

Table 6.30 Bubble Point Calculations for Residue

Component	x_i	p_{vi}, kPa	$y_i = \frac{x_i p_{vi}}{p_t}$
Toluene	0.005	54.959	0.013
Ethyl benzene	0.475	24.588	0.548
Styrene	0.52	18	0.439
Total			$\Sigma y_i = 1$ (check)

Bubble point of residue $T_{bp} = 363.55$ K (90.55°C)

Empirical heat capacity equation for liquids

$$C_{ml} = a + bT + cT^2 + dT^3$$

where, C_{ml} is in kJ / (kmol · K) and T is in K.

Table 6.31 Heat Capacity Equation Constants[10]

Component	a	$b \times 10^3$	$c \times 10^6$	$d \times 10^9$
Toluene	1.8083	812.223	–1512.67	1630.01
Ethyl benzene	4.3143	900.174	–1450.05	1433.6
Styrene	–38.019	1197.21	–2195.65	1933.12
Benzene	–7.2733	770.541	–1648.18	1897.94

These heat capacity equation constants are used for calculation of heat-flow rates of various streams.

Table 6.32 Heat Capacity Equation Constants for Residue

Component	x_i	$a_i \cdot x_i$	$b_i \cdot x_i \times 10^3$	$c_i \cdot x_i \times 10^6$	$d_i \cdot x_i \times 10^9$
Toluene	0.005	0.009	4.061	–7.563	8.15
Ethyl benzene	0.475	2.049	427.583	–688.774	680.96
Styrene	0.52	–19.77	622.549	–1141.738	1005.222
Total	1.00	–17.712	1054.193	–1838.075	1694.332

Enthalpy of residue,

$$H_w = \Sigma a_i x_i (T_{bp} - T_0) + \Sigma b_i x_i \times 10^{-3} \left(\frac{T_{bp}^2 - T_o^2}{2}\right) + \Sigma c_i x_i \times 10^{-6} \left(\frac{T_{bp}^3 - T_o^3}{3}\right) + \Sigma d_i x_i \times 10^{-9} \left(\frac{T_{bp}^4 - T_o^4}{4}\right)$$

$$H_w = -17.712(363.55 - 331.5) + 1054.193 \times 10^{-3} \left(\frac{363.55^2 - 331.5^2}{2}\right)$$

$$- 1838.075 \times 10^{-6} \times \left(\frac{363.55^3 - 331.5^3}{3}\right)$$

$$+ 1694.332 \times 10^{-9} \times \left(\frac{363.55^4 - 331.5^4}{4}\right)$$

$H_w = 6338.32$ kJ/kmol

$\phi_W = H_W W = 6338.32 \times 90.35 = 572\,667.2$ kJ/h $\equiv 159.074$ kW

$\phi_F = H_F F$:

Table 6.33 Heat Capacity Equation Constants for Feed

Component	x_i	$a_i \cdot x_i$	$b_i \cdot x_i \times 10^3$	$c_i \cdot x_i \times 10^6$	$d_i \cdot x_i \times 10^9$
Benzene	0.022	–0.16	16.952	–36.26	41.755
Toluene	0.074	0.134	60.105	–111.93	120.621
Ethyl benzene	0.434	1.872	390.676	–629.322	622.182
Styrene	0.47	–17.869	562.689	–1031.956	908.566
Total	1.00	–16.023	1030.422	–1809.476	1693.124

At bubble point $\sum \frac{x_i p_{vi}}{p_t} = \Sigma y_i = 1$

Trial: At $T = 357.75$ K, $p_t = 21.3$ kPa

Table 6.34 Bubble Point Calculations

Component	x_i	p_{vi}, kPa	$y_i = \frac{x_i p_{vi}}{p_t}$
Benzene	0.022	116.227	0.12
Toluene	0.074	45.422	0.158
Ethyl benzene	0.434	19.923	0.406
Styrene	0.47	14.445	0.319
Total	1.00		1.003 (check)

Bubble point of feed, $T_{bp} = 357.75$ K (84.75 °C)

$$H_F = -16.023(357.75 - 331.5) + 1030.422 \times 10^{-3}\left(\frac{357.75^2 - 331.5^2}{2}\right)$$
$$- 1809.476 \times 10^{-6}\left(\frac{357.75^3 - 331.5^3}{3}\right)$$
$$+ 1693.124 \times 10^{-9}\left(\frac{357.75^4 - 331.5^4}{4}\right)$$
$$= 5079 \text{ kJ/kmol}$$

$$\phi_F = H_F \dot{n}_F = 507\,900 \text{ kJ/h} \equiv 141.08 \text{ kW}$$

Heat duty of kettle-type reboiler

$$\phi_B = \phi_C + \phi_D + \phi_W - \phi_F + \phi_L \tag{6.79}$$

$$\phi_B = 663.7 + 0 + 159.074 - 141.08 + \phi_L$$

$$\phi_B = 681.7 + \phi_L = 681.7 \times 1.05 = 716 \text{ kW (considering 5\% heat loss)}$$
$$\equiv 716 \times 10^3 \text{ W}$$

In continuous operation at steady state, boiling temperature of liquid in kettle-type reboiler = Bubble point temperature of residue = Dew point temperature of vapour leaving the reboiler = Steady state equilibrium temperature of kettle-type reboiler = T_{bp}.

$$T_{bp} = 363.55 \text{ K} \quad (90.55°\text{C})$$

Critical temperature drop of light organics is ranging from 20°C to 50°C. For the precise calculations, value of critical temperature drop and critical heat flux at the actual operating condition must be determined by experiment.

Temperature of heating medium $t_h = t_{BP} + \Delta\mathcal{T}_m$ (6.80)

$\Delta\mathcal{T}_m$ should be close to and less than $\Delta\mathcal{T}_c$.

Let $\Delta\mathcal{T}_m \cong 30°\text{C}$

t_h = Temperature of saturated steam = 393.38 K = 120.23°C

$\Delta\mathcal{T}_m = 393.38 - 363.55 = 29.83°\text{C}$ (acceptable)

Saturated steam pressure = 0.2 MPa

Latent heat, $\lambda_s = 2201.6$ kJ/kg (Ref: Steam Tables)

Steam required:

$$\dot{m}_s = \frac{\phi_B}{\lambda_s} = \frac{716}{2201.6} = 0.325\,218 \text{ kg/s} \equiv 1170 \text{ kg/h}$$

For the first trial calculations, assume $U_o = 1000$ W/(m^2 · °C)

Heat transfer area based on assumed value of U_o:

$$A_o = \frac{\phi_B}{U_o \Delta\mathcal{T}_m} = \frac{716 \times 10^3}{1000 \times 29.836} = 24 \text{ m}^2 = A_{o \text{ provided}}$$

$$A_{o \text{ provided}} = 24 \text{ m}^2 = N_t\, \pi\, d_o\, L$$

Nominal U-tube length = 3.6576 m (12 ft)

$d_o = 25.4$ mm, $d_i = 21.1836$ mm (14 BWG)

$$A_{o \text{ provided}} = 24 \text{ m}^2 = N_t\, \pi \times (0.0254) \times 3.6576$$

Number of tubes = 82 (U-tubes)

Number of tube holes in tube sheet = 82 × 2 = 164

Use square pitch arrangement.

Tube pitch $P_t = 1.5\, d_o = 38.1$ mm

Minimum U bend radius = 1.5 d_o = 38.1 mm

$h_o = h_{nbo}$ = Nucleate boiling heat transfer coefficient for mixture = $f_m \cdot h_{nb}$ (6.89)

where, h_{nb} can be determined by Mostinski equation[2]

$$h_{nb} = 0.104\, p_c^{0.69} \left(\frac{\phi_B \times 10^3}{A}\right)^{0.7} \left[1.8\left(\frac{p}{p_c}\right)^{0.17} + 4\left(\frac{p}{p_c}\right)^{1.2} + 10\left(\frac{p}{p_c}\right)^{10}\right] \quad (6.82)$$

$$\frac{\phi_B}{A} = \frac{716 \times 10^3}{24} = 29\,833.33 \text{ W/m}^2$$

$p = 21.3$ kPa = 0.213 bar

No reliable method is available for estimating critical pressure (p_{cm}) of a mixture.

$p_{cm} \cong \Sigma\, y_i p_{ci}$ (Kay's rule)

f_m = correction factor

Table 6.35 Composition in Reboiler

Component	Residue composition		Composition of vapour		Composition of liquid entering reboiler	
	x_i	$\dot{n}_W x_i$, kmol/h	$y_i = p_{vi} x_i / p_t$	$\dot{n}_V y_i$, kmol/h	$\dot{n}_L x_i$, kmol/h	x_{1i}
Toluene	0.005	0.45175	0.013	0.856	1.30775	0.0084
Ethyl benzene	0.475	42.9163	0.548	36.07	78.9863	0.5058
Styrene	0.52	46.982	0.439	28.895	75.877	0.4859
Total	1.00	90.35	1.00	65.82	156.17	1.000

Total material balance around reboiler

$$\bar{L} = \dot{n}_L = \dot{n}_V + \dot{n}_W$$

and component balance: $\dot{n}_L x_{1i} = \dot{n}_W \cdot x_i + \dot{n}_V \cdot y_i$

where, correction factor

$$f_m = \exp[-0.0083(t_{bo} - t_{bi})] \tag{6.90}$$

where, t_{bo} = Temperature of vapour mixture leaving the reboiler = 90.4°C

t_{DP} = Dew point temperature of vapour leaving the reboiler, °C

or t_{BP} = Bubble point temperature of residue, °C

t_{bi} = Temperature of liquid entering to the reboiler = Bubble point temperature of liquid entering the reboiler.

At t_{bi}

$$\Sigma \frac{x_{1i} p_{vi}}{p_t} = 1$$

To find $\bar{L}(= \dot{n}_L)$ and x_{1i} by Eq. (6.87) and (6.88), value of $\bar{V}$ is required.

$$\bar{V} = \dot{n}_V = \frac{\phi_B}{\lambda_{av}}$$

Table 6.36 Properties of Residue Components

Component	x_i	Critical temperature T_c, K	T_1, K	λ_i, kJ/kmol at T_1 K
Toluene	0.005	591.79	383.7	33 179
Ethyl benzene	0.475	617.2	409.3	35 564
Styrene	0.52	647	418.2	36 819
Total	1.00			

Watson equation

$$\frac{\lambda}{\lambda_1} = \left[\frac{T_c - T}{T_c - T_1}\right]^{0.38}$$

To be precise, λ must be determined at average temperature of T_{bo} and T_{bi} and also for average composition of liquid. Here, λ_{av} is determined at T_{bo} and for the residue composition as there is a negligible change in temperature and composition.

At $T = T_{bo} = 363.55$ K

Using Watson equation

$$\lambda_T = 34\,365 \text{ kJ/kmol}, \quad \lambda_{EB} = 38\,356 \text{ kJ/kmol}, \quad \lambda_S = 39\,941 \text{ kJ/kmol}$$

$$\lambda_{\text{av}} \cong 0.005 \times 34\,365 + 0.475 \times 38\,356 + 0.52 \times 39\,941$$

$$= 39\,160 \text{ kJ/kmol}$$

$$\bar{V} = \dot{n}_V = \frac{716}{39\,160} \times \frac{3600}{1} = 65.82 \text{ kmol/h}$$

$$\bar{L} = \dot{n}_V = \dot{n}_L + \dot{n}_W = 65.82 + 90.35 = 156.17 \text{ kmol/h}$$

Values of x_{1i} are calculated by using Eqs (6.91) and (6.92) and are given in Table 6.35.

At $T_{bi} = 363.1$ K (Trial)

$$p_{vT} = 54.1658 \text{ kPa}, \quad p_{vEB} = 24.1972 \text{ kPa}, \quad p_{vS} = 17.7 \text{ kPa}$$

$$p_t = 21.3 \text{ kPa}$$

$$\sum \frac{x_{1i} p_{vi}}{p_t} = \frac{0.0084 \times 54.1658}{21.3} + \frac{0.5058 \times 24.1972}{21.3} + \frac{0.4859 \times 17}{21.3}$$

$$= 1 \quad \text{(check)}$$

$$T_{bi} = 363.1 \text{ K}$$

Correction factor $f_m = \exp[-0.0083(363.55 - 363.1)]$

$$f_m = 0.996\,27$$

$$h_o = h_{nb\text{mix}} = f_m \cdot h_{nb} = 0.996\,27 \times h_{nb}$$

$$p_{\text{cm}} = \Sigma p_{ci} y_i \quad \text{(Kay's rule)}$$

Table 6.37 Critical Pressure Data[10]

Component	p_c, bar	y_i
Toluene	41.1	0.013
Ethyl benzene	36.1	0.548
Styrene	39.9	0.439
Total		1.000

$$p_{cm} = 0.013 \times 41.1 + 0.548 \times 36.1 + 0.439 \times 39.9$$

$$= 37.83 \text{ bar},$$

$$\frac{p}{p_{cm}} = \frac{0.213}{37.83} = 0.005\,63 = p_{rm}$$

$$h_{nb} = 0.104 \times (37.83)^{0.69} \times (29\,833.33)^{0.7} \left[1.8 p_{rm}^{0.17} + 4 p_{rm}^{1.2} + 10 p_{rm}^{10}\right]$$

$$= 1304.72 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$h_o = h_{n_b\text{mix}} = 0.996\,27 \times 1304.72 = 1299.85 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

For the condensation of saturated steam consider

$$h_i' = 6000 \text{ W/(m}^2 \cdot \text{°C)}$$

This value also includes fouling resistance.
Shell side fouling coefficient, $h_{od} = 5000$ W/(m^2 · °C)
Thermal conductivity of steel tube material, $k_w = 50$ W/(m · °C)
Using Eq. (6.46):

$$\frac{1}{U_o} = \frac{1}{1299.85} + \frac{1}{5000} + \frac{0.0254 \ln\left(\frac{25.4}{21.1836}\right)}{2 \times 50} + \frac{25.4}{21.1836} \times \frac{1}{6000}$$

$$U_o = 822.86 \text{ W/(m}^2 \cdot \text{°C)}$$

Heat transfer area required

$$A_{o\,\text{req}} = \frac{\phi_B}{U_o \Delta T_m} = \frac{716 \times 10^3}{822.86 \times 29.83} = 29.17 \text{ m}^2$$

Heat transfer area provided, $A_{o\text{pro}} = 24$ m^2

$A_{o\,\text{req}} > A_{o\text{pro}}$ (not acceptable)

Increase in heat transfer area will decrease the value of h_{nb} and the value of U_o. Value of overall coefficient removed [($U_o = 822.86$ W/(m$^{2\cdot}$ °C)] is within normal range (See Table 6.7). Further decrease in the value of U_o is not required. To decrease the value of $A_{o\text{req}}$ increase the pressure and temperature of saturated steam.
Let saturation temperature = 404.35 K

Pressure = 0.28 MPa

$$\lambda_s = 2170.1 \text{ kJ/kg}$$

Steam required $\dot{m}_s = \dfrac{\phi_B}{\lambda_s}$

$$= \frac{716}{2170.1} = 0.33 \text{ kg/s} \equiv 1188 \text{ kg/h}$$

$$\Delta T_m = 404.35 - 363.55 = 40.8\text{°C}$$

$$A_{o\text{req}} = \frac{\phi_B}{U_o \Delta T_m} = \frac{716 \times 10^3}{822.86 \times 40.8} = 21.327 \text{ m}^2$$

Excess heat transfer area $= \left(\dfrac{24}{21.327} - 1\right) \times 100$

$= 12.533\%$ (satisfactory)

Actual heat flux, $\dfrac{\phi_a \times 10^3}{A} = \dfrac{\phi_B \times 10^3}{A_{o\text{pro}}} = 29\,833.33$ W/m^2

To verify condition of $\phi_a \leq 0.7\ (\phi_B)_c$, critical heat flux is determined by modified Zuber's equation.

$$\left(\frac{\phi_B \times 10^3}{A}\right)_c = K_b\left(\frac{P_t}{d_o}\right)\left(\frac{\lambda}{\sqrt{N_t}}\right)(\sigma g(\rho_L - \rho_v)\rho_v^2)^{0.25} \tag{6.85}$$

$K_b = 0.44, \quad P_t = 38.1$ mm, $\quad d_o = 25.4$ mm

$N_t = 82 \times 2 = 164, \quad g = 9.81$ m/s^2

$\lambda =$ Latent heat of vaporization, J/kg

$\lambda_s = 39\ 160$ kJ/kmol

Average molar mass of residue

$$M_{av} = \Sigma M_i x_i = 0.005 \times M_T + 0.475 \times M_{EB} + 0.52 \times M_S$$

$$M_{av} = 0.005 \times 92.141 + 0.475 \times 106.168 + 0.52 \times 104.152 = 105.05 \text{ kg/kmol}$$

$$\lambda = \frac{39\ 160}{105.05} \times 10^3$$

$$= 372\ 774.9 \text{ J/kg}$$

$$\rho_v = \frac{pM_{av}}{RT} = \frac{pM_{av}}{T} \times \frac{T_s}{p_s V_s} \quad (s = \text{standard conditions})$$

Average molar mass of vapour

$$M_{av} = \Sigma M_i y_i = 0.013 \times M_T + 0.548 \times M_{EB} + 0.439 \times M_S$$

$$M_{av} = 0.013 \times 92.141 + 0.548 \times 106.168 + 0.439 \times 104.152 = 105.1 \text{ kg/kmol}$$

$$\rho_v = \frac{21.3 \times 105.1}{363.55} \times \frac{273}{101.325 \times 22.414}$$

$$\rho_v = 0.74 \text{ kg/m}^3$$

$$\rho_L = \text{Density of liquid mixture} = \frac{1}{\Sigma \dfrac{w_i}{\rho_{Li}}}$$

For Residue:

Table 6.38 Properties of Residue Components

Component	mole fraction x_i	w_i (mass fraction) $w_i = \dfrac{x_i M_i}{\Sigma x_i M_i}$	Liquid density, ρ_{Li} kg/m^3
Toluene	0.005	0.0044	867
Ethyl benzene	0.475	0.48	867
Styrene	0.52	0.5156	906
Total	1.00	1.00	

$$\rho_L = \frac{1}{\left(\dfrac{0.0044}{867} + \dfrac{0.48}{867} + \dfrac{0.5156}{906}\right)}$$

$$= 886.68 \text{ kg/m}^3$$

σ = surface tension of liquid mixture = σ_m

Surface tension of liquid mixture can be estimated by the following equation: (Eq. (3.152) of Ref. 2)

$$\sigma_m^{1/4} = \Sigma[\mathcal{P}_i](\rho_{Lm}x_i - \rho_{Vm}y_i) \tag{6.93}$$

where, σ_m = Surface tension of liquid mixture, dyn/cm

$[\mathcal{P}_i]$ = Parachor of component i to be obtained from Table 3.343 of Ref. 2

ρ_{Lm} = Liquid mixture density, mol/cm^3

ρ_{Vm} = Vapour mixture density, mol/cm^3

$[\mathcal{P}_i]$ for toluene = 189.6 (For C_6H_5 – group) + 55.5 (For—CH_3 group).

$$[\mathcal{P}_{\text{toluene}}] = 245.1$$

$$[\mathcal{P}_{EB}] = 189.6 + 55.5 + 40 \text{ (For —CH}_2\text{— group)}$$
$$= 285.1$$

$$[\mathcal{P}_{\text{styrene}}] = 189.6 + (15.5 + 9) + 40 = 254.1$$

$$\rho_{Lm} = 886.68 \text{ kg/m}^3 \equiv \frac{886.68}{105.05} \equiv 8.44 \frac{\text{kmol}}{\text{m}^3}$$
$$\equiv 8.44 \times 10^{-3} \text{ mol/cm}^3$$

$$\rho_{Vm} = 0.74 \text{ kg/m}^3 \equiv \frac{0.74}{105.1} \times 10^{-3} \equiv 7.041 \times 10^{-6} \text{ mol/cm}^3$$

$$\sigma^{1/4} = \sigma_m^{1/4} = 245.1(8.44 \times 10^{-3} \times 0.005 - 7.041 \times 10^{-6} \times 0.013)$$
$$+ 285.1(8.44 \times 10^{-3} \times 0.475 - 7.041 \times 10^{-6} \times 0.548)$$
$$+ 254.1(8.44 \times 10^{-3} \times 0.52 - 7.041 \times 10^{-6} \times 0.439)$$
$$= 2.2666$$

$$\sigma = 26.3936 \text{ dyn/cm}$$
$$\equiv 26.3936 \times 10^{-3} \text{ N/m}$$

$$\left(\frac{\phi_B \times 10^3}{A}\right)_c = 0.44 \times \left(\frac{38.1}{25.4}\right) \times \frac{372\,774.9}{\sqrt{164}} \times (26.3936 \times 10^{-3} \times 9.81 \times (886.68 - 0.74) \times 0.74^2)^{0.25}$$
$$= 64\,317 \text{ W/m}^2$$

$$0.7\left(\frac{\phi_B \times 10^3}{A}\right)_c = 45\,022 \text{ W/m}^2$$

Thus $\phi_a \leq 0.7\,(\phi_B)_c$

To find the tube bundle diameter for square pitch arrangement with U-tube bundle, following equation can be written:

$$\frac{\pi}{4}D_b = N_t P_t^2 + \text{Area of central portion}$$

$$\frac{\pi}{4}D_b = N_t P_t^2 + D_b \times P_t$$

(for P_t = 38.1 mm (1.5 in) = minimum U-tube radius)

$$\frac{\pi}{4}D_b - D_b P_t - N_t P_t^2 = 0$$

$$D_b = \frac{P_t \pm \sqrt{P_t^2 + 4(\pi/4)N_t P_t^2}}{2\left(\frac{\pi}{4}\right)}$$

$$= \frac{P_t + \sqrt{P_t^2 + \pi N_t P_t^2}}{(\pi/2)}$$

Minor correction in this equation:

$$D_b = \frac{P_t + \sqrt{P_t^2 + \pi N_t P_t^2}}{\pi/2} + d_o$$

$$= \frac{38.1 + \sqrt{38.1^2 + \pi \times 164 \times 38.1^2}}{\pi/2} + 25.4 \text{ (82 U-tubes} = 2 \times 82 = 164 \text{ tubes)}$$

$$= 600.74 \cong 600 \text{ mm}$$

Ideally tube bundle diameter must be determined based on actual tube sheet layout as shown in Fig. 6.31.

Fig. 6.31 *Tube Sheet Layout Drawing for D_b = 600 mm, N_t = 162*

Let the height of weir which maintains the liquid level in kettle-type reboiler be 700 mm.

Shell ID, D_s:

$D_s = 700 + 250 = 950$ mm

(where 250 mm is the vertical distance between shell ID and liquid level.)

or $D_s = 1.6\, D_b$ (From Table 6.26)

Hence, $D_s = 960$ mm

Based on Tube sheet layout drawing for $D_b = 600$ mm, $N_t = 162$

Let shell ID, $D_s = 960$ mm (greater of the two values)

Checking of liquid entrainment

To avoid the excessive liquid entrainment

$$v < v_{max}$$

where, v = Actual vapour velocity at liquid surface, m/s

v_{max} = Maximum permissible vapour velocity at liquid surface, m/s

$$v = \frac{(\dot{m}_v/\rho_v)}{L'W}$$

where, $L'W$ = Liquid surface area

Fig. 6.32 *Checking of Liquid Entrainment*

$$L' \approx \text{Tube length} = \frac{3.6576}{2} \quad \text{(U-tube)}$$

$$L' = 1.8288 \text{ m}$$

From geometry, width of liquid surface,

$$\text{Liquid level} - \frac{960}{2} = 220$$

$$\left(\frac{W}{2}\right)^2 = \left(\frac{960}{2}\right)^2 - 220^2$$

$$W = 853.23 \text{ mm}$$

Liquid surface area, $L'W = 1.8288 \times 0.853$

$$= 1.5604 \text{ m}^2$$

$$v = \frac{(\dot{n}_V \times \text{Average molar mass of vapour})/\rho_v}{1.5604}$$

$$v = \frac{(65.82 \times 105.1)/0.74}{1.5604} \times \frac{1}{3600}$$

$$v = 1.664 \text{ m/s}$$

$$v_{\max} = 0.2\left(\frac{\rho_L - \rho_v}{\rho_v}\right)^{1/2} \tag{6.86}$$

$$v_{\max} = 0.2\left(\frac{886.68 - 0.74}{0.74}\right)^{1/2} = 6.92 \text{ m/s}$$

$v < v_{\max}$ Hence, there is no excessive liquid entrainment.

Shell side or boiling side pressure drop Δp_s in case of kettle-type reboiler is negligible.

Tube Side Pressure Drop Δp_t

On tube side, condensation of saturated steam is taking place.

$$\Delta p_t = 0.5\,\Delta p_t' = 0.5\,N_p\left(8\,J_f\frac{L}{d_i}\left(\frac{\mu}{\mu_w}\right)^{-m} + 2.5\right)\frac{\rho u_t^2}{2} \tag{6.32}$$

Here $N_p = 2, \quad L = \dfrac{3.6576}{2} = 1.8288$ m

$d_i = 0.021\ 1836$ m

ρ = Density of saturated steam at 0.28 MPa

$$= \frac{1}{\text{Specific volume of vapour}} = \frac{1}{0.646\ 04} = 1.548 \text{ kg/m}^3$$

Tube side mass velocity, $G_t = \dfrac{\dot{m}_s}{\left(\dfrac{N_t}{N_p}\right) \times \dfrac{\pi}{4} d_i^2}$

$$G_t = \frac{0.33}{\left(\dfrac{162}{2}\right) \times \dfrac{\pi}{4}(0.021\ 1836)^2} = 11.56 \text{ kg/(m}^2 \cdot \text{s)}$$

Tube side Reynolds number

$$Re = \frac{d_i G_t}{\mu} = \frac{0.021\ 1836 \times 11.56}{1350 \times 10^{-8}} = 18\ 139.44$$

Viscosity of saturated steam at 404.35 K

$\mu_v = 1350 \times 10^{-7}$ P $= 1350 \times 10^{-8}$ kg/(m · s))

$J_f = 0.0041$ (From Fig. 6.13)

$$u_t = \frac{G_t}{\rho} = \frac{11.56}{1.548} = 7.468 \text{ m/s}$$

$$\Delta p_t = 0.5 \times 2\left(8 \times 0.0041\left(\frac{1.8288}{0.021\ 1836}\right) \times 1 + 2.5\right)\frac{1.548 \times 7.468^2}{2}$$

$$= 230.15 \text{ Pa} \equiv 0.23 \text{ kPa (acceptable)}$$

6.13.2 Design of Vertical Thermosyphon Reboiler

As a vertical thermosyphon reboiler, BEM type fixed tube sheet heat exchanger is normally used. It is installed in the perfect vertical position. Figure 6.33 shows standard location of vertical thermosyphon reboiler in which normal level of liquid in distillation column is same as level of top tube sheet of heat exchanger. Inside the tubes, liquid is partially vaporized by using the heating medium like saturated steam or hot oil on shell side. It decreases the density of fluid (vapour–liquid mixture) in "hot" leg compared to the density of saturated liquid in relatively "cold" leg and starts natural fluid circulation in the anticlockwise direction as shown in Fig. 6.33 by arrows. In this type of reboiler, syphoning effect is created by heating (thermally), hence it is called thermosyphon reboiler.

HTRI and HTFS have developed their own methods for the process design of thermosyphon reboiler which are not available in open literature. The oldest and the simplest method for the process design of vertical thermosyphon reboiler is Kern's method which is outlined as follows.

Fig. 6.33 *Standard Arrangement of Vertical Thermosyphon Reboiler*

1. Calculate the heat duty of reboiler based on energy balance equation for distillation column using Eqs. (6.8) and (6.74)
2. Fix the value of mean temperature difference, $\Delta\mathcal{T}_m$. It should be less than and close to critical temperature drop. Then, average temperature of heating medium $t_h = \Delta\mathcal{T}_m + t_B$.
3. Select the suitable heating medium. Based on the energy balance, find the mass flow rate of heating medium required.
4. Assume the value of overall heat transfer coefficient, $U_o = 1000\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$. Find the heat transfer area based on assumed value of U_o.

$$A_o = \frac{\phi_B \times 10^3}{U_o \Delta\mathcal{T}_m} = A_{\text{pro}} = N_t \pi d_o L \qquad \text{(6.47 and 6.17)}$$

   Find or decide the required dimensions of the heat exchanger.
5. Assume the recirculation ratio equal to 4 for the first trial.

$$\text{Recirculation ratio} = \frac{\text{Mass flow rate of liquid recirculated}}{\text{Vaporization rate}}$$

$$= \frac{\dot{m}_{Lo}}{\dot{m}_{Vo}} \qquad (6.94)$$

   where, $\dot{m}_{Lo}$ = Mass flow rate of liquid at outlet of reboiler
   $\dot{m}_{Vo}$ = Mass flow rate of vapour at outlet of reboiler.
6. Carry out the pressure balance. At steady state,
   Available differential head = Pressure loss in the circuit
   or Δp_{av} = Pressure loss in the base of distillation column
   + Pressure loss in associated piping and nozzles
   + Pressure loss in reboiler (i.e., tube side pressure drop)
   Velocity of liquid through the shell of distillation column is very low compared to the velocity of liquid through pipes. Hence, pressure loss in the base of distillation column may be neglected. If piping arrangement is smooth and higher sizing for the pipes is provided, then pressure loss in piping may also be neglected. Hence, $\Delta p_{\text{av}} \cong \Delta p_t$, i.e., at steady state.

Δp_t = Tube side pressure drop (loss) of reboiler, Pa.

Available differential head, Δp_{av} is calculated by following equation:

$$\Delta p_{av} = L\,(\rho_L - \rho_{av})\,g \tag{6.95}$$

where, L = Length of tube, m

ρ_L = Density of liquid in cold leg, kg/m^3

ρ_{av} = Average density of liquid vapour mixture in hot leg, kg/m^3

g = Acceleration of gravity = 9.81 m/s^2

$$\rho_{av} = \frac{\ln(V_o/V_i)}{V_o - V_i} \quad \text{(Ref. 5)} \tag{6.96}$$

where, V_i = Specific volume of saturated liquid at the inlet of reboiler, m^3/kg

$V_i = 1/\rho_L$

V_o = Specific volume of liquid-vapour mixture at the outlet of reboiler, m^3/kg

$$V_o = \frac{(\dot{m}_{Vo}/\rho_v) + (\dot{m}_{Lo}/\rho_L)}{(\dot{m}_{Vo} + \dot{m}_{Lo})} \tag{6.97}$$

where, $\dot{m}_{Vo}$, $\dot{m}_{Lo}$ = Mass flow rates of vapour phase and liquid phase at the outlet of reboiler, kg/s

In Kern's method, it is suggested to find Δp_t by same equation that is used for no phase change in the tube side fluid [Eq. (6.31)].

In this equation, $\rho = \rho_{av}$ = average density of tube side fluid.

7. Compare the value of available differential head Δp_{av} and pressure drop in the system or tube side pressure drop, (Δp_t), obtained based on assumed value of recirculation ratio. There are total five possibilities:
   (a) $\Delta p_{av} \cong \Delta p_t$
   (b) $\Delta p_{av} > \Delta p_t$
   (c) $\Delta p_{av} >> \Delta p_t$
   (d) $\Delta p_{av} < \Delta p_t$
   (e) $\Delta p_{av} << \Delta p_t$

(a) $\Delta p_{av} \cong \Delta p_t$ implies that the assumed value of recirculation ratio (4 for first trial calculation) is equal to the actual value of recirculation ratio. Proceed for the next step of calculation.

(b) $\Delta p_{av} > \Delta p_t$, implies that the assumed value of recirculation ratio is less than the actual recirculation ratio. Hence, assume higher value of recirculation ratio, repeat the calculations and again compare the new values of Δp_{av} and Δp_t. By trial and error calculations, find the recirculation ratio for which $\Delta p_{av} \cong \Delta p_t$. Recirculation ratio can have a fractional value.

(c) $\Delta p_{av} >> \Delta p_t$ or $\Delta p_{av}/\Delta p_t > 50$ implies that the assumed value of recirculation ratio is very much less than actual recirculation ratio. In this case also, one can find the value of recirculation ratio for which $\Delta p_{av} \cong \Delta p_t$, by trial and error calculations but very high recirculation ratio and hence, very high tube side pressure drop may be obtained which cannot be permitted. Very high tube side pressure drop will result in significant boiling point elevation inside the tubes of the reboiler and decrease the mean temperature difference.

Very high tube side velocity gives erosion and vibrations in tubes. Hence, in such a case one of the following options can be considered:

(i) Provide the control valve with flow meter in the inlet line to the reboiler. Then, pressure balance is $\Delta p_{av} = \Delta p_t$ + Pressure loss in flow meter + Pressure drop offered by control valve.

Pressure drop of control valve can be adjusted to the desired value. This option also provides flexibility in the actual operation. In actual plant operation, recirculation ratio or circulation rate can be corrected depending on the actual performance. This facility is not available with other options.

(ii) Elevate the level of top tube sheet of reboiler above the liquid level at the base of distillation column as shown in Fig. 6.34.

Fig. 6.34 *Increase of Elevation of Reboiler*

In this option,

$$\Delta p_{av} = L' (\rho_L - \rho_{av})g - (L - L') \rho_{av} g \tag{6.95a}$$

Hence, this modification decreases the value of available differential head Δp_{av} and may equate it with Δp_t at the lower value or reasonable value of recirculation ratio.

(d) If $\Delta p_{av} < \Delta p_t$, then one of the following options can be considered:

(i) Increase the number of tubes and decrease the tube length. This modification will decrease the values of both Δp_{av} and Δp_t. But, the percentage decrease in the value of Δp_t will be more compared to the same in the value of Δp_{av}. Hence, it may be possible to equate Δp_{av} and Δp_t for the lower value of recirculation rate.

(ii) $\Delta p_{av} < \Delta p_t$, implies that the actual recirculation ratio is less than assumed or minimum recommended value of recirculation ratio. In this case, actual recirculation ratio can be increased by elevating liquid level at the base of the distillation column above the top tube sheet of reboiler as shown in Fig. 6.35.

In this case, equation of Δp_{av} is changed.

$$\Delta p_{av} = \rho_L g h + L (\rho_L - \rho_{av})g \tag{6.95b}$$

But this modification may increase the height of support of distillation column. If increase in the height of support is not possible, then one can go for underground construction. If none of these options is permitted, then

use a pump in the inlet line to the reboiler to increases the circulation rate. Then this reboiler is called a forced circulation reboiler.

Fig. 6.35 *Decrease of Elevation of Reboiler*

(e) $\Delta p_{av} << \Delta p_t$ implies that the actual recirculation ratio is very low or very much less than minimum recommended value, i.e., 4. This is possible when the viscosity of bottom product of distillation column is very high. In this case use a pump in the inlet line to the reboiler to get the desired value of circulation rate and desirable value of tube side heat transfer coefficient. But then this reboiler also becomes a forced circulation reboiler.

8. After finding the recirculation ratio for which available differential head becomes nearly equal to pressure loss in the system, calculate the tube side heat transfer coefficient. In the latest method, tube length is divided in different sections. For the different sections, different regimes of two phase flow are considered. Hence, for the different sections different values of heat transfer coefficients are calculated by different correlations (Ref. 12, 13 and 14). But in the simplified Kern's method, it is suggested to use the same forced convection correlation which are actually derived for the sensible heat transfer (cooling or heating) of tube side fluid. Hence, in Kern's method, h_i is determined by Eq. (6.18) or (6.19). In addition to that Kern has specified the maximum limit for the value of h_i as under.

   If $h_i \geq 300$ Btu/(h · ft^2 · °F) [= 1700 W/(m^2 · °C)],

   take $h_i = 1700$ W/(m^2 · °C)

9. Calculate the shell side or heating medium side heat transfer coefficient. If shell side heating medium is saturated steam then take

$$h_o' = 6000 \text{ W/(m}^2 \cdot \text{°C)}$$

   This figure also includes fouling resistance. If hot oil is used as heating medium, then use the Eq. (6.40) or Eq. (6.41) for the calculation of h_o.

10. Determine the overall heat transfer coefficient by Eq. (6.46).
11. Calculate the required heat transfer area by using Eq. (6.47). If ratio of provided HTA to required HTA is 10 to 20% design is acceptable. If it is not, then change the value of $A_{o\text{pro.}}$, either by increasing the tube length or by increasing number of

tubes. An increase in tube length will keep the tube side heat transfer coefficient unchanged but it will increase the value of Δp_t. Increase in number of tubes will decrease the value of h_i and also of h_o, If hot oil is used as heating medium it will decrease the value of Δp_t. In any case, both pressure balance calculations and heat transfer coefficient calculations must be repeated.

12. Find the shell side pressure drop (Δp_s). It should be less than the maximum allowable value.

Example 6.8 Design a vertical thermosyphon reboiler which must provide 10 000 kg/h of *n*-pentane vapour to a distillation tower at 10 bar a pressure.

Solution

Determination of boiling point of pentane:
Antoine equation[9] for *n*-pentane

$$\ln p_v = A - \frac{B}{(T + C)}, \text{ where, } p_v \text{ in Torr, } T \text{ in K} \tag{2.1}$$

$$\ln p_v = 15.8333 - \frac{2477.07}{T - 39.94}$$

$$p_v = p_t = 10 \text{ bar} = 7500.6 \text{ Torr}$$
$$T = 398.38 \text{ K} \equiv 125.4°\text{C}$$

Heat duty, $\phi = \dot{m}\lambda \times 1.05$ (5% heat loss)
where, λ = Latent heat of vaporization at the boiling point
Latent heat of vaporization of pentane at 125.4°C:
At 25°C, λ_{v1} = 366.512 kJ/kg
Critical temperature of pentane, t_c = 197°C, (Ch 3, of Ref. 2)
From Watson's equation,

$$\lambda_2 = \lambda_1 \left(\frac{1 - T_{r_2}}{1 - T_{r_1}} \right)^{0.38} = 366.512 \left(\frac{1 - \left(\frac{125.4 + 273}{197 + 273} \right)}{1 - \left(\frac{25 + 273}{197 + 273} \right)} \right)^{0.38}$$

$$= 366.512 \times 0.7167$$
$$= 262.68 \text{ kJ/kg}$$

Heat duty, $\phi = 1.05 \times 10\,000 \times 262.68$
$\phi = 2758\,140$ kJ/h $\equiv$ 766.15 kW $\equiv 766.15 \times 10^3$ W

Let $\Delta\mathcal{T}_m$ = 39.6°C. Assume that this value of $\Delta\mathcal{T}_m$ is less than and close to the critical temperature drop.
Average temperature of heating medium

$$t_h = t_B + \Delta\mathcal{T}_m = 125.4 + 39.6 = 165°\text{C}$$

From Steam Tables, for the steam temperature t_s = 165°C, saturation steam pressure = 700 kPa and latent heat of condensation of steam, λ_s = 2064.9 kJ/kg.

$$\phi_t = \dot{m}_s \lambda_s$$

$$\dot{m}_s = \frac{766.15}{2064.9} = 0.371 \text{ kg/s} \equiv 1335.6 \text{ kg/h}$$

For the first trial calculations, assuming overall heat transfer coefficient $U = 1000$ W/(m^2 · °C)

$$A_{\text{pro}} = \frac{\phi_t}{U\Delta T_m} = \frac{766.15 \times 10^3}{1000 \times 39.6}$$

$$A_{\text{pro}} = 19.3472 \text{ m}^2 = N_t\, \pi\, d_o\, L$$

Let length of tube $L = 6$ ft = 1.8288 m

$$d_o = 25.4 \text{ mm}$$

$$19.3472 = N_t \times \pi \times (0.0254) \times 1.8288$$

$$N_t = 132.57 \cong 133 \text{ tubes}$$

For 25.4 mm OD tube, 31.75 mm tube pitch ($P_t/d_o = 1.25$) triangular pitch arrangement and 1-1 shell and tube heat exchanger,

Shell ID, $D_i = 438$ mm (From Table 6.1(f))

Assume recirculation ratio = 4

Available differential head

$$\Delta p_{\text{av}} = L(\rho_L - \rho_{\text{av}})g \tag{6.95}$$

ρ_L = Density of liquid pentane at 125.4°C = 626 kg/m^3

Density of pentane vapour at 10 bar a pressure and at 125.4°C

$$\rho_v = \frac{pM}{RT} = \frac{10 \times 72}{(273 + 125.4)} \times \frac{273}{1.013\,25 \times 22.414}$$

$$\rho_v = 21.724 \text{ kg/m}^3$$

Average density of liquid-vapour mixture

$$\rho_{\text{av}} = \frac{\ln(V_o/V_i)}{V_o - V_i} \tag{6.96}$$

V_i = Specific volume of liquid at inlet

$$V_i = \frac{1}{\rho_L} = 1.597\,44 \times 10^{-3} \text{ m}^3/\text{kg}$$

For a recirculation ratio = 4

Flow rate of liquid at the outlet of reboiler

$$\dot{m}_{Lo} = 4 \times 10\,000 \text{ kg/h} = 40\,000 \text{ kg/h}$$

Flow rate of vapour at the outlet of reboiler $\dot{m}_{Vo} = 10\,000$ kg/h

Specific volume of vapour at outlet

$$V_o = \frac{(\dot{m}_{Vo}/\rho_v) + (\dot{m}_{Lo}/\rho_L)}{(\dot{m}_{vo} + \dot{m}_{Lo})} = \frac{\left(\dfrac{10\,000}{21.724} + \dfrac{40\,000}{626}\right)}{50\,000}$$

$$V_o = 0.010\,4843 \text{ m}^3/\text{kg}$$

$$\rho_{av} = \frac{\ln\left(\dfrac{0.010\,4843}{1.597\,44 \times 10^{-3}}\right)}{0.010\,4843 - 1.597\,44 \times 10^{-3}} = 211.715 \text{ kg/m}^3$$

$$\Delta p_{av} = 1.8288 \times (626 - 211.715) \times 9.81 = 7432.5 \text{ Pa}$$

Assume that higher sizes of the piping will be used in between base of distillation column and thermosyphon reboiler.
Pressure loss in the system ≅ Tube side pressure drop, Δp_t.
Tube side pressure drop,

$$N_p = 1, \quad \frac{\mu}{\mu_w} \cong 1, \quad \rho_{av} = 211.715 \text{ kg/m}^3$$

Tube inside diameter

$$d_i = 22.098 \text{ mm (16 BWG tube)}$$

Tube inside flow area, Eq. (6.26)

$$a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2 = \frac{133}{1} \times \frac{\pi}{4}(0.022\,098)^2 = 0.051 \text{ m}^2$$

Tube side mass velocity

$$G_t = \frac{\dot{m}}{a_t} = \frac{\dot{m}_{Vo} + \dot{m}_{Lo}}{a_t} = \frac{50\,000 \times \dfrac{1}{3600}}{0.051}$$

$$= 272.33 \text{ kg/(m}^2 \cdot \text{s)}$$

Tube side velocity, $u_t = \dfrac{G_t}{\rho_{av}} = \dfrac{272.33}{211.715}$

$$= 1.2863 \text{ m/s}$$

$$Re_t = \frac{d_i G_t}{\mu_L} = \frac{0.022\,098 \times 272.33}{0.12 \times 10^{-3}} = 50\,149.6$$

(Kern suggests to use liquid viscosity, instead of viscosity of liquid–vapour mixture)

$$J_f = 3.2 \times 10^{-3} \qquad \text{(From Fig. 6.13)}$$

Equation (6.31): $\Delta p_t = \left(8 \times 3.2 \times 10^{-3} \times \left(\dfrac{1.8288}{0.022\,098}\right) + 2.5\right) \times \left(\dfrac{211.715 \times 1.2863^2}{2}\right)$

$$\Delta p_t = 808.94 \text{ Pa}$$

$$\Delta p_{av} > \Delta p_t$$

Assume the higher value of recirculation ratio (greater than 4) and by trial and error calculations find the value of recirculation ratio for which $\Delta p_{av} \cong \Delta p_t$.
Trial I: Assume circulation ratio = 15.5

$$\dot{m}_{Lo} = 15.5 \times 10\,000 = 155\,000 \text{ kg/h}, \ \dot{m}_{vo} = 10\,000 \text{ kg/h}$$

$$V_o = \frac{\left(\dfrac{10\,000}{21.724}\right) + \left(\dfrac{155\,000}{626}\right)}{165\,000} = 4.2904 \times 10^{-3} \text{ m}^3\text{/kg}$$

$$V_i = 1.597\,44 \times 10^{-3}\ \text{m}^3/\text{kg}$$

$$\rho_{\text{av}} = \frac{\ln[(4.2904 \times 10^{-3})/(1.597\,44 \times 10^{-3})]}{(4.2904 \times 10^{-3} - 1.597\,44 \times 10^{-3})} = 366.874\ \text{kg/m}^3$$

$$\Delta p_{\text{av}} = 1.8288 \times (626 - 366.874) \times 9.81 = 4648.9\ \text{Pa}$$

Tube side mass velocity, $G_t = \dfrac{\left(\dfrac{165\,000}{3600}\right)}{0.051} = 898.693\ \text{kg/(m}^2 \cdot \text{s)}$

Tube side Reynolds number

$$Re_t = \frac{d_i G_t}{\mu_L} = \frac{0.022\,098 \times 898.693}{0.12 \times 10^{-3}} = 165\,494$$

Tube side fraction factor, $J_f = 2.64 \times 10^{-3}$ (From Fig. 6.13)

Tube side velocity $u_t = \dfrac{G_t}{\rho_{av}} = \dfrac{898.693}{366.874} = 2.45$ m/s

Tube side pressure drop

$$\Delta p_t = 1\left(8 \times 2.64 \times 10^{-3} \times \left(\frac{1.8288}{0.022\,098}\right) \times 1 + 2.5\right) \times \frac{(366.874 \times 2.45^2)}{2}$$

$$= 4677.2\ \text{Pa}$$

$$\Delta p_{\text{av}} \cong \Delta p_t$$

Hence, recirculation ratio of 15.5 is acceptable.

Heat transfer coefficients:

(a) Tube side heat transfer coefficient

$$Re_t = 165\,494$$

Kern suggests to use liquid properties.

Properties of liquid pentane at 125.4°C:

Specific heat $C_L = 2.2483$ kJ/(kg · °C)

Viscosity $\mu_L = 0.12$ cP

Thermal conductivity, $k_L = 0.124\,56$ W/(m · °C)

$$Pr = \frac{2.2483 \times 0.12 \times 10^{-3} \times 10^3}{0.124\,56} = 2.166$$

$$\frac{h_i d_i}{k} = 0.023\ Re^{0.8}\ Pr^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.19}$$

$$h_i = 0.023 \times \frac{0.124\,56}{0.022\,098} \times (165\,494)^{0.8} \times (2.166)^{0.33} \times 1$$

$$h_i = 2503.5\ \text{W/(m}^2 \cdot \text{°C)} \geq 1702.6\ \text{W/(m}^2 \cdot \text{°C)}$$

Take $h_i = 1702.6$ W/(m^2 · °C)

Shell side heat transfer coefficient $h_o' = 6000$ W/(m^2 · °C)

(This value includes fouling resistance)

Thermal conductivity of tube material $k_w = 45$ W/(m · K) (mild steel tubes).

Tube side fouling coefficient $h_{id} = 5000$ W/(m^2 · °C)

Eq. (6.46):

$$\frac{1}{U_o} = \frac{1}{6000} + \frac{0.0254 \ln\left(\frac{25.4}{22.098}\right)}{2 \times 45} + \frac{25.4}{22.098} \times \frac{1}{1702.6} + \frac{25.4}{22.098} \times \frac{1}{5000}$$

$$U_o = 900.13 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area required

$$A_{\text{req}} = \frac{\phi}{U_o \Delta T_m} = \frac{766.15 \times 1000}{900.13 \times 39.6} = 21.494 \text{ m}^2$$

$$\frac{A_{\text{pro}}}{A_{\text{req}}} = \frac{19.3472}{21.494} = 0.9 < 1.1$$

Assume new ratio of 1.2 for the tube length.

Revising tube length $L_{\text{rev}} = 6 \times \frac{1.2}{0.9} = 8 \text{ ft} \equiv 2.4384 \text{ m}$

$$A_{\text{pro}} = N_t \, \pi \, d_o \, L$$
$$= 133 \times \pi \times 0.0254 \times 2.4384 = 25.8786 \text{ m}^2$$

For the new tube length and with recirculation ratio = 15.5

$$\Delta p_{\text{av}} = 4648.9 \times \frac{2.4384}{1.8288} = 6198.53 \text{ Pa}$$

Tube side pressure drop,

$$\Delta p_t = 5182.63 \text{ Pa}$$

$\Delta p_{\text{av}} > \Delta p_t$, hence assume the higher value of recirculation ratio.

Trial: For the recirculation ratio = 17

$$\dot{m}_{Lo} = 17 \times 10\,000 = 170\,000 \text{ kg/h}, \quad \dot{m}_{Vo} = 10\,000 \text{ kg/h}$$

Specific volume of liquid-vapour mixture at the outlet of reboiler

$$V_o = \frac{\left(\frac{170\,000}{626} + \frac{10\,000}{21.724}\right)}{180\,000} = 4.066 \times 10^{-3} \text{ m}^3\text{/kg}$$

Average density, $\rho_{\text{av}} = \dfrac{\ln\left(\dfrac{4.066 \times 10^{-3}}{1.597\,44 \times 10^{-3}}\right)}{(4.066 \times 10^{-3} - 1.597\,44 \times 10^{-3})}$

$$= 378.46 \text{ kg/m}^3$$

$$\Delta p_{\text{av}} = 2.4384 \times (626 - 378.46) \times 9.81$$
$$= 5921.33 \text{ Pa}$$

Tube side pressure drop:

Tube side mass velocity

$$G_t = \frac{180\,000/3600}{0.051} = 980.392 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_i G_t}{\mu} = \frac{0.022\,098 \times 980.392}{0.12 \times 10^{-3}}$$

$$= 180\,539.2$$

$$J_f = 2.4 \times 10^{-3} \quad \text{(From Fig. 6.13)}$$

$$u_t = \frac{G_t}{\rho_{av}} = 2.59 \text{ m/s}$$

$$\Delta p_t = 1 \times \left(8 \times 2.4 \times 10^{-3} \times \left(\frac{2.4384}{0.022\,098}\right) + 2.5\right) \times \frac{378.46 \times 2.59^2}{2}$$

$$= 5862.75 \text{ Pa}$$

$$\Delta p_{\text{av}} < \Delta p_t \quad \text{(acceptable)}$$

For the recirculation ratio = 17

$$Re_i = 180\,539.2$$

$$h_i = 2503.5 \times \left(\frac{180\,539.2}{165\,494}\right)^{0.8}$$

$$= 2684 \text{ W/(m}^2 \cdot \text{°C)} \geq 1702.6 \text{ W/(m}^2 \cdot \text{°C)}$$

Take $h_i = 1702.6 \text{ W/(m}^2 \cdot \text{°C)}$

Heat transfer area required $A_{\text{red}} = 21.494 \text{ m}^2$

$$\text{Excess heat transfer area} = \left(\frac{25.8786}{21.494} - 1\right) \times 100$$

$$= 20.4\%$$

Shell side pressure drop Δp_s:

For the condensation of saturated steam, shell side pressure drop is calculated by Eq. (6.45).

Shell side Friction Factor, j_f:

To find this factor, first shell side flow area must be determined by Eq. (6.33).

Let B_s = Baffle spacing = D_s = Shell inside diameter

$B_s = D_s = 0.438$ m, $d_o = 25.4$ mm, $P_t = 31.75$ mm

$$A_s = \frac{31.75 - 25.4}{31.75} \times 0.438^2 = 0.038\,37 \text{ m}^2$$

$$\text{Shell side mass velocity } G_s = \frac{\dot{m}_s}{A_s} = \frac{0.371}{0.038\,37} = 9.67 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\text{Density of steam } \rho_v = \frac{1}{\text{Specific volume}}$$

From Steam Tables specific volume of saturated steam at 700 kPa pressure,

$$V_s = 0.272\,68 \text{ m}^3\text{/kg}$$

$$\rho_v = 3.6673 \text{ kg/m}^3$$

$$u_s = \frac{G_s}{\rho_v} = \frac{9.67}{3.6673} = 2.64 \text{ m/s}$$

$$\text{Equivalent diameter, } d_e = \frac{1.1}{d_o}(P_t^2 - 0.907 d_o^2) \tag{6.37}$$

$$d_e = \frac{1.1}{0.0254}\,(0.031\,75^2 - 0.907(0.0254)^2)$$

$$d_e = 0.0183 \text{ m}$$

Shell side Reynolds number:

$$Re_s = \frac{d_e G_s}{\mu}$$

Viscosity of steam at 165°C and at 700 kPa pressure $\mu_v = 14.5 \times 10^{-6}$ (N · s)/m^2 (Table 3.302, of Ref. 2)

$$Re_s = \frac{0.0183 \times 9.67}{14.5 \times 10^{-6}} = 12\,204.2$$

$$J_f = 4.78 \times 10^{-2} \text{ (For 25\% baffle cut)} \qquad \text{(Ref. Fig. 6.15)}$$

$$\Delta p_s = 0.5 \times 8 \times 4.78 \times 10^{-2} \left(\frac{0.438}{0.0183}\right)\left(\frac{2.4384}{0.438}\right) \times \left(\frac{3.6673 \times 2.64^2}{2}\right)$$

$$\Delta p_s = 325.6 \text{ Pa} \equiv 0.326 \text{ kPa} << 13.8 \text{ kPa}$$

It is well within permissible limit.

6.13.3 Criteria of Selection between Kettle-type Reboiler and Thermosyphon Reboiler

1. Boiling fluid side (process fluid side) pressure drop in kettle-type reboiler is negligible. While the same is high and significant in thermosyphon reboiler. Hence, with vacuum distillation, kettle-type reboiler is preferred. Thermosyphon reboiler is not preferred for use with absolute pressure below 0.3 bar. For example, in separation of the mixture of monochloroacetic acid and acetic acid by distillation, kettle-type reboiler is the only choice. Cracking temperature of monochloroacetic acid is 120°C and is obtained as the bottom product. Vapour pressure of monochloroacetic acid at 118°C is 60 Torr. Hence, to restrict the boiling point of monochloroacetic acid below 120°C, pressure drop in distillation column plus pressure drop in overhead condenser plus pressure drop in reboiler plus pressure drop in associated piping must be less than 60 Torr (0.08 bar). So, kettle-type reboiler is the only choice.
2. If the viscosity of bottom product of distillation column is very high, then thermosyphon reboiler cannot be used. In such a case, forced circulation reboiler is preferred.
   In fatty acid, separation in a distillation tower under vacuum, forced circulation reboiler is used due to viscous nature of fatty acids. However, for the vacuum service and corrosive system with highly viscous bottom product kettle-type reboiler is used. For example, separation of pure $POCl_3$ (phosphorous oxitrichloride) from treated crude $POCl_3$, residue or bottom product of distillation is highly viscous. Thermosyphon reboiler cannot be used for this application.
3. With atmospheric and high pressure distillation column, thermosyphon reboilers are preferred as size and fixed cost of thermosyphon reboiler is less than the same of kettle-type reboiler. Because of the fluid circulation, thermosyphon reboiler provides higher heat transfer coefficient than kettle-type reboiler. Even for the

same value of heat transfer area, fixed cost of kettle-type reboiler is higher than the same of the thermosyphon reboiler.

4. If the ratio of vaporization rate required to mass flow rate of residue (bottom product of distillation column) is 0.8 or above, kettle-type reboiler is preferred.

   Kettle-type reboilers are also use as "Vaporizers" in industry. Thermosyphon reboiler cannot be used as vaporizer. Separation taking place in kettle-type reboiler can be considered as equivalent to one theoretical stage. If Murphree efficiency of the column is 50% (approx.), it can result in reduction of two trays.

6.14 AIR COOLED HEAT EXCHANGERS AND AIR HEATERS

6.14.1 Introduction

Atmospheric air (having temperature varying from 20 to 45°C, depending on season) can be used as cooling medium if the process fluid is to be cooled up to 60°C as minimum driving force required for heat transfer in air-cooled heat exchanger is 10°C.

Advantages and disadvantages of air as a cooling medium over water as a cooling medium are given in section on air cooling.

As shown in Fig. 6.36, air cooled heat exchanger consists of banks of finned tubes over which air is blown by forced draft fan mounted below or drawn by an induced draft fan(s), mounted above the tube bundle. Tube bundle consists of tubes with spiral wound transverse fins in the majority of applications. Forced draft unit consumes less power. Induced draft provides comparatively even distribution of air across the bundle since the air velocity approaching the bundle is relatively low. Percentage recirculation of hot exhaust air is less in induced draft unit compared to the forced draft unit since the exist air velocity is several times that of forced draft unit. Induced draft arrangement gives more protection to heat transfer surface against rain fall, snow fall, sleet, etc.

Fig. 6.36 *Forced Draft Air Cooled Heat Exchanger*

If the difference between process fluid outlet temperature (required) and ambient-air temperature is less than 30°C, induced draft unit is preferred because of the less

% recirculation of hot exhaust air. But if the same is more than 30°C, forced draft unit is preferred as it consumes less power. 3/4 in (19.05 mm) OD and 1 in (25.4 mm) OD tubes are more commonly used. Fin spacing from 3.6 to 2.3 mm (7 to 11 fins per linear inch) and triangular pitch from 43 to 63.5 mm are common. Tube ends are left bare to permit the attachment or insertions of tubes into appropriate holes of box-type headers. Fin material can be aluminum, copper, mildsteel, stainless steel, etc.

The same heat exchanger is also used for heating the air by saturated steam or cooling of the atmospheric air by chilled water. Hot air is used as drying medium in the dryers while chilled air is used for air conditioning. In areas of water scarcity, air cooled refrigerant condenser is considered. Refer to Exercise 6.10(c) for design of such a condenser.

For all above-mentioned applications of the air heat exchangers, worst condition or design condition is different.

1. If atmospheric air is used as cooling medium then for the design calculations the maximum possible temperature of ambient air in the entire year must be considered.
2. If air is heated by saturated steam to get the hot air for dryer, then for the design calculations minimum possible temperature of ambient air in the entire year must be considered.
3. When atmospheric air is cooled to lower temperature (say to 14°C) by chilled water, then sizeable percentage of heat duty is utilized in the condensation of water vapour which is always present in atmospheric air. Hence, for this case, for the design calculations atmospheric air with the maximum possible absolute humidity (and with corresponding temperature) must be considered.

Fins are used to increase the heat transfer area and thereby to decrease the size of heat exchanger required. Needless to say, fins should have good contact with the tubes. Many techniques are used to fix fins on the tubes. For the heat transfer, fin area is not as effective as bare tube area. Hence, in design calculations to consider the effectiveness of fin area, fin efficiency is determined.

Design method for air side or finside is completely different from the same used for shell and tube heat exchanger, but for finding tube side heat transfer coefficient and tube side pressure drop same correlations are applicable that are used for a shell and tube heat exchanger.

6.14.2 Method for Finding Air Side or Fin Side Heat Transfer Coefficient and Pressure Drop

Method is detailed in Example 6.9 for calculating air side flow area (a_s).

Example 6.9 Consider

Tube OD = 19.05 mm, Fin OD = 38 mm

Tube pitch = 43 mm, Fin height = 9.475 mm

Number of fins per liner inch = 8 or fin spacing = 3.15 mm

Fin thickness = 0.889 mm

Type of fin = Spiral wound transverse fin

Solution

(a) Fin area per unit length of tube,

$$A_f = \frac{\pi}{4}\ (\text{Fin OD}^2 - \text{Tube OD}^2) \times 2 \times \text{No. of fins per one meter of tube length} \tag{6.98}$$

$$= \frac{\pi}{4}\ (0.038^2 - 0.019\ 05^2) \times 2 \times \frac{8}{25.4} \times 1000$$

$$= 0.534\ 86\ \frac{\text{m}^2}{\text{m of tube length}}$$

Bare tube area/m of one tube $= \pi\, d_o \times 1 - \pi d_o t_f \cdot n_f$ (6.99)

where, d_o = OD of bare tube, m

t_f = thickness of fin, m

n_f = number of fins per 1 m of tube length

$$= 8 \times \frac{1000}{25.4} = 315$$

Bare tube area,

$$A_b = \pi \times 0.019\ 05 \times 1 - \pi \times 0.019\ 05 \times \left(\frac{0.889}{1000} \times 1\right) \times 8 \times \frac{1000}{25.4}$$

$$= 0.043\ 09\ \text{m}^2/\text{m of one tube}$$

Fig. 6.37 *Projection of Finned Tube*

(b) Projected Perimeter:

$$= 2(\text{Fin OD} - \text{tube OD}) \times n_f + 2 \times (1 - t_f \cdot n_f) \tag{6.100}$$

$$= 2(0.038 - 0.01905) \times 8 \times \frac{1000}{25.4} + 2\left(1 - 0.889 \times 10^{-3} \times 8 \times \frac{1000}{25.4}\right)$$

$$= 13.377\ \text{m/m of one tube}$$

Air or finside heat transfer coefficient:

Find the equivalent diameter by using following equation:

$$d_e = \frac{2(\text{fin area} + \text{bare tube area})}{\pi\ (\text{projected perimeter})} \tag{6.101}$$

$$d_e = \frac{2 \times (0.534\,86 + 0.043\,09)}{\pi \times 13.377} = 0.0275 \text{ m}$$

Shell side or air side flow area:

$$a_s = \text{Air facing area} - \text{Area occupied by finned tubes}$$

$$\text{Air facing area} = \text{Length of area} \times \text{Width of area}$$
$$= \text{Tube length} \times \text{Width of area}$$

In majority of applications, width of air facing area is kept equal to tube length.

Fig. 6.38 *Air Facing Area*

For example, let tube length $L = 1500$ mm and

$$W = L \cong 1500 \text{ mm}$$

$$a_s = WL - n_p \times d_o \times L - n_p \times (\text{fin OD} - \text{tube OD})\, t_f \cdot n_f L \tag{6.102}$$

where, n_p = Number of tubes in bank perpendicular to the direction of flow of air

n_f = Number of fins per 1 m of tube length = 315

$$n_p = \frac{1500}{43} = 34.88 \simeq 35$$

Let $W = 1510$ mm (revised) for $n_p = 35$ tubes in one bank

$$W = (n_p - 1) \times p_t + \text{Fin OD} + x$$

$$W = (35 - 1) \times 43 + 38 + x \quad \text{or} \quad x = 10 \text{ mm}$$

Hence, there will be 5 mm clearance on either side.

$$a_s = 1.51 \times 1.5 - 35 \times 0.019\,05 \times 1.5 - 35 \times (0.038 - 0.019\,05) \times (0.889 \times 10^{-3}) \times 8 \times \frac{1500}{25.4}$$

$$= 0.986\,31 \text{ m}^2$$

After calculating air side flow area (a_s), following method is recommended for heat transfer calculations.

(c) After calculating d_e and a_s, calculate the air side mass velocity

$$G_s = \frac{\dot{m}_a}{a_s} \tag{6.103}$$

where, $\dot{m}_a$ = Mass flow rate of air, kg/s

a_s = Air side or shell side flow area, m^2

(d) Calculate air side Reynolds number.

$$Re = \frac{d_e G_s}{\mu} \tag{6.104}$$

where, d_e = Equivalent diameter, m
G_s = Mass velocity of air, kg/(m^2 · s)
μ = Viscosity of air at average temperature, kg/(m · s)

(e) Calculate air side Prandtl number.
All properties must be determined at average temperature on shell side.

(f) Calculate fin side or air side heat transfer coefficient (clean and for 100% fin efficiency).

$$h_f = J_{ha} \frac{k}{d_e} Pr^{1/3} \tag{6.105}$$

where, h_f = Heat transfer coefficient, W/(m^2 · °C) (clean)
J_{ha} = Factor for heat transfer coefficient
k = Thermal conductivity of air, W/(m · °C)
d_e = Equivalent diameter, m
Pr = Prandtl number of air

J_{ha}, factor of heat transfer coefficient, can be calculated by following equation:

$$J_{ha} = 0.085\,2072\, Re^{0.7324} \tag{6.106}$$

(g) Calculate dirty fin side heat transfer coefficient, h_f' (for 100% fin efficiency)

$$\frac{1}{h_f'} = \frac{1}{h_f} + \frac{1}{h_{do}} \tag{6.107}$$

where, h_{do} = Shell side fouling coefficient, W/(m^2 · °C)
h_f' = Dirty fin side heat transfer coefficient, W/(m^2 · °C)

(h) Calculate dirty fin side heat transfer coefficient based on inside heat transfer area and considering fin efficiency

$$h_{f_t}' = (\Omega \times A_f + A_o') \frac{h_f'}{A_i} \tag{6.108}$$

where, h_{f_t}' = Dirty fin side or air side heat transfer coefficient based on inside heat transfer area, W/(m^2 · °C)
Ω = Fin efficiency
A_f = Total fin area, m^2
A_o' = Total bare tube area, m^2
A_i = Inside heat transfer area, m^2

(i) Fin efficiency Ω can be determined from Fig. 6.39 (a) to (d) in which graph is drawn for $(r_e - r_b)\sqrt{\frac{h_f'}{k_m y_b}}$ vs Ω for the different values of $\frac{r_e}{r_b}$.

where, $r_e = \frac{\text{Fin OD}}{2}$, m, $r_b = \frac{\text{Tube OD}}{2}$, m

(a) Efficiency of Annular Fins of Constant Thickness

(b) Efficiency of Annular Fins with Metal Area for Constant Heat Flux

Fig. 6.39 *(c) Fin Efficiency of Several Types of Straight Fin*

$$y_b = \frac{\text{Fin thickness}}{2}, \text{ m}$$

k_m = Thermal conductivity of fin material, W/(m · °C)

h_f' = Dirty fin side heat transfer coefficient determined by Eq. (6.107), W/(m^2 · °C)

$I_n(u)$ = Modified Bessel function of the first kind and order n, dimensionless

$K_n(u)$ = Modified Bessel function of the second kind and order n, dimensionless

u = A function

β = Constant

(j) Determine the tube side heat transfer coefficient h_i as per correlations applicable for shell and tube heat exchangers. Decide the tube side fouling coefficient h_i. Find the overall heat transfer coefficient based on inside heat transfer area, U_i

(d) Efficiency Curves for Four Types of Spine

Fig. 6.39 *Fin Efficiencies for Various Types of Fins*[5]

$$U_i = \frac{1}{\dfrac{1}{h'_{fi}} + \dfrac{1}{h_i} + \dfrac{1}{h_{id}}} \tag{6.109}$$

(k) Then calculate the inside heat transfer area required

$$A_i = \frac{\phi_t}{U_i \Delta\mathcal{T}_m} \tag{6.110}$$

where, ϕ_t = Total heat duty, W

$\Delta\mathcal{T}_m$ = Mean temperature difference, °C

$= F_t\, \Delta\mathcal{T}_{\text{lm}}$ (6.11)

where, F_t = LMTD correction factor for cross flow can be determined, from Fig. (6.40)

Fig. 6.40 (a) and (b) *Cross Flow Temperature Difference Correction Factors*[5]

Fig. 6.40 (c) and (d) *Cross Flow Temperature Difference Correction Factors*[5]

(l) Heat transfer area provided (inside surface)

$$A_i = N_t \, \pi \, d_i \, L \tag{6.111}$$

Provided area should be greater by 10 to 20% than the required area.

(m) Calculate, the tube side pressure drop by using the same correlation that is used for shell and tube heat exchanger. It should be less than maximum allowable pressure drop.

(n) Calculate the volumetric equivalent diameter by following equation:

$$D_{ev} = \frac{4 \times \text{net free volume}}{(A_f + A_o')\left(\dfrac{n_p}{n_t}\right)} \tag{6.112a}$$

Fig. 6.41 *Finned Tube Arrangement*

S_L and S_T are tube pitches (P_t).

Net free volume,

$$V_{fv} = W \times L \times x' - n_p \left(\frac{\pi}{4}\right) d_o^2 L$$

$$- n_p \times \left(\frac{\pi}{4}\right) (\text{Fin OD}^2 - d_o^2) \times t_f n_f L \tag{6.112b}$$

where, W = Width of air facing area, m

L = Length of tube, m

x' = Horizontal distance between two vertical bank (see Fig. 6.41)

= $P_t \sin 60$ (For equilateral triangular pitch arrangement)

n_t = Total number of tubes

n_p = Number of tubes in one bank, perpendicular to the direction of the flow of air

d_o = Outside diameter of (bare) tube, m

t_f = Thickness of fin, m

n_f = Number of fins per 1 m of tube length
A_f = Total fin area, m^2
A_o' = Total bare tube area, m^2

For Example 6.9

Net free volume, $V_{fv} = 1.51 \times 1.5 \times 0.037\,24 - 35 \times \frac{\pi}{4}(0.019\,05)^2 \times 1.5$

$$- 35 \times \frac{\pi}{4}\,(0.038^2 - 0.019\,05^2) \times (0.889 \times 10^{-3})$$

$$\times \left(8 \times \frac{1000}{25.4} \times 1.5\right) = 0.0569 \text{ m}^3$$

$$A_f = (0.534\,86 \text{ m}^2/1 \text{ m of one tube}) \times L \times n_t$$
$$= 0.534\,86 \times 1.5 \times n_t = 0.802\,29 \times n_t \text{ m}^2$$
$$A_o' = (0.043\,09 \text{ m}^2/\text{m of one tube}) \times L \times n_t = 0.064\,635\, n_t \text{ m}^2$$

$$(A_f + A_o')\frac{n_p}{n_t} = (0.802\,29 + 0.064\,635) \times 35 = 30.3424 \text{ m}^2$$

$$D_{ev} = \frac{4 \times 0.0569}{30.3424} = 0.0075 \text{ m}$$

Reynolds number for the calculation of pressure drop

$$Re_s = \frac{D_{ev} G_s}{\mu_a} \tag{6.113}$$

where, G_s = Mass velocity of air based on flow area a_s, kg/($m^2 \cdot s$)
μ_a = Viscosity of air at average temperature, kg/(m · s)

Calculate the air side (or shell side) pressure drop

$$\Delta p_s = \frac{J_{fa} G_s^2 L_p}{D_{ev}\, \rho_a}\left(\frac{D_{ev}}{S_T}\right)^{0.4}\left(\frac{S_L}{S_T}\right)^{0.6} \tag{6.114}$$

where, Δp_s = Pressure drop, N/m^2 or Pa
J_{fa} = Air side friction factor can be calculated by following equation.

$$= 1.085\,58 \times Re^{-0.128\,025} \tag{6.115}$$

S_L = Center-to-center distance to the nearest tube in the next bank, m (as shown in Fig. 6.41)
S_T = Pitch in transverse beak, m (as shown in Fig. 6.41)
L_P = Effective path length of pressure drop, m
ρ_a = Average density of air, kg/m^3

L_P can be determined by following equation:

$$L_p = x' \times \frac{n_t}{n_p} \tag{6.116}$$

Calculated pressure drop should be less than maximum allowable pressure drop.

Example 6.10 Mobil therm oil is used as heating medium (i.e., thermic fluid) in chemical industry. Its operating range is from –1.1°C to 316°C.

It is required to cool 9000 kg/h of Mobil therm oil from 260°C to 200°C by using atmospheric air as a cooling medium in air cooler. Design the suitable air cooler.

Properties of Mobil therm oil at 230°C temperature

Density, $\rho = 850$ kg/m^3

Specific heat, $C_L = 2.5$ kJ/(kg · °C)

Viscosity, $\mu = 0.595$ cP or mPa · s

Thermal conductivity, $k = 0.1159$ W/(m · °C)

Solution

Heat duty required

$$\phi_t = \dot{m}C_L\,\Delta t = 9000 \times 2.5 \times (260 - 200)$$
$$= 1350\,000 \text{ kJ/h}$$
$$\equiv 375 \text{ kW} \equiv 375 \times 10^3 \text{ W}$$

Assume maximum possible temperature of atmospheric air in the area as 48°C.

Let outlet temperature of air = 75°C

Mass flow rate of air required

$$\dot{m}_a = \frac{\phi_t}{C_{pa}\Delta t'} = \frac{375}{C_{pa}(75 - 48)}$$

Specific heat of air at 61.5°C

$C_{pa} = 0.25$ kcal/(kg · °C) = 1.0467 kJ/(kg · °C) (Ref. 10)

$$\dot{m}_a = 13.2692 \text{ kg/s} \equiv 47\,769 \text{ kg/h}$$

$\Delta\mathcal{T}_m$ Mean temperature difference

$$\Delta\mathcal{T}_m = \Delta\mathcal{T}_{lm} \cdot F_t$$

Oil temperatures are very much greater than air temperatures, hence, forced draft fan can be selected to reduce the power consumption. Atmospheric air will enter from the bottom of tube bundle. Hot oil is also introduced from the bottom of the header. Hence, overall there is a cocurrent contacts. But, actually there is cross flow contact with multipass flow of oil.

$$\Delta\mathcal{T}_{lm} = \frac{(260 - 48) - (200 - 75)}{\ln\left(\dfrac{260 - 48}{200 - 75}\right)} = \frac{212 - 125}{\ln\left(\dfrac{212}{125}\right)} = 164.69 \text{ °C}$$

$$R = \frac{t_1 - t_2}{t_2' - t_1'} = \frac{(260 - 200)}{(75 - 48)} = 2.222$$

$$S = \frac{t_2' - t_1'}{t_1 - t_1'} = \frac{(75 - 48)}{(260 - 48)} = 0.127\,36$$

$F_t = 0.98$ (From Fig. 6.40a)

$\Delta\mathcal{T}_m = 164.69 \times 0.98 = 161.396$°C

Evaluation of h_o:

Equivalent diameter $d_e = \dfrac{2(\text{Fin area + Bare tube area})}{\pi(\text{Projected perimeter})}$ (6.101)

Let tube OD = 19.05 mm,
Type of fin = Spiral wound transverse fin
Fin OD = 38 mm, Tube pitch = 43 mm (Equilateral triangular)
Number of fins per linear inch = 8
Fin thickness = 0.889 mm
Fin and tube material = mild steel

Fin area, $A_{f1} = 2 \times \dfrac{\pi}{4}(\text{fin OD}^2 - d_o^2) \times \text{No. of fins per 1 m length}$ (6.100)

$$A_{f1} = 2 \times \frac{\pi}{4}(0.038^2 - 0.019\,05^2) \times 8 \times \frac{1000}{25.4}$$

$$= 0.534\,86 \text{ m}^2$$

Bare tube area $A_{o1} = \pi d_o \times 1 - \pi d_o t_f n_f$ (6.99)

(where n_f = no. of fins/1 m of one tube)

$$A_{o1} = \pi \times 0.01905 \times 1 - \pi \times 0.01905 \times (0.889 \times 10^{-3}) \times 8 \times \frac{1000}{25.4}$$

$$A_{o1} = 0.043\,09 \text{ m}^2$$

Projected perimeter per 1 m of one tube

$= 2\,(\text{fin OD} - d_o) \times n_f + 2\,(1 - t_f n_f)$ (6.100)

$$= 2(0.038 - 0.019\,05) \times 8 \times \frac{1000}{25.4} + 2\left(1 - 0.889 \times 10^{-3} \times 8 \times \frac{1000}{25.4}\right) = 13.377 \text{ m}$$

$$d_e = \frac{2(0.534\,86 + 0.043\,09)}{\pi \times 13.377} = 0.0275 \text{ m}$$

Flow Area:

For the first trial calculation,
Let tube length = 900 mm = L
and width of air facing area, W = 900 mm
Let n_p = number of tubes in one bank, perpendicular to the flow of air

$$n_p \cong \frac{W}{p_t} \cong \frac{900}{43} \cong 20.93$$

Let $n_p = 21$

$W = (n_p - 1)\,p_t + \text{Fin OD} + x$

$W = 20 \times 43 + 38 + x$

$x = 2$ mm

Let $W = 910$ mm

$x = 12$ mm (Revised)

Flow area

$$a_s = WL - n_p \times d_o \times L - n_p\,(\text{fin OD} - \text{tube OD})\,t_f n_f L \qquad (6.102)$$

$$= 0.91 \times 0.9 - 21 \times 0.019\,05 \times 0.9 - 21(0.038 - 0.019\,05)$$
$$\times 0.889 \times 10^{-3} \times 8 \times \frac{1000}{25.4} \times 0.9$$
$$= 0.358\,67 \text{ m}^2$$

Mass velocity, $G_s = \dfrac{\dot{m}_a}{a_s} = \dfrac{47\,769/3600}{0.358\,67} = 36.995 \text{ kg/(m}^2 \cdot \text{s)}$

Reynolds number, $Re = \dfrac{d_e G_s}{\mu_a}$ (6.104)

Viscosity of air at 61.5°C

$$\mu_a = 2000 \times 10^{-7} \text{ P} = 2000 \times 10^{-8} \text{ kg/(m} \cdot \text{s)}$$

(Figure 3.42 of Ref. 2)

$$Re = \frac{0.0275 \times 36.995}{2000 \times 10^{-8}} = 50\,868$$

Factor for heat transfer coefficient

$$J_{ha} = 0.085\,2072\, Re^{0.7324} \quad (6.106)$$
$$= 0.085\,2072(50\,868)^{0.7324}$$
$$= 238.49$$

Clean heat transfer coefficient, for 100% fin efficiency

$$h_f = J_{ha} \frac{k}{d_e} Pr^{1/3} \quad (6.105)$$

Prandtl number of air at 61.5°C:
Thermal conductivity of air at 61.5°C, $k = 0.028\,784$ W/(m · °C)
(Table 3.314, of Ref. 2)

$$Pr = \frac{1.0467 \times 2000 \times 10^{-8}}{0.028\,784} \times 10^3$$
$$= 0.7273$$
$$h_f = 238.49 \times \frac{0.028\,784}{0.0275} \times (0.7273)^{1/3}$$
$$= 224.49 \text{ W/(m}^2 \cdot \text{°C)}$$

Dirty fin side heat transfer coefficient

$$\frac{1}{h_f'} = \frac{1}{h_f} + \frac{1}{h_{do}} \quad (6.107)$$

$$h_{do} = 5000 \text{ W/(m}^2 \cdot \text{°C)} \quad \text{(From Table 6.10)}$$

$$\frac{1}{h_f'} = \frac{1}{224.49} + \frac{1}{5000}$$
$$h_f' = 214.84 \text{ W/(m}^2 \cdot \text{°C)}$$

Dirty fin side heat transfer coefficient based on inside heat transfer area and fin efficiency.

$$h'_{fi} = (\Omega \times A_f + A'_o)\frac{h'_f}{A_i} \tag{6.108}$$

$A_f = n_t A_{f1} L$, where, n_t = Total number of tubes

Total fin area $A_f = n_t \times 0.53\,486 \times 0.9 = 0.481\,374\, n_t\ \text{m}^2$

A'_o = Total bare tube area

$A_o{}' = n_t A_{o1} L = 0.043\,09 \times 0.9 \times n_t = 0.038\,781\, n_t$

$A_i = n_t \pi d_i L$

Tube inside diameter, d_i = 14.8336 mm (For 14 BWG tube)

$A_i = \pi(0.014\,8336) \times 0.9 \times n_t = 0.041\,94\, n_t\ \text{m}^2$

Fin efficiency (Ω):

Evaluation of function, $(r_e - r_b)\sqrt{\dfrac{h'_f}{k_m y_b}}$:

where, $r_e = \dfrac{\text{fin OD}}{2} = \dfrac{38}{2} = 19\ \text{mm} \equiv 0.019\ \text{m}$

$r_b = \dfrac{\text{Tube OD}}{2} = \dfrac{19.05}{2} = 9.525\ \text{mm} \equiv 0.009\,525\ \text{m}$

k_m = Thermal conductivity of fin material (mild steel)
= 45 W/(m · °C)

$y_b = \dfrac{\text{Fin thickness}}{2}$ (For fins of uniform thickness)

$= \dfrac{0.889 \times 10^{-3}}{2} = 4.445 \times 10^{-4}\ \text{m}$

$$\text{Function} = (0.019 - 0.009\,525)\sqrt{\frac{214.84}{45 \times 4.445 \times 10^{-4}}} = 0.98$$

$\dfrac{r_e}{r_b} = 1.994\,75, \quad \Omega = 0.68$ [from Fig. 6.39(a)]

$$h'_{fi} = \frac{(0.68 \times 0.481\,374\, n_t + 0.038\,781\, n_t) \times (214.84)}{0.041\,94\, n_t}$$

$= 1875.45\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$

Evaluation of h_i:

Tube side flow area, a_t

Let number of tubes per pass = n_p = 21

$$a_t = 21 \times \frac{\pi}{4} d_i^2$$

$$= 21 \times \frac{\pi}{4} \times (0.0148\ 336)^2 = 3.629 \times 10^{-3}\ \text{m}^2$$

Tube side mass velocity,

$$G_t = \frac{\dot{m}}{a_t} = \frac{9000/3600}{3.629 \times 10^{-3}} = 688.9\ \text{kg/(m}^2 \cdot \text{s)}$$

Tube side velocity, $u_t = \dfrac{G_t}{\rho} = \dfrac{688.9}{850}$

$$= 0.81\ \text{m/s (low)}$$

Tube side Reynolds number

$$Re_t = \frac{0.0148\ 336 \times 688.9}{0.595 \times 10^{-3}} = 17\ 174.57$$

Prandtl number $Pr = \dfrac{C_L \mu}{k} = \dfrac{2.5 \times 0.595 \times 10^{-3} \times 10^3}{0.1159}$

$$= 12.8343$$

Using Dittus–Bolter equation

$$\frac{h_i d_i}{k_f} = 0.023 \times \frac{0.1159}{0.014\ 8336} \times (17\ 174.57)^{0.8}(12.8343)^{0.33}$$

$$= 1019.15\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

$$U_i = \frac{1}{\dfrac{1}{h'_{ft}} + \dfrac{1}{h_i} + \dfrac{1}{h_{id}}} \tag{6.109}$$

$$U_i = \frac{1}{\left(\dfrac{1}{1875.45} + \dfrac{1}{1019.15} + \dfrac{1}{5000}\right)} = 583.29\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area (inside) required

$$A_{i\text{req}} = \frac{\phi_t}{U_i \Delta T_m} = \frac{375 \times 1000}{583.29 \times 161.396} = 3.98\ \text{m}^2$$

Let heat transfer area to be provided

$$A_{i\text{pro}} = 1.1 \times A_{i\text{req}} = 4.378\ \text{m}^2$$

Heat transfer area per bank $= n_p\, \pi\, d_i\, L = 21 \times \pi \times 0.014\ 8336 \times 0.9 = 0.880\ 76\ \text{m}^2$

Number of banks required $= \dfrac{4.378}{0.880\ 76} = 4.971$

Let number of banks = 5

Excess heat transfer area = 10.65%

Total number finned tubes $n_t = 21 \times 5 = 105$

Total fin area $A_f = 105 \times 0.534\ 86 \times 0.9 = 50.544\ \text{m}^2$

Total bare tube area $A'_o = 105 \times 0.043\ 09 \times 0.9 = 4.072\ \text{m}^2$

Total outside heat transfer area = 54.616 m^2

Air side pressure drop

Net free volume, $V_{fv} = W\,L\,x' - n_p \times \frac{\pi}{4} d_o^2\,L - n_p \times \frac{\pi}{4}(\text{Fin OD}^2 - d_o^2)\,t_f\,n_f\,L$ (6.112b)

where, x' = Horizontal distance between two vertical banks, mm
= P_t sin 60 (For equilateral triangular pitch arrangement), mm
= 43 mm × sin 60 = 37.239 mm

Net free volume, $V_{fv} = 0.91 \times 0.9 \times 0.037\,239 - 21 \times \frac{\pi}{4}(0.019\,05)^2 \times 0.9$

$$-21 \times \frac{\pi}{4}(0.038^2 - 0.019\,05^2)(0.889 \times 10^{-3})\left(8 \times \frac{1000}{25.4}\right) \times 0.9$$

$$= 0.020\,62 \text{ m}^3$$

Volumetric equivalent diameter

$$D_{ev} = \frac{4 \times \text{Net free volume}}{(A_f + A_o')\left(\frac{n_p}{n_t}\right)} \quad (6.112a)$$

$$= \frac{4 \times 0.020\,63}{(50.544 + 4.072) \times \frac{21}{105}} = 0.007\,555 \text{ m}$$

Reynolds number for calculating pressure drop

$$Re_s = \frac{D_{ev} G_s}{\mu} = \frac{0.007\,555 \times 36.995}{2000 \times 10^{-8}} = 13\,974.86$$

$$J_{fa} = 1.085\,58 \times Re^{-0.128\,025} \quad (6.115)$$

$$= 1.085\,58 \times (13\,974.86)^{-0.128\,025} = 0.319\,86$$

$$\Delta p_s = \frac{J_{fa} G_s^2 L_p}{D_{ev}\rho}\left(\frac{D_{ev}}{S_T}\right)^{0.4}\left(\frac{S_L}{S_T}\right)^{0.6} \quad (6.114)$$

where, L_p = Effective path length for pressure drop, m

$$L_p = x' \frac{n_t}{n_p} = 0.037\,239 \times \frac{105}{21} = 0.1862 \text{ m}$$

Density of air $\rho = \frac{PM}{RT} = \frac{1 \times 29}{0.0821 \times (61.5 + 273)} = 1.056 \text{ kg/m}^3$

$$\Delta p_s = \frac{0.319\,86 \times 36.995^2 \times 0.1862}{0.007\,555 \times 1.056}\left(\frac{0.007\,555}{0.043}\right)^{0.4} \times \left(\frac{0.043}{0.043}\right)^{0.6}$$

(For equilateral triangular pitch $S_L = S_T = P_t$)

$$\Delta p_s = 5096 \text{ N/m}^2 = 519.6 \text{ mm WC} > \Delta p_{s\max} = 350 \text{ mm WC}$$

Discharge pressure of fans are generally less than 350 mm WC.

To decrease the pressure drop, flow area a_s must be increased

Let the revised tube length, $L = 1200$ mm and
width of flow area (air facing area), $W = 1200$ m
Number of tubes in one bank

$$n_p \cong \frac{W}{p_t} = \frac{1200}{43} = 27.9 = 28$$

Let $n_p = 28$

$$W = (n_p - 1)p_t + \text{Fin } OD + x$$

$$W = 27 \times 43 + 38 + x = 1200 \Rightarrow x = 1 \text{ mm}$$

Let $W = 1210$ mm and $x = 11$ mm (Revised)

$$a_s = WL - n_p d_o L - n_p\,(\text{fin OD} - d_o)t_f n_f L \tag{6.102}$$

$$= 1.21 \times 1.2 - 28 \times 0.019\,05 \times 1.2$$

$$- 28(0.038 - 0.019\,05) \times 0.889 \times 10^{-3} \times 8 \times \frac{1200}{25.4}$$

$$= 0.633\,64 \text{ m}^2$$

Mass velocity, $G_s = \dfrac{47\,769/3600}{0.633\,64} = 20.941 \text{ kg/(m}^2 \cdot \text{s)}$

Reynolds number, $Re = \dfrac{d_e G_s}{\mu_a}$

$$Re = \frac{0.0275 \times 20.941}{2000 \times 10^{-8}} = 28\,794$$

Factor for heat transfer coefficient

$$J_{ha} = 0.085\,2072\, Re^{0.7324} = 157.2$$

Clean heat transfer coefficient, for 100% fin efficiency

$$h_f = J_{ha} \frac{k}{d_e} P_r^{1/3} \tag{6.105}$$

$$= 157.2 \times \frac{0.028\,784}{0.0275} \times (0.7273)^{1/3}$$

$$= 147.97 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Dirty finside coefficient

$$\frac{1}{h'_f} = \frac{1}{147.97} + \frac{1}{5000}$$

$$h'_f = 143.72 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$(r_e - r_b)\sqrt{\frac{h'_f}{k_m y_b}} = (0.019 - 0.0095\,25)\sqrt{\frac{143.72}{45 \times 4.445 \times 10^{-4}}}$$

$$= 0.8$$

$$r_e/r_b = 1.994\,75,\ \Omega = 0.75 \quad \text{(Fig. 6.31(a))}$$

Dirty finside heat transfer coefficient based on inside heat transfer area and fin efficiency

$$h'_{fi} = (\Omega \times A_f + A'_o)\frac{h'_f}{A_i} \tag{6.108}$$

$$= (0.75 \times 0.534\,86 \times 1.2 \times n_t + 0.043\,09 \times 1.2 \times n_t)\frac{143.72}{\pi \times (0.014\,336) \times 1.2 \times n_t}$$

$$= (0.75 \times 0.534\,86 + 0.04309) \times \frac{143.72}{0.0466}$$

$$= 1370 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Evaluation of h_i

$$n_p = 28$$

$$a_t = 28 \times \frac{\pi}{4}(0.014\,8336)^2 = 4.84 \times 10^{-3} \text{ m}^2$$

$$G_t = \frac{\dot{m}}{a_t} = \frac{9000/3600}{4.84 \times 10^{-3}} = 516.53 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_t = \frac{G_t}{\rho} = \frac{516.53}{850} = 0.608 \text{ m/s,}$$

$$Re = \frac{0.014\,8336 \times 516.53}{0.595 \times 10^{-3}} = 12\,877.3$$

$$h_i = 0.023 \times \frac{0.1159}{0.0148\,336} \times (12\,877.3)^{0.8}(12.8343)^{0.33}$$

$$= 809.445 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Overall coefficient based on inside heat transfer surface, Eq. (6.109):

$$\frac{1}{U_i} = \frac{1}{h'_{fi}} + \frac{1}{h_i} + \frac{1}{h_{id}} = \frac{1}{1370} + \frac{1}{809.445} + \frac{1}{5000}$$

$$U_i = 461.82 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$A_{i\text{req}} = \frac{\phi_t}{U_i \Delta T_m} = \frac{375 \times 1000}{461.82 \times 161.396} = 5.03 \text{ m}^2$$

Heat transfer area per one bank = $\pi d_i L \times n_p = 1.5658 \text{ m}^2$

Number of banks required = $\dfrac{5.03}{1.5658} = 3.2124$

Let number of banks = 4 = $\dfrac{n_t}{n_p}$

Excess heat transfer area = $\dfrac{(4 - 3.2124)}{3.2124} \times 100 = 24.5\ \%$

Total number of finned tubes $n_t = 28 \times 4 = 112$

Tube OD = 19.05 mm

Fin OD = 38 mm
Number of fins per inch = 8
Thickness of fin = 0.889 mm
Type of fin = spiral wound transverse fin
Tube length = 1200 mm
Width of air facing area = 1210 mm
Tube pitch = 43 mm (equilateral triangular)
Total inside heat transfer area, $A_i = 6.263\ \text{m}^2$ (provided)
Total fin area $A_f = 71.885\ \text{m}^2$
Total bare tube area, $A_o' = 5.7913\ \text{m}^2$

Air side pressure drop

Net free volume

$$V_{fv} = WLx' - n_p \times \frac{\pi}{4} d_o^2 L - n_p \times \frac{\pi}{4}\ (\text{Fin OD}^2 - d_o{}^2)\ t_f n_f L \qquad (6.112b)$$

$$= 1.21 \times 1.2 \times 0.037\ 239 - 28 \times \frac{\pi}{4}(0.019\ 05)^2 \times 1.2 - 28$$

$$\times \frac{\pi}{4}\ (0.038^2 - 0.019\ 05^2) \times 0.889 \times 10^{-3} \times 8 \times \frac{1200}{25.4}$$

$$= 0.0365\ \text{m}^3$$

$$D_{ev} = \frac{4 \times \text{net free volume}}{(A_f + A_o')\dfrac{n_p}{n_t}} \qquad (6.112a)$$

$$= \frac{4 \times 0.0365}{(71.885 + 5.7913) \times \dfrac{28}{112}}$$

$$= 0.007\ 518\ \text{m}$$

Reynolds number for calculating pressure drop

$$Re = \frac{D_{ev} G_s}{\mu} = \frac{0.007\ 518 \times 20.941}{2000 \times 10^{-8}}$$

$$= 7871.7$$

Friction factor for calculating pressure drop

$$J_{fa} = 1.085\ 58 \times Re^{-0.128\ 025} \qquad (6.115)$$

$$= 1.085\ 58 \times (7871.7)^{-0.128\ 025} = 0.344\ 25$$

Effective path length for pressure drop

$$L_p = x' \cdot \frac{n_t}{n_p} = 0.037\ 239 \times \frac{112}{28} = 0.148\ 956\ \text{m} \qquad (6.116)$$

$$\Delta p_s = \frac{J_{fa} G s^2 L_p}{D_{ev} \rho} \left(\frac{D_{ev}}{S_T}\right)^{0.4} \left(\frac{S_L}{S_T}\right)^{0.6} \qquad (6.114)$$

$$= \frac{0.344\,25 \times 20.941^2 \times 0.148\,956}{0.007\,518 \times 1.056} \times \left(\frac{0.007\,518}{0.043}\right)^{0.4} \times 1^{0.6}$$

$$= 1410 \text{ N/m}^2 \equiv 143.8 \text{ mm WC} < 350 \text{ mm WC } (\Delta p_{s\max})$$

Tube side pressure drop

Tube side pressure drop can be calculated by following equation:

$$\Delta p_t = N_p\left(8J_f(L/d_i)\left(\frac{\mu}{\mu_w}\right)^{-m} + 2.5\right)\frac{\rho u_t^2}{2} \tag{6.31}$$

N_p = No. of tube side passes = 4

For Re = 128 77.3, J_f = 0.0045 (From Fig. 6.13)

$$L = 1.2 \text{ m}, d_i = 0.014\,8336 \text{ m}$$

$$\rho = 850 \text{ kg/m}^3, u_t = 0.608 \text{ m/s}, \frac{\mu}{\mu_w} \cong 1$$

$$\Delta p_t = 4\left(8 \times 0.0045\left(\frac{1.2}{0.014\,8336}\right) \times 1 + 2.5\right) \times \frac{850 \times 0.608^2}{2}$$

$$= 3401.25 \text{ N/m}^2 \equiv 3.401 \text{ kPa}$$

Calculated pressure drop is well within maximum allowable (68 kPa).

Hence, to optimize the design, tube side velocity and tube side heat transfer coefficient can be increased. With a special arrangement in header, number of tube side passes can be doubled.

Let number of tubes per pass = 14

$$a_t = 14 \times \frac{\pi}{4}(0.014\,8336)^2 = 2.4194 \times 10^{-3} \text{ m}^2$$

$$G_t = 1033.3 \text{ kg/(m}^2 \cdot \text{s)}, Re = 25\,760.75, u_t = 1.2156 \text{ m/s}$$

$$h_i = 1409.6 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$U_i = 610 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$A_{i\text{req}} = \frac{375 \times 1000}{610 \times 161.396} = 3.809 \text{ m}^2$$

$$\text{Number of banks required} = \frac{3.809}{1.5658} = 2.4326$$

$$\text{Let number of banks} = 3 = \frac{n_t}{n_p}$$

$$\text{Excess area} = \frac{3 - 2.4326}{2.4326} \times 100 = 23.32\%$$

Total number of finned tubes $n_t = 28 \times 3 = 84$

Tube OD = 19.05 mm, Fin OD = 38 mm, Number of fins per inch = 8

Thickness of fin = 0.889 mm, Type of fin = Spiral wound transverse fin

Tube length = 1200 mm

Width of air facing area = 1210 mm

Tube pitch = 43 mm (equilateral triangular)

Total fin area, $A_f = 53.9139\ \text{m}^2$

Total bare tube area, $A_o' = 4.343\ 47\ \text{m}^2$

Number of tubes per pass = 14

Number of tube side passes = 6

Inside heat transfer area provided, $A_i = 4.6974\ \text{m}^2$

Air side pressure drop

$$D_{ev} = \frac{4 \times 0.0365}{(53.9139 + 4.343\ 47) \times \dfrac{28}{84}} = 0.007\ 518\ \text{m} \quad \text{(no change)}$$

$$Re_s = 7871.7,\ J_{fa} = 0.344\ 25 \text{ (no change)}$$

$$L_p = x' \frac{n_t}{n_p} = 0.037\ 239 \times \frac{84}{28} = 0.111\ 72\ \text{m} \quad \text{(Revised)}$$

Revised air side pressure drop

$$\Delta p_s = 1410 \times \left(\frac{0.111\ 72}{0.148\ 956}\right) = 1057.53\ \text{N/m}^2 \equiv 1.058\ \text{kPa}$$

$$\equiv 107.85\ \text{mm WC} < \Delta p_{\text{smax}}\ (= 350\ \text{mm WC})$$

Tube side pressure drop (revised)

$$N_p = 6, \quad Re_t = 25\ 760.75 \quad J_f = 0.0038$$

$$u_t = 1.2156\ \text{m/s}$$

$$\Delta p_t = 6\left(8 \times 0.0038 \times \left(\frac{1.2}{0.014\ 8336}\right) \times 1 + 2.5\right) \times \frac{850 \times 1.2156^2}{2}$$

$$= 18\ 687\ \text{N/m}^2 \equiv 18.687\ \text{kPa} < 68\ \text{kPa}$$

Since both side pressure drops are within desired range, design is acceptable.

6.15 PLATE HEAT EXCHANGERS [3,4,11]

6.15.1 Introduction

The Plate Heat Exchanger (PHE) consists of a frame in which closely spaced metal plates are clamped between a head and a follower. The plates have circular holes in a corner and these holes are sealed around by gasket which also attach over the plate edges. Holes at the corner are known as *ports*. Typical plate and gasketed plate heat exchanger is shown in Fig. 6.42.

Plates are pressed from stainless steel, titanium, Hastelloy C, Incoloy 825, nickel 200, Monel 400, aluminum brass, tantalum, etc. The thickness of plate ranges from 0.5 to 3 mm. The gap between the plates ranges from 1.5 to 5 mm. This gap is maintained by a gasket, which is sandwiched between two metal surfaces. Plate is having corrugated surface. Corrugated surface design strengthens the plates, increases the effective heat transfer area and produces turbulence in the fluids flowing through gaps. Plates are

easily cleaned and replaced. The heat transfer area can be readily increased or decreased by adding or removing plates, respectively. Hot and cold fluids are allowed to flow in alternate channels. For any one plate, one side corrugated surface is in contact with cold fluid while the other side of corrugated surface is in contact with hot fluid. Variety of flow arrangements are possible.

Fig. 6.42 *Plate-and-Frame Heat Exchanger. Hot Fluid Flows down between Alternate Plates and Cold Fluid Flows up between Alternate Plates.*

Heat transfer area per plate ranges from 0.03 to 1.5 m^2. Height-to-width ratio of plate ranges from 2 to 3. The upper limit of standard gasketed PHE is reported as 650 m^2 of heat transfer surface. Total number of plates of this PHE is 400. Variety of gasket materials (Table 6.39) are used in plate size of this PHE having 2.8 m height and 1.1 m width.

Table 6.39 Recommended Gasket Materials for PHE[2]

Gasket material	Temperature limit, °C
Styrene butadine rubber	85
Medium nitrile rubber	135
Butyl rubber	150
Fluorocarbon rubber	175
Compressed asbestos fiber	260

(a) Counter Flow

(b) Series Flows

(c) Looped (Parallel) Flows

(d) 2 Pass/2 Pass Counter-Current Flows

Fig. 6.43 *Various Arrangements of Flows in Plate and Frame Heat Exchangers*

6.15.2 Advantages and Disadvantages of Plate Heat Exchangers Over Shell and Tube Heat Exchangers

Advantages

1. Plate heat exchanger provides higher heat transfer coefficient as compared to shell and tube heat exchanger in available space.
2. Maintenance and cleaning is easier with PHE as compared to shell and tube heat exchanger.

3. Fouling resistance is less with PHE.
4. Minimum driving force required for heat transfer is 1°C for PHE while the same is 3 to 5°C for shell and tube heat exchanger.
5. PHE is more flexible. It is easy to increase or decrease the heat transfer area by adding or removing the plates.
6. With highly viscous slurry, PHE is better as it can be easily cleaned.

Disadvantages

1. Fixed cost of plate and hence, of PHE is higher. PHEs are not as widely used as shell and tube heat exchangers.
2. Gasketed PHE cannot be used if operating pressure is more than 30 bar g or operating temperature is more than 250°C. While for the same case shell and tube heat exchanger can be used.
3. Internal leakage or mixing of two fluids is more common with PHE compared to shell and tube heat exchanger.
4. Liquid containing suspended particles tend to plug the flow area in PHE easily and so frequent cleaning becomes necessary. In shell and tube heat exchanger, chocking can be delayed/avoided by keeping higher velocity in tubes or by selecting bigger size tubes.

6.15.3 Design of Plate Heat Exchanger

General process design steps[3,4,17,18] for PHE are same as that for shell and tube heat exchanger. Only differences are mentioned as follows:

1. LMTD correction factor (F_t) for PHE is determined from the value of number of transfer units, (NTU).

$$F_t = f(\text{NTU})$$

where, NTU = number of transfer units = $\dfrac{\Delta t}{\Delta \mathcal{T}_m}$ (6.117)

Δt = Temperature change required in process stream, °C.

$\Delta \mathcal{T}_m$ = Logarithmic mean temperature difference, °C

For series flow, F_t can be taken as 0.95. For 1-1 pass and higher passes (2-2, 3-3, 4-4) curves of F_t vs NTU are available (refer to Fig. 6.44).

2. If there is no phase change in the fluid, then forced convective heat transfer coefficient in conduits or plate film coefficient

$$\frac{h_p d_e}{k_f} = c Re^a Pr^b \left(\frac{\mu}{\mu_w}\right)^x \tag{6.118}$$

Reported values of constant and exponents are in the range of

c = 0.15 to 0.4

a = 0.65 to 0.85

b = 0.3 to 0.45

x = 0.05 to 0.2

Typical values are $c = 0.26$, $a = 0.65$, $b = 0.4$, and $x = 0.14$.

where, h_p = Plate film coefficient, W/(m^2 · °C)

d_e = Equivalent diameter, m

Fig. 6.44 *Log Mean Temperature Correction Factor for Plate Heat Exchanger*[3] *(Reproduced with the Permission of Elsevier, UK)*

$d_e = 2y$

y = Gap between the plates, m

Re = Reynold number = $(d_e\, G_p)/\mu = (d_e\, u_p\, \rho)/\mu$ (6.119)

G_p = Mass velocity of fluid, kg/(m$^2 \cdot$ s)

$= \dot{m}/(n_c \cdot A_f)$

$\dot{m}$ = Mass flow rate of fluid per channel, kg/s

A_f = Cross-sectional area of channel or gap (i.e., flow area), m^2

u_p = Channel velocity, m/s

$A_f = yW$, m^2

n_c = No. of channels per pass

W = Effective width of plate, m

3. Fouling coefficients for PHEs are given in Table 6.40. Fouling coefficients of PHEs are higher than the same for shell and tube heat exchangers.

Table 6.40 Recommended Fouling Coefficients for Plate Heat Exchangers

Fluid	Fouling coefficient, W/(m$^2 \cdot$ °C)
Cooling water	10 000
Steam	100 000
Demineralized water	100 000
Soft water	50 000
Lubricating oil	6 000
Light organic solvent	10 000
Process fluids	5 000 – 20 000

4. Pressure drop in plate heat exchanger can be calculated by the following equation:

$$\Delta p = \Delta p_p + \Delta p_{po} \tag{6.120}$$

where, Δp = Total pressure drop, Pa
Δp_p = Conduit or channel pressure drop, Pa
Δp_{po} = Port pressure drop, Pa

Pressure drop occurs, when fluid is flowing through conduit or channel

$$\Delta p_p = 8J_{fp}\left(\frac{L_p}{d_e}\right)\frac{\rho u_p^2}{2} \tag{6.121}$$

where, d_e = Equivalent diameter for conduit, m
$d_e = 2y = 2 \times$ gap between plate, m
ρ = Density of fluid, kg/m^3
u_p = Channel velocity, m/s
L_p = Path length, m
L_p = Plate length × Number of passes
J_{fp} = Friction factor = $f(Re)$

Value of J_{fp} depends on the design of plate used. For the turbulent flow following approximate equation can be used:

$$J_{fp} = 0.6\, Re^{-0.3} \quad \text{(Ref. 3)} \tag{6.122}$$

Δp_{po} = Pressure drop occurs due to the flow of fluid through ports

$$= 1.3\,\frac{\rho u_h^2}{2} \times N_p \text{ per port} \tag{6.123}$$

where, u_h = Velocity of fluid through the holes, m/s

$$= \frac{\dot{m}}{\rho A_h} \tag{6.124}$$

A_h = Area of hole = $\frac{\pi}{4}d_h^2$, m^2

d_h = Diameter of hole, m
N_p = Number of passes

Example 6.11 Ethanol liquid at the rate of 25 kg/s is to be cooled from 78°C to 40°C in gasketed plate heat exchanger. Operating pressure at inlet of heat exchanger is 2 atm g. Cooling water, available in plant at 32°C, is used as a cooling medium in heat exchanger. Design the suitable plate heat exchanger.

Solution

Heat duty, $\phi_t = \dot{m}C_L\Delta t$

C_L = Specific heat of ethanol liquid at average temperature (59°C)
= 2.93 kJ/(kg · °C)

$\phi_t = 25 \times 2.93 \times (78 - 40) = 2783.5$ kW $\equiv 2783.5 \times 10^3$ W

Let outlet temperature of cooling water = 40°C

Fig. 6.45

ϕ_t = 2783.5 kW = $\dot{m}_w \times 4.1868 \times (40 - 32)$

$\dot{m}_w$ = Mass flow rate of cooling water

= 83.1 kg/s ≡ 301 m^3/h

Logarithmic mean temperature difference for the perfect counter current contact

$$\Delta T_{lm} = \frac{(78-40)-(40-32)}{\ln\left(\frac{38}{8}\right)} = 19.254°C$$

Number of transfer unit (NTU) based on the maximum temperature difference

$$\text{NTU} = \frac{\Delta t}{\Delta T_{lm}} = \frac{78-40}{19.254°C} = 1.9736 \qquad (6.117)$$

For 1 : 1 pass arrangement, from Fig. 6.44:

$F_t = 0.96$

$\Delta T_m = F_t \Delta T_{lm} = 0.96 \times 19.254 = 18.484°C$

Assuming overall heat transfer coefficient as

$U = 2000\ W/(m^2 \cdot °C)$

$$A = \frac{\phi_t}{U_o \Delta T_m} = \frac{2783.5 \times 10^3}{2000 \times 18.484} = 75.3\ m^2$$

Selecting the plate having effective width 0.5 m and effective length 1.5 m.

Effective area of one plate = $1.5 \times 0.5 = 0.75\ m^2$

For the first trial calculation

Area provided = 75.3 m^2 = No. of plates × 0.75 m^2

Number of plates = 100.4

Let number of plates = 101

Area provided, $A_{pro} = 101 \times 0.75 = 75.75\ m^2$

Number of channels per pass, $n_c = (101 - 1)/2 = 50$

Let gap between successive plate, $y = 3$ mm

Equivalent diameter $d_e = 2y = 6\ mm = 6 \times 10^{-3}$ m

A_f = Flow area = Cross-sectional area of gap

$A_f = yW = 0.003 \times 0.5 = 1.5 \times 10^{-3}\ m^2$

Ethanol side heat transfer coefficient

$$\frac{h_p d_e}{K_p} = c Re^a Pr^b \left(\frac{\mu}{\mu_w}\right)^x \qquad (6.118)$$

Let $c = 0.26$, $a = 0.65$, $b = 0.4$ and $\dfrac{\mu}{\mu_w} \cong 1$

$$h_p \cdot \frac{d_e}{k_f} = 0.26 Re^{0.65}\, Pr^{0.4}$$

For the flow of ethanol, $Re = \dfrac{d_e G_p}{\mu}$

Mass velocity of ethanol, $G_{pE} = \dfrac{\dot{m}_E}{\text{Total gap area}}$

$$= \frac{25}{50 \times 1.5 \times 10^{-3}} = 333.33 \text{ kg/(m}^2 \cdot \text{s)}$$

Channel velocity, $u_{pE} = G_{pE}/\rho$

ρ = Density of ethanol at 59°C = 775 kg/m^3

$$u_{pE} = \frac{333.33}{775} = 0.43 \text{ m/s}$$

$$Re = \frac{6 \times 10^{-3} \times 333.33}{0.6 \times 10^{-3}} = 3333.3$$

Viscosity of ethanol at 59°C, $\mu = 0.6$ cP

$$Pr = \frac{C_L \mu}{k}$$

Thermal conductivity of ethanol at 59°C, $k = 0.147$ W/(m · °C)

$$Pr = \frac{2.93 \times 0.6 \times 10^{-3} \times 10^3}{0.147} = 11.96$$

$$h_{pE} = 0.26 \frac{k_f}{d_e} Re^{0.65} Pr^{0.4}$$

$$h_{pE} = 0.26 \times \frac{0.147}{6 \times 10^{-3}} \times (3333.3)^{0.65} (11.96)^{0.4}$$

$$h_{pE} = 3350.43 \text{ W/(m}^2 \cdot \text{°C)}$$

Cooling water side heat transfer coefficient:

Mass velocity of water, $G_{pW} = \dfrac{\dot{m}_w}{\text{Total gap area}}$

$$= \frac{83.1}{50 \times 1.5 \times 10^{-3}} = 1108 \text{ kg/(m}^2 \cdot \text{s)}$$

Density of water at 36°C, $\rho = 993.684$ kg/m^3

Viscosity of water at 36°C $\mu = 0.72$ cP

$$h_{pW} = \frac{G_p}{\rho} = \frac{1108}{993.684} = 1.115 \text{ m/s}$$

Reynold number $Re = \dfrac{d_e G_p}{\mu}$

$$Re = \frac{6 \times 10^{-3} \times 1108}{0.72 \times 10^{-3}} = 9233$$

Thermal conductivity of water at 36°C, $k = 0.6228$ W/(m · °C)
Prandtl number for water at 36°C

$$Pr = \frac{C_{LW}\mu}{k} = \frac{4.1868 \times 0.72 \times 10^{-3} \times 10^{3}}{0.6228}$$

$$Pr = 4.84$$

$$h_{pW} = 0.26 \frac{k}{d_e} Re^{0.65} Pr^{0.4}$$

$$h_{pW} = \frac{0.26 \times 0.6228}{6 \times 10^{-3}} \times (9233)^{0.65} \times (4.84)^{0.4}$$

$$h_{pW} = 19\ 168.66 \text{ W/(m}^2 \cdot \text{°C)}$$

Overall heat transfer coefficient:

$$\frac{1}{U_o} = \frac{1}{h_{pE}} + \frac{1}{h_{fE}} + \frac{t_p}{k_p} + \frac{1}{h_{pW}} + \frac{1}{h_{fW}}$$

h_{fE} = Fouling coefficient for ethanol = 10 000 W/(m^2 · °C)
t_p = Plate thickness = 1 mm = 0.001 m
k_p = Thermal conductivity of plate material

Let the plate material be titanium.

k_p = 21 W/(m · °C)
h_{fW} = Fouling coefficient for cooling water in PHE
= 10 000 W/(m^2 · °C) (From Table 6.40)

$$\frac{1}{U_o} = \frac{1}{3350.43} + \frac{1}{10\ 000} + \frac{0.001}{21} + \frac{1}{19\ 168.66} + \frac{1}{10\ 000}$$

$$= 1671.52 \text{ W/(m}^2 \cdot \text{°C)}$$

Area required = $\dfrac{\phi_t}{U_o \Delta T_m} = \dfrac{2783.5 \times 1000}{1671.52 \times 18.484} = 90 \text{ m}^2$

Area required > Area provided
For the second trial calculations
Let number of plates be 121 with 2-2 pass arrangement.

Number of channels per pass = $\dfrac{121 - 1}{2 \times 2} = 30$

Area provided, $A_{pro} = 121 \times 0.75 = 90.75\ m^2$

For NTU = 1.9736 $F_t = 0.95$ (From Fig. 6.44)

$\Delta T_m = 0.95 \times 19.254°C = 18.2913°C$

$\boldsymbol{h_{pE}}$

Mass velocity, $G_{pe} = 333.33 \times \dfrac{50}{30} = 555.55\ kg/(m^2 \cdot s)$

Channel velocity, $u_{pe} = 0.43 \times \dfrac{5}{3} = 0.7167\ m/s$

$$Re = \frac{d_e G_{pE}}{\mu} = \frac{6 \times 10^{-3} \times 555.55}{0.6 \times 10^{-3}} = 5555.5$$

$$\boldsymbol{h_{pE}} = 3350.43 \times \left(\frac{5555.5}{3333.3}\right)^{0.65} = 4669.84\ W/(m^2 \cdot °C)$$

$\boldsymbol{h_{pW}}$

Mass velocity, $G_{pW} = 1108 \times \dfrac{5}{3} = 1846.67\ kg/(m^2 \cdot s)$

Channel velocity, $u_{pW} = 1.115 \times \dfrac{5}{3} = 1.8583\ m/s$

$$Re = 9233.33 \times \frac{5}{3} = 15\ 389$$

$$h_{pW} = 19\ 168.66 \times \left(\frac{15\ 389}{9233.33}\right)^{0.65} = 26\ 717.5\ W/(m^2 \cdot °C)$$

Overall coefficient

$$\frac{1}{U_o} = \frac{1}{4669.84} + \frac{1}{10\ 000} + \frac{0.001}{21} + \frac{1}{26\ 717.5} + \frac{1}{10\ 000}$$

$$U_o = 2003.25\ W/(m^2 \cdot °C)$$

Area required, $A_{req} = \dfrac{\phi_t}{U \Delta T_m} = \dfrac{2783.5 \times 1000}{2003.25 \times 18.2913} = 75.9646\ m^2$

Area provided, $A_{pro} = 90.75\ m^2$

Excess area $= \left(\dfrac{90.75}{75.9646} - 1\right) \times 100 = 19.46\%$ (acceptable)

Pressure drops

Ethanol side pressure drop

$$\Delta p = \Delta p_p + \Delta p_{po} \tag{6.120}$$

Channel pressure drop

$$J_{fp} = 0.6\ Re^{-0.3} \tag{6.122}$$

$$= 0.6(5555.5)^{-0.3} = 0.045\ 158$$

Path length, L_p = Plate length × Number of passes = 1.5 × 2 = 3 m

$$\Delta p_p = 8 \times 0.045\ 158 \times \left(\frac{3}{0.006}\right) \times \frac{775 \times 0.7167^2}{2}$$

$$= 35\ 953.5\ \text{N/m}^2 \equiv 35.954\ \text{kPa}$$

Let port diameter (hole diameter) = 120 mm

$$\text{Hole area} = \left(\frac{\pi}{4}\right) 0.12^2 = 0.011\ 31\ \text{m}^2$$

u_h = velocity of ethanol through hole (port)

$$u_h = \frac{\dot{m}_E}{\rho A_h} = \frac{25}{775 \times 0.011\ 31}$$

$$= 2.8522\ \text{m/s}$$

Port pressure drop

$$\Delta p_{po} = 1.3\,\frac{\rho u_h^2}{2} \times N_p$$

$$= 1.3 \times \frac{775 \times 2.8522^2}{2} \times 2 = 8196\ \text{N/m}^2 \equiv 8.196\ \text{kPa per port}$$

Assuming 2 ports, ethanol side pressure drop

$$\Delta p = 35.954 + 2 \times 8.196 = 51.746\ \text{kPa}$$

It may be noted that port pressure drop is 31.7% of total pressure drop which is significant.

Cooling water side pressure drop:

$$Re = 15\ 389$$

$$J_{fp} = 0.6\,Re^{-0.3} = 0.6 \times (15\ 389)^{-0.3} = 0.033\ 265$$

L_f = Plate length × No. of passes = 1.5 × 2 = 3 m

Channel pressure drop by Eq. (6.121):

$$\Delta p_p = 8 J_{fp} \left(\frac{L_p}{d_e}\right) \frac{\rho u_p^2}{2}$$

$$= 8 \times 0.033\ 265 \times \left(\frac{3}{0.006}\right) \times \frac{993.684 \times 1.8583^2}{2}$$

$$= 228\ 295.6\ \text{N/m}^2 \equiv 228.296\ \text{kPa per port} \quad \text{(very high)}$$

Trial-2:

To decrease the cooling water side pressure drop, let number of cooling water side passes = 1, i.e., 2 : 1 arrangement. Ethanol side heat transfer coefficient, velocity and pressure drop are same as that for the previous case (2 : 2 arrangement).

For 2 : 1 arrangement

u_p = 0.7167 m/s, h_{pE} = 4669.84 W/(m^2 · °C), Δp = 44.15 kPa (ethanol side)

For cooling water,

No. of plates = 121, No. of passes = 1

For the flow of water,

Number of channels per pass = $\dfrac{121-1}{2} = 60$

Mass velocity, $G_{pW} = \dfrac{1846.67}{2} = 923.33$ kg/(m^2 · s)

Velocity of water through channel, $u_p = \dfrac{1.8583}{2} = 0.929$ m/s

$$Re = \frac{15\,389}{2} = 7694.5$$

$$h_{pW} = 26\,717.5 \times \left(\frac{1}{2}\right)^{0.65} = 17\,026.54 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Overall heat transfer coefficient

$$\frac{1}{U_o} = \frac{1}{2003.25} + \frac{1}{17\,026.54} - \frac{1}{26\,717.5}$$

$$U_o = 1921.26 \text{ W(m}^2 \cdot {}^\circ\text{C)}$$

Area required $A_{\text{req}} = \dfrac{\phi_t}{U_o \Delta\mathcal{T}_m} \dfrac{2783.5 \times 1000}{1921.26 \times 18.2913} = 79.206$ m^2

Excess area = $\left(\dfrac{90.75}{79.206} - 1\right) \times 100 = 14.575\%$

Cooling water side pressure drop

$$Re = 7694.5$$

$$J_{fp} = 0.6\, Re^{-0.3} = 0.6 \times (7694.5)^{-0.3} = 0.040\,95$$

Channel pressure drop

$$\Delta p_p = 8 J_{fp} \left(\frac{L_p}{d_e}\right) \times \frac{\rho u_p^2}{2} \tag{6.121}$$

Path length L_p = Plate length × Number of passes = 1.5 m

$$\Delta p_p = 8 \times 0.040\,95 \times \left(\frac{1.5}{0.006}\right) \times \frac{993.684 \times 0.929^2}{2}$$

$$= 35\,118.3 \text{ N/m}^2 \equiv 35.118 \text{ kPa}$$

Hole or port diameter = 120 mm

Hole area = 0.011 31 m^2

u_h = Velocity of water through hole (port)

$$u_h = \frac{\dot{m}_W}{\rho A_h} = \frac{83.1}{993.684 \times 0.011\,31} = 7.3942 \text{ m/s} \quad \text{(high)}$$

Increase the port size to 150 mm.

$$u_h = \frac{\dot{m}_w}{\rho A_h} = \frac{83.1}{993.684 \times \frac{\pi}{4}(0.15)^2} = 4.732 \text{ m/s} \quad \text{(acceptable)}$$

$$\Delta p_{pt} = 1.3 \times \frac{\rho u_h^2}{2} \times N_p$$

$$= 1.3 \times \frac{993.684 \times 4.732^2}{2} \times 1$$

$$= 14462.7 \text{ N/m}^2 \equiv 14.463 \text{ kPa per port}$$

Assuming 2 ports, pressure drop for the flow of cooling water

$$\Delta p = 35.118 \text{ kPa} + 2 \times 14.463 \text{ kPa} = 64.044 \text{ kPa}$$

Here also, port pressure drop is 46.7% of total pressure drop.

6.16 SPIRAL FLOW HEAT EXCHANGERS

6.16.1 Introduction

Spiral flow heat exchangers provide a spiral flow path for one or both (cold and hot) fluids being handled. They are of two types; (i) spiral tube exchanger and (ii) spiral-plate exchanger. These heat exchangers offer the following advantages over conventional shell and tube heat exchangers:

1. Compactness
2. Centrifugal force increases the heat transfer coefficient, particularly of highly viscous liquid slurries or sludges.
3. Spiral plate heat exchangers foul at much lower rate than the shell and tube heat exchanger because of the single flow passage and curved flow path which does not allow easy settlement of solids.
4. Relative ease of cleaning.
5. Spiral configuration reduces stress associated with differential thermal expansion.
6. Maintenance cost is less.

Disadvantages of spiral-tube and spiral-plate heat exchangers over shell and tube heat exchangers are as follows.

1. Spiral flow heat exchangers are more expensive (requires higher fixed cost) than shell and tube heat exchangers having the same heat transfer surface.
2. Design of spiral flow heat exchangers is not well established like the same of shell and tube heat exchanger.
3. Pressure drop (loss) is higher than the same in shell and tube heat exchanger.
4. Maximum design pressure for spiral plate heat exchanger is 10 bar *g* as the spiral construction limits the design pressure.
5. Gasket is special and assembly requires skill.

6.16.2 Spiral Plate Heat Exchanger

Spiral plate heat exchanger is fabricated from two relatively long strips of plate which are spaced apart and wound around an open split center to form a pair of concentric spiral passages. Spacing is maintained uniformly along the length of the spiral by

spacer studs welded to plate. Commonly used material for the fabrication of spiral plate heat exchanger are carbon steel, stainless steel, Hastelloys, nickel and nickel alloys, titanium, copper-alloys, etc.

Figure 6.46 shows spiral flow in both channels. Flow channels are closed by welding alternate channels at both sides of spiral plate.

Fig. 6.46 *Spiral Flow in Both Channels*

Figure 6.47 shows flow is spiral in one channel, axial in other. One channel is completely open on both ends and other closed at both sides of plate.

Fig. 6.47 *Spiral Flow in One Channel, Axial in Another*

Figure 6.48 shows combination flow.

Fig. 6.48 *Combination Flow*

Spiral flow in both channels This type of flow arrangement is used for liquid-to-liquid services. With this type of flow arrangement, spiral plate heat exchanger is covered by flat heads on both sides. Usually two liquids flow counter currently with the cold liquid entering at the periphery and flowing toward the core and the hot liquid entering at the core and flowing toward the periphery. Use of this type spiral heat exchanger is common in cooling hot hydrogenated edible oil from deodorizer as it offers good heat transfer coefficient for viscous oil.

Spiral flow in one channel and axial flow in other This type of flow arrangement is used for condensing or boiling. With this type of flow arrangement, spiral flow heat exchanger is covered by conical heads on one or both sides. Condensation or boiling takes place in axial direction. This arrangement is preferred where there is a large difference in the volumes of two fluids. In case of condensation, difference between volumetric flow rates of condensing vapour and cooling medium is always large. In case of boiling, if hot oil is used as heating medium, then also the difference between volumetric flow rate of boiling liquid and the same of hot oil is large.

Combination flow Combination flow is used to condense vapours. In this arrangement, condensing vapour flows axially and then condensate flows spirally. This arrangement is used for condensation with subcooling. Part of the open spiral is kept closed at the top. Entering fluid (condensing vapour) flows axially through the center part of assembly in downward direction. Condensate at bottom flows spirally and coming out from the side bottom. With this arrangement, spiral heat exchanger is equipped with conical head at top and flat cover at bottom.

6.16.3 Process Design Steps for Spiral Plate Heat Exchanger

General process design steps[18,19] are as follows:

1. Calculate heat duty (ϕ in kW).
2. Select cooling medium or heating medium.
3. Based on energy balance calculate the mass flow rate of heating medium or cooling medium.
4. Calculate the LMTD.
5. Assume the value of overall heat transfer coefficient for the first trial calculations.
6. Determine the heat transfer area required based on the assumed value of overall heat transfer coefficient for first trial calculations.

$$A = \frac{\phi_t \times 10^3}{U \times \Delta T_{lm}}$$

7. Heat transfer area for spiral plate heat exchanger

$$A = 2 \times L \times H \tag{6.125}$$

where, A = Heat transfer area, m^2
L = Length of plate, m
H = Width of plate = Height or length of heat exchanger, m

Fix the value of H and find the value of L

$$L = \frac{A}{2 \times H} \tag{6.126}$$

Diameter of heat exchanger or diameter of outside spiral can be calculated by following equation:

$$D_s = [1.28 \times L(d_c + d_h + 2t) + c^2)^{1/2} \tag{6.127}$$

where, D_s = Outside spiral diameter, m
L = Length of plate, m
d_c = Channel spacing of cold side, m
d_h = Channel spacing of hot side, m
t = Plate thickness, m
c = Core diameter, m

Some dimensions of spiral plate heat exchanger are standardized and are as follows.

(a) Plate width H	:	101.6 mm (4 in), 152.4 mm (6 in), 304.8 mm (12 in), 457.2 mm (18 in), 609.6 mm (24 in), 762 mm (30 in) 914.4 mm (36 in), 1219.2 mm (48 in), 1524 mm (60 in), 1778 mm (72 in)
(b) Core Diameter, C	:	203.2 mm (8 in), 304.8 mm (12 in)
(c) Channel spacing, d_c and d_h	:	4.76 mm (3/16 in) (for 304.8 mm maximum width) 6.35 mm (1/4 in) (for 1219.2 mm maximum width)

7.94 mm (5/16 in), 9.525 mm (3/8 in), 12.7 mm (1/2 in), 19.05 mm (3/4 in), 25.4 mm (1 in)

(d) Plate thickness, t : For carbon steel 3.175 mm (1/8 in), 4.76 m (3/16 in), 6.35 mm (1/4 in), 7.94 mm (5/16 in)
For stainless steel 14 to 3 US gauge

In spiral plate heat exchanger to arrive at the best compact design outside diameter of spiral D_s and plate width (H) are kept approximately equal.

8. Calculate the equivalent diameter of flow channel by following equation:

$$D_e = \frac{4 \times \text{Flow area}}{\text{Wetted perimeter}} = \frac{4[\text{Spacing} \times \text{Width}]}{2(\text{Spacing} + \text{Width})}$$

For hot fluid

$$D_{eh} = \frac{4 \times (d_h \times H)}{2(d_h + H)} \tag{6.128a}$$

For cold fluid

$$D_{ec} = \frac{4 \times (d_c \times H)}{2(d_c + H)} \tag{6.128b}$$

9. Calculate the Reynolds number by following equation:

$$Re = \frac{2\dot{m}}{H \times \mu} \tag{6.129}$$

where, $\dot{m}$ = Mass flow rate of fluid, kg/s
H = Width of plate, m
μ = Viscosity of fluid, kg/(m · s) or Pa · s

Reynolds number is calculated for both; cold fluid and hot fluid.

10. Calculate the critical Reynolds number Re_c. Critical Reynolds number is the value of Reynolds number above which turbulent flow is achieved.

$$Re_c = 20\,000\left(\frac{D_e}{D_s}\right)^{0.32} \tag{6.130}$$

where, D_e = Equivalent diameter of flow channel, m
D_s = Outside diameter of spiral, m

11. Calculate the cold fluid side and hot fluid side heat transfer coefficients by using the suitable correlations. Various correlations for calculating heat transfer coefficient for spiral plate heat exchangers are given as follows.

(a) For spiral flow with no phase change and $Re > Re_c$ (i.e., turbulent flow):

$$\frac{h}{C_p G} = \left(1 + 3.5\left(\frac{D_e}{D_s}\right)\right)(0.023\, Re^{-0.2} \times Pr^{-2/3}) \tag{6.131}$$

For spiral flow with, no phase change and, $Re < Re_c$ (i.e., laminar flow)

$$\frac{h}{C_p G} = 1.86\, Re^{-2/3} Pr^{-2/3} \left(\frac{d}{D_s}\right)^{1/6} \left(\frac{\mu_f}{\mu_b}\right)^{-0.14} \tag{6.132}$$

where, h = Heat transfer coefficient, W/(m^2 · °C)
C_p = Specific heat of fluid, J/(kg · °C)
G = Mass velocity of fluid, kg/(m^2 · s)
Re = Reynolds number
Pr = Prandtl number
μ = Viscosity of fluid, kg/(m · s) or Pa · s
k = Thermal conductivity of fluid, W/(m · °C)
d = Channel spacing, d_c or d_h, m
D_s = Outside spiral diameter, m

Here mass velocity is calculated by following equation:

$$G = \frac{\dot{m}}{(d \times H)} \tag{6.133}$$

where, $\dot{m}$ = Mass flow rate, kg/s
$d = d_c$ or d_h, m
H = Width of plate, m

(b) For axial flow with no phase change and $Re > 10\,000$

$$\frac{h}{C_p G} = 0.023\, Re^{-0.2}\, Pr^{-2/3} \tag{6.134}$$

12. Calculate the overall heat transfer coefficient, U_o, by following equation.

$$\frac{1}{U_o} = \frac{1}{h_c} + \frac{1}{h_h} + \frac{1}{h_{cd}} + \frac{1}{h_{hd}} + \frac{t}{k_m} \tag{6.135}$$

where, h_c = Cold fluid side heat transfer coefficient, W/(m^2 · °C)
h_h = Hot fluid side heat transfer coefficient, W/(m^2 · °C)
h_{cd} = Cold fluid side fouling coefficient, W/(m^2 · °C)
h_{hd} = Hot fluid side fouling coefficient, W/(m^2 · °C)
t = Thickness of plate, m
k_m = Thermal conductivity of plate material, W/(m · °C)

13. Calculate the heat transfer area required

$$A_r = \frac{\phi_t \times 10^3}{U_o \Delta \mathcal{T}_{\ln}}$$

14. Calculate the % excess heat transfer area. Ideally it should be in between 10 to 20%.
15. Calculate the cold fluid side and hot fluid side pressure drops by using the suitable correlations for spiral flow heat exchanger.

(a) Spiral flow with no phase change and $Re > Re_c$

$$\Delta p = 0.0789\frac{L}{\rho}\left[\frac{W}{d_s H}\right]^2\left[\frac{1.3\mu_f^{1/3}}{(d_s+0.0032)}\left(\frac{H}{W}\right)^{1/3}+1.5+\frac{16}{L}\right] \tag{6.136a}$$

(b) Spiral flow with no phase change and $100 < Re < Re_c$

$$\Delta p = 36.84\frac{L}{\rho}\left[\frac{W}{d_s H}\right]\left[\frac{1.035\mu_f^{1/2}}{(d_s+0.0032)}\left(\frac{H}{W}\right)^{1/2}\left(\frac{\mu_f}{\mu_b}\right)+1.5+\frac{16}{L}\right] \tag{6.136b}$$

(c) Spiral flow with no phase change and $Re < 100$

$$\Delta p = 5.5256\times 10^{-5}\left[\frac{L\rho\mu_f}{d_s^{2.75}}\right]\left(\frac{\mu_f}{\mu_b}\right)^{0.17}\left(\frac{W}{H}\right) \tag{6.136c}$$

(d) Axial flow with no phase change and $Re > 10\,000$

$$\Delta p = 253.85\left(\frac{\dot{m}}{L}\right)^{1.8}\left(0.0115\mu_f^{0.2}\frac{H}{d_s}+1+0.03\,H\right) \tag{6.136d}$$

where, L = Total length of plate, m
ρ = Density of fluid, kg/m^3
$\dot{m}$ = Mass flow rate of fluid, kg/s
d_s = Channel spacing (d_c or d_h), m
H = Width of plate, m
μ_f = Viscosity of fluid, kg / (m · s) or Pa · s
μ_b = Bulk fluid viscosity, kg/(m · s)

Calculated pressure drop must be less than or equal to maximum allowable pressure drop or optimum pressure drop.

Example 6.12 Design a spiral flow plate heat exchanger for cooling the malamine suspension liquid from 47°C to 33°C by using cooling water as cooling medium based on the following data. Cooling water is available at 32°C.

Data

(i)

Table 6.41 Composition of Malamine Suspension Liquid

Component	$\dot{m}$, kg/h	mass %	mole %
Water	16 052	57.21	74.2
Carbon dioxide	3 151	11.23	5.49
Ammonia	3 055	10.89	14.96
Urea	1 329	4.74	1.84
Melamine	4 470	15.93	2.95
Total	28 057	100.00	100.00

(ii) Physical properties of melamine suspension liquid
  (a) Specific heat $C_L = 3.349$ kJ/(kg · °C)
  (b) Thermal conductivity $k = 0.4935$ W/(m · °C)
  (c) Viscosity: At 47°C, $\mu_f = 1.8$ cP
  At 33°C, $\mu_f = 2.3$ cP
  (d) Density: At 47°C, $\rho = 1120$ kg/m^3
  At 33°C, $\rho = 1126$ kg/m^3
(iii) Suitable material of construction: SA 240 Gr 316 L
(iv) Maximum allowable pressure drops
  For malamine suspension liquid = 1.5 bar
  For cooling water = 1 bar

Solution

Heat duty $$\phi_t = \dot{m}C_L\Delta t = \left(28\,057 \times \frac{1}{3600}\right) \times 3.349 \times (47 - 33)$$
$$= 365.41 \text{ kW} \equiv 365.41 \times 10^3 \text{ W}$$

Mass flow rate of cooling water

Energy balance

Energy balance $\phi_t = \dot{m}_w\, C_{Lw}\, \Delta t' = \dot{m}_w \times 4.1868 \times (36 - 32)$

For the first trial calculations let the outlet temperature of cooling water = 36°C

$$\dot{m}_w = \frac{365.41}{4.1868 \times 4} = 21.819 \text{ kg/s} \equiv 78.55 \text{ m}^3\text{/h}$$

$$\Delta\mathcal{T}_{\text{lm}} = \frac{(47 - 36) - (33 - 32)}{\ln\left(\dfrac{47 - 36}{33 - 32}\right)}$$
$$= 4.17°\text{C} = \Delta\mathcal{T}_m$$

Fig. 6.49

Assuming the value of overall heat transfer coefficient
$$U = 1500 \text{ W/(m}^2 \cdot °\text{C)}$$

Heat transfer area
$$A = \frac{\phi_t}{U\Delta\mathcal{T}_m} = \frac{365.41 \times 1000}{1500 \times 4.17} = 58.42 \text{ m}^2$$

Heat transfer area provided for the first trial calculations
$$A_p = 58.42 \text{ m}^2 = 2 \times L \times H \tag{6.125}$$

Let $H = 24$ in $= 0.6096$ m
$$L = \frac{58.42}{2 \times 0.6096} = 47.9167 \text{ m}$$

To get the best compact design of spiral plate heat exchanger, width of plate (or length of heat exchanger) should be approximately equal to outside spiral diameter (or outside diameter of heat exchanger).

Let other dimensions of heat exchanger:

d_c = Channel spacing for cold side fluid = 12.7 mm

d_h = Channel spacing for hot side fluid = 6.35 mm

c = Core diameter = 203.2 mm

t = Plate thickness = 3.175 mm

Outside spiral diameter

$$D_s = [1.28 \times L(d_c + d_h + 2t) + c^2]^{1/2} \quad (6.121)$$

$$= [1.28 \times 47.9167(0.0127 + 0.006\,35 + 2 \times 0.003\,175) + 0.2032^2]^{1/2}$$

$$= 1.2646 \text{ m}$$

$$H \neq D_s$$

For second trial calculations let H = 36 in = 914.4 mm

$$L = 31.944 \text{ m}$$

$$D_s = [1.28 \times 31.944(0.0127 + 0.006\,35 + 2 \times 0.003\,175) + 0.2032^2]^{1/2}$$

$$= 1.04 \text{ m}$$

$$H \cong D_s$$

H = 914.4 mm, D_s = 1040 mm, L = 31.944 mm

d_c = 12.7 mm, d_h = 6.35 mm, C = 203.2 mm

t = 3.175 mm

Note: These dimensions of heat exchanger are not the final dimensions. These can be changed after heat transfer coefficient calculations and/or pressure drop calculations.

Hot fluid side heat transfer coefficient calculations:

Equivalent diameter

$$D_{eh} = \frac{4 \times (d_h \times H)}{2(d_h + H)} \quad (6.128a)$$

$$D_{eh} = \frac{4 \times (6.35 \times 914.4)}{2(6.35 + 914.4)} = 12.6 \text{ mm}$$

Reynolds number

$$Re = \frac{2\dot{m}}{H \times \mu} \quad (6.129)$$

$$\mu_f = \frac{1.8 + 2.3}{2} = 2.05 \text{ cP}$$

$$Re = \frac{2 \times 7.7936}{0.9144 \times 2.05 \times 10^{-3}} = 8315.3$$

Critical Reynolds number: $Re_c = 20\,000\left(\frac{D_{e_H}}{D_s}\right)^{0.32} = 20\,000\left(\frac{12.6}{1040}\right)^{0.32}$

$$= 4871.9$$

$Re > Re_c$, i.e., process fluid will flow in turbulent region.

For spiral flow with no phase change and $Re > Re_c$

$$\frac{h_h}{c_p G} = \left(1 + 3.5\left(\frac{D_{eh}}{D_s}\right)\right)(0.023\, Re^{-0.2} \times Pr^{-2/3}) \quad 6.131)$$

$$C_p = 3.349 \text{ kJ/(kg} \cdot \text{°C)}$$

Prandtl number, $$Pr = \frac{3.349 \times \frac{10^3}{1} \times (2.05 \times 10^{-3})}{0.4935}$$

$$= 13.912$$

Mass velocity, $$G = \frac{\dot{m}}{d_h \times H} = \frac{7.7936}{0.006\,35 \times 0.9144}$$

$$= 1342.234 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\frac{h_h}{3.349 \times 10^3 \times 1342.234} = \left(1 + 3.5\left(\frac{12.6}{1040}\right)\right) \times 0.023 \times 8315.3^{-0.2} \times 13.912^{-2/3}$$

$$h_h = 3063.9 \text{ W/(m}^2 \cdot \text{s)}$$

Cold fluid side heat transfer coefficient calculations:

$$De_c = \frac{4 \times (d_c \times H)}{2(d_c + H)} \quad (6.128b)$$

$$= \frac{4 \times (12.7 \times 914.4)}{2(12.7 + 914.4)} = 25.052 \text{ mm}$$

Viscosity of water at 34°C, $\mu = 0.738$ cP (Ref. 2)

Reynolds number $$Re = \frac{2\dot{m}}{H \times \mu} = \frac{2 \times 21.819}{0.9144 \times 0.738 \times 10^{-3}} \quad (6.129)$$

$$= 64\,665.44$$

Critical Reynolds number $$Re_c = 20\,000\left(\frac{De_c}{D_s}\right)^{0.32} = 20\,000\left(\frac{25.052}{1040}\right)^{0.32}$$

$$= 6070.28$$

$$Re > Re_c$$

For spiral flow with no phase change and $Re > Re_c$

$$\frac{h_c}{C_p G} = \left(1 + 3.5\left(\frac{De_c}{D_s}\right)\right)(0.023\, \text{Re}^{-0.2}\, \text{Pr}^{-2/3}) \quad (6.131)$$

$$C_p = 4186.8 \text{ J/(kg} \cdot \text{°C)}$$

Mass velocity $$G = \frac{\dot{m}}{d_c \times H} = \frac{21.819}{0.0127 \times 0.9144}$$

$$= 1878.9 \text{ kg/(m}^2 \cdot \text{s)}$$

Thermal conductivity of water at 34°C, $k = 0.62$ W/(m · k)

Prandtl number $$Pr = \frac{4186.8 \times 0.738 \times 10^{-3}}{0.62} = 4.9836$$

$$\frac{h_c}{4186.8 \times 1878.9} = \left(1 + 3.5\left(\frac{25.052}{1040}\right)\right) \times 0.023 \times 64665.44^{-0.2} \times 4.9836^{-2/3}$$

$$h_c = 7336.76 \text{ W/(m}^2 \cdot \text{°C)}$$

Overall heat transfer coefficient

$$\frac{1}{U_o} = \frac{1}{h_c} + \frac{1}{h_h} + \frac{1}{h_{c_d}} + \frac{1}{h_{h_d}} + \frac{t}{k_m} \tag{6.135}$$

where, $h_c = 7336.76$ W/(m^2 · °C), $h_h = 3063.9$ (m^2 · °C),
$h_{cd} = 15\,000$ W/(m^2 · °C), $h_{hd} = 15\,000$ W/(m^2 · °C),
$t = 0.003\,175$ m
$k_m = 16.26$ W/(m · °C) (of stainless steel)

$$\frac{1}{U_o} = \frac{1}{7336.76} + \frac{1}{3063.9} + \frac{1}{15\,000} + \frac{1}{15\,000} + \frac{0.003\,175}{16.26}$$

$$U_o = 1263.78 \text{ W/(m}^2 \cdot \text{°C)}$$

Heat transfer area required, $A_{r1} = \dfrac{\phi_t}{U \Delta \mathcal{T}_m}$

$$= \frac{365.41 \times 1000}{1263.78 \times 4.17} = 69.34 \text{ m}^2$$

Required area, A_{r1} > Area provided, A_{p1} which is not acceptable.
For the second trial calculations let $L = 42$ m
Area provided $A_{p2} = 2 \times L \times H$
$= 2 \times 42 \times 0.9144 = 76.81$ m^2
Revised value of D_s (outside diameter of spiral)

$$D_s = [1.28 \times L(d_c + d_h + 2t) + c^2]^{1/2}$$
$$= [1.28 \times 42(0.0127 + 0.006\,35 + 2 \times 0.003\,175) + 0.2032^2]^{1/2}$$
$$= 1.186 \text{ m}$$

Critical Reynolds number for hot fluid

$$Re_c = 20\,000\left(\frac{12.6}{1186}\right)^{0.32} = 4671.34$$

$$Re > Re_c$$

Revised value of hot fluid side heat transfer coefficient

$$h_h = \frac{\left(1 + 3.5\left(\frac{12.6}{1186}\right)\right)}{\left(1 + 3.5\left(\frac{12.6}{1040}\right)\right)} \times 3063.9$$

$$= 3048.56 \text{ W/(m}^2 \cdot \text{°C)}$$

Critical Reynolds number for cold fluid

$$Re_c = 20\,000\left(\frac{25.052}{1186}\right)^{0.32} = 5820.4$$

$$Re > Re_c$$

Revised value of cold fluid side heat transfer coefficient

$$h_c = \frac{\left(1 + 3.5\left(\frac{25.052}{1186}\right)\right)}{\left(1 + 3.5\left(\frac{25.052}{1040}\right)\right)} \times 7336.76 = 7266.53 \text{ W/(m}^2 \cdot \text{°C)}$$

Overall heat transfer coefficient

$$\frac{1}{U_o} = \frac{1}{3048.56} + \frac{1}{7266.53} + \frac{1}{15000} + \frac{1}{15000} + \frac{0.003\,175}{16.26}$$

$$U_o = 1259.07 \text{ W/(m}^2 \cdot \text{°C)}$$

Heat transfer area required

$$A_{r2} = \frac{365.41 \times 1000}{1259.07 \times 4.17} = 69.6 \text{ m}^2 < A_{p2}$$

Excess heat transfer area = $\frac{(76.81 - 69.6)}{69.6} \times 100 = 10.36\%$ (acceptable)

Malamine suspension liquid side pressure drop

$$\Delta p = 0.0789\frac{L}{\rho}\left[\frac{W}{d_s H}\right]^2\left[\frac{1.3\mu_f^{1/3}}{(d_s + 0.0032)}\left(\frac{H}{W}\right)^{1/3} + 1.5 + \frac{16}{L}\right] \qquad (6.136a)$$

$L = 42$ m, $\rho = 1123$ kg/m^3, $W = 7.7936$ kg/s

$d_s = d_h = 0.006\,35$ m, $H = 0.9144$ m

$\mu_f = 2.05 \times 10^{-3}$ kg/(m · s)

$$\Delta p = 0.0789 \times \frac{42}{1123}\left[\frac{7.7936}{0.006\,35 \times 0.9144}\right]^2$$

$$\left[\frac{1.3(2.05 \times 10^{-3})^{1/3}}{0.006\,35 + 0.0032} \times \left(\frac{0.9144}{7.7936}\right)^{1/3} + 1.5 + \frac{16}{42}\right]$$

$$= 55\,004.5 \text{ Pa} \equiv 55 \text{ kPa} \equiv 0.55 \text{ bar} < 1 \text{ bar} \qquad \text{(acceptable)}$$

Cooling water side pressure drop

$L = 42$ m, $\rho = 1000$ kg/m^3, $W = 21.819$ kg/s

$d_s = d_c = 0.0127$ m, $H = 0.9144$ m, $\mu_f = 0.738 \times 10^{-3}$ kg/(m · s)

$$\Delta p = 0.0789 \times \frac{42}{1000}\left[\frac{21.819}{0.0127 \times 0.9144}\right]^2$$

$$\left[\frac{1.3 \times (0.7378 \times 10^{-3})^{1/3}}{(0.0127 + 0.0032)}\left(\frac{0.9144}{21.819}\right)^{1/3} + 1.5 + \frac{16}{42}\right]$$

$$= 52\ 023.5 \text{ Pa} \equiv 0.52 \text{ bar} < 1 \text{ bar} \qquad \text{(acceptable)}$$

Comments: In the given case, malamine suspension liquid solution is a slurry solution. Hence, spiral plate heat exchanger is a better choice as compared to shell and tube heat exchanger because of the following reasons:

(i) Slurry solution must be allocated to shell side of shell and tube heat exchanger to facilitate the ease of flow. Shell and tube heat exchanger must be U-tube or floating head heat exchanger to facilitate removal of tube bundle and for frequent cleaning. Also, malamine solution required the use of special material, SS 316 L. Hence, shell, tubes, tube sheets, baffles, floating head and/or one of the heads must be of SS 316 L.

(ii) Shell and tube heat exchanger provides less heat transfer coefficient for the slurry solution as compared to spiral plate heat exchanger. Also, fouling coefficients are lesser with shell and tube heat exchanger compared to spiral plate heat exchanger. Hence, shell and tube heat exchanger requires more heat transfer area.

(iii) Cleaning is easier with spiral plate heat exchanger but requires skill.

(iv) Because of the compactness spiral plate heat exchanger will occupy less space as compared to shell and tube heat exchanger.

(v) Here cooling water is available at 32°C. It can cool the malamine solution up to 33°C with spiral plate heat exchanger, wherein driving force is only 1°C. Minimum driving force required for heat transfer in shell and tube heat exchanger is 3°C.

Based on (i) and (ii) it can be concluded that the cost difference between spiral plate heat exchanger and shell and tube heat exchanger is less.

6.17 BRAZED ALUMINUM PLATE-FIN HEAT EXCHANGER

Typical brazed aluminum plate-fin heat exchanger handling multiple streams is shown in Fig. 6.50. This type of heat exchanger is preferred for low temperature (subzero temperature) cryogenic applications as it offers the following features:

1. It can handle temperature approaches down to less than 2°C. In cryogenic duties, operating cost is dominated by the cost of energy required to generate the low temperatures; hence, such a close temperature approach is very important.
2. It is the most compact and lightest heat exchanger and it provides the maximum heat transfer area density.
3. It can operate with a thermal effectiveness up to 98%.
4. It has multistream capability. All cold streams produced in process can be passed through single heat exchanger to cool the incoming warm streams.
5. It is an expensive heat exchanger.

Fig. 6.50 *Brazed Aluminum Plate-fin Heat Exchanger*[20]

6.17.1 Construction and Working

It consists of alternating layers of plates and corrugated fins. Flow passes are formed between the consecutive plates (plates are known as *parting sheets*). These sheets provide the primary surface and fins provide the secondary surface for heat transfer.

Brazed or corrugated fins hold the heat exchanger together. At the end of heat exchanger, pads of finning are laid at an angle and serve as distributors, while in major part of heat exchanger finning are laid parallel to the axis. Streams are flowing truly in countercurrent directions. Each stream flows in number of layers, each of which is divided into numerous parallel nearly rectangular subchannels by the fins. Fin heights and frequencies decide the size of these subchannels. Fin heights are in between 5 and 9 mm and fin frequencies in the main heat transfer region is 590 to 787 fins/m (15 to 20 fins/in). The equivalent hydraulic diameters of these subchannels are only a few millimeters. These small passages result in very high heat transfer area density in the range of 800 to 1500 m^2/m^3. For comparison, heat transfer area density of shell and tube heat exchanger is about 50 to 250 m^2/m^3 and the same of plate and frame heat exchangers are about 200 to 1000 m^2/m^3. Such high area density and with aluminum construction make this heat exchanger the smallest as well as the lightest among all other type of heat exchangers. Typical size of such a heat exchanger can be 1.2 m wide, 1.2 m deep and 6.2 m long. This heat exchanger can also be used as vaporizer or condenser.

Brazed aluminum plate heat exchangers can be used at pressures up to 100 bar and within a temperature range of –269°C to 100°C. With appropriate alloys for the headers and nozzles, they can be used up to 200°C. However, the maximum permissible temperautre for aluminum alloys decreases rapidly with increasing pressure.

6.18 OTHER TYPES OF HEAT EXCHANGE EQUIPMENTS

Several other types of heat exchange equipments are in use in chemical process industry. To name a few; serpentine coolers, scraped surface heat exchangers, helical coil-type heat exchangers, etc. Each one has specific area in which they are used extensively due to convenience. For example, serpentine coolers are used for condensing ammonia vapour in cold storages. Similarly for cooling Vanaspati (hydrogenated edible oils), crystallizing menthol from its solution, wax cooling, manufacture of ice cream, and for freeze crystallization applications, scrapped surface heat exchangers are extensively used. Heat transfer in jackets and half coils (of CSTR) with agitators are covered in Chapter 10. Process engineer should evaluate all possible designs for selecting a best suited equipment for a given application. Due to space limitations, it is not possible to cover process design of all types of heat exchange equipments in this book but reader is advised to refer literature for specific equipment design.

6.19 HEAT EXCHANGER NETWORKING FOR ENERGY INTEGRATION

Energy requirement of a chemical plant can be considerably reduced by process integration. For example, in acetaldehyde plant, exothermicity of the reaction (e.g., partial oxidation of ethanol) is utilized as a part of heat duty required by the reboiler of the distillation column of the same plant. This integration reduces saturated steam required for the reboiler of distillation column and eliminates the requirement of cooling medium for the reactor. Large scale chemical plant uses large number of heat exchangers. These heat exchanges are either heaters and vaporizers or coolers and condensers. Integration between few of them reduces hot utility requirement (i.e., requirement of heating medium) as well as cold utility requirement. (i.e., requirement of cooling medium). Systematic method is developed by Linnhoff and others[21] for the energy integration via systematic heat exchanger networking which is known as *pinch technology*.

In heat exchanger networking via pinch technology, following facts are considered:

1. The heat transferred from a hot stream must be equal to heat transferred from cold stream (heat losses are neglected).
2. Heat can only be transferred from a hotter fluid to a colder fluid (second law of thermodynamics). Therefore, the temperature of a cold fluid must be less than that of a hot fluid at all points along the path of heat exchanger.

ΔT_{min}: In actual energy integration temperature of hot stream must be reasonably greater than the same of cold stream to get an acceptable rate of heat transfer. This acceptable minimum temperature difference is also called *pinch temperature difference*. Choice of pinch temperature difference (ΔT_{min}) is important. The lower value of ΔT_{min} decreases the energy requirement but increases the fixed cost of heat exchangers. Minimum suggested value of ΔT_{min} is 10°C. This implies that in any of the exchangers to be used in the network, the temperature difference between the hot stream and cold stream will **not** be less than 10°C. For any network, there will be a best value for the minimum temperature difference (ΔT_{min}) that will give the lowest total annual operating cost. It is usually in between 10°C to 20°C.

"Hot streams" and "Cold streams": For heat exchanger networking total streams considered are divided into two types. "Hot streams" are the streams that need cooling (heat sources) while "Cold streams" are the streams that need heating (heat sinks).

6.19.1 Representation of Heat Exchanger Network

Heat exchanger network is represented as a grid. The process streams are drawn as horizontal lines, with the stream numbers shown in square boxes, as shown in Fig. 6.51. Hot streams are drawn at the top of grid and flow from left to right. The cold streams are drawn at bottom and flow from right to left.

Fig. 6.51 *Grid Presentation*

Coolers and heaters are represented as circles; (C) and (H), respectively. Heat exchangers which are used to exchange the heat between the two process streams are marked by two circles and the two circles are connected by vertical line. Two circles with vertical line connect the two streams between which heat is being exchanged.

As shown in Fig. 6.51, in this case a is hot stream number, n is cold stream number. A is a heat exchanger exchanging the heat between hot stream a and cold stream n. Cooler C cools the stream further after it leaves the heat exchanger A. Heater H heats the stream n after it leaves the heat exchanger A.

Calculations for finding minimum utility requirements for the given stream data: First step for this is selection of the value of minimum temperature difference, ΔT_{min} between hot streams and cold streams. Then for the chosen value of ΔT_{min}, minimum utility required can be determined by the following method, developed by Hohmann[22] and Lockhart. This method is illustrated by following example.

Example 6.13 Synthesize the heat exchanger network for the following four process streams such that resulting heat exchanger network will require the minimum hot and cold utilities. Also, find the values of minimum utilities (hot and cold) required.

Table 6.42 Process Stream Data

Stream	Type	$\dot{m}C_p$, kW/°C	t_{in}, °C	t_{out}, °C
1	Hot	10	250	130
2	Hot	8	160	90
3	Cold	7	60	160
4	Cold	6	110	260

Solution

Select the value of ΔT_{min} = 10°C. (Selected value of ΔT_{min}, may not be equal to optimum temperature difference.) For ΔT_{min} = 10°C, Table 6.43 is prepared by calculations, presented below.

Method for Preparing the Table:

The hottest temperature in the data is cold stream 4 outlet temperature (260°C). List it at the top of the cold stream temperature column (column 2). Note down the corresponding hot stream temperature (270°C), which is 10°C (= ΔT_{min}) hotter at the top of the hot stream temperature column. Next highest temperature is of hot stream 1 temperature; 250°C. Corresponding cold stream temperature based on ΔT_{min} = 10°C is 240°C and written second in column 2. Similarly, temperatures of hot streams and cold streams are written in descending order either in column 1 (if it is hot stream temperature) or in column 2 (if it is cold stream temperature). While corresponding temperatures for ΔT_{min} = 10°C are written in other columns.

There are seven temperature intervals in this problem. In column 3 available heat by hot streams for each temperature interval is calculated. No hot stream is available at 270°C, hence, available heat for seventh interval is zero. Since hot stream temperature of 270°C is of academic interest only as it is shown in brackets in column 1.

For sixth interval (in column 3) available heat is calculated as $(\dot{m}C_p)_1$ (250°C–170°C) = 10 (250 – 170) = 800 kW. So, in sixth interval hot stream 1 is cooled from 250°C to 170°C. Stream 1 is also the only hot stream in interval 5, contributing another 100 kW, when it cools from 170°C to 160°C. Interval 4 has both hot streams 1 and 2. In interval 4 both streams are cooled from 160°C to 130°C. Hence available heat for interval 4 is calculated as follows.

$$[(\dot{m}C_p)_1 + (\dot{m}C_p)_2]\ (160°C - 130°C) = ((10 + 8) \times 30) = 540 \text{ kW}$$

In column 4 cascaded heat available indicates cumulative or total heat available at temperature t, when hot streams cool from 250°C to temperature t°C.

In column 5 required heat by cold streams for each temperature interval is calculated. Stream 4 is present only in interval 7 and requires 120 kW heat to increase its temperature from 240°C to 260°C, $(\dot{m}C_p)_4$ (260 – 240) = 6(260 – 240) = 120 kW.

In interval 5 both streams 3 and 4 are present. Hence, required heat for interval 5 is $[(\dot{m}C_p)_3 + ((\dot{m}C_p)_4]$ (160 – 150) = ((6 + 7) × 10) = 130 kW. Cascaded required heat by cold streams at different temperatures are shown in column 6.

Column 7 represents the net available heat for each interval.

Net available heat (column 7) of any interval = Heat available with hot streams (column 3) in this interval – Heat required by cold streams (column 5) in the same interval.

Column 8 represents the cascaded net available heat or cumulative net available heat at the end of each interval, starting from the seventh interval to first interval. Cascaded heat is the amount of heat available from the hot streams over that required by cold streams at any temperature t, as one starts the calculation from the highest temperature to the lowest temperature. Find the highest negative number in column 8. In this example it is –120 kW. It implies that 120 kW is the minimum amount of heat that must be supplied from hot utilities. The same amount of heat (120 kW) is added in each number of eighth column. This gives ninth column. The point in the

Table. 6.43 Cascaded Heat Flow Calculations for Example 6.13

1		2	3	4	5	6	7	8	9
Hot stream temperature ° C	Interval	Cold streams temperature ° C	Composite avail. heat kW	Hot streams cascaded heat kW	Composite reqd. heat kW	Cold streams cascd. heat kW	Grand composite net heat kW	Hot and cold streams	
								Cascaded heat kW	Adjusted cascaded heat, kW
(270°C)		260°C				0		0	120
	7		0		120		–120		
250° C		(240°C)		0		120		–120	0
	6		800		480		320		
(170° C)		160°C		800		600		200	320
	5		100		130		–30		
160°C		(150°C)		900		730		170	290
	4		540		390		150		
130°C		(120°C)		1,440		1,120		320	440
	3		80		130		–50		
(120°C)		110°C		1,520		1,250		270	390
	2		240		210		30		
90°C		(80°C)		1,760		1,460		300	420
	1		0		140		–140		
(70°C)		60°C		1,760		1,600		160	280

last column where the value of adjusted cascaded heat is zero is the pinch point. Top number in last column (120 kW), is the minimum amount of heat that must be supplied from hot utilities and the bottom number (280 kW) is the minimum amount of heat that must be removed by cold utilities.

6.19.2 Grand Composite Curve (GCC)

It is an important curve or graph to understand heat exchanger network synthesis problem. To draw this curve for the given problem, Table 6.43 must be prepared, based on given data. Then GCC is the plot of adjusted cascaded heat obtained in the last column (ninth column) vs hot stream/cold stream temperatures. (Columns 1 and 2). GCC for this example is shown in Fig. 6.52.

Fig. 6.52 *Grand Composite Curve for Example 6.13*

In this curve each interval is represented by line segment. Each interval is either consuming heat (heat sink) or producing heat (heat source). Interval which acts as a heat sink, represented by a line segment that moves down and to the left. Interval which acts as a heat source, represented by a line segment that moves down and to the right. For example, seventh interval locally requires 120 kW of heat input (heat

sink), hence it is represented by a line segment which moves downward and to the left. Interval 6 produces 320 kW of heat (heat source), hence it is represented by line segment moving downward and to the right.

Whenever there is a heat source segment just above a heat sink segment right facing noze appear in the Grand Composite Curve. Such right facing nozes can be self-integrated. While the bold segment above the pinch point requires heat from hot utilities and bold segment below the pinch up to the dashed line represents the heat which must be removed by cold utilities.

Heat exchanger network synthesis for the maximum energy recovery:

Network design for the maximum energy recovery and minimum use of hot and cold utilities can be divided in the following steps:

1. Select the value of $\Delta T_{\min}$.
2. Prepare the table (like Table 6.43) for drawing the Grant Composite Curve.
3. Calculate the minimum hot utility required and the minimum cold utility required. Identify the pinch point.
4. For the maximum heat recovery do not use cold utilities above the pinch and do not use hot utilities below the pinch. Also, do not transfer heat across the pinch.
5. Estimate the number of exchangers above the pinch equal to $N - 1$, where N is the number of streams above the pinch. Similarly, estimate the number of exchangers below the pinch equal to $M - 1$, where M is the number of streams below the pinch.
6. Develop a suitable network.

Applying the same steps to the given Example 6.13:

1. $\Delta T_{\min} = 10°C$ (selected)
2. GCC is drawn based on the value of ΔT_m (Fig. 6.52).
3. Minimum hot utility required is to supply 120 kW heat and the minimum cold utility required is for removing 280 kW heat. Pinch point occurs at 250°C hot stream temperature and equivalent cold stream temperature of 240°C.
4. Above the pinch only stream 4 exists. Hot oil (another stream) can be used to increase the temperature of stream 4 from 240°C to 260°C. Thus $N = 2$ which means one heat exchanger is needed for this heat duty. Heat duty of this heater is 120 kW. Below the pinch all four process streams and one cold utility stream exists ($M = 5$). Thus there is a need of four exchangers for this part or below the pinch.
5. One possible heat exchanger network for the maximum energy recovery is shown in Fig. 6.53.

Hot stream 1 contains $(\dot{m}C_p)\ \Delta t = 10 \times (250 - 130) = 1200$ kW heat. Part of this heat can be utilised to increase the temperature of stream 4 from 110°C to 240°C by using the heat exchanger A. Heat duty of heat exchanger A is $6 \times (240 - 110) = 780$ kW. Stream 1 is divided in two parts a and b. Stream a is used to heat stream 4.

$$(\dot{m}C_p)_{1a} = \frac{780}{(250 - 130)} = 6.5 \text{ kW/°C}$$

$$(\dot{m}C_p)_{1b} = 10 - 6.5 = 3.5 \text{ kW/°C}$$

Stream	1a	1b	2	3	4
$(\dot{m}C_p)$	6.5	3.5	8	7	6
Total Heat Flow, kW	780	420	560	700	900

Fig. 6.53 *Heat Exchanger Network for Example 6.13*

Hot stream 2 contains $(\dot{m}C_p)\ \Delta t = 8 \times (160 - 90) = 560$ kW heat. The same can be used to increase the temperature of cold stream 3 from 60°C to 140°C, i.e., $[7 \times (140 - 60)] = 560$ kW. For this heat exchange, heat exchanger *B* is used.

To heat the stream 3 from 140°C to 160°C, heat duty required is $[7 \times (160 - 140)] = 140$ kW. Same can be furnished by hot stream 1b. This is done in heat exchanger *C*. Outlet temperature of hot stream 1 from heat exchange *C* is t_o.

Then $$(\dot{m}C_p)_b\,(250 - t_o) = 140$$
$$3.5\,(250 - t_o) = 140$$
$$t_o = 210°C$$

Remaining heat of hot stream 1b is $[3.5 \times (210 - 130)] = 280$ kW. If there exists an opportunity to heat demineralised water for boiler feed make-up purpose, 280 kW heat exchange can be effected in an economiser (*EC*). If such an opportunity is not available, stream 1b can be cooled to 130°C with the help of cooling water in a cooler.

Stream 4, available at 240°C from heat exchanger, can be heated to the desired temperature of 260°C in a heater (*H*) with the help of circulating thermic fluid.

In all there will be 5 heat exchangers in the network; *A*, *B*, *C*, *H* and *EC*, the last two use hot and cold utilities, respectively.

Comments:
Here stream 1 is splitted into two parts. Stream splitting has one advantage and two disadvantages.
Advantage: Stream splitting removes the use of one extra heat exchanger in network. Here if the stream 1 is not splitted, then total number of heat exchangers required in network will be 6 instead of 5.
Disadvantages:
(i) In stream splitting, control of the flows in two branches is required.
(ii) Splitting a stream means each branch has a lower flow rate than that for the entire stream. In case of no phase change heat transfer, lower flow rate means lower heat transfer coefficient and larger exchanger area.

Suggested heat exchanger network design is based on the value of $\Delta\mathcal{T}_{min}$ = 10°C. It is not the optimum design. To arrive at the optimum network design detailed cost analysis is required for the different values of $\Delta\mathcal{T}_{min}$. The value of $\Delta\mathcal{T}_{min}$ which gives the minimum total cost is the optimum $\Delta\mathcal{T}_{min}$, and design of heat exchanger network based on that is optimum network design. A simple computer program can be developed for carrying out iterative calculations, varying $\Delta\mathcal{T}_{min}$ values.

Example 6.14 Design the suitable heat exchanger network for atmospheric crude fractionation unit based on the data given in Table 6.44[22, 23].

Solution

1. Selection of pinch temperature difference: Fractions of crude petroleum oil provides relatively lower heat transfer coefficients. Hence, the optimum value of $\Delta\mathcal{T}_m$ is relatively higher. Select the value of $\Delta\mathcal{T}_m$ = 20°C.

Table 6.44 Table of Stream Data (Ref. 23, 24)

Stream No.	Name of stream	Temperature in, °C	Temperature out, °C
1	Fuel oil	349	90
2	Gas oil	341	65
3	Kerosene	268	38
4	Reflux	251	77
5	Heavy Naphtha	235	38
6	Light Naphtha (Main)	168	71
7	Light Naphtha (Pre flash)	136	71
8	Desalted feed	15.6	121
9	Pre-flash feed	120	194
10	Crude tower feed	189	368

Table 6.45 $\dot{m}C_p$ Data of Streams (Ref. 23, 24)

Stream No.	Temperature range, °C			$\dot{m}C_p$, MW/°C
1	349	to	243	0.215
	243	to	213	0.197
	213	to	167	0.178
	167	to	90	0.168
2	341	to	210	0.105
	210	to	172	0.1
	172	to	111	0.087
	111	to	65	0.076
3	268	to	135	0.065
	135	to	38	0.054
4	251	to	169	0.105
	169	to	77	0.091
5	235	to	127	0.008
	127	to	38	0.007
6	168	to	136	0.6
	136	to	118	0.478
	118	to	108	0.41
	108	to	71	0.303
7	136	to	118	0.256
	118	to	108	0.21
	108	to	71	0.159
8	15.6	to	121	0.379
9	120	to	122	0.4
	122	to	163	0.422
	163	to	186	0.6
	186	to	194	0.725
10	189	to	237	0.477
	237	to	265	0.496
	265	to	368	0.66

2. Table is to be prepared for drawing Grand Composite Curve.

Sample calculations for available heat in column 3 for the interval of temperature change of hot streams from 214°C to 168°C: In this interval first five streams exist (all hot streams from 1 to 5). Cold stream 10 also exists in this interval. But, it cannot be considered in calculations of available heat (with hot streams).

Available heat within this interval

= 0.197 (214 – 213) + 0.178 (213 – 168) (of Stream 1) + (0.105 (214 – 210) + 0.1 (210 – 172) + 0.087 (172 – 168)) (of Stream 2) + (0.065 (214 – 168)) (of Stream 3) + [0.105 (214 – 169) + 0.091 (169 – 168)] (of Stream 4) + 0.008 (214 – 168) (of Stream 5)

= 20.949 MW.

In column 4, available heat indicates the cumulative or total heat available from the hot streams at temperature t, when all hot streams (Stream No. 1 to 7) cool from the hottest temperature 388°C to the temperature t°C.

In column 5, required heat by cold streams for each temperature interval is calculated.

Sample calculations for the required heat in column 5 for the interval of temperature from 148°C to 194°C of cold streams are as follows. In this interval two cold streams; No. 9 and No. 10 exist. Required heat by cold streams in this interval

= [(194 – 186) × 0.725 + (186 – 163) × 0.6 + (163 – 148) × 0.422] (of Stream 9) + [(194 – 189) × 0.477] (of Stream 10)

= 28.315 MW

In column 6, cascaded required heat by cold streams at temperature t' indicates the total heat required by the cold streams to increase the temperature of cold streams from t' to the maximum required temperatures:

Column 7 represents the net heat available for each interval.

Net heat available (column 7) = Heat available with hot streams (column 3) – Heat required by cold streams (column 5)

Column 8 represents the cascaded or cumulative net heat available at any temperature t. The most negative number in column 8 is –60.344 MW. It implies minimum amount of heat that must be supplied from the hot utilities. The same amount of heat 60.344 MW, is added in each number of 8th column. Resulting numbers are written in the last column (column 9) as adjusted heat in MW. The point in the last column where the adjusted cascaded heat is zero, represent the pinch point. Topmost number in the last column, 60.344 MW is the minimum amount of heat that must be supplied from the hot utilities and the bottommost number of last column; 44.0974 MW is the minimum amount of heat that must be removed by cold utilities.

3. Grand Composite Curve (GCC):

For this example, GCC is shown in Fig. 6.54. Pinch point occurs at 168°C of hot streams temperature and 148°C of cold streams temperature.

4. Hot streams and cold streams exist above the pinch and below the pinch are shown in Fig. 6.54.

For the maximum heat or energy recovery cold utilities cannot be used above the pinch and hot utilities cannot be used below the pinch. Also, heat cannot be transferred across the pinch.

Figure 6.55 also shows heat load (heat available or required) above and below the pinch.

Sample calculation of heat load above the pinch for Stream No. 1:

Heat load above the pinch for Stream No. 1 = $\sum(\dot{m}C_p)_i\,\Delta t_i$

= 0.215 (349 – 243) + 0.197 (243 – 213) + 0.178 (213 – 168)

= 36.71 MW

Fig. 6.54 *Grand Composite Curve for Example 6.14*

Above the pinch design

For the maximum energy recovery cold utility cannot be used above the pinch. But hot utility can be used above the pinch. One of the possible network designs above the pinch is shown in Fig. 6.56. Here five hot streams and two cold steams exist above the pinch. Heat available with Stream No. 5 above the pinch is 0.536 MW and is very low compared to heat loads (heat available or required) of other streams existing above the pinch. Hence the heat exchange above the pinch heat load of Stream No. 5 is not considered but the same load is transferred or included in the design of network below

the pinch. Here, Stream No. 9 (cold stream) is splitted into four streams to exchange the heat with four hot streams. Stream No. 10 (cold stream) is splitted into two streams to exchange the heat with two hot streams. Streams are splitted near the pinch while heater *H* is designed far away from the pinch.

Table 6.46 Cascaded Heat Flow Calculations for Example 6.14

1	2	3	4	5	6	7	8	9
Hot stream temperature °C	**Cold stream temperature °C**	**Available heat MW**	**Cascaded heat MW**	**Required heat MW**	**Cascaded heat MW**	**Net heat MW**	**Cascaded heat MW**	**Adjusted cascaded heat MW**
(388)	368		0		0		0	60.344
		0		25.74		–25.74		
349	(329)		0		25.74		–25.74	34.604
		1.72		5.28		–3.56		
341	(321)		1.72		31.02		–29.3	31.044
		23.36		45.392		–22.032		
268	(248)		25.08		76.412		–51.332	9.012
		6.545		8.318		–1.773		
251	(231)		31.625		84.73		–53.105	7.239
		7.696		7.632		0.064		
235	(215)		39.321		92.362		–53.041	7.303
		10.08		10.017		0.063		
214	194		49.401		102.379		–52.978	7.366
		20.949		28.315		–7.366		
168	(148)		70.35		130.694		–60.344	0
		27.523		11.372		16.151		
(141)	121		97.873		142.066		–44.193	16.151
		1.019		0.779		0.24		
(140)	120		98.892		142.845		–43.953	16.391
		4.076		1.516		2.56		
136	(116)		102.968		144.361		–41.393	18.951
		48.165		17.434		30.731		
90	(70)		151.133		161.795		–10.662	49.682
		8.97		4.927		4.043		
77	(57)		160.103		166.722		–6.619	53.725
		3.594		2.274		1.32		
71	(51)		163.697		168.996		–5.299	55.045
		0.822		2.274		–1.452		
65	(45)		164.519		171.27		–6.751	53.593
		1.647		10.233		–8.586		
38	(18)		166.166		181.503		–15.337	45.007
		—		0.9096		–.9096		
(35.6)	15.6		166.166		182.4126		–16.2466	44.0974

Fig. 6.55 *Division of Hot Streams and Cold Stream around Pinch Point for Example 6.14*

Below the pinch design

For the maximum energy recovery hot utility cannot be used below the pinch (Refer to Fig. 6.57). If the stream splitting is required, then split it near the pinch. Design the heat exchangers which utilize the utilities far away from the pinch. Here, below the pinch, Stream No. 9 (cold stream) is again splitted at the pinch into four streams to exchange the heat with the same four streams, from Stream No. 1 to 4. Another cold stream, Stream No. 8, is also splitted into two streams to exchange the heat with Stream No. 1 and Stream No. 6. At the end, Stream No. 9 exchanges the heat with Stream No. 5. Total six coolers are designed, far away from the pinch, at the end of network to meet the process requirements.

Complete design

Complete design of heat exchanger network for the maximum energy recovery is obtained by merging the two network designs, above and below the pinch. To reduce the number of heat exchangers used for Stream No. 9 above the pinch, *C*, *D*, *E* and

Fig. 6.56 *Network Design above the Pinch for Example 6.14*

Fig. 6.57 *Network Design below the Pinch for Example 6.14*

Fig. 6.58 *Combined Network for Example 6.14*

F are combined with *C*2, *D*2, *E*2 and *F*2, respectively. *C*2, *D*2, *E*2 and *F*2 are the heat exchangers used below the pinch. For these combinations minor adjustments are done in the temperatures of branch streams. Combined design or completed design is shown in Fig. 6.58.

6.19.3 Limitations of Heat Exchanger Networking

Heat exchanger networking is normally carried out on the basis of available data on heat sources/sinks. However, in actual practice, certain overriding considerations need attention which may exclude certain services from networking. Following limitations need be carefully evaluated for each service before being considered for networking:

1. Networking makes the system more complex and therefore control of the system is more difficult.
2. Networking requires additional connecting piping and fittings. These pipings may introduce additional pressure drops in the systems; thereby networking of long distance heat exchangers may not be economically feasible.
3. Failure of any one heat exchanger can deteriorate the performance of entire network.
4. Networking may call for lower $\Delta\mathcal{T}_m$ of heat exchangers which may call for high fixed cost due to bigger size of heat exchangers.
5. 'Odd' heat exchanger should not be included in networking. Any heat exchanger requiring special or costlier material of construction or that provides very low or very high heat transfer coefficients in comparison to other heat exchangers of network should not be included in the network.
6. Many networking of heat exchangers involve stream splitting (refer to Example 6.14). Stream splitting of liquid-vapour mixed stream (i.e., two-phase flow) is difficult. Other disadvantages of stream splitting are mentioned in Example 6.13.
7. A specific heat exchanger of network may be needed during start-up or shut-down. During this period, heating/cooling service of the heat exchanger may not be available and therefore additional service will be required to be provided in it. This will increase the cost of networking system.

6.20 HEAT TRANSFER IN SOLIDS

Unlike heat transfer in liquids and gases, heat transfer in solids takes place chiefly by conduction. Cooling of solids is often required after a dryer, a calciner, a roaster or a combustor. Metal powders, burnt lime, coal, etc., are cooled from high temperature (in excess of 500°C). Urea, furnace grade carbon black, etc., require cooling after drying. Pharmaceutical products (fine powder) require cooling after drying. In food processing, coffee beans are cooled after roasting. Beans, corn and peas are frozen by cooling with refrigerated air for preservation.

Cooling of hygroscopic solids is required for more than one reason. Granulated or prilled fertilizer and carbon black with some moisture may agglomerate on natural cooling due to moisture migration in silo. This results in lump formation. These lumps are quite hard and when broken by mechanical means produce dusty material making it

unsuitable for end use. However, caking tendency is not observed in the same material if it is cooled (say below 60°C) in absence of moisture.

Several technologies are available for cooling solids. Although compact plate-type heat exchangers are developed for cooling of solids, their application is limited due to cost and possible adherence of sticky solids to the heat exchanger surface.

Direct contact coolers with or without indirect cooling (with cooling water) are popular for cooling of solids. Adham[25] has described various solids cooling technologies at length. Heat recovery from hot gases, coming out of the cooler is possible depending on temperature of gases and its possible use elsewhere in the plant. Chiefly, four types of coolers are in use for most applications of solids cooling.

1. Stationary cooler
2. Rotary cooler
3. Fluid bed cooler
4. Conveyor cooler

6.20.1 Stationary Coolers

Simple version of a stationary cooler (Fig. 6.59) has a bin with internal cooling surface. It is recommended for free flowing material and is unsuitable for wet, very fine and sticky powders. Overall heat transfer rate in the cooler is quite low and hence, larger cooling surface is required.

Fig. 6.59 *Stationary Cooler with Water Cooled Plates*

Solids travel in a near plug flow manner and a temperature gradient from top to bottom is observed. Stationary coolers are recommended for moderate inlet temperature (say 300 to 400°C) and for heat duties not exceeding 150 kW. Free flowing powder up to 100 μm size can be cooled in such a cooler. Barring these limitations, stationary coolers are compact, require minimum or no auxiliary equipment, require no off-gas

treatment and are simple to operate. Cooling up to 60 to 70°C should be possible with this cooler with circulating water maintained at a temperature of 30 to 35°C.

6.20.2 Rotary Coolers

This versatile cooler can handle different type of solids and can be used as direct or an indirect cooler with or without heat recovery. Although it can be used for a wide range of solids, it is a preferred equipment for coarse particles. In this types of coolers, hot air (or gas) flows in counter current direction of solids, increasing the thermal efficiency. Cooling coils can be embedded in the bed to provide indirect cooling. Heat from the hot air, coming out from the cooler, can be recovered in a number of ways.

Figure 6.60 is a simple sketch of a rotary cooler. Diameter of the cooler is restricted below 3 m for the considerations of fabrication and transportation. Drive mechanism for the cooler also imposes restrictions on the dimensions. Length-to-diameter ratio is kept in the range of 4 to 10. To avoid passing of solids without coming in contact with air, depth of material is kept in the range of 30–40% of radius. Such equipment can be used for drying as well as cooling. In carbon black industry, ceramic industry, metal powder industry, etc., this equipment is popular.

Fig. 6.60 *Rotary Cooler with Direct Air and Spray Water Cooling*

6.20.3 Fluid Bed Cooler

Coarse small particles (below 2–2.5 mm) can be fluidized with air or gas. However, very fine powders (less than 10 μm) resists smooth fluidization. So fluidized bed cooler can be considered for the intermediate size solids. In Fig. 6.61, four-stage fluid bed cooler with embedded cooling coils is shown.

Spray cooling is an option both in rotary as well as fluid bed coolers but it has to be executed with caution. Hot air wet bulb temperature should not be allowed to exceed 80°C and fluid or moving bed temperature is not allowed to fall below 150°C to prevent moisture absorption by the solids.

Fluid bed cooler can handle free flowing and non-sticky solids. Heat transfer rate is excellent in a fluid bed cooler among all solid-cooling equipments. However, it needs an auxiliary equipment such as a cyclone or a bag filter for off-gas treatment to trap fines. This equipment is relatively simple in operation and easy to maintain.

Fig. 6.61 *Four Stage Fluidized Bed Cooler with Indirect Water Cooling*

Removal of fines from the solids may be considered as an advantage. The product after cooling is free from dust and its particle size distribution is in the narrow range making it a preferred marketable product.

6.20.4 Conveyor Coolers

Variety of conveyor coolers are available. Vertical spiral cooler, moving grate cooler, vibrating pan cooler, screw conveyor coolers, etc., are a few of them. The screw conveyor cooler is normally used for indirect cooling of solids with cooling water which flows in the conveyor shell jacket and the hollow shaft. Screw conveyor coolers can be used for free flowing solids, normally coarse in nature (0.1–12 mm). Screw conveyor coolers can be advantageously used for cooling as well as conveying. Single or multiple screw designs are available but double screw design is popular up to a maximum length of 8.5 m.

Process design of cooling equipments of solids can be well explained by the following example.

Example 6.15 Urea prills from a prill tower in a fertilizer plant have a temperature of 100°C and moisture content of 1.5 to 2% (by mass). They need to be cooled to 60°C before sending to silo for storage to prevent caking and lump formation. Average sieve analysis of prills is given in Table 6.47.

Table 6.47 Typical Distribution of Urea Prills by Size

Size of prills, mm	mass %
+2.4	0.8
+2.0	9.0
+1.7	56.3
+1.4	26.6
+1.0	7.3

Physical properties of urea:

Particle density, $\rho_s = 1.335$ kg/L

Bulk density, $\rho_{sb} = 0.758$ kg/L (without compaction)

Thermal conductivity, $k_s = 80$ W/(m · °C)

Heat capacity, $C_s = 1.775$ kJ/(kg · °C)

I. Cooling Mediums

(a) **Circulating condensate** is proposed to be used in the stationary cooler to avoid possibility of fouling in the plates. Supply and return temperatures may be taken as 35 and 45°C, respectively. Hot condensate is passed through an external shell and tube heat exchanger in which it is cooled to 35°C with the help of cooling water of 30°C which is recirculated.

(b) **Ambient air** at 44°C is to be used in rotary and fluid bed coolers. Outlet air temperature from the coolers may be fixed at 75°C.

Work out process design of stationary, rotary and fluid bed coolers to process 60 t/h urea prills. While there is hardly any change in moisture content in the stationary cooler, moisture content of prills gets reduced to 0.5 mass % in rotary and fluid bed coolers.

Overall heat balance:

$$\text{Average temperature of air} = \frac{75 + 44}{2}$$
$$= 59.5°\text{C}$$
$$\equiv 332.65 \text{ K}$$

$$\text{Molar volume of air} = \frac{22.414 \times 332.65}{273.15}$$
$$= 27.296 \text{ m}^3/\text{kmol}$$

Density of air, $$\rho_g = \frac{28.96}{27.296} = 1.061 \text{ kg/m}^3$$

Moisture removal of urea = 60 000 (0.015 – 0.005) = 600 kg/h

Total heat load of prill cooling in rotary and fluid bed coolers,

$$\phi_1 = 60\,000 \times 1.775\,(100 - 60) + 600 \times 2644$$
$$= 4260\,000 + 1586\,400$$
$$= 5846\,400 \text{ kJ/h}$$
$$\equiv 1624.0 \text{ kW} \equiv 1624.0 \times 10^3 \text{ W}$$

Average heat capacity of air at 59.5°C,

$$C_{pa} = 1.005 \text{ kJ/(kg} \cdot \text{°C)}$$

Air is heated from 44°C to 75°C.

$$\text{Mass flow rate of air, } q_{ma} = \frac{5846\,400}{1.005(75-44)}$$

$$= 187\,655 \text{ kg/h}$$

Volumetric flow rate of air, $q_{va} = 187\,655/1.061$

$$= 176\,866 \text{ m}^3\text{/h}$$

Viscosity of air at 59.5°C, $\mu_a = 0.072$ kg/(m · h)

In case of stationary cooler, heat lead,

$$\phi_2 = 4260\,000 \text{ kJ/h} \quad \text{(no evapouration load)}$$

$$\equiv 1183.33 \text{ kW}$$

Flow rate of circulating condensate,

$$q_{mc} = \frac{4260\,000}{4.1868(45-35)} = 101\,748 \text{ kg/h}$$

$$\equiv 101.7 \text{ m}^3\text{/h}$$

Process design of stationary (bulk flow) cooler:

Refer to Fig. 6.59.

Assume $2b = 5$ cm

$b = 2.5$ cm $\equiv 0.025$ m

In a stationary cooler, solids travel in plug flow and, therefore, a temperature gradient in bulk solids is observed. Following equation[25] relating to the unsteady-state heat transfer from a solid, can be used to calculate cooling time of the solids.

$$\frac{t_2 - t_2'}{t_1 - t_2'} = \sum_{n=0}^{\infty} \frac{2}{(n+0.5)^2 \pi^2} e^{\left[-(n+0.5)\frac{2\pi^2 \alpha/\theta}{b^2}\right]} \tag{6.137}$$

where, t_1 = Inlet solid temperature, °C

t_2 = Outlet solid temperature °C

t_1' = Inlet circulating condensate temperature, °C

t_2' = Outlet circulating condensate temperature, °C

α = Thermal diffusivity in solid, m^2/h

$$= k_s/(\rho_s \cdot C_s) \tag{6.138}$$

k_s = Thermal conductivity of solid, kJ/(h · m · °C)

C_s = 1.775 kJ/(kg · °C) for urea

θ = cooling time, h

Above equation assumes that convective transfer coefficient of cooling water and thermal conductivity of the plate material (SS in this case) is much larger than k_s. This assumption may be considered valid for urea prills.

$$k_s = 80 \text{ W/(m} \cdot \text{°C)}$$

$$= 288 \text{ kJ/(h} \cdot \text{m} \cdot \text{°C)}$$

$$\alpha = \frac{288}{1335 \times 1.775}$$

$$= 0.121\,54 \text{ m}^2\text{/h}$$

$$\text{LHS of Eq. (6.137)} = \frac{60-45}{100-45}$$
$$= 0.272\ 73$$

Spreadsheet or Mathcad can be useful in solving Eq. (6.134). Substituting values of b and α in RHS of the equation and solving by trial and error,

$$\theta = 0.0023 \text{ h} = 8.28 \text{ s}$$

Hold-up required for solids cooling,

$$= \frac{60\ 000 \times 8.28}{3600 \times 0.758} = 182.06 \text{ m}^3$$

Single silo or bin of such large operating volume with cooling plates would not be desirable for operational and maintenance reasons. Cooling height of at least 2 m is recommended.

Overall heat transfer coefficient of 175 W/(m$^2 \cdot$°C) will be safe to design the cooling surface.

$$\Delta\mathcal{T}_1 = 100 - 45 = 55°\text{C}$$
$$\Delta\mathcal{T}_2 = 60 - 45 = 15°\text{C}$$
$$\Delta\mathcal{T}_{lm} = \frac{55-15}{\ln\left(\frac{55}{15}\right)} = 30.79°\text{C}$$

$$\text{Heating surface requirement, } A_1 = \frac{4260\ 000}{30.79 \times 175 \times 3.6}$$
$$= 219.61 \text{ m}^2 \text{ (min.)}$$

Four or more silos may be provided. Distance between plates for cooling water flow should be kept at least 2 cm. Silos and plate coolers with HTA, exceeding 220 m^2, can be easily sized for the required heat duty.

For four silos,

Hold-up volume of solids = 182.06/4 = 45.515 m^3 per silo

Assume cooling (plate) height = 3 m

Cross-sectional area of silo = 45.515/3 = 15.172 m^2 (each)

Consider square construction of the silo.

$$\text{Each side} = \sqrt{15.172} = 3.895 \text{ say } 3.9 \text{ m}$$
$$\text{Plate length} = 3.5 \text{ m}$$
$$\text{Width between two plates} = 5 \text{ cm}$$
$$\text{No. of pairs of plates} = \frac{350}{5+5}$$
$$= 35$$
$$\text{HTA per pair of plates} = 3.5 \times 3 \times 2$$
$$= 21 \text{ m}^2$$
$$\text{HTA per silo} = 21 \times 35 = 735 \text{ m}^2$$
$$\text{Total HTA} = 735 \times 4 = 1960 \text{ m}^2$$
$$\frac{\text{Available HTA}}{\text{Required HTA}} = \frac{2940}{219.61} = 13.38 \text{ (much in excess)}$$

Note: In order to avoid adherence of urea dust, present in the feed of silo, a small quantity of dry air is injected from bottom. The exhaust air from the cooler can be directed to prill tower bottom.

II. Process Design of Rotary Cooler

Assume diameter of rotary cooler as 2.75 m. Air is passed in countercurrent (horizontal) fashion to solids flow.

$$\text{Cross-sectional area of cooler, } A_2 = (\pi/4)\,(2.75)^2 = 5.939\,57 \text{ m}^2$$

$$\text{Velocity of air} = \frac{176\,866}{3600 \times 5.939\,57} = 8.272 \text{ m/s}$$

This is a very high velocity and will blow dust and fine area prills. Normally in a rotary cooler, velocity up to 1.5 m/s is preferred. This will call for six rotary coolers to be used in parallel.

$$\text{Flow of urea prills/cooler} = \frac{60\,000}{6} = 10\,000 \text{ kg/h} \equiv 10 \text{ t/h per silo cooler}$$

Flow of air through each cooler,

$$q_{v\,\text{air}} = \frac{176\,866}{6} = 29\,477.7 \text{ m}^3\text{/h at inlet}$$

$$\text{Velocity of air at inlet} = \frac{29\,477.7}{3600 \times 5.939\,57} = 1.379 \text{ m/s } (< 1.5 \text{ m/s})$$

Rotary cooler length can be calculated by the following equation:

$$L = N_t H_t \tag{6.139}$$

where, L = Rotary cooler length, m
N_t = Number of transfer units
H_t = Height (length) of transfer unit, m

$$N_t = \frac{\Delta t_G}{\Delta \mathcal{T}_{\text{lm}}} \tag{6.140}$$

where, $\Delta t_G = t_{g,\,\text{out}} - t_{g,\,\text{in}}$, °C

$$\Delta \mathcal{T}_{\text{lm}} = \frac{(t_{s,\,\text{in}} - t_{g,\,\text{out}}) - (t'_{s,\,\text{out}} - t'_{g,\,\text{in}})}{\ln \dfrac{(t_{s,\,\text{in}} - t_{g,\,\text{out}})}{(t'_{s,\,\text{out}} - t'_{g,\,\text{in}})}}$$

$$= \frac{(100 - 75) - (60 - 44)}{\ln\left(\dfrac{100 - 75}{60 - 44}\right)} = 20.17°\text{C}$$

$$N_t = \frac{75 - 44}{20.17} = 1.537$$

Preferred range of N_t is 1.5 to 2.5.

$$H_t = \frac{1000\, G\, C_a}{U_v} \tag{6.141}$$

where, G = Mass flow rate per unit area of air, kg/(m^2 · s)

C_a = Heat capacity of air = 1.005 kJ/(kg · s)

U_v = Overall volumetric heat transfer coefficient, kJ/(m^3 · s · °C)

$$= 237\, G^{0.67}/D \tag{6.142}$$

D = Rotary cooler diameter, m

$$G = \frac{187\,655}{6} \times \frac{1}{3600 \times 5.939\,57}$$

$$= 1.462\,69 \text{ kg/(m}^2 \cdot \text{s)}$$

$$U_v = 237\,(1.462\,69)^{0.67}/2.75$$

$$= 111.2 \text{ kJ/(m}^3 \cdot \text{s} \cdot {}^\circ\text{C)}$$

Note that units of U_v are volumetric and different from normal units.

$$H_T = \frac{1000 \times 1.462\,69 \times 1.005}{111.2}$$

$$= 13.2$$

$$L = 1.537 \times 13.2 = 20.29 \text{ m or } 20.3 \text{ m}$$

$$L/D = 20.3/2.75 = 7.38$$

Rotary coolers, commonly in use, have L/D ratio in the range of 4 to 10. Bed depth of about a meter is recommended in the cooler. Above design is for cooling with air only. If cooling surface is provided across the length for indirect cooling with cooling water, heat transfer rate can be estimated by considering overall heat transfer coefficient of 55–110 W/(m^2 · °C).

III. Process Design of Fluid Bed Cooler

For determining the fluidizing velocity, mean particle diameter (d_p) is required. Mean particle diameter,

$$d_p = \sum \frac{1}{\left(\dfrac{w_i}{d_i}\right)} \tag{6.143}$$

where, d_p = Mean particle diameter and

w_i = Mass fraction of particle diameter, d_i

$$\sum\left(\frac{w_i}{d_i}\right) = \frac{0.008}{2.4} + \frac{0.09}{2} + \frac{0.563}{1.7} + \frac{0.266}{1.4} + \frac{0.073}{1}$$

$$= 0.6425$$

$$d_p = 1/0.6425 = 1.556 \text{ mm}$$

For estimating fluidization velocity, use of following equation[25] is recommended:

$$Re_m = \left[33.7^2 + \frac{5 \times 10^5\, d_p^3 \rho_g (\rho_s - \rho_g) g}{\mu_g^2}\right]^{1/2} - 33.7 \tag{6.144}$$

where, Re_m = Reynolds number at minimum fluidization velocity
d_p = 1.556 mm = 0.001 556 m
ρ_g = 1.061 kg/m^3
ρ_s = 1.335 kg/L = 1335 kg/m^3
g = gravitational acceleration
= 9.81 m/s^2
μ_g = 0.072 kg/(m · h)

$$Re_m = \left[1135.65 + \frac{5\times10^5(0.001\,556)^3\ 1.061(1335-1.061)9.81}{(0.072)^2}\right]^{1/2} - 33.7$$

$$= (1135.65 + 5044.89)^{1/2} - 33.7$$

$$= 44.92$$

Minimum fluidization velocity is related with Reynolds number as 44.92:

$$u_m = \frac{Re_m \mu_g}{3600\ \rho_g d_p} \tag{6.145}$$

$$= \frac{44.92\times0.072}{3600\times1.061\times0.001\,556}$$

= 0.54 m/s at ambient temperature

At the minimum velocity, fluidization starts. Terminal velocity (u_t) is the velocity above which the solids particles will be pneumatically conveyed. Reynolds number (Re_t) at the terminal velocity is derived by

$$C_d Re_t^2 = 1.6\times10^7\ g\ d_p^3 \frac{\rho_g(\rho_s-\rho_g)}{\mu^2} \quad \text{(Ref. 26)} \tag{6.146}$$

where, C_d = Drug coefficient which depends on sphericity (ϕ_s) of the particles
ϕ_s = 0.6 for broken solids and the same can be taken for urea prills
C_d = 6 for ϕ_s = 0.6

$$Re_t^2 = \frac{1.6\times10^7\times(0.001\,556)^3\times1.061(1355-1.061)}{(0.072)^2\times6} = 26\,906$$

$$Re_t = 164$$

$$u_t = \frac{Re_t \mu_g}{3600\ \rho_g d_p} \tag{6.147}$$

$$= \frac{164\times0.072}{3600\times1.061\times0.001\,556} = 1.987 \text{ m/s}$$

Actual fluidization velocity will be between u_m and u_t. Select u = 1 m/s for normal fluidization.

Volumetric flow rate of air, q_{va} = 176 866 m^3/h

$$\text{Area of fluidization, } A_3 = \frac{176\,866}{1\times3600}$$

$$= 49.129 \text{ m}^2$$

Selected width, $W = 3$ m

Length, $L = 49.129/3 = 16.376$ m

To improve thermal efficiency, total length will be divided into a number of stages. Select 4 stages for fluidization.

$$\text{Length of each stage} = \frac{16.376}{4} = 4.094 \text{ m, say } 4.1 \text{ m}$$

Staging will be beneficial for crossflow of solids. Bed depth of 0.4 to 0.5 m is adequate in each stage. Assuming bed depth of 0.5 m,

$$\text{Hold-up of urea in fluid bed cooler} = 49.129 \times 0.5 \times 758$$
$$= 18\,620 \text{ kg}$$

This means residence time (θ) of urea prills in the fluids bed cooler will be 18.62 min; or approximately 20 min.

Peak pressure drop in the bed[26],

$$\frac{\Delta p_p}{H \rho_b g} = \left[\frac{6.8}{\tan \gamma}\left(\frac{D_i}{D_c}\right) + 0.8\right] - 34.4\,\frac{d_p}{H} \tag{6.148}$$

where, Δp_p = Peak pressure drop, kN/m^2 or kPa

H = Height of bed = 0.5 m

ρ_b = Bulk density = 0.758 kg/L

g = 9.81 m/s^2

V = Angle of internal friction.

For granular solids, consider it to be 55°.

D_i = Fluid inlet orifice diameter

= 2 cm = 0.02 m (assumed)

D_c = diameter of equivalent cross section

$$= \sqrt{\frac{4}{\pi}(49.129)} = 7.909 \text{ m}$$

$$\frac{\Delta p_p}{0.5 \times 0.758 \times 9.81} = \left[\frac{6.8}{\tan 55}\left(\frac{0.02}{7.909}\right) + 0.8\right] - 34.4 \times \frac{0.001556}{0.5}$$

$$= 0.812 - 0.107 = 0.705$$

$$\Delta p_p = 2.62 \text{ kPa} \equiv 267 \text{ mm WC}$$

Note: In normal course, pressure drop will vary between 30 and 50% of peak pressure drop (i.e., 80–135 mm WC) across the bed. To this add friction loss in the ducts which will give the fan pressure requirement.

During fluidization, broken and prills with poor mechanical strength will break and form dust due to attrition. This is expected to give higher dust load than the other two types of coolers. Dust will be separated from exhaust air and will be allowed to fall in a dust dissolving tank for reprocessing. Urea prills, coming out from the fluid bed coolers will be of near uniform size and with definite mechanical strength which will not break during storage in silo or during transportation.

It is recommended to test the desired product in a pilot batch fluidizer. The fluidization velocity, orifice size, bed depth, pressure drop across the bed and other design parameters can be verified in the pilot unit. Process design, based on the verified parameters, will give an optimum design.

EXERCISES

6.1 Select the correct answer.

(a) For an exchanger with no phase change service design conditions are: Hot fluid is cooled in counter current manner from 213°C to 57°C with a cold fluid which is heated from 29.5°C to 40.5°C. Other parameters being maintained the same, if hot fluid inlet temperature is reduced to 201°C and cold fluid inlet temperature is increased to 35°C, then

(i) Hot fluid and cold fluid outlet temperatures will change to 60°C and 45°C, respectively.

(ii) Hot fluid outlet temperature will change to 60°C while cold fluid outlet temperature will remain as 40.5°C.

(iii) Hot fluid outlet temperature will remain 57°C while cold fluid outlet temperature will change to 45°C or

(iv) Data not sufficient to predict.

(b) In the heat transfer equation for heat exchanger, the heat transfer rate appears to be directly proportional to the heat transfer area. Hence (other parameters remaining the same) for double the heat transfer area, the heat transfer rate will be

(i) doubled

(ii) more than doubled or

(iii) less than doubled.

(c) A shell and tube, horizontally installed heat exchanger with 2-tube passes, has pressure gages (P_1 and P_2) installed, respectively on the inlet pipe to the lower nozzle and on the outer pipe from the upper nozzle of the channel. The pressure gages are approximately 1.4 m apart. The difference in the readings ($p_1 - p_2$) is 0.42 bar. Each nozzle accounts for 0.07 bar pressure drop. Estimate the difference in pressure reading (as $p_1 - p_2$) for the following cases:

(i) Tube side passes are changed from 2 to 4 and

(ii) Tube side flow rate is doubled.

[**Answer:** (a) 1.42 bar, (b) 0.85 bar or (c) 0.42 bar]

6.2 A fired tube waste-heat boiler generate steam at a pressure of 10.34 bar g. It is designed for a gas flow of 80 000 kg/h from an incinerator. Gas is designed to be cooled from 600°C to 260°C. On a particular day, the gas flow is 55 000 kg/h and inlet temperature to the boiler is 500°C and steam is generated at 10.34 bar g. What will be the expected gas outlet temperature if the overall heat transfer coefficient is proportional to mass flow rate of gas to the power of 0.8? Assume other properties of flue gases to be nearly the same.

Assume overall thermal efficiency of the boiler to be 95% in both the cases. Average heat capacity of flue gases may be taken as 1 kJ/(kg · K). Demineralized water enters steam drum at 35°C. Calculate steam generation from the boiler in both the cases.

Data: Saturation temperature of steam at 10.34 bar g = 185.5°C
Enthalpy of saturated steam at 10.34 bar g = 2780.8 kJ/kg
Enthalpy of water at 35°C = 146.6 kJ/kg
Reference state: Enthalpy of saturated liquid water at 0.01°C = 0 kJ/kg

6.3 120 000 kg/h of ethanol is to be cooled from 78°C to 40°C by cooling water as coolant. Cooling water enters at 32°C and leaves at 40°C. Design shell and tube heat exchangers for the different tube lengths 1.5 m, 3 m, 4.5 m and 6 m.

6.4 3000 kg/h of nearly pure saturated phosphorous oxychloride ($POCl_3$) vapour at 15 kPa g is to be condensed and cooled to 60°C by cooling water which is available in plant at 32°C. Design the suitable shell and tube heat exchanger for the given duty using following data:
Tube material: Graphite
Tube OD: 25 mm
Tube ID: 20 mm
Thermal conductivity of graphite: 150 W/(m · K)
Molar mass of $POCl_3$: 153.5 kg/kmol
Specific heat of liquid $POCl_3$: 0.92 kJ/(kg · °C)
Latent heat of vaporization of $POCl_3$: 229 kJ/kg
Thermal conductivity of liquid $POCl_3$: 0.173 W/(m · °C)
Viscosity of liquid $POCl_3$ = 1.1 cP or mPa · s
Liquid density of $POCl_3$ = 1675 kg/m^3
Condensation temperature of $POCl_3$ at 15 kPa g = 110°C
Viscosity of $POCl_3$ vapour, $\mu_v = 1000 \times 10^{-7}$ Poise

6.5 Determine shell side heat transfer coefficient and pressure drop by Kern's method and Delaware method for following heat exchange duty and compare results:
(i) Type of HE: AET as per TEMA (pull through floating head heat exchanger)
(ii) Shell side fluid: Oil Flow rate = 10 000 kg/h
(iii) Inlet/Outlet temperatures: 110/50 °C
(iv) Properties of oil at average temperature
C_L = 2.16 kJ/(kg · °C)
μ_L = 8 cP or mPa · s
k_L = 0.14 W/(m · °C)
ρ_L = 850 kg/m^3
(v) Tube data:
Number of tubes, N_t = 918
Tube OD/ID = 19.05/15.748 mm
Tube length = 3 m
MOC of tube: Carbon steel
Type of tube layout: Triangular
Tube pitch, P_t = 25.4 mm
Tube bundle diameter, D_b = 846 mm
Shell ID = 926 mm
No. of tube passes = 8
Pass partition plate thickness = 12.7 mm
No. of vertical pass partition plate = 1 (Hence, 2 tube passes)
(vi) Type of baffle: 25% cut segmental
Baffle spacing = 200 mm (central)
Entrance and exit baffle spacing = 300 mm
Clearance between shell ID and baffle diameter = 4.8 mm (diametric)
Clearance between tube hole in baffle and tube OD = 0.8 mm (diametric)

6.6 Determine the shell side heat transfer coefficients and shell side pressure drops by Kern's method and Delaware method for following heat exchange duty and compare results.
(i) Type of HE: AET as per TEMA (pull through floating head heat exchanger)
(ii) Shell side fluid: Liquid methanol Flow rate: 100 000 kg/h
(iii) Inlet/Outlet temperatures: 95°C/40°C

(iv) Properties of methanol at average temperature:
$C_L = 2.84$ kJ/(kg · °C)
$\mu_L = 0.34$ cP or mPa · s
$k_L = 0.19$ W/(m · K)
$\rho_L = 750$ kg/m^3

(v) Tube data:
Number of tubes, $N_t = 918$
Tube OD/ID = 20/16 mm
Tube length = 4880 mm
MOC of tube: Cupro-nickel
Type of tube layout: Triangular
Tube pitch, $P_t = 25$ mm
Tube bundle diameter, $D_b = 826$ mm
Shell ID = 894 mm
No. of tube passes = 2
Pass partition plate thickness = ?

(v) Type of baffle: 25: cut segmental (horizontal)
Baffle spacing, $B_s = 356$ mm
Clearance between shall ID and baffle diameter = 4.8 mm (diametric)
Clearance between tube hole in baffle and tube OD = 0.8 mm (diametric)

6.7 Design vertical thermosyphon reboiler for acetic acid dehydration column based on following data:

(a) Feed is liquid at 30°C.
Feed flow rate = 12 000 kg/h
Feed composition = 80% acetic acid and 20% water (by mass)

(b) Distillate is saturated liquid at 100.6°C
Flow rate of distillate = 3000 kg/h
Distillate composition = 20% acetic acid and 80% water (by mass)

(c) Residue is nearly pure saturated acetic acid at 138°C and at 179.5 kPa a.

(d) Reflux ratio, $R = 4.2$

(e) Latent heat of vaporization

$$\lambda_{AA} = 25\,732 \text{ kJ/kmol at } 293.8 \text{ K}$$
$$\lambda_W = 40\,677 \text{ kJ/kmol at } 373.15 \text{ K}$$

(f) Crtical temperature of acetic acid = 592.71 K
Crtical temperature of water = 647.11 K

(g) Heat capacity equation for liquids

$$C_{\text{ml}} = a + bT + cT^2 + dT^3$$

where, C_{ml} = Liquid heat capacity, kJ/(kmol · K)
T = Absolute temperature, K

Table 6.48 Heat Capacity Data or Liquids[10]

	a_i	$b_i \times 10^3$	$c_i \times 10^6$	$d_i \times 10^9$
Acetic acid	–36.0814	604.681	–393.957	–561.602
Water	18.2964	472.118	–1338.78	1314.24

6.8 50 000 kg/h cold air is required at 18°C temperature. Chilled water with 6°C supply temperature and 8°C return temperature is to be used as cooling medium. Design a suitable finned tube heat exchanger with box type shell.

Hint: Atmospheric air contains water vapour. Hence, condensation load of water vapour must be considered in the calculation of heat duty. Air saturated with water vapour at 40°C can be considered as design condition.

6.9 Design a kettle-type reboiler which must provide 2000 kg/h of ethylene glycol vapour to a distillation column at 30 kPa a pressure at bottom.

6.10 A chilling plant is designed for achieving 703 kW (≈ 200 TR) heat exchange capacity to supply aqueous ethylene glycol brine at –25°C by evaporating liquid ammonia in the evaporator at –33.3°C (and 1 atm). Ammonia vapour at –28°C leaves the evaporator and sent to a compressor.

(a) In one alternative, a shell and tube-type condenser is considered. Superheated vapour at 120°C and 16.435 bar a from the compressor enters the shell of the condenser and condenses at 42°C. Cooling water enters the tubes at 32°C and leaves at 38°C. For this duty design a BEM type horizontal condenser. Consider tube OD and length as 19.05 mm (16 BWG) and 2.4383 m (8 ft), respectively. Triangular (Δ) pitch equals 1.25 d_o.

(b) In another alternative, an evaporative condenser is considered for condensing ammonia. As shown in Fig. 6.62 evaporative condenser is basically a rectangular tube bundle installed in a cooling tower.

Cooling water falls over the tubes at a required rate at essentially a constant temperature; 4.5°C above the design wet bulb temperature of air. Ambient air at 40°C dry bulb (*DB*) temperature and 28°C wet bulb (*WB*) temperature is supplied from sides of the tower. An induced draft fan sucks the air from the louvers and discharges it from top with 95% relative humidity (*RH*). Ammonia gas enters at 120°C the tubes and condenses at 1.5°C higher than the cooling water temperature. Design the tube bundle to be placed in the evaporative condenser. For the evaporation of water film over tube bundle, heat transfer coefficient can be determined by Kallam's equation.

Fig. 6.62 *Evaporative condenser*

$$h_o = 7.3 \times 10^{-9} \times N^{0.05} \, [(G^{0.3} d_o \, YZ)/(\mu \, P_V)]^{4.4} \quad \text{(Ref. 5)} \tag{6.149}$$

where h_o = water side film coefficient, W/(m^2 · °C)

$$G = \dot{m}_c/(L \cdot B) \tag{6.150}$$

$\dot{m}_c$ = circulation rate of cooling water, kg/s

L = Length of tube = 2.4384 m

B = Width of tube bundle, m

μ = Viscosity of water, kg/(m · s)

P_V = Vertical tube pitch = 0.030 48 m

d_o = OD of tube = 19.05 mm

N = Number of horizontal tube rows

$$Y = (P_H - d_o)/d_o \tag{6.151}$$

$$Z = (P_H - d_o)/P_H \tag{6.152}$$

P_H = Horizontal tube pitch = 0.0381 m

Here, evaporation temperature of water film can be taken as 34°C. Evaporation rate of water can be taken as 2% of circulation rate.

(c) In a water scarce area, an air cooler is considered for condensing ammonia. Ambient air at 45°C is supplied to the condenser while ammonia is condensed at 60°C in the tubes. Compressed ammonia gas enters the condenser at 130°C. A pressure drop of 15 kPa is permitted in the condenser. Condenser will have two-pass arrangement in finned tubes and leaves from top at 50°C. Each horizontal tube row can be considered as a pass.

Effective tube details: SS 304 with OD = 19.05 mm and triangular (Δ) pitch of 43 mm

Fin details: Type of fin: Spiral wound transverse fins, SS 304 MOC, Fin OD = 38 mm, 8 fins per linear inch and fin thickness = 0.889 mm

Tube length = 3 m

Width of air cooler = 3 m

Design an air cooler for ammonia condensation duty.

Properties of ammonia are listed in Table 6.49.

Table 6.49 Properties of Ammonia**

Temp. °C	Pressure bar a	State	Enthalpy*, kJ/kg		Gas/Vapour		
					C_p	k	μ
			Liquid	Vapour	kJ/(kg·K)	mW/(m·K)	μPa·s
–33.3	1.013 25	Satd, liq.	48.8	1418.3	2.2969	20.968	8.054
–28	1.013 25	Sup. vap.	–	1428.1	2.2707	21.171	8.197
34	13.124	Satd, liq.	361.2	1488.0	3.3488	27.433	10.126
42	16.435	Satd, liq.	400.5	1490.4	3.5683	28.713	10.393
60	26.156	Satd, liq.	492.0	1489.3	4.2084	32.262	11.049
120	13.25	Sup. vap.	–	1723.7	2.5166	37.461	13.641
130	26.30	Sup. vap.	–	1714.1	2.7897	40.862	14.030

* Reference state: Enthalpy of saturated liquid at 0°C = 200 kJ/kg

** Ref.: REFPROP Ver. 8.0, NIST Standard Reference Data Base 23, 2007.

6.11 Synthesize the heat exchanger network for the following five process streams such that resulting heat exchanger network will require the minimum hot and cold utilities. Also find the values of minimum utilities (hot and cold) required.

Table 6.50 Process Stream Data[27]

Stream	Type	$\dot{m}C_p$, kW/°C	t_{in}, °C	t_{out}, °C
1	Hot	228.5	159	77
2	Hot	20.4	267	88
3	Hot	53.8	343	90
4	Cold	93.3	26	127
5	Cold	196.1	118	265

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Process Design of Liquid-Liquid Extractors

7

7.1 INTRODUCTION

Liquid-liquid extraction is the separation of the components of a liquid solution by contacting it with another insoluble liquid solvent. One of the components or certain group of the components of the solution (feed) is selectively solubalised in the solvent. It is called *solute*. After the contact, components of liquid feed are distributed between two immiscible liquids; called *extract phase* and *raffinate phase*. Solvent rich phase is called extract phase and solvent lean phase is called raffinate phase. A component which is left behind in the raffinate phase after extraction or non-solute part of feed is called *diluent*.

Solvent extraction finds many applications in industry. Take an example of separation of solution containing 10% by mass acetic acid and 90% by mass water. Separation of these two is difficult by ordinary distillation as this mixture provides very low relative volatility. In liquid-liquid extraction, acetic acid-water solution is contacted with a solvent such as ethyl acetate. The solvent makes the immiscible or partially miscible liquid mixture with water. Acetic acid is selectively extracted in ethyl acetate. At the end of single stage extraction two immiscible liquid layers are obtained. Ethyl acetate rich layer is called extract phase and water rich layer is called raffinate phase.

In another application, *n*-hexane is used as a solvent to extract sterol (a nutracentical) from deodorised distillate, derived from soybean oil. Extract phase is rich in sterol, leaving behind a raffinate, containing mixed tocopherols (antioxidants), squalline, etc.

Liquid-liquid extraction is an important unit operation and finds applications in refinery, pharmaceutical industries, metallurgical industries and other industries. In refinery, after distillation, liquid-liquid extraction is the most widely used unit operation.

Invariably extraction will have to be followed by another unit operation, such as distillation or crystallisation for separation of solute from the extract phase.

7.2 IMPORTANT INDUSTRIAL APPLICATIONS OF LIQUID-LIQUID EXTRACTION[1, 2]

1. Separation of 10% to 30% acetic acid in aqueous solution by using ethyl acetate or ethyl ether as solvent.
2. Recovery of uranium from ore rich liquor. Solvent for this case is alkyl phosphate in kerosene.
3. Separation of long chain fatty acids from vegetable oil by using propane as solvent.
4. Propane deasphalting. Separation of lube oil from residuum (residue of vacuum distillation column of refinery) by using propane as solvent.
5. Separation of tantalum and niobium. It involves dissolution of this mixutre in hydrofluoric acid, followed by extraction with Methyl Isobutyl Ketone (MIBK). Similarly, liquid-liquid extraction is used for the metal separations such as uranium–vanadium, hafnium–zirconium and tungsten–molybdenum mixtures.
6. Phosphoric acid and boric acid are finally purified by liquid-liquid extraction.
7. Liquid-liquid extraction is used for the separation of the mixture of aromatic and paraffinic hydrocarbons of nearly the same molar mass and having close boiling points (e.g., a mixture of benzene and haxane). For this case, solvents used are liquid sulphur dioxide, diethylene glycol or sulpholane. Extractive distillation is also used for the same application.
8. Many pharmaceutical products are produced in the form of complex mixture. From this mixture, pure product is separated by liquid-liquid extraction; e.g., penicillin, resorcinol, etc.
9. In the petroleum refinery the separation of pure benzene and toluene from aromatic rich feed stock is carried out by extraction. In this case, sulpholane is used as solvent.
10. For dearomatisation of raw hexane fraction (or for the production of food grade hexane) using N–methyl pyrrolidone (NMP) as solvent.
11. For dearomatisation of the straight run kerosene fraction (or for the production of superior kerosene) using sulpholane as solvent.
12. For the purification of lube oil using furfural or NMP as solvent.
13. In an aqueous effluent containing very high COD, organic solvent (such as toluene or xylene) is effectively used to extract out COD causing components.
14. In the manufacturing process of MTBE, methanol is separated from the mixture of methanol and C_4 stream by using water as solvent.
15. For extraction of unsaturated fatty acids from a mixture of fatty acids using fulphural as solvent.

7.3 THEORY OF LIQUID-LIQUID EXTRACTION OPERATIONS

7.3.1 Continuous and Dispersed Phases

In extraction operation, two immiscible liquid phases are brought in contact with each other. In Section 7.1, differentiation of extract and raffinate phases was clarified. But before

these two phases are separated from an extractor, two immiscible liquids are brought in contact which are known as continuous and dispersed phases. Dispersed phase is a liquid which forms the droplets and are dispersed in a continuum of the other; known as a continuous phase. Ordinarily, the liquid with smaller flow rate will be dispersed in another. For a batch operation, the liquid in which impeller is immersed when at rest is usually a continuous phase. Normally, dispersed phase has higher viscosity and lower interfacial tension. In continuous extraction, interchanging of dispersed phase and continuous phase is possible. Also inflammable liquid should preferably be a dispersed phase. A few industrial examples illustrate both phases.

1. Refer to Table 8.78. A heterogeneous azeotrope of *n*-butanol and water when condenses, two phases are separated. Bottom aqueous layer contains 6 to 7.5% (by mass) *n*-butanol, depending on temperature, which should be recovered for economical reasons. Toluene or xylene is used as solvent for the extraction purpose. In this case, aqueous layer; i.e., feed (dense phase) is a continuous phase while organic solvent (light phase) is a dispersed phase.
2. Acetic acid is extracted from acetic acid-water (feed) mixture (dense phase) with the help of ethyl acetate as solvent (light phase). In this case, heavy phase is dispersed in light phase.
3. Feed mixture of dimethyl formamide (DMF)-water (light phase) is contacted with methylene chloride (dense phase) for extraction of DMF in which light phase is a dispersed phase.
4. Feed mixture of benzene-*n*-hexane (light phase) is contacted with liquid sulphur dioxide (heavy phase) for extraction of *n*-hexane. In this operation, light phase is a dispersed phase.

7.3.2 Phase Equilibrium

Understanding of a phase diagram is necessary before attempting design of liquid-liquid extraction system. Brief discussions in this section will be limited to a binary feed and a solvent. Thus the system comprises three components. Ternary phase diagram acts as a map for liquid extraction applications. Ternary phase diagrams are unique in that they show all three components of the system on one plot. The following general principles govern ternary phase diagrams. Each apex point of the triangle represents one of the pure components. Any point of a side of the triangle represents a binary mixture. Lines may be drawn parallel to the sides of the equilateral triangle for the plotting of the compositions. Any point in the triangle represents a ternary mixture and its composition can be read easily from the apex lines. The ternary phase diagram can be constructed from experimental data. Figure 7.1 represents the ternary diagram for water-ethanol-benzene system[1] at 25°C and atmospheric pressure.

The saturation curve or bimodal curve (miscibility boundary) on Fig. 7.1 is represented by DEFGHIJKK′J′I′H′G′F′E′D′. On this diagram DD′, EE′, …, JJ′ represent tie lines which connect two equilibrium points on the miscibility boundary. For example, a mixture representing point L can be prepared which has composition 45% benzene, 35% ethanol and rest water (by mass) by mixing a feed containing water (43.75%) and ethanol (56.25%) and nearly pure benzene. After shaking it, if it allowed to equilibrate (similar to that in a mixer-settler), it will separate in two layers. Top light layer (point I′) is known as the raffinate phase which will contain 84.4% benzene and 13.85% ethanol

Fig. 7.1 *Phase Diagram for Benzene–Ethanol–Water at 25°C (Reproduced with the Permission of the American Chemical Society, USA)*

Note: All percentages are mass %

while bottom heavy layer (point I) is called the extract phase which will contain 13.7% benzene and 52.2% ethanol. Point M is called plait point, representing critical composition; 37.4% ethanol, 52.0% benzene and 10.6% water. The plait point represents ternary homogeneous liquid mixture, saturated with both; solvent and diluent. Curve on the right of the plait point is called saturated extract phase while curve on the left is called saturated raffinate phase. Extract phase is a ternary homogeneous liquid mixture, saturated with diluent, while raffinate phase is the ternary homogeneous liquid mixture, saturated with solvent. The area under the binodal curve is the region where two liquid phases will exist. Above the curve, there will be only one liquid phase.

Material balance equations can be established to find quantities of the separated layers, knowing their compositions. Alternately using line ratio principle of geometry, one can find the quantities from the diagram (Fig. 7.1).

IL/LI′ = Quantity of raffinate phase/Quantity of extract phase

= 0.7125 linear units/0.885 linear units = 0.805 : 1

Using above relation, one can find a mass ratio of extract to raffinate phases as 44.6:55.4. In addition to the above considerations, equilibrium constraints must be satisfied. Distribution coefficient or partition coefficient K or m at equilibrium is defined as

$$K \text{ or } m = \frac{\mathcal{Y}}{\mathcal{X}} = \frac{\text{Mass fraction of solute in extract phase}}{\text{Mass fraction of solute in raffinate phase}} \tag{7.1}$$

In liquid-liquid extraction, all the components are present in both extract and raffinate phases at equilibirum. At equilibirum, acitivity of any component is same in both phases.

$$\mathring{A}_r = \mathring{A}_e \Rightarrow \gamma_r x = \gamma_e y \tag{7.2}$$

where, $\mathring{A}_r$ = activity of component in raffinate phase
$\mathring{A}_e$ = activity of component in extract phase
γ_r, γ_e = activity coefficients of components in raffinate/extract phase
y = mole fraction of component in extract phase
x = mole fraction of component in raffinate phase

$$K' = \frac{y}{x} = \frac{\gamma_r}{\gamma_e} \tag{7.3}$$

where, K' = distribution coefficient in mole fraction units

Large collections of experimental equilibrium data are now available for liquid-liquid systems. One source is *"Liquid-liquid Equilibrium Data Collection"* by Sorenson and Arit, DECHEMA, Frankfurt, Germany. In *Perry's Chemical Engineers' Handbook* (Ref. 4), distribution coefficients for a few ternary systems are given (Table 15.5 of 6th Ed.)

7.4 DESIRABLE SOLVENT PROPERTIES OR CHOICE OF SOLVENT

7.4.1 Selectivity

The relative separation or selectivity is defined as the ratio of ratios of solute to nonsolute components in extract phase to the same in raffinate phase at equilibrium. It is represented as α (or β).

For all useful extractions, $\alpha > 1$. If $\alpha = 1$, it means no separation or no extraction. It represents the separation power of the solvent. Higher the selectivity, lesser is the number of theoretical stages required for the desired separation. Dilute solute concentration generally gives the highest selectivity.

7.4.2 Distribution Coefficient

Distribution coefficient K or m is the ratio of mass fraction of solute in extract phase to mass fraction of solute in raffinate phase at equilibrium.

It is not necessary that K should be greater than 1. K can be less than 1. But higher value of K is desirable because higher the distribution coefficient, lesser is the amount of solvent required for desired separation. Unfortunately solvents which provide the higher value of K normally provide poor value of α.

7.4.3 Recoverability

It is always necessary to recover the solvent from extract phase for reuse. Hence, selected solvent must be easily recoverable from the extract phase. In most of the cases, solvent is separated from extract phase by distillation. If selected solvent is to be separated or recovered by distillation than it should preferably not form an azeotrope with the solute and also it should provide higher value of relative volatility. Lesser value

of latent heat of vaporisation of solvent is also desirable for low cost of recovery. Water is not an attractive solvent for extraction as it forms azeotrope or non-ideal solution with many organics and it has very high value of latent heat of vaporisation.

7.4.4 Capacity

Capacity of a solvent to extract the solute is measured in terms of maximum solubility of the solute in solvent or in terms of solubility limit. Higher the solubility limit, lesser is the amount of solvent required. Solute should be significantly soluble in the selected solvent.

7.4.5 Insolubility of Solvent in Raffinate

Low solubility of solvent in the raffinate phase is desirable. Solvent which is more insoluble is preferred. More insoluble solvent can separate wide range of composition of the feed mixture. Also, it gives less solvent loss and adds less impurity in the final raffinate. Generally, a solvent which is more insoluble provides higher selectivity.

7.4.6 Density

Higher density difference between solvent and diluent or extract and raffinate phase is desirable. It makes the decantation easier. In most of the systems, density difference decreases to zero at plait point, but in some system it can become zero at an intermediate solute concentration and can invert the phases at higher concentration. For such a system, in the desired range of separation in the extractor, if density difference passes through zero then use of continuous contact equipment like packed tower is not preferred but mixer-settler-type stagewise extractor can be used. Other option is to use two continuous contact extractors in series.

7.4.7 Interfacial Tension

A high interfacial tension promotes rapid coalescence and generally requires high mechanical energy for agitation to produce small droplets. Conversely, low interfacial tension requires lesser mechanical energy for agitation to produce small droplets but at the same time it provides slow coalescence rates. Usually interfacial tension decreases with increase in solute concentration and increase in mutual solubility and falls to zero at plait point. Very high and very low interfacial tension of selected solvent is not desirable, otherwise higher collision rate is more important, hence moderately high interfacial tension is preferred.

7.4.8 Chemical Reactivity

The solvent should be inert towards the other components of extraction system and to the common materials of construction.

7.4.9 Viscosity, Vapour Pressure and Freezing Point

All these properties of solvent should be low for ease in handling, transportation and storage.

7.4.10 Toxicity and Flammability

The solvent should preferably be non-toxic, non-flammable and of low cost.

7.5 DESIGN OF COUNTER CURRENT MULTISTAGE EXTRACTOR

Process design of extractor can be divided in the following steps:

1. Selection of a suitable solvent and determination of liquid-liquid equilibrium data. Actually both these steps are interrelated.
2. Determine the minimum amount of solvent required for the desired separation.
3. Determine or decide the actual amount of solvent. Actual amount of solvent must be the optimum amount of solvent. Then complete the material balance.
4. Determine the number of theoretical stages (for stagewise extractor) or number of transfer units (for differential continuous contact extractor) required for the desired separation.
5. Find the tower diameter of tower-type extractor or carry out the process design of agitator for agitated vessel-type extractor and also decide the height and diameter of agitated vessel.
6. Find the stage efficiency for stagewise extractor or height of transfer units for differential or continuous contact-type extractor.
7. Find the actual number of stages for stagewise extractor or height of tower for tower-type extractor.
   (a) First step is explained in previous Section 7.4.
   (b) S_m: Minimum amount of solvent is the maximum amount of solvent for which infinite number of stages are required for desired separation.

Fig. 7.2 *Multistage Countercurrent Extraction*

$$F + S_m = E_{1m} + R_{NP} \tag{7.4}$$

$$R_{NP} - S_m = F - E_{1m} = \Delta R_m \tag{7.5}$$

ΔR_m is a difference point. It represents net flow outward at the last stage N_P and is equal to net flow inward at first stage. On *an equilateral* triangular coordinates, when operating line from ΔR matches with tie line, infinite number of stages are required for the desired separation. To find S_m, extend the tie lines and allow them to intersect $\overleftrightarrow{SR_{NP}}$. A line joining the equilibrium compositions of extract and raffinate phases is called a *tie line*.

Among all such points, a point that is farthest from S, if all points are on the left-hand side of S or a point which is nearest to S, if all points are on the right-hand side of S, represents ΔR_m, the difference point for minimum solvent.

(a) ΔR_m Nearest from S for $m < 1$

(b) ΔR_m Farthest from S for $m > 1$

Fig. 7.3 *Minimum Solvent for Countercurrent Extraction*[4]

In most of the cases but not in all cases, tie line which is on extension passes through F gives the location of ΔR_m. After finding the location of ΔR_m, locate E_{1m}. Draw the line $\overline{E_{1m}R_{NP}}$ which intersects line $\overline{FS}$ at X_{Mm}.

$$F + S_m = M_m \tag{7.6}$$

$$FX_F + S_m Y_s = X_{Mm} M_m = X_{Mm}(F + S_m) \tag{7.7}$$

By substituting the value of X_{Mm} from the graph, one can find S_m using Eq. (7.7).

If A and B are insoluble liquids or can be considered as insoluble liquids at operating temperature and in desired concentration change, then solute balance is given by following equation:

$$A(X'_F - X'_{NP}) = B(Y'_1 - Y'_S) \tag{7.8}$$

where, $X' = \dfrac{\text{kg of solute (C)}}{\text{kg of non-solute (A)}}$ and $Y' = \dfrac{\text{kg of solute (C)}}{\text{kg of solvent (B)}}$

$$\frac{A}{B} = \frac{Y'_1 - Y'_S}{X'_F - X'_{NP}} \tag{7.9}$$

$$\frac{A}{B_m} = \frac{Y'_{1k} - Y'_S}{X'_F - X'_{NP}} \tag{7.10}$$

where, B_m = Minimum amount of solvent

Y'_{1k} is an equilibrium with X'_F. From equilibrium data, one can find Y'_{1k} that is in equilibrium with X'_F. Then from Eq. (7.10), one can find B_m. Operating line starts from point (X'_{NP}, Y'_s).

Fig. 7.4 *Determination of B_m*

8. To decide the actual amount of solvent, find the number of theoretical stages required for the desired separation for the different values of actual amount of solvent, like

$S = 1.1\ S_m$, $S = 1.2\ S_m$, $S = 1.3\ S_m$ … and so on.

On increasing the values of S from S_m, value of N will decrease. In initial range, N will significantly decrease with increase in S, but after a certain increase in S, N may not decrease appreciably.

For example, in this case Fig. 7.4 one can say optimum value of S is very close to $1.4\ S_m$.

Fig. 7.5 *Determination of S_{opt}*

Ideally actual amount of solvent = S_{opt}

But to find optimum values of S detailed costing calculations are required.

9. Procedure to find number of theoretical stages for desired separation N from the value of S:

(a) If A and B are not completely insoluble liquids:

Refer to Fig. 7.2.

Overall Material Balance.

$$F + S = E_1 + R_{NP} \tag{7.11}$$

Solute balance:

$$FX_F + SY_s = E_1Y_1 + X_{NP}\,R_{NP} = MX_M \tag{7.12}$$

$$F - E_1 = R_{NP} - S = \Delta R = \text{Net flow outward} = \text{Net flow inward} \tag{7.13}$$

Fig. 7.6 *Determination of Operating Curve*

Procedure:

Find $X_m = \dfrac{FX_F + SY_S}{F + S}$. Locate point M on graph which lies on $\overline{FS}$ line. Extend the line $\overline{R_{NP}M}$ that will intersect the binodal curve at E_1. Draw the lines $\overleftrightarrow{E_1F}$ and $\overleftrightarrow{R_{NP}S}$. These will intersect at the point ΔR. Draw the random lines from ΔR that give the points of operating curve (X_s, Y_{s+1}).

Operating curve starts from point (X_F, Y_1) and terminates at point (X_{NP}, Y_s). Start the stagewise construction from (X_F, Y_1) to (X_{NP}, Y_s) in between equilibrium curve and operating line and find number of theoretical stages, required for the desired separation.

(b) If A and B are immiscible liquids then overall material balance will be

$$A(X'_F - X'_{NP}) = B(Y'_1 - Y'_s) \tag{7.14}$$

By using Eq. (7.14), for the given actual amount of solvent B and its composition Y'_s, one can find Y'_1. Operating line starts at (X'_F, Y'_1) and terminates at (X'_{NP}, Y'_s).

Stagewise construction starts from point (X'_F, Y'_1) and terminates at the point (X'_{NP}, Y'_s) of the operating line.

In a special case, where equilibrium curve is also the straight line then the number of theoretical stages required for the desired separation can be obtained from Eq. (7.16a) or Eq. (7.16b) (Ref. 3).

Fig. 7.7 *Determination of Equilibrium Stages*

Fig. 7.8 *Countercurrent Extraction with Insoluble Solvent*

Let $$m' = \frac{Y'}{X'} = \text{slope of equilibrium line}$$

Ratio of the slope of equilibrium line to the slope of operating line is given by following equation:

$$\varepsilon = \frac{m'B}{A} \tag{7.15}$$

If, $\varepsilon \neq 1$

$$N = \frac{\ln\left[\left(\dfrac{\mathcal{X}'_F - (\mathcal{Y}'_s/m')}{\mathcal{X}'_{NP} - (\mathcal{Y}'_s/m')}\right)\left(1 - \dfrac{1}{\varepsilon}\right) + \dfrac{1}{\varepsilon}\right]}{\ln \varepsilon} \tag{7.16a}$$

When ε = 1

$$N = \frac{\mathcal{X}'_F - (\mathcal{Y}'_s/m')}{\mathcal{X}'_{NP} - (\mathcal{Y}'_s/m')} - 1 \tag{7.16b}$$

Multistage counter current extraction requires less number stages for the given amount of solvent than the cross-current extraction or it requires less amount of solvent for the fixed number of stages than cross-current extraction.

Number of Transfer Units (Ref. 4)

In case of differential or continuous contact-type extractor, instead of number of theoretical stages, number of transfer units are determined. Then the working height of continuous contact-type extractor is given by the equation

$$Z = N_t H_t \tag{7.17}$$

where, N_t = Number of transfer units

H_t = Height of a transfer unit

Number of overall mass transfer units based on raffinate phase is given by equation

$$N_{toR} = \int_{\mathcal{X}_{NP}}^{\mathcal{X}_F} \frac{d\mathcal{X}}{\mathcal{X} - \mathcal{X}^*} + \frac{1}{2}\ln\left(\frac{1 - \mathcal{X}_{NP}}{1 - \mathcal{X}_F}\right) + \frac{1}{2}\ln\left(\frac{\mathcal{X}_{NP}(r-1)+1}{\mathcal{X}_F(r-1)+1}\right) \tag{7.18}$$

where, $\mathcal{X}_F$ = Mass fraction of solute in feed

$\mathcal{X}_{NP}$ = Mass fraction of solute in final raffinate

r = Ratio of molar masses of non-solute to solute

$\mathcal{X}^*$ = For any point ($\mathcal{X}_a$, $\mathcal{Y}_a$) of operating curve, $\mathcal{X}_a^*$ is the value of $\mathcal{X}$ that is in equilibrium with $\mathcal{Y}_a$.

Fig. 7.9 *Determination of Number of Transfer Units*

If solute component or fraction of feed (A) and solvent (B) can be considered as completely immiscible liquid mixture, then it is better to use mass ratio concentrations, rather than mass fractions. For this special case N_{toR} is given by the equation

$$N_{toR} = \int_{X'_{NP}}^{X'_F} \frac{dX'}{X' - X'^*} + \frac{1}{2}\ln\left(\frac{1 + r\,X'_{NP}}{1 + rX'_F}\right) \tag{7.19}$$

where, X'_F = Mass ratio of solute to non-solute in feed

X'_{NP} = Mass ratio of solute to non-solute in final raffinate

X^*_a = For any point (X'_a, Y'_a) of operating line, X^*_a is the value of X' that is in equilibrium with Y'_a

r = Ratio of molar masses of non-solute to solute

In another special case where operating line and equilibrium curve both are straight lines, number of overall mass transfer units based on raffinate phase (N_{toR}) is given by the equation,

$$N_{toR} = \frac{\ln\left[\left(\dfrac{X'_F - (Y'_s/m')}{X'_{NP} - (Y'_s/m')}\right)\left(1 - \dfrac{1}{\varepsilon}\right) + \dfrac{1}{\varepsilon}\right]}{\left(1 - \dfrac{1}{\varepsilon}\right)} \quad (\text{if } \varepsilon \neq 1) \tag{7.20}$$

or

$$N_{toR} = [(X'_F - Y'_s/m')/(X'_{NP} - Y'_s/m')] - 1 \quad (\text{if } \varepsilon = 1) \tag{7.21}$$

where, $\varepsilon = \dfrac{m'B}{A}$ and $m' = \dfrac{Y'}{X'}$

Example 7.1 A feed stream having flow rate of 200 kg/h and containing 20 mass % acetic acid in water is to be extracted at 25°C with 400 kg/h of recycled MIBK (Methyl IsoButyl Ketone) that contains 0.1 per cent acetic acid and 0.01 per cent water. The aqueous raffinate is to be extracted down to 1 per cent acetic acid. How many theoretical stages will be required and what will the extract composition be? Extraction is to be carried out in counter current manner.

Table 7.1 Equilibrium Data[4] of Water – Acetic Acid – Methyl Isobutyl Ketone at 25°C

mass % in Raffinate				mass % in Extract			
Water	**Acetic acid**	**MIBK**	X'	**Water**	**Acetic acid**	**MIBK**	Y'
98.45	0	1.55	0	2.12	0	97.88	0
95.46	2.85	1.7	0.0299	2.8	1.87	95.33	0.0196
85.8	11.7	2.5	0.1364	5.4	8.9	85.7	0.1039
75.7	20.5	3.8	0.2708	9.2	17.3	73.5	0.2354
67.8	26.2	6.0	0.3864	14.5	24.6	60.9	0.4039
55	32.8	12.2	0.5964	22	30.8	47.2	0.6525
42.9	34.6	22.5	0.8065	31	33.6	35.4	0.9492

(Ref. Table 15.1 of Ref. 4)

$\mathcal{X}'$ = mass ratio of solute to diluent (C/A) in raffinate phase
$\mathcal{Y}'$ = mass ratio of solute to extraction solvent (C/B) in extract phase

Fig. 7.10 *Ternary Diagram for AcOH-H_2O-MIBK System*[4]

Solution

$$F + S = E_1 + R_{NP} = M \tag{7.11}$$

$$200 + 400 = M = 600 \text{ kg/h}$$

$$\mathcal{X}_m = \frac{F\mathcal{X}_F + S\mathcal{Y}_s}{F + S}$$

$$= \frac{(200 \times 0.2) + (400 \times 0.001)}{600} = 0.067\ 33,$$

$$\mathcal{Y}_1 = 0.088 \quad \text{(From Fig. 7.10)}$$

$$M\mathcal{X}_M = E_1\mathcal{Y}_1 + \mathcal{X}_{NP}R_{NP}$$

$$40.4 = 0.088\ (600 - R_{NP}) + 0.01R_{NP}$$

$$R_{NP} = 158.97 \text{ kg/h}, \quad E_1 = 441.03 \text{ kg/h}$$

Random lines from ΔR give the points of operating line ($\mathcal{X}_s$, $\mathcal{Y}_{s+1}$).

$\mathcal{X}_s$	0.2	0.14	0.08	0.065	0.01
$\mathcal{Y}_{s+1}$	0.085	0.06	0.035	0.025	0.001

Equilibrium data:

$\mathcal{X}$	0	0.0285	0.117	0.205	0.262	0.328	0.346
$\mathcal{Y}^*$	0	0.0187	0.089	0.173	0.246	0.308	0.336

Stepwise construction from $\mathcal{X}_{sF}$ ($\mathcal{X}_s$ corresponds to $\mathcal{X}_F$) to $\mathcal{X}_{NP}$ gives number of theoretical stages required for desired separation.

Fig. 7.11 *Determination of Number of Stages for Example 7.1*

From graph (Fig. 7.11), $N = 4.62$.

Example 7.2 Solve Example 7.1 considering MIBK (B, solvent) and water (A, non-solute component) of feed as completely immiscible liquids and equilibrium curve as a straight line in terms of mass ratio concentrations.

Solution

Overall solute balance

$$A(\mathcal{X}'_F - \mathcal{X}'_{NP}) = B(\mathcal{Y}'_1 - \mathcal{Y}'_s) \tag{7.14}$$

$A = 200\ (1 - 0.2) = 160$ kg/h

$B = 400\ (1 - 0.001) = 399.6$ kg/h

$$\mathcal{X}'_F = \frac{20}{80} = 0.25 \quad \mathcal{X}'_{NP} = \frac{1}{99} = 0.0101$$

$$\mathcal{Y}'_1 = ? \quad \mathcal{Y}'_s = \frac{0.1}{(100 - 0.1 - 0.01)} = 0.001$$

$$160\ (0.25 - 0.0101) = 399.6\ (\mathcal{Y}'_1 - 0.001)$$

$$\mathcal{Y}'_1 = 0.097\ \frac{\text{kg acetic acid}}{\text{kg MIBK}}$$

$\mathcal{X}'_F = 0.25,$

For operating range $\mathcal{X}' < 0.25$ slope of equilibrium curve

$$m' = \left[\left(\frac{0.0196}{0.0299}\right) \times \left(\frac{0.1039}{0.1364}\right) \times \left(\frac{0.2354}{0.2708}\right)\right]^{1/3} = 0.7571$$

$$\varepsilon = \frac{m'B}{A} = 0.7571 \times \frac{399.6}{160} = 1.89$$

$$N = \frac{\ln\left[\left(\frac{X'_F - (Y'_s/m')}{X'_{NP} - (Y'_s/m')}\right)\left(1 - \frac{1}{\varepsilon}\right) + \frac{1}{\varepsilon}\right]}{\ln \varepsilon} \tag{7.16a}$$

$$N = \frac{\ln\left[\left(\frac{0.25 - (0.001/0.7571)}{0.0101 - (0.001/0.7571)}\right)\left(1 - \left(\frac{1}{1.89}\right)\right) + \left(\frac{1}{1.89}\right)\right]}{\ln 1.89}$$

$N = 4.13$

Example 7.3 For the extraction, described in Example 7.1, calculate

(a) N_{toR} considering MIBK and water as partially miscible liquids.
(b) N_{toR} considering MIBK and water as completely immiscible liquids and equilibrium curve as straight line (in terms of mass ratio concentration).

Solution

(a) Number of overall mass transfer units based on raffinate phase is given by the equation

$$N_{toR} = \int_{X_{NP}}^{X_F} \frac{dX}{X - X^*} + \frac{1}{2}\ln\left(\frac{1 - X_{NP}}{1 - X_F}\right) + \frac{1}{2}\ln\left(\frac{X_{NP}(r-1)+1}{X_F(r-1)+1}\right) \tag{7.18}$$

$X_F = 0.2$, $X_{NP} = 0.01$

Data for equilibrium curve

X	0	0.0285	0.117	0.205	0.262	0.328	0.346
Y	0	0.0187	0.089	0.173	0.246	0.308	0.336

Data for operating curve

X_s	0.2	0.14	0.08	0.01
Y_{s+1}	0.085	0.06	0.035	0.001

Table 7.2 Data for Finding N_{toR} [from Fig. (7.12)]

X	0.2	0.15	0.1	0.05	0.025	0.01
X^*	0.113	0.089	0.063	0.032	0.014	0.001
$X - X^*$	0.087	0.061	0.037	0.018	0.011	0.009
$1/(X - X^*)$	11.494	16.393	27.027	55.555	90.909	111.111

Fig. 7.12 *Equilibrium Data for AcOH – Water – MIBK System*

Fig. 7.13 *Determination of N_{toR}*

From Fig. (7.13), $\int_{0.01}^{0.2} \frac{dX}{X - X^*}$ = 1.7236 square units = 7.0351 (Integral value)

$$r = \frac{\text{Molar mass of non-solute}}{\text{Molar mass of solute}} = \frac{18}{60} = 0.3$$

$$N_{toR} = 7.0351 + \frac{1}{2}\ln\left(\frac{1 - 0.01}{1 - 0.2}\right) + \frac{1}{2}\ln\left(\frac{0.01\,(0.3 - 1) + 1}{0.2\,(0.3 - 1) + 1}\right)$$

$$= 7.0351 + 0.106\,55 + 0.0719 = 7.213\,55$$

(b) If water (A) and MIBK (B) are immiscible liquids and equilibrium curve is straight line (in mass ratio concentration) for the given operating range

$$N_{toR} = \frac{\ln\left[\left(\dfrac{X'_F - (Y'_s/m')}{X'_{NP} - (Y'_s/m')}\right)\left(1 - \dfrac{1}{\varepsilon}\right) + \dfrac{1}{\varepsilon}\right]}{\left(1 - \dfrac{1}{\varepsilon}\right)} \tag{7.20}$$

X' $\dfrac{\text{kg of Acetic acid}}{\text{kg of water}}$	0	0.0299	0.1364	0.2708
Y' $\dfrac{\text{kg of Acetic acid}}{\text{kg of MIBK}}$	0	0.0196	0.1039	0.2354

$$X'_F = 0.25,\ m' = \left[\left(\frac{0.0196}{0.0299}\right) \times \left(\frac{0.1039}{0.1364}\right) \times \left(\frac{0.2354}{0.2708}\right)\right]^{1/3}$$
$$= 0.7571$$

$$\varepsilon = \frac{m'B}{A} = 0.7571 \times \frac{399.6}{160} = 1.89$$

$$N_{toR} = \frac{\ln\left[\left(\dfrac{0.25 - (0.001/0.7571)}{0.0101 - (0.001/0.7571)}\right)\left(1 - \left(\dfrac{1}{1.89}\right)\right) + \dfrac{1}{1.89}\right]}{\left[1 - \left(\dfrac{1}{1.89}\right)\right]}$$
$$= 5.584$$

Example 7.4 For the separation of dimethylformamide (DMF) from its dilute solution in water, liquid-liquid extraction may require lower operating cost than atmospheric distillation. Methylene chloride is considered to be a preferred solvent for extracting DMF from its aqueous solution.

DMF-water solution having a flow rate of 1000 kg/h and containing 20% DMF by mass is to be counter currently extracted with methylene chloride to reduce the DMF concentration to 1% in the final raffinate. Determine (a) the minimum amount of solvent which can be used and (b) the number of theoretical stages if actual amount of solvent is double than the minimum required.

Data: Phase equilibrium equation[6] is given by equation $Y = 0.5555\ X$ at 25° C where, Y and X are mass fractions of solute.

Solution

Mutual solubilities of water (A) and methylene chloride (B) in each other at room temperature are very low. Hence, to simplify the calculations, water (A) and methylene chloride (B) may be assumed as completely insoluble liquids.

(a) For completely immiscible liquids, solute balance is given by the equation

$$A(X'_F - X'_{NP}) = B(Y'_1 - Y'_s) \tag{7.14}$$

Table 7.3 Determination of Data for Equilibrium Line

Y	0	0.013 89	0.0278	0.042	0.055 55	0.0694	0.0833	0.1111	0.1389	0.166 65
X	0	0.025	0.05	0.075	0.1	0.125	0.15	0.2	0.25	0.3
Y'	0	0.014	0.0286	0.04384	0.0588	0.0746	0.09	0.125	0.1613	0.2
X'	0	0.0256	0.0526	0.081	0.1111	0.1429	0.1765	0.25	0.3333	0.4286

Fig. 7.14 *Determination of Equilibrium Stages for Example 7.4*

From graph $Y'_{1m} = 0.125$ [From Fig. (7.14)]

Solute balance:

$A(X'_F - X'_{NP}) = B_m(Y'_{1m} - Y'_s)$

$A = F(1 - X_F) = 1000\ (1 - 0.2) = 800$ kg/h, $X'_{NP} = 0.01/0.99 = 0.0101$

$800\ (0.25 - 0.0101) = B_m\ (0.125 - 0)$

$B_m = 1535.36$ kg/h

(b) Actual amount of solvent $B = 2 \times 1535.36 = 3070.72$ kg/h

Solute balance:

$A(X'_F - X'_{NP}) = B(Y'_1 - Y'_s)$

$800\ (0.25 - 0.0101) = 3070.72\ (Y'_1 - 0)$

$Y'_1 = 0.0625$

From Fig. 7.14, $N = 3.85$

If equilibrium curve is considered as a straight line in terms of mass ratio concentration, then number of theoretical stages required for desired separation is given by the following equation:

$$N = \frac{\ln\left[\left(\dfrac{X'_F - (Y'_s/m')}{X'_{NP} - (Y'_s/m')}\right)\left(1 - \dfrac{1}{\varepsilon}\right) + \dfrac{1}{\varepsilon}\right]}{\ln \varepsilon} \tag{7.16a}$$

$$\varepsilon = \frac{m'B}{A}$$

$$m' = \left[\left(\frac{0.014}{0.0256}\right)\left(\frac{0.04384}{0.081}\right)\left(\frac{0.0746}{0.1429}\right)\left(\frac{0.1613}{0.3333}\right)\right]^{1/4}$$

$$= 0.523$$

$$\varepsilon = \frac{0.523 \times 3070.72}{800} = 2$$

$$N = \frac{\ln\left[\left(\frac{0.25}{0.0101}\right)\left(1 - \frac{1}{2}\right) + \frac{1}{2}\right]}{\ln(2)}$$

$$N = 3.687$$

Example 7.5 For the liquid-liquid extraction system, given in Example 7.4, calculate N_{toR} for the actual amount of solvent $B = 2\,B_m$.

Solution

Water (A) and methylene chloride (B) are considered as completely immiscible liquids. Also, as shown in Fig. 7.14, equilibrium curve is a straight line in terms of mass ratio concentration. With these assumptions, formula for finding N_{toR} is

$$N_{toR} = \frac{\ln\left[\left(\frac{X'_F - (Y'_s/m')}{X'_{NP} - (Y'_s/m')}\right)\left(1 - \frac{1}{\varepsilon}\right) + \frac{1}{\varepsilon}\right]}{\left(1 - \frac{1}{\varepsilon}\right)} \tag{7.20}$$

$$B = 2B_m = 3070.72 \text{ kg/h}, \quad A = 800 \text{ kg/h},$$

$$X'_f = 0.25, \quad Y'_s = 0, \quad m' = 0.523$$

$$\varepsilon = \frac{0.523 \times 3070.72}{800} = 2, \quad X'_{NP} = 0.0101$$

$$N_{toR} = \frac{\ln\left[\left(\frac{0.25 - 0}{0.0101 - 0}\right)\left(1 - \frac{1}{2}\right) + \frac{1}{2}\right]}{\left(1 - \frac{1}{2}\right)}$$

$$N_{toR} = 5.11$$

7.6 INDUSTRIALLY IMPORTANT EXTRACTORS

Many types of extractors are available for achieving mass transfer (Ref. 4, 5, 7, 8, 9, 10). Each has its own particular advantage. As per general classification, there are two fundamental types of extractors; stagewise and differential. The stagewise extractors

consist of discrete units, in each of which the phases are brought into contact, mixed, allowed to separate and sent to next unit. Differential extractors provide continuous contact and mass transfer along the full length of the device and phases are separated only at the end.

7.6.1 Categorizing Industrially Important Extractors

The decisive criteria for the selection of the contactor device is the physical properties of the system feed/solvent as it is characterised by the density difference and interfacial tension, and the difficulty of the separation in terms of required number of theoretical stages (NTS) to reach the desired raffinate and extract concentrations. On this basis, industrial extractors can be divided into four categories:

1. Mixer-settler for low NTS requirement.
2. Unagitated static columns for low NTS requirement.
3. Agitated columns, including pulsating equipments, for high NTS requirement.
4. Centrifugal extractors for difficult separations and for low NTS requirement.

Salient features of various extractors are discussed below.

Mixer-settlers

Each mixer-settler unit provides a single stage of extraction. The two phases enter the mixing section where they are mixed using an impeller. Later the two-phases are allowed to separate by gravity due to their density differences. Typical mixer settler has mixing time of the order of a few minutes and settling times of several minutes.

Advantages

1. Provides better contact of extract and raffinate phases and thus provides higher rate of mass transfer.
2. Higher efficiency or attain separation closer to equilibrium.
3. Requires limited instrumentation.
4. Reliable scale-up.
5. Requires low head room.
6. Low capital (fixed) cost.
7. Provides more stagewise flexibility.
8. Offers wide range of capacities and simple operation.

Disadvantages

1. Requires large hold-up.
2. Higher power requirement.
3. High solvent inventory.
4. Requires large floor space.

If number of theoretical stages, required for desired separation, is less than or equal to four, mixer settler is probably the most economical amongst all types of extractors. In this extractor, turbines or propellers are used as agitators. Amongst all types of agitators, pitched blade turbine agitator [Fig. 10.2(iv)] is found to be most effective for mixing of two immiscible liquids. For smaller vessels, propellers are selected for mixing.

Types of mixer settlers There are basically two types of mixer settlers. Batch vessels are used for mixing solvent and feed solution. After mixing for a few minutes, two phases are allowed to settle. Top organic layer (generally light) is decanted out.

Figure 7.15 depicts a continuous mixer-settler system. Solvent and feed solution are pumped in the vessel with help of an axial pump. Mixer is typically equipped with slow speed, large diameter, low shear pump-mixer-type agitator(s). Pump mixers commonly have variable speed drives and are designed to avoid fine droplet formation and air entrainment which hinder coalescence in the settler. If required, auxiliary mix boxes with agitators can be used for large plants to avoid mixing inefficiencies which can occur with large mix boxes.

Inner View of
Mixer Vessel

Fig. 7.15 *Continuous Mixer Settler*

In the adjoining settler, the phases coalesce and separate by gravity and are removed via separate weir boxes as shown in the figure. Mix boxes can be cylindrical or square. Settlers are generally rectangular. Devices such as flow distributors, picket fences and various coalescence aids are installed in the settler to improve performance. Phase separation rates in the settlers are relatively slow, and the specific area is typically 3 to 5 m^3 per h (of combined aqueous and organic phases) per m^2 of settler area, depending on operating temperature, aqueous feed composition and concentration of the extract phase in the organic phase.

Mixer settlers have become popular in nuclear industry. For this purpose, Krebs mixer-settler is a popular design. This design is similar to Fig. 7.15 but the agitator is fitted with a low head, high volume, low shear conical pump, consisting of a static

tulip shaped stator and a trapezoidal, typically six bladed rotor. Settler is of compact design and thus occupies less floor space. It is usually designed for flow rate of 8 to 12 $m^3/(m^2 \cdot h)$.

Unagitated static columns There are two types of columns that are used for extraction operation; packed column and sieve plate column.

In industry, the use of packed columns (random or structured) for liquid-liquid extraction applications will be in most cases found limited to operations where the density differences between the carriers is higher than 30–50 kg/m^3, the volumetric phase ratio (φ) between dispersed drop phase and continuous phase is in the magnitude of $0.5 < \varphi < 5$ and the number of theoretical stages (NTS) ≤ 10. Figure 7.16 schematically represents variety of packed columns for this service. It may be noted that arrangements for heavy or light dispersed phases are different for packed beds.

(a) Typical Column Arrangement for Light Liquid Dispersed

(b) Typical Column Arrangement for Heavy Liquid Dispersed

Fig. 7.16 *Typical Column Arrangement for Packed Columns for Liquid Extractions*

There is a different function of the column internals in vapour/liquid services like distillation or absorption, where the specific surface of the packing provides the required area to perform mass transfer. In contrast, liquid-liquid extraction columns the mass transfer area is almost independent of the packing surface, but is directly linked to the drop phase holdup in the tower. The main function of the packing is to provide an increased flow path length and more residence time for the drop phase.

Coalesced drops are redispersed at the sharp edges of the packing and the residence time distribution of both phases should be kept as narrow as possible (plug flow) by minimizing any axial back mixing effect. It is preferred to select a packing that is wetted by continuous phase. Historically random packings were the choice in packed towers but with technical developments, structured packings also find applications in extraction due to better turn down ratio and higher throughputs.

Typical column internals for extraction service are also shown in Fig. 7.17. It may be seen that dispersion tray and heavy phase inlet distributor are somewhat different from those used for distillation/absorption service. Two designs of coalescers are included. This piece of internals plays an important role as both phases should separate well before they are allowed to come out from the extractor. These coalescers are produced by two dissimilar filaments, knitted together to form the mesh. One filament is a metallic wire made of stainless steel or an alloy and the other is made of PP, PTFE, glass fibres, etc. In cartridge form, the liquid flows from the centre radially outwards. For extraction columns, mesh is made up of say 75 μm size filaments.

Spray Nozzle Distributor

Random Packing Support Grid

Hold Down Plate

Dispersion Plate

Structured Packings Support Grid

Fig. 7.17 *(Contd.)*

Fig. 7.17 *Packed Tower Internals for Liquid-Liquid Extraction*

Advantages

1. High specific throughput capacity.
2. Easy operation and maintenance because no moving parts are involved.
3. For corrosive media there are almost unlimited options for the material selection available, from stainless steel to high-alloyed metal, from polypropylene to PTFE, technical ceramics or pure carbon.
4. A simple operation also at high pressure or temperature.

Disadvantages

1. Although empirical equations are available for design of packed columns, it is still advisable to conduct pilot studies for fixing mass transfer coefficient $(K_L a)$ and deciding on flooding velocity.
2. Packed columns are costly as compared to mixer settlers.
3. Selection of internals plays important role in its functioning. Hence, design of packed columns is required to be carried out with care.

Sieve tray column of special design can also be used for liquid-liquid extraction (Fig. 7.18). Unlike sieve tray column for distillation/absorption, it has coalescer/disperser below each sieve tray and weirs are not provided on the trays. Sieve tray column is less preferred to a packed column for extraction service.

One of the more common liquid-liquid extraction applications is the removal of sulphur compounds from liquefied petroleum gas (LPG) using an amine solution. A packed tower sizing may be based on an amine superficial velocity of 5 $m^3/(m^2 \cdot h)$. Normally LPG is a dispersed phase in this application.

Another application of a packed column extractor is of organic contaminants removal from waste water. When organic compound's boiling point is more than that of water, liquid-liquid extraction offers a better option. Acetic acid, pyridine, aniline, nitrobenzene, etc., are some organic contaminants in waste water that can be extracted with a solvent and recovered with low energy requirements.

Agitated columns Several designs of agitated columns are available in industry for liquid-liquid extraction service. In this category, two types of extractors are considered. One is pulsed column and another is a rotating disc contactor.

Fig. 7.18 *Sieve Plate Extractor*

1. Pulsed Columns: The pulsed column [Fig. 7.19(a)] consists of a vertical cylindrical contacting section with settlers at the top and bottom. The contacting section is fitted with either sieve plates or "disc and doughnut" plates. Generally, organic phase is continuous and aqueous phase is dispersed. Both are continuously fed in countercurrent directions. Energy is applied to pulse the liquids in the column up and down. One way is done by injecting pressurized air into a pulse leg that pushes liquid into the column, then venting the pulse leg to fill the pulse leg with solution from the column. Alternately energy can be supplied by mechanical devices, such as pistons, that move back and forth to displace the solution in the column. The pulse action lifts and lowers the solution in the column, usually only a fraction of a metre.

Advantages

1. Potentially lower organic inventory than conventional mixer settlers.
2. Small floor space area.
3. Ability to operate with the solution with the smaller flow as the continuous phase.
4. No phase recycle needed.
5. Can incorporate a large number of stages in one column.
6. Totally enclosed system, excludes dust, minimises evaporative losses, improves fire protection, and (excludes air in mechanically agitated extractor).
7. Has greater tolerance for suspended solids.
8. No internal moving parts; low shear; low entrainment; low maintenance.

Fig. 7.19(a) *Agitated Extractors*

9. Ability to vary droplet size, by varying pulse frequency and amplitude.
10. Opportunity to use inexpensive FRP for construction.

Disadvantages

1. HTS requirement is high.
2. High head room requirement.
3. Less flexible for large feed flow variations.
4. Pilot scale testwork needed to develop scale-up data.
5. Less accessible for clean-out.
6. Visual monitoring of process difficult.
7. Power is required for pulse generation.

2. Rotating Disc Columns (RDC) Two designs are figured in this category. Scheibel extractor is shown in Fig. 7.19(b). The impellers are turbine type and disc and doughnut baffles surround them. In another design [Fig. 7.19(c)], a simple disc is provided in the centre and horizontal baffles are provided near the wall. The disc rotates at much higher speed than turbine-type impellers. In Kuhni design (third design), instead of baffles, sieve plates are provided between two discs.

(b) Scheibel Extractor

(c) Rotating Disc Extractor

Fig. 7.19(b) and 7.19(c)

The disc contactor consists of a cylindrical column that is divided into number of compartments formed by a series of stator rings. Each compartment contains a centrally located, horizontal rotor disk that creates a high degree of turbulence inside the column. Typically, the disk diameter is 1/3rd to 2/3rd of the column diameter. The disk disperses the liquid and forces it outward toward the wall where the stator rings create quite zones where the two phases can separate. Most extraction takes place in the mixing section but some does take place in the calming section so that efficiency of each mixer settler unit is sometimes greater than 100%. Typically, each mixer-settler compartment is 300 to 600 mm high.

Advantages

1. Good throughput.
2. High efficiency means more stages per metre height.
3. Less head room and less floor space.

Disadvantages

1. Corrosive liquids can damage the internal moving parts.
2. Pilot study is recommended for scale-up.

Centrifugal extractors

Centrifugal contactors are high-speed rotary machines with the advantage of very low residence time. Centrifugal contactors utilise a mechanical device to agitate the mixture to increase the interfacial area and decrease the mass transfer resistance. In this machine, heavier phase is transported to the periphery and the lighter phase to the centre which means centrifugal force is responsible for the countercurrent flow of the phases. These are ideal for systems in which the density difference is less than 4%.

Centrifugal extractors are expensive but provide many theoretical stages of separation with very short hold-up time (about 4 s). While it is possible to obtain many theoretical stages in a single machine, 3 to 5 theoretical stages are more common. They are therefore used to extract valuables and heat sensitive products such as vitamins and antibiotics.

Figure 7.20(a) and (b) show single stage and a four stage centrifuges for extraction service, respectively.

Advantages

1. High throughput
2. Less amount of solvent is required
3. Short residence time by fast reaching the equilibrium

Disadvantage

1. High initial cost

Centrifugal extractors have become popular in pharmaceutical industry.

(a) Single Stage Centrifugal Extractor

(b) Four Stage Centrifugal Extractor

Fig. 7.20 *Centrifugal Extractors*

7.6.2 Comparison of Various Industrial Extractors

With availability of different extractors, it is necessary to compare them for their efficiencies and their processing capability. Process engineer can use Fig. 7.21 for selection of the best suited extractor for his application.

MS = Krebs Mixer Settler
SE = Sieve Plate Extractor
FK = Random Packed Column
PSE = Pulsed Sieve Plate Column
RDC-1 = Rotating Disc Contractor, Recommended by Dr. J. Stichlmair
RDC-2 = Rotating Disc Contractor, Recommended by Dr. T. Holmes, et al.
Kuhni = Kuhni Extractor
York-Scheibel = Scheibel Column
PFK = Pulsed Packed Column

Fig. 7.21 *Comparison Plot of Various Commercial Extractors*[4]
(Reproduced with Permission from Perry's Chemical Engineers' Handbook, 7th Edition)

Selection criteria and performance of various extractors are qualitatively compared in Tables 7.4 and 7.5.

Table 7. 4 Selection Criteria for Continuous Extractors

Type of extractor	Number of stages	Flow rate	Residence time	Interfacial tension	Viscosity	Density difference	Floor space
Mixer-settler	Low	High	Very high	Moderate to high	Low to high	Low to high	High
Static (unagitated) columns	Moderate	Moderate	Moderate	Low to moderate	Low to moderate	Low to moderate	Low
Agitated columns	High	Moderate	Moderate	Low to high	Low to high	Low to high	Low
Centrifugal extractor	High	Low	Very low	Low to moderate	Low to moderate	Low to moderate	Moderate

Table 7.5 Performance Comparison of Extractors

Type of Extractor	HETS, m	Superficial velocity of continuous and dispersed phases, m/h	Liquid capacity of combined streams, $m^3/(m^2 \cdot h)$	Stage efficiency, %
Mixer settler (single stage)	0.75–1		3–5	75–100
Sieve tray tower	0.5–3.6	27–75	3–60	6–24
Packed Tower	0.6–1.5	12–40 (Pall rings) 65–90 (Structured)	6–45	–
Pulsed column	0.8–1.3	25–35	–	–
Rotating disc contactor	0.3–0.5	7–30	15–30	80–100
Scheibel extractor	0.2–0.4	6–14	15–30	80–100
Centrifugal extractor	–	75–110	–	300–1000

7.6.3 Process Design of Mixer

It includes two steps:

(i) Determining the volume of the mixer.

(ii) Process design of agitator; e.g. deciding shape factors and power requirement.

(iii) Mixer settlers are operated in batchwise mode and also in continuous manner. For batch mixer, volume of mixer is decided by the total volume of liquid mixture, charged to the mixer. For continuous mixer, volume of the mixer depends on desired hold up time.

Ideally, hold up time in a continuous mixer is the time required to reach equilibrium. It also depends on the rate of mass transfer. It can be decided on the basis of pilot plant or lab scale experiments of the given system.

(iv) **Process Design of Agitator (Ref. 2 and 11)**

The work of Miller and Mann and of Olney and Carlson have established that to calculate power required for agitating two liquid phases one can use the power data available for agitating single phase liquids, if density and viscosity of two phase liquid mixture are determined by following equations.

$$\rho_{av} = \rho_C Z_C + \rho_D Z_D \tag{7.22}$$

$$\mu_{av} = \frac{\mu_C}{Z_C}\left(1 + \frac{1.5\mu_D Z_D}{\mu_D + \mu_C}\right) \tag{7.23}$$

where, μ_{av} = Average viscosity of liquid mixture cP or mPa · s
ρ_{av} = Average density of two-phase liquid mixture, kg/m^3
ρ_C = Density of continuous phase, kg/m^3
ρ_D = Density of dispersed phase, kg/m^3
Z = Volume fraction of appropriate phase
μ_C = Viscosity of continuous phase, cP or mPa · s
μ_D = Viscosity of dispersed phase, cP or mPa · s

Fig. 7.22 *Agitator Power Correlations*[4]

Curve 1: Six Flat Blade Turbine, D_a/W_i = 5, Four Baffles Each $D_T/12$, (Refer to Fig. 10.2 (v))
Curve 2: Six Flat Blade Open Turbine, D_a/W = 8, Four Baffles Each Equal to $D_T/12$, (Refer to Fig. 10.2 (ii)
Curve 3: 45° Pitched Blade Turbine, D_a/W_i = 8, Four Baffles, Each $D_T/12$, (Refer to Fig. 10.2 (iv). but with six blades)
Curve 4: Propeller Pitch Equal to $2D_a$, Four Baffles, Each $D_T/12$, (Refer to Fig. 10.2 (i))
Curve 5: Propeller Pitch Equal to D_a, Four Baffles, Each $D_T/10$, (Refer to Fig. 10.2 (i))
(Reproduced with the Permission of McGraw-Hill Education, U.S.A.)

Power data for agitating single phase liquids in baffled vessel are available in the form of graph N_p vs Re [Fig. (7.22)]. The same can be used to calculate the power required for mixing two phase liquid mixture.

$$N_p = \frac{Pg_c}{\rho_{av} n^3 D_a^5} \tag{7.24}$$

$$Re = \frac{nD_a^2 \rho}{\mu_{av}} \tag{7.25}$$

where, N_p = Power number
Re = Reynolds number
P = Power required for agitation, W
g_c = Newton's law of gravitational constant, (g_c = 1 for SI system)
n = Impeller rotational speed, s^{-1}
D_a = Impeller diameter, m
ρ_{av}, μ_{av} = Density and viscosity of two-phase liquid mixture, can be determined by Eqs. (7.22) and (7.23), respectively.

7.6.4 Process Design of Batch Settlers (Ref. 3, 4, 11)

Settlers are also known as *decanters*. They are used to separate two immiscible liquids or two partially miscible liquids. The mixture of liquids leaving a mixer, is a cloudy dispersion which must be settled, coalesced and separated into its liquid phases. It requires sedimentation and coalescence of the drops of the dispersed phase. Decanters used are of the various shapes as shown in Fig. (7.23). It can be horizontal or vertical.

Volume of settler is estimated by taking hold-up time equal to 5 to 10 min. Laboratory experiments for deciding hold-up time could prove valuable.

(a) Horizontal Gravity Settler

(b) Vertical Gravity Settler

Fig. 7.23 *Batch Settlers*

Diameter or size of the decanter is decided on the basis of fact that velocity of continuous phase should be less than settling velocity of the droplets of dispersed phase. Velocity of continuous phase is calculated by the equation

$$u_c = \frac{q_c}{A_i} < u_d \tag{7.26}$$

where, u_c = Velocity of continuous phase, m/s
q_c = Continuous phase volumetric flow rate, m^3/s
A_i = Area of interface, m^2
u_d = Settling velocity of the dispersed phase droplets, m/s

Stokes law is used to determine the settling velocity of the droplets.

$$u_d = \frac{d_d^2 g(\rho_d - \rho_c)}{18\mu_C} \tag{7.27}$$

where, d_d = Droplet diameter, m
u_d = Settling velocity of the dispersed phase droplet, m/s
g = Acceleration due to gravity = 9.81 m/s^2
ρ_c = Density of continuous phase, kg/m^3
ρ_d = Density of dispersed phase, kg/m^3
μ_C = Viscosity of continuous phase, $N \cdot s/m^2$ or $kg/(m \cdot s)$

Droplet size (d_d) can be assumed equal to 150 μm. In decanter, feed droplet sizes are normally well above 150 μm. If the calculated settling velocity is greater than 4×10^{-3} m/s, then a figure of 4×10^{-3} m/s is to be used.

For vertical decanter, take $L = 2\ D_i$, thickness of dispersion band = 0.1 L and residence time of droplets in the dispersion band = $0.1\ L / u_d$. The residence time of droplets in dispersion band should be 2 to 5 min.

For a horizontal cylindrical decanter, the interfacial area A_i depends on the position of the interface.

$$A_i = WL \tag{7.28}$$

$$W = 2\sqrt{2rZ - Z^2} \tag{7.29}$$

where, W = Width of interface, m
Z = Height of the interface from the base of the vessel, m
L = Length of cylinder, m
r = Radius of cylinder, m

Fig. 7.24 *Geometry of Horizontal Separator*

In the approximate method, for horizontal decanter,

take $L/D_i = 4$ and $D_i = 8.4\sqrt{q_c + q_d}$ (7.30)

where, D_i = Inside diameter, m

q_d, q_c = Volumetric flow rate, m^3/s (for horizontal decanter)

Example 7.6 For the extraction system described in Example 7.1, design a mixer-setler-type extractor. Following data are obtained from pilot study:

(i) Residence time (hold-up time) of liquid mixer in pilot plant mixer, $\theta = 6$ min
(ii) Stage efficiency = 0.92
(iii) Type of agitator = 45° pitch blade turbine
(iv) Shape factors of pilot plant mixer: $D_a/W_i = 8$, four baffles, $D_T/J = 12$, six blades, $H/D_T = 1$, $D_a/D_T = 0.33$
where, D_a = Diameter of agitator, m
D_T = Inside diameter of mixing vessel, m
J = Width of baffle, m
H = Depth of liquid tank, m
W_i = Width of agitator blade, m
(v) Power required per unit volume in pilot plant = 220 W/m^3

Solution

Treybal[2] demonstrated that for geometrically similar agitated vessels with equal holding time and equal power per unit volume on the two scales, the stage efficiency is likely to increase on scale-up. Based on this proposition, residence time in commercial scale mixer can safely be taken as 6 min and stage efficiency is 0.92.

Total number of mixer settlers = $4.62/0.92 \cong 5$

Working volume of mixer = Hold up time × Volumetric flow rate of liquid mixture

$$V_w = \theta \times q_v$$

$$q_v = (\text{Mass flow rate of feed} + \text{Mass flow rate of final extract})/\rho_{av}$$

$$\rho_{av} = \rho_C Z_C + \rho_D Z_D$$

$$\rho_{MIBK} = 801 \text{ kg/m}^3, \quad \rho_{water} = 1000 \text{ kg/m}^3, \quad \rho_{Acetic\ acid} = 1049 \text{ kg/m}^3$$

This mixer–settler will be operated in continuous manner. If first mixer is considered, then extract phase (MIBK rich phase) is the lighter phase and feed (water rich phase) is the heavier phase. Feed density is estimated as

$$\rho_F = \frac{1}{\left(\dfrac{w_1}{\rho_1} + \dfrac{w_2}{\rho_2}\right)} \tag{6.55}$$

where, w_1, w_2 = Mass fractions of components

ρ_1, ρ_2 = Densities of components

$$\rho_F = \frac{1}{\left(\frac{0.2}{1049} + \frac{0.8}{1000}\right)} = 1009.4 \text{ kg/m}^3$$

$$\rho_E = \frac{1}{\left(\frac{0.088}{1049} + \frac{0.892}{801} + \frac{0.02}{1000}\right)} = 821.36 \text{ kg/m}^3$$

Volumetric flow rate of feed = 200/1009.4 = 0.198 m^3/h

Volumetric flow rate of final extract = 441.03/826.36 = 0.5337 m^3/h

Final extract is having higher volumetric flow rate than feed. Hence, feed or water-rich layer is the dispersed phase and final extract (solvent rich layer) is the continuous phase.

$$Z_D = \frac{200/1009.4}{\left(\frac{200}{1009.4} + \frac{441.03}{821.36}\right)} = 0.27$$

$$Z_C = 1 - Z_D = 0.73$$

$$\rho_{av} = \rho_C Z_C + \rho_D Z_D$$

$$= 821.36 \times 0.73 + 1009.4 \times 0.27 = 872.13 \text{ kg/m}^3$$

$$q_v = (200 + 441.03)/872.13 = 0.735 \text{ m}^3\text{/h}$$

$$\frac{H}{D_T} = 1, \quad V_w = 0.735\left(\frac{6}{60}\right) = 0.0735 \text{ m}^3$$

$$\frac{\pi}{4} D_T^2 H = 0.0735$$

$$\frac{\pi}{4} D_T^3 = 0.0735 \text{ m}^3$$

$$D_T = 0.454 \text{ m}$$

For 45° pitch blade turbine, $D_a/D_T = 0.33$, where D_a = diameter of agitator, m

$$D_a = 0.1498 \text{ m}$$

Power required per unit volume = 220 W/m^3

$$P = 220 \times 0.0735 = 16.17 \text{ W}$$

For first trial calculations, let tip velocity of agitator, v = 200 m/min = $\pi D_a n$

$$\pi \times 0.1498 \times n = 200$$

$$n = 424.98 \text{ rpm} \simeq 425 \text{ rpm}$$

For pure components:

$$\mu_{\text{MIBK}} = 0.5 \text{ mPa} \cdot \text{s}, \mu_{\text{water}}$$

$$= 0.8 \text{ mPa} \cdot \text{s}, \mu_{\text{Acetic acid}} = 1.1 \text{ mPa} \cdot \text{s}$$

For organic liquid mixture[12],

$$\frac{1}{\mu_m} = \frac{w_1}{\mu_1} + \frac{w_2}{\mu_2} + \frac{w_3}{\mu_3} \tag{7.31}$$

$$\frac{1}{\mu_C} = \frac{0.088}{1.1} + \frac{0.02}{0.8} + \frac{0.892}{0.5} = 1.889$$

$$\mu_C = 0.5294 \text{ mPa} \cdot \text{s}$$

$$\frac{1}{\mu_D} = \frac{w_{AA}}{\mu_{AA}} + \frac{w_{\text{water}}}{\mu_{\text{water}}} = \frac{0.2}{1.1} + \frac{0.8}{0.8} = 1.1818$$

$$\mu_D = 0.846 \text{ mPa} \cdot \text{s}$$

$$\mu_{\text{av}} = \frac{0.5294}{0.73}\left(1 + \frac{1.5 \times 0.27 \times 0.846}{(0.846 + 0.5294)}\right) = 0.9059 \text{ mPa} \cdot \text{s}$$

$$Re = \frac{nD_a^2\rho_{\text{av}}}{\mu_{\text{av}}} = \frac{(425/60) \times 0.1498^2 \times 872.13}{0.9059 \times 10^{-3}} = 153\,025$$

$$N_p = \frac{Pg_c}{\rho_{\text{av}} n^3 D_a^5} = 1.5 \qquad \text{[From Fig. (7.22)]}$$

$$P = \frac{1.5 \times 872.13 \times \left(\frac{425}{60}\right)^3 \times 0.1498^5}{1}$$

$$= 35 \text{ W} >> 16.17 \text{ W}$$

Hence, decrease the value of tip velocity to match this value.
Let for second trial calculation, tip velocity = 155 m/min

$$n = \frac{155}{(\pi \times 0.1498)} = 329.36 \text{ rpm} \simeq 329 \text{ rpm}$$

$$Re = 153\,025 \times \frac{329}{425} = 118\,459$$

$$N_p = 1.5 \quad \text{(From Fig. 7.22)}$$

$$P = \frac{1.5 \times 872.13 \times \left(\frac{329}{60}\right)^3 \times 0.1498^5}{1} = 16.27 \text{ W, close to } 16.17 \text{ W}$$

Use 45° pitch blade turbine, having diameter $D_a = 0.1498$ m and rotational speed, $n = 330$ rpm.

(b) Process design of settler

(i) Let position of settler be horizontal.

Approximate diameter of horizontal settler

$$D_i = 8.4\sqrt{q_c + q_d} \tag{7.30}$$

$$q_c = \frac{441.03/3600}{826.36} = 1.4825 \times 10^{-4} \text{ m}^3/\text{s}$$

$$q_d = \frac{200/3600}{1009.4} = 5.503\,82 \times 10^{-5} \text{ m}^3/\text{s}$$

$$D = 8.4\sqrt{q_c + q_d} = 0.1197 \simeq 0.12 \text{ m}$$

This equation is valid for $L/D_i = 4$.
Length of horizontal decanter $L = 0.48$ m
D and L by other method:
Settling velocity of the droplets of dispersed phase; Eq. (7.27)

$$d_d = 150 \times 10^{-6} \text{ m}, \quad g = 9.8 \text{ m/s}^2, \quad \rho_d = 1009.4 \text{ kg/m}^3$$

$$\rho_c = 821.36 \text{ kg/m}^3, \quad \mu_c = 0.5294 \text{ mPa} \cdot \text{s}$$

$$u_d = \frac{(150 \times 10^{-6})^2 \times 9.81 \times (1009.4 - 821.36)}{18 \times 0.5294 \times 10^{-3}}$$

$$= 0.004\,355 \text{ m/s} \quad > \quad 4 \times 10^{-3} \text{ m/s}$$

Hence, $\quad u_D = 0.004$ m/s

$$A_{\min} = \frac{L_c}{u_d} = \frac{q_c}{u_d} = \frac{(0.5337/3600)}{0.004} = 0.037 \text{ m}^2$$

$$A_{\min} = (W_i L)_{\min}$$

If the position of interface is fixed at the centreline of settler then $W_i = D_i$, let $L = 4D_i$.

$$A_{\min} = 4D_i^2 = 0.037 \text{ m}^2$$

$$D_{i\,\min} = 0.096 \text{ m} < D_i = 0.12 \text{ m}, L = 0.48 \text{ m}$$

To fix the position of interface at centreline of decanter $Z_i = D_i/2$, locations of outlet nozzles can be determined based on the pressure balance.

Fig. 7.25 *Location of Nozzles for Horizontal Decanter*

Pressure balance:

$$(z_h - z_i)\,\rho_H = (z_l - z_i)\,\rho_L$$

ρ_H = density of heavier phase = 1009.4 kg/m^3

ρ_L = density of lighter phase = 821.36 kg/m^3

$$z_i = D_i/2 = 0.12/2 = 0.06 \text{ m}$$

Let $\quad z_l = 0.8\,D_i = 0.8 \times 0.12 \text{ m} = 0.096$ m

$$(z_h - 0.06) \times 1009.4 = (0.096 - 0.06) \times 821.36$$
$$z_h = 0.0893 \text{ m}$$

Vertical decanter (other option):

$$A_{i\min} = (\pi/4)\, D_{i\,\min}^2 = 0.037 \text{ m}^2$$
$$D_{i\,\min} = 0.217 \text{ m}$$

Let $D_i = 0.25$ m $\quad L = 2 \times 0.25 = 0.5$ m

Width of dispersion bend = 0.1 L = 0.05 m

Residence time of the droplets of dispersed phase in dispersion band,

$$\theta = \frac{0.05}{u_d} = \frac{0.05}{0.004\,355} = 11.481 \text{ s}$$

As per the guidelines, this should preferably be 120 s. For 120 second residence time, L = 5.226 m (very high). This guide line is given for higher flow rates (1500 kg/h to 600 kg/h).

Hence, let D_i = 0.25 m and L = 1 m which means θ = 23 seconds.

To fix the interface at the centre of vertical decanter, locations of nozzles must be determined.

Fig. 7.26 *Location of Nozzles for Vertical Decanter*

Pressure balance:

$$(z_l - z_i)\, \rho_L g + Z_i\, \rho_H\, g = z_h\, \rho_H\, g$$

$$z_h = (z_l - z_i)\frac{\rho_L}{\rho_H} + z_i$$

$$z_i = 0.5 \text{ m}, \qquad z_l = 0.9\, L = 0.9 \text{ m}$$

$$z_h = (0.9 - 0.5) \times \frac{821.36}{1009.4} + 0.5 = 0.8255 \text{ m}$$

Two criteria may be used for scale-up of agitated vessel; (a) constant tip velocity and (b) constant power per unit volume.

Criteria of constant tip velocity is used for miscible liquids where heat is transferred from coil or jacket or where suspended solids are involved.

Criteria of constant power per unit volume can be used for immiscible liquid systems gas-liquid systems, emulsions and pastes.

Incorporation of coalescer pads, such as that shown in Fig. 7.17, can improve the separation of phases in the decanters.

7.6.5 Process Design of Packed Tower-Type Extractor (Ref. 4, 7, 8, 9)

In packed tower-type extractor, standard commercial packings, used in vapour-liquid system, are used. This includes structured packings, Raschig and Pall rings, Berl and Intalox saddles and other random packings. Packing reduces the vertical back mixing of the continuous phase and thereby reduces height required for mass transfer. It is recommended to redistribute the dispersed phase at about every 1.5 to 3 m distance to generate new droplets. Size of packing should preferably be less than one-eighth of the tower diameter to minimise the wall effect. Flooding velocity in packed tower is the velocity of dispersed phase or continuous phase at which dispersed phase is coming out from the outlet pipe of continuous phase or vice versa.

1. **Tower diameter:** Diameter of packed tower is determined based on value of flooding velocity. To find the flooding velocity in packed tower many correlations are available. One of the best correlations; Crawford–Wilke correlation, is represented in Fig. 7.27.

Fig. 7.27 *Flooding in Packed Towers, Crawford-Wilke Correlation*[3]
(Reproduced with the Permission of McGraw-Hill Education, U.S.A.)

where, σ_c = Interfacial tension of continuous phase, lbf/ft
g_c = Newton's law gravitational constant
= 4.18×10^8 (lb · ft)/(lbf · h^2)
ρ_C = Density of continuous phase, lb/ft^3
μ_C = Viscosity of continuous phase, lb/(ft · h)
$\Delta\rho$ = Difference in density, lb/ft^3
a_p = Specific packing surface, ft^2/ft^3
ε = Fraction void volume in packed section
v_{CF}, v_{DF} = Superficial flooding velocity, ft/h

Actual velocity of the continuous phase and the same of the dispersed phase should be less than or equal to 50% of the flooding values.

2. **Height of packed tower:** Height of packed tower H is given by equation.

$$H = N_{toR} \cdot H_{toR} = N_{toE} \cdot H_{toE} \tag{7.32}$$

Value of H_{toR} or H_{toE} should be decided by actual experiments at pilot plant scale. Value of H_{toR} in packed tower depends on type of system, type of packing, size of packing, tower diameter, etc. Hence, it is difficult to find the suitable correlation for finding H_{toR} for the given system.

For example, equation for finding H_{toR} for acetic acid-water-MIBK system, in which water rich phase is the raffinate phase and MIBK rich phase is the extract phase, is given by equation[1].

$$H_{toR} = 0.2347\left(\frac{v_C}{v_D}\right)^{0.648} \tag{7.33}$$

where, H_{toR} = Height of equivalent overall transfer unit based on raffinate phase, m
v_D = Actual superficial velocity of dispersed phased, m/s
v_C = Actual superficial velocity of continuous phase, m/s

This equation was derived by Sherwood *et al.*[1] for the packed tower having 0.09 m diameter, 12.7 mm carbon rings at 25°C for the certain range of flow rates. Hence, the use of this equation for the same system with larger diameter packed tower, different type and size of packing and for different values of flow rates, may not be reliable. So the equation of H_{toR} or H_{toE} should be determined on the same system at pilot plant scale with similar type of equipment and with similar conditions like same values of superficial velocities, same operating temperature, etc. However, Eq. (7.33) may be used for the preliminary design of the pilot plant equipment.

3. **Criteria of selection:** If number of theoretical stages required for desired separation is less and limited floor area is available in the plant than packed tower can be selected as extractor. Packed tower consumes much less power than mixer settler.

Another method for finding height of packing for the packed-type extractor or height of other continuous extractor, based on HETS (height equivalent to theoretical

stage), is as follows. The height of extraction zone cane be determined by the following equation:

$$H = \text{HETS} \times N \tag{7.34}$$

where, H = Height of extraction zone, m
N = Number of theoretical stages required for desired separation

HETS value can be selected from Table 7.6.

Table 7.6 Approximate HETS Values of Extractors

Type of Extractor	HETS, m	Superficial velocity of continuous and dispersed phase, m/h
Sieve tray tower	0.5–3.6	27–75
Packed tower – Pall rings	0.6–1.5	12–40
Packed tower – Structured packing	0.6–1.3	65–90
Rotating disc contactor	0.3–0.5	7–30
Scheibel extractor	0.2–0.4	6–14
Pulsed sieve column	0.8–1.3	25–35

For the design of pilot plant extractor, Table 7.6 and Eq. (7.34) can be used. However, the same cannot be used directly for the design of commercial scale extractor.

Example 7.7 For the extraction system described in Example 7.1, design the packed tower-type extractor.

(a) Tower Diameter:
Let type of packing: SS Pall rings
Size: 16 mm (for first trial calculations)
From Table 9.1 of Chapter 9,

a_p = specific packing surface = 340 m^2/m^3
ε = fraction of void volume = 0.92

To find the flooding velocities, first the value of following function must be determined:

$$f = \left(\frac{\sigma_C g_C}{\rho_C}\right)^{0.2} \left(\frac{\mu_C}{\Delta\rho}\right)\left(\frac{a_p}{\varepsilon}\right)^{1.5} \tag{7.35}$$

where, σ_C = Surface tension of continuous phase, lbf/ft

From Example 7.6, volumetric flow rate of feed = 0.198 m^3/h

Volumetric flow rate of final extract = 0.5337 m^3/h.

Volumetric flow rate of extract is very much greater than the same of feed, hence extract is the continuous phase and feed (heavy phase) is the dispersed phase. Figure 7.16(b) represents the system to be adopted.

Final extract contains 89.2% MIBK, 8.8% acetic acid and 2% water by mass.

Surface Tension of MIBK:

Structure of MIBK:

(Molar mass = 12 × 6 + 16 + 12 = 100)

For hydrogen bonded compound, Sugden equation[4] can be used to predict surface tension of a pure compound.

$$\sigma^{1/4} = [\mathcal{P}]\,(\rho_L - \rho_G) \tag{7.36}$$

where, σ = Surface tension, dyn/cm

ρ_L = Liquid density, mol/cm^3

$[\mathcal{P}]$ = Temperature independent parachor value

For MIBK,

$$\rho_L = 801 \text{ kg/m}^3$$

$$\rho_G = \frac{pM}{RT} = \frac{1 \times 100}{(273 + 30)} \times \frac{273}{1 \times 22.414}$$

$$\rho_G = 4 \text{ kg/m}^3$$

$$\rho_L = 801 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{10\,0} = 0.008 \text{ mol/cm}^3$$

$$\rho_G = 4 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{100} = 4 \times 10^{-5} \text{ mol/cm}^3$$

From Table 3.343, Ref. 4,

$$[\mathcal{P}] = 171.9 + 55.5 + 29 = 256.4$$

$$\sigma_M^{1/4} = 256.4\,(0.008 - 4 \times 10^{-5}) = 2.0409$$

$$\sigma_M = 17.35 \text{ dyn/cm}$$

For acetic acid,

$$\rho_L = 1049 \text{ kg/m}^3$$

$$\rho_G = \frac{pM}{RT} = \frac{1 \times 60}{(273 + 30)} \times \frac{273}{1 \times 22.414} = 2.41 \text{ kg/m}^3$$

$$\rho_L = 1049 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{60} = 0.017\,48 \text{ mol/cm}^3$$

$$\rho_G = 2.41 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{60} = 4.016 \times 10^{-5} \text{ mol/cm}^3$$

$$[\mathcal{P}] = 55.5 + 73.8 = 129.3$$

$$\sigma_A^{1/4} = 129.3\,(0.01748 - 4.016 \times 10^{-5})$$

$$\sigma_A = 25.86 \text{ dyn/cm}$$

For water, $\sigma_W = 71.4$ dyn/cm (Ref. 4)

Surface tension of liquid mixture[4] is given by the following equation:

$$\sigma_m^{1/4} = \Sigma \sigma_i^{1/4} x_i \tag{7.37}$$

where, x_i = mole fraction of component i in liquid phase

$$x_{\text{MIBK}} = \frac{0.892/100}{\left(\frac{0.088}{60} + \frac{0.892}{100} + \frac{0.02}{18}\right)} = 0.7758$$

$$x_{\text{AcOH}} = \frac{0.088/60}{\left(\frac{0.088}{60} + \frac{0.892}{100} + \frac{0.02}{18}\right)} = 0.1276$$

$$x_{\text{H}_2\text{O}} = 1 - 0.7758 - 0.1276 = 0.0966$$

$$\sigma_C^{1/4} = 17.35^{1/4} \times 0.7758 + 25.86^{1/4} \times 0.1276 + 71.4^{1/4} \times 0.0966$$

$$\sigma_C = 21.443 \text{ dyn/cm}$$

$$\equiv 0.021\,443 \text{ N/m}$$

From Example 7.6,

$$\rho_C = 821.36 \text{ kg/m}^3,\ \mu_C = 0.5294 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$g_c = 1, \quad \Delta\rho = \rho_D - \rho_C = 1009.4 - 821.36 = 188.04 \text{ kg/m}^3$$

To use Fig. 7.20 (Crawford – Wilke correlation), all variables must be in US customary units.

$$\sigma_C = 0.021\,443 \times \frac{1}{4.448\,22} \times \frac{1}{3.28} = 1.47 \times 10^{-3} \text{ lbf/ft}$$

$$g_c = 4.18 \times 10^8 \text{ lb} \cdot \text{ft/(lbf} \cdot \text{h}^2)$$

$$\rho_C = 821.36 \times \frac{1}{1.601\,846 \times 10} = 51.276 \text{ lb/ft}^3$$

$$\mu_C = 0.5294 \times 10^{-3} \times \frac{1}{4.133789 \times 10^{-4}} = 1.28 \text{ lb/(ft} \cdot \text{h)}$$

$$\Delta\rho = 188.04 \times \frac{1}{16.01846} = 11.739 \text{ lb/ft}^3$$

$$a_p = 340 \text{ m}^2/\text{m}^3 = 340 \times \frac{0.3048}{1} = 103.632 \text{ ft}^2/\text{ft}^3$$

$$\varepsilon = 0.92$$

$$f = \left(\frac{1.47 \times 10^{-3} \times 4.18 \times 10^8}{51.276}\right)^{0.2} \left(\frac{1.28}{11.739}\right)\left(\frac{103.632}{0.92}\right)^{1.5} = 852.81$$

From Fig. 7.27 and from Example 7.6,

$$(v_{CF}^{1/2} + v_{DF}^{1/2})^2 \rho_C / (a_p \mu_C) = 175$$

$$(v_{CF}^{1/2} + v_{DF}^{1/2})^2 = \frac{175\, a_p \mu_C}{\rho_C} = \frac{175 \times 103.632 \times 1.28}{51.276} = 452.72 \text{ ft/h}$$

$$q_C = 1.4825 \times 10^{-4}\ \text{m}^3/\text{s}$$
$$q_D = 5.503\,82 \times 10^{-5}\ \text{m}^3/\text{s}$$
$$\frac{v_{CF}}{v_{DF}} = \frac{q_C}{q_D} = \frac{1.4825 \times 10^{-4}}{5.503\,82 \times 10^{-5}} = 2.6936$$
$$\left((2.6936 v_{DF})^{1/2} + v_{DF}^{1/2}\right)^2 = 452.72$$
$$\left(2.6412 v_{DF}^{1/2}\right)^2 = 452.72 \times \frac{1}{3600} \times \frac{0.3048}{1} = 0.038\,33$$
$$v_{DF} = 0.0055\ \text{m/s and } v_{CF} = 0.0148\ \text{m/s}$$

Let actual velocities be 50%.

$$v_C = 0.5\ v_{CF} \text{ and } v_D = 0.5\ v_{DF}$$
$$v_D = 0.002\,75\ \text{m/s}, \quad v_C = 0.0074\ \text{m/s}$$
$$v_C = \frac{q_C}{(\pi/4)D_i^2}$$
$$\frac{\pi}{4} D_i^2 = \frac{1.4825 \times 10^{-4}}{0.0074} = 0.02\ \text{m}^2$$
$$D_i = 0.16\ \text{m}$$

Let $D_i = 0.2$ m

Total flow in the packed column $= q_C + q_D$
$= 2.0329 \times 10^{-4}\ \text{m}^3/\text{s}$
$= 0.731\,84\ \text{m}^3/\text{h}$

Cross-sectional area of column $= (0.2)^2\ \pi/4$
$= 0.031\,42\ \text{m}^2$

Superficial velocity (capacity) $= 0.731\,84/0.031\,42$
$= 23.3\ \text{m}^3/(\text{m}^2 \cdot \text{h})$

Based on curve FK in Fig. 7.21, this is acceptable.

Revised values of actual velocities based on final value of inside diameter.

$$v_C = 0.0047\ \text{m/s}, \quad v_D = 0.001\,75\ \text{m/s}$$

Height of packing:

Total height of packing $H = N_{toR} \cdot H_{toR}$

$N_{toR} = 7.213\,55$ (From Example 7.3)

H_{toR} for the given system

$$H_{toR} = 0.2347\ (v_C/v_D)^{0.648}$$

where, H_{toR} is in m.

$$H_{toR} = 0.2347 \left(\frac{0.0047}{0.001\,75}\right)^{0.648} = 0.4452\ \text{m}$$
$$H = 7.213\,55 \times 0.4452 = 3.211\ \text{m}$$

Results: Type of packing: 16 mm, SS Pall rings, inside diameter of tower: 0.2 m, total height of packing section = 3.25 m

How to find H_{toR} or H_{toE} from experiment (Ref. 12, 13)

To find the H_{toR} from the experiment at pilot plant or at lab scale, one can use the following equation for the packed tower-type extractor:

$$H_{toR} = \frac{\text{Superficial velocity of raffinate}}{K_{Ra}} = \frac{v_R}{K_{Ra}} \tag{7.37}$$

where, K_{Ra} = Overall mass transfer coefficient based on raffinate phase

$$= \frac{\text{kmol of solute transferred/second}}{\text{Volume of packing} \times \Delta C_{lm}}$$

ΔC_{lm} = Logarithmic mean driving force

Similarly,

$$H_{toE} = \frac{V_E}{K_{Ea}} \tag{7.39}$$

$$K_{Ea} = \frac{\text{kmol of solute transferred/second}}{\text{Volume of packing} \times \Delta\, C_{lm}}$$

Example 7.8 In the extraction of acetic acid from an aqueous solution by benzene in a pilot plant scale packed tower type extractor, following data were measured:

(i) Cross-sectional area of tower = 0.0045 m^2
(ii) Height of packing = 1.4 m
(iii) Concentration of acetic acid in feed = 0.69 kmol/m^3
(iv) Concentration of acetic acid in final raffinate = 0.684 kmol/m^3
(v) Flow rate of extract phase = 5.6×10^{-6} m^3/s
(vi) Concentration of acetic acid in solvent = 0.004 kmol/m^3
(vii) Concentration of acetic acid in final extract = 0.0115 kmol/m^3

Determine H_{toE} from above data.
Equilibrium relationship for this system[13] is $C_B^* = 0.0247\ C_w$,
where C_B^*, C_w = concentration of acetic acid, kmol/m^3

Solution

Total acetic acid transferred to extract (benzene) phase

$$= q_{VE}\,(C_{B2} - C_{B1})$$

$$= 5.6 \times 10^{-6}\,(0.0115 - 0.004) = 4.2 \times 10^{-8} \text{ kmol/s}$$

Equilibrium concentration of acetic acid in benzene phase at inlet and outlet,

$$C_{B1}^* = 0.0247 \times 0.684 = 0.0169 \text{ kmol/m}^3$$

$$C_{B2}^* = 0.0247 \times 0.69 = 0.0171 \text{ kmol/m}^3$$

Driving force at bottom, $\Delta C_1 = C_{B1}^* - C_{B1} = 0.0169 - 0.004 = 0.0129$ kmol/m^3
Driving force at top, $\Delta C_2 = C_{B2}^* - C_{B2} = 0.0171 - 0.0115 = 0.0056$ kmol/m^3
Logarithmic mean driving force

$$\Delta C_{lm} = \frac{\Delta C_1 - \Delta C_2}{\ln\left(\frac{\Delta C_1}{\Delta C_2}\right)} = \frac{0.0129 - 0.0056}{\ln\left(\frac{0.0129}{0.0\,056}\right)}$$

$$= 0.008\,748 \text{ kmol/m}^3$$

$$H_{toE} = \frac{\text{Superficial velocity of extract phase}}{\text{Overall mass transfer coefficient based on extract}} = \frac{V_E}{K_{Ea}}$$

$$V_E = \frac{\text{Volumetric flow rate of extract}}{\text{Cross-sectional area}} = \frac{5.6 \times 10^{-6}}{0.0045}$$

$$= 1.244 \times 10^{-3} \text{ m/s}$$

$$K_{Ea} = \frac{\text{kmol transferred}}{(\text{Volume of packing} \times \Delta C_{lm})}$$

$$= \frac{4.2 \times 10^{-8}}{(1.4 \times 0.0045 \times 0.008\,748)} = 7.62 \times 10^{-4} \text{ s}^{-1}$$

$$H_{toE} = \frac{1.244 \times 10^{-3}}{7.62 \times 10^{-4}} = 1.6325 \text{ m}$$

Example 7.9 For the extraction system described in Examples 7.4 and 7.5, design the packed tower-type extractor.

Solution

Feed flow rate $F = 1000$ kg/h. It contains 80% DMF and 20% water.
At steady state it is contacted with final extract E_1.

$$E_1 = B\,(1 + \mathcal{Y}_1')$$

$$E_1' = 3070.72\,(1 + 0.0625) = 3262.64 \text{ kg/h}$$

$$\mathcal{Y}_1 = \frac{\mathcal{Y}_1'}{1 + \mathcal{Y}_1'} = \frac{0.0625}{1 + 0.0625} = 0.0588$$

$$\rho_F = \frac{1}{\left(\frac{w_1}{\rho_1} + \frac{w_2}{\rho_2}\right)} \tag{7.30}$$

$$\rho_{\text{DMF}} = 944.5 \text{ kg/m}^3, \qquad \rho_{\text{water}} = 1000 \text{ kg/m}^3$$

$$\rho_F = \frac{1}{\left(\frac{0.8}{944.5} + \frac{0.2}{1000}\right)} = 955.1 \text{ kg/m}^3$$

$$\rho_{CH_2Cl_2} = 1336 \text{ kg/m}^3 \qquad \rho_{\text{DMF}} = 944 \text{ kg/m}^3$$

$$\rho_E = \frac{1}{\left(\frac{0.0588}{944.5} + \frac{(1 - 0.0588)}{1336}\right)} = 1304.2 \text{ kg/m}^3$$

$$q_{vF} = \frac{F}{\rho_F} = \frac{1000}{955.1} = 1.047 \text{ m}^3/\text{h}$$

$$q_{vE} = \frac{E_1}{\rho_E} = \frac{3262.64}{1304.2} = 2.502 \text{ m}^3/\text{h}$$

Tower Diameter

Let type of packing: SS Pall rings, size = 25 mm

a_p = surface area of packing = 205 m^2/m^3

ε = fraction of void volume = 0.94

To find the flooding velocities by Crawford – Wilke correlation, first the following function must be determined:

$$f = \left(\frac{\sigma_c g_c}{\rho_c}\right)^{0.2} \left(\frac{\mu_c}{\Delta\rho}\right)\left(\frac{a_p}{\varepsilon}\right)^{1.5} \tag{7.34}$$

$$\sigma_C^{1/4} = \Sigma x_i\, \sigma_i^{1/4} \tag{7.37}$$

$$\sigma_{\text{DMF}} = 35.2 \text{ dyn/cm}$$

Molar mass of DMF, $M = 73.09$

For pure DMF, $\sigma^{1/4} = [\mathcal{P}]\,(\rho_L - \rho_G)$ (Sudgen's equation)

ρ_L, ρ_G in mol/cm^3

$$[\mathcal{P}] = 40 + 55.2 \times 2 = 150.4 \quad \text{(Table 3–343, of Ref. 3)}$$

$$\rho_L = 1336 \text{ kg/m}^3$$

$$\rho_G = \frac{pM}{RT} = \frac{1 \times 85}{(273 + 25)} \times \frac{273}{1 \times 22.414} = 3.474 \text{ kg/m}^3$$

$$\rho_L = 1336 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{85} = 0.0157 \text{ mol/cm}^3$$

$$\rho_G = 3.474 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{85} = 4.087 \times 10^{-5} \text{ mol/cm}^3$$

$$\sigma_{CH_2Cl_2}^{1/4} = 150.4\,(0.0157 - 4.087 \times 10^{-5}) = 2.355$$

$$\sigma_{CH_2Cl_2} = 30.765 \text{ dyn/cm}$$

Mole fraction of DMF in extract phase,

$$x_1 = \frac{0.0588 / 73.09}{\left(\frac{0.0588}{73.09}\right) + \left(\frac{1 - 0.0588}{85}\right)} = 0.0677$$

$$\sigma^{1/4} = x_1\, \sigma_{\text{DMF}}^{1/4} + (1 - x_1)\sigma_{CH_2Cl_2}^{1/4} \tag{7.37}$$

$$\sigma_C^{1/4} = 0.0677 \times 35.2^{1/4} + (1 - 0.0677) \times 30.765^{1/4}$$

$$\sigma_C = 31.05 \text{ dyn/cm} \equiv 0.031 \text{ N/m}$$

For organic mixtures[12],

$$\frac{1}{\mu_C} = \frac{w_1}{\mu_1} + \frac{w_2}{\mu_2}$$

$$\mu_{\text{DMF}} = 0.802 \text{ mPa} \cdot \text{s}, \quad \mu_{CH_2Cl_2} = 0.6 \text{ mPa} \cdot \text{s}$$

$$\frac{1}{\mu_C} = \frac{0.0588}{0.802} + \frac{(1-0.0588)}{0.6}$$

$$\mu_C = 0.61 \text{ mPa} \cdot \text{s} \equiv 0.61 \times 10^{-3} \text{ kg / (m} \cdot \text{s)}$$

$$\Delta\rho = \rho_c - \rho_d = 1304.2 - 955.1 = 349.1 \text{ kg/m}^3$$

To use Fig. (7.27), all variables must be in US customary units.

$$\sigma_C = 0.031\,05 \times \frac{1}{4.448\,222} \times \frac{1}{3.28} = 2.128 \times 10^{-3} \text{ lbf/ft}$$

$$g_c = 4.18 \times 10^8 \text{ lb} \cdot \text{ft / (lbf} \cdot \text{h}^2)$$

$$\rho_C = 1304.2 \times \frac{1}{1.601\,846 \times 10^1} = 81.42 \text{ lb/ft}^3$$

$$\mu_C = 0.61 \times 10^{-3} \times \frac{1}{4.133\,789 \times 10^{-4}} = 1.4756 \text{ lb/(ft} \cdot \text{h)}$$

$$\Delta\rho = 349.1 \times \frac{1}{16.018\,46} = 21.7936 \text{ lb/ft}^3$$

$$a_p = 205 \text{ m}^2/\text{m}^3$$

$$= 205 \times \frac{0.3048}{1} = 62.484 \text{ ft}^2/\text{ft}^3$$

$$\varepsilon = 0.94$$

$$f = \left(\frac{\sigma_c g_c}{\rho_c}\right)^{0.2} \left(\frac{\mu_c}{\Delta\rho}\right) \left(\frac{a_p}{\varepsilon}\right)^{1.5} \tag{7.35}$$

$$= \left(\frac{2.128 \times 10^{-3} \times 4.18 \times 10^8}{81.42}\right)^{0.2} \left(\frac{1.4756}{21.7936}\right) \times \left(\frac{62.484}{0.94}\right)^{1.5}$$

$$= 235.66$$

From Fig. (7.27),

$$\left(v_{CF}^{1/2} + v_{DF}^{1/2}\right)^2 \rho_c / (a_\rho \mu_c) = 600$$

$$\left(v_{CF}^{1/2} + v_{DF}^{1/2}\right)^2 = 600 \times \frac{62.484 \times 1.4756}{81.42} = 679.45$$

$$\frac{v_{CF}}{v_{DF}} = \frac{q_{VC}}{q_{VD}} = \frac{q_{VE}}{q_{VF}}$$

$$= \frac{2.502}{1.047} = 2.39$$

$$\left((2.39\, v_{DF})^{1/2} + v_{DF}^{1/2}\right)^2 = 679.45$$

$$6.482\, v_{DF} = 679.45$$

$$v_{DF} = 104.82 \text{ ft/h and } v_{CF} = 250.52 \text{ ft/h}$$

or

$$v_{DF} = 104.82 \times \frac{1}{3600} \times \frac{0.3048}{1} = 0.009 \text{ m/s}$$

and $v_{CF} = 0.0215$ m/s

Let $v_D = 0.5\ v_{DF} = 0.0045$ m/s,
$v_C = 0.5\ v_{CF} = 0.010\ 75$ m/s

$$\frac{\pi}{4} D_i^2 = \frac{q_{VC}}{v_C}$$

$$= \frac{2.502 / 3600}{0.010\ 75} = 0.065 \text{ m}^2$$

$$D_i = 0.288 \text{ m}$$

Total volumetric flow (capacity) of tower = 2.502 + 1.047 = 3.549 m^3/h
Superficial flow rate = 3.549/0.065 = 54.6 $m^3/(m^2 \cdot h)$

In Fig. 7.21, area FK represents random packed columns. Maximum superficial capacity can be read as 30 $m^3/(m^2 \cdot h)$ which is less than calculated value 54.6 $m^3/(m^2 \cdot h)$. Hence, packed column diameter will have to be increased to achieve 30 $m^3/(m^2 \cdot h)$.

Cross-sectional area required of the column = $\frac{3.549}{30} = 0.1183 \text{ m}^2$

Column diameter, $D_i = 0.3881$ m
Let $D_i = 0.4$ m = 400 mm

Since column diameter is increased, there is no need to check superficial velocities, etc.

Packing height:

$$H = N_{toR} \times H_{toR}$$

$$N_{toR} = 5.345$$

Suitable correlation for H_{toR} is not available in open literature for the given system. It must be determined by pilot plant study. Here H_{toR} is determined by the same equation that is derived for acetic acid – water – MIBK system.

$$H_{toR} = 0.2347 \left(\frac{v_C}{v_D} \right)^{0.648}$$

$$= 0.2347 \left(\frac{0.010\ 75}{0.0045} \right)^{0.648}$$

$$H_{toR} = 0.41 \text{ m}$$

$$H = 5.345 \times 0.41 = 2.19 \text{ m}$$

Let $H = 2.5$ m

Resulting data:
Type of packing: 25 mm SS Pall rings
Inside diameter of tower = 300 mm
Height of packing section = 2.5 m

Feed density (955.1 kg/m^3) is lower than solvent density (1336 kg/m^3). Also feed flow rate (1000 kg/h) is lower than solvent flow rate (1535.36 kg/h). Hence, lighter phase is dispersed in heavier phase. System shown in Fig. 7.16(a) can be adopted.

7.7 SUPERCRITICAL EXTRACTION (SCE)

In liquid-liquid extraction solvents like water, ethylene glycol, chloroform, hexane, ethyl acetate, etc., are contacted with feed mixture to separate the desired product. Desired product is transferred to liquid solvent. Then desired product is to be separated from solvent. In short by liquid-liquid extraction from a given feed solution one can make a new solution (solvent + solute). If separation of new solution is easier than the same of the given solution, then extraction is called useful extraction.

Separation of solute from solvent is normally done by distillation. It consumes large amount of energy. Also it is difficult to get 100% pure solute; absolutely free from solvent by distillation.

In supercritical extraction (Ref. 14 to 21), supercritical fluid is used as a solvent. In Sections 7.1 to 7.6, it was discussed that the fluid can exist as liquid or gas. But in addition to that a fluid can also exist as supercritical fluid (known as SCF).

Supercritical fluid is any fluid at a temperature higher than critical temperature and at a pressure higher than critical pressure.

In Fig. 7.28 and Fig. 7.29, phase diagrams of carbon dioxide and water are given. Supercritical regions for both the compounds are shown in the figures.

7.7.1 Properties of Supercritical Fluid Solvents (SCFS) (Ref. 13, 14, 22)

Physical properties of supercritical fluids are between those gaseous and liquid states. Typical values of important physical properties are listed in Table 7.7.

Table 7.7 Comparison of Physical Properties of Gas, Liquid and Supercriitical Fluids[22]

Physical property	Gas	SCF	Liquid
Density, ρ, kg/m^3	0.6 – 2	200 – 500	600 – 1000
Dynamic viscosity, μ, mPa · s	0.01 – 0.3	0.01 – 0.03	0.2 – 3
Kinematic viscosity, $\gamma \times 10^6$, m^2/s	5 – 500	0.2 – 0.1	0.1 – 5
Thermal conductivity, k, W/(m · K)	0.01 – 0.025	High*	0.1 – 0.2
Diffusion coefficient, $D \times 10^5$, m^2/s	10 – 40	0.07 – 0.08	0.0002 – 0.002

* Thermal conductivity values are high in the near critical region.

1. In supercritical state, density of fluid is just like liquid density and hence solvent power for extraction is also equivalent to liquid solvents. In supercritical region density of the fluid is sensitively changing with pressure. Slight change in pressure creates a dramatic change in density.
2. Viscocities of SCFs are equivalent to viscocities of gases. Hence, they consume less power for transportation. Also they provide lesser resistance to heat transfer and mass transfer.
3. Molecular diffusivities of SCFs are intermediate to liquids and gases.
   D_L is in the order of $C \times 10^{-9}$ m^2/s (D_L = diffusivity of liquid).
   D_G is in the order of $C \times 10^{-5}$ m^2/s (D_G = diffusivity of gas).
   Where C is a numerical value.

Fig. 7.28 *Phase Diagram (p-t Diagram) of Carbon dioxide*
Based on Data Contained in Thermodynamic Properties of Carbon dioxide, by: R. Span and W. Wagner, J. Phys. Chem. Ref. Data, ***25****(6), p. 1509, 1996.*

Advantages and disadvantages of SCF solvents over liquid solvents

Advantages

1. SCF solvents are easily and completely separated from extracted products. This means SCF solvents give the product, nearly 100% free from solvent.
2. Reduction in pressure or increase in temperature separates the SCF from extracted products. Hence, they require lesser energy for separation. Thus with SCE, near 100% recovery of solvent from extracted product is possible with low cost, compared to liquid-liquid extraction with liquid solvents.
3. SCF solvent has very low viscosity and higher diffusivity compared to liquid solvent. Hence, it provides lesser resistance to mass transfer and also it provides lesser friction during transportation.
4. SCFs provide very high value of selectivity, compared to conventional liquid solvents.

Fig. 7.29 *Phase Diagram (p-t Diagram) of Water*
Based on Data Contained in
1. *Vacuum Technology, Its Foundations, Formulae & Tables, by Leybold AG, Germany, 1987.*
2. *1980 SI JSME STEAM TABLES, Japan.*
3. *Publication No. NRISTIR 5078 of National Institute of Standards & Technology, USA, 1998.*

Selectivity:

$$\alpha = \frac{\text{Mass of solute/mass of non-solute component of extract phase}}{\text{Mass of solute/mass of non-solute component of raffinate}}$$

$$\alpha = \frac{(C/A)_{\text{extract}}}{(C/A)_{\text{raffinate}}}$$

Extraction is possible only if $\alpha > 1$. If $\alpha >> 1$, it implies that lesser number of theoretical stages are required for desired separation. Hence, with SCF, lesser

number of theoretical stages are required for desired separation. Consequently they require lesser height of extractor as compared to conventional liquid solvents.

5. With common SCFs like CO_2, solvent recovery is possible at room temperature or at temperature close to the room temperature.
6. Common SCFs like CO_2 is easily available with relatively low price as compared to conventional liquid solvent.
7. SCFs like CO_2 (dry) is non-corrosive and non-toxic in nature.

Disadvantages

1. It is found by experiments that supercritical fluids, provide poor value of distribution coefficient for extraction as compared to conventional liquid solvents.
   Distribution coefficient,
   $$m = \frac{\text{Mass fraction of solute in extract phase at equilibrium}}{\text{Mass fraction of solute in raffinate phase at equilibrium}} \quad (7.1)$$
   Hence, solvent/feed ratio, required with SCFs, is very much higher than conventional liquid solvents.
2. Except the few solvents like CO_2, for other solvent like hexane, ethylene glycol, etc., it is very difficult to achieve the supercritical state of fluid due to the practical limitations. Choice of solvent is narrow with SCFs.
3. Certain SCFs are flammable (like propane, butanes, etc.) and hence, adequate safety measures are required in design.

Properties of CO_2, H_2O and *n*-hexane in supercritical region are given in Ref. 23. Properties of ethane, propane and butanes in supercritical region can be found in Ref. 24.

Table 7.8 List of Supercritical Solvents and their Properties[25]

Fluid	t_c (°C)	p_c (bar)	Remarks
Carbon dioxide, (CO_2)	31.00	73.77	Most Favourable
Dinitrogen monoxide, (N_2O)	36.45	72.40	Not stable
Xenon, (Xe)	16.60	58.68	High price, poor availability
Ethane, (C_2H_6)	32.27	48.80	Inflammable
Ethylene, (C_2H_4)	9.19	50.39	Inflammable
Water, (H_2O)	374.15	221.20	High temperature and pressure operation, corrosive, high latent heat of vaporisation
Propane (C_3H_8)	96.67	42.47	Inflammable
n–Butane (C_4H_{10})	152.03	37.97	Inflammable
i–Butane (C_4H_{10})	135.00	36.48	Inflammable

Among all these solvents, CO_2 and propane are used in commercial plants. Water is also a useful SCF but requires a very high pressure operation.

CO_2 is most preferred as SCE solvent. Reasons are:

(a) It has low critical temperature (t_c = 31.0°C).
(b) It has relatively low critical pressure (p_c = 73.77 bar).
(c) It is non-toxic and non-corrosive in nature (when dry).
(d) It is easily available at low cost. It is available as a byproduct from many industries; such as fermentation of molasses, ammonia plant, etc. It is present in substantial amount in atmosphere as green house gas.

Advantages and disadvantages of SCE over conventional unit operations

Advantages

1. It consumes lesser energy compared to liquid-liquid extraction and distillation, combined together. Hence, it requires lesser operating cost as compared to extraction and distillation. It requires only electricity as a utility to drive a compressor and a positive displacement liquid pump. With CO_2 or N_2O, chilled water or brine is required for condensation.
2. Compared to the liquid-liquid extraction it gives purer products, nearly 100% free from solvent or foreign impurities.
3. SCF provides very high value of selectivity as compared to conventional liquid solvents. Hence, supercritical extractor requires lesser number of theoretical stages and lesser height, compared to liquid-liquid extractor.
4. Compared to (atmospheric) distillation, supercritical extraction can be operated at lesser temperature, closer to room temperature. Liquid-liquid extraction requires higher temperature in solvent recovery step. This advantage is beneficial particularly for heat sensitive products.
5. Products obtained by biotechnology route is usually a dilute solution (< 10%) in water. For the separation of desired product from water by distillation requires very high amount of energy and operating cost. While for the same separation, SCFE consumes very less energy and has low operating cost.

   Several examples of herbal and heat sensitive products are given in Ref. 21 with the use of CO_2 as SCF.

   One research paper mentions that operating cost required to separate ethanol from its dilute solution by distillation is 5 times higher than the same by SCFE while fixed cost of distillation unit could be half that of the SCFE unit.

Disadvantages

1. It requires higher capital investment as this operation is carried out at high pressure. Fixed cost of compressor is relatively higher than cost of other equipments. Also the cost of the extractor is high. There are only a few suppliers of the technology and equipments of the plant of SCFE at present. Hence, the specialisation of technology further increases fixed cost of plant at present.
2. It provides very low value of distribution coefficient hence large amount of solvent is required for separation.
3. Limited experience with its use in pilot and commercial applications. However, now pilot studies with CO_2 as SCF are available at certain research institutes with sufficient data for scale-up.

4. Limited knowledge of design data. Also thermodynamics of supercritical system is not fully understood till date.

7.7.2 Flow Sheet of Batch Supercritical Extraction (Ref. 14, 15)

Figure 7.30 is the flow sheet of SC Extraction plant which is operated in batchwise manner. Operational steps are as follows.

1. In bath extraction, feed is initially charged before starting carbon dioxide circulation.
2. In CO_2 vessel, CO_2 is in compressed gas form but $p < p_c$ and temperature is equal to room temperature.
3. From CO_2 vessel, compressed gas passes through chilled water condenser or brine condenser where CO_2 is liquefied.
4. Liquefied CO_2 is pumped by metering pump (a positive displacement pump) to high pressure. At the discharge of pump, CO_2 is liquid with $p > p_c$ and $t < t_c$.
5. Compressed liquefied CO_2 which is in liquid state at $p > p_c$ and at temperature less than t_c is converted to SCF by increasing the temperature above critical temperature.
6. Supercritical CO_2 is contacted with feed which was initially charged in the batch extractor. Sufficient contact time is allowed to reach equilibrium
7. From the top of the extractor, extract phase (SC CO_2 + solute) is obtained. It is heated to the temperature at which soluble solute is separated into insoluble solid or liquid.

Fig. 7.30 *Flow Sheet of Batch SCE Plant*

8. Then pressure is decreased and SC CO_2 is converted to gaseous CO_2 with $p < p_c$ and at temperature which is close to t_c.
9. CO_2 is circulated till the desired degree of extraction is obtained. A level indicator can be provided on the separator.
10. Once desired degree of extraction is obtained, system is depressurised. Raffinate phase is separated from the extractor at atmospheric pressure. New feed then can be charged to the extractor.

7.7.3 Application of SCE (Ref. 13 to 20)

SCE finds its application in food, pharmaceutical, petroleum refineries, solid fuel industries and polymer industries. Extensive research work is going on other areas of applications.

1. Established Commercial Applications

(a) Decaffenation of coffee
(b) Deasphalting of oil
(c) Extraction of essence from fragrant materials
(d) Separation of aromas and oils from spices
(e) Spray painting and coating
(f) Chromatographic analysis of polymer
(g) Supercritical fluid polymerisation, e.g., manufacturing of polytetrafluoroethylene
(h) Extraction of herbs
(i) Purification of active pharmaceutical intermediates (API)
(j) Isolation of eico sapentanoic acid (EPA) from fish oils
(k) Purification of a vitamin intermediate
(l) Purification of a reactive multifunctional acrylic monomer
(m) Extraction of cholesterol from butter
(n) Extraction of oligomers from high vacuum sealants.
(o) Extraction of sterols from deodorised distillate of edible/non-edible oils.

2. Applications under Research and Development

(a) Extraction of various chemicals from coal and wood
(b) Extraction of organic chemicals from its dilute solution in water. (This application is useful in biotechnology route and also in the recovery of chemicals from wastewater stream)
(c) In separation of liquid hydrocarbon mixtures which are difficult to separate by conventional techniques
(d) Separation of aromatics from waste solid material such as tobacco dust
(e) Regeneration of adsorbent materials
(f) Extraction of oil from oil containing conventional and unconventional seeds, e.g., soybeans, ground nuts, jojoba beans, coriander, etc.
(g) Deodorisation of vegetable and animal oils.
(h) Extraction of oil from potato chips
(i) Recovery of citrous oil from peelings
(j) Extraction of anticancer drugs and other substances from plants (herbs)
(k) Refining of pyrethrum to obtain pyrethrin insecticides

(l) Destruction in hazardous waste and production of biocrude from organic waste (water is used as SCF)
(m) Extraction of chemicals from used automotive tires
(n) Separation of monoglycerides from the mixture of glycerides
(o) Paper deacidification and preservation
(p) Plastic recycling
(q) Textile dyeing
(r) Generation of nano particles
(s) Supercritical phase oxidation

Many other applications are under R&D and can be found from literature.

7.7.4 Use of Cosolvents and Surfactants (Ref. 16)

One of the major limitations of SCF is availability of very few suitable solvent. Most suitable solvent is supercritical CO_2 which is non-polar in nature.

Many non-volatile polar substances cannot be dissolved easily in non-polar SC CO_2. Most of the organic compounds are polar in nature, e.g., ethanol, ethylene glycol, etc.

Polar solvents have high critical temperature. Hence, they involve very high cost in extraction. Also other problems like thermal cracking may take place of such solvent as SC solvent results in its rejection. It is found by experiment that an addition of small amount of polar liquid solvent (1 to 10 mole %) in SC CO_2, dramatic improvement in the solubility can be achieved.

Take for example case of hydroquinone which dissolves very little in pure SC CO_2. Addition of 2.8 mole % methanol increases hydroquinone's solubility by a factor of 6. Addition of 2 mole % tributyl phosphate (TBP) increases hydroquinone's solubility by a factor of 250.

Hence, TBP is much more effective than methanol as cosolvent. Selection of the correct cosolvent requires detailed knowledge of chemistry and molecular structure of polar and non-polar compounds. Hence, selection of right cosolvent is very important. Cosolvent is also known as *modifier*.

7.7.5 Few Commercial Applications

It will be interesting to study two case studies of supercritical extraction/processing.

Decaffeination of coffee (Ref. 18) Coffee industry is an important food grade industry. With industrialisation and increasing demand of refreshers, there has been an increase in consumption of coffee in most of the parts of world. Coffee seeds contain 1–3% caffeine. Consumption of large dosage of caffeine produces adverse effect on human body, but at moderate level no major harmful effects are observed.

Pure caffeine is a white powder, soluble in water and various organic solvents. Hot water is an excellent solvent for caffeine but the same cannot be selected as a solvent for extraction as other ingradients of the coffee beans are also soluble in hot water while caffeine is selectively soluble in SCF CO_2. There are many plants in the world which extract caffeine from raw coffee. One of them is coffee decaffeination plant in Houston, USA, built by Kraft General Foods Ltd. The process, used by Kraft, is shown in Fig. (7.31).

Fig. 7.31 *Decaffeination Process*

Process description The moist green coffee beans are charged to an extraction vessel having 7 ft (2.13 m) diameter and 70 ft (21.33 m) height. Here moisture of water act as modifier. Carbon dioxide is dried and charged from the bottom of extractor in supercrifical state. From supercritical extract, caffeine is then recovered by countercurrent water wash, carried out nearly at the same pressure.

The depleted carbon dioxide is dried and recycled to the extractor. Caffeine rich aqueous stream is concentrated by reverse osmosis.

The Lipton tea decaffeination process is similar to Kraft process (in Germany) except that the caffeine is removed from the carbon dioxide by passing the solution through a bed of activated carbon.

The caffeine recovery from the extract is the key part of the process. As the decaffeinated green coffee is finally contacted with solvent, equilibrium distribution of caffeine between beans and solvent decide the percentage of unrecovered caffeine in the beans.

If caffeine concentration in the solvent (SC CO_2) is reduced to 0.002 mass % in recovery step, then it gives equilibrium concentration of caffeine remaining in the processed beans to about 0.16 mass %.

Hence, it is very much necessary to remove caffeine from extract phase as much as possible. One way is to reduce the pressure of SC extract to atmospheric pressure, but it is not an economical option.

It is suggested not to try to reduce the concentration of caffeine from coffee below 0.16% as it is not economical. Hence, the selection of recovery process and maximum extraction of caffeine is very important.

ROSE process (Deasphalting of oil) (Ref. 25) Solvent extraction has been used to separate valuable components from heavy oil and residuals from less desirable material. One of them is propane deasphalting. In the refinery, bottom product of vacuum distillation column is known as *residuum*. Propane deasphalting is used to separate the residuum into three fractions; (i) lube oil (ii) resin, and (iii) asphalt.

Fig. 7.32 *ROSE Process*

It uses liquid propane as solvent to extract lube oil and resin from residuum and to reject high molar mass asphalt, as reffinate. Solvent recovery in conventional propane deasphalting consists of series of flash vessels and steam stripping. Entire propane is recovered as vapour at lower pressure. It is converted into high pressure liquid propane by compression and liquefaction and recycled back to extractor. Thus, energy consumption in conventional propane deasphalting is very high and it demands the use of new technique. The ROSE (Residuum Oil Supercritical Extraction) process is a new process developed by Kerr-McGee's R&D group. ROSE process produces identical products from residuum (lube oil, resin, and asphalt) but it consumes less energy.

Details of three products obtained from residuum are as follows:

1. **Asphalt:** It is the heaviest, most viscous and the most hydrogen deficient fraction of crude petroleum oil. At room temperature it is a dark brown to black friable solid. Its ring and ball softening point ranges from 70 to 200°C. Carbon to hydrogen ratio is 9 to 11.6 by mass. It is extremely difficult to remove sulphur, nitrogen or metals from asphalt. Hydrotreating and cracking catalysts are rapidly coked, deactivated and poisoned by asphalt.
2. **Resin:** It is an intermediate fraction, lighter than asphalt and heavier than deasphalted oil or lube oil. Resin can be hydrotreated, hydrocracked, thermally-cracked or even coked to produce more volatile products. Resins are less viscous, low in metals, sulphur and carbon. Resins have less C/H ratio than asphaltene.
3. **DAO or Lube Oil:** Deasphalted oil (DAO) or lube oil is a clean product containing substantially lower metals and carbon residue than either asphalt or resin. DAO can be sent for hydrotreating, hydrocracking or catalytic cracking, without any further purification.

Process description The residuum is charged to extractor where it is contacted with liquid propane at elevated pressure. In ROSE process also, extraction is liquid-liquid extraction, similar to conventional propane deasphalting. It is not the supercritical extraction. As an extractor, either Mixer-settler or Rotating Disc Contractor (RDC) is used. From the extractor, asphalt is obtained as raffinate phase, but it is contaminated by very less amount of propane. Propane vapour is separated from raffinate phase by flashing and stripping in tower T_1 which is a flash vessel cum stripper.

Extract phase, containing large amount of liquid propane, resin and lube oil, is passed through heater E_1 where it is heated to the temperature at which resin becomes insoluble and is separated from the extract phase. Propane rich layer and resin layer are separated in decanter D_1. Traces of propane from resin layer is separated by flashing and stripping in the tower T_2.

The remaining propane-oil solution is heated to higher temperature by two heaters E_2 and H_2 in series. At this temperature liquid propane is converted into supercritical propane. DAO or lube oil is not soluble in supercritical propane, hence it is separated from propane in decanter D_2. Traces of propane from DAO or lube oil is separated by flashing and stripping in tower T_3. Supercritical propane from D_2 is passed through heat exchangers E_2 and E_1, where its sensible heat is utilised for heating. Then in heat exchanger C_2 it is liquefied. Liquefied propane is recycled back to extractor by pump P_1, which develops only differential pressure necessary to overcome system pressure drop.

Small amount of propane vapour, stripped off from the towers T_1, T_2 and T_3, is condensed in condenser C_1. Condensate is sent to tank S_1. This intermediate hold-up tank is maintained at lower temperature and at lower pressure, where make-up liquefied propane is also added. Small amount of propane from this tank (7 to 15% of the total required in extractors) is pumped by pump P_2 and sent to suction line of pump P_1. Pressure difference around pump P_2 is quite higher than the same around pump P_1.

Thus in ROSE process, extraction is carried out in subcritical region or as liquid-liquid extraction. Only properties of supercritical fluid are utilised in solvent recovery step.

Advantages of Rose process over conventional propane deasphalting are as follows.

1. 85 to 93% of the extraction solvent is recovered as supercritical fluid in the oil separator without evaporation and pressure reduction. Also, the concept of energy integration is applied in two heat exchangers (E_1 and E_2) to save the utilities. Hence, energy required by ROSE process is substantially lower than the same required for conventional propane deasphalting.
2. ROSE process requires less maintenance cost.
3. Increase in propane to feed ratio improves the quality of products. With ROSE process, higher solvent to feed ratio is affordable, hence it results in better quality of products.

ROSE process is competitive, well established and commercially proven technology. It is licensed by more than 15 companies worldwide.

Decaffeination of coffee and ROSE process were discussed in detail to bring forth the process engineering problems and heat integration aspects. For development of other solvent extraction systems, process engineer should consider similar aspects so that an efficient system can be designed. Useful guidelines for process design of SCF extractor obtained from Ref. 21.

Conclusion

In the early stages of development of the Supercritical Fluid Extraction, the requirement of expensive equipments, lack of proper and appropriate techniques were the main hurdles of adoption and commercialisation of the technology.

But now in the changed scenario of improvement in the SCE techniques, increasing cost of energy and increasing cost of conventional solvents makes this unit operation more feasible and attractive. Still there is a large scope for the development of supercritical extraction technology. More research work is required to improve the distribution coefficient of SCE system. In future, it is certain that this unit operation will be applied in many processes.

EXERCISES

7.1 A feed stream having flow rate of 1000 kg/h and containing 40 mass % styrene and 60 mass % ethyl benzene is to be extracted with ethylene glycol as solvent. Extraction is to be carried out in multistage countercurrent extractor. Final raffinate is to be extracted down to 1% styrene. Determine (a) minimum amount of solvent required for the desired separation and (b) number of theoretical stages required for desired separation if the actual amount of solvent is 5000 kg/h.

Table 7.9 Liquid-liquid Equilibrium Data[4] of Ethyl Benzene-Styrene-Ethylene Glycol at 25°C

mass % in Raffinate			mass % in Extract		
Ethyl benzene	Styrene	Ethylene glycol	Ethyl benzene	Styrene	Ethylene glycol
90.56	8.63	0.81	9.85	1.64	88.51
80.4	18.67	0.93	9.31	3.49	87.2
70.49	28.51	1.00	8.72	5.48	85.8
60.93	37.98	1.09	8.07	7.45	84.48
53.55	45.25	1.2	7.35	9.25	83.4
52.96	45.84	1.2	7.31	9.49	83.2
43.29	55.32	1.39	6.3	12.00	81.7
41.51	57.09	1.4	6.06	12.54	81.4

(From Table 15.2 of Ref. 4)

7.2 For the extraction described in Exercise 7.1, if a packed tower is used as extractor then (i) find the value of N_{toR} for $S = 5000$ kg/h, and (ii) determine the tower diameter.

7.3 A feed mixture containing 60 mass % methyl cyclohexane and 40 mass % *n*-heptane is contacted with pure aniline as solvent. Extraction is carried out in countercurrent manner in four mixer-settler vessels. Stage efficiency of mixer-settler may be assumed equal to 1. Solvent to feed ratio (mass basis) is kept 4. Feed flow rate is 200 kg/h. Find the resultant product flow rates and product compositions.

Table 7.10 Equilibrium Data at 25°C

Hydrocarbon phase, mass %		Aniline rich phase, mass %	
Methyl cyclohexane	*n*-Heptane	Methyl cyclohexane	*n*-Heptane
0.0	92.6	0.0	6.2
9.2	83.1	0.8	6.0
18.6	73.4	2.7	5.3
22.0	69.8	3.0	5.1
33.8	57.6	4.6	4.5
40.9	50.4	6.0	4.0
46.0	45.0	7.4	3.6
59.0	30.7	9.2	2.8
67.2	22.8	11.3	2.1
71.6	18.2	12.7	1.6
73.6	16.0	13.1	1.4
83.3	5.4	15.6	0.6
88.1	0.0	16.9	0.0

(Ref. 8, p. 325)

(**Hint:** Assume *n*-heptane and aniline as completely immiscible liquids to simplify the calculations.)

7.4 Design a mixer settler for Exercise 7.3.
Residence time required in mixer to achieve equilibrium = 8 min
Type of agitator of mixer = 45° pitched blade turbine
Tip velocity of agitator = 200 m/min

7.5 A feed mixture containing 20 mass % acetic acid and 80 mass % water is contacted with nearly pure butyl acetate as solvent to recover acetic acid. Extraction is carried out in a packed tower. Feed flow rate is 1000 kg/h. Final raffinate leaving the packed tower should contain less than or equal to 1% (by mass) acetic acid. Determine the following:

(i) Minimum amount of solvent required for desired separation.
(ii) Value of N_{toR} for actual amount of solvent $S = 2\ S_m$.
(iii) Tower diameter required for packed tower.
(iv) Determine the height of packing. Assuming that Eq. (7.32) is valid for acetic acid-water-butyl acetate system for finding H_{toR}.

Data:

Table 7.11 Liquid-Liquid Equilibrium Data (mass %) at 25°C

Solvent rich layer			Water rich layer		
Water	**Butyl acetate**	**Acetic acid**	**Water**	**Butyl acetate**	**Acetic acid**
1.8	94.8	3.4	90.5	0.8	8.7
3.7	84.6	11.5	78.9	1.1	20.0
4.7	82.0	13.3	75.6	1.2	23.2
7.8	73.0	19.2	68.0	1.6	30.4
8.9	68.0	23.1	65.0	2.3	32.7
14.7	56.0	29.3	56.3	5.6	38.1

All percentages in Table 7.11 are mass %.

7.6 A feed stream having flow rate of 400 kg/h and containing 30% ethanol in water is to be extracted at 25°C with 600 kg/h of recycled benzene that contains 0.5% ethanol. The aqueous raffinate is to be extracted down to 1% ethanol. How many theoretical stages will be required? Extraction is to be carried out in counter manner in a packed tower. Design the extractor.

Table 7.12 Equilibrium Data of Benzene-Ethanol-Water System[1] at 25°C.

mass % in Extract			mass % in Raffinate		
Benzene	**Ethanol**	**Water**	**Benzene**	**Ethanol**	**Water**
99.35	0.60	0.05	0.13	11.20	88.67
98.00	1.85	0.15	0.28	22.00	77.72
96.30	3.50	0.20	0.53	30.90	68.57
95.25	4.45	0.30	1.02	36.00	62.98
92.00	7.40	0.60	3.35	43.50	53.15
90.75	8.45	0.80	4.80	45.90	49.30
84.40	13.85	1.75	13.70	52.20	34.10
80.00	17.45	2.55	20.45	52.15	27.40
70.60	24.75	4.65	33.20	48.00	18.80
64.10	29.30	6.60	40.95	44.00	15.05

Figure 7.1 represents phase diagram for the system.

7.7 Sulphonation of various aromatic compounds is carried out with either sulphuric acid (98%) or oleum, having different strengths of SO_3 in normal course. Generation of spent dilute sulphuric acid with high chemical oxygen demand (COD) is a serious effluent problem.

Consider following sulphonation reactions.

(a) Sulphonation of *p*-nitrotoluene

(b) Sulphonation of aniline

(c) Sulphonation of Tobias Acid

(d) Sulphonation of 2-Naphthol

As a part of Green Engineering, experiments are planned to carry out reactions with liquid sulphur trioxide (SO_3) at supercritical conditions. Assume that reactants and products are soluble in any of the supercritical fluids, listed in Table 7.8. Also assume that all reactions will be carried out with stoichiometric proportions of the reactants and with 100% conversions. For this process research suggest a suitable

supercritical fluid for each of the above reactions with reasonings. Also consider whether a cosolvent is recommended for any of the above reactions. Why?

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8 Process Design of Distillation Columns

8.1 INTRODUCTION

In simple terms, vaporisation followed by condensation is known as distillation. It is a method of separating components of a solution which are relatively volatile. Distillation is the most widely used separation method in chemical industries. Simple distillation or batch distillation was known in the first century. Multistage countercurrent distillation was invented in 1830 by Aeneas Coffey of Dublin[1]. He used tray tower for separating 95.6% ethanol (by mass) from its dilute solution in water. But as of today 100% reliable method for designing of distillation column, which can be applied to all systems without any limitation, is not available. All methods have limitations. Hence, process design engineers use more than one method for the design of distillation column. For an entirely a new mixture, it is better to verify the design of its distillation column first on a pilot plant scale column before applying it at commercial or large scale.

8.2 CRITERIA OF SELECTION

For a given mixture, if all the components are relatively volatile and are required to be separated in pure form, then distillation is the first choice.

If the relative volatility of the two adjacent components is very high, then evaporation is preferred. For example, concentration of orange juice.

If relative volatility of two adjacent components is very low: close to 1 (e.g., 10 to 30%, by mass acetic acid in water) or equal to 1 (e.g., azeotropic mixture, 95.6% by ethanol in water), special type of distillation like Azeotropic Distillation or Extractive Distillation is used. For the same case, liquid-liquid extraction, membrane separation or crystallization may be more economical. Examples, (a) For the separation of pure acetic acid from 10% (by mass) aqueous acetic acid solution, liquid-liquid extraction using ethyl acetate as solvent is more economical than azeotropic distillation of the same solution using butyl acetate or ethyl acetate as an entrainer, (b) For the separation of pure ethanol from its azeotropic mixture with water [95.6% ethanol (by mass) in ethanol-water solution], pervaporation unit consumes less than 20% of the energy than that used in azeotropic distillation. Also for the same separation, pervaporation unit requires low pressure steam (to maintain 80°C temperature which can be obtained by flashing the condensate of high pressure steam, and (c) For the separation of *p*-xylene from the mixed xylenes, crystallization is used.

If the separation of only one component is required rather than a separation of all the components of the mixture, then absorption, stripping or liquid-liquid extraction may be more economical than distillation.

Examples:

1. For the separation of penicillin from the complex mixture, liquid-liquid extraction is used.
2. For the removal of hydrogen and methane from aromatics, stripping is used.
3. For the removal of propane from the mixture called asphalt, stripping is used. Volatility of propane is very high compared to volatilities of other components of the asphalt.
4. For the removal of hydrogen sulphide from the natural gas mixture, absorption using ethanolamines as solvent is used.
5. For removal of ammonia from lean aqueous solution, steam stripping is preferred.
6. If all components of air are to be separated in pure form (nitrogen, oxygen, argon, etc.) cryogenic distillation is preferred. But if only nitrogen in pure form is to be separated from air then pressure swing adsorption (PSA) is preferred.

8.3 SELECTION OF EQUIPMENT FOR DISTILLATION[1,2]

In most of the cases, distillation is carried out either in a tray tower or in a packed tower. Criteria of selection between tray tower and packed tower are as follows.

1. For high vacuum distillation, a packed tower is selected. Packed tower generally provides lower pressure drop as compared to the tray tower. Pressure drop in packed tower is usually in the range of 1/5th to 1/8th of the same in tray tower. For example, in the distillation of the mixture of monochloroacetic acid and acetic acid, absolute operating pressure in the reboiler should be less than 50 Torr a to avoid thermal cracking of bottom product (monochloroacetic acid). For this case, packed tower should be selected. Packed tower provides lower residence time for liquid as compared to tray tower. The rate of thermal cracking is directly proportional to residence time. For highly heat sensitive product, decrease in boiling point means improvement in the quality. The rate of thermal cracking increases exponentially with the temperature. For very high vacuum distillation, short path distillation is used. For example, separation of vitamin E from its crude mixture (such as soybean oil deodoriser distillate) is carried out in short path distillation equipment. Operating absolute pressure in short path distillation can be as low as 0.05 mbar or 50 μbar.

   For low pressure distillation applications, liquid flow rates tend to be very low. For such applications, packed column can be opted provided liquid rates are above minimum wetting rates for the selected packing.
2. For very small tower diameter packed tower is preferred. With large tower diameter, tray tower is preferred. With very small diameter (less than 150 mm), fabrication of trays is difficult while with large diameter, liquid distribution is difficult in the packed tower.
3. For handling higher ratio of liquid flow rate to vapour flow rate, packed towers are preferred. For very low liquid flow rate and for very low L/V ratio, tray tower is selected. Packed tower requires minimum wetting rate to work efficiently.

4. For corrosive system, packed tower with plastic packings like polypropylene packings, glass fibre reinforced plastic packings, etc., is more economical than tray tower made from special metal alloys. For example, for the separation of methyl iodide and acetic acid, packed tower with glass fibre reinforced plastic packings is more economical than tray tower which should be made entirely from Hastalloy–B.
5. If the side products are to be drawn from the distillation column, like the distillation columns of refinery, tray tower is preferred.

8.4 DISTILLATION COLUMN DESIGN

The design of a distillation column can be divided into the following steps:

1. If a feed mixture contains more than two components, then select two key components from all the components of the feed mixture. For a binary feed mixture determine more volatile and less volatile components.
2. Decide the operating pressure of distillation column. At the operating pressure, find the vapour–liquid equilibrium data.
3. Specify the degree of separation required. In case of a binary distillation, decide the top (distillate) and the bottom (residue) product compositions. In case of multicomponent distillation, decide the distribution of key components in distillate and residue. Also, find the distribution of non key components in the same. Complete the material balance.
4. Determine the minimum reflux ratio R_m.
5. Determine the optimum reflux ratio R and find the number of theoretical or equilibrium stages required for the desired separation.
6. Select the type of tower: tray tower or packed tower (as discussed in Section 8.3). Select the type of tray in tray tower or type of packing in packed tower.
7. Find tower diameter and pressure drop across the tower. Decide the top disengagement and bottom disengagement spaces.
8. Determine the tray efficiency and find the actual number of trays in case of tray tower or find HETP (height equivalent to theoretical plate) and decide the height of packing in packed tower. In case of packed tower, design the liquid distributor, redistributor, packing support, etc.
9. Select the material of construction for all parts. Design the suitable insulation system.
10. Design the condenser and reboiler.
11. Decide the control methodology for the distillation column, line sizes and locations of all nozzles.
12. Mechanical design of distillation column is to be carried out which also includes design of suitable supports for the distillation column.

Calculation and equations for the first five steps are different for binary and multicomponent distillation. In this chapter first eight steps [(1) to (8)] are discussed in detail.

8.4.1 Step 1: Selection of Key Components[1,2]

If the feed mixture to distillation column contains only two components, then it is called *binary distillation*. In many cases, feed mixture actually contains more than two components, but it is considered as binary mixture because the amounts of other components (except two key components) are negligible.

When a feed mixture to distillation column contains more than two components, it is called *multicomponent distillation*. In such a distillation, it is not possible to obtain more than one pure component in single column. For the separation of N' component mixture by distillation, if all the components are to be separated in pure form, then at least N'–1 distillation columns are required.

In multicomponent distillation, one cannot specify the complete composition of the top and bottom products independently. For example, a feed mixture to distillation column is 100 kmol/h containing 20 kmol/h ethane, 30 kmol/h propane and 50 kmol/h of butane. One cannot fix distillate composition: 20 kmol/h ethane, 29 kmol/h propane, 1 kmol/h butane and residue composition: 1 kmol/h propane, 49 kmol/h of butane. One can fix that distillate will contain 29 kmol/h of propane and 1 kmol/h of butane and residue will contain 1 kmol/h of propane and 49 kmol/h of butane.

Based on the degree of freedom analysis for the multicomponent distillation, one can fix only distribution of any two components of feed mixture in distillate and residue but not the distribution of all components[3]. Before starting the design of multicomponent distillation, two key components are selected. Key components are the two components of the feed mixture between which one likes to make the sharp separation. Two key components are further divided as light key component and heavy key component. More volatile key component is called *light key* and less volatile component is called *heavy key*.

Light key component It is a component of the feed mixture which is desired to be kept out of the bottom product. It is the component of feed which is present in residue in finite amount, while components lighter than light key are either not present or present in very small amount. If all components of feed are present in residue with sufficient concentrations, then most volatile component of residue is the light key component.

Heavy key component It is a component of feed mixture which is desired to be kept out of the top product. It is the component of feed which is present in distillate in finite amount, while all components heavier than the heavy key are not present or present in negligible amount. If all components of feed are found in distillate with sufficient concentrations, then the least volatile is the heavy key component.

If the light key and heavy key components are selected as per the order of volatility then they are known as *adjacent keys*. If any other component lies in between them, then they are known as *split keys*. If a component has very low concentration in feed, then it is not selected as key component.

Components of the feed mixture other than key components are known as *non-key components*. The non-key components which are distributed in top and bottom

products are known as *distributed components*. Any component which does not appear in either top or bottom product up to significant extent is known as *non-distributed component*.

8.4.2 Step 2: Selection of Operating Pressure and Determination of Vapour-Liquid Equilibrium Data

Selection of operating pressure In many cases of high pressure distillation column, operating pressure in distillation column is fixed by the temperature of the cheapest cooling medium (e.g., cooling water), which must be able to condense the distillate vapour. For this, bubble point of distillate (in case of total condenser) should be sufficiently greater than cooling water temperature. In case of partial condenser, bubble point of reflux (is equal to dew point of distillate vapour) should be sufficiently greater than temperature of cooling water. But if, this operating pressure (based on cooling water temperature) is calculated to be very high, then chilled water or brine can be considered as cooling medium for the overhead condenser. Operating pressure in distillation column is always less than the critical pressure of top product or temperature of cooling medium of overhead condenser cannot be greater than critical temperature of top product. In many cases, distillation is carried out under vacuum to avoid the thermal degradation of heat sensitive product or to facilitate the use of cheaper heating medium like saturated steam in reboiler or to alter the vapour-liquid equilibrium data.

Examples for the selection of operating pressure

1. Distillation of acetaldehyde-ethanol-water mixture is carried out at about 3 atm g pressure. Pure acetaldehyde is the top product. Normal boiling point (t_B at 1 atm) of acetaldehyde is 20.2°C. To facilitate the use of cooling water as a cooling medium in the overhead condenser, boiling temperature or condensation temperature of acetaldehyde should be sufficiently greater than the design temperature of cooling water from cooling tower (say 32°C). Also the product acetaldehyde is stored in bullet-type storage tanks which are exposed to atmosphere. Hence, to reduce the cost of storage or to avoid the use of insulation around storage tank and to avoid the use of any refrigeration system for the storage tank, boiling point of acetaldehyde should be sufficiently greater than atmospheric temperature (50°C maximum). Considering all these aspects, condensation temperature (is also equal to boiling temperature of pure component) of acetaldehyde is fixed at around 60°C. For getting 60°C as a condensation temperature of acetaldehyde, required operating pressure at the top of distillation column is around 3.6 atm g (vapour pressure of acetaldehyde at 60°C). It also eliminates the use of pump for the transfer of acetaldehyde to storage tank.
2. For the separation of methyl chloride from the mixture of chloromethanes, normal boiling point or condensation temperature of pure methyl chloride is –24°C. At 9 atm a pressure, its condensation temperature is 40°C. Hence, to facilitate the use of cooling water as a cooling medium in overhead condenser, this distillation column can be operated at 9 atm a pressure. Similarly, in case of distillation of

ammonia (Example 8.7), saturation pressure of ammonia at 40°C is 1555.5 kPa a. Hence, the column is operated at about 15 bar g to facilitate condensation of ammonia at 40°C in the condenser with cooling water at 32°C.

3. For the separation of ethane from the mixture of ethane, propane and butanes by distillation, cooling water cannot be used as a cooling medium in the overhead condenser because critical temperature of ethane (t_c = 32.3°C) is not sufficiently greater than the temperature of cooling water (say 32°C). Minimum driving force required for heat transfer in shell and tube-type overhead condenser is 3 to 5°C. At 10°C, vapour pressure of ethane is 30 atm a, hence chilled water (at 5 to 7°C) can be used as a cooling medium in the overhead condenser, but the required operating pressure is 30 atm a, which is high particularly for larger diameter column. At 20 atm a pressure, condensation temperature of pure ethane is –6.4°C, hence, suitable brine system can be used as a cooling medium in the overhead condenser, but it will require higher cost of refrigeration and insulation. In the plant, if this column is following a demethaniser column, then top product stream of demethaniser can be used as a cooling medium (heat pump arrangement) in the overhead condenser of deethaniser column and operating pressure can be reduced to very low value, close to atmospheric. Optimum solution should therefore be worked out.
4. In some high pressure distillation columns, operating pressure is limited by undesirable side reactions which otherwise take place at higher pressure and higher temperature. For example, to avoid the polymerisation of butadiene, operating pressure of debutaniser, depropaiser, de-ethaniser, etc., columns of naphtha or natural gas cracker units are kept at lower pressure.

Vacuum distillation For heat sensitive material or to facilitate the use of cheaper heating medium in reboiler or to alter the vapour-liquid equilibrium data, distillation column is operated under vacuum. Advantages and disadvantages of vacuum distillation are given below.

Advantages

1. It prevents thermal decomposition of heat sensitive material. For example, in separation of monochloroacetic acid and acetic acid by distillation, monochloroacetic acid is obtained as bottom product. Boiling point of monochloroacetic acid must be restricted below 120°C, otherwise thermal degradation can take place. If the boiling point of monochloroacetic acid is kept at 109°C, then operating pressure at bottom of column must be equal to 40 Torr a.
2. It allows the use of more economical heating medium such as saturated steam against hot oil in high capacity plant. For example, for the separation of ethylene glycol-water mixture by distillation, ethylene glycol is obtained as bottom product. Its normal boiling point is 197°C. Use of saturated steam as a heating medium could be economical up to 180°C. Hence, this column is better to be operated under vacuum to facilitate the use of saturated steam instead of use of hot oil.
3. It requires fewer stages or lower reflux ratio or both in many cases as relative volatility increases with decrease in the operating pressure.

4. In some case vacuum breaks the azeotrope and make the separation easier. For example, if the distillation of ethanol–water mixture is carried out below 70 Torr absolute pressure, azeotrope does not form.
5. It reduces undesirable side reactions, if they are taking place. For example, distillation of ethylbenzene-styrene mixture under vacuum reduces polymerisation.
6. Vacuum distillation provides safety in the distillation of toxic or hazardous material. For example, for the separation of nearly 100% pure nitric acid from the azeotropic mixture of nitric acid and water, extractive distillation is used. This extractive distillation uses concentrated Sulphuric acid as solvent (Example 8.21). Material of construction of this column is borosilicate or high quality glass. This column is operated under slight vacuum for safety purpose. Here, vacuum also facilitates the use of saturated steam in reboiler.
7. It increases product recovery when dealing with maximum temperature constraints. For example, refinery vacuum tower.

Disadvantages

1. It requires larger column diameter and larger diameter vapour lines.
2. Because of the reduction in condensation temperature, costlier cooling medium may be required for condensation.
3. Presence of air as a non-condensable reduces the efficiency of main condenser. Also air leaving the condenser is saturated with vapour of top product.
4. Increases the possibility of contamination of products by air or other components of atmosphere.

Determination of vapour-liquid equilibrium (VLE) data

VLE data must be determined for the given mixture for distillation and at the operating pressure of distillation column. To find the VLE data, there are three options. Priority-wise these three options are given below.

1. VLE data can be determined by an experiment. If the operating pressure is atmospheric or vacuum, then VLE data-experiment is easier. Only difficult part is analysis. But if VLE data are required at high pressure, then this experiment is difficult. This is the most reliable option or method for finding the VLE data for a given mixture because the given feed mixture obtained from lab scale or pilot plant scale process for distillation may contain some unknown impurities which may not be considered or ignored in other two options or methods given in the coming section.
2. Find the VLE data for the given system from the literature, if they are available at the same operating pressure. In the literature, VLE data are available for the several thousand binary and many multicomponent systems. All these data were determined by actual experiments. Major sources for these data are as follows.
   (i) **Vapour-Liquid and Liquid-Liquid Equilibrium Data Collection**, DECHEMA Chemistry Data Series[4], Volume-1, Parts 1 to 10, by Gmehling and Onken (1977).
   (ii) **Vapour-Liquid Equilibrium Data**[5] by Chu, Ju-chin *et al.* (1956).

(iii) **Vapour-Liquid Equilibrium Data Bibliography**[6] by Wichterle, Linek and Hala, Elsevier, The Netherlands, 1976.

(iv) **Vapour-Liquid Equilibrium Data at Normal Pressures**[7] by Boublik, Polak, Wichterle ad Hala, Pergamon, Oxford, UK, 1968

(v) **Perry's Chemical Engineers Handbook**[8], 7th Edition by Perry and Green, McGraw-Hill, USA, 1998.

(vi) **Azeotropic Data**[9], 3 Vol., 2nd Ed. by Gmehling, J., Wiley-VCH, Germany, 2004.

3. VLE data can be determined theoretically by using phase equilibrium equation. In the given feed mixture for distillation, if all components are from the same group and operating pressure for distillation is atmospheric then usually vapour-liquid equilibrium for such mixture can be assumed as ideal vapour-liquid equilibrium. For example, feed mixture of benzene and toluene at atmospheric pressure.

For the ideal VLE, Roult's law is applicable.

$$y_i p_t = x_i p_v \quad \text{(Roult's law)} \tag{8.1}$$

where, y_i = Mole fraction of component i in vapour phase
x_i = Mole fraction of component i in liquid phase
p_t = Operating pressure, kPa
p_v = Vapour pressure of pure liquid i at the given temperature, kPa

General form of vapour-liquid equilibrium equation is $y_i = K_i x_i$ (8.2)

where, K_i = Equilibrium constant for component i

K_i – values for some hydrocarbons for the range of operating pressure from 101.3 kPa to 6000 kPa and for the range of operating temperature from –70°C to 200°C, can be obtained-from Dadyburjor chart (refer to Fig. 6.20).

For non-ideal system VLE data is determined by following equation:

$$y_i \varphi_i p_t = x_i \gamma_i p_v \tag{8.3}$$

where, φ_i = Fugacity coefficient of component i
γ_i = Activity coefficient of component i

Fugacity coefficient φ_i represents the deviation of vapour phase from ideal gas mixture. If the operating pressure is close to atmospheric pressure then vapour phase can be assumed as ideal gas and φ_i is unity. φ_i can be calculated from an appropriate equation of state like Redlich–Kwong equation, Peng–Robinson equation, etc.

Activity coefficient γ_i represents the deviation of the behaviour of component in given solution from the behaviour of same component in ideal solution. γ_i is a function of temperature and liquid composition.

$$\gamma_i = f(x_i, T) \tag{8.4}$$

γ_i is not dependent on pressure.

Activity coefficient γ_i can be determined by van Laar equation, UNIFAC method or UNIQUAC method or by NRTL equation. Group contribution method like UNIFAC method or UNIQUAC method are more reliable, but group contribution parameters are available for limited number of groups in literature. For example, for the mixture of ethane and propane, if VLE data are required at 10 atm a pressure, then $\varphi_i \neq 1$, but γ_i can be assumed equal to 1 for both components.

For the mixture of ethanol-ethyl iodine, if VLE data required are at 1 atm pressure, then for both components

$$\varphi_i \cong 1, \text{ but } \gamma_i \neq 1.$$

8.4.3 Step 3: Degree of Separation

In case of binary distillation, specify x_D and x_W considering equilibrium limitations in mind, (where x_D = mole fraction of more volatile component in liquid distillate and x_W = mole fraction of more volatile component in residue). Find the molar flow rate of distillate D and molar flow rate of residue W, by using following two material balance equations:

$$F = D + W \tag{8.5}$$

$$Fz_F = Dx_D + W\,x_W \tag{8.6}$$

where z_F represents mole fraction of more volatile component in feed. Since feed can be saturated liquid, saturated vapour or a mixture of liquid and vapour, symbol z_F is used. If it is only liquid, symbol x_F can be used.

To establish the material balance in multicomponent distillation is not easy. In case of multicomponent distillation, specify the distribution of two key components in distillate (top product) and in residue (bottom product), considering equilibrium limitations in mind. Exact distribution of non-key components in top product and bottom predict is not possible at the starting of design. But, the same can be determined approximately by Hegstebeck and Geddes equation[10].

$$\log\left(\frac{d_i}{b_i}\right) = A + C \log \alpha_i \tag{8.7}$$

where, d_i = Moles of ith component in distillate
b_i = Moles of ith component in residue
α_i = Average relative volatility of ith component with respect to heavy key component
A, C = Constants

Material balance calculations (approximate) for multicomponent distillation is illustrated by following example.

Example 8.1 Feed mixture to a distillation column contains 30% *n*-hexane, 32% *n*-propane, 20% *n*-butane and 18% *n*-pentane (by mole). Total flow rate of the feed is 100 kmol/h. If butane and pentane are selected as light key and heavy key components, respectively then (a) fix the operating pressure of distillation column, and (b) find the product compositions.

Solution

Generally, the mixture of propane and butane is stored and sold as LPG (Liquefied Petroleum Gas). Hence in that case, top product (mixture of propane and butane) must be in liquid phase or a total condenser must be used. To facilitate the use of cooling water as cooling medium in overhead total condenser, bubble point temperature of distillate must be sufficiently greater than design temperature of cooling water from cooling tower (say 32°C). Also to facilitate the storage of liquid distillate without using insulation and refrigeration system, bubble point temperature must be sufficiently

greater than atmospheric temperature. Here distillate is highly flammable liquid. Hence, to avoid the use of a pump between distillation column and a storage tank, let bubble point of the distillate be 60°C.

For the first trial calculations, assuming that non-key components, propane will appear in distillate only and hexane will appear in residue only. For the first trial calculations material balance is assumed as follows.

Table 8.1 Material Balance of Distillation Column (1st Trial Calculations)

Component	Distillate, kmol/h	Residue, kmol/h
n-Propane	32.0	—
n-Butane	19.8	0.2
n-Pentane	0.2	17.8
n-Hexane	—	30.0
Total	52.0	48.0

Bubble point temperature of distillate = 60°C. At bubble point, $\Sigma K_i\, x_i = 1$

$$K_{Pr}\, x_{Pr} + K_{Pe}\, x_{Pe} + K_{Bu}\, x_{Bu} = 1$$

$$x_{Pr} = \frac{32}{52} = 0.6154,\ x_{Pe} = \frac{0.2}{52} = 0.0038,\ x_{Bu} = \frac{19.8}{52} = 0.381$$

$$0.6154\, K_{Pr} + 0.0038\, K_{Pe} + 0.381\, K_{Bu} = 1 \qquad \text{(a)}$$

Here values of equilibrium constants are to be determined at the temperature 60°C and operating pressure p_t which is unknown. Hence by trial and error calculations find the value of p_t which will satisfy Eq. (a). Values of equilibrium constants are determined by using Fig. 6.20.

At p_t =1500 kPa and t = 60°C

$K_{Pr} = 1.4,\quad K_{Pe} = 0.21,\quad K_{Bu} = 0.52$

$\Sigma K_i\, x_i = 0.6154 \times 1.4 + 0.0038 \times 0.21 + 0.381 \times 0.52 = 1.06$

At p_t = 1600 kPa and 60°C,

$K_{Pr} = 1.35,\quad K_{Pe} = 0.2,\quad K_{Bu} = 0.5$

$\Sigma K_i x_i = 0.6154 \times 1.35 + 0.0038 \times 0.2 + 0.38 \times 0.5 = 1.022 \simeq 1$

Operating pressure at top of column = 1600 kPa a

Assume pressure drop in column = 30 kPa

Operating pressure at the base of column = 1630 kPa a

To decide the distribution of non-key components Hegstebeck–Gedde's equation can be used.

$$\log\left(\frac{d_i}{b_i}\right) = A + C \log \alpha_i \qquad (8.7)$$

where, $$\alpha_i = \alpha_{avi} = (\alpha_{itop} \cdot \alpha_{ibottom})^{1/2} \qquad (8.8)$$

To find out the average value of relative volatility, top and base (bottom) temperatures of distillation column are to be determined.

For distillation column with total condenser, composition of overhead vapour and distillate are same. Topmost temperature of distillation column is equal to dew point temperature of overhead vapour.

Topmost temperature

At dew point, $$\Sigma \frac{y_i}{K_i} = 1$$

$$\frac{0.6154}{K_{\text{Pr}}} + \frac{0.381}{K_{\text{Bu}}} + \frac{0.0038}{K_{\text{Pe}}} = 1$$

At operating pressure p_t = 1600 kPa and at temperature t, values of equilibrium constants must be determined, where t is unknown.

For the first trial calculations, Let t = 75°C

At 75°C, $K_{Pr} = 1.65$, $K_{Bu} = 0.65$ $K_{Pe} = 0.26$

$$\Sigma \frac{y_i}{K_i} = \frac{0.6154}{1.65} + \frac{0.381}{0.65} + \frac{0.0038}{0.26} = 0.974$$

At 72°C, $K_{Pr} = 1.55$, $K_{Bu} = 0.6$, $K_{Pe} = 0.25$

$$\Sigma \frac{y_i}{K_i} = \frac{0.6154}{1.55} + \frac{0.381}{0.6} + \frac{0.0038}{0.25} \approx 1$$

Bottommost temperature

Bottommost temperature of distillation column is the bubble point of residue. Operating pressure at the base of column, p_t' = 1630 kPa.

At bubble point $\Sigma K_i x_i = 1$

For residue, $x_{\text{Bu}} = \frac{0.2}{48} = 0.004\ 17$, $x_{\text{Pe}} = \frac{17.8}{48} = 0.37$, $x_{\text{He}} = \frac{30}{48} = 0.625$

For the first trial calculations, let t = 150°C (and at 1630 kPa)

$K_{\text{Bu}} = 1.9$, $K_{\text{Pe}} = 0.95$, $K_{\text{He}} = 0.52$

$\Sigma K_i x_i = 0.004\ 17 \times 1.9 + 0.37 \times 0.95 + 0.625 \times 0.52 = 0.684$

Let t = 180°C. $K_{\text{Bu}} = 2.5$, $K_{\text{Pe}} = 1.3$, $K_{\text{He}} = 0.79$

$\Sigma K_i x_i = 0.004\ 17 \times 2.5 + 0.37 \times 1.3 + 0.625 \times 0.79 = 0.99 \approx 1$

Table 8.2 Relative Volatilities

Feed Component	$\alpha_{72°\,C}$	$\alpha_{180°\,C}$	$\alpha_{avi} = (\alpha_{72} \cdot \alpha_{180})^{0.5}$
n-Propane	$\frac{1.55}{0.25} = 6.20$	$\frac{4.25}{1.3} = 3.270$	4.500
n-Butane (LK)	$\frac{0.6}{0.25} = 2.40$	$\frac{2.5}{1.3} = 1.923$	2.150
n-Pentane (HK)	1.00	1.000	1.000
n-Hexane	$\frac{0.11}{0.25} = 0.44$	$\frac{0.79}{1.3} = 0.6077$	0.517

At 72°C, $K_{\text{He}} = 0.11$ and at 180°C, $K_{\text{Pr}} = 4.25$.

For butane (light key component):

$$\log\left(\frac{d_i}{b_i}\right) = A + C \log \alpha_i$$

$$\log\left(\frac{19.8}{0.2}\right) = A + C \log (2.15)$$

$$1.9956 = A + 0.3324\, C$$

For pentane (heavy key component)

$$\log\left(\frac{0.2}{17.8}\right) = A + C \log (1)$$

$$-1.949\ 39 = A + 0$$

Solving two equations

$$A = -1.949\ 39 \text{ and } C = 11.8682$$

For propane:

$$\log\left(\frac{d_i}{b_i}\right) = -1.949\ 39 + 11.8682 \log (4.5)$$

$$\log\left(\frac{d_i}{b_i}\right) = 5.803$$

$$\left(\frac{d_i}{b_i}\right) = 635\ 331 \text{ (very high)}$$

$$d_i = 32 \text{ kmol/h}$$
$$b_i = 5 \times 10^{-5} \text{ kmol/h} \approx 0 \text{ kmol/h}$$

For hexane:

$$\log\left(\frac{d_i}{b_i}\right) = -1.949\ 39 + 11.8682 \log (0.517)$$

$$\left(\frac{d_i}{b_i}\right) = 4.47 \times 10^{-6}$$

$$b_i = 30 \text{ kmol/h}$$
$$d_i = 1.34 \times 10^{-4} \text{ kmol/h} \approx 0 \text{ kmol/h}$$

Propane does not appear in the residue and hexane does not appear in the distillate. Hence, assumption made before deciding the distribution of components is valid. No correction is required. So Table 8.1 shows the final product composition.

8.4.4 Step 4: Minimum Reflux Ratio

Determine the minimum reflux ratio R_m. It is the maximum reflux ratio for which infinite number of trays are required for the desired separation. It corresponds to the minimum condenser cooling load and the minimum reboiler heating load required for the desired separation. In other words, on decreasing reflux ratio, a value of reflux ratio obtained for which desired separation becomes impossible, is called *minimum reflux ratio;* R_m.

Determination of R_m for binary distillation For determination of R_m, it is important to know quality of feed which is characterised by a parameter q. It is defined as moles of liquid flow to stripping section of the column per mole of feed and it can be determined by the following equation:

$$q = \frac{H_G - H_F}{H_G - H_L} \tag{8.9}$$

where, H_G = Enthalpy of saturated vapour, kJ/kmol
H_F = Enthalpy of feed, kJ/kmol
H_L = Enthalpy of saturated liquid, kJ/kmol

Equation (8.9) can be modified for calculations as given in Eq. (8.16).

If 1. feed is below bubble point, $q > 1$
2. feed is at bubble point, $q = 1$ (i.e., saturated liquid)
3. feed is partially vaporised, $q < 1$
4. feed is at dew point, $q = 0$ and
5. feed is superheated vapour, $q < 0$.

(a) In case of binary distillation with ideal vapour-liquid equilibrium curve or with the equilibrium curve which is always concave downward, R_m can be calculated by any of the following equations[1]:

(i) For feed liquid at the bubble point or with, the saturated liquid feed (i.e., for $q = 1$)

$$R_m = \frac{1}{(\alpha - 1)}\left[\frac{x_D}{x_F} - \frac{\alpha(1 - x_D)}{1 - x_F}\right] \tag{8.10}$$

where, α = Average relative volatility
x_D = Mole fraction of more volatile component in distillate
x_F = Mole fraction of more volatile component in feed liquid

(ii) For the saturated vapour feed or for feed vapour at dew point (i.e., for $q = 0$)

$$R_m = \frac{1}{(\alpha - 1)}\left[\frac{\alpha x_D}{y_F} - \frac{1 - x_D}{1 - y_F}\right] - 1 \tag{8.11}$$

where, y_F = Mole fraction of more volatile component in feed vapour

(iii) For any other condition of feed (for any value of q), R_m can be determined by solving the following equation:

$$\frac{R_m z_F + q x_D}{R_m(1 - z_F) + q(1 - x_D)} = \frac{\alpha[x_D(q - 1) + z_F(R_m + 1)]}{(R_m + 1)(1 - z_F) + (q - 1)(1 - x_D)} \tag{8.12}$$

(b) In case of binary distillation with ideal or non-ideal vapour-liquid equilibrium curve, minimum reflux ratio R_m can be determined by McCabe–Thiele method (Ref. 1, 11). Following assumptions are made in McCabe–Thiele method:

(i) For any equilibrium stage, molar latent heat of vaporization of liquid on tray is equal to molar latent heat of condensation of vapour leaving the tray.

(ii) Heat losses and heat of solution (or say mixing) are negligible.

(iii) Total condenser is used but there is no subcooling. Condenser removes all the latent heat from the overhead vapour but does not cool the resulting liquid further.

Because of the first two assumptions, molar flow rate of liquid and molar flow rate of vapour in each section (enriching section and stripping section) is assumed to remain constant throughout the section. Because of the third assumption, composition of overhead vapour is equal to composition of distillate and also equal to composition of reflux.

$L, G, \bar{L}, \bar{G}$ = constant

$x_D = y_i = x_o$

where, L, G = Molar flow rates of liquid and vapour, respectively in rectification or enriching section, kmol/h.

$\bar{L}, \bar{G}$ = Molar flow rates of liquid and vapour, respectively in stripping or exhausting section, kmol/h.

x_D = Mole fraction of more volatile component in overhand vapour

x_o = Mole fraction of more volatile component in reflux.

Method for determining R_m by McCabe Thiele method:

(i) Draw the vapour–liquid equilibrium curve, y vs x. Also, draw the 45° diagonal ($x = y$) line.

(ii) Mark the points $x = y = x_D$, $x = y = z_F$ and $x = y = x_W$ on the 45° diagonal line.

(iii) Draw the q-line. It starts from the point $x = y = z_F$. The equation of q-line is

Thus, $$y = \frac{q}{q-1}x - \frac{z_F}{q-1} \tag{8.13}$$

Slope of q-line is $\dfrac{q}{q-1}$.

(iv) Draw the line from $x = y = x_D$ point to the point of intersection of q-line and equilibrium curve. Extend the same line to y-axis. Intercept of this line gives the value of $\dfrac{x_D}{R_m + 1}$ from which the value of R_m can be calculated. But this procedure is valid for ideal vapour-liquid equilibrium curves and also for non-ideal vapour-liquid equilibrium curves except the curves as shown in Figs. 8.1(b) and (c). For the non-ideal VLE curve like Fig. 8.1(b) tangent line of equilibrium curve decides the value of R_m. For the non-ideal VLE curve as shown in Fig. 8.1(c), a tangent operating line of stripping section may set the value of R_m.

In case of multicomponent distillation, R_m can be determined by Underwood's method[1,8,12]. In this method, R_m is determined by solving the following two equations:

$$\Sigma \frac{\alpha_i x_{iD}}{\alpha_i - \vartheta} = R_m + 1 \tag{8.14}$$

$$\Sigma \frac{\alpha_i z_{iF}}{\alpha_i - \vartheta} = 1 - q \tag{8.15}$$

where, α_i = Average relative volatility of each component i with respect to heavy key

x_{iD} = Mole fraction of component i in distillate

z_{iF} = Mole fraction of component i in feed

q = Heat required to convert 1 mole of feed from its condition to a saturated vapour divided by latent heat of vaporisation [Eq. (8.9)].

$$\vartheta = \text{Constant}$$

Here $\alpha_i = (\alpha_{i\text{top}} \cdot \alpha_{i\text{bottom}})^{1/2}$ (8.8)

Fig. 8.1 *Determination of Minimum Reflux Ratio for Binary Distillation*

Topmost temperature of distillation column is dew point temperature of overhead vapour. Bottommost temperature of distillation column is bubble point temperature of residue.

For accuracy purpose, it is suggested that ϑ, must be determined correct up to four decimal places[13]. Equation (8.15) may give many values of ϑ, but correct value of ϑ is that belongs to the interval starting from 1 to the value of α_{LK}.

$$\vartheta \in \{\alpha_{HK}, \alpha_{LK}\} \text{ or } \alpha_{LK} > \vartheta > \alpha_{HK}$$

where, α_{LK} = Relative volatility of light key component with respect to heavy key component.

α_{HK} = Relative volatility of heavy key component with respect to heavy key component = 1

Example 8.2 Determine the minimum reflux ratio for the binary distillation at standard atmospheric pressure based on the following data:

Feed = 100 kmol/h

Feed mixture: benzene-toluene

Mole fraction of benzene in feed, $x_F = 0.4$
Condition of feed = Saturated liquid
Mole fraction of benzene in distillate required, $x_D = 0.99$
Mole fraction of benzene in residue required, $x_W = 0.02$
Average relative volatility, $\alpha = 2.25$

Solution

Benzene-toluene mixture behaves as an ideal mixture.

$$R_m = \frac{1}{(\alpha - 1)}\left[\frac{x_D}{x_F} - \frac{\alpha\,(1 - x_D)}{1 - x_F}\right] \tag{8.10}$$

$$R_m = \frac{1}{(2.25 - 1)}\left[\frac{0.99}{0.4} - \frac{2.25\,(1 - 0.99)}{1 - 0.4}\right]$$

Minimum reflux ratio $R_m = 1.95$

Example 8.3 In the previous example, if the feed is at room temperature (30°C), what is the value of R_m?

Solution

To find the value of R_m, first value of q must be determined using Eq. (8.9).

$$q = \frac{H_G - H_L + (H_L - H_F)}{H_G - H_L} = \frac{\lambda + C_p \Delta t}{\lambda} \tag{8.16}$$

t_F = Temperature of feed = 30°C
t_b = Bubble point temperature of feed

At bubble point:

$$\Sigma x_i K_i = 1$$

$$\Sigma x_i \frac{p_{vi}}{p_t} = 1 \text{ (for ideal VLE)}$$

Antoine equations[14]

For benzene, $$\ln p_{vB} = 15.9008 - \frac{2788.51}{T - 52.36}$$

For toluene, $$\ln p_{vT} = 16.0137 - \frac{3096.52}{T - 53.67}$$

For the first trial calculations let $t_B = \Sigma t_{Bi}\, x_i$
where, t_{Bi} = normal boiling point of pure component i
Normal boiling point of benzene = 80.1°C
Normal boiling point of toluene = 110.6°C

For first trial calculations, $t_B = 0.4 \times 80.1 + 0.6 \times 110.6 = 98.4$°C (371.4 K)

$$\ln p_{vB} = 15.9008 - \frac{2788.51}{371.4 - 52.36} = 7.16$$

$$p_{vB} = 1287.54 \text{ Torr, similarly}$$
$$p_{vT} = 527.4 \text{ Torr}$$
$$\Sigma x_i p_{vi} = 0.4 \times 1287.54 + 0.6 \times 527.4 = 831.456 \text{ Torr} \neq 760 \text{ Torr}$$

After trial and error calculations,

$$\text{at } t_B = 95.3°\text{C}, \quad p_{vB} = 1181.72 \text{ Torr}, \quad p_{vT} = 479.1 \text{ Torr}$$
$$\Sigma x_i p_{vi} = 0.4 \times 1181.72 + 0.6 \times 479.1 = 760.15 \text{ Torr}$$

Bubble point of feed mixture, $t_{BP} = 95.3°\text{C}$

Specific heat must be determined at the average temperature

$$t_{\text{av}} = \frac{95.3 + 30}{2} = 62.65°\text{C}$$

Table 8.3 Property Data of Benzene and Toluene

Component	C_L at 62.65°C, kJ/(kmol · °C)	λ at 95.3°C, kJ/kmol
Benzene	146.96	29 391.3
Toluene	173.33	34 666.7

(Figure 3.11 and Table 3–179, of Ref. 14)

Average molar heat capacity, $C_L = \Sigma C_{Li}\, x_i$

$$C_L = 146.96 \times 0.4 + 173.33 \times 0.6$$
$$= 162.78 \text{ kJ/(kmol} \cdot °\text{C)}$$

Average latent heat

$$\lambda = \Sigma x_i\, \lambda_i = 0.4 \times 29\,391.3 + 0.6 \times 34\,666.7$$
$$= 32\,556.54 \text{ kJ/kmol}$$
$$q = \frac{32\,556.54 + 162.78 \times (95.3 - 30)}{32\,556.54}$$
$$= 1.3265$$

R_m can be determined by Eq. (8.12).

$$\frac{(R_m \times 0.4) + (1.3265 \times 0.99)}{R_m(1 - 0.4) + 1.3265\,(1 - 0.99)} = \frac{2.25\left[0.99(1.3265 - 1) + 0.4(R_m + 1)\right]}{(R_m + 1)\,(1 - 0.4) + (1.3265 - 1)\,(1 - 0.99)}$$

$$\frac{0.4R_m + 1.313\,235}{0.6R_m + 0.013\,265} = \frac{0.7273 + 0.9\,(R_m + 1)}{0.6(R_m + 1) + 0.003\,265}$$

After the trial-and-error calculations, for $R_m = 1.67$, LHS (1.9514) ≃ RHS (1.95)

Use of a mathematical software such as Mathcad® or Maple® can be made to solve such an equation.

Minimum reflux ratio, $R_m = 1.67$

Example 8.4 A distillation column is to separate 4750 mol/h of feed composed of 37% *n*-butane, 32%. *iso*-pentane, 21% *n*-pentane and 10% *n*-hexane. The column operates at an average pressure of 2 atm a and will produce a distillate product containing 95% *n*-butane and 5% *iso*-pentane. The bottom

product is allowed to contain no more than 570 mol/h of *n*-butane. Use Underwood's method to determine the minimum reflux for the required separation. Feed is 25% (by mole) vapour. Assume ideal vapour–liquid equilibrium. All compositions are mole %.

Solution

In this example, distillate composition is given. Hence, residue composition can be determined by material balance. Distillate composition indicates that *n*-butane is light key component and *iso*-pentane is heavy key component.

Overall material balance:

$$F = D + W$$

$$4750 = D + W$$

n-butane balance:

$$0.37 \times 4750 = 0.95D + 570$$

$$D = 1250 \text{ mol/h and } W = 3500 \text{ mol/h}$$

Table 8.4 Feed and Product Compositions

Component	Feed		Distillate		Residue	
	mole %	mol/h	mole %	mol/h	mole %	mol/h
n-butane	37	1757.5	95	1187.5	16.3	570.0
iso-pentane	32	1520.0	5	62.5	41.6	1457.5
n-pentane	21	997.5	—	—	28.5	997.5
n-hexane	10	475.0	—	—	13.6	475.0
Total	100	4750.0	100	1250.0	100.0	3500.0

In Underwood's method, to find average relative volatilities, topmost and bottommost temperatures of distillation column are required.

Topmost temperature = Dew point temperature of overhead vapour

At dew point

$$\Sigma \frac{y_i p_t}{p_{vi}} = 1 \text{ (for ideal VLE) – Raoult's law}$$

$$y_i = x_{iD} \text{ (for total condenser)}$$

$$\left(\frac{0.95}{p_{\text{vnB}}} + \frac{0.05}{p_{\text{viP}}}\right) \times 2 = 1$$

where, p_{vnB} = Vapour pressure of *n*-butane

p_{viP} = Vapour pressure of *iso*-pentane.

Antoine equations[10]:

$$n\text{-butane: } \ln p_{\text{vnB}} = 15.6782 - \frac{2154.9}{T - 34.42}$$

$$iso\text{-pentane: } \ln p_{\text{viP}} = 15.6338 - \frac{2348.67}{T - 40.05}$$

where, T is temperature in K and p_{vnB} is in Torr.
After a trial-and-error calculations dew point temperature can be determined.
For $t = 22°C, \quad T = 295$ K

$$p_{vnB} = 1650 \text{ Torr} = 2.17 \text{ atm}$$
$$p_{viP} = 615 \text{ Torr} = 0.8 \text{ atm}$$
$$\text{LHS} = \left(\frac{0.95}{2.17} + \frac{0.05}{0.8}\right) \times 2 = 1 = \text{RHS}$$

Dew point temperature of overhead vapour = 22°C.
Bottommost temperature = Bubble point of residue
At bubble point:

$$\Sigma x_i\, p_i^{sat} = p_t \quad \text{(for ideal VLE)}$$
$$0.163\, p_{vnB} + 0.416\, p_{viP} + 0.285\, p_{vnP} + 0.136\, p_{vnH} = 2$$

Antoine equations[10]:

n-Pentane: $\ln p_{vnP} = 15.8333 - \dfrac{2477.07}{T - 39.94}$

n-hexane $\ln p_{vnH} = 15.8366 - \dfrac{2697.55}{T - 48.78}$

After a trial and error calculations, final $T = 320$ K or $t = 47°C$

$$p_{vnB} = 3403.3 \text{ Torr} = 4.478 \text{ atm}$$
$$p_{viP} = 1399.95 \text{ Torr} = 1.842 \text{ atm}$$
$$p_{vnP} = 1084.1 \text{ Torr} = 1.426 \text{ atm}$$
$$p_{vnH} = 361.6 \text{ Torr} = 0.476 \text{ atm}$$
$$\text{LHS} = 0.163 \times 4.478 + 0.416 \times 1.842 + 0.285 \times 1.426 + 0.136 \times 0.476$$
$$= 1.97 \approx 2 = \text{RHS}$$

Table 8.5 Relative Volatility Calculations

Component	$\alpha_{22} = \dfrac{p_{vi}}{p_{vHK}}$	$\alpha_{47} = \dfrac{p_{vi}}{p_{vHK}}$	$\alpha_{av} = (\alpha_{22} \times \alpha_{47})^{1/2}$
n-butane (LK)	$\dfrac{2.17}{0.8} = 2.7125$	$\dfrac{4.478}{1.842} = 2.43$	2.567
iso-pentane (HK)	$\dfrac{0.8}{0.8} = 1$	1	1
n-pentane	$\dfrac{0.6}{0.8} = 0.75$	$\dfrac{1.426}{1.842} = 0.774$	0.762
n-hexane	$\dfrac{0.173}{0.8} = 0.216$	$\dfrac{0.476}{1.842} = 0.258$	0.236

Feed contains 25% vapour.

$$H_F = 0.25 \times H_G + 0.75 \times H_L$$

$$q = \frac{H_G - H_F}{H_G - H_L} = \frac{H_G - (0.25H_G + 0.75H_L)}{H_G - H_L} \tag{8.9}$$

$$q = 0.75$$

Underwood's equations; Eq. (8.14) and Eq. (8.15):

Equation (8.15) $\dfrac{2.567 \times 0.37}{2.567 - \vartheta} + \dfrac{1 \times 0.32}{1 - \vartheta} + \dfrac{0.762 \times 0.21}{0.762 - \vartheta} + \dfrac{0.236 \times 0.1}{0.236 - \vartheta} = 0.25$

For $\vartheta = 1.6045, \quad f(\vartheta) = 0.250\,25 = \text{LHS} \approx 0.25 = \text{RHS}$

Also $\vartheta \in \{1, \ldots, 2.567\}$ (check)

Using Eq. (8.14)

$$\frac{2.567 \times 0.95}{2.567 - 1.6045} + \frac{1 \times 0.05}{1 - 1.6045} = R_m + 1$$

$$R_m + 1 = 2.450\,95$$

$$R_m = 1.450\,95$$

8.4.5 Step 5: Determination of Optimum Reflux Ratio

Ideally the reflux ratio which should be used for a new design should be optimum or the most economical. Optimum reflux ratio is the value of reflux ratio for which the total cost of distillation is minimum. Value of optimum reflux ratio depends on the type of system or feed mixture, specified separation and operating conditions. Change in desired purity of products changes the value of optimum reflux ratio and also the minimum reflux ratio. Based on cost analysis, optimum reflux ratio is defined as that value of reflux ratio which causes the total variable annual cost (annual fixed charges plus annual operating costs) to be a minimum.

At the minimum reflux ratio, distillation column requires infinite number of trays or infinite packing height and consequently the fixed cost is infinite but minimum operating cost (cost for the heating medium of reboiler, cooling medium for the condenser, power for reflux pump, etc., minimum). As the reflux ratio increases above the minimum, number of trays required or packing height required rapidly decreases but column diameter, sizes of condenser and reboiler, and size of reflux pump increases. Despite the increase in column diameter and increase in sizes of condenser and reboiler, the annual fixed cost for the distillation goes down as the increase in reflux ratio above minimum results in saving in tower height which more than offsets other fixed costs. This is not continued as the reflux ratio increases further. At certain value of reflux ratio the tray requirement

Fig. 8.2 *Determination of Optimum Reflux Ratio*

or height of packing requirement approaches a near constant value, characteristic of the minimum trays or height requirement, while the diameter and sizes of heat exchangers continue to increase and hence, at some value of reflux ratio, fixed cost passes through minimum point. Increase in the value of reflux ratio further increases the fixed cost again. Operating cost always increases with increase in reflux ratio. The total annual cost, which is sum of operating and fixed costs, must therefore pass through a minimum at the optimum reflux ratio.

For many hydrocarbon distillations, optimum reflux ratio lies between 1.2 to 1.5 times the minimum reflux ratio. But for the difficult distillation, like distillation of acetic acid-water system, optimum reflux ratios are higher than 1.5. For azeotropic mixtures, reflux ratio is usually high. For the optimum design of distillation column, optimum values of reflux ratio, operating pressure and purity of products must be determined.

Determination of theoretical stages To determine number of theoretical stages or equilibrium stages, required for desired separation, many methods are available. But they can be broadly divided in two groups:

1. Methods valid for binary distillation
2. Methods valid for multicomponent distillation

1. Methods Valid for Binary Distillation: Both shortcut methods and detailed methods (rigorous methods) are available. Shortcut methods available for binary distillation are Fensky's equation[1], Smoker's equation[10], etc. Detailed methods available for binary distillation are McCabe–Thiele method[11], Ponchon–Savarit method[1], etc. Also to find the number of theoretical stages for high purity products, Kremser equation[1], Robinson and Gilliland equation[10], etc., are available.

(a) McCabe–Thiele Method[1, 11] Assumptions made in McCabe–Theile method are discussed in the Section 8.4.4. McCabe–Thiele method cannot be used for the following cases:

(i) Cases where heat of solution or heat losses are significantly large. For example, separation of ammonia from dilute aqueous solution by distillation.

(ii) For the cases in which high purity of products are required. In such cases, other method is used to support the McCabe–Thiele method. However, for the same cases one can use only McCabe–Thiele method, but more than one graph are to be drawn at different scales or a software like AutoCad® can be used.

Stepwise procedure for applying McCabe–Thiele method is as follows.

- Draw the vapour liquid equilibrium curve from the VLE data available at the operating pressure of distillation column.
- Mark the point x_D, z_F, and x_W on 45° diagonal line.
- Draw the q-line. It starts from $x = y = z_F$ point and its slope is $q/(q-1)$.
- Determine the minimum reflux ratio R_m.
- Select the value of actual reflux ratio R. Draw the enriching (or rectification) section (nth tray) operating line. The equation of this line is

$$y_{n+1} = \frac{R}{R+1} x_n + \frac{x_D}{R+1} \tag{8.17}$$

This line starts from $x = y = x_D$ point. Its intercept is $x_D/(R + 1)$.

- Draw the stripping or exhausting section operating line. It starts from the point $x = y = x_W$ passing through the point of intersection of enriching section operating line and the q line.
  Operating line of the exhausting (or stripping section) is represented by the following equation:

$$y_{m+1} = \frac{\bar{L}}{\bar{L} - W} x_m - \frac{W}{\bar{L} - W} x_W \tag{8.18}$$

  where y_{m+1} is the vapour composition of light component of the tray above m-th tray and x_m is the liquid composition of light component on m-th tray.

$$\bar{L} = L + F \cdot q \tag{8.19}$$

  and

$$\bar{V} = \bar{L} - W \tag{8.20}$$

- Starting from x_D to x_W or from x_W to x_D, carry out the stepwise construction between operating lines and equilibrium curve. Total number of steps is equal to number of theoretical or equilibrium stages required for the desired separation.

Example 8.5 Acetic acid is to be separated from a process stream containing 80% acetic acid and 20% water (by mass) by continuous distillation at atmospheric pressure. Concentration of water in bottom product (pure acetic acid) should not be greater than 50 ppm. Top product (distillate) contains 80% water and 20% acetic acid (by mass). The feed is liquid at 30°C. Estimate the number of theoretical stages required.

Table 8.6 Equilibrium Data for Acetic Acid-Water Data at 101.3 kPa

Temperature, °C	Mole fraction of water				Relative volatility
	x	$X = x/(1-x)$	y	$Y = y/(1-y)$	$\alpha_i = Y/X$
118.3	0		0		
110.6	0.1881	0.2317	0.3063	0.4415	$\alpha_1 = 1.905\ 851$
107.8	0.3084	0.4459	0.4467	0.8073	$\alpha_2 = 1.810\ 489$
105.2	0.4498	0.8175	0.5973	1.4832	$\alpha_3 = 1.814\ 312$
104.3	0.5195	1.0812	0.6580	1.9240	$\alpha_4 = 1.779\ 539$
103.5	0.5824	1.3946	0.7112	2.4626	$\alpha_5 = 1.765\ 768$
102.8	0.6750	2.0769	0.7797	3.5393	$\alpha_6 = 1.704\ 09$
102.1	0.7261	2.6510	0.8239	4.6786	$\alpha_7 = 1.764\ 862$
101.5	0.7951	3.8804	0.8671	6.5245	$\alpha_8 = 1.681\ 374$
100.8	0.8556	5.9252	0.9042	9.4384	$\alpha_9 = 1.592\ 925$
100.8	0.8787	7.2440	0.9186	11.2850	$\alpha_{10} = 1.557\ 838$
100.5	0.9134	10.5473	0.9409	15.9205	$\alpha_{11} = 1.509\ 43$
100.2	0.9578	22.6967	0.9708	33.2466	$\alpha_{12} = 1.464\ 821$
100.0	1.0000		1.0000		

(Date source: Table 13.1 of Ref. 8)

Solution

Feed contains 20% water and 80% acetic acid by mass. Molar mass of water is 18 and molar mass of acetic acid is 60. Water is the lighter component.

Mole fraction of water in liquid feed:

$$z_F = x_F = \frac{\left(\frac{20}{18}\right)}{\left(\frac{20}{18}\right) + \left(\frac{80}{60}\right)} = 0.4545$$

Mole fraction of water in distillate:

$$x_D = \frac{\left(\frac{80}{18}\right)}{\left(\frac{80}{18}\right) + \left(\frac{20}{60}\right)} = 0.9302$$

Mole fraction of water in bottom product:

$$x_W = \frac{(50 \times 10^{-6}/18)}{\left(\frac{50 \times 10^{-6}}{18}\right) + \left(\frac{1 - 50 \times 10^{-6}}{60}\right)} = 1.666 \times 10^{-4}$$

***q*-line**

Feed is liquid at 30°C temperature. From *t*-*x*-*y* data, bubble point of feed can be easily determined. t = bubble point temperature of feed when $x = x_F$.

For $x = 0.4545$, corresponding value of $t = 105.14$°C.

Bubble point of feed = 105.14°C

Latent heat of water at 105.14°C, $\lambda_W = 40\ 392$ kJ/kmol

Latent heat of acetic acid at 105.14°C, $\lambda_A = 20\ 096.6$ kJ/kmol

Specific heat at $\frac{30 + 105.14}{2} = 67.57$°C,

$$C_{p\mathrm{W}} = 75.36 \text{ kJ/(kmol} \cdot \text{°C)}$$

$$C_{p\mathrm{A}} = 131.88 \text{ kJ/(kmol} \cdot \text{°C)}$$

Latent heat of feed, $\lambda_\mathrm{F} = 0.4545 \times 40\ 392 + (1 - 0.4545) \times 20\ 096.6$

$= 29\ 320.86$ kJ/kmol

Mean specific heat of feed,

$$C_{pF} = 0.4545 \times 75.36 + (1 - 0.4545) \times 131.88$$

$$= 106.19 \text{ kJ/(kmol} \cdot \text{°C)}$$

$$q = \frac{H_G - H_F}{H_G - H_L} = \frac{(H_G - H_L) + (H_L - H_F)}{H_G - H_L} = \frac{\lambda_F + C_{pF}(t_B - t_F)}{\lambda_F}$$

$$= \frac{29\ 320.86 + 106.19\ (105.14 - 30)}{29\ 320.86} = 1.272$$

Equation of q-line:

$$y = \frac{q}{q-1}x - \frac{z_F}{q-1} \tag{8.13}$$

$$y = \frac{1.272}{0.272}x - \frac{0.4545}{0.272}$$

$$= 4.676\,47x - 1.67$$

q-line starts from $x = y = z_F$ or $x = y = 0.4545$ point.

To find another point, when $y = 0.6$, $x = 0.4854$ using q-line equation.

q-line passes through (0.4854, 0.6) point.

Draw the q-line in the graph. From Fig. (8.3), intercept for the operating line at minimum reflux,

$$0.304 = \frac{x_D}{R_m + 1}$$

$$\frac{0.9302}{R_m + 1} = 0.304$$

$$R_m = 2.1$$

Let $$R = 2\,R_m = 2 \times 2.1 = 4.2$$

$$\frac{x_D}{R + 1} = \frac{0.9302}{4.2 + 1} = 0.1789$$

Fig. 8.3 *McCabe–Thiele Diagram for Acetic Acid-Water System*

On stagewise construction starting from $x = y = x_D$, as shown in Fig. 8.3, up to $x = x_r = 0.02$, number of theoretical or equilibrium stages required is 16 in which 8th theoretical stage from the top is corresponding to feed tray.

Similarly, for the different values of R, number of equilibrium stages, N, required for the same separation are determined and listed in Table 8.7.

Table 8.7 Theoretical Stages (N) for Different Values of R/R_m

R/R_m	2	2.5	3	3.5	4
N	16	15.0	14	14.0	13.5

On increasing the value of R/R_m above 2, decrease in the value of N is very marginal. Hence, $R = 2\ R_m$ is considered to be nearer to the optimum value.

To find the number of equilibrium stages required to increase the purity of bottom product (acetic acid) from $x = 0.02$ to $x_w = 1.666 \times 10^{-4}$, Robinson and Gilliland equation[10] can be used.

For the stripping section, Robinson and Gilliland correlation is

$$N_s = \frac{\log\left[\dfrac{(K'/S' - 1)(x_r'/x_w - 1)}{1/S'(K' - 1)} + 1\right]}{\log\,(K'/S')} + 1 \tag{8.21}$$

where, N_s = Number of ideal stages required from the reference point x_r' to x_W
x_W = Mole fraction of the more volatile component in the residue (bottom product)
x_r' = Mole fraction of more volatile component at the reference point
S' = Slope of the stripping section operating line
K' = Equilibrium constant for more volatile component

For the enriching section, Robinson and Gilliland correlation is

$$N_r = \frac{\log\left[\dfrac{(1 - S) + \dfrac{x_r}{x_d'}(S - K)}{(1 - K)}\right]}{\log\left(\dfrac{S}{K}\right)} - 1 \tag{8.22}$$

where, N_r = Number of ideal stages required from reference point x_r to x_d'
x_d' = Mole fraction of least volatile component in distillate
x_r = Mole fraction of least volatile component at reference point
K = Equilibrium constant for the least volatile component
S = Slope of enriching section operating line

Here, in this example, Eq. (8.21) can be used to find the number of theoretical stages required to purify the bottom product (acetic acid) from $x_r' = 0.02$ to $x_W = 1.666 \times 10^{-4}$.

S' = Slope of stripping section operating line = 1.2
K' = Slope of equilibrium line in the portion from 0.02 to 0. [From Fig. 8.3] = 2.5

$$N_s = \frac{\log\left\{\dfrac{[(2.5/1.2)-1][(0.02/1.666\times10^{-4})-1]}{(1/1.2)(2.5-1)}+1\right\}}{\log\,(2.5/1.2)}+1$$

$$N_s = \frac{\log\,(104.174\ 95)}{\log\,(2.083\ 33)}+1 = 7.33$$

As discussed earlier, second graph with different scale can be drawn for finding theoretical stages in the stripping section below $x_r = 0.02$. Actual plotting also gives additional stages of 7.

Total number of theoretical stages required for desired separation = 16 + 7.33 = 23.33, say 24.

Note: It can be seen that nearly 30% theoretical stages are required to reduce the water content in the bottom product from 0.02 mole fraction to 50 mg/L.

As stated earlier for the separation of pure acetic acid from its dilute aqueous solution containing less than 30% by mass acetic acid, liquid-liquid extraction and azeotropic distillation are more economical than ordinary distillation. Figure 8.4 shows that for separating nearly pure water and pure acetic acid from 25.18% by mass acetic acid solution, 22 + 7 = 29 theoretical stages required for the value of reflux ratio $R = 3.5823$. Comparison between the separation of 80% acetic acid solution and 25.18% acetetic acid solution is given in Table 8.8.

Fig. 8.4 *McCabe–Thiele Diagram for Acetic Acid-Water System*

Table 8.8 Comparison between Two Cases

Feed flow rate, kg/h	% by mass acetic acid in feed	R	N	Top vapour kg /h	λ_{av} kJ/kg	Condenser duty, kW	Reboiler duty, kW
12 000	80	4.2	23	3000	1886.7	8 175.7	9 000
12 000	25.18	3.5813	29	8978	2256.9	25 785.7	29 000

(Condenser duty, $\phi_C = (R + 1)\, D\, \lambda_{AC}$)

Table 8.8 shows that for the separation of dilute solution, about 20% more stages and approximately three times higher condenser duty and reboiler duty are required. Hence, for the separation of dilute aqueous acetic acid solution liquid-liquid extraction or azeotropic distillation is more economical.

(b) Use of Open Steam in Distillation While distilling an aqueous solution, if non-aqueous component is more volatile then water is removed as residue product. In such a case, instead of using separate reboiler at bottom, heat required can be provided by admission of steam directly to the bottom of tower. For the same value of reflux ratio and the given degree of separation, use of open steam requires more number of trays or equilibrium stages. However, it is usually cheaper than the separate reboiler system as it avoids fixed cost and cleaning cost of reboiler. It also facilitates the use of lower pressure steam as heating medium but with the use of open steam, condensate cannot be recycled back to boiler feed tank.

Overall material balance,

$$F + S = D + W \tag{8.23}$$

where, S = Molar flow rate of saturated steam entering at bottom, kmol/h

More volatile component balance

$$Fz_F = D\, x_D + W\, x_W \tag{8.6}$$

In this case stripping section operating line starts from ($x = x_W$, $y = 0$) point and has a slope equal to $\bar{L}/S$ or W/S.

where, $\bar{L}$ = Molar flow rate of liquid in stripping section, kmol/h
= W, molar flow rate of residue, kmol/h

If superheated steam is used as open steam, then S and $\bar{L}$ are calculated by the following equations:

$$S = S_s\left(1 + \frac{H_s - H_{\text{sat}}}{\lambda M}\right) \tag{8.24}$$

$$\bar{L} = S - S_s + W \tag{8.25}$$

where, S_s = Molar flow rate of superheated steam, kmol/h
H_s = Enthalpy of superheated steam, kJ/kmol
H_{sat} = Enthalpy of saturated steam, kJ/kmol
λ = Latent heat of vaporisation at operating temperature, kJ/kg
M = Molar mass of water = 18.0153 kg/kmol

Design of distillation column with open steam can be better explained with following example.

Example 8.6 Aqueous solution of ethanol contains 6% by mass ethanol. It is to be concentrated in the distillation column to 30% ethanol (by mass). Open steam, saturated at 80 kPa g pressure will be used for the distillation

column. Feed flow rate is 5000 kg/h and it is saturated liquid at its bubble point. Residue should not contain more than 0.02 mass per cent ethanol. Sparging steam flow is 0.2 kg per kg of feed. Calculate the number of theoretical stages required for the desired separation.

Solution

Basis: 5000 kg/h feed

$$F = \frac{5000 \times 0.06}{46} + \frac{5000 \times 0.94}{18}$$

$$= 6.52 + 261.11 = 267.63 \text{ kmol/h}$$

$$S = (0.2 \times 5000)/18 = 55.556 \text{ kmol/h}$$

$$F + S = D + W \tag{8.23}$$

$$D + W = 323.186 \text{ kmol/h}$$

$$z_F = \frac{(0.06/46)}{(0.06/46) + (0.94/18)} = 0.024\ 37$$

$$x_D = \frac{(0.3/46)}{(0.3/46) + (0.7/18)} = 0.1436$$

$$x_W = \frac{0.0002/46}{(0.0002/46) + (0.9998/18)} = 7.827 \times 10^{-5}$$

$$Fz_F = D\,x_D + W\,x_W \tag{8.6}$$

$$267.63 \times 0.024\ 37 = 0.1436\ D + 7.827\ 10^{-5}\ W$$

$$6.522 = 0.1436\ D + 7.827 \times 10^{-5}\ (323.186 - D)$$

$$D = 45.266 \text{ kmol/h}$$

$$W = 277.92 \text{ kmol/h}$$

Feed is saturated liquid at its bubble point. Hence, $q = 1$

$$\bar{L} = L + Fq = L + F = W$$

$$\bar{G} = G + F(q - 1) = G$$

$$S = \bar{G} = G = 55.556 \text{ kmol/h}$$

$$G = (R + 1)\ D$$

$$55.556 = (R + 1)\ 45.266$$

$$R = 0.2273$$

Table 8.9 Equilibrium Data for Ethanol-Water System at 101.3 kPa

x	0.019	0.0721	0.0966	0.1238	0.1661
y	0.17	0.3891	0.4375	0.4704	0.5089

(Table 13.1 of Ref. 8)

Intercept of enriching section

operating line = $x_D/(R + 1) = 0.1436/(0.2273 + 1) = 0.117$

Slope of stripping section operating line = $\bar{L}/S = W/S = \dfrac{277.92}{55.556} = 5$

Stripping section operating line starts from (x_W, 0) point.
From Fig. 8.5, number of equilibrium stages required is $N = 7$.

Fig. 8.5 *McCabe–Thiele Diagram for Steam Stripping of Ethanol-Water Solution*

(c) Ponchon–Savarit Method This method is more rigorous than McCabe–Thile method. This method requires detailed enthalpy data and it is particularly applied to a binary system in which heat of solution is significant. This method is explained by following illustration.

Example 8.7 Determine the minimum reflux ratio and number of theoretical stages required for the distillation column of ammonia-water absorption refrigeration plant based on following data. Also find the heat duties of overhead condenser (ϕ_C) and reboiler (ϕ_B) for the given value of reflux ratio R.
Data: (Example 8.4 of Ref. 14)

(a) Feed:
Feed is strong aqueous (SA) ammonia solution which enters the distillation column at 102.5°C.
Enthalpy of feed = 325.2 kJ/kg
Ammonia in SA = 36.3% (by mass)
Feed flow rate = 7523.28 kg/h

(b) Distillate: 100% pure ammonia in the form of saturated liquid at 40°C

(c) Residue: Weak aqueous (WA) solution of ammonia, having 29.8% by mass

(d) Operating pressure of column = 1555.5 kPa a (assumed constant throughout the column)
(e) Cooling water at 32°C is used as cooling medium in overhead condenser and saturated steam at 3 bar a is used as heating medium in generator (reboiler).
(f) *H–x–y* data (Fig. 5.19, of Ref. 8)

Table 8.10 Enthalpies of Saturated Liquid and Vapour Mixtures

x, y	H_L, kJ/kmol	H_G, kJ/kmol
0	15 480.7	50 310.7
0.15	10 887.3	48 194.8
0.30	7 519.0	45 665.1
0.40	5 808.0	43 985.9
0.45	5 614.9	42 997.0
0.50	4 900.0	42 078.8
0.60	4 523.2	40 024.9
0.70	5 189.7	38 234.3
0.80	6 449.3	36 122.0
0.90	7 181.6	33 334.7
1.00	9 159.6	27 832.4

[Reference states: Enthalpies of pure saturated liquids at triple points (0.01°C for water and –77.8°C for ammonia) = 0 kJ/kmol]

where, H_L = Enthalpy of saturated liquid at 1555.5 kPa a pressure, kJ/kmol
H_G = Enthalpy of saturated vapour at 1555.5 kPa a pressure, kJ/kmol
x, y = Mole fraction of ammonia in ammonia-water liquid solution and in ammonia–steam mixture, respectively.

(g) VLE data at p_t = 1555.5 kPa a
(Table 3.23 and Table 3.24 of Ref. 8)

Table 8.11 Equilibrium Data for Ammonia-Water System

x	y
0	0
0.30	0.9120
0.35	0.9456
0.40	0.9690
0.45	0.9800
0.50	0.9880
0.60	0.9950
0.75	0.9980
1.00	1.0000

(h) Actual or operating reflux ratio, $R = 0.3137$ which is nearly equal to 0.08 kg per kg ammonia (as NH_3) in the feed.

Solution

Material balance

Feed flow rate, $\dot{m}_F = 7523.28$ kg/h

$$x_F = \frac{(0.363/17)}{(0.363/17) + (0.637/18)} = 0.3763$$

Average molar mass of feed

$$M_{av} = \Sigma M_i x_i$$

$$M_{av} = 17\,(0.3763) + 18\,(1 - 0.3763) = 17.6237 \text{ kg/kmol}$$

$$F = \frac{7523.28}{17.6237} = 426.88 \text{ kmol/h}$$

$$x_W = \frac{0.298/17}{(0.298/17) + (0.702/18)} = 0.31$$

$$x_D \cong 1$$

$$F = D + W \text{ or } 426.88 = D + W \qquad \text{(a)}$$

Ammonia balance

$$Fx_F = D\,x_D + W\,x_W$$

$$426.88 \times 0.3763 = D + 0.31\,W \qquad \text{(b)}$$

Solving Eq. (a) and (b),

$$D = 41.0176 \text{ kmol/h}, \; W = 385.8624 \text{ kmol/h}$$

Minimum reflux ratio, R_m

Refer to Fig. 8.6(a) and (b). To find R_m, in $H - x, y$ diagram, draw the tie–lines. A line connecting equilibrium values of x and y is called a tie line. Extend the tie lines and allow to intersect $x = x_D$ and $x = x_W$ lines. Intersection point with $x = x_D$ is represented as ΔD and with $x = x_W$ as ΔW. Extended tie lines which give the farthest locations of ΔD_m and ΔW_m and are corresponding to minimum reflux ratio. But if x, y equilibrium curve is everywhere concave downward, then a tie line which when extended passes through F corresponds to minimum amount of reflux.

Here x, y equilibrium curve is everywhere concave downward. Hence, the minimum reflux ratio is established by the tie line which when extended passes through F.

Mark the point F in H–x, y diagram. $x_F = 0.3763$

$H_F = 325.2 \times 17.6237 = 5731.23$ kJ/kmol

Find the tie line which when extended, passes through point F. One method is, draw the two nearest tie lines from the given equilibrium data. Here, these two tie lines are (i) $x = 0.35$ and $y = 0.9456$, and (ii) $x = 0.4$ and $y = 0.969$. Then draw the line having average slope of two tie lines and passes through point F. Extend this tie line and allow it to intersect $x = x_D$ line. Intersection point is ΔD_m.

From Fig. 8.6 at ΔD_m, $H_m = 31\,818$ kJ/kmol.

$$R_m = \frac{H_m - H_{G1}}{H_{G1} - H_{L0}}$$

(a) Enthalpy-Concentration Diagram for Aqueous Ammonia Solution

(b) Theoretical Stage Calculations for Distillation of Aqueous Ammonia Solution

Fig. 8.6 *Separation of Ammonia from Aqueous Solution*

$$R_m = \frac{31\,818 - 27\,832.4}{27\,832.4 - 9159.6} = 0.213\,44$$

Actual R is 0.3137. Thus actual reflux ratio is 1.47 times the minimum reflux ratio.

For
$$R = 0.3137$$
$$= \frac{H - H_{G1}}{H_{G1} - H_{L0}}$$
$$0.3137 = \frac{H - 27\,832.4}{27\,832.4 - 9159.6}$$
$$H = 33\,690 \text{ kJ/kmol}$$

Mark ΔD on $x = x_D$ line corresponding to the value of H.

$$FH_F = DH + WH'$$
$$426.88 \times 5731.23 = 41.0176 \times 33\,690 + 385.8624\,H'$$
$$H' = 2759.18 \text{ kJ/kmol}$$

Mark ΔW on $x = x_W$ line corresponding to value of H'. ΔD, F and ΔW are on the same line. In Fig. 8.6(a), ΔD at $x_D = 1$, $H = 33\,690$ kJ/kmol and ΔW at $x_W = 0.31$, $H' = 2759.18$ kJ/kmol are plotted. Auxiliary random lines from the Δ points are drawn and allowed to intersect the saturated vapour and saturated liquid curves at values of y and x, respectively. These (x, y) points are the points of operating curves. Operating curves are plotted in Fig. 8.6(b). Stagewise construction from x_D to x_W between equilibrium curve and operating curve gives number of theoretical stages required for the desired separation.

Table 8.12 x, y Values for Enriching Section Operating Curve and Stripping Section Operating Curve

Enriching		Stripping	
x	y	x	y
1.00	1.000	0.381	0.942
0.95	0.990	0.373	0.900
0.90	0.982	0.360	0.800
0.80	0.970	0.350	0.700
0.70	0.962	0.339	0.600
0.60	0.955	0.329	0.500
0.50	0.949	0.324	0.400
0.381	0.942	0.310	0.310

Here stagewise construction is started from $x_D = 0.999\,72$ to $x_W = 0.31$. Total number of theoretical stages required (as shown in Fig. 8.6(b)) is 5.36.

$$N = 5.36.$$
$$H = H_D + \frac{\phi_C}{D} = 9159.6 + \frac{\phi_C}{41.0176}$$

$$\phi_C = 1006\ 178.1 \text{ kJ/h} \equiv 279.493 \text{ kW - condenser duty}$$

$$H' = H_W - \frac{\phi_B}{W} = 7347.86 - \frac{\phi_B}{385.8624}$$

$$\phi_B = 1770\ 599.1 \text{ kJ/h} \equiv 491.83 \text{ kW - reboiler duty}$$

Methods Valid for Multicomponent Distillation To determine by number of theoretical stages or equilibrium stages required for desired separation in multicomponent distillation many methods are available. None of them is totally reliable. Hence, for a new case of distillation, it is recommended to use more than one method for the determination of theoretical stages. Also it is better to design the distillation column, first for pilot plant and then modify the design based on the results of pilot plant column rather than designing the column directly for the commercial scale plant. Methods available for multicomponent distillation can be broadly divided into two groups:

1. Short cut methods: FUG method, FUE method, Smith–Brinkley method
2. Rigorous methods: Lewis Matheson method, Thiele–Geddes method, Relaxation methods, Tridiagonal Matrix method, Linear Algebra methods

FUG Method (Fenskey–Underwood–Gilliland's Method)[8] It is the most widely used short-cut method for determining number of equilibrium stages for multicomponent distillation.

In this method, first minimum reflux ratio R_m is determined by Underwood's method, which was explained in the Section 8.4.4.

Then Fenskey's equation [Eq. (8.26)] is used to find the minimum number of theoretical stages required for the desired separation.

$$N_m = \frac{\log\left[\left(\dfrac{x_{LK}}{x_{HK}}\right)_d \left(\dfrac{x_{HK}}{x_{LK}}\right)_b\right]}{\log \alpha_{LK}} \tag{8.26}$$

where, α_{LK} = Average relative volatility of light key with respect to heavy key

$(x_{LK}, x_{HK})_d$ = Mole fractions of light key and heavy keys in distillate

$(x_{LK}, x_{HK})_b$ = Mole fractions of light key and heavy keys in residue

If there is a wide difference in the values of α_{LK} at the topmost temperature and at bottommost temperature, then use Eq. (8.27). Fenskey's equation in such cases gives lesser value of N_m than actual N_m required, if α_{LK} is determined by Eq. (8.8).

$$\alpha_{LK} = (\alpha_{LK,\ \text{top}} \times \alpha_{LK,\ \text{bottom}})^{1/2} \tag{8.8}$$

$$\alpha_{LK} = (\alpha_{LK,\ \text{top}} \times \alpha_{LK,\ \text{feed}} \times \alpha_{LK,\ \text{bottom}})^{1/3} \tag{8.27}$$

where, $\alpha_{LK,\ \text{feed}}$ = relative volatility of light key with respect to heavy key at the bubble point of feed.

After finding the values of R_m and N_m, number of theoretical stages required or number of equilibrium stages required for desired separation can be determined by Gilliland's correlation. It was originally presented in terms of graph. Many attempts are

made to represent Gilliland's correlation empirically. One of the empirical equations for Gilliland's correlation is developed by Malokanov[8].

$$f(N) = \frac{N - N_m}{N + 1} = 1 - \exp\left[\left(\frac{1 + 54.4\psi}{11 + 117.2\psi}\right)\left(\frac{\psi - 1}{\psi^{0.5}}\right)\right] \tag{8.28}$$

where, $$\psi = \frac{R - R_m}{R + 1} \tag{8.29}$$

Figure 8.7 is the plot representing Gilliland correlation.

Fig. 8.7 *Gilliland Correlation*
(Reproduced from Perry's Chemical Engineeris' Handbook, 6th Ed., McGraw-Hill, USA)

Example 8.8 Determine the number of theoretical stages required for desired separation by FUG method for the case given in Example 8.4.

Solution

Minimum reflux ratio determined by Underwood's method in the Example 8.4. $R_m = 1.4509$

Using Fenskey's equation to find the minimum number of theoretical stages required for desired separation, N_m for total reflux.

$$N_m = \frac{\log\left[\left(\frac{x_{LK}}{x_{HK}}\right)_d \left(\frac{x_{HK}}{x_{LK}}\right)_b\right]}{\log \alpha_{LK}} \tag{8.26}$$

$$N_m = \frac{\log\left[\left(\frac{0.95}{0.05}\right)\left(\frac{0.416}{0.163}\right)\right]}{\log (2.567)}$$

$$N_m = 4.117$$

Using Eqs. (8.28) and (8.29), Table 8.13 is prepared.

Table 8.13 Values of *N* for Different Values of *R*

R	ψ	*f*(*N*)	*N*
1.7	0.092 26	0.5616	10.67
2.0	0.183 03	0.4752	8.75
2.5	0.299 74	0.3811	7.27
3.0	0.387 30	0.3198	6.52
3.5	0.455 36	0.2761	6.07
4.0	0.509 82	0.2433	5.76

Above the value of reflux ratio $R = 3$, there is a tapering in values of N. Hence, the optimum reflux ratio is nearer to $R = 3$.

Using Eq. (8.29), $\psi = 0.3873$. Substituting ψ in Eq. (8.28),

Number of theoretical stages required for the desired separation $N = 6.52$.

For $\psi = 0.3873$;

Read $$f(N) = \frac{N - N_m}{N + 1} = 0.32 \text{ from Fig. 8.7}$$

$$N = 6.525$$

Example 8.9 A fatty acid mixture contains 11% palmitic acid, 4% stearic acid, 28.5% oleic acid and rest linoleic acid (by mass). It is to be distilled to separate in pure components in distillation columns operating at 2 Torr absolute at top.

Fig. 8.8(a) *Separation of Fatty Acids*

Table 8.14 Pressure vs Boiling Point Data of Fatty Acids

Absolute Pressure		Boiling Point, °C			
kPa	Torr	Palmitic acid	Stearic acid	Oleic acid	Linoleic acid
0.133	1	167.4	183.6	177.6	178.5
0.267	2	179.0	195.9	189.5	190.1
0.533	4	192.2	209.2	202.6	202.8
1.07	8	206.1	224.1	217.0	216.9
2.13	16	221.5	240.0	232.9	232.6
4.27	32	238.4	257.1	250.6	250.0
8.53	64	257.1	276.8	270.3	269.7
17.1	128	278.7	299.7	292.5	291.9

(Contd.)

Table 8.14 (*Contd.*)

Absolute Pressure		Boiling Point, °C			
kPa	Torr	Palmitic acid	Stearic acid	Oleic acid	Linoleic acid
34.1	256	303.6	324.8	317.7	317.2
68.3	512	332.6	355.2	346.5	346.5
101.325	760	351.5	376.1	364.9	365.2

(Table 2 of Ref. 15)

Table 8.15 Chemical Formulas and Molar Masses of Fatty Acids

Fatty acid	Chemical formula	Molar mass	Melting point, °C
Palmitic acid	$C_{16}H_{32}O_2$	256.42	63.1
Stearic acid	$C_{18}H_{36}O_2$	284.48	69.6
Oleic acid	$C_{18}H_{34}O_2$	282.46	16.3
Linoleic acid	$C_{18}H_{32}O_2$	280.45	–5.0

Feed is a saturated liquid. Determine the number of theoretical stages required for the desired separation.

Solution

Boiling points of oleic acid and linoleic acid are very close. Hence, they are near impossible or very difficult to separate by distillation. Study of melting points of fatty acid (Table 8.15) suggests that fractional crystallization can be explored for separation of oleic and linoleic acids. It is planned to separate the following three products from the feed mixture.

Products	*Purity in % by mole*
1. Palmitic acid	99%
2. Mixture of linoleic and oleic acids	97% (Combinedly)
3. Stearic acid	95%

There are three (N') products. Hence, two distillation columns ($N' - 1$) are required as discussed in Section 8.4.1. First column will separate 99% by mole pure palmitic acid as top product and remaining as bottom product. Second column will give 97% by mole pure (combinedly) mixture of linoleic and oleic acid as the top product and 95% by mole stearic acid as the bottom product. Both columns will be operated under vacuum and operating pressure at the top of both columns will be 2 Torr a.

Determination of number of theoretical stages for first distillation column This column operates as a multicomponent (3) distillation column. Top product is nearly pure palmitic acid. At 179°C vapour pressure of palmitic acid is 2 Torr (Table 8.14). Hence, top temperature of column will be very close to 179°C. Let operating pressure at the base of first column be 30 Torr a. (This is decided based on trial calculations). Bottommost temperature of distillation column is bubble point temperature of residue. To carry out the material balance and to find the feed composition in mole %, let the feed flow rate be 1000 kg/h (basis).

Mass flow rate of feed, $\dot{m}_F$ = 1000 kg/h

Table 8.16 Feed Composition

Component	mass %	Molar mass	kmol/h	mole %
Palmitic acid	11	256.42	0.428 98	11.94
Stearic acid	4	284.48	0.140 61	3.91
Oleic acid	28.5	282.46	1.008 99	28.08
Linoleic acid	56.5	280.45	2.014 62	56.07
Total	100.0		3.593 20	100.00

Average molar mass of feed, $M_F = 1000/3.5932$
$= 278.3$

Table 8.17 Composition of Distillate and Residue of First Column

Component	Distillate, *D*			Bottom, *W*		
	kmol/h	kg/h	mole %	kmol/h	kg/h	mole %
Palmitic acid	0.3971	101.824	99.0	0.031 88	8.175	1.00
Stearic acid	—	—	—	0.140 61	40.001	4.40
Oleic acid	0.002	0.565	0.5	1.006 99	284.434	31.55
Linoleic acid	0.002	0.561	0.5	2.012 62	564.439	63.05
Total	0.4011	102.950	100.0	3.192 10	897.049	100.00

Vapour pressures of oleic acid and linoleic acid are very close. Hencte, in distillation calculations they are considered as one product. For first column palmitic acid is the light key and mixture of oleic acid-linoleic acid is the heavy key.
Component balance around first distillation column: Palmitic acid balance.

$$0.1194\ F = 0.99\ D + 0.01\ W$$

Let mole % of palmitic acid in residue = 1%. (refer to Table 8.17)

$$0.1194 \times 3.5932 = 0.99\ D + 0.01\ (3.5932 - D)$$

$$D = 0.4011 \text{ kmol/h}, W = 3.1921 \text{ kmol/h}$$

At bubble point temperature of residue,

$$\Sigma x_{wi}\, p_{vi} = p_t = 30 \text{ Torr}$$

Trial temperature = 250°C

Table 8.18 Vapour Pressures as Read from Fig. 8.8(b)

Component	Vapour pressure, Torr	
	At 179°C	At 250°C
Palmitic acid	2	51.71
Stearic acid	0.714	23.29
Linoleic acid/Oleic acid	0.903	31.43

Fig. 8.8(b) *Vapour Pressures of Fatty Acids based on Table 8.14*

Using Fig. 8.8(b) and Table 8.17,
$0.01 \times 51.71 + 0.044 \times 23.29 + (0.3155 + 0.6305) \times 31.43 = 31.27$ Torr $\cong 30$ Torr
Hence, bottommost temperature of first distillation column $\cong 250°C$

Table 8.19 Relative Volatility Calculations

Component	α_{top}	α_{bottom}	$\alpha_{av} = (\alpha_{top} \cdot \alpha_{bottom})^{0.5}$
Palmitic acid (LK)	2.2148	1.6452	1.9089
Stearic acid	0.7907	0.741	0.7654
Linoleic acid/Oleic acid (HK)	1	1	1

Here FUG method is used to find the number of theoretical stages required for desired separation.
Minimum number of theoretical stages required by using Fenskey's method, N_m:

$$N_m = \frac{\log\left[\left(\frac{x_{LK}}{x_{HK}}\right)_d \left(\frac{x_{HK}}{x_{LK}}\right)_b\right]}{\log \alpha_{LK}} \tag{8.26}$$

$$= \frac{\log\left[\left(\frac{0.99}{0.01}\right)\left(\frac{0.3155 + 0.6305}{0.01}\right)\right]}{\log (1.9089)} = 14.144$$

Underwood's method

$$\sum \frac{\alpha_i x_{if}}{\alpha_i - \vartheta} = 1 - q \tag{8.15}$$

Feed is saturated liquid. Hence, $q = 1$

$$\frac{1.9089 \times 0.1194}{1.9089 - \vartheta} + \frac{0.7654 \times 0.0391}{0.7654 - \vartheta} + \frac{1 \times (0.2808 + 0.5607)}{1 - \vartheta} = 1 - 1 = 0$$

Solving by Mathcad,
For $\vartheta = 1.719\ 32$, $f(\vartheta) = 0.001 \cong 0$
Also, $\vartheta \in \{1, \ldots, 1.9089\}$

$$\sum \frac{\propto_i x_{id}}{\propto_i - \vartheta} = R_m + 1$$

$$\frac{1.9089 \times 0.99}{1.9089 - \vartheta} + \frac{1 \times (0.005 + 0.005)}{1 - \vartheta} = R_m + 1$$

$$R_m = 8.9545$$

Gilliland's correlations; Eqs. (8.28) and (8.29):

Table 8.20 Evaluation of Parameters

R	ψ	$f(N)$	N
10	0.095	0.5586	33.31
12	0.234	0.4318	25.65
15	0.378	0.326	21.47
17	0.447	0.2813	20.07

Let, $R = 15$, for which $N = 21.47 \cong 22$

For $$\psi = \frac{R - R_m}{R + 1} = \frac{15 - 8.9545}{15 + 1} = 0.378$$

From Fig. 8.7 $$\frac{N - N_m}{N + 1} = 0.3062 \text{ or } N = 20.83$$

FUG method does not give information about feed tray location. Feed tray location can be determined by using following equation (Kirkbride equation)[10]:

$$\log\left(\frac{N_r}{N_s}\right) = 0.206 \log\left[\left(\frac{W}{D}\right)\left(\frac{x_{HK}}{x_{LK}}\right)_f \left(\frac{x_{bLK}}{x_{dHK}}\right)^2\right] \tag{8.30}$$

where, N_r = Number of stages above the feed
N_s = Number of stages below the feed

$$\log\left(\frac{N_r}{N_s}\right) = 0.206 \log\left[\left(\frac{3.1921}{0.4011}\right)\left(\frac{0.2808 + 0.5607}{0.1194}\right)\left(\frac{0.01}{0.01}\right)^2\right]$$

$$\frac{N_r}{N_s} = 2.292$$

$$N_r + N_s = 22$$

$$3.292\, N_s = 22$$

$N_s = 6.683 \cong 7, \quad N_r = 15$

Hence, seventh stage from bottom will be the feed stage.

Second column acts as a binary distillation column. For second distillation column $W = F'$ (as shown in Fig. 8.7(a)), where F' is molar flow rate of feed to second distillation column.

Average molar mass of feed, $M_{F'} = 897.049/3.1921$
$= 281.02$

Linoleic-Oleic acids balance around second column:

$$F' \times (0.3155 + 0.6305) = 0.97\ D' + 0.05\ W'$$
$$3.1921 \times 0.946 = 0.97\ D' + 0.05\ (3.1921 - D')$$
$$2.860\ 12 = 0.92\ D'$$
$$D' = 3.1088 \text{ kmol/h}, \ W' = 0.0833 \text{ kmol/h}$$

Table 8.21 Composition of Distillate and Residue of Second Column

Component	Distillate, D'			Bottom, W'		
	kmol/h	kg/h	mole %	kmol/h	kg/h	mole %
Palmitic acid	0.0319	8.180	1.026	—	–	—
Stearic acid	0.0615	17.496	1.978	0.0791	22.502	95.00
Oleic acid	1.0056	284.042	32.347	0.0014	0.395	1.68
Linoleic acid	2.0098	563.648	64.649	0.0028	0.785	3.36
Total	3.1088	873.366	100.000	0.0833	23.682	100.00

Operating pressure at the top of second column = 2 Torr a.

Topmost temperature of distillation column is dew point temperature of overhead vapour.

At dew point temperature

$$\sum \frac{y_i p_t}{p_{vi}} = 1$$

Let topmost temperature = 190.1°C (Trial) $\quad y_i = x_{Di}$

At 190.1°C temperature, from Fig. 8.7(b) vapour pressure of components are as follows.

Table 8.22 Vapour Pressures as Read from Fig. 8.8(b)

Component	Vapour pressure, Torr	
	At 190°C	At 268.2°C
Linoleic/oleic acid	2	63.5
Stearic acid	1.64	50

Table 8.23 Relative Volatilities Calculations

Component	α_{top}	α_{bottom}	$\alpha_{av} = (\alpha_{top} \cdot \alpha_{bottom})^{0.5}$
Linoleic/oleic acid	1.22	1.27	1.245
Stearic acid	1	1	1

For the calculations of number of theoretical stages required for desired separation feed to second column can be approximated as binary system.

$$F' = 3.1921\ \frac{\text{kmol}}{\text{h}},\quad D' = 3.1088\ \frac{\text{kmol}}{\text{h}},\quad W' = 0.0833\ \frac{\text{kmol}}{\text{h}},$$

$$z_F = 0.946,\qquad x_D = 0.97,\qquad x_W = 0.05$$

Vapour-liquid equilibrium data can be determined by using following equation:

$$y = \frac{\alpha x}{1 + (\alpha - 1)x}$$

Table 8.24 VLE Data for Second Column

x	y
0	0
0.2	0.2374
0.4	0.4536
0.6	0.6513
0.8	0.8328
1.0	1.0000

Feed is saturated liquid; $q = 1$.

Fig. 8.8(c) *McCabe–Thiele Diagram for Second Column*

From Fig. 8.7(c)

$$\frac{x_D}{R_m + 1} = 0.4301$$

$$R_m = 1.2553$$

Let $$R = 3 \times R_m = 3.7659$$

$$\frac{x_D}{R + 1} = \frac{0.97}{4.7659} = 0.203\ 53$$

For R = 3.7659, number of theoretical stages required for desired separation, N = 33.
Feed tray location = 4th equilibrium stage from top

Rigorous Methods Rigorous or detailed methods available for multicomponent distillation for finding or verifying number of theoretical stages required for desired separation are as follows.

(a) Lewis–Matheson method
(b) Theile–Geddes method
(c) Relaxation methods
(d) Equation tearing procedures using tridiagonal matrix algorithm.

Among these methods, only Lewis–Matheson method calculates the number of theoretical stages. In other methods, number of equilibrium stages required above and below the feed point must be specified by the user. In other methods, for the given number of theoretical stages, product compositions are determined.

(a) Lewis–Matheson Method[13]

In this method, constant molar overflow is assumed. In other words, it is assumed that for each stage, molar latent heat of vaporization of liquid phase is equal to latent heat of condensation of vapour phase. Hence, moles of liquid vaporized over each theoretical stage is equal to moles of vapour condensed on that stage. This implies that, molar flow rates of liquid and vapour in enriching section (L and V) and also in stripping section ($\bar{L}$ and $\bar{V}$) remain constant. To use this method following information must be specified:

(i) Feed flow rate, conditions and compositions
(ii) One product flow (D or W)
(iii) Product compositions or distribution of key and non-key components
(iv) Reflux ratio
(v) Column pressure

Stepwise Lewis–Matheson method is as given below:

- Assuming that total condenser is used. $L = RD$ and $V = (R + 1)D$. Determine the dew point temperature of overhead vapour by using the equation,

$$\sum \frac{y_{Ni}\varphi_i p_t}{\gamma_i p_{vi}} = 1 \text{ (For non-ideal system)} \tag{8.3}$$

or $$\sum \frac{y_{Ni} p_t}{p_{vi}} = 1 \text{ (For ideal vapour-liquid equilibrium)} \qquad (8.1)$$

where, $y_{Ni} = x_{Di} = x_{oi}$

y_{Ni} = Mole fraction of component i in vapour phase leaving the Nth stage

x_{Di} = Mole fraction of component i in distillate

x_{oi} = Mole fraction of component i in reflux

p_t = Operating pressure at top of distillation column, kPa a

p_{vi} = Vapour pressure of component i at dew point temperature of overhead vapour, kPa a

φ_i = Fugacity coefficient of component i in vapour phase

γ_i = Activity coefficient of component i in liquid phase

Same calculations (dew point temperature calculation) also give the value of x_{Ni}

$$x_{Ni} = \frac{y_{Ni} p_t}{p_{vi}} \text{ (For ideal VLE) – Raoult's Law} \qquad (8.1)$$

where x_{Ni} = mole fraction of component i in liquid phase leaving the Nth tray.

Composition of vapour leaving the $(N - 1)^{th}$ tray can be determined by a material balance around the top section as shown in Fig. (8.9).

$$V\, y_{(N-1)i} = L x_{Ni} + D x_{Di} \qquad (8.31)$$

Fig. 8.9(a) *Material Balance of Top Tray* **Fig. 8.9(b)** *Material Balance for (N–1)th Tray*

By using above equation $y_{(N-1)i}$ is determined. After finding $y_{(N-1)i}$, i.e., composition of vapour leaving the $(N - 1)^{th}$ tray, find the dew point of this vapour.

$$\sum \frac{y_{(N-1)_i} p_t}{p_{vi}} = 1 \text{ (For ideal VLE)} \qquad (8.32)$$

Same calculations also give the values of $x_{(N-1)i}$, composition of liquid phase leaving the $(N - 1)^{th}$ tray.

Material balance around $(N-1)^{th}$ tray gives composition of vapour leaving the $(N-2)^{th}$ tray.

$$Vy_{(N-2)i} = Vy_{(N-1)i} + Lx_{(N-1)i} - Lx_{Ni} \tag{8.33}$$

These calculations (i.e., dew point temperature calculations and material balance calculations) are repeated up to the feed tray.

- *Feed Tray Identification* To identify the feed tray following conditions must be satisfied.

  If feed is liquid then mole fraction ratio or molar ratio of light key to heavy key in the feed should lie in between the same ratios of the liquid leaving the feed tray and of the liquid leaving the tray, next above the feed tray.

$$\left(\frac{x_{LK}}{x_{HK}}\right)_{\text{Feed tray}} \le \left(\frac{x_{LK}}{x_{HK}}\right)_{\text{Feed}} \le \left(\frac{x_{LK}}{x_{HK}}\right)_{\text{Tray above the feed tray}}$$

or

$$\left(\frac{x_{LK}}{x_{HK}}\right)_{N_F} \le \left(\frac{x_{LK}}{x_{HK}}\right)_{\text{Feed}} \le \left(\frac{x_{LK}}{x_{HK}}\right)_{N_F+1} \tag{8.34}$$

  If the feed is a mixture of liquid and vapour then also the above condition is valid. However, the difference is only liquid phase molar ratio is to be considered for satisfying this condition.

  If feed is vapour then condition is

$$\left(\frac{x_{LK}}{x_{HK}}\right)_{\text{Tray beneath feed tray}} \le \left(\frac{y_{LK}/K_{LK}}{y_{HK}/K_{HK}}\right)_{\text{Feed}} \le \left(\frac{x_{LK}}{x_{HK}}\right)_{N_F}$$

or

$$\left(\frac{x_{LK}}{x_{HK}}\right)_{N_F-1} \le \left(\frac{y_{LK}/K_{LK}}{y_{HK}/K_{HK}}\right)_{\text{Feed}} \le \left(\frac{x_{LK}}{x_{HK}}\right)_{N_F} \tag{8.35}$$

  In a distillation column, any tray from top to bottom can be the feed tray. Hence, in tray-to-tray calculations from top, this condition must be checked for each tray.

- *For Stripping Section:*

  Molar flow rate of liquid, $\bar{L} = L + qF$

  Molar flow rate of vapour, $\bar{V} = \bar{L} - W$

  where, W = Molar flow rate of residue

  Calculate the bubble point temperature of residue using either Eq. (8.3) or Eq. (8.1).

  Same calculations also give the values of y_{Wi} composition of vapour leaving the reboiler.

$$y_{Wi} = \frac{x_{Wi}p_{vi}}{p_t'} \tag{8.36}$$

  The material balance around the reboiler gives

$$\bar{L}x_{1i} = \bar{V}y_{Wi} + Wx_{Wi} \tag{8.37}$$

Calculate the bubble point temperature of liquid leaving the first tray

$$\sum \frac{x_{1i} p_{vi}}{p_t'} = 1 \text{ (For ideal VLE)} \tag{8.38}$$

where, p_t' = Pressure in reboiler
$= p_t + \Delta p$

Same calculations also give y_{1i}, composition of vapour leaving the first tray. Then material balance around 1st tray gives x_{2i}.

$$\bar{L}x_{2i} = \bar{L}x_{1i} + \bar{V}y_{1i} - \bar{V}y_{Wi} \tag{8.39}$$

x_{2i} is the composition of liquid leaving the second tray. These iterative calculations are repeated from bottom to feed tray. For the liquid leaving the feed tray x_{LK}/x_{HK} ratios, one obtained from enriching section calculations and other obtained from stripping section calculations, must be very close.

- Normally, in the first trial calculations, it happens that on getting close values of the ratios x_{LK}/x_{HK}, one from enriching section and other from stripping section, mole fractions of the components of liquid leaving the feed tray, one obtained from top section and other from bottom section do not match. If they are different then it is called mismatch ".

Fig. 8.10 *Material Balance around Reboiler*

If composition of liquid phase, leaving the feed tray, obtained from top section calculations and the same from bottom section calculations are different, then to match them one has to change product distribution and product rates. In other words one has to change x_{Di}, x_{Wi}, D and W. For the new values, entire calculations must be repeated until the entire composition of liquid leaving the feed tray calculated from top and bottom closely match.

In the first trial calculations, if composition of liquid leaving the feed tray from top to down calculations and the same from bottom to up calculations are not matching, then Smith suggests the following method to make the change in product composition for second trial calculations[12]:

Change in moles of component i in distillate for second trial calculations, Δd_i is calculated by following equation:

$$\Delta d_i = \frac{(x_{FB} - x_{FD})_i}{\left(\frac{x_{FD}}{d}\right)_i + \left(\frac{x_{FB}}{b}\right)_i} \tag{8.40}$$

where, x_{FBi} = Mole fraction of component i in the liquid from feed tray as found from the bottom to up calculations

x_{FDi} = Mole fraction of component i in the liquid from feed tray as found from the top to down calculations

d_i, b_i = Moles of component i in top and bottom products in last iteration

Then corresponding changes required in bottom product for component i is $\Delta b_i = -\Delta d_i$. For new distribution, entire calculation is repeated. These calculations are continued until compositions of liquid, leaving the feed tray, obtained from top–down calculations and bottom–up calculations are approximately same.

For more exact calculations, enthalpy balance should also be done for each tray to find the variation in liquid rate and vapour rate on each tray. But these calculations require detailed and exact enthalpy data. Also, if one considers it, then calculations become more laborious and complicated. Use of a computer is then essential.

Example 8.10 A saturated liquid, consisting of phenol and cresols with some xylenols, is fractioned to give a top product of 95.3 mole % phenol. Metacresol is heavy key and phenol is light key component. Total condenser is used. The composition of the top product and of the phenols in the bottoms are given in Table 8.25.

(a) Complete the material balance over the still for a feed rate of 100 kmol/h.
(b) Calculate the minimum reflux ratio by Underwood's method.
(c) For $R = 3R_m$, calculate the composition of vapour entering to the topmost tray by Lewis–Matheson method.

Table 8.25 Distillation Column Data and Relative Volatilities

Component	α_{av}	Feed, mole %	Top product, mole %	Bottom product, mole %
Phenol	1.98	35	95.30	5.24
o–Cresol	1.59	15	4.55	?
m–Cresol	1.00	30	0.15	?
Xylenol(s)	0.59	20	—	?
Total		100	100.00	

Solution

(a) Overall material balance

$$F = D + W$$

$$100 = D + W \tag{1}$$

Phenol balance:

$$F \times 0.35 = D \times 0.953 + W \times 0.0524 \tag{2}$$

From Eqs. (1) and (2), $D = 33$ kmol/h, $W = 67$ kmol/h.

Component balance gives the composition of residue.

o–Cresol balance:

$$F \times 0.15 = 0.0455 \times D + x_{Wo} \times W$$

$$100 \times 0.15 = 0.0455 \times 33 + x_{Wo} \times 67$$

$$x_{Wo} = 0.2015$$

Similarly, $x_{Wm} = 0.447$, $x_{Wx} = 0.2985$

where, x_{Wm} = mole fraction of *m*–cresol in residue

x_{Wx} = mole fraction of xylenol in residue

(b) R_m by Underwood's method

$$\frac{1.98 \times 0.953}{1.98 - \vartheta} + \frac{1.59 \times 0.0455}{1.59 - \vartheta} + \frac{1 \times 0.0015}{1 - \vartheta} = R_m + 1 \tag{3}$$

$q = 1.0$ (saturated liquid feed)

$$\frac{1.98 \times 0.35}{1.98 - \vartheta} + \frac{1.59 \times 0.15}{1.59 - \vartheta} + \frac{1 \times 0.3}{1 - \vartheta} + \frac{0.59 \times 0.2}{0.59 - \vartheta} = 1 - 1 = 0 \tag{4}$$

By trial-and-error calculations, or by Mathcad, determine ϑ.

Also $\vartheta \in \{1, \ldots 1.98\}$.

$\vartheta = 1.22$

On substituting this value of ϑ in Eq. (3) gives

$R_m = 1.67$

(c) $R = 3\,R_m = 3 \times 1.67 = 5.01$

$L = RD$, $V = (R + 1)\,D$

$L = 5.01 \times 33 = 165.33$ kmol/h

$V = (5.01 + 1) \times 33 = 198.33$ kmol/h

Refer to Fig. 8.9(a).

Component balance around top segment

$$Vy_{(N-1)i} = Lx_{Ni} + Dx_{Di}$$

Composition of vapour entering to topmost tray

$$y_{(N-1)i} = \frac{L}{V}x_{Ni} + \frac{D}{V}x_{Di} \tag{5}$$

At dew point of overhead vapour

$$\sum \frac{y_{N_i}\,p_t}{p_{vi}} = \Sigma x_{Ni} = 1 \tag{6}$$

But here operating pressure p_t is not specified. So x_{Ni} cannot be determined by Eq. (6). x_{Ni} can be determined based on the data of average relative volatility, α_{avi}.

$$\Sigma x_{Ni} = x_{NP} + x_{NO} + x_{Nm} + x_{Nx} = 1$$

where, x_{Ni} = Mole fraction of component i in the liquid leaving the Nth tray

x_{NP} = Mole fraction of phenol in the same

x_{No} = Mole fraction of o–cresol in the same

x_{Nm} = Mole fraction of m–cresol in the same

x_{Nx} = Mole fraction of xylenol in the same

$$\frac{x_{NP}}{x_{Nm}} + \frac{x_{No}}{x_{Nm}} + 1 + \frac{x_{Nx}}{x_{Nm}} = \frac{1}{x_{Nm}}$$

Average relative volatility of phenol with respect to heavy key component,

$$\alpha_{\text{av}P} = \frac{y_{NP}/x_{NP}}{y_{Nm}/x_{Nm}}$$

$$\frac{x_{NP}}{x_{Nm}} = \frac{y_{NP}}{y_{Nm}\alpha_{\text{av}P}} \tag{7}$$

$$\frac{y_{NP}}{\alpha_{\text{av}P}} = \frac{x_{NP}}{x_{Nm}} y_{Nm}$$

$$\frac{y_{NP}}{\alpha_{\text{av}P}} + \frac{y_{No}}{\alpha_{\text{av}o}} + \frac{y_{Nm}}{\alpha_{\text{av}m}} + \frac{y_{Nx}}{\alpha_{\text{av}x}} = \frac{y_{Nm}}{x_{Nm}} \Sigma x_{Ni} = \frac{y_{Nm}}{x_{Nm}}$$

$$\sum \frac{y_{Ni}}{\alpha_{\text{av}i}} = \frac{y_{Nm}}{x_{Nm}} \tag{8}$$

From Eqs. (7) and (8):

$$x_{NP} = \frac{y_{NP} / \alpha_{\text{av}P}}{\sum \frac{y_{Ni}}{\alpha_{\text{av}i}}} = \frac{0.953/1.98}{(0.953/1.98) + (0.0455/1.59) + (0.0015/1)}$$

$$x_{NP} = 0.9411$$

Since total condenser is used, $y_{Ni} = x_{Di}$

Similarly,

$$x_{No} = \frac{\left(\frac{0.0455}{1.59}\right)}{\left(\frac{0.953}{1.98}\right) + \left(\frac{0.0455}{1.59}\right) + \left(\frac{0.0015}{1}\right)} = 0.055\ 95$$

$$x_{Nm} = 0.002\ 95$$

On substituting the values of x_{Ni} in Eq. (5)

$$y_{(N-1)i} = \frac{165.33}{198.33} \times x_{Ni} + \frac{33}{198.33} \times x_{Di}$$

$$y_{(N-1)P} = \frac{165.33}{198.33} \times 0.9411 + \frac{33}{198.33} \times 0.953$$

$$y_{(N-1)P} = 0.8336 \times 0.9411 + 0.1664 \times 0.953$$
$$= 0.943$$

$$y_{(N-1)o} = 0.8336 \times 0.055\ 95 + 0.1664 \times 0.0455 = 0.0542$$

$$y_{(N-1)m} = 0.8336 \times 0.002\ 95 + 0.1664 \times 0.0015 = 0.0027$$

Example 8.11 Find the number of theoretical stages required for the desired separation by Lewis–Matheson method for the following feed mixture.

Table 8.26 Distillation Column Details

Component	Feed, mole %	Distillate, mole %	Residue, mole %
n-Butane	37	95.0	16.3
i-Pentane	32	5.0	41.6
n-Pentane	21	0.0	28.5
n-Hexane	10	0.0	13.6
Total	100	100.0	100.0

In Table 8.26, the distillate and residue compositions are specified for the first trial calculations. These are not the final values.

1. $F = 4750$ mol/h, $D = 1250$ mol/h, $W = 3500$ mol/h and $q = 1$
2. *n*–Butane is light key component and *i*–Pentane is heavy key component.
3. Operating pressure $p_t = 2$ atm a
4. Reflux ratio $R = 3$
5. Vapour pressures of components can be calculated using Antoine equation.

$$\ln p_v = A - \frac{B}{(T + C)}, \quad p_v \text{ in Torr and } T \text{ in K} \tag{2.1}$$

Table 8.27 Antoine Constants[10]

Component	*A*	*B*	*C*
n-Butane	15.6782	2154.9	– 34.42
i-Pentane	15.6338	2348.67	– 40.05
n-Pentane	15.8333	2477.07	– 39.94
n-Hexane	15.8366	2697.55	– 48.78

Solution

Lewis–Matheson method

Molar flow rates of liquid and vapour in enriching section

$$L = RD, \quad V = (R + 1)D$$

$$L = 3 \times 1250 = 3750 \text{ mol/h}, V = (3 + 1) \times 1250 = 5000 \text{ mol/h}$$

For total condenser,

$$y_{Ni} = x_{Di} = x_{oi}$$

To find the dew point of overhead vapour

$$\sum \frac{y_{Ni} p_t}{p_{vi}} = 1$$

For $T = 295$ K (i.e., $t = 22°C$), $p_{vB} = 1650$ Torr, $p_{viP} = 615$ Torr

$$\frac{0.95 \times (2 \times 760)}{1650} + \frac{0.05 \times (2 \times 760)}{615} \cong 1$$

or $\quad 0.875\,15 + 0.1236 = 0.998\,75 \cong 1$

Hence, dew point temperature of overhead vapour, $t_{DP} = 22°C$.

The same calculations also give

$$x_{NB} = 0.875\,15$$

$$x_{NiP} = 0.1236$$

Material balance around the top segment, shown in Fig. 8.9(a).

$$Vy_{(N-1)i} = Lx_{Ni} + Dx_{Di}$$

$$y_{(N-1)i} = \frac{L}{V}x_{Ni} + \frac{D}{V}x_{Di}$$

$$y_{(N-1)B} = \frac{3750}{5000} \times x_{NB} + \frac{1250}{5000} \times x_{DB}$$

$$y_{(N-1)B} = 0.75 \times 0.875\ 15 + 0.25 \times 0.95 = 0.8939$$

$$y_{(N-1)iP} = 0.75 \times 0.1236 + 0.25 \times 0.05 = 0.1052$$

Find the dew point temperature of vapour leaving the $(N-1)^{\text{th}}$ tray by trial-and-error calculations.

At $t = 24°\text{C}$ or $T = 297$ K

$p_{viB} = 1757.3$ Torr, $p_{viP} = 660.65$ Torr

$$\sum \frac{y_{(N-1)}p_t}{p_{vi}} = \frac{0.8911 \times (2 \times 760)}{1757.3} + \frac{0.1050 \times (2 \times 760)}{660.65} = 1.012 \approx 1$$

Dew point temperature of vapour leaving the $(N-1)^{\text{th}}$ tray is 24°C.

The same calculations also give

$$x_{(N-1)B} = 0.77 \text{ and } x_{(N-1)iP} = 0.24$$

Here $\Sigma x_{(N-1)i} = 1.01$. To make this total = 1.000, normalised values of $x_{(N-1)i}$ are determined as follows.

$$x_{(N-1)B} = \frac{0.77}{0.77 + 0.24} = 0.7624$$

$$x_{(N-1)\,iP} = \frac{0.24}{0.77 + 0.24} = 0.2376$$

Check whether the $(N-1)^{\text{th}}$ tray is feed tray or not.

$$\frac{x_{(N-1)B}}{x_{(N-1)_P}} = \frac{0.7624}{0.2376} = 3.2 \nless \frac{37 \text{ (moles of } nC_4 \text{ in feed)}}{32 \text{ (moles of } iC_5 \text{ in feed)}} \quad (= 1.156)$$

Hence, $(N-1)^{\text{th}}$ tray is not the feed tray.

Component balance around $(N-1)^{\text{th}}$ tray

Refer to Fig. 8.9(b):

$$Vy_{(N-2)i} = Lx_{(N-1)i} + Vy_{(N-1)i} - Lx_{Ni}$$

$$y_{(N-2)i} = \frac{L}{V}x_{(N-1)i} + y_{(N-1)i} - \frac{L}{V}x_{Ni}$$

$$y_{(N-2)B} = 0.75 \times 0.7624 + 0.8911 - 0.75 \times 0.875\ 15$$

$$y_{(N-2)B} = 0.8065, \quad y(N-2)_{iP} = 1 - y_{(N-2)B} = 0.1935$$

Find the dew point temperature of vapour leaving the $(N-2)^{\text{th}}$ tray.

At, $T = 301$ K or $t = 28°$ C, $p_{vB} = 1987.6$ Torr, $p_{viP} = 760$ Torr

$$\sum\left(\frac{y_{(N-2)i}p_t}{p_{vi}}\right) = \frac{0.8065\times(2\times760)}{1987.6} + \frac{0.1935\times(2\times760)}{760}$$

$$= 0.6168 + 0.387 = 1.0038 \approx 1$$

The same calculations also give

$$x_{(N-2)B} = 0.6168 \text{ and } x_{(N-2)iP} = 0.387$$

Normalised values of $x_{(N-2)i}$

$$x_{(N-2)B} = 0.6182, \quad x_{(N-2)iP} = 0.3818$$

Check whether $(N-2)^{\text{th}}$ tray is feed tray or not.

$$\frac{x_{(N-2)B}}{x_{(N-2)_{iP}}} = \frac{0.6182}{0.3818} = 1.62 \nless 1.156\left(=\frac{37}{32}\right)$$

$(N-2)^{\text{th}}$ tray is not the feed tray.

Component balance around $(N-2)^{\text{th}}$ tray

$$Vy_{(N-3)i} = Lx_{(N-2)i} + Vy_{(N-2)i} - Lx_{(N-1)i}$$

$$y_{(N-3)i} = \frac{L}{V}x_{(N-2)i} + y_{(N-2)i} - \frac{L}{V}x_{(N-1)i}$$

$$y_{(N-3)B} = 0.75\times0.6182 + 0.8065 - 0.75\times0.7624 = 0.6984$$

$$y_{(N-3)iP} = 0.3016$$

Find the dew point of vapour, leaving the $(N-3)^{\text{th}}$ tray.

At $T = 305$ K or $t = 32°C$, $p_{vB} = 2239.9$ Torr, $p_{viP} = 870.63$ Torr.

$$\sum\frac{y_{(N-3)i}p_t}{p_{vi}} = \frac{0.6984\times1520}{2239.9} + \frac{0.3016\times1520}{870.63} = 1.00$$

$$= 0.4789 + 0.5266 = 1.0005 \approx 1$$

Dew point temperature of vapour leaving the $(N-3)^{\text{th}}$ tray, $t_{DP} = 32°C$

The same calculations also give

$$x_{(N-3)B} = 0.47 \text{ and } x_{(N-3)iP} = 0.53$$

Check $(N-3)^{\text{th}}$ tray is feed tray or not.

$$\frac{x_{(N-3)B}}{x_{(N-3)iP}} = \frac{0.47}{0.53} = 0.887 < 1.156\left(=\frac{37}{32}\right)$$

$(N-3)^{\text{th}}$ tray or 4th tray from the top is the feed tray.

Molar flow rate of liquid and vapour in stripping or exhausting section:

$$\bar{L} = L + Fq = 3750 + 4750 = 8500 \text{ kmol/h}$$

$$\bar{V} = \bar{L} - W = 8500 - 3500 = 5000 \text{ kmol/h}$$

Correct mole fractions in liquid leaving the feed tray

$$x_{BF}\bar{L} = x_B L + x_B' F$$

$$x_{BF} = 0.47\times\frac{3750}{8500} + 0.37\times\frac{4750}{8500} = 0.414$$

(where x_B' is mole fraction of n–Butane in feed)

$$x_{iPF} = 0.53 \times \frac{3750}{8500} + 0.32 \times \frac{4750}{8500} = 0.413$$

$$x_{nPF} = \frac{0.21 \times 4750}{8500} = 0.1173$$

$$x_{nHF} = \frac{0.1 \times 4750}{8500} = 0.056$$

Composition of the liquid, leaving the feed tray obtained from top to down calculations.

$$x_{BF} = 0.412, \quad x_{iPF} = 0.413, \quad x_{nPF} = 0.1173, \quad x_{nHF} = 0.056$$

$$\frac{x_{BF}}{x_{iPF}} \cong 1 < 1.156$$

Bottom to up calculations:

Assume $\Delta p \approx 0$, i.e., column operates at nearly same pressure from top to bottom.

Find the bubble point of residue via trial-and-error calculations:

$$\sum \frac{x_{Wi} p_{vi}}{p_t} = \frac{0.163 \times p_{vB}}{1520} + \frac{0.416\ p_{viP}}{1520} + \frac{0.285 p_{vnP}}{1520} + \frac{0.136 p_{vnH}}{1520}$$

At $t = 48°\text{C}$ or $T = 321\text{K}$

$$\sum \frac{x_{Wi} p_{pvi}}{p_t} = \frac{0.163 \times 3494 + 0.416 \times 1442.4 + 0.285 \times 1118.8 + 0.136 \times 375}{1520}$$

$$= 1.01 \cong 1$$

Hence, bubble point temperature, $t_{BP} = 48°\text{C}$

The same calculations also give the vapour composition, leaving the reboiler.

$$y_{WB} = \frac{x_{WB} p_{vB}}{p_t} = 0.375$$

$$y_{WiP} = 0.395, \quad y_{WnP} = 0.2098, \quad y_{WnH} = 0.033$$

Component balance around reboiler gives (Ref. Fig. 8.10):

$$\bar{L} x_{1i} = \bar{V} y_{Wi} + W x_{Wi}$$

$$x_{1i} = \frac{\bar{V}}{\bar{L}} y_{Wi} + \frac{W}{\bar{L}} x_{Wi} = \frac{5000}{8500} y_{Wi} + \frac{3500}{8500} x_{Wi}$$

$$= 0.5882\ y_{Wi} + 0.4118\ x_{Wi}$$

Composition of liquid, leaving the first tray

$$x_{1B} = 0.5882 \times 0.375 + 0.4118 \times 0.163 = 0.2877$$

$$x_{1iP} = 0.5882 \times 0.395 + 0.4118 \times 0.416 = 0.4036$$

$$x_{1nP} = 0.5882 \times 0.2098 + 0.4118 \times 0.285 = 0.24$$

$$x_{1nH} = 0.5882 \times 0.033 + 0.4118 \times 0.136 = 0.075$$

$$\frac{x_{1B}}{x_{1iP}} = \frac{0.2877}{0.4036} = 0.7128, \quad \frac{x_{BF}}{x_{iPF}} = 1.0036$$

Hence, $\frac{x_{1B}}{x_{1iP}} < \frac{x_{BF}}{x_{iPF}}$ and first tray (from bottom) is not the feed tray.

Find the bubble point of liquid leaving the first tray by trial-and-error calculations.

At $t = 41°C$ or $T = 314$ K

$p_{vB} = 2894.5$ Torr, $p_{viP} = 1164.97$ Torr, $p_{vnP} = 893.26$ Torr

$p_{vnH} = 288.76$ Torr, (by using Antoine constants)

$$\sum \frac{x_{1i}p_{vi}}{p_t} = \frac{0.2877 \times 2894.5}{1520} + \frac{0.4036 \times 1164.97}{1520} + \frac{0.24 \times 893.26}{1520} + \frac{0.075 \times 288.76}{1520} = 1.012 \approx 1 = y_{1i}$$

Bubble point of liquid leaving the first tray, $t_{BP} = 41°C$. Same calculations also give the composition of vapour, leaving the first tray.

$$y_{1B} = \frac{x_{1B}p_{vB}}{p_t} = \frac{0.2877 \times 2894.5}{1520} = 0.548$$

$$y_{1iP} = 0.309, \quad y_{1nP} = 0.141, \quad y_{1nH} = 0.014$$

Normalised values of y_{1i}

$$y_{1B} = \frac{0.548}{\Sigma y_{1i}} = \frac{0.548}{1.012} = 0.5415$$

$$y_{1iP} = \frac{0.309}{1.012} = 0.305$$

$$y_{1nP} = \frac{0.141}{1.012} = 0.139$$

$$y_{1nH} = \frac{0.014}{1.012} = 0.0138$$

Component balance around the first tray gives

$$\bar{L}x_{2i} = \bar{V}y_{1i} + \bar{L}x_{1i} - \bar{V}y_{wi}$$

$$x_{2i} = \frac{\bar{V}}{\bar{L}}y_{1i} + x_{1i} - \frac{\bar{V}}{\bar{L}}y_{wi}$$

$$x_{2B} = \frac{5000}{8500} \times 0.5415 + 0.2877 - \frac{5000}{8500} \times 0.375 = 0.3856$$

$$x_{2iP} = \frac{5000}{8500} \times 0.305 + 0.4036 - \frac{5000}{8500} \times 0.395 = 0.3506$$

$$x_{2nP} = \frac{5000}{8500} \times 0.139 + 0.24 - \frac{5000}{8500} \times 0.2098 = 0.1984$$

$$x_{2nH} = \frac{5000}{8500} \times 0.0138 + 0.075 - \frac{5000}{8500} \times 0.033 = 0.0637$$

$$\frac{x_{2B}}{x_{2iP}} = \frac{0.3856}{0.3506} = 1.1 \cong \frac{x_{BF}}{x_{iPF}} \text{ (= 1.025) – close enough}$$

Second tray from bottom can be called the feed tray. Ideally values of this ratio $\left(\frac{x_{BF}}{x_{iPF}}\right)$ obtained from bottom to up calculations should be same as that obtained from top to down calculation. Here composition of liquid leaving the feed tray obtained from top to down calculations and the same obtained from bottom to up calculations are not matching.

Table 8.28 Summary of First Trial Calculations of Feed Tray Liquid

Component	Calculated mole fractions	
	Composition based on top–down calculation	Composition based on bottom–up calculation
n-Butane	0.412	0.3856
i-Pentane	0.413	0.3506
n-Pentane	0.1173	0.1984
n-Hexane	0.056	0.0637

To match this composition and to get the exact value of number of theoretical stages required for the desired separation of key components, second set of calculations are required. Based on first trial calculations number of theoretical stages required for the desired separation of key components is 6 (Reboiler, 1st , 2nd = $N - 3$ = Feed tray, $N - 2$, $N - 1$, N).

How to start the second trial calculations?

To start the second trial calculations, product composition x_{Di} and x_{Wi} (hence, D and W also) must be changed. Based on the Smith's suggestion following equation can be used to make the change in product composition for the 2nd trial calculations.

Change in moles of component in distillate.

$$\Delta d_i = \frac{(x_{FB} - x_{FD})_i}{\left(\frac{x_{FD}}{d}\right)_i + \left(\frac{x_{FB}}{b}\right)_i} \tag{8.40}$$

For *n*–Butane or for nC_4

$$\Delta d_{nC_4} = \frac{0.3856 - 0.412}{\left(\frac{0.412}{1250 \times 0.95}\right) + \left(\frac{0.3856}{0.163 \times 3500}\right)} = -25.81 \text{ mol/h}$$

nC_4 in distillate for the second trial calculation = $1250 \times 0.95 - 25.81 = 1161.69$ mol/h

nC_4 in residue = $0.163 \times 3500 + 25.81 = 596.31$ mol/h

Note that total of molar flow rates of each component should be constant.

For *iso*–Pentane or iC_5:

$$\Delta d_{iC_5} = \frac{0.3506 - 0.413}{\left(\frac{0.413}{1250 \times 0.05}\right) + \left(\frac{0.3506}{3500 \times 0.416}\right)} = -9.1 \text{ mol/h}$$

iC_5 in distillate = 1250 × 0.05 – 9.1 = 53.4 mol/h

iC_5 in residue = 3500 × 0.416 + 9.1 = 1465.1 mol/h

For *n*-Pentane or nC_5:

$$\Delta d_{nC_5} = \frac{0.1984 - 0.1173}{\left(\frac{0.1173}{d_{nC_5}}\right) + \left(\frac{0.1984}{3500 \times 0.285}\right)} = 0.6913 \text{ mol/h}$$

In first trial calculation $d_{nC_5} = 0$. But to use above equation one cannot put $d_{nC_5} = 0$. Let $d_{nC_5} = 1$ mol/h (negligible value)

nC_5 in distillate = 1 + 0.6913 = 1.7 mol/h

nC_5 in residue = 3500 × 0.285 – 1.7 = 995.8 mol/h

For *n*-Hexane or nC_6:

$$\Delta d_{nC_6} = \frac{0.0637 - 0.056}{\left(\frac{0.056}{d_{nC_6}}\right) + \left(\frac{0.0637}{3500 \times 0.136}\right)}$$

Let $d_{nC_6} = 1$ mol/h (negligible)

$\Delta d_{nC_6} = 0.137$ mol/h

nC_6 in distillate = 1.137 mol/h

nC_6 in residue = 3500 × 0.137 – 1.137 = 474.86 mol/h

Table 8.30 Product Composition for Second Trial Calculations

Component	Distillate, *D*		Residue, *W*	
	mol/h	mole fraction	mol/h	mole fraction
n_{C_4}	1161.69	0.954	596.31	0.169
i_{C_5}	53.4	0.044	1465.1	0.415
n_{C_5}	1.7	0.001	995.8	0.282
n_{C_6}	1.137	0.001	474.86	0.134
Total	1217.93	1.000	3532.07	1.000

Based on this new product distribution one can start the second set of calculations. These trial calculations are repeated until the composition of liquid leaving the feed tray obtained from top–down calculations match with the same obtained from bottom–up calculations. Results obtained at the end of first trial calculations are matching with the same given by FUG method.

(b) Thiele Geddes Method[1]:

Original method of Thiele and Geddes was developed for manual calculations in 1933. Thereafter computer programs are developed for this method to reduce the labour of calculations. This method is a rating method in which distribution of components between distillate and bottoms is predicted for a specified number of stages.

In this method, following variables are to be specified as input data:

(i) Number of equilibrium stages above and below the feed.
(ii) Column pressure
(iii) Temperatures (stagewise) – Dew point temperature of vapour leaving the equilibrium stage is equal to bubble point temperature of liquid leaving the same stage and this temperature is called the stage temperature.
(iv) Reflux flow rate
(v) Distillate rate
(vi) Feed flow rate, feed condition, and feed composition
(vii) Liquid and vapour flow rates, leaving each stage

Calculation procedure is as follows.

Fig. 8.11 *Material Balance around Overhead Condenser*

Material balance for any component around the condenser is

$$G_1\, y_1 = L_o\, x_o + Dz_D$$

$$\frac{G_1 y_1}{Dz_D} = \frac{L_o x_o}{Dz_D} + 1$$

For a total condenser, $y_1 = x_o = z_D = x_D$

and $\dfrac{L_o}{D} = R$

$$\frac{G_1 y_1}{Dz_D} = \frac{L_o}{D} + 1 = R + 1 = A_o + 1$$

where, $A_o = \dfrac{L_o}{D} = R$ (for total condenser)

For a partial condenser, $z_D = y_D = K_o\, x_o$

where, K_o = equilibrium constant for partial condenser

$$\frac{G_1 y_1}{Dz_D} = \frac{L_o x_o}{Dz_D} + 1 = \frac{L_o}{DK_o} + 1 = \frac{R}{K_o} + 1 = A_o + 1$$

where, $A_o = \dfrac{R}{K_o}$ (for partial condenser)

Component balance up to tray 1 gives

$$G_2\, y_2 = L_1\, x_1 + Dz_D$$

$$\frac{G_2 y_2}{Dz_D} = \frac{L_1 x_1}{Dz_D} + 1 \tag{8.41}$$

Fig. 8.12 *Material Balance around Top Section up to First Tray*

For Tray 1, absorption factor

$$A_1 = \frac{L_1}{K_1 G_1} = \frac{L_1 x_1}{G_1 y_1} \tag{8.42}$$

$$L_1\, x_1 = G_1\, y_1\, A_1$$

From Eqs. (8.41) and (8.42),

$$\frac{G_2 y_2}{Dz_D} = \frac{G_1 y_1 A_1}{Dz_D} + 1$$

$$= (A_o + 1)\, A_1 + 1 = A_1\, A_o + A_1 + 1 \tag{8.41a}$$

Similarly, for the vapour coming from any n^{th} tray of enriching section

$$\frac{G_n y_n}{Dz_D} = \frac{A_{n-1}\, G_{n-1}\, y_{n-1}}{Dz_D} = A_0\, A_1\, A_2 \ldots A_{n-1}$$

$$+ A_1\, A_2 \ldots A_{n-1} + \ldots + A_{n-1} + 1 \tag{8.43}$$

For the vapour coming from feed tray

$$\frac{G_F y_F}{Dz_D} = A_0\, A_1\, A_2 \ldots A_{F-1} + A_1\, A_2 \ldots A_{F-1} + \ldots + A_{F-1} + 1 \tag{8.44}$$

For stripping or exhausting section:
Component balance around reboiler

$$\bar{L}_N x_N = \bar{G}_{N+1}\, y_{N+1} + W x_W$$

$$\frac{\bar{L}_N x_N}{W x_W} = \frac{\bar{G}_{N+1} y_{N+1}}{W x_W} + 1$$

$$\frac{\bar{L}_N x_N}{W x_W} = S_W + 1 \tag{8.45}$$

where, S_W is stripping factor of reboiler.

For kettle-type reboiler ($y_{N+1} = K_b\, x_W$) stripping factor

$$S_W = \frac{\bar{G}_{N+1} y_{N+1}}{W x_W} = \frac{\bar{G}_{N+1} K_b}{W} \tag{8.46}$$

where, K_b = equilibrium constant for kettle-type reboiler

For thermosyphon reboiler $y_{N+1} \cong x_W$

Fig. 8.13 *Material Balance around Reboiler*

Stripping factor for thermosyphon reboiler

$$S_w = \frac{\bar{G}_{N+1}}{W} \tag{8.47}$$

Component balance around bottom section up to the last tray.

Fig. 8.14 *Material Balance around Bottom Section up to Final Tray*

$$\bar{L}_{N-1}\, x_{N-1} = \bar{G}_N\ y_N + W x_W$$

$$\frac{\bar{L}_{N-1}\ x_{N-1}}{W x_W} = \frac{\bar{G}_N y_N}{W x_W} + 1 \tag{8.48}$$

Stripping factor for N^{th} tray

$$S_N = \frac{\bar{G}_N K_N}{\bar{L}_N} = \frac{\bar{G}_N\ y_N}{\bar{L}_N x_N}$$

where, K_N = Equilibrium constant for N^{th} tray

$$\bar{G}_N \; y_N = \bar{L}_N \; x_N \; S_N \tag{8.49}$$

From the Eqs. (8.48) and (8.49),

$$\frac{\bar{L}_{N-1} \; x_{N-1}}{Wx_W} = \frac{\bar{L}_N \; x_N}{Wx_W} \; S_N + 1 \tag{8.50}$$

From Eqs. (8.45) and (8.50),

$$\frac{\bar{L}_{N-1} \, x_{N-1}}{Wx_W} = (S_W + 1) \, S_N + 1 = S_w \, S_N + S_N + 1 \tag{8.51}$$

For any m^{th} tray of stripping section

$$\frac{\bar{L}_m \, x_m}{Wx_W} = S_{m+1} \cdot S_{m+2} \ldots S_{N-1} \cdot S_N \cdot S_w + S_{m+1} \cdot S_{m+2} \ldots S_{N-1} \, S_N$$
$$+ S_{m+1} \cdot S_{m+2} \ldots S_{N-1} + \ldots + S_{m+1} + 1 \tag{8.52}$$

For the liquid leaving the feed tray

$$\frac{\bar{L}_F x_F}{Wx_W} = S_{F+1} \cdot S_{F+2} \ldots S_N \cdot S_w + S_{F+1} \cdot S_{F+2} \ldots S_N$$
$$+ S_{F+1} \ldots S_{N-1} + \ldots + S_{F+1} + 1 \tag{8.53}$$

Absorption factor for feed tray

$$A_F = \frac{\bar{L}_F}{K_F G_F} = \frac{\bar{L}_F \; x_F}{G_F \; y_F} \tag{8.54}$$

where, K_F = Equilibrium constant for feed tray

$$\frac{Wx_W}{Dz_D} = \frac{G_F y_F / Dz_D}{\bar{L}_F x_F / Wx_w} \frac{\bar{L}_F x_F}{G_F y_F} = \frac{(G_F y_F / Dz_D)}{(\bar{L}_F x_F / Wx_W)} \cdot A_F \tag{8.55}$$

Symbol z_D is mole fraction of distillate which can be liquid or vapour.

In calculations, $G_F y_F / Dz_D$ ratio is determined by Eq. (8.44). $(\bar{L}_F \; x_F)/(Wx_W)$ ratio is determined by Eq. (8.53).

Absorption factor for feed tray is determined by Eq. (8.54). The values are substituted in Eq. (8.55) and the ratio Wx_w/Dz_D is determined.

Overall component balance

$$Fz_F = Wx_W + Dz_D \tag{8.56}$$

$$\frac{Fz_F}{Dz_D} = \frac{Wx_W}{Dz_D} + 1$$

$$Dz_D = \frac{Fz_F}{\dfrac{Wx_W}{Dz_D} + 1} \tag{8.57}$$

Equation (8.57) gives the value of Dz_D, then Eq. (8.56) gives Wx_W. Thus finally one can get the product composition.

This product composition is correct, if all input data are correct. Normally, in the first trial calculations temperatures at the trays are assumed to calculate the equilibrium constants; K_i. To verify whether assumed values of temperatures are correct or not, find y_n by following equation:

$$y_n = \frac{G_n \; y_n}{Dz_D} \times \frac{Dz_D}{G_n} \tag{8.58}$$

where, $\dfrac{G_n y_n}{Dz_D}$ is determined by Eq. (8.43).

For any tray n, y_ns for all components must be determined by Eq. (8.58). Then check whether $\Sigma y_{ni} = 1$ or not.

If $\Sigma y_{ni} = 1$, it means assumed value of temperature for tray n is correct. If $\Sigma y_{ni} \neq 1$, find the normalized values of y_{ni} and find new dew point temperature. Same calculations should be done for all the trays.

Similarly, in stripping section for each tray (m) find

$$x_m = \frac{\overline{L}_m x_m}{Wx_W} \times \frac{Wx_W}{\overline{L}_m} \tag{8.59}$$

and check whether $\Sigma x_{mi} = 1$ or not.

Here $\dfrac{\overline{L}_m x_m}{Wx_W}$ is determined by Eq. (8.52).

If $\Sigma x_{mi} \neq 1$, then find the normalized values of x_{mi} and find new bubble point.

For new temperatures, carry out the material balance and enthalpy balance simultaneously and find the new values of molar liquid flow rates and molar vapour flow rates (new values of $L's$, $G's$, $\overline{L}'s$, $\overline{G}'s$). Then repeat the calculations.

Example 8.12 Find the product composition by Thiele–Geddes method based on the following data (same as Table 8.26):

(i)

Table 8.31 *Feed Composition*

Component	mole %
n–Butane	37
i–Pentane	32
n–Pentane	21
n–Hexane	10

n–Butane is light key component and i–Pentane is heavy key component.

(ii) Feed flow rate $F = 4750$ mol/h. Feed is saturated liquid at its bubble point.

(iii) Reflux ratio, $R = 3$

(iv) $D = 1250$ mol/h, $W = 3500$ mol/h

(v) Operating pressure, $p_t = 2$ atm a

(vi) Assume constant molar overflow. Total condenser is used.

Table 8.32 Equilibrium Constant Data ($K_i s$)

Tray number	Temperature	K_{C4}	K_{iC5}	K_{nC5}	K_{C6}
1	22°C	1.086	0.4046	0.3000	0.0870
2	24°C	1.156	0.4346	0.3230	0.0950
3	28°C	1.308	0.5000	0.3747	0.1125
4 = feed	32°C	1.474	0.5730	0.4324	0.1330
5	41°C	1.904	0.7660	0.5877	0.1900
Reboiler	48°C	2.300	0.9500	0.7360	0.2500

(Read from Fig. 6.20)

Solution

Molar flow rates of liquid and vapour in enriching section.

$$L = R\,D = 3 \times 1250 = 3750 \text{ mol/h}$$

$$G = (R + 1)\,D = 4 \times 1250 = 5000 \text{ mol/h}$$

$$\frac{Wx_W}{Dx_D} = \frac{G_F y_F / Dx_D}{L_F x_F / Wx_W} \times A_F$$

$$\frac{G_F y_F}{Dx_D} = A_0 A_1 A_2 \ldots A_{F-1} + \ldots + A_{F-1} + 1 \tag{8.44}$$

Here fourth tray is feed tray.

$$\frac{G_F y_F}{Dx_D} = A_0 A_1 A_2 A_3 + A_1 A_2 A_3 + A_2 A_3 + A_3 + 1$$

for n–Butane

$$A_0 = R = 3$$

$$A_1 = \frac{L_1}{K_1 G} = \frac{L}{K_{C_4} G} = \frac{3750}{1.086 \times 5000} = 0.6906$$

$$A_2 = \frac{L}{K_{C_4} G} = \frac{3750}{1.156 \times 5000} = 0.6488$$

$$A_3 = \frac{3750}{1.308 \times 5000} = 0.5734$$

$$\frac{G_F y_F}{Dx_D} = 3 \times 0.6906 \times 0.6488 \times 0.5734 + 0.6906 \times 0.6488 \times 0.5734 + 0.6488 \times 0.5734 + 0.5734 + 1 = 2.973$$

$$A_F = \frac{\overline{L}_F}{K_F G_F} \tag{8.54}$$

$$\bar{L} = L + Fq = 3750 + 4750 \times 1 = 8500 \text{ mol/h}$$

$K_F = K_{C_4}$ at feed tray temperature (equilibrium constant for *n*–Butane)

$$K_F = 1.474$$

$$A_F = \frac{8500}{1.474 \times 5000} = 1.1533$$

$$\frac{\bar{L}_F x_F}{W x_W} = S_{F+1} \cdot S_{F+2} \ldots S_N \cdot S_W + S_{F+1} \ldots S_N + \ldots + S_{F+1} + 1 \quad (8.53)$$

$$\bar{G} = G + F(q-1) \text{ or } \bar{G} = \bar{L} - W = 8500 - 3500 = G = 5000 \text{ mol/h}$$

$$\frac{\bar{L}_F x_F}{W x_W} = S_5 S_w + S_5 + 1$$

$$S_5 = \frac{K_5 \bar{G}}{\bar{L}} = \frac{1.904 \times 5000}{8500} = 1.12$$

$$S_W = \frac{K_W \bar{G}}{W} = \frac{2.3 \times 5000}{3500} = 3.286$$

$$\frac{\bar{L}_F x_F}{W x_W} = 1.12 \times 3.286 + 1.12 + 1 = 5.8003$$

$$\frac{W x_W}{D x_D} = \frac{(G_F y_F / D x_D)}{(\bar{L}_F x_F / W x_W)} \times A_F = \frac{2.973}{5.8003} \times 1.1533 = 0.5911 \quad (8.55)$$

$$D z_D = \frac{F z_F}{\frac{W x_W}{D z_D} + 1} = \frac{4750 \times 0.37}{0.5911 + 1} = 1104.58 \text{ mol/h} \quad (8.57)$$

$$W x_w = 652.92 \text{ mol/h}$$

$$z_D = 0.8837, \; x_W = 0.1865$$

For *iso*–Pentane:

$$A_0 = R = 3, \quad A_1 = \frac{L}{K_1 G} = \frac{3750}{K_1 \times 5000}$$

$$A_1 = \frac{0.75}{K_{iC_5} \text{ at } 22°\text{C}} = \frac{0.75}{0.4046} = 1.8537$$

$$A_2 = \frac{L}{K_2 G} = \frac{0.75}{0.4346} = 1.7257$$

$$A_3 = \frac{L}{K_3 G} = \frac{0.75}{0.5} = 1.5$$

$$\frac{G_F y_F}{D x_D} = 3 \times 1.8537 \times 1.7257 \times 1.5 + 1.8537 \times 1.7257 \times 1.5 + 1.7257 \times 1.5 + 1.5 + 1$$

$$= 24.282$$

Absorption factor for feed tray

$$A_F = \frac{\overline{L}}{K_F G} = \frac{8500}{0.573 \times 5000} = 2.967$$

$$\frac{\overline{L}_F x_F}{W x_W} = S_5 S_W + S_5 + 1$$

$$S_5 = \frac{K_5 \overline{G}}{\overline{L}} = \frac{0.766 \times 5000}{8500} = 0.45$$

$$S_W = \frac{K_W \overline{G}}{W} = \frac{0.95 \times 5000}{3500} = 1.357$$

$$\frac{\overline{L}_F x_F}{W x_W} = 0.45 \times 1.357 + 0.45 + 1 = 2.060\,65$$

$$\frac{W x_W}{D x_D} = \frac{24.282}{2.060\,65} \times 2.967 = 34.96$$

$$D x_D = \frac{4750 \times 0.32}{(34.96 + 1)} = 42.27 \text{ mol/h}$$

$$W x_W = 1477.73 \text{ mol/h}$$

$$x_D = 0.0338, \quad x_W = 0.4222$$

For *i*-pentane, similarly one can calculate, $x_D = 0.005\,58$, $x_W = 0.283$ and for *n*-hexane $x_D \cong 0$ and $x_W = 0.1357$

Table 8.33 Resulting Product Compositions

Component	x_D	$x_{D\text{nor}}$	x_W	$x_{W\text{nor}}$
n-Butane	0.8837	0.9571	0.1865	0.1815
i-Pentane	0.0338	0.0366	0.4222	0.4110
n-Pentane	0.0059	0.0063	0.2829	0.2754
n-Hexane	—	—	0.1357	0.1321
Total	0.9234	1.0000	1.0273	1.0000

$x_{D\text{nor}}$ = Normalised values of x_{Di}

$x_{W\text{nor}}$ = Normalised values of x_{Wi}

Note: Results of Tables 8.30 and 8.33 are close enough.

(c) Equation–Tearing Procedures Using Tridiagonal Matrix Algorithm[8]:
This method require the help of a computer. This method is more general and flexible, compared to Lewis–Matheson method and Thiele–Geddes method.

Consider a general continuous flow steady state, multicomponent multistage distillation operation. Assume that phase equilibrium between an existing vapour

phase and existing liquid phase is achieved at each stage, that no chemical reaction takes place, and that neither of the existing phases entrains the other phase. Consider the general equilibrium stage j as shown in Fig. 8.15.

Fig. 8.15 *General Equilibrium Stage*

Here F_j represent molar flow rate of feed to a stage j at temperature T_{Fj} (in K) and pressure p_{fj} and with overall composition in mole fractions z_{ij}. If stage j is not the feed stage then in calculation $F_j = 0$. Heat is transferred from stage j (+ve) or to stage j (–ve) at rate ϕ_j. If there is no external heat exchanger, attached with stage j, then $\phi_j = 0$. Equilibrium vapour and liquid phases leave the stage j at temperature (in K) and pressure p_j and with mole fraction y_{ij} and x_{ij}, respectively. The vapour may be partially withdrawn from the column as side stream at a molar flow rate W_j and the remainder V_j is sent to adjacent stage $j - 1$ above. Similarly, existing liquid may be split into a side stream at a molar flow rate of U_j, with the remainder L_j, sent to adjacent stage j as follows:

For each stage j, following independent equations can be written:

(i) Component material balance equations (M)

$$L_{j-1}\, x_{i\,j-1} + V_{j+1}\, y_{i\,j+1} + F_j\, z_{ij} - (L_j + U_j)\, x_{ij} - (V_j + W_j)\, y_{ij} = 0 \tag{8.60}$$

(ii) Phase equilibrium equation (E)

$$y_{ij} - K_{ij}\, x_{ij} = 0 \tag{8.61}$$

(iii) Mole fraction summation equation (S)

$$\Sigma y_{i\,j} - 1.0 = 0$$

$$\Sigma x_{ij} - 1.0 = 0 \tag{8.62}$$

(iv) Energy balance or enthalpy balance equation (H)

$$L_{j-1} H_{Lj-1} + V_{j+1} H_{vj+1} + F_j H_{Fj} - (L_j + U_j) H_{Lj} - (V_j + W_j) H_{vj} - \phi_j = 0 \tag{8.63}$$

These equations are known as MESH equations. For evaluating different variables namely; flow rates, compositions, temperatures and pressures, number of MESH equations are necessary. For this purpose, degree of freedom dictates that for any stage j total $2C + 3$ MESH (= C No. of material balance equations + C No. of phase equilibrium equations + 2 No. of mole fraction summation equations + 1 No. of energy balance equation) equations (where, C = number of components) can be written. For total N number of equilibrium stages, $N(2C + 3)$ MESH equations can be written. Total number of variables are $N(3C + 10) + 1$. It includes $(C - 1)$ numbers of z_{ij}, y_{ij} and x_{ij}, flow rates; F_j, L_j, V_j, U_j, W_j, enthalpies; H_{Fj}, H_{Vj}, H_{Lj}, pressures; p_{Fj}, p_j, temperatures; t_{Fj}, t_j and heat transfer rates; ϕ_j as stagewise variables and equilibrium stages 'N' as variable.

Total number of variables = $[3(C - 1) + 5 + 3 + 2 + 2 + 1) N + 1]$
$= (3C + 10) N + 1$

Difference between total number of variables and number of equations is $N(C + 7) + 1$. Hence, $N(C + 7) + 1$ variables must be specified.

On combining MESH equations to eliminate y_is and L_js, following $N \cdot C$ equations are obtained:

$$A_j x_{i\,j-1} + B_{i\,j} x_{ij} + C_{ij} x_{i,\,j+1} = D_{ij} \tag{8.64a}$$

where, $A_j = V_j + \sum_{m=1}^{j-1} (F_m - W_m - U_m) - V_i \quad 2 \le j \le N$

$$B_{ij} = -\left[V_{j+1} + \sum_{m=1}^{j} (F_m - W_m - U_m) - V_1 + U_j + (V_j + W_j) K_{ij} \right] \quad 1 \le j \le N$$

$$C_{ij} = V_{j+1} K_{i\,j+1} \quad 1 \le j \le N - 1$$

$$D_{ij} = -F_j Z_{ij} \quad 1 \le j \le N$$

Equation (8.64a) is a general representation of total $N \cdot C$ equations. Each set of N equations is a tridiagonal matrix equation. For example, if $N = 5$, for each component following equations can be written in tridiagonal matrix form:

$$\begin{bmatrix} B_1 & C_1 & 0 & 0 & 0 \\ A_2 & B_2 & C_2 & 0 & 0 \\ 0 & A_3 & B_3 & C_3 & 0 \\ 0 & 0 & A_4 & B_4 & C_4 \\ 0 & 0 & 0 & A_5 & B_5 \end{bmatrix} \begin{bmatrix} x_1 \\ x_2 \\ x_3 \\ x_4 \\ x_5 \end{bmatrix} = \begin{bmatrix} D_1 \\ D_2 \\ D_3 \\ D_4 \\ D_5 \end{bmatrix} \tag{8.64b}$$

Each set of above equations give the mole factions of one component in the liquids, leaving all the trays (equilibrium stages). Substitution of x_{ij} values in the phase equilibrium equations give the values of y_{ij}. Like that all unknown variables are determined. For solving such equations, a mathematical software (such as Mathcad®) is useful.

8.4.6 Step 6: Selection of Type of Tower

Basically two types of distillation columns are in use: packed towers and tray columns. Criteria for selection of the type of column were discussed in Section 8.3.

Packed column is widely used in distillation operation. Various packed column designs were discussed in Chapter 7. Figure 7.16 gives schematics for towers, packed with different types of packings. Characteristics of various packings are listed in Table 9.1 Number of theoretical stages are calculated as per general procedure outlined in the Section 8.4.5. Height Equivalent to Theoretical Stage (Plate) – HETP is obtained from a packing supplier. Both these values gives required height of a packed bed for the service. Refer to Table 8.36 for the recommended values of HETP. Packed tower internals for distillation service are different from those used for liquid-liquid extraction and are similar to those discussed in Chapter 9.

In tray towers, distillation trays provide hold-up for a portion of liquid through which vapour flows to accomplish the mass transfer required for separation. Variety of tray tower designs are available for distillation service. Trays are essentially used for distillation service when

(a) towers are very large in diameter (multi-pass trays),
(b) liquids contain solids or foulants,
(c) there are many internal transitions, and
(d) liquid loads are high.

Most commonly used tray tower is a cross-flow tray tower which is less expensive. Sieve trays, bubble cap trays and valve trays fall in this category. Modified sieve trays (e.g., Linde trays) and other high capacity trays are also developed for variety of applications.

Sieve trays A typical conventional cross-flow type sieve tray is shown in Fig. 8.16. This tray has become a dominate tower internal for mass transfer columns owing to its simple shape and ease of manufacture. Liquid travels horizontally across the tray and into a vertical *downcomes* (also called a *downspout*) while vapour flows up through the holes across the flow of liquid.

Fig. 8.16 *Typical Sieve Tray*

A detailed sketch of the sieve tray arrangement is shown in Fig. 8.26 for a specific design. Space occupied by downcomer vary from 8 to 12% of total tray area. Perforated holes occupy 5 to 15% of the active tray area which excludes two downcomer areas from total cross-sectional area. Hole diameter (D_H) varies from 4 to 8 mm. Distance between two trays normally varies from 150 to 600 mm in general but it can be more (up to 900 mm) for foamy liquids. Typical weir height (h_w) varies from 30 to 90 mm. Clear liquid depth ($h_L = h_W + h_{ow}$) also decides liquid inventory on the tray. It can be safely assumed that liquid head on the tray will be equal to h_L or higher (Refer to Eq. (8.70)). When h_L/D_H exceeds about 2, vapour momentum is exchanged with significant quantity of liquid, two-phase mixture is continuous in liquid and behaves as froth, offering good mass transfer.

There are several other flow patterns offered by different design engineers which are shown in Fig. 8.21. Reverse flow pattern, shown in Fig. 8.21(b), is typically developed by Linde, Germany and these trays are known as *Linde trays*. Double pass and four pass trays are selected when tower diameter is large and it is feared that liquid gradient (Δ) could be high in a single pass cross-flow tray. Linde trays are claimed to offer at least 10% more efficiency than that obtained with single cross-flow trays.

High capacity sieve (also known as dual-flow) trays are developed without downcomers. As a result tray diameter reduces considerable. In these type of trays, hole diameter varies from 12.5 to 25 mm. Holes are drilled in the entire active area of the tray. Dual-flow trays have a higher capacity and lower pressure drop, compared to conventional distillation trays. However, dual-flow trays have a very narrow efficient operating range. Vapour and liquid flow counter currently through the same holes. The majority of commercial applications of high capacity trays are in small to moderate sized columns. Alternately, slots are provided on the trays for countercurrent flow of both fluids.

Bubble cap trays Bubble cap trays were in use until the late 1950s when it was superseded by sieve and valve trays. It consists of a flat perforated deck in which the holes are enclosed with vapour risers and caps in the form of inverted cups mounted on top of the risers. The caps can either be equipped with slots or holes, through which vapour escapes. Vapour is forced into the surrounding cross-flowing liquid such that aerated liquid is trapped on the tray floor to a depth at least equal to the weir height. This gives the bubble cap tray the advantage to operate at extremely low liquid and vapour rates. Figure 8.17 shows a bubble cap tray along with enlarged view of a sample cap design and three other designs of bubble caps.

Bubble cap trays, generally, have the ability to handle wide ranges of liquid and vapour flow rates satisfactorily due to their leakproof performance. Standard cap sizes of 75, 100 and 150 mm are available. Typical distance between two bubble caps is kept 1.25 to 1.4 times of bubble cap size. Weir height (h_w) is kept more than bubble cap height such that it is immersed in liquid during operation. Downcomers are similar to those for cross-flow sieve trays.

Fig. 8.17 *Typical Bubble Cap Tray*

Valve trays Valve trays are essentially flat perforated trays with moveable or fixed valve units with or without a cage structure covering the holes. Moveable valves are disk-shaped-type devices which are enclosed within a cage structure or contain legs formed out of the valve disc. Fixed valves are units with integral legs formed out of the tray deck. Figure 8.18 represents a valve tray and Fig. 8.19 shows with different valves that are in common use.

Fig. 8.18 *Typical Floating Cage Valve Tray*

Fig. 8.19 *Different Types of Valves*

The open or vertical curtain area of the valve through which vapour escapes in a horizontal direction is defined by the restrictive legs integral with the valve unit or the leg rise of the cage structure attached to the tray deck. As the vapour rate is increased, the movable valve units rise and the upper limit of opening is controlled by the valve leg height. Valve trays perform over a wider operating range and has a higher turndown compared with the sieve trays.

Variations in valve diameter (d_v) from 38 to 50 mm, valve lift from 8 to 20 mm and valve slot area from 12 to 30% of active tray area are common in designs. Minimum and maximum distance between two valves is maintained at 15 mm and 30 mm, respectively. Weir height (h_w = 20 to 40 mm) for valve tray is slightly less than that for cross-flow sieve tray. There are several other design variations of valve trays offered by the manufacturers. For example in one design, alternate rows of valves can have different weight (up to double), claiming better vapour distribution on the tray.

Apart from the above three types of trays, there are many other tray designs offered by various suppliers. It is not possible to cover all designs in the book.

Selection of tray type

Different types of trays used in tray tower are (i) sieve tray, (ii) bubble cap tray, (iii) valve tray, (iv) Linde trays and other modified sieve trays.

Various factors that must be considered for the selection of tray type are: (a) cost, (b) capacity, (c) operating range, (d) efficiency, and (e) pressure drop.

(a) *Cost*: Bubble cap tray is the costliest type of tray. With mild steel as a material of construction, the ratios of cost

Bubble cap: Valve: Sieve trays ≅ 3:1.5:1 (general guideline)

In other words, cost of bubble cap tray is three times the cost of sieve tray and double the cost of valve tray with mild steel as material. With different materials, these ratios could be somewhat different.

(b) *Capacity*: If tower diameter required for given flow rates is more it means capacity of tray is less. The ranking for capacity is in the order of modified sieve tray, sieve tray, valve tray and bubble cap tray. In other words, for the given flow rates, tower diameter required is maximum with bubble cap trays and minimum with modified sieve tray. However, the difference in the values of tower diameters is not large.

(c) *Operating range*: It is an important factor for the selection of tray. Operating range of tray means range of liquid and vapour flow rates over which tray works satisfactorily. Bubble cap tray provides the maximum operating range while sieve tray provides the minimum operating range. Operating process plants are designed and operated with some maximum capacity and also for some minimum capacity. Hence, distillation columns are operated in the range of some maximum feed flow rate to minimum feed flow rate. The ratio of design flow rate to operating flow rate is called *turn-down ratio*. Some process plants are frequently operated at very high turndown ratio to meet the product's demand and to avoid the shut down and start-up conditions. For higher turn-down ratio, bubble cap trays are preferred. Valve trays also offer more flexibility or higher

turn down ratio than the sieve trays. However, in actual plant operation, movable float valves get stuck-up or fly-away if operating conditions are frequently changed. In such a case, valve trays loose efficiency.

(d) *Efficiency*: This factor is not generally considered for the selection of tray type. Efficiency of trays are almost same, if they are operated at design flow rates, or with minimum turn down. However, bubble cap columns give good efficiency over a wider range of turn down in general.

(e) *Pressure drop*: Pressure drop over the tray and for entire column is important for vacuum distillation for which a packed tower is the first choice. But for the low vacuum (up to 700 Torr) operation, tray tower can be selected to get other advantages. For example, to get the better vapour-liquid contact in large diameter column or to facilitate the side draws. Among the trays, sieve tray provides the lowest pressure drop, followed by valve tray, while bubble cap provides the highest pressure drop in general.

Sieve trays and modified sieve trays are the cheapest and are satisfactory for the majority of the applications. Nowadays, majority of chemical plants are having automatic and precise control system and hence, flow rates are not much fluctuating. However, to meet the fluctuations in the demand of product, some plants are intentionally operated with different flow rates or capacities. To handle the higher turn-down ratio, valve trays or bubble cap trays are considered. Valve tray perforations are covered with liftable caps. Vapour flow lifts the cap. Thus lifting cap directs the flow of vapour horizontally into the liquid which provides intense mixing as compared to a sieve tray. Valve trays are cheaper than bubble cap trays but they are not suitable for very low vapour rate. Bubble cap tray provides higher efficiency even with low vapour flow rate.

8.4.7 Step 7: Process Design of Tray Towers

Process design of a packed tower for absorber duty is detailed in Sec. 9.3.2 of Chapter 9. In analogous manner, a packed tower can be designed for distillation system. Process design of a sieve tray tower is detailed below.

Determining the tower diameter of sieve tray tower Flooding in sieve tray tower fixes the upper limit of vapour velocity through the tower. Velocity of vapour at which flooding in sieve tray tower occurs is called *flooding velocity*; v_F. Tower diameter of sieve tray column is decided based on the value of v_F. Actual velocity or design velocity through distillation column should be in the range of 70 to 90% of flooding velocity. Ideally it should be in between 80 and 85%.

Flooding is an inoperative condition. Flooding phenomena or flooding velocity are defined differently for different equipments. For example, in case of knock back condenser, flooding velocity is the velocity of vapour at which entrainment of liquid droplets in vapour just starts or the velocity of vapour at which % entrainment of liquid droplets in vapour becomes greater than zero. While in distillation column entrainment of liquid droplets in vapour up to 10% is normally accepted.

Flooding in tray tower of distillation column can occur or observed in either of the following ways[16]:

1. **Jet flooding:** In distillation operation froth of liquid-vapour mixture forms on each tray from which nearly clear vapour is separated and rises upward to meet the liquid on the next above tray. When froth of liquid-vapour mixture touches the next above tray (i.e. unstable operation) it is called *jet flooding* or *operational flooding*. Actually the vapour flows through perforations of tray forms a free flowing jet after leaving the orifice. Liquid droplets are entrained in these free flowing jets. These free flowing jets combinedly form the froth.
2. **Downcomer flooding:** In distillation column, liquid flows in downward direction by gravitational force but it flows against the pressure. When liquid flows from one tray to next below tray, it flows from lower pressure to higher pressure. Hence, to compensate that, it elevates certain level inside the downcomer. When the liquid level in downcomer on any tray rises above the weir, it is called *downcomer flooding* or *hydraulic flooding*.

Nowadays, it is possible to find tray or trays which restrict the capacity of existing tower or which decide the maximum capacity of existing tower. To find these trays of existing tower, one can intentionally operate the tower with maximum flow rates or even in flooding condition for a short period of time. During that time photograph of each section of tower can be taken with γ-rays. In these photographs, one can clearly see the froth zone above the trays and liquid level inside the downcomers. Based on the observation one can find a tray or more than one tray which decide the maximum capacity of tower. If these sieve trays, which restrict the capacity of sieve tray tower, are replaced by modified or high capacity sieve trays, then it is possible to increase the capacity of existing tower (may be up to 20%).

In calculations, tower diameter is decided based on jet flooding velocity. Downcomer flooding conditions are checked in a calculation of liquid back-up in downcomer (Refer Eq. (8.83)). Flooding velocity through sieve tray tower can be determined by the following equation:

$$v_F = C_f\left(\frac{\sigma}{0.02}\right)^{0.2}\left(\frac{\rho_L - \rho_V}{\rho_V}\right)^{0.5} \tag{8.65}$$

where, v_F = Flooding vapour velocity based on net cross-sectional area, m/s
C_f = Flooding constant which can be obtained from Fig. 8.20.
σ = Surface tension of liquid, N/m
ρ_v = Density of vapour, kg/m^3
ρ_L = Density of liquid kg/m^3

$$F_{LV} = \text{liquid-vapour flow factor} = \frac{\dot{m}_L}{\dot{m}_V}\left(\frac{\rho_v}{\rho_L}\right)^{0.5} \tag{8.66}$$

$\dot{m}_L$ = mass flow rate of liquid, kg/s
$\dot{m}_V$ = mass flow rate of vapour, kg/s

Figure (8.20) gives flooding velocity with ±10% error, subject to the following restrictions:

(a) System is non-foaming.
(b) Weir height is less than 15% of plate spacing.
(c) Sieve plate perforations are 13 mm or less in diameter.

(d) Value of C_f obtained from Fig. (8.20) is valid only if $A_h/A_a \geq 0.1$. If $A_h/A_a <$ 0.1 then C_f value obtained from Fig. (8.20) must be corrected by multiplying it with following correction factor.

A_h/A_a	*Correction Factor*
0.08	0.9
0.06	0.8

where, A_h = Total area of all active holes
A_a = Active or bubbling area of tray
A_n = Net area available for vapour-liquid disengagement

For a single pass tray, $A_n = A_c - A_d$ and $A_a = A_c - 2A_d$ (max.)

where, A_c = Total column cross-sectional area
A_d = Cross-sectional area of downcomer. Downcomer area A_d usually ranges from 8 to 12 % of A_c

Actual active area excludes calming area, area occupied by support devices, etc., and is less than A_a.

For a double pass tray, $A_a = A_c - 4\,A_d$ (max.)

In sieve trays, hole layout can be triangular pitch (similar to tube layout on a tubesheet of shell and tube heat exchanger) or could have equal diametrical pitch, depending on A_n/A_c ratio.

Fig. 8.20 *Flooding Velocity (Sieve Plates)*[8]
(Reproduced with the permission of McGraw-Hill Education, USA)

Selection of liquid flow pattern After finding the tower diameter, liquid flow pattern over sieve tray is decided. Common liquid flow patterns of cross-flow sieve tray are shown in Fig. 8.21. In most of the cases single pass (cross flow, (Fig. 8.21(a)) pattern is selected. Liquid flow pattern depends on the liquid flow rate and tower diameter. Table 8.34 can be used for the selection of liquid flow pattern.

Fig. 8.21 *Common Liquid Flow Patterns in Cross-Flow Plates*[8]
(Reproduced with the permission of McGraw-Hill Education, USA)

Table 8.34 Selection Guidelines for Liquid Flow Pattern

Column diameter, m	Range of liquid flow rate, m^3/s			
	Reverse flow	Cross flow		Cascade double pass
		Single pass	Double pass	
0.9	$0 - 1.90 \times 10^{-3}$	$1.90 \times 10^{-3} - 0.0126$		
1.2	$0 - 2.50 \times 10^{-3}$	$2.50 \times 10^{-3} - 0.0190$		
1.8	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0250$	0.025 – 0.0440	
2.4	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	0.0315 – 0.050	
3	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	0.0315 – 0.057	0.057 – 0.0880
3.66	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	0.0315 – 0.063	0.063 – 0.1000
4.6	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	0.0315 – 0.070	0.070 – 0.1136
6	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	0.0315 – 0.070	0.070 – 0.1260

Tower diameter also depends on tray spacing. Value of flooding constant depends on the value of tray spacing (Fig. 8.20). Tray spacing ranges from 0.15 m to 1 m. At design stage, if the tray spacing is increased, required tower diameter decreases, tray efficiency increases and hence, actual number of trays required for desired separation decreases but overall height for the tower increases. Ideally one should find the optimum tray spacing or the tray spacing for which total cost of tower is minimum. If column diameter is less than 1 m, tray spacing is kept 0.2 to 0.3 m. For columns above 1 m diameter, tray spacing of 0.3 to 0.6 m is used. For the first trial calculations one can start with tray spacing equal to 0.3 m but if column diameter is calculated to be more than 1 m then for the second trial calculations, tray spacing can the taken equal to 0.4 to 0.5 m. Even with smaller tray spacing of 0.15 to 0.3 m, larger spacing is needed between certain plates to accommodate nozzles for feed and side streams, for manholes, etc.

Checking of liquid entrainment As a rough guide, the % of liquid entrainment should be less than 10%.

$$\% \text{ liquid entrainment} = \frac{\text{kg of liquid droplets entrained}}{\text{kg of gross liquid flow}} \times 100 \tag{8.67}$$

or % liquid entrainment = 100ψ

where, ψ = Fractional liquid entrainment

$$\psi = \frac{\text{kg of liquid entrained}}{\text{kg of gross liquid flow}} = \frac{\text{Moles of liquid entrained}}{\text{Moles of gross liquid flow}}$$

Value of ψ as a function of liquid-vapour factor (F_{LV}) with % of flood as a parameter is given by Fair[17]. The same is reproduced in Fig. 8.22. ψ should be less than 0.1 to get higher tray efficiency. Optimum value of ψ can be greater than 0.1.

$$\% \text{ of flood} = \frac{\text{Actual vapour velocity based on net area}}{\text{Flooding velocity}} \times 100$$

$$= \frac{v_n}{v_f} \times 100 \tag{8.68}$$

An elaborate empirical approach for entrainment is given by Bennett et al in Ref. 16.

Weir design The height of weir decides the height of liquid over the tray. In most cases, weirs are straight and rectangular plates. With bubble cap trays, to compensate the fluctuations in the flow rate, V–notch-type weirs are also used. Increase in weir height increases depth of liquid pool (h_L) and contact time. Hence, it increases tray efficiency of the tray with which it is attached. However, increase in weir height increases liquid entrainment in vapour, leaving the tray and hence decreases tray efficiency of the next above tray. Therefore, effect of increase of weir height on the tray efficiency cannot be predicted. However, increase in weir height increases pressure drop. For a distillation column operating above atmospheric pressure, weir height is kept in at 30 mm to 90 mm; while 30 mm to 50 mm is more common. For vacuum operation, to control the

Fig. 8.22 *Entrainment Correlation for Sieve Plates*[8]
(Reproduced with the permission of Chemical Engineering by Access Intelligence, USA)

tray pressure drop, weir height is reduced and it is kept in between 6 and 12 mm. In old designs, inlet weir was also used, but in recent designs inlet weir is not recommended as it results in *hydraulic jump*.

Weir length fixes area of segmental downcomer. Weir length is normally kept in between 0.6 to 0.85 times column diameter. For the first trial calculations, weir length can be taken as $0.77D_i$ and equivalent down comer area is 12%. Decrease in weir length increases the net area of tray and decreases the tower diameter, but it decreases the downcomer area, increases the pressure drop in downcomer and also decreases the volume of downcomer. It also elevates the liquid level in downcomer. Increase in weir length increases the tower diameter. Hence, balanced weir length and weir height are required. Relation between weir length and downcomer area is given in Table 8.35.

Table 8.35 Relationship of Weir Length and Downcomer Area

$\frac{\text{Weir length}}{ID \text{ of tower}} = \frac{l_w}{D_i}$	$\frac{\text{Downcomer area } A_d}{\text{Column area } A_c} \times 100$
0.600	5.25
0.650	6.80
0.700	8.80
0.705	9.00
0.715	9.50
0.727	10.00
0.745	11.00
0.770	12.00
0.780	13.00
0.800	14.00
0.815	15.00
0.826	16.00
0.840	17.00
0.850	18.00

Clear liquid depth over the tray is equal to height of the weir, h_w, plus the depth of the crest of liquid over the weir, h_{ow}. The height of the liquid crest over the weir can be estimated by using Francis formula (Ref. 9).

$$h_{ow} = 750\left(\frac{\dot{m}_L}{\rho_L l_w}\right)^{\frac{2}{3}} \tag{8.69}$$

where, h_{ow} = Weir crest of liquid, mm
$\dot{m}_L$ = Mass flow rate of liquid, kg/s
l_w = Weir length, m
ρ_L = Liquid density, kg/m^3

$$h_L = h_w + h_{ow} \tag{8.70}$$

Checking for weeping At a very low value of vapour velocity, liquid rains down through the perforations. This phenomenon is known as *weeping*. It is again an inoperative condition. Minimum vapour velocity required to avoid the weeping is given by Eduljee's equation:

$$v_{h\,\min} = \frac{K - 0.9(25.4 - d_h)}{\sqrt{\rho_V}} \tag{8.71}$$

where, $v_{h\,\min}$ = Minimum vapour velocity through the holes, m/s
d_h = Hole diameter, mm
ρ_V = Density of vapour, kg/m^3

K = Constant, a function of depth of clear liquid (h_L) on the plate, which can be obtained from Fig. (8.23).

Fig. 8.23 *Weep-Point Correlation (Eduljee, 1959)*[10]
(Reproduced with the permission of Elsevier, UK)

Another empirical approach for checking weeping is developed by Bennett *et al.* (Ref. 16) based on liquid Froude number.

$$Fr_L = \left[\frac{(\rho_V \; v_h^2)}{(\rho_L \; g \; h_L)} \right]^{0.5} \tag{8.72}$$

where, ρ_V = Vapour density, kg/m^3
v_h = Vapour velocity through holes, m/s
ρ_L = Liquid density, kg/m^3
g = Gravitational constant = 9.81 m/s^2
h_L = Liquid head over the plate, m

Froude number should be greater than 0.5 to ensure no weeping.

Basically Froude number (Fr_L) specifies the ratio of inertia force to gravity force on an element of fluid. Hence, this equation is more appropriate in defining the weeping condition.

Total pressure drop in sieve tray tower Total pressure drop in sieve tray tower, Δp_T is given by the following equation:

Δp_T = Pressure drop in inlet nozzle + Pressure drop/tray × No. of trays + Pressure drop in outlet nozzle

Major pressure drop in vapour is provided by trays.

$$\Delta p_T \cong N \times \Delta p_t \tag{8.73}$$

where, N = Total number of sieve trays of tower

Δp_t = Tray pressure drop, Pa

Tray pressure drop in Pa or N/m^2 can be determined from the tray pressure drop in mm of liquid column by following equation:

$$\Delta p_t = 9.8 \times 10^{-3}\, h_t\, \rho_L \tag{8.74}$$

where, Δp_t = Tray pressure drop, N/m^2 or Pa

h_t = Tray pressure drop, mm of liquid column (LC)

ρ_L = Density of liquid, kg/m^3

To find the tray pressure drop simple additive model is used.

$$h_t = h_d + (h_w + h_{ow}) + h_r \tag{8.75}$$

where, h_d = Pressure drop through dry plate, mm LC

h_w = Height of weir, mm

h_{ow} = Height of liquid crest, mm

h_r = Residual pressure drop, mm LC

Tray pressure drop (h_t) is the sum of pressure drops which occurs as vapour rises through the perforations of dry plate (h_d), pressure drop which occurs as vapour rises through the pool of clear liquid ($h_w + h_{ow}$) and pressure drop which occurs in vapour-liquid disengagement space (h_r). Due to the high vapour velocity, froth formation takes place over the tray. Some liquid particles are entrained in vapour phase. Hence, some additional energy loss is taking place in carrying the liquid particles. These liquid particles are disengaged from the vapour after travelling a certain distance. This additional energy loss is counted by residual pressure drop, h_r.

Pressure drop through dry plate is given by following equation:

$$h_d = 51\left(\frac{v_h}{C_o}\right)^2 \frac{\rho_V}{\rho_L} \tag{8.76}$$

where, h_d = Dry plate pressure drop, mm LC

v_h = Velocity of gas through the holes, m/s

$$= \frac{\text{Volumetric flow rate of gas, m}^3\text{/s}}{\text{Total hole area, m}^2} = \frac{q_v}{A_h} \tag{8.77}$$

ρ_V, ρ_L = Densities of vapour phase and liquid phases, respectively, kg/m^3

C_o = Orifice coefficient which is a function of ratio of plate thickness to hole diameter and % perforated area of plate. It can be determined by using Fig. 8.24.

$$\%\ \text{Perforated area} = \frac{A_h}{A_a} \times 100 \tag{8.78}$$

where, A_h = Total area of all active holes of tray, m^2

A_a = Active area of tray = $A_c - 2\,A_d$, m^2 for single pass

A_c = Inside area of column, m^2

A_d = Downcomer area of tray, m^2

Fig. 8.24 *Discharge Coefficient (Sieve Plates)*[8]
(Reproduced with the permission of McGraw-Hill Education, USA)

Residual pressure drop can be estimated by the following equation:

$$h_r = \frac{12.5 \times 10^3}{\rho_L} \tag{8.79}$$

where, h_r = Residual pressure drop, mm LC
ρ_L = Density of liquid, kg/m^3

Design of downcomers There are two functions of a downcomer:

1. It provides the passage for the flow of liquid.
2. Foamy or frothy liquid enters into downcomer. Hence, downcomer also acts as a vapour-liquid separator. To facilitate the vapour-liquid disengagement, certain minimum residence time (at least 3 seconds) is required in the downcomer.

Following types of downcomers are used:

(a) *Straight segmental downcomer:* It is the simplest and cheapest and is preferred for most applications. In this type, downcomer channel is formed by a flat plate. The portion of the flat plate which extends down below the plate from

the outlet weir is called *apron*. With this type of downcomer, apron is straight and vertical.

(b) *Inclined segmental downcomer:* With this type of segmental downcomer, apron is inclined towards the wall. It is preferred with foamy liquid, with which vapour-liquid disengagement is difficult. Inclined or sloppy apron provides more resistance to flow and makes the phase separation easier. It also increases tray area available for perforations. But with this type, liquid back-up in downcomer is higher compared to straight segmental downcomer.

(c) *Circular downcomers or pipes:* They are used for small liquid flow rates in a few cases.

Liquid is falling from tray to tray by passing through the downcomer. Due to the pressure drop across the tray, liquid is flowing from lower pressure to higher pressure. To overcome the pressure difference liquid elevates its level in the downcomer. This is called liquid back-up in the downcomer. Also some pressure drop occurs when liquid flows through the downcomer area. Downcomer pressure drop increases the liquid back-up further. Head loss or pressure drop in the downcomer is estimated by following equation:

$$h_{dc} = 166\left[\frac{\dot{m}_d}{\rho_L A_m}\right]^2 \tag{8.80}$$

where, h_{dc} = Downcomer pressure drop, mm LC

$\dot{m}_d$ = Liquid flow rate in downcomer, kg/s

ρ_L = Density of liquid, kg/m^3

$A_m = A_d$ or A_{ap} whichever is smaller, m^2

A_d = Downcomer area, m^2

A_{ap} = Clearance area under downcomer apron, m^2

$$A_{ap} = h_{ap}\, l_w/10^6 \tag{8.81}$$

where, h_{ap} = Height of the bottom edge of the apron above the plate, mm

l_w = Length of weir, mm

$$h_{ap} = h_w - (5 \text{ to } 10) \text{ mm} \tag{8.82}$$

where, h_w = Height of weir, mm

Liquid back-up in downcomer in terms of clear liquid is given by following equation:

$$h_b = (h_w + h_{ow}) + h_t + h_{\mathrm{dc}} \tag{8.83}$$

= Downcomer back-up measured from plate surface, mm (i.e., elevation of clear liquid in downcomer)

Due to the formation of froth and foam, actual height or foamy or frothy liquid in downcomer is quite great, nearly double than h_b.

Density of froth or "aerated" liquid is in between 0.4 to 0.7 times that of the clear liquid. For design purpose, it is taken as 0.5 times the density of liquid. Hence, to avoid the downcomer flooding.

$$h_b \ngtr \frac{I_t + h_w}{2} \tag{8.84}$$

where, h_b = Liquid back-up in the downcomer measured from plate surface, mm
I_t = Tray spacing, mm
h_w = Height of weir, mm

Certain minimum residence time must be allowed in the downcomer to facilitate the disengagement of vapour and liquid phases. Residence time of at least 3 seconds is recommended. Residence time in downcomer is given by following equation:

$$\theta_r = \frac{A_d \cdot h_{bc} \cdot \rho_L}{\dot{m}_d} \tag{8.85}$$

where, A_d = Downcomer area, m^2
h_{bc} = Liquid back-up in downcomer in terms of clear liquid, m
ρ_L = Density of liquid, kg/m^3
$\dot{m}_d$ = Liquid flow rate in downcomer, kg/s

Design of bubble cap tray column and valve tray column Design of bubble cap columns can be made in accordance with the procedure described in Refs. 2, 8, 20 to 23. Hydraulic design of valve tray column is quite complicated and most data for different valve systems are proprietary. Reference 23 gives some date for valve tray columns. A preliminary design can be made on the basis of these data and it can be checked with the supplier of the valve tray based on his hydraulic data.

8.4.8 Step 8: Tray Efficiency

Like VLE data, in case of tray efficiency also, it is better to predict the tray efficiency based on actual data on the same system or similar system of commercial scale plant or of pilot plant. There is no entirely satisfactory method available for finding the tray efficiency. Various correlations are available for finding tray efficiency, but they are used only if the reliable actual data on the same or similar system are not available. Overall efficiency of a tray tower is given by the following equation:

$$\eta_{oc} = \frac{N_t}{N_a} \tag{8.86}$$

where, N_t = Number of theoretical stages required for the desired separation.
N_a = Actual number of trays required for the desired separation.

Tray efficiency depends on (i) system composition and properties, (ii) rate of throughput, and (iii) geometry of tray tower (geometry of tray, tray spacing, etc.).

Two methods are given here to find the tray efficiency:

1. Van Winkle's method
2. AIChE method.

If actual data for predicting tray efficiency for the given system are not available, then it can be predicted approximately by using these two methods. For the safe design calculation, it is suggested that if the value of tray efficiency obtained based on any of these methods is more than 0.5 then consider the value of overall efficiency as 0.5.

1. Van Winkle's Correlation or Method[19] Van Winkle's correlation can be used to predict the tray efficiency of a binary system of distillation. This correlation can be used for both bubble-cap and sieve trays.

$$\eta = 0.07\, D_g^{\,0.14}\, Sc_L^{\,0.25}\, Re^{0.08} \tag{8.87}$$

where, D_g = Surface tension number = $\sigma_L/(\mu_L \cdot u_V)$ (8.88)

σ_L = Liquid surface tension, N/m

μ_L = Liquid viscosity, N · s/m^2 or kg/(m · s)

u_v = Superficial vapour velocity, m/s

Sc_L = Liquid Schmidt number = $\mu_L/(\rho_L \cdot D_{LK})$ (8.89)

D_{LK} = Liquid diffusivity of light key component, m^2/s

$$Re = \text{Reynolds number} = \frac{h_w u_v \rho_V \times 10^{-3}}{\mu_L (f_A)} \tag{8.90}$$

where, h_w = Weir height, mm

ρ_V = Vapour density, kg/m^3

f_A = Fractional area = A_h/A_c

2. AIChE Method[10] The point efficiency $\dot{\eta}$ is related to number of transfer units by the following equation:

$$\frac{1}{\ln(l - \dot{\eta})} = -\left[\frac{1}{N_G} + \frac{mV}{L} \times \frac{1}{N_L}\right] \tag{8.91}$$

where, $\dot{\eta}$ = Point efficiency

N_G = Number of gas phase transfer units

m = Slope of equilibrium curve at the point in consideration

V, L = Molar flow rates of vapour and liquid phases, respectively at the point in consideration

N_L = Number of liquid phase transfer units

The number of gas phase transfer units is given by the following equation:

$$N_G = \frac{(0.776 + 4.57 \times 10^{-3} h_w - 0.24 F_V + 105 L_p)}{\left(\dfrac{\mu_v}{\rho_v D_V}\right)^{0.5}} \tag{8.92}$$

where, h_w = Weir height, mm

$F_V = v_a\sqrt{\rho_V}$

v_a = Vapour velocity based on active tray area, m/s

$$v_a = \frac{q_v}{A_a} = \frac{\text{Volumetric flow rate of vapour}}{\text{Active tray area}}$$

L_p = Volumetric liquid flow rate across the plate divided by average width of plate, m^3/(s · m)

$$\text{Average width of plate} = \frac{A_a}{Z_L} \tag{8.93}$$

Z_L = Length of liquid path across the tray, m
μ_V = Viscosity of vapour, N · s/m^2
ρ_V = Density of vapour, kg/m^3
D_V = Vapour diffusivity, m^2/s

The number of liquid phase transfer unit is given by the following equation:

$$N_L = (4.13 \times 10^8\, D_L)^{0.5}\,(0.21\, F_V + 0.15)\theta_L \tag{8.94}$$

where, D_L = Liquid diffusivity, m^2/s
θ_L = Liquid contact time, s

Liquid contact time θ_L can be determined by the following equation:

$$\theta_L = \frac{Z_c Z_L}{L_p} \tag{8.95}$$

where, Z_L = Length of liquid path from inlet downcomer to outlet weir, m
Z_c = Liquid hold-up on the plate, m^3 per m^2 active area

For sieve trays,

$$Z_c = 0.006 + 0.73 \times 10^{-3}\, h_w - 0.24 \times 10^{-3}\, F_V\, h_w + 1.22\, L_p \tag{8.96}$$

Tray efficiency η is equal to the point efficiency $\dot{\eta}$, if the liquid on tray is perfectly mixed. For the real tray, this may not be true.

Tray efficiency η can be determined from point efficiency by using Fig. (8.25). In this figure, graph of $\frac{\eta}{\dot{\eta}}$ vs $\frac{mV}{L}\dot{\eta}$ is drawn for different values of Peclet number. For a tray, Peclet number is given by the following equation:

$$Pe = \frac{Z_L^2}{D_e \theta_L} \tag{8.97}$$

where, D_e = Eddy diffusivity, m^2/s

For sieve trays, the Eddy diffusivity can be estimated by the following equation.

$$D_e = (0.0038 + 0.017\, v_a + 3.86\, L_p + 0.18 \times 10^{-3}\, h_w)^2 \tag{8.98}$$

In actual operation liquid droplets get entrained in vapour phase. This reduces the value of tray efficiency η. Actual tray efficiency η_a considering the liquid entrainment can be calculated by following equation.

$$\eta_a = \frac{\eta}{1 + \eta\left(\frac{\psi}{1-\psi}\right)} \tag{8.99}$$

where, ψ = Fractional liquid entrainment.

In Sec. 8.4.7, various parameters relating to the process design of a distillation tower were discussed in general. However, detailed design of the tower internals are well covered in the book by Billet[20]. For preparation of the specifications of a tray tower, useful guidelines can be derived from the article by Mukherjee[21].

Fig. 8.25 *Relationship between Plate and Point Efficiencies*[10]
(Reproduced with the permission of McGraw-Hill Education, USA)

8.4.9 HETP of Packings

For the distillation in packed tower, height of packed bed (Z), required for the desired separation, is determined by following equation:

$$Z = N_t \times \text{HETP} \tag{8.100}$$

where, N_t = Number of theoretical stages required for the desired separation

HETP = Height equivalent to a theoretical plate.

The height of an equivalent equilibrium stage or theoretical plate (HETP) is the height of packing that gives the same separation as an equilibrium stage. HETP for the given type and size of random packing is nearly constant and is nearly independent of the system physical properties. HETP values available in literature are valid only for good liquid distribution and for the reasonable pressure drop.

HETP values for various packing can be obtained from Table 8.36.

Table 8.36 Recommended HETP for Various Packings

Sr. No.	Packing types	Packing size, mm	HETP, m
1.	Pall ring, random	25	0.40 – 0.50
	(for Δp > 17 mm WC	38	0.60 – 0.75
	per m of packing)	50	0.75 – 1.00

(*Contd.*)

Table 8.36 (*Contd.*)

Sr. No.	Packing types	Packing size, mm	HETP, m
2.	Berl saddle and	25	0.40 – 0.50
	Intalox sadle, random	38	0.60 – 0.75
	(for $\Delta p > 29$ mm WC	50	0.75 – 1.00
	per m of packing)		
3.	Raschig rings	25 to 50	1.0
4.	Structured packings*		≤ 0.5

*HETP of a structured packing depends on a factor known as F_V which is defined as

$$F_V = v_g \sqrt{\rho_V} \tag{8.101}$$

where, v_g = Superficial vapour velocity, m/s

ρ_V = Vapour density, kg/m^3

HETP equal to around 0.5 m is reported for F_V factor, not exceeding 3, beyond which pressure drop increases steeply.

Bennett (Ref. 16) has given empirical equations for estimation of HETP for different types of packings.

Example 8.13 Design a crossflow-type sieve tray tower for the distillation of acetic acid-water system, specified in Example 8.5. Maximum feed flow rate is 12 000 kg/h and minimum feed flow rate is 8400 kg/h (70% of the maximum).

Solution

Data obtained from Example 8.5:

(i) Feed composition: 80% acetic acid and 20% water (by mass) or
54.55% acetic acid and 45.45% water (by mole)

(ii) Mole fractions of water: In distillate $x_D = 0.9302$, In residue $x_W = 1.666 \times 10^{-4}$

(iii) $q = 1.272$ for liquid feed at 30°C

(iv) Reflux ratio, $R = 4.2$

(v) Number of theoretical stages required for desired separation, $N = 24$

Flow rates

Average molar mass of feed,

$$M_{av} = \Sigma x_i M_i = 0.5455 \times 60 + 0.4545 \times 18 = 40.911 \text{ kg/ kmol}$$

Molar flow rate of feed, $F = \dfrac{12\ 000}{40.911} = 293.32$ kmol/h

$$F = D + W$$
$$Fx_F = Dx_D + Wx_W$$
$$D + W = 293.22$$
$$0.9302\, D + 1.666 \times 10^{-4}\, W = 0.4545 \times 293.32 = 133.314$$
$$0.9302\, D + 1.666 \times 10^{-4}\, (293.32 - D) = 133.314$$

$D = 143.29$ kmol/h

$W = 293.32 - 143.29 = 150.03$ kmol/h

Molar flow rates of vapour and liquid at the top in enriching section:

$L = RD = 4.2 \times 143.29 = 601.818$ kmol/h

$V = (R + 1)\, D = 5.2 \times 143.29 = 745.108$ kmol/h

Molar flow rates of vapour and liquid in stipping section:

$\bar{L} = L + Fq = 601.818 + 293.32 \times 1.272 = 974.92$ kmol/h

$\bar{V} = F\,(q - 1) + V = 293.32\,(1.272 - 1) + 745.108 = 824.89$ kmol/h

Tower diameter calculation

(a) Tower diameter required at top

Operating pressure at top of column = 1 atm = 101.325 kPa

Here total condenser is used, hence, composition of vapour leaving the topmost tray and composition of liquid entering to the topmost tray (reflux) are equal.

$$\frac{\dot{m}_L}{\dot{m}_V} = \frac{L}{V} = \frac{601.818}{745.108} = 0.8077$$

where, $\dot{m}_L, \dot{m}_V$ = Mass flow rates of liquid and vapour at the top, kg/s

Density of vapour, ρ_v

$$\rho_v = \frac{pM_{\text{av}}}{RT}$$

Average molar mass of vapour at top

$M_{\text{av}} = \Sigma x_i M_i = (1 - 0.9302) \times 60 + 0.9302 \times 18 = 20.9316$ kg/ kmol

$$\rho_v = \frac{20.9316}{(t_T + 273)} \times \frac{273}{22.414}$$

To find the temperature of vapour at top, in t–x–y data, put $y = x_D = 0.9302$ and find the corresponding value of temperature from Table 8.6.

$y = x_D = 0.9302 \quad t = t_T = 100.64°\text{C}$

$\rho_v = 0.6823$ kg/m^3

Density of liquid at top, ρ_L

$$\rho_L = \frac{1}{\Sigma \dfrac{w_i}{\rho_i}} \tag{6.55}$$

Density of pure water at 100.64°C, $\rho_w = 958$ kg/m^3

Density of pure acetic acid at 100.64°C, $\rho_{AA} = 1010$ kg/m^3

Mass fraction of water in distillate = 0.8

Mass fraction of acetic acid in distillate = 0.2

$$\rho_L = \frac{1}{\left(\dfrac{0.8}{958}\right) + \left(\dfrac{0.2}{1010}\right)} = 967.97 \text{ kg/m}^3$$

Liquid–vapour flow factor at top,

$$F_{LV} = \frac{\dot{m}_L}{\dot{m}_V}\left(\frac{\rho_v}{\rho_L}\right)^{0.5} \tag{8.66}$$

$$F_{LV} = 0.8077\left(\frac{0.6823}{967.97}\right)^{0.5} = 0.021\ 444$$

For the first trial calculations, tray spacing = 0.3 m (assumed)
From Fig. 8.20,

$$C_f = 0.0635$$

Flooding velocity,

σ = Surface tension of liquid, N/m

$$\sigma^{1/4} = \Sigma\sigma_i^{1/4} x_i = (1 - 0.9302)\ \sigma_{AA}^{1/4} + 0.9302\ \sigma_W^{1/4}$$

where, σ_W = Surface tension of pure water, N/m = 58×10^{-3} N/m

σ_{AA} = Surface tension of pure acetic acid, N/m

σ_{AA} at 100.64°C:

$$\sigma_{AA}^{1/4} = [\mathcal{P}](\rho_L' - \rho_V') \tag{6.93}$$

σ_{AA} = Surface tension of acetic acid, dyn/cm

ρ_L' = Density of liquid acetic acid, mol/cm^3

$$\rho_L' = \frac{1010 \times 10^{-3}}{60} = \frac{1.01}{60} = 0.016\ 833\ \frac{\text{mol}}{\text{cm}^3}$$

ρ_V' = Density of acetic acid vapour, mol/cm^3

$$\rho_V' = \frac{1}{(273 + 100.64)} \times \frac{T_s}{p_s \cdot V_s}$$

$$\rho_V' = \frac{1}{(273 + 100.64)} \times \frac{273}{22\ 414} = 3.26 \times 10^{-5}\ \text{mol/cm}^3$$

$[\mathcal{P}]$ = Parachor can be calculated from Table 3–343, of Ref. 14.
$[\mathcal{P}]$ = 55.5 (Contribution of CH_3 –) + 73.8 (Contribution of – COOH)
$[\mathcal{P}]$ = 129.3

$$\sigma_{AA}^{1/4} = 129.3\ (0.016\ 833 - 3.26 \times 10^{-5}) = 2.1723$$

Surface tension of mixture,

σ_{AA} = 22.268 dyn/cm ≡ 22.268×10^{-3} N/m

$$\sigma^{1/4} = (1 - 0.9302) \times (22.268 \times 10^{-3})^{1/4} + 0.9302 \times (58 \times 10^{-3})^{1/4}$$

$\sigma = 55 \times 10^{-3}$ N/m

Equation (8.65):

$$v_F = 0.0635\left(\frac{55 \times 10^{-3}}{0.02}\right)^{0.2}\left(\frac{967.97 - 0.6823}{0.6823}\right)^{0.5}$$

$$= 2.93 \text{ m/s}$$

Let actual vapour velocity through tower, $v = 0.85\ v_F = 0.85 \times 2.93$
$= 2.49$ m/s

Volumetric flow rate of vapour at top

$$q_V = \frac{VM_{av}}{\rho_v} = \frac{745.108 \times 20.9316}{0.6823} = 22\,858.4 \text{ m}^3/\text{h} \equiv 6.3496 \text{ m}^3/\text{s}$$

Net area required at top

$$A_n = \frac{q_V}{v} = \frac{6.3496}{2.49} = 2.55 \text{ m}^2$$

Let downcomer area, $A_d = 0.12\, A_c$

$$A_n = A_c - A_d = A_c - 0.12\, A_c = 0.88\, A_c$$

where, A_c = Inside cross-sectional area of tower

$$0.88\, A_c = 2.55$$

$$A_c = \frac{2.55}{0.88} = 2.8977 \text{ m}^2$$

Inside diameter of column required at top

$$D_i = \sqrt{\frac{4 \times A_c}{\pi}} = \sqrt{\frac{4 \times 2.8977}{\pi}} = 1.92 \text{ m say } 1.95 \text{ m at the top}$$

(b) Tower diameter required at bottom

Operating pressure at the base of column = Operating pressure at top + Δp_T

where, Δp_T = Total pressure drop in sieve tray tower

h_t = 120 mm WC per tray

$\Delta p_T \cong$ Actual no. of trays $\times\, \rho_L\, g\, h_t$

Assuming tray efficiency = 0.5

Actual number of trays, $N_a = \dfrac{N}{0.5} = \dfrac{24}{0.5} = 48$

$$\Delta p_T = 48 \times 1000 \times 9.81 \times 120 \times 10^{-3}$$
$$= 56\,506 \text{ Pa} \equiv 56.5 \text{ kPa (assumed)}$$

Operating pressure at base

$$p_t' = p_t + \Delta p_T = 101.325 + 56.5 = 157.825 \text{ kPa} \equiv 1.558 \text{ atm}$$

Bottom product is nearly pure acetic acid. Antoine equation[14] for acetic acid

$$\ln p_v = 15.8667 - \frac{4097.86}{T - 27.4937}$$

When $p_v = p_t'$, $T = T_b$ = Base temperature of column

$$\ln (157.825) = 15.8667 - \frac{4097.86}{T - 27.4937}$$

$$T = 406.74 \text{ K} \quad t = 133.6°\text{C (Estimated)}$$

Molar flow rates of vapour and liquid at bottom

$$\bar{V} = 824.89 \text{ kmol/h}, \quad \bar{L} = 974.92 \text{ kmol/h}$$

At the base of column,

$$\frac{\dot{\bar{m}}_L}{\dot{\bar{m}}_V} \cong \frac{\bar{L}}{\bar{V}} = \frac{974.92}{824.89} = 1.182$$

where $\dot{\bar{m}}_L$, $\dot{\bar{m}}_V$ = Mass flow rates of vapour and liquid at bottom, kg/s

Density of vapour at bottom

$$\rho_V = \frac{p_t' M}{RT_b} = \frac{157.825 \times 60}{(273 + 133.6)} \times \frac{273}{101.325 \times 22.414}$$

$$\rho_V = 2.8368 \text{ kg/m}^3$$

Density of liquid (nearly pure acetic acid) at bottom

ρ_L = Density of acetic acid at 134.26°C and at 160.185 kPa
= 1000 kg/m^3 – saturated liquid

Liquid–vapour flow factor at bottom; Eq. (8.66):

$$F_{LV} = 1.182\left(\frac{2.8368}{1000}\right)^{0.5} = 0.062\,96$$

For tray spacing = 0.3 m, from Fig. 8.20;

$C_f = 0.0616$

Flooding velocity

Surface tension of pure acetic at 134.26°C temperature:

$$\sigma_{AA}^{1/4} = [\mathcal{P}](\rho_L'' - \rho_V'')$$

$$\rho_L'' = 1000 \times 10^{-3} \times \frac{1}{60} = 0.016\,67 \text{ mol/cm}^3$$

$$\rho_V'' = 2.8368 \times \frac{10^{-3}}{60} = 4.728 \times 10^{-5} \text{ mol/cm}^3$$

$$\sigma_{AA}^{1/4} = 129.3\ (0.016\,67 - 4.728 \times 10^{-5}) = 2.1493$$

$$\sigma_{AA} = 21.34 \text{ dyn/cm} \equiv 21.34 \times 10^{-3} \text{ N/m}$$

$$v_F = 0.0616\left(\frac{21.34 \times 10^{-3}}{0.02}\right)^{0.2}\left(\frac{1000 - 2.8368}{2.8368}\right)^{0.5}$$

$$v_F = 1.17 \text{ m/s}$$

Actual vapour velocity, $v = 0.85 \times 1.17 = 0.9945$ m/s

Volumetric flow rate of vapour at bottom

$$\dot{Q} = \frac{\bar{V} M_{\text{av}}}{\rho_V''} = \frac{(824.89 \times 60)/3600}{2.8368} = 4.8464 \text{ m}^3\text{/s}$$

Net area required at bottom

$$A_n = \frac{4.8464}{0.9945} = 4.8732 \text{ m}^2$$

Inside area of column

$$A_c = \frac{4.8732}{0.88} = 5.538 \text{ m}^2$$

Inside diameter of column required at base

$$D_i = \sqrt{\frac{4 \times A_c}{\pi}} = \sqrt{\frac{4 \times 5.538}{\pi}} = 2.655 \text{ m}$$

Up to 1 m diameter 0.3 m tray spacing is sufficient. Here, tower diameters are greater than 1 m. Hence, for the 2nd trial calculations, increase the tray spacing to 0.45 m.
For tray spacing = 0.45 m

(a) Tower diameter required at top

For $F_{LV} = 0.021\ 44$ and tray spacing = 0.45 m
From Fig. 8.20, $C_f = 0.0814$

$$v_F = \frac{0.0814}{0.0635} \times 2.93 = 3.756 \text{ m/s}$$

Actual vapour velocity, $v = 0.85\ v_F = 3.1926$ m/s
Net area required

$$A_n = \frac{6.3496}{3.1926} = 1.989 \text{ m}^2$$

$$A_c = \frac{A_n}{0.88} = 2.26 \text{ m}^2$$

Inside diameter at top

$$D_i = \sqrt{\frac{4 \times 2.26}{\pi}} = 1.7 \text{ m}$$

(b) Tower diameter required at bottom

For $F_{LV} = 0.062\ 96$ and tray spacing = 0.45 m
From Fig. 8.20, $C_f = 0.0794$

$$v_F = \frac{0.0794}{0.0616} \times 1.17 = 1.5 \text{ m/s}$$

Actual vapour velocity, $v = 0.85\ v_F = 1.275$ m/s

Net area required, $A_n = \dfrac{4.8464}{1.275} = 3.8 \text{ m}^2$

Inside diameter of column required at bottom

$$A_c = \frac{A_n}{0.88} = 4.32 \text{ m}^2$$

Inside diameter of column required at bottom

$$D_i = \sqrt{\frac{4 \times 4.32}{\pi}} = 2.345 \text{ m say } 2.36 \text{ m}$$

Provide different diameters in two different sections.

Table 8.37 Summary of Calculations

Parameter	Enriching Section	Stripping Section
Inside diameter of column (D_i), m	1.7	2.36
Inside area of column (A_c), m^2	2.27	4.374
Net column area (A_n), m^2	1.998	3.85
Downcomer area, (A_d), m^2	0.2724	0.525
Maximum active area, ($A_a = 0.76\ A_c$), m^2	1.7252	3.3242
Hole area, (A_h), m^2	0.172 52	0.332 42

For the first trial, hole area A_h is 10% of active area, A_a.

Volumetric flow rate of liquids

In enriching section, $q_L = \dfrac{LM_{\text{av}}}{\rho_L} = \dfrac{601.818 \times 20.9316}{967.97}$

$$= 13.0138\ \text{m}^3/\text{h} \equiv 3.6 \times 10^{-3}\ \text{m}^3/\text{s}$$

In stripping section,

$$q_L = \frac{\overline{L}M_{\text{av}}}{\rho_L} = \frac{974.92 \times 60}{1000} = 54.495\ \text{m}^3/\text{h} \equiv 16.25 \times 10^{-3}\ \text{m}^3/\text{s}$$

Referring Table 8.34, single pass tray can be used for both sections.

Check for weeping

Minimum vapour velocity through holes to avoid the weeping is given by the following equation:

$$v_{\text{h min}} = \frac{K - 0.9(25.4 - d_h)}{\sqrt{\rho_v}} \tag{8.71}$$

where, K = constant can be obtained from Fig. 8.23 as a function of ($h_w + h_{ow}$)

Take

Weir height, h_w = 50 mm (For both sections)

Hole diameter, d_h = 5 mm (For both sections)

Plate thickness, t = 5 mm (For both sections for MS trays)

= 3 mm (For both sections for SS trays)

(a) For enriching section

Height of liquid crest over the weir

$$h_{ow} = 750\left(\frac{\dot{m}_L}{\rho_L l_w}\right)^{2/3} \tag{8.69}$$

For the checking of weeping conditions minimum value of h_{ow} at 70% turn down must be determined. Minimum value of

$$\dot{m}_L = 0.7\ LM_{\text{av}} = 0.7 \times 601.818 \times 20.9316$$

$$= 8817.9\ \text{kg/h} \equiv 2.45\ \text{kg/s (minimum)}$$

$$\rho_L = 967.97 \text{ kg/m}^3$$

Referring to Table 8.35, for $A_d/A_c = 0.12$, $l_w/D_i = 0.77$

$$l_w = \text{Length of weir} = 0.77\ D_i$$

$$l_w = 0.77 \times 1.7 = 1.309 \text{ m}$$

$$\text{Minimum } h_{ow} = 750\left(\frac{2.45}{967.97 \times 1.309}\right)^{2/3} = 11.64 \text{ mm}$$

At minimum rate, $h_L = h_w + h_{ow} = 50 + 11.64 = 61.64$ mm

From Fig. 8.23, $K = 30.38$

$$v_{h\,\min} = \frac{30.38 - 0.9\,(25.4 - 5)}{(0.6823)^{0.5}} = 14.552 \text{ m/s}$$

Actual vapour velocity through holes at minimum vapour flow rate,

$$v_{ha} = \frac{0.7\,q_v}{A_h} = \frac{0.7 \times 6.3496}{0.172\,52} = 25.76 \text{ m/s}$$

Since, $v_{ha} > v_{h\,\min}$, there will be no weeping.

A check by Eq. (8.72) would also confirm no weeping.

$$Fr_L = \left[\frac{0.6823\,(25.76)^2}{(967.97 \times 9.81 \times 0.061\,64)}\right]^{0.5}$$

$$= 0.88 > 0.5$$

Thus in the enriching section, minimum operating rate is well above weep point.

(b) For stripping section

Minimum value of $\dot{\bar{m}}_L = 0.7 \times \dot{m}_L = 0.7 \times 974.92 \times 60$

Minimum $\dot{\bar{m}}_L = 40\,946.64$ kg/h $\equiv 11.374$ kg/s

$$\rho_L = 1000 \text{ kg/m}^3$$

$$l_w = 0.77 \times 2.36 = 1.8172 \text{ m}$$

$$\text{Minimum } h_{ow} = 750\left(\frac{11.374}{1000 \times 1.8172}\right)^{2/3} = 25.47 \text{ mm}$$

At minimum rate, $h_L = h_w + h_{ow} = 50 + 25.47 = 75.47$ mm

From Fig. 8.23, $K = 30.69$

$$v_{h\,\min} = \frac{30.69 - 0.9\,(25.4 - 5)}{(2.8368)^{0.5}} = 7.32 \text{ m/s}$$

Actual vapour velocity through holes at minimum vapour flow rate

$$v_{ha} = \frac{0.7 \times q_v}{A_h} = \frac{0.7 \times 4.8464}{0.332\,42} = 10.2054 \text{ m/s}$$

In this case also $v_{ha} > v_{h\,\min}$. Hence, no weeping will take place in the stripping section.

$$\text{Eq. (8.72): } Fr_L = \left[\frac{2.8368\,(10.2054)^2}{(1000 \times 9.81 \times 0.075\,47)}\right]^{0.5}$$

$$= 0.6317 > 0.5$$

Note: Fr_L of the stripping section is close to the limit which indicates that stripping section is more sensitive to weeping than enriching section. Since column's operational stability depends on both the sections, overall stability will be governed by the performance of the stripping section in this case.

Tray pressure drop

(a) Tray pressure drop for enriching section

Dry plate pressure drop

$$h_d = 51\left(\frac{v_h}{C_o}\right)^2 \frac{\rho_V}{\rho_L} \tag{8.76}$$

$$v_h = \frac{q_v}{A_h} = \frac{6.3496}{0.172\,52} = 36.8 \text{ m/s (maximum)}$$

$$\rho_V = 0.6823 \text{ kg/m}^3, \quad \rho_L = 967.97 \text{ kg/m}^3$$

From Fig. 8.24, for $\dfrac{\text{Plate thickness}}{\text{Hole diameter}} = 1$ and $\dfrac{A_h}{A_p} \cong \dfrac{A_h}{A_a} = 0.1$, $C_o = 0.8422$

A_p = Perforated area is slightly less than active aera, A_a, which excludes some calming zone.

However, for calculation purpose, $A_p = A_a$

$$h_d = 51\left(\frac{36.8}{0.8422}\right)^2 \times \frac{0.6823}{967.97} = 68.64 \text{ mm LC}$$

$$h_w = 50 \text{ mm LC for } \rho_L = 967.97 \text{ kg/m}^3$$

Maximum height of liquid crest over the weir

$$\text{Maximum } h_{ow} = 750\left(\frac{\dot{m}_L}{\rho_L l_w}\right)^{2/3} = 750\left(\frac{2.45/0.7}{967.97 \times 1.309}\right)^{2/3} = 14.765 \text{ mm LC}$$

Residual pressure drop,

$$h_r = \frac{12.5 \times 10^3}{\rho_L} \tag{8.79}$$

$$= \frac{12.5 \times 10^3}{967.97} = 12.9136 \text{ mm LC}$$

Total tray pressure drop

$$h_t = h_d + (h_w + h_{ow}) + h_r \tag{8.75}$$

$$h_t = 68.64 + (50 + 14.765) + 12.9136$$

$$= 146.32 \text{ mm LC for } \rho_L = 967.97 \text{ kg/m}^3$$

Note: This is equivalent to 142 mm WC. Tray pressure drop was assumed equal to 120 mm WC to estimate the base pressure. The calculations can be repeated with a revised value of tray pressure drop but small changes in physical properties will have little effect on the tray design.

(b) Tray pressure drop for stripping section

Dry plate pressure drop, h_d

$$v_h = \frac{4.8464}{0.332\,42} = 14.58 \text{ m/s}$$

$\rho_V = 2.8368$ kg/m^3,
$\rho_L = 1000$ kg/m^3,
$C_o = 0.8422$ (From Fig. 8.20)

$$h_d = 51\left(\frac{14.58}{0.8422}\right)^2 \times \frac{2.8368}{1000}$$

$h_d = 43.36$ mm LC
$h_w = 50$ mm

Maximum height of liquid crest over the weir

$$h_{ow} = 750\left(\frac{11.374/0.7}{1000 \times 1.8172}\right)^{2/3} = 32.31 \text{ mm LC}$$

Residual pressure drop

$$h_r = \frac{12.5 \times 10^3}{1000} = 12.5 \text{ mm LC}$$

Total tray pressure drop

$$h_t = 43.36 + (50 + 32.31) + 12.5 = 138.17 \text{ mm LC or WC}$$

Note: In this section also h_t is higher than 120 mm WC. Calculations with revised h_t may not significantly effect the tray design. However, total pressure drop with correct h_t values in both sections should be worked out and base temperature at the bottom should be recalculated which is important for reboiler design. Considering h_t = 150 mm WC for tray, bottom temperature can be recalculated as 136.63°C (409.78 K).

Checking of downcomer design

Types of downcomer: Straight and segmental downcomer area, $A_d = 0.12\ A_c$ (For both sections).

(a) For enriching section

Pressure drop in downcomer is given by the following equation:

$$h_{dc} = 166\left[\frac{\dot{m}_d}{\rho_L A_m}\right]^2 \tag{8.80}$$

$\dot{m}_d$ = Liquid flow rate through downcomer, kg/s
$= LM_{av}$

$$= 601.818 \times 20.9316 \times \frac{1}{3600}$$

$= 3.5$ kg/s
$\rho_L = 967.97$ kg/m^3
$A_m = A_d$ or A_{ap}, whichever is smaller, m^2
$A_d = 0.2724$ m^2 (Table 8.37)

$$A_{ap} = h_{ap} \times l_w$$
$$l_w = 1.309,$$
$$h_{ap} = h_w - 10$$
$$= 50 - 10 = 40 \text{ mm} \equiv 0.04 \text{ m}$$
$$A_{ap} = 0.04 \times 1.309 = 0.052\,36 \text{ m}^2$$
$$A_{ap} \ll A_d. \text{ Therefore, take } A_m = A_{ap}.$$
$$A_m = 0.052\,36 \text{ m}^2$$
$$h_{\text{dc}} = 166\left(\frac{3.5}{967.97 \times 0.052\,36}\right)^2$$
$$= 0.79 \text{ mm, say 1 mm}$$

Liquid back-up in downcomer

$$h_b = h_w + h_{ow} + h_t + h_{dc} \tag{8.83}$$
$$= 50 + 14.765 + 146.32 + 1 = 212 \text{ mm}$$
$$\frac{l_t + h_w}{2} = \frac{450 + 50}{2} = 250 \text{ mm}$$
$$h_b = 212 < \frac{l_t + h_w}{2} \quad \text{which satisfies Eq. (8.84).}$$

Downcomer area and tray spacing are acceptable.
Check residence time:

$$\theta_r = \frac{A_d h_{bc} \rho_L}{L_{md}} \tag{8.85}$$
$$= \frac{0.2724 \times 0.212 \times 967.97}{3.5}$$
$$= 15.97 \text{ s} > 3 \text{ s} - \text{satisfactory}$$

(b) For stripping section

Pressure drop in downcomer

$$h_{dc} = 166\left(\frac{11.374/0.7}{1000 \times A_m}\right)^2$$

Downcomer area, $A_d = 0.525 \text{ m}^2$

$$A_{ap} = h_{ap} \cdot l_w$$
$$h_{ap} = 50 - 10 = 40 \text{ mm}, l_w = 1.8172 \text{ m}$$
$$A_{ap} = 0.04 \times 1.8172 = 0.0727 \text{ m}^2$$
$$A_m = 0.0727 \text{ m}^2$$
$$h_{dc} = 8.29 \text{ mm}$$

Liquid back-up in downcomer

$$h_b = h_w + h_{ow} + h_t + h_{\text{dc}}$$
$$h_b = 50 + 32.31 + 140 + 8.29 = 230.6 \text{ mm}$$
$$\frac{l_t + h_w}{2} = \frac{450 + 50}{2} = 250 \text{ mm}$$

$$h_b = 230.6 < \frac{l_t + h_w}{2} \quad \text{which satisfies Eq. (8.84).}$$

Here also downcomer area and tray spacing are acceptable.
Residence time in downcomer

$$\theta_r = \frac{A_d h_b \rho_L}{\dot{m}_d} = \frac{0.525 \times 0.23\,06 \times 1000}{(11.374/0.7)}$$
$$= 7.45 \text{ s} > 3 \text{ s}$$

Hence, it is satisfactory.

Checking for entrainment
(a) For enriching section

Vapour velocity based on net area, $v_n = \dfrac{q_v}{A_n}$

$$v_n = \frac{6.3496}{1.998} = 3.178 \text{ m/s}$$

$$\text{Flooding} = \frac{v_n}{v_f} \times 100 = \frac{3.178}{3.756} \times 100 = 85\%$$

$$F_{LV} = 0.021\,444, \quad \psi = 0.21 \quad \text{(from Fig. 8.22)}$$

Entrainment = 21% > 10% – somewhat high

If ψ is less, then higher tray efficiency is obtained. But to decrease the value of ψ, tower diameter must be increased further. Ideally, optimum value of ψ must be determined for the given case. Optimum value of ψ can be greater than 0.1 or 10%.

(b) For stripping section

% Flooding = 85 %, $F_{LV} = 0.062\,96$

From Fig. 8.22, $\psi = 0.084$

% entrainment = 8.4% < 10%

It will provide higher tray efficiency.

Tray efficiency by AIChE method
(a) For enriching section

Physical properties of liquid and vapour for the same tray are:

$\rho_L = 967.97$ kg/m^3, $\sigma_L = 55 \times 10^{-3}$ N/m

μ_L = Viscosity of reflux or distillate (water–acetic acid solution at 100.64°C)

$$\ln \mu_L = w_1 \ln \mu_1 + w_2 \ln \mu_2 \tag{6.56}$$

$w_1 = 0.8$ = mass fraction of water

$w_2 = 0.2$ = mass fraction of acetic acid

μ_w = Viscosity of pure water at 100.64°C = 0.28 mPa · s or cP

μ_{AA} = Viscosity of pure acetic acid at 100.64°C = 0.48 mPa · s or cP (Fig. 3–43 of Ref. 8)

$\mu_L = 0.312$ mPa · s or cP $= 0.312 \times 10^{-3}$ (N · s)/m^2

Diffusivity of acetic acid in water at 100.64°C:

$D_L = 1.24 \times 10^{-5}$ cm^2/s at 25°C
(Table 3.319 of Ref. 8)

To find D_L at 100.64°C,

$$\frac{D_L \mu}{T} = \text{constant}$$

where, μ = Viscosity of solvent (water)

μ_w at 25°C = 0.95 mPa · s or cP

$$D_L = \frac{1.24 \times 10^{-5} \times 0.95}{(273 + 25)} \times \frac{(273 + 100.64)}{0.28}$$

$$D_L = 5.275 \times 10^{-5}\ \text{cm}^2/\text{s} \equiv 5.275 \times 10^{-9}\ \text{m}^2/\text{s}$$

For vapour:

$\rho_V = 0.6823\ \text{kg/m}^3$

μ_v = Viscosity of vapour mixture

$$\mu_v = \Sigma \frac{y_i \mu_i}{\Sigma y_j \Theta_{ij}} \tag{6.77}$$

$$\Theta_{ij} = \frac{\left[1 + \left(\mu_i / \mu_j\right)^{1/2} \left(M_j / M_i\right)^{1/4}\right]^2}{\left[8\left(1 + \frac{M_i}{M_j}\right)\right]^{1/2}}$$

and $$\Theta_{ji} = \left(\frac{\mu_j}{\mu_i}\right)\left(\frac{M_i}{M_j}\right)\Theta_{ij}$$

[(Eq. (3–87) to (3–89) of Ref. 8)]

Viscosity of acetic acid vapour at 100.64°C:

$\mu_{AA} = 1000 \times 10^{-7}$ poise = 0.01 cP or mPa · s (i for acetic acid)

Viscosity of water vapour at 100.64°C

$\mu_W = 1250 \times 10^{-7}$ poise = 1250×10^{-5} cP or mPa · s (j for water)

$$\Theta_{ij} = \frac{\left[1 + \left(1000/1250\right)^{1/2} \left(18/60\right)^{1/4}\right]^2}{\left[8\left(1 + \frac{60}{18}\right)\right]^{1/2}} = 0.4691$$

$$\Theta_{ji} = \left(\frac{1250}{1000}\right)\left(\frac{60}{18}\right) \times 0.4691 = 1.9546$$

$y_i = 1 - 0.9302 = 0.0698, \quad y_j = 0.9302$

$$\mu_v = \frac{(0.0698 \times 0.01) + (0.9302 \times 0.0125)}{(0.9302 \times 0.4691) + (0.0698 \times 1.9546)}$$

$$\mu_v = 0.0215 \text{ cP or mPa} \cdot \text{s} \equiv 0.0215 \times 10^{-3} \text{ (N} \cdot \text{s)/m}^2$$

D_V = Diffusivity of acetic acid vapour in water vapour.

$$D_V = \frac{10^{-3} T^{1.75} [(M_A + M_w)/(M_A M_w)]^{1/2}}{p[(\Sigma V_{AA})^{1/3} + (\Sigma V_W)^{1/3}]^2} \tag{8.102}$$

where, D_V is in cm^2/s. T in K, p is operating pressure in atmosphere, M_A and M_W are molar masses and ΣV is atomic diffusion volume.

(Equation 3–133 of Ref. 8. ΣV can be determined by using Table–342 of the same reference.)

For acetic acid (CH_3COOH)

$$\Sigma V_{AA} = 2 \times 16.5 + 4 \times 1.98 + 2 \times 5.48 = 51.88$$

For water (H_2O),

$$\Sigma V_W = 2 \times 1.98 + 5.48 \times 1 = 9.44$$

$$T = 273 + 100.64 = 373.64 \text{ K}$$

$$D_V = \frac{10^{-3} \times (373.64)^{1.75} [(60 + 18)/(60 \times 18)]^{1/2}}{1 \times [51.88^{1/3} + 9.44^{1/3}]^2}$$

$$D_V = 0.25 \text{ cm}^2/\text{s} = 0.25 \times 10^{-4} \text{ m}^2/\text{s}$$

Number of gas phase transfer units is calculated by the following equation:

$$N_G = \frac{0.776 + 4.57 \times 10^{-3} h_w - 0.24 F_V + 105 L_p}{\left(\dfrac{\mu_V}{\rho_V D_V}\right)^{0.5}} \tag{8.92}$$

h_w = weir height = 50 mm

$$F_V = v_a \sqrt{\rho_v} \tag{8.84}$$

$$v_a = \text{Vapour velocity based on active tray area} = \frac{q_{v\,\min}}{A_a} = \frac{0.7\, q_v}{A_a}$$

$$= \frac{0.7 \times 6.3496}{1.7252} = 2.5764 \text{ m/s}$$

$$F_V = 2.5764 \sqrt{0.6823} = 2.128$$

$$L_p = \frac{\text{Volumetric flow rate of liquid}}{\text{Average width of plate}}$$

$$L_p = \frac{0.7 \times q_L}{(A_a / Z_L)} = \frac{0.7 \times 3.6 \times 10^{-3}}{1.7252/Z_L} \tag{8.103}$$

where, Z_L = Length of liquid path
= Horizontal distance between weir and downcomer apron

$$\frac{l_w}{D/2} = 2\frac{l_w}{D} = 2 \times 0.77 = 1.54 \tag{8.104}$$

Using Mathematical Table 1.19a of Ref. 8.
Chord h is given by

$$\frac{h}{R} = 0.362$$

$$h = \frac{0.362 \times D_i}{2} = 0.181\, D_i$$

$$Z_L = D_i - 2h = D_i - 2 \times 0.181\, D_i = D_i\,(1 - 2 \times 0.181)$$

$$= 0.638 \times D_i = 0.638 \times 1.7 = 1.0846 \text{ m}$$

$$L_p = 1.5843 \times 10^{-3} \text{ m}^2/\text{s}$$

$$\frac{\mu_V}{\rho_V D_V} = \frac{0.0215 \times 10^{-3}}{0.6823 \times 0.25 \times 10^{-4}} = 1.26$$

$$N_G = \frac{(0.776 + 4.57 \times 10^{-3} \times 50 - 0.24 \times 2.128 + 105 \times 1.5843 \times 10^{-3})}{\sqrt{1.26}}$$

$$= 0.588$$

Liquid hold-up on the plate, m^3 per m^2 active area, is given by the following equation:

$$Z_c = 0.006 + 0.73 \times 10^{-3}\, h_w - 0.24 \times 10^{-3}\, F_v\, h_w + 1.22\, L_p \tag{8.96}$$

$$= 0.006 + (0.73 \times 10^{-3} \times 50) - (0.24 \times 10^{-3} \times 2.128 \times 50)$$

$$+ (1.22 \times 1.5846 \times 10^{-3}) = 0.0189 \text{ m}^3/\text{m}^2$$

Liquid contact time

$$\theta_L = \frac{Z_c Z_L}{L_p} \tag{8.95}$$

$$= \frac{0.0189 \times 1.0846}{1.5843 \times 10^{-3}} = 12.94 \text{ s}$$

Number of liquid phase transfer unit

$$N_L = (4.13 \times 10^8\, D_L)^{0.5}\,(0.21\, F_V + 0.15)\,\theta_L \tag{8.94}$$

$$= (4.13 \times 10^8 \times 5.275 \times 10^{-9})^{0.5}\,(0.21 \times 2.128 + 0.15) \times 12.94 = 11.4$$

Eddy diffusivity D_e for sieve tray can be estimated by the following equation:

$$D_e = (0.0038 + 0.017\, v_a + 3.86\, L_p + 0.18 \times 10^{-3}\, h_w)^2$$

$$= (0.0038 + 0.017 \times 2.5764 + 3.86 \times 1.5843 \times 10^{-3} + 0.18 \times 10^{-3} \times 50)^2$$

$$= 0.003\,93 \text{ m}^2/\text{s}$$

Peclet number $Pe = \dfrac{Z_L^2}{D_e \theta_L} = \dfrac{1.0846^2}{0.003\,93 \times 12.94} = 23.13$ (8.96)

Point efficiency, $\dot{\eta}$

$$\frac{1}{\ln(1-\dot{\eta})} = -\left(\frac{1}{N_G} + \frac{mV}{L} \times \frac{1}{N_L}\right) \tag{8.91}$$

Slope of equilibrium curve of top tray

$$m = \frac{y}{x} = \left[\left(\frac{0.9703}{0.9578}\right) \times \left(\frac{0.9409}{0.9134}\right)\left(\frac{0.9186}{0.8787}\right)\right]^{1/3}$$

(Value of y and x are obtained from vapour-liquid equilibrium data, given in Example 8.5.)

$$m = (1.0136 \times 1.0301 \times 1.0454)^{1/3} = 1.0296$$

$$\frac{mV}{L} = \frac{1.0296 \times 745.108}{601.818} = 1.2747$$

Note: At 70 % turndown, individual values of V and L will change, but value of ratio V/L will remain constant.

$$\frac{1}{\ln(1-\dot{\eta})} = -\left(\frac{1}{0.588} + 1.2747 \times \frac{1}{11.4}\right)$$

$$\dot{\eta} = 0.424$$

$$\frac{mV}{L}\dot{\eta} = 1.2747 \times 0.424 = 0.54 \text{ and } Pe = 23.13$$

From Fig. (8.25) $\dfrac{\eta}{\dot{\eta}} = 1.297$

Efficiency of sieve tray $\eta = 1.297 \times 0.424 = 0.55$

Actual tray efficiency η_a, considering liquid entrainment

$$\eta_a = \frac{\eta}{1 + \eta\left(\dfrac{\psi}{1-\psi}\right)} \tag{8.99}$$

$$\eta_a = \frac{0.55}{1 + 0.55\left(\dfrac{0.21}{1-0.21}\right)} = 0.4798$$

(b) For stripping section

To find the tray efficiency of the bottommost tray of stripping section, physical properties of liquid and vapour of the same tray are required.

Liquid

$$\rho_L = 1000 \text{ kg/m}^3$$

$$\sigma_L = 21.334 \times 10^{-3} \text{ N/m}$$

Viscosity of acetic acid liquid at 134.26°C

$$\mu_L = 0.34 \text{ mPa} \cdot \text{s or cP} = 0.34 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

Diffusivity D_L at 134.26°C

$$D_L = \frac{1.24 \times 10^{-5} \times 0.95}{(273 + 25)} \times \frac{(273 + 134.26)}{0.28}$$

$$= 5.75 \times 10^{-5} \text{ cm}^2\text{/s} \equiv 5.75 \times 10^{-9} \text{ m}^2\text{/s}$$

Vapour

$$\rho_V = 2.8368 \text{ kg/m}^3$$

$$\mu_V = 0.009 \text{ mPa} \cdot \text{s or cP}$$

$$D_V = \frac{10^{-3} \times (407.26)^{1.75}[(60 + 18)/(60 \times 18)]^{1/2}}{(1.58)\,[51.88^{1/3} + 9.44^{1/3}]^2}$$

$$= 0.1839 \text{ cm}^2\text{/s} \equiv 0.1839 \times 10^{-4} \text{ m}^2\text{/s}$$

Number of gas phase transfer unit

Vapour velocity based on active area

$$v_a = \frac{0.7 \times q_v}{A_a} = \frac{0.7 \times 4.8464}{3.3242} = 1.02 \text{ m/s}$$

$$F_V = 1.02\sqrt{2.8368} = 1.718$$

$$L_p = \frac{0.7 \times q_L}{(A_a/Z_L)} = \frac{0.7 \times 16.25 \times 10^{-3}}{3.3242/Z_L}$$

$$Z_L = 0.638\, D_i = 0.638 \times 2.36 = 1.5 \text{ m}$$

$$L_p = 5.1328 \times 10^{-3} \text{ m}^2\text{/s}$$

$$\frac{\mu_V}{\rho_V D_V} = \frac{0.009 \times 10^{-3}}{2.8368 \times 0.1839 \times 10^{-4}} = 0.1725$$

$$N_G = \frac{0.776 + (4.57 \times 10^{-3} \times 50) - (0.24 \times 1.718) + (105 \times 5.1328 \times 10^{-3})}{\sqrt{0.1725}}$$

$$= 2.7234$$

Liquid hold-up on the plate,

$$Z_c = 0.006 + (0.73 \times 10^{-3} \times 50) - (0.24 \times 10^{-3} \times 1.718 \times 50) + (1.22 \times 5.1328 \times 10^{-3}) = 0.028 \text{ m}^3\text{/m}^2$$

Liquid contact time

$$\theta_L = \frac{Z_c Z_L}{L_p} = \frac{0.028 \times 1.5}{5.1328 \times 10^{-3}} = 8.18 \text{ s}$$

Number of liquid phase transfer units,

$$N_L = (4.13 \times 10^8 \times 5.75 \times 10^{-9})^{0.5} (0.21 \times 1.718 + 0.15) \times 8.18$$
$$= 6.44$$

Point efficiency, $\dot{\eta}$

$$\frac{1}{\ln(1-\dot{\eta})} = -\left(\frac{1}{N_G} + m\frac{\overline{V}}{\overline{L}}\frac{1}{N_L}\right)$$

$$= -\left(\frac{1}{2.7234} + 2.5 \times \frac{824.89}{974.92} \times \frac{1}{6.44}\right) \quad (8.91)$$

$$\dot{\eta} = 0.7625$$

$$\frac{m\overline{V}}{\overline{L}} \cdot \dot{\eta} = 2.5 \times 0.7625 \times \frac{824.89}{974.92} = 1.613$$

Eddy diffusivity

$$D_e = (0.0038 + 0.017\, v_a + 3.86\, L_p + 0.18 \times 10^{-3}\, h_w)^2 \quad (8.98)$$
$$= (0.0038 + 0.017 \times 1.02 + 3.86 \times 5.1328 \times 10^{-3} + 0.18 \times 10^{-3} \times 50)^2$$
$$= 0.0025 \text{ m}^2/\text{s}$$

Peclet number

$$Pe = \frac{Z_L^2}{D_e \theta_L} = \frac{1.5^2}{0.0025 \times 8.18} = 110 \quad (8.97)$$

From Fig. 8.25,

$$\frac{\eta}{\dot{\eta}} = 2.46$$

$$\eta = 2.46 \times 0.7625 = 1.876$$

Efficiency cannot be greater than 1.

Hence $\eta = 1$

Actual tray efficiency η_a considering liquid entrainment

$$\eta_a = \frac{1}{1 + \left(\frac{0.084}{1 - 0.084}\right)} = 0.916$$

Tray efficiency by Van Winkle's correlation[19]

For the topmost tray

$$\eta = 0.07\, D_g^{0.14}\, Sc_L^{0.25}\, Re^{0.08} \quad (8.87)$$

where, D_g = Surface tension number = $\dfrac{\sigma_L}{u_L u_v}$ (8.88)

$\sigma_L = 55 \times 10^{-3}$ N/m, $\mu_L = 0.312 \times 10^{-3}$ (N · s)/m^2

u_V = Superficial vapour velocity

$$u_V = \frac{6.3496}{\pi/4\, D_i^2} = \frac{6.3496}{(\pi/4)(1.7)^2} = 2.797 \text{ m/s}$$

$$D_g = \frac{55 \times 10^{-3}}{0.312 \times 10^{-3} \times 2.797} = 63.6$$

$$f_A = \text{Fractional area} = \frac{A_h}{A_c} = 0.077\,18$$

Liquid Schmidt number, Eq. (8.89):

$$\rho_L = 967.97 \text{ kg/m}^3, \quad D_{LK} = 5.275 \times 10^{-9} \text{ m}^2\text{/s}$$

$$Sc_L = \frac{0.312 \times 10^{-3}}{967.97 \times 5.275 \times 10^{-9}} = 61.1$$

Reynolds number,

$$Re = \frac{h_w u_V \rho_V}{\mu_L (f_A)} \times 10^{-3} = \frac{0.05 \times 2.797 \times 0.6823}{0.312 \times 10^{-3} \times 0.077\,18} = 3962.6 \qquad (8.90)$$

$$\eta = 0.07\,(63.6)^{0.14}\,(61.1)^{0.25}\,(3962.6)^{0.08}$$
$$= 0.679,$$

$$\eta_a = \frac{0.679}{1 + 0.679\left(\dfrac{0.21}{1 - 0.21}\right)} = 0.5752$$

For the bottommost tray

Surface tension number

$$D_g = \frac{\sigma_L}{\mu_L u_v} \qquad (8.88)$$

$$\sigma_L = 21.334 \times 10^{-3} \text{ N/m}, \ \mu_L = 0.34 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$u_V = \text{Superficial vapour velocity} = \frac{4.8464}{(\pi/4)\,(2.36)^2} = 1.108 \text{ m/s}$$

$$D_g = \frac{21.334 \times 10^{-3}}{0.34 \times 10^{-3} \times 1.108} = 56.63$$

$$\rho_L = 1000 \text{ kg/m}^3, \quad D_L = 5.75 \times 10^{-9} \text{ m}^2\text{/s}$$

$$Sc_L = \frac{\mu_L}{\rho_L D_L} = \frac{0.34 \times 10^{-3}}{1000 \times 5.75 \times 10^{-9}} = 59.13$$

$$f_A = \frac{A_h}{A_c} = \frac{0.332\,42}{4.374} = 0.076$$

$$Re = \frac{h_w u_V \rho_V \times 10^{-3}}{\mu_L (f_A)} = \frac{0.05 \times 1.108 \times 2.8368}{0.34 \times 10^{-3} (0.076)}$$

$$= 6082$$

$$\eta = 0.07\,(56.63)^{0.14}\,(59.31)^{0.25}\,(6082)^{0.08}$$

$$= 0.6863$$

$$\eta_a = \frac{0.6863}{1 + 0.6863\left(\dfrac{0.084}{1 - 0.084}\right)} = 0.6457$$

Table 8.37 Resulting Data of Tray Efficiency

Method	Tray Efficiency	
	Topmost Tray	Bottommost Tray
AIChE Method	0.4798	0.916
Van Winkle's Method	0.5752	0.6457

Let tray efficiency for enriching section = 0.45

Tray efficiency for stripping section = 0.5

$$\text{Actual number of trays in enriching section} = \frac{7}{0.45} \cong 16$$

Actual number of trays in stripping section = 17/0.5 = 34

Actual number of trays = 18 + 34 = 52, assume 55.

Feed nozzles can be provided at 32nd, 36th and 40th trays. More number of feed nozzles are provided to facilitate the flexibility in the operation. For example, in actual operation if the feed is introduced on 36th tray, purity of distillate could be more than desired but bottom product (in this case it is pure acetic acid) may not be of desired purity. In such a case to get desired result, feed can be introduced on 32nd tray.

Pressure drop

Pressure drop in the topmost tray = 146.32 mm LC

Pressure drop in the bottommost tray = 138.17 mm LC

Assume tray pressure drop for the entire column = 145 mm WC

$$\rho_L \text{ (at top)} = 967.97 \text{ kg/m}^3, \quad \rho_L \text{ (at bottom)} = 1000 \text{ kg/m}^3$$

$$\Delta p_T = 55 \times 145 = 7975 \text{ mm WC}$$

$$\Delta p_T = 7975 \times 10^{-3} \times 1000 \times 9.81 = 78\,235 \text{ N/m}^2 \equiv 78.2 \text{ kPa}$$

Value of 78.2 kPa is higher than that assumed value of 56.5 kPa (p. 522). With the correct Δp_T, temperature of boiling liquid in the reboiler is to be reworked.

Refer to Fig. 8.26 for crossflow sieve trays' details for enriching and stripping sections.

E = Enriching Section S = Stripping Section All Dimensions are in mm (NTS).

Fig. 8.26 *Crossflow Sieve Tray Details*

8.5 BATCH DISTILLATION

It is a very common unit operation used in the manufacturing of chemicals which are produced in batch processes. Batch distillation is carried out with or without using a rectification column (tray tower or packed tower). Thus, it is divided in two types: (i) simple distillation, and (ii) batch distillation with rectification.

8.5.1 Simple Distillation

This type of batch distillation consists of a heated vessel (coiled vessel or jacketed vessel or kettle-type reboiler), a condenser and one or more receiving tanks. Feed mixture is charged into the vessel and brought to boiling. Vapours are condensed and condensate is collected in the receiver. No reflux is returned (Fig. 8.27). Simple batch distillation provides only one theoretical plate of separation. In some batch reactors of small plants, at the end of reactions, volatile components are separated from high boiling liquid solution by simple distillation. Following equation is applicable to the simple batch distillation.

Fig. 8.27 *Simple Batch Distillation*

$$\ln\left(\frac{n_F}{n_W}\right) = \int_{x_B}^{x_F} \frac{dx}{y - x} \tag{8.105}$$

where, n_F = Moles of liquid in initial condition
n_W = Moles of liquid in final condition
x_F = Mole fraction of more volatile component in liquid phase in initial conditions
x_B = Mole fraction of more volatile component in liquid phase in final conditions
x, y = Mole fraction of more volatile component in liquid and vapour phases at a given time

If the liquid mixture is a binary mixture for which relative volatility α is constant, then above Eq. (8.105) can be converted into the following equation:

$$\ln\left(\frac{n_F}{n_W}\right) = \left[\frac{1}{(\alpha - 1)}\right] \ln\left[\frac{x_B(1 - x_F)}{x_F(1 - x_B)}\right] + \ln\left[\frac{1 - x_F}{1 - x_B}\right] \tag{8.106}$$

For a binary system and for simple batch distillation, the following equation is also applicable.

$$\ln\left(\frac{n_{Af}}{n_{Ai}}\right) = \alpha \ln\left(\frac{n_{Bf}}{n_{Bi}}\right) \tag{8.107}$$

where, n_{Af}, n_{Ai} = Moles of component A in the pot after and before distillation, respectively

n_{Bi}, n_{Bf} = Moles of component B in the pot before and after distillation, respectively.

In analogue manner, for simple or differential batch distillation of a multi-component mixture, the following equation is applicable:

$$\ln\left(\frac{n_{if}}{n_{ii}}\right) = \alpha_i \ln\left(\frac{n_{rf}}{n_{ri}}\right) \tag{8.108}$$

where, n_{if}, n_{ii} = Moles of component i in the residue and in the feed in the pot after and before distillation, respectively

n_{rf}, n_{ri} = Moles of reference component (r) in the residue and in the feed, respectively

α_i = Relative volatility of component i with respect to reference component r

Example 8.14 1000 kg of feed, containing 30% by mass ethylene glycol and remaining water, is charged to a batch still. Simple batch distillation is carried out at 30.4 kPa absolute pressure to get the residue which must contain 95% by mass ethylene glycol. Find the amount of residue (final product).

Table 8.39 Vapour – liquid Equilibrium Data of Ethylene Glycol–Water System at 30.4 kPa a[8]

Temperature, °C	x	y
69.5	1.00	1.000
76.1	0.77	0.998
78.9	0.69	0.997
83.1	0.60	0.990
89.6	0.46	0.980
103.1	0.27	0.940
118.4	0.15	0.870
128.0	0.10	0.780
134.7	0.07	0.700
145.0	0.03	0.530
160.7	0.00	0.000

Solution

Feed contains 300 kg ethylene glycol and 700 kg water.
Molar mass of water = 18
Molar mass of ethylene glycol = 62

$$n_F = \frac{300}{62} + \frac{700}{18} = 43.7276 \text{ kmol}$$

Mole fraction of water in feed, $x_F = \dfrac{700/18}{43.7276} = 0.8893$

Mole fraction of water in residue, $x_B = \dfrac{5/18}{(5/18) + (95/62)} = 0.153\,47$

$$\ln\left(\frac{43.7276}{n_W}\right) = \int_{0.153\,47}^{0.8893} \frac{dx}{y - x}$$

To solve this integration, Table 8.40 is prepared based on the given VLE data as well as based on the equilibrium curve drawn.

Table 8.40 Batch Distillation Data

x	y	$y - x$	$F(x) = \dfrac{1}{y - x}$
$x_B = 0.153\,47$	0.875	0.721 53	1.385 94
0.270 00	0.940	0.670 00	1.492 54
0.350 00	0.970	0.620 00	1.612 90
0.460 00	0.980	0.520 00	1.923 08
0.600 00	0.990	0.390 00	2.564 10
0.690 00	0.997	0.307 00	3.257 33
0.770 00	0.998	0.228 00	4.385 96
$x_F = 0.889\,30$	0.999	0.109 70	9.115 77

Draw the graph of $\dfrac{1}{y - x}$ vs x (Fig. 8.28).

$$\int_{0.153\,47}^{0.8893} \frac{dx}{y - x} = \text{area under curve from } x_B \text{ to } x_F = 2.0643 \text{ (integral value)}$$

$$\ln\left(\frac{43.7276}{n_W}\right) = 2.0643$$

$$n_W = 5.5493 \text{ kmol}$$

Batch distillation data, presented in Table 8.40, were fitted (using Excel*) in an equation form.

$F(x) = (138.6652x^4 - 232.9768x^3 + 142.0108x^2 - 33.8861x + 4.0347)$ ($\boldsymbol{R}^2 = 0.9987$)

Fig. 8.28 *Evaluation of Integral for Batch Distillation*

$$\text{Integral} \int_{0.153\,47}^{0.8893} F(x)\,dx = 2.116\,27$$

Thus integral value can be obtained by fitting an equation without drawing the graph. The value differs by 2.51% from the integral value, obtained from the graph. Advantage of the equation form is that it can be integrated in any desired range easily.

$$\text{Recovery of ethylene glycol in final product } (n_W) = \frac{(1 - x_B)\, n_W}{(1 - x_F)\, n_F} \times 100$$

$$= \frac{(1 - 0.153\,47)\, 5.5493}{(1 - 0.8893)\, 43.7276} \times 100$$

$$= 97.05\%$$

Example 8.15 A mixture of hydrocarbons at 5.44 atm a is to be differentially distilled until the moles of propane is reduced to 10 moles in the residue. Total moles of feed is 100 mol.

Feed composition

Component	mole %
Ethane (C_2)	10

Propane (C_3)	25
n-butane ($n - C_4$)	35
i-Butane ($i - C_4$)	30

Solution

To find the average relative volatilities, bubble point of feed and bubble point of final residue are required.

At 10°C and 5.44 atm a for feed solution

$$K_{C_2} = 4.5, \quad K_{C_3} = 1.18, \quad K_{nC_4} = 0.33, \quad K_{iC_4} = 0.48 \qquad \text{(Ref. Fig. 6.20)}$$

$$\sum K_i x_i = 4.5 \times 0.1 + 1.18 \times 0.25 + 0.33 \times 0.35 + 0.48 \times 0.3 = 1$$

Hence, 10°C is the bubble point of feed.

Bubble point of Residue:

1st trial calculations

Assume that bubble point of final residue is 40°C.

At 40°C and 5.44 atm a,

$$K_{C_2} = 7.2, \quad K_{C_3} = 2.2, \quad K_{nC_4} = 0.75, \quad K_{iB} = 1.0 \quad \text{(Ref. Fig. 6.20)}$$

Here propane is reference component.

Average relative volatilities with respect to C_3

$$\alpha_{C_2} = \sqrt{\left(\frac{4.5}{1.18}\right)\left(\frac{7.2}{2.2}\right)} = 3.53$$

$$\alpha_{C_3} = 1, \quad \alpha_{nC_4} = 0.309, \quad \alpha_{iC_4} = 0.43$$

Moles of components in residue

$$n_{C_{3f}} = 10 \text{ moles}$$

$$n_{C_{2f}} = n_{C_{2i}} \left(\frac{n_{C_{3f}}}{n_{C_{3i}}}\right)^{\alpha_{C2}}$$

$$= 10\left(\frac{10}{25}\right)^{3.53} = 0.3938 \text{ mol}$$

$$n_{C_{4f}} = 35\left(\frac{10}{25}\right)^{0.309} = 26.37 \text{ mol}$$

$$n_{iC_{4f}} = 30\left(\frac{10}{25}\right)^{0.43} = 20.23 \text{ mole}$$

Total moles of the residue = 0.3938 + 10 + 26.37 + 20.23 = 56.9938 mole

Calculated composition of residue

$$x_{C_2} = 0.0069, \quad x_{C_3} = 0.175, \quad x_{nC_4} = 0.463, \quad x_{iC_4} = 0.355 \text{ at } 40°\text{C}$$

$$\sum K_i x_i = 7.2 \times 0.0069 + 0.175 \times 2.2 + 0.463 \times 0.75 + 1 \times 0.355 = 1.13 > 1$$

Hence, actual bubble point is less than 40°C.

At 35°C

$$K_{C_2} = 6.7, \quad K_{C_3} = 2, \quad K_{nC_4} = 0.67, \quad K_{iC_4} = 0.92$$

$$\alpha_{C_2} = 3.58, \quad \alpha_{C_3} = 1, \quad \alpha_{nC_4} = 0.307, \quad \alpha_{iC_4} = 0.432$$

$$n_{C_{2f}} = 0.378 \text{ mol}, \quad n_{C_{3f}} = 10 \text{ mol}$$

$$n_{nC_{4f}} = 26.4 \text{ mol}, n_{iC_{4f}} = 20.2 \text{ mol}$$

Total moles of residue = 56.97 mol

Calculated composition of residue

$$x_{C_2} = 0.0066, x_{C_3} = 0.175, x_{nC_4} = 0.464, x_{iC_4} = 0.354$$

$$\sum K_i x_i = 6.7 \times 0.0066 + 2 \times 0.175 + 0.67 \times 0.464 + 0.92 \times 0.354 = 1.03 \approx 1.$$

Thus residue has 56.97 mol with bubble point of 35°C.

8.5.2 Batch Distillation with Rectification

Simple batch distillation is a single stage separation hence it does not give good separation unless the relative volatility is very high. In many cases, a rectification column which may be a tray tower or a packed tower with reflux is used to improve the performance of batch distillation. If the column is not too large, then it is mounted on top of the still or heated vessel (Fig. 8.29).

Fig. 8.29 *Batch Distillation with Rectification Column*

This type of batch distillation consists of a still (heated vessel or reboiler), a rectification column, a condenser, some means of splitting off a portion of the condensed vapour (distillate) as reflux and one or more top product receivers. In

operation, a batch of liquid feed is charged to the reboiler or still. Then first, batch distillation is carried out under total reflux. After achieving steady state, portion of the overhead condensate is continuously withdrawn in accordance with the established reflux policy. To get the constant overhead composition, the amount of reflux returned to the column must be continuously increased throughout the run.

Process design of binary batch rectification with constant overhead composition[8] Since compositions within column are continuously changing during the batch distillation, rigorous calculation methods are extremely complex. For a binary system, approximate but useful method is based on McCabe–Thiele graphical method. In addition to the assumptions of McCabe–Thiele method (negligible heat of solution and equimolar overflow on the trays), this approximate method assumes negligible hold-up of liquid on the trays in the column and in the condenser. Stepwise procedure of this approximate method is as follows:

1. Determine the minimum reflux ratio by using the following equation:

$$R_{\min} = \frac{(L/V)_{\min}}{1-(L/V)_{\min}} \tag{8.109}$$

where, $\left(\frac{L}{V}\right)_{\min}$ = Minimum internal reflux ratio

$$= \frac{y_D - y_{pi}}{x_D - x_{pi}} \tag{8.110}$$

where, $x_D = y_D$ = Mole fraction of more volatile component in distillate, if total condenser is used.

For partial condenser,

y_D = Mole fraction of more volatile component in distillate vapour

x_D = Mole fraction of more volatile component in reflux

$x_{pi} = x_F$ = Mole fraction of more volatile component in liquid feed

y_{pi} = Value of y which is in equilibrium with x_{pi} which can be determined from the vapour-liquid equilibrium curve or data or equation.

x_{pi} and y_{pi} defined above are valid only for normal equilibrium curve, as shown in Fig. 8.30.

For the equilibrium curve with inflection as shown in Fig. 8.31, x_{pi} and y_{pi} are equilibrium compositions corresponding to the inflection point, "*P*".

2. Actual reflux ratio is 1.5 to 10 times minimum. Decide the actual reflux ratio R for starting of batch distillation. Determine the value of internal reflux ratio L/V required for starting condition.
3. Draw the operating line for starting of batch distillation. Slope of operating line is L/V (for starting condition). Determine the number of theoretical stages required for the desired separation at the start of batch distillation. To find this, start the staircase construction from x_D to x_F in between equilibrium curve and operating line.

Fig. 8.30 *Determination of Minimum Reflux for Equilibrium Curve without Inflection*

Fig. 8.31 *Determination of Minimum Reflux for Equilibrium Curve with Inflection*

4. Select the slightly higher value of R and L/V than the same of starting condition. For this new value of L/V draw the operating line from x_D. Draw the same number of steps (or equilibrium stages) from x_D which was obtained for starting condition. Find the composition of liquid in reboiler (x_{Wi}) corresponding to new value of R and L/V.
5. Select again slightly greater value of R and L/V. Draw the operating line for the new value of L/V. Find the composition of liquid in reboiler or still (residue); x_{Wi}, by drawing the same number of steps from x_D which was required for starting condition. Continue these calculations until, new value of x_{Wi} obtained, is equal to x_B, where x_B is equal to mole fraction of more volatile component required in bottom product.
6. Time required for batch distillation rectification and with constant overhead composition can be calculated by Bogart equation[8]:

$$\theta = \frac{n_F(x_D - x_F)}{V} \int_{x_B}^{x_F} \frac{dx_W}{(1 - L/V)(x_D - x_W)^2} \tag{8.111}$$

where, θ = Total time required in batch distillation with rectification, s
n_F = Total moles of feed-charged, kmol
V = Molar flow rate of vapour phase, kmol/s
L = Molar flow rate of liquid phase, kmol/s
x_D = Mole fraction of more volatile component in distillate
x_B = Mole fraction of more volatile component in bottom product or final residue.
x_W = Mole fraction of more volatile component in liquid inside the still or reboiler
x_F = Mole fraction of more volatile component in feed

Above equation can also be written as

$$\theta_i = \frac{n_F(x_D - x_F)}{V} \int_{x_{Wi}}^{x_F} \frac{dx_W}{(1 - L/V)(x_D - x_W)^2} \tag{8.112}$$

where, x_{Wi} = Intermediate composition (mole fraction of more volatile component) of liquid in still or reboiler.

Define $$F'(x_W) = \frac{1}{(1 - L/V)(x_D - x_W)^2} \tag{8.112a}$$

By using Eq. (8.112), for the different values of θ, L/V and R can be determined. Based on these data, the graph of R vs θ can be developed. This graph or curve is also useful in actual operation, giving information about how to increase the value of reflux ratio R with respect to time to maintain the constant composition of overheard (top) product.

7. The quantity of distillate at any time θ can be determined by using the following equation:

$$n_F - n_{Wi} = \frac{n_F(x_F - x_{Wi})}{x_D - x_{Wi}} \tag{8.113}$$

where, n_{Wi} = Moles of liquid in still at any time θ_i, kmol

At the end of distillation, quantity of material distilled

$$n_D = n_F - n_B = \frac{n_F(x_F - x_B)}{(x_D - x_B)} \tag{8.114}$$

where, n_B = Moles of final bottom product, kmol

8. Carry out the material of construction of packed tower or tray tower.
9. Select the type of tower; tray tower or packed tower.
10. Determine the tower diameter and pressure drop per unit height or tray.
11. Determine the tray efficiency of tray tower or HETP in case of packed tower.
12. Determine the actual number of trays required in tray tower or height of packing required in packed tower.
13. Determine the actual total pressure drop.
14. Design the condenser, reboiler, etc.

Example 8.16 1000 kg of feed, containing 40% by mass methanol and balance water, is to be separated in batch distillation with a rectification column. Top product must contain 99.9% by mass of methanol. Composition of distillate should remain constant throughout the batch distillation. Desired composition of residue is 95% by mass water.

(a) Design the rectification column.
(b) Determine the operating (actual) reflux ratio vs. time data.

Operating pressure in rectification column is atmospheric.

Table 8.41 Equilibrium Data for Methanol-Water Data at 101.3 kPa

Temperature, °C	Mole fraction of methanol				Relative volatility
	x	$X = x/(1-x)$	y	$Y = y/(1-y)$	$\alpha_i = Y/X$
100.0	0.0		0		–
96.4	0.02	0.0204	0.134	0.1547	7.5820
93.5	0.04	0.0417	0.230	0.2987	7.1688
91.2	0.06	0.0638	0.304	0.4368	6.8429
89.3	0.08	0.0870	0.365	0.5748	6.6102
87.7	0.100	0.1111	0.418	0.7182	6.4639
84.4	0.150	0.1765	0.517	1.0704	6.0656
81.7	0.200	0.2500	0.579	1.3753	5.5012
78.0	0.300	0.4286	0.665	1.9851	4.6318
75.3	0.400	0.6667	0.729	2.6900	4.0351
73.1	0.500	1.0000	0.779	3.5249	3.5249
71.2	0.600	1.50000	0.825	4.7143	3.1429
69.3	0.700	2.3333	0.870	6.6923	2.8681
67.5	0.800	4.0000	0.915	10.7647	2.6912
66.0	0.900	9.0000	0.958	22.8095	2.5344
65.0	0.950	19.0000	0.979	46.6190	2.4536
64.5	1.000		1.000		–

(Table 13-1 of Ref. 8)

Average volatility, $\alpha_{avg} = \sqrt[15]{\alpha_1 \times \alpha_2 \times \ldots \times \alpha_{15}} = 4.448$

Solution

(a) Graphical Method:

Basis: 1000 kg feed consisting 40% methanol (by mass)

$$n_F = \frac{0.4 \times 1000}{32} + \frac{0.6 \times 1000}{18} = 45.833 \text{ kmol}$$

Mole fraction of methanol in feed, $x_F = \dfrac{(400/32)}{45.833} = 0.2727$

Mole fraction of methanol in distillate, $x_D = \dfrac{(99.9/32)}{(99.9/32) + (0.1/18)} = 0.9982$

Mole fraction of methanol in residue,

$$x_B = \frac{(5/32)}{(5/32) + (95/18)} = 0.028\ 75$$

$$n_D = \frac{n_F (x_F - x_B)}{(x_D - x_B)} \tag{8.114}$$

$$= \frac{45.833\ (0.2727 - 0.028\ 75)}{(0.9982 - 0.028\ 75)}$$

$$= 11.5333 \text{ kmol}$$

$$n_B = 45.833 - 11.5333 = 34.2997 \text{ kmol}$$

Draw the equilibrium curve and find the minimum reflux ratio, R_m.

$$R_m = \frac{(L/V)_{\min}}{1 - (L/V)_{\min}} \tag{8.109}$$

where, $$(L/V)_{\min} = \frac{y_D - y_{pi}}{x_D - x_{pi}} \tag{8.110}$$

Figure 8.32 shows equilibrium curve for methanol-water system at 101.325 kPa.

$$x_D = y_D = 0.9982$$

$$x_{pi} = x_F = 0.2727$$

Corresponding equilibrium value of y of x_{pi}

$$y_{pi} = 0.6415$$

$$(L/V)_{\min} = \frac{0.9982 - 0.6415}{0.9982 - 0.2727} = 0.491\ 66$$

$$R_m = \frac{0.491\ 66}{1 - 0.491\ 66} = 0.9672$$

Let the reflux ration R at the starting of batch distillation (at $\theta = 0$) is equal to $2R_m$.

$$R_1 = 2 \times 0.9672 = 1.9344$$

Slope of operating line

$$L/V = \frac{R_1}{R_1 + 1} = 0.6592$$

Point on *y*-axis	R	$\frac{x_D}{R+1}$	x_B
1	1.9344	0.3400	0.6354
2	3.0	0.249 55	0.2296
3	3.5	0.2218	0.1427
4	4.0	0.2000	0.1036
5	4.5	0.1815	0.0774
6	5.0	0.1664	0.0616
7	6.0	0.1426	0.0447
8	8.0	0.1109	0.0294
9	9.0	0.1000	0.0288

Fig. 8.32 *McCabe–Thiele Diagram for Batch Rectification of Methanol-Water Solution*

Intercept of this operating line

$$x_D/(R_1 + 1) = 0.9982/2.9344 = 0.34$$

Figure 8.32 shows that for $R_1 = 1.9344$ it is not possible to get the bottom composition of $x_B = 0.028\ 75$. Hence, it is planned to increase the reflux ratio gradually from $R_1 = 1.9344$ to $R_9 = 9$.

For reflux ratio $R_9 = 9$ and intercept $\frac{x_D}{R_9+1} = 0.1$, approximately 10 number of theoretical stages required to get $x_B = 0.028\ 75$ as inferred from Fig. 8.32. Based on this the value of number of theoretical stages $N = 10$ is fixed for all values of reflux ratio. For each value of reflux ratio, value of bottom composition x_{Wi} is determined for $N = 10$.

Table 8.42 Batch Distillation Data

R	L/V	$\frac{x_D}{R+1}$	x_W	$F'(x_W) = \frac{1}{(1-L/V)(x_D - x_W)^2}$
$R_1 = 1.9344$	0.65921	0.34017	0.63540	—
$R_2 = 3$	0.75000	0.24955	0.27270	7.5995
$R_2 = 3$	0.75000	0.24955	0.22960	6.7711
$R_3 = 3.5$	0.77778	0.22182	0.14270	6.1485
$R_4 = 4$	0.80000	0.19964	0.10360	6.2476
$R_5 = 4.5$	0.81818	0.18149	0.07740	6.4868
$R_6 = 5$	0.83333	0.16637	0.06160	6.8398
$R_7 = 6$	0.85714	0.14260	0.04470	7.6994
$R_8 = 8$	0.88889	0.110 91	0.02940	9.5890
$R_9 = 9$	0.90000	0.09982	0.02875	10.6402

Note: Table 8.42 shows that for $R_1 = 1.9344$ and $N = 10$, $x_w = 0.6354 > x_F$. It means that for $R_1 = 1.9344$, more than 10 number of equilibrium stages are required to get the desired separation. In actual operation for $R = 1.9344$ and $N = 10$, distillate composition obtained will be less than x_D even at the starting of batch distillation. It is calculated to be 0.9917 from the graph (not shown in Fig. 8.32). For $R_2 = 3$ and $N = 10$, $x_w = 0.2296 < x_F$. Hence, it is decided to keep $R = 3$ (min) at the start of batch distillation.

Time required for batch distillation and with constant overhead composition is given by Bogart equation [Eq. (8.111)].

$$\theta = \frac{45.833\,(0.9982 - 0.2727)}{V} \int_{0.028\,75}^{0.2727} \frac{dx_w}{(1 - L/V)\,(x_D - x_W)^2}$$

Draw the graph of $F'(x_W) = \frac{1}{(1 - L/V)\,(x_D - x_W)^2}$ vs x_W and find the area under the curve.

Fig. 8.33 *Evaluation of Integral for Batch Distillation*

From Fig. 8.33, area under the curve from $x_W = 0.028\ 75$ to $x_W = 0.2727$ is equal to 1.637 47 (integral value).

Batch rectification data, presented in Table 8.42, were fitted (using Excel®) in an equation form.

$$F'(x_W) = 4500\ 637.98x_W^6 - 3944\ 492x_W^5 + 1356\ 915.1x_W^4 - 234\ 196.7x_W^3 + 21\ 563.33x_W^2 - 1024.75x_W + 26.56 \qquad (\boldsymbol{R^2} = 0.9794)$$

$$\text{Integral} = \int_{0.028\ 75}^{0.2727} F'(x_W)\ dx_W = 1.649\ 81$$

$$\theta = \frac{45.833\ (0.9982 - 0.2727)}{V} \times 1.637\ 47$$

$$V\theta = 54.449 \text{ kmol}$$

If the total time of batch distillation is fixed at $\theta = 6$ h,

$$V = \frac{54.449}{6} = 9.0748 \text{ kmol/h}$$

Total time for batch distillation in any batch process is fixed based on the time required by the equipment which is governed by the batch process. In most of the cases, time required by the reaction in batch reactor decides the time of batch process and time of other separation operations like batch distillation.

Total distillation time, $\theta = 6$ h

Here reflux ratio R at the starting of batch distillation is 3. In operation R can be kept equal to 3 from $\theta = 0$ to $\theta = \theta_1$.

$$\theta_1 = \frac{\text{Area of segment from } x_W = x_F \text{ to } x_W = 0.2296}{\text{Total area under the curve}} \times \theta$$

$$\theta_1 = \frac{0.3062}{1.637\,47} \times 6 = 1.122 \text{ h}$$

For $R_3 = 3.5$, area of segment from $x_W = 0.2296$ to $x_W = 0.1427$ is 0.558 89 (determined from Fig. 8.33)

Hence, $$\theta_2 = \frac{0.558\,89}{1.637\,47} \times 6 = 2.048 \text{ h}$$

Similarly,

For $R_4 = 4$, $$\theta_3 = \frac{0.242\,43}{1.637\,47} \times 6 = 0.8883 \text{ h}$$

For $R_5 = 4.5$, $$\theta_4 = \frac{0.150\,34}{1.637\,47} \times 6 = 0.5509 \text{ h}$$

For $R_6 = 5$, $$\theta_5 = \frac{0.122\,41}{1.637\,47} \times 6 = 0.4485 \text{ h}$$

For $R_7 = 6$, $$\theta_6 = \frac{0.121\,94}{1.637\,47} \times 6 = 0.4468 \text{ h}$$

For $R_8 = 8$, $$\theta_8 = \frac{0.129\,01}{1.637\,47} \times 6 = 0.473 \text{ h}$$

For $R_9 = 9$, $$\theta_9 = \frac{0.006\,13}{1.637\,47} \times 6 = 0.0225 \text{ h}$$

Table 8.43 Reflux Ratio vs Time

R	θ, h			x_W		
3	0	to	1.122	0.2727	to	0.2296
3.5	1.122	to	3.17	0.2296	to	0.1427
4	3.17	to	4.0583	0.1427	to	0.1036
4.5	4.0583	to	4.6092	0.1036	to	0.0774
5	4.6092	to	5.0577	0.0774	to	0.0616
6	5.0577	to	5.5045	0.0616	to	0.0447
8	5.5045	to	5.9775	0.0447	to	0.0294
9	5.9775	to	6	0.0294	to	0.02875

(b) Numerical Integration Method

Alternate method of obtaining the integral value is given by Koppel[24].
The following equation can be derived by combining material balance equation with a vapour-liquid equilibrium equation:

$$y_{n-1} = \left(\frac{L}{V}\right)x_n + \left(\frac{D}{V}\right)x_D \tag{8.115}$$

and

$$y_{n-1} = \frac{\alpha x_{n-1}}{1 + x_{n-1}(\alpha - 1)} \tag{8.116}$$

Combining the above equations,

$$x_{n-1} = \frac{x_n + (D/L)x_D}{\alpha(V/L) - x_n(\alpha - 1) - x_D(\alpha - 1)(D/L)} \tag{8.117}$$

For total condensation in the overhead condenser, $x_D = y_1$. Also for constant molar flow rates on each tray, $L/D = R$ and $V/L = (R + 1/R)$. Substitution of these ratios in terms of reflux ratio, Eq. (8.117) can be solved.

By selecting R and x_D, x_{n-1} can be calculated from Eq. (8.117) which represents the mole fraction of light component on tray 2 from top. Similarly, for the next tray (i.e., number 3), x_{n-2} can be calculated from x_{n-1}. In this manner x_n can be calculated for all theoretical trays; the last one will represent x_W. Tray-to-tray calculations are summarised in Table 8.44.

For $x_D = 0.9982$, $N = 10$ and $\alpha = 4.448$,

Table 8.44 Tray-to-Tray Calculations for Varying Reflux Ratios

Tray No.	$R = 3$		$R = 4$	
	x_n	x_{n-1}	x_n	x_{n-1}
1	$x_D = 0.9982$	0.9920	$x_D = 0.9982$	0.9920
2	0.9920	0.9721	0.9920	0.9708
3	0.9721	0.9114	0.9708	0.9024
4	0.9114	0.7581	0.9024	0.7253
5	0.7581	0.5029	0.7253	0.4433
6	0.5029	0.2740	0.4433	0.2185
7	0.2740	0.1581	0.2185	0.1186
8	0.1581	0.1158	0.1186	0.0858
9	0.1158	0.1023	0.0858	0.0762
10	0.1023	$0.0982 = x_W$	0.0762	$0.0734 = x_W$

Similar calculations cab be made for different reflux ratios. These are summarised in Table 8.45.

Table 8.45 Computation of Integral between x_W and x_F

R	x_W	$1/(x_D - x_W)$	$R/(R + 1)$	$F'(x_W)$ Eq. (8.112a)	Integral value $F'(x_W)_{avg} \cdot \Delta x_W$
3	$x_F = 0.2727$	1.3784	0.7500	7.5995	
3	0.0981	1.1110	0.7500	4.9373	1.094 41
3.5	0.0840	1.0938	0.7778	5.3842	0.072 84

(*Contd.*)

Table 8.45 (*Contd.*)

R	x_W	$1/(x_D - x_W)$	$R/(R+1)$	$F'(x_W)$ Eq. (8.112a)	Integral value $F'(x_W)_{avg} \cdot \Delta x_W$
4	0.0731	1.0813	0.8000	5.8463	0.059 45
4.5	0.0652	1.0718	0.8182	6.3183	0.049 90
5	0.0587	1.0643	0.8333	6.7970	0.042 94
6	0.0489	1.0534	0.8571	7.7670	0.071 34
7	0.0419	1.0457	0.8750	8.7474	0.057 65
8	0.0366	1.0400	0.8889	9.7341	0.048 32
9	0.0326	1.0356	0.9000	10.7249	0.041 56
10	0.0293	1.0321	0.9091	11.7184	0.036 45
10.5	0.0279	1.0307	0.9130	12.2159	0.016 65 = x_W
Total					1.591 50

Compare integral value 1.637 47 (to attain $x_W = 0.028\ 75$) from graph (Fig. 8.33) with the value 1.5915 (to attain $x_W = 0.016\ 65$), obtained in Table 8.45. Both values differ by 2.81%. This difference can be attributed to the use of average volatility (α_{avg}) for numerical calculations.

Design of distillation column

Type of tower = Packed tower (because of small capacity)
Type of packing = Stainless Steel Pall rings
Size of packing = 16 mm
Void space = 92%
Packing factor $F_p = 230\ \text{m}^{-1}$ (Table 9.1)
Tower diameter required at bottom:

$$F_{LG} = \frac{L_w}{G_w}\left(\frac{\rho_G}{\rho_L}\right)^{\frac{1}{2}} \tag{9.15}$$

Molar flow rate of vapour, $V = 9.0748$ kmol/h

$$V = L + D \text{ and } L/D = R$$

Maximum value of actual or operating reflux ratio in the entire operation is 9.

$$L_{max} = R_{max}\, D = 9\, D$$

$$V = L + D = 9D + D = 10\, D$$

$$D = \frac{V}{10} = 0.907\ 48 \text{ kmol/h}$$

$$L = 8.1673 \text{ kmol/h}$$

Composition and average molar mass of vapour and liquid phases at bottom are changing with time. Average molar mass is maximum at the starting of distillation.

At the starting of distillation

$$M_{av} \simeq 0.2727 \times 32 + (1 - 0.2727) \times 18 \simeq 21.8178 \text{ kg/kmol}$$

$$\frac{L_W}{G_W} = \frac{L \times M_{av} / \left[\left(\frac{\pi}{4}\right) D^2\right]}{V \times M_{av} / \left[\left(\frac{\pi}{4}\right) D^2\right]} = \frac{L}{V} = 0.9$$

Bubble point temperature at $x = 0.2727$ is 79°C (from t–x–y data).

$$\rho_G = \frac{pM_{av}}{RT} = \frac{101.325 \times 21.8178}{8.314 \times (273 + 79)} = 0.7554 \text{ kg/m}^3$$

$$\rho_L = \frac{1}{\sum \frac{w_i}{\rho_{Li}}} = 1 / \left(\frac{0.4}{753} + \frac{0.6}{972.42}\right)$$

$$\rho_L = 870.9 \text{ kg/m}^3$$

$$F_{LG} = 0.9 \left(\frac{0.7554}{870.9}\right)^{\frac{1}{2}} = 0.0265$$

From Fig. 9.5, $K_F = 0.2$

Let actual velocity of vapour through packed tower = 70% of flooding velocity.

$$\sqrt{\frac{K}{K_F}} \times 100 = 70$$

$$K = (0.70)^2 \text{ or } K_F = 0.098$$

Corresponding $\Delta p = 83.3$ mm WC/m of packing (From Fig. 9.5).

$$G_W = \left(\frac{K \rho_G \rho_L g}{F_p \psi \mu_L^{0.2}}\right)^{\frac{1}{2}} \tag{9.18}$$

$K = 0.098$, $\rho_G = 0.7554$ kg/m^3, $\rho_L = 870.9$ kg/m^3

$F_p = 230$ m^{-1}

(Density of water is from Table 3.28, of Ref. 14)

$$\psi = \frac{\text{Density of water}}{\text{Density of liquid}} = \frac{972.42}{870.9} = 1.1166$$

ψ is inverse of specific gravity of liquid.

μ_L = viscosity of liquid solution

Viscosity of 40% methanol solution (by mass) at 79°C,

$\mu_L = 0.45$ cP $= 0.45$ mPa · s

(Fig. 3.43 of Ref. 8)

$$G_W = \left(\frac{0.098 \times 0.7554 \times 870.9 \times 9.81}{230 \times 1.1166 \times 0.45^{0.2}}\right)^{\frac{1}{2}}$$

$$= 1.7 \text{ kg/(m}^2 \cdot \text{s)}$$

Mass flow rate of vapour, $\dot{m}_v = 9.0748 \times 21.8178 = 198$ kg/h ≡ 0.055 kg/s

$$\text{Area required at bottom} = \frac{0.055}{1.7} = 0.032\,35 \text{ m}^2$$

$$A = 0.032\,35 = \left(\frac{\pi}{4}\right) D_i^2$$

$$D_i = 0.203 \text{ m}$$

Tower diameter required at top

$$\frac{L_W}{G_W} = \frac{L}{V} = 0.9$$

$$M_{av} = 0.9982 \times 32 + (1 - 0.9982) \times 18 = 31.9748 \text{ kg/kmol}$$

$$\rho_G = \frac{pM_{av}}{RT} = \frac{101.325 \times 31.9748}{8.314 \times (273 + 64.5)} = 1.1546 \text{ kg/m}^3$$

Top temperature is the dew point temperature of overhead vapour. It can be determined from *t-x-y* data. Find the value of t for $y = x_D = 0.9982$.

$$\rho_L = \text{Density of methanol at 64.5 °C} = 760 \text{ kg/m}^3$$

$$F_{LG} = 0.9\left(\frac{1.1546}{760}\right)^{1/2} = 0.035$$

From Fig. 9.3, $K_F = 0.18$

Let actual velocity of vapour is equal to 70% of flooding velocity.

$$K = (0.7)^2 K_F = 0.0882$$

Corresponding $\Delta p = 90$ mm WC/m of packing

$$\mu_L = \text{Viscosity of methanol at 64.5°C} = 0.34 \text{ cP} = 0.34 \text{ mPa} \cdot \text{s}$$

$$G_W = \left(\frac{0.0882 \times 1.1546 \times 760 \times 9.81}{230 \times 1.29 \times 0.34^{0.2}}\right)^{\frac{1}{2}}$$

$$= 1.7819 \text{ kg/(m}^2 \cdot \text{s)}$$

Mass flow rate of vapour, $\dot{m}_v = 9.0748 \times 31.9748 = 290.165$ kg/h ≡ 0.0806 kg/s

$$\text{Area required at top} = \frac{0.0806}{1.7819} = 0.045\,23 \text{ m}^2$$

$$D_i = \sqrt{\frac{4 \times 0.04523}{\pi}} = 0.24 \text{ m}$$

Select higher value of the tower diameter (D_i) of two values. Take $D_i = 0.24$ m for the entire tower.

HETP (height equivalent theoretical plate) for Pall rings = 0.5 m
Total height of packing = HETP × N = 0.5 × 10 = 5 m
Let the total height of packing = 5.1 m
Number of packing sections = 3 each with 1.7 m packed height
Type of liquid distributor = Pipe type
Number of holes required in pipe-type reflux distributor,

Fig. 8.34 *Pipe-Type Liquid Distributor*

$$n_h = \frac{(\pi/4)\, D_i^2 \times 10^4}{194}$$

For each 30 in^2 (194 cm^2) area, one distribution point is required.

$$n_h = \frac{(\pi/4)\,(0.24)^2 \times 10^4}{194} = 2.33$$

Let $n_h = 4$

However, the pipe of 25 mm NB is proposed for distribution.
Let the velocity of reflux through pipe = 2 m/s
Volumetric flow rate of reflux

$$= \frac{LM_{\text{av}}}{\rho_L \times 3600} = \frac{(0.9 \times 9.0748) \times 31.9748}{760 \times 3600}$$

$$= 9.545 \times 10^{-5}\ \text{m}^3/\text{s}$$

Inside diameter of pipe:

$$d_i = \sqrt{\frac{9.545 \times 10^{-5} \times 4}{2 \times \pi}} = 0.007\ 79 \text{ m}$$

Let $d_i = 8$ mm

Let the velocity of solvent through holes = 3 m/s

Let d_h = diameter of hole, mm

$$4 \times \frac{\pi}{4} d_h^2 = \frac{9.545 \times 10^{-5}}{3} = 3.181\ 67 \times 10^{-5}$$

$d_h = 0.0032$ m

Let $d_h = 3.5$ mm

Packing support

Packing support should be selected such that flow area provided by packing support for the flow of gas should be greater than flow area provided by packing material.

Void space for 16 mm size SS Pall rings = 92% (Table 9.1)

Hence with Pall rings, any gas-injection-type packing support must be used. Let the type of packing support is cap-type packing support.

Actual outer diameter of packing support is greater than 240 mm as some portion of packing support is sandwiched between two flanges.

Fig. 8.35 *Cap-Type Packing Support*

Let h = Height of slot in riser, mm
a = Width of slot in riser, mm
d_r = Diameter of riser, mm
n = Number of risers, mm
D_i = Inside diameter of column, mm

Let $$d_r = \frac{240}{6} = 40 \text{ mm}$$

Number of risers, $n = 9$

Let n_s = number of slots per riser

Total area of slots of risers = $n_s\, n\, h\, a$ where, distance between two successive slots is a mm.

Let $a = 5$ mm

$$n_s = \frac{\pi d_i}{2a} = \frac{\pi \times 40}{2 \times 5} = 12.566$$

Let $n_s = 12$

Hence, total area of slots of risers = $12 \times 9 \times h \times 5 \geq 0.92 \times \frac{\pi}{4}(240)^2$

$$h \geq 77 \text{ mm}$$

Let $h = 80$ mm

Process design of a binary batch rectification to attain definite minimum concentration of distillate In many cases of batch distillation, it is not required to attain high purity of distillate as discussed in the previous section. Since the recovered distillate is reused in the plant, purity requirement can be lower but a certain minimum concentration of more volatile component is desired. For design of such a system, at times reflux ratio is kept constant and distillation is carried out for a known period. For such designs, graphical[2] and numerical procedures[24] differ from the procedures described in the previous section. Reader is advised to see the original references for the procedures.

8.5.3 Process Design of Multicomponent Batch Rectification

Stepwise procedure for process design for multicomponent batch rectification[2] with constant overhead composition is as under.

1. Select light key and heavy key components.
2. Fix the distribution of light key and heavy key component in distillate and final residue. Calculate the distribution of non-key components in distillate and residue and complete the material balance using Eq. (8.108).
3. Calculate dewpoint of distillate, bubble point of feed and bubble point of final residue.
4. Calculate the average relative volatilities of all components with respect to heavy key component.

5. Calculate the minimum number of equilibrium stages (N_m) required for the desired separation by Fenskey's equation Eq. (8.26).
6. Fix the number of equilibrium stages required for the desired separation. N can be 1.5 to 3 times N_m.
7. Keep mole fraction of light key component in distillate (x_{LD}) and mole fraction of heavy key component in distillate (x_{HD}) as constant. Change the values of mole fraction of light key in residue (x_{LW}) from x_{LF} to x_{LB}, where, x_{LF} and x_{LB} are mole fractions of light key in feed and in final residue, respectively. For any intermediate value of x_{LW}, corresponding value of x_{HW} can be determined by material balance.

$$n_F = n_{Di} + n_{Wi} \qquad (i = \text{intermediate value}) \tag{8.118}$$

$$n_F \cdot x_{LF} = n_{Di} \cdot x_{LD} + n_{Wi} \cdot x_{LW} \tag{8.119}$$

From equations (8.118) and (8.119), calculate n_{Di} and n_{Wi}. Then find x_{HW} by using following material balance equations.

$$n_F \cdot x_{HF} = n_{Di} \cdot x_{HD} + n_{Wi} \cdot x_{HW} \tag{8.120}$$

8. Find R_m by following equation:

$$R_m = \frac{1}{(\alpha - 1)}\left[\left(\frac{x_{LD}}{x_{LW}}\right) - \alpha\left(\frac{x_{HD}}{x_{HW}}\right)\right] \tag{8.121}$$

where α is average relative volatility of light key with respect to heavy key.

9. Find the actual reflux ratio R based on the values of R_m, N_m and N by using Gilliland's correlation, Eq. (8.28) and Eq. (8.29) or Fig. 8.7.
10. Calculate $\frac{L}{V}$ by the following equation:

$$\frac{L}{V} = \frac{R}{R+1} \tag{8.122}$$

Thus for the different values of x_{LW} from x_{LF} to x_{LB}, corresponding values of x_{HW}, R_m, R and L/V can be determined.

11. Time required for multi component batch distillation with rectification and with constant overhead composition can be calculated by Bogart equation:

$$\theta = \frac{n_F(x_{LD} - x_{LF})}{V} \int_{x_{LB}}^{x_{LF}} \frac{dx_{LW}}{(1 - L/V)(x_{LD} - x_{LW})^2} \tag{8.123}$$

12. Develop R vs θ curve.

Example 8.17 Determine the reflux ratio vs time data for the batch distillation with rectification for the following mixture.

Feed: 200 kmol with 50% benzene, 25% toluene and 25% *ortho* xylene (by mole)
Distillate: 99% pure benzene (by mole)

Distillate composition should remain constant throughout the course of distillation. Distillation is to be continued until the moles of benzene is reduced to 10 kmol in the residue.
Data: Antoine equation:

$$\text{Log}_{10}\, p_v = A - \frac{B}{T + C} \tag{2.1}$$

where, p_v is vapour pressure in bar and T is the temperature in K.

Table 8.46 Antoine Constants

Component	*A*	*B*	*C*
Benzene	4.725 83	1660.652	–1.461
Toluene	4.078 27	1343.943	–53.773
o-Xylene	4.129 28	1478.244	–59.076

Solution

Here benzene is light key and toluene are heavy key components.
Assume that remaining 1% of distillate is toluene.

Table 8.47 Material Balance

Component	Feed		Distillate		Residue	
	kmol	x_F	kmol	x_{iD}	kmol	x_{iB}
Benzene	100	0.5	90	0.99	10	0.0917
Toluene	50	0.25	0.91	0.01	49.09	0.4500
o-Xylene	50	0.25	–	–	50	0.4583
Total	200	1.00	90.91	1.00	109.09	1.0000

Bubble point temperature of feed

Operating pressure = 1 atm = 1.013 25 bar

At 96°C (369 K)

$$p_{vB} = 1.6126 \text{ bar}, \quad p_{vT} = 0.6529 \text{ bar}, \quad p_{vX} = 0.2289 \text{ bar}$$

$$\sum p_{vi} \cdot x_{Fi} = 1.6126 \times 0.5 + 0.6529 \times 0.25 + 0.2289 \times 0.25 = 1.027 \text{ bar}$$
$$\cong 1 \text{ atm}$$

Hence bubble point of feed = 96°C

Dewpoint of distillate

At dewpoint temperature, $\sum \frac{y_{Di}\ p_t}{p_{vi}} = 1$

$$\left(\frac{0.99}{p_{vB}}+\frac{0.01}{p_{vT}}\right)\times 1.013\,25=1$$

At 81°C (354 K),

$$\left(\frac{0.99}{1.0358}+\frac{0.01}{0.4}\right)\times 1.013\,25=0.9937\cong 1$$

Hence, dewpoint of distillate = 81°C (354 K)

Bubble point of final residue

At 117°C (390 K)

$$p_{vB}=2.8297 \text{ bar},\quad p_{vT}=1.2054 \text{ bar},\quad p_{vX}=0.4595 \text{ bar}$$

$$\sum x_{Bi}\,p_{vi}=0.0917\times 2.8297+0.45\times 1.2054+0.4583\times 0.4595=1.0125 \text{ bar}$$
$$\equiv 1.013\,25$$

Hence bubble point of final residue = 117°C (390 K)

Average relative volatilities of components with respect to heavy key

$$\alpha_{Bi}=\left[\left(\frac{1.6126}{0.6529}\right)\left(\frac{1.0358}{0.4}\right)\right]^{1/2}=2.529$$

$$\alpha_{Bf}=\left[\left(\frac{1.0358}{0.4}\right)\left(\frac{2.8297}{1.2054}\right)\right]^{1/2}=2.4655$$

$$\alpha_B=(2.529\times 2.4655)^{1/2}=2.497$$
$$\alpha_T=1$$

Vapour pressure of *o*-xylene at 81°C,

$$p_{vX}=0.1309 \text{ bar}$$

$$\alpha_{Xi}=\left[\left(\frac{0.1309}{0.4}\right)\left(\frac{0.2289}{0.6529}\right)\right]^{1/2}=0.3387$$

$$\alpha_{Xf}=\left[\left(\frac{0.1309}{0.4}\right)\times\left(\frac{0.4595}{1.2054}\right)\right]^{1/2}=0.3532$$

$$\alpha_X=0.3459$$

Minimum number of theoretical stages required for the desired separation

$$N_m=\frac{\log\left[\left(\frac{x_{LK}}{x_{HK}}\right)_d\left(\frac{x_{HK}}{x_{LK}}\right)_b\right]}{\log\alpha_{LK}}$$

$$N_m = \frac{\log\left[\left(\frac{0.99}{0.01}\right)_d \left(\frac{0.45}{0.0917}\right)_b\right]}{\log(2.497)}$$

$$= 6.7598$$

Let actual number of theoretical stayes required for the desired separation, $N = 12$

$$\frac{N - N_m}{N+1} = \frac{12 - 6.7598}{12+1} = 0.4031$$

Using Eq. (8.28) and Eq. (8.29),

$$\frac{R - R_m}{R+1} = 0.2615$$

From Fig. 8.7, $\dfrac{R - R_m}{R+1} = 0.2678$

For $x_{LW} = x_{LF} = 0.5$

$$R_m = \frac{1}{(\alpha - 1)}\left[\left(\frac{x_{LD}}{x_{LW}}\right) - \alpha\left(\frac{x_{HD}}{x_{HW}}\right)\right]$$

$$R_m = \frac{1}{(2.497 - 1)}\left[\left(\frac{0.99}{0.5}\right) - 2.497\left(\frac{0.01}{0.25}\right)\right]$$

$$R_m = 1.256$$

$$\frac{R - 1.256}{R+1} = 0.2615$$

$$R = 2.055, \qquad \frac{L}{V} = \frac{R}{R+1} = \frac{2.055}{3.055} = 0.6726$$

$$\frac{1}{(1 - L/V)(x_{LD} - x_{LW})^2} = \frac{1}{(1 - 0.6726)(0.99 - 0.5)^2} = 12.721$$

For $x_{LW} = 0.4$

$$R_m = \frac{1}{(2.497 - 1)}\left[\left(\frac{0.99}{0.4}\right) - 2.497\left(\frac{0.01}{x_{HW}}\right)\right]$$

$$n_F = n_D + n_W = 200$$

$$= n_{Di} + n_{Wi}$$

$$n_F \cdot x_{LF} = n_{Di} \cdot x_{LD} + n_{Wi} \cdot x_{LW}$$

$$200 \times 0.5 = n_{Di} \times 0.99 + n_{Wi} \times 0.4$$
$$= n_{Di} \times 0.99 + (200 - n_{Di}) \times 0.4$$
$$n_{Di} = 33.9 \text{ kmol}, \quad n_{Wi} = 166.1 \text{ kmol}$$
$$n_F x_{HF} = n_{Di} x_{HD} + n_{Wi} x_{HW}$$
$$200 \times 0.25 = 33.9 \times 0.01 + 166.1 \times x_{HW}$$
$$x_{HW} = 0.299$$
$$R_m = 1.5975$$

$$\frac{R - 1.5975}{R + 1} = 0.2615$$

$$R = 2.5173 \quad \frac{L}{V} = \frac{2.5173}{1 + 2.5173} = 0.7157$$

$$\frac{1}{\left(1 - \frac{L}{V}\right)\left(x_{LD} - x_{LW}\right)^2} = \frac{1}{(1 - 0.7157)\,(0.99 - 0.4)^2} = 10.1046$$

Similarly, following table can be prepared:

Table 8.48 Multicomponent Batch Rectification Data

x_{LW}	x_{HW}	R_m	R	L/V	$F''(x_{LW}) = \frac{1}{\left[(1 - L/V)\,(x_{LD} - x_{LW})^2\right]}$
0.5	0.25	1.256	2.055	0.6727	12.7239
0.4	0.299	1.5975	2.5173	0.7157	10.1043
0.3	0.348	2.156	3.2735	0.7660	8.9761
0.2	0.3969	3.2646	4.7747	0.8268	9.2528
0.15	0.4214	4.369	6.2701	0.8625	10.3034
0.0917	0.45	7.1747	10.0693	0.9097	13.7176

Figure (8.36) is the plot of x_{LW} vs $F''(x_{LW}) = \frac{1}{\left[(1 - L/V)\,(x_{LD} - x_{LW})^2\right]}$.

Fig. 8.36 *Evaluation of Integral for Batch Distillation with Rectification*

Total area under the curve = 4.1211 (integral value)

$$\theta = \frac{n_F\,(x_{LD} - x_{LF})}{V} \int_{x_{LB}}^{x_{LF}} \frac{dx_{LW}}{\left(1 - \dfrac{L}{V}\right)(x_{LD} - x_{LW})^2}$$

$$\theta = \frac{200\,(0.99 - 0.5)}{V} \times \text{Area under curve from } x_{LW} = 0.0917 \text{ to } 0.5$$

$$V\theta = 200\,(0.99 - 0.5) \times 4.1211 = 403.87 \text{ kmol}$$

Batch rectification data, presented in Table 8.48, were fitted (using Excel®) in an equation form.

$$F''(x_{LW}) = 1204.42x_{LW}^4 - 1626.18x_{LW}^3 + 861.85x_{LW}^2 - 201.72x_{LW} + 26.12$$

$$(\boldsymbol{R}^2 = 1.00)$$

$$\text{Integral} = \int_{0.0917}^{0.5} F''(x_{LW})\,dx_{LW} = 4.1326$$

Let θ = 6 h (total time period for batch distillation)

V = 403.87/6 = 67.31 kmol/h

Area of segment [from $x_W = x_F = 0.5$ to $x_W = 0.4$]

For R = 2.5173

= 1.1197

$$\theta_1 = \frac{1.1197 \times 6}{4.1211} = 1.6031 \text{ h}$$

Area of segment [from $x_W = x_F = 0.5$ to $x_W = 0.3$] = 2.0665

For R = 3.2735

$$\theta_2 = \frac{2.0665 \times 6}{4.1211} = 3.0087 \text{ h}$$

Area of segment [from $x_W = x_F = 0.5$ to $x_W = 0.2$] = 2.9069

For R = 4.7747

$$\theta_3 = \frac{2.9609}{4.1211} \times 6 = 4.3109 \text{ h}$$

Area of segment [from $x_W = x_F = 0.5$ to $x_W = 0.15$] = 3.4466

For R = 6.2710

$$\theta_4 = \frac{3.4466}{4.1211} \times 6 = 5.018 \text{ h}$$

Using R_i vs θ_i data, Fig. 8.37 is prepared. For integral values of θ_i, R_i values are read from Fig. 8.37 which are tabulated in Table 8.49.

Fig. 8.37 *Evaluation of Integral for Batch Distillation with Rectification*

Table 8.49 Calculated R vs θ

θ, h	R
0	2.055
1	2.35
2	2.691
3	3.267
4	4.28
5	6.226
6	10.07

8.6 SHORT PATH DISTILLATION[25,26]

Short path distillation is very high vacuum distillation. Operating pressure at distillation surface in short path distillation unit (SPDU) could be as low as 10^{-6} bar (1 μbar). Short path distillation provides the distillation at the minimum possible temperature which is desirable for heat sensitive products.

In distillation, maximum boiling point is of the bottom product. If the normal boiling point of any product of distillation is higher than the decomposition temperature or cracking temperature, distillation cannot be carried out at atmospheric pressure. Reduction in absolute pressure results in the reduction in distillation temperature or boiling temperature. In lower pressure range, more reduction in boiling temperature of pure component is observed for a small reduction in operating pressure. For example, boiling point of stearic acid is reduced by 0.2°C when operating pressure is reduced from 251 mbar to 250 mbar. But, its boiling point is reduced by 3.8°C when operating pressure is reduced from 5 mbar to 4 mbar. In both cases, reduction in operating pressure is 1 mbar but in lower pressure range reduction in boiling point is quite high. Hence, reduction of pressure in lower pressure range is important as it creates considerable reduction in boiling point. For a food grade product (for example, orange juice) and for a highly heat sensitive product (for example, vitamin E, i.e., α–tocopherol) reduction in boiling point means better recovery. Maximum recovery is obtained at the minimum boiling temperature.

Decreases in pressure results in the increase of vapour volume per unit mass and in turn it results in increase in vapour velocity. In the lower pressure range, even low vapour flow rate or non-condensable flow rate results in relatively higher value of pressure drop.

It will be interesting to note change in properties of ice and water vapour from Table 8.50 at low, high and ultra high vacuum.

Table 8.50 Properties of Water, Ice and Water Vapour at Low Temperatures[27]

Melting point		Saturation vapour pressure, p_s		Density of ice, ρ_s	Specific volume of saturated vapour, V	Specific enthalpy, kJ/kg		
T	t							
K	°C	Pa	bar	kg/m^3	m^3/kg	Ice	Sublimation	Vapour
273.15	0	611.65	0.611×10^{-2}	916.708	205.99	–333.4	2834.3	2500.9
270	–3.15	470.06	0.470×10^{-2}	917.170	264.96	–340.0	2835.1	2495.1
265	–8.15	305.92	0.306×10^{-2}	917.886	399.64	–350.2	2836.2	2486.0
260	–13.15	195.81	0.196×10^{-2}	918.594	612.65	–360.3	2837.1	2476.8
255	–18.15	123.15	0.123×10^{-2}	919.295	955.48	–370.2	2837.8	2467.6
250	–23.15	76.02	0.760×10^{-3}	919.980	1517.5	–380.0	2838.3	2458.3
245	–28.15	46.01	0.460×10^{-3}	920.658	2457.3	–389.5	2838.6	2449.1
240	–33.15	27.27	0.273×10^{-3}	921.319	4061.4	–398.9	2838.7	2439.8
235	–38.15	15.81	0.158×10^{-3}	921.973	6860.7	–408.0	2838.6	2430.6

(*Contd.*)

Table 8.50 (*Contd.*)

Melting point		Saturation vapour pressure, p_s		Density of ice, ρ_s	Specific volume of saturated vapour, V	Specific enthalpy, kJ/kg		
T	t							
K	°C	Pa	bar	kg/m^3	m^3/kg	Ice	Sublimation	Vapour
230	–43.15	8.95	0.895×10^{-4}	922.611	11 861	–417.0	2838.4	2421.4
225	–48.15	4.94	0.494×10^{-4}	923.233	21 021	–425.9	2838.0	2412.1
220	–53.15	2.65	0.265×10^{-4}	923.839	38 249	–434.5	2837.3	2402.8
215	–58.15	1.39	0.139×10^{-4}	924.437	71 588	–443.0	2836.6	2393.6
210	–63.15	0.702	0.702×10^{-5}	925.018	1.3809×10^5	–451.3	2835.6	2384.3
205	–68.15	0.344	0.344×10^{-5}	925.583	2.7514×10^5	–459.4	2834.4	2375.0
200	–73.15	0.1626	1.626×10^{-6}	926.132	5.6759×10^5	–467.3	2833.1	2365.8
195	–78.15	0.7404	0.740×10^{-6}	926.655	1.2155×10^6	–475.1	2831.6	2356.5

Reference State:
Specific enthalpy of saturated liquid water at 273.16 K (0.01°C) = 0 kJ/kg

Specific volume of saturated water vapour at 0.1 bar (boiling point = 45.83°C), 1.232×10^{-3} bar (or 1.232 mbar) and 1.626×10^{-6} bar (or 1.626 μbar) is 14.675 m^3/kg, 955.48 m^3/kg and 5.6759×10^5 m^3/kg, respectively. Thus several fold increase in vapour volume will have to be handled at ultra high vacuum. This will call for big size pipes (ducts) for transporting vapours at ultra high vacuum so that low pressure drops are achieved.

Hence, in all vacuum distillation columns, minimum possible value of absolute pressure at vaporisation surface of boiling liquid in reboiler is decided by pressure drop of vapour flowing from vaporisation surface of liquid in reboiler to the heat transfer surface of external condenser. In conventional packed tower-type vacuum distillation column, vapour travels a long path from reboiler to external condenser. Also, in conventional design, no extra care is generally taken to reduce the flow of non-condensables.

To break this physical barrier of pressure drop of vapour and non-condensables travelling very long path from vapourising surface to external condenser, in short path distillation, an internal condenser is used and because of the same, vapour travels through a very short distance, in order of few centimetres, from vaporisation surface of liquid film over heating surface to the surface of internal condenser. Because of the shortening of the path length of vapour and non-condensables, this new unit is known as short path distillation unit (SPDU).

There is a confusion in between the words "Molecular Distillation" and "Short Path Distillation". In the latter, if the distance travelled by vapour molecules from the vaporisation surface to the surface of internal condenser is less than mean free path of molecules between two successive collisions, then short path distillation is called molecular distillation. If in this path length, there is no collision between vapour molecules then pressure drop in vapour flow is the minimum. Decrease in pressure increases the mean free path of molecules between two successive collisions. For

commercial applications, it is not necessary to achieve the condition of molecular distillation for each application, hence, only a few short path distillation units are operated as molecular distillation units.

8.6.1 Design and Working of Short Path Distillation Unit

A short path distillation unit (Fig. 8.38) consists of a vertically mounted double walled vacuum chamber with a central inner condenser and roller wiper system.

Fig. 8.38 *Typical Short Path Distillation Unit*

Feed is continuously fed on a rotating distributor plate. At the bottom of rotating distribution plate, 3 to 12 guiding rods are mounted. These guiding rods contain the rollers which are passed over the rods. Distribution plate with rods and rollers is rotated by an electric motor. This entire rotating system is known as Roller–Wiper system. Feed material is thrown by rotating distributor to wall or to heat transfer surface. Then material flows down by gravity over the heat transfer surface. Rollers do not touch the heat transfer surface but they create the pressure and turbulence in liquid film. Rollers are rotated about the central axis of the unit and are also rotated about their own axis. Friction between liquid film and rollers create the automatic rotation in rollers about their own axis. This roller wiper system creates effectively mixed, uniform and thin film over the heating surface. Heating is provided by thermic fluid, saturated steam or hot water. Liquid film is partially vaporized. Vapour travel through the shortest path from liquid film surface to the surface of inner central condenser. Cooling medium may be cooling water, chilled water, oil or brine.

Residue and condensate (distillate) both are falling down by gravity. Partition is provided to avoid the possibility of the mixing of residue and distillate.

Vacuum nozzle located at the side bottom of vessel, through which non-condensables (and some vapours) are drawn out by vacuum system. Vapours are condensed in a cold trap. Vacuum measuring instrument is also provided in the bottom part.

Unit is kept in the perfect vertical position and hence, residence time of liquid on the heating surface is in the order of few seconds. The distillation takes place in "single pass" or in "once through". In this operation, in the short residence time and the lowest, vaporisation temperature reduces the possibility of thermal degradation of product. Thus SPDU provides the most gentle conditions for the distillation of heat sensitive products.

Sometimes it is advisable to have a degassing stage in front of short path distillation unit to remove the dissolved noncondensable gases like oxygen, nitrogen, etc. In addition to this maximum tightness of the system, the prevention of gaseous cracked products and the lack of stripping gas reduce size of vacuum system required and also reduces the operating cost of the vacuum system.

Standard short path distillation units with heating surface area range from 0.01 m^2 to 42 m^2, heating temperature up to 450°C and product throughput range from a few g/h to several t/h (multiple units) are available.

Theoretical treatment of agitated thin film evaporator is given by Salden[28] in which heat transfer surface area requirement can be calculated (i.e., estimated) by knowing properties of the fluid to be processed in the evaporator and the heating medium. This area correspondences to the jacket heat transfer surface. However, most recognised suppliers of such equipments provide trial facility on a pilot model. It is recommended to conduct some trials on the pilot plant and the model suitable for the service is selected.

8.6.2 Applications of SPDU[25,26]

The use of short path distillation to modify the traditional processes or for the development of a new process requires special knowledge because many classical rules and laws of traditional vacuum distillation cannot apply to short path distillation.

Short path distillation finds its application in pharmaceutical industries, fine chemicals industry, polymer industries, food industries, oil industries and refineries. Some important applications are:

1. Separation of Vitamin E: The short path distillation is applied for synthetically produced vitamin-E as well as for natural vitamin-E. The synthesised vitamin-E exists in the final stage as acetic acid ester (as vitamin E-acetate). Typically 100 kg crude vitamin-E acetate contains 78.75 kg pure vitamin-E acetate, 13.75 kg residue and 7.5 kg low boiling impurities. In two-stage short path distillation, pure vitamin E acetate is obtained as light yellow pure oil.

Deodoriser distillate of an edible oil refinery is a source of natural vitamin-E. Its concentration varies from 3 to 10% (by mass) in the distillate of soybean oil, depending on the operating conditions of the deodoriser. Concentration up to 40% (by mass) can be carried out in SPDU as shown in Fig. 8.39.

Fig. 8.39 *Distillation of Vitamin-E*

2. Separation of Monoglyceride:

Monoglycerides are mono fatty acid esters of glycerol.

Mono fatty acid + Glycerol → Monoglyceride + Water

Monoglycerides are used as emulsifiers in many of food products. The modern food product technology demands the concentration of monoglyceride more than 90%. It is a heat sensitive product. More than 90% concentration of monoglycerides is achieved by two-stage short path distillation (Fig. 8.40).

3. Carotene: It is a pro-vitamin which is used as a supplement in many food products. Red palmoil ester contains about 0.06% carotene. Three-stage short path distillation plant (Fig. 8.41) is required to increase the concentration up to 40%.

4. Dimeric Fatty Acids: They are basic materials for polyester or alkyd resins. They are produced by dimerisation of oleic acid. After reaction a mixture of monomer, dimer and oligomer fatty acids are formed. The fractionation of this mixture takes place in two or there stage short path distillation plant (Fig. 8.42).

5. To recover more middle distillate from the residuum, coming from vacuum distillation unit of refinery, currently vacuum distillation is carried out in conventional distillation equipment in which operating pressure at the base of column or in reboiler is decided by pressure drop in the column. The same operating pressure also decides the certain boiling range of residuum. Currently, considerable amount of middle distillate remains in the residuum. With conventional vacuum distillation unit, one can

Fig. 8.40 *Manufacturing of Monoglyceride*

Fig. 8.41 *Concentration of Cartone in Palm Oil Ester*

increase the % recovery of middle distillate by increasing base temperature but the same cannot be permitted because increase in the base temperature above the certain limiting value results in considerable thermal cracking of the residuum. In short path distillation, the same residuum can be boiled or distilled at much lower temperature range. Reduction in operating pressure in SPDU creates the sizeable reduction in boiling temperatures. Hence, with SPDU, it is possible to recover the middle distillate further without causing thermal cracking.

6. Oleoresin: Oleoresin is a red colour natural pigment separated from paprika. It is used as a natural colour additive in food products. The oleoresin is extracted by hexane. From the extracted product, which contains 96% oleoresins and 4% hexane (by mass), hexane can be completely removed from oleoresin (down to 10 ppm) by two stage short path distillation (Fig. 8.43).

Fig. 8.42 *Separation of Dimeric Fatty Acids*

Fig. 8.43 *Manufacturing of Oleoresins*

7. Deacidification of Ricebran Oil and Palm Oil: Ricebran oil and palm oil are preferred to be used as cooking mediums because of their high nutritional values. But because of considerable high content of free fatty acids, deacidification by conventional

distillation is difficult. With short path distillation, these oils can be gently deacidified to the concentrations of less than 0.1% without loss of nutritional values.

8. Separation of Monomers from Prepolymers: Polyurethanes are polymers with different compositions and wide range of characteristics. They are produced by polyadduct-type polycondensation reaction from toluene diisocyanate and polyols. In the first step, so-called prepolymers are formed. These prepolymers still contain a certain amount of non-reacted diisocyanate monomers. It is necessary to reduce monomer content before selling to end manufacturers because these monomers are toxic in nature. It can be reduced by short path distillation.

8.6.3 Economics

It is a general impression that cost of short path distillation unit is quite high. The reason for this is historical. The first industrial short path distillation plants were operated with very low throughputs to maintain the very low value of operating pressure at distillation surface. Vacuum technology was not advanced at that time and this was the limiting factor for the throughput. Also in initial stages, there were high overdimensioning of the unit. These all reasons created the general impression that short path distillation is very expensive operation and the distillation cost can only be borne by very expensive products. But now, reality is different.

Improved vacuum technology, considerately higher throughput quantities, increased availability of units from different manufacturers and low personnel requirements with automated units have led to a substantial reduction in cost of distillation. Results have shown that on increasing product throughput from 150 kg/h to 3600 kg/h, total cost of short-path distillation in ₹/kg of product reduces by more than 85%.

Short-path distillation unit requires only one week shutdown time per year. Hence, it requires relatively less maintenance.

8.7 REACTIVE AND CATALYTIC DISTILLATION[29,30,35]

In most of the chemical plants, one can find the reaction followed by separation. Usually reaction and separation are performed one by one. First reactants are allowed to react in the reactor, then in a separate equipment product mixture is separated from unconverted reactants and/or from byproducts and inerts. It is rarely seen that both reaction and separation combined and carried out simultaneously in one equipment.

However, by combining reaction and separation of product in one equipment one can get two advantages:

1. Removal of the product from the reaction mass at the reaction conditions increases equilibrium conversion of reactant or increases the extent of reaction. As per Le Chātelier's principle, for the reaction in equilibrium, if any change in any process variable like pressure, temperature or composition of reaction mass is made, reaction or process is moved in such a direction which will nullify the effect of the change in the variable. In reactive distillation, change in the composition of reaction mass is made by continuously removing the product from the reacting mass. Hence, to nullify the effect of this change, reaction

proceeds in the forward direction and produces more and more products and so higher conversion is achieved.

2. Separate equipment for the separation is not required. Also separate piping, instruments, etc., are not required.

   There are four commonly used techniques to combine reaction and separation:

   (a) Reactive distillation (Reaction + Distillation)
   (b) Membrane reaction (Reaction + Membrane separation)
   (c) Extractive reaction (Reaction + Extraction)
   (d) Reaction with absorption (Reaction + Absorption)

In reactive distillation, reaction and distillation both are carried out simultaneously in one equipment. If reactive distillation is carried out in presence of heterogeneous solid catalyst, then it is called *catalytic reactive distillation*.

The concept of reactive distillation is not new. This technique was first applied in 1920 to esterification process using homogeneous liquid phase catalyst. In manufacturing of ethyl acetate, reaction between ethanol and acetic acid and separation of crude ethyl acetate from reaction mass by distillation were carried out simultaneously in one equipment. Ethanol was used as excess reactant. Overhead vapour from reactive distillation column is a ternary azeotrope having composition 82.16% ethyl acetate, 8.4% ethanol and 9% water (by mass). On condensation heteroazeotrope liquid is converted into two layers, ester rich layer and ethanol-water solution layer. Part of ester rich layer is taken out as top product (distillate) and remaining is recycled back to column as reflux. Sulphuric acid was added in feed as catalyst. Waste water (water + sulphuric acid) is continuously taken out as bottom product. Alcohol-water stream is taken out as an intermediate product.

In manufacturing of butyl acetate overhead vapour is a ternary heteroazeotrope of butyl acetate, butanol and water. On condensation, it is converted into two immiscible liquid layers.

Table 8.49 Ternary Azeotrope Separation at 90°C[31]

	Layer % by mass	% by mass		
		Ester	Butanol	Water
Upper	75	80.5	13.4	6.1
Lower	25	1.92	1.69	97.39

(Table 12.7 of Ref. 31)

Lower aqueous layer is continuously taken out from decanter for recovery of butanol (separately) while upper layer or ester rich layer is recycled back to column as a source of reflux. Bottom product is crude butyl acetate. It is sent for further purification. Thus in both cases, concept of reactive distillation, invented in 1920, was applied. But after that for a long period of time very little work was done in this direction. Almost after 50 years, an important research work was done by Sermewald in 1971[32], that applying reactive distillation with heterogeneous catalyst is far more useful than homogeneous catalyst. Nowadays, this technique is being successfully applied to some processes like synthesis of methyl tertiary butyl ether (MTBE), synthesis of ethyl tertiary butyl

ether (ETBE), selective hydrogenation of butadine of C_4 stream, synthesis of ethyl propionate, methyl acetate, etc.

If a solid catayst is used, then the same catalyst can also act as packing material of packed tower. In that case solid catalyst plays double role to enhance the rate of chemical reaction as well as to enhance the rate of mass transfer for distillation. This special type of reactive distillation which uses solid catalyst is known as catalytic distillation.

Even though reactive distillation has two major advantages over conventional reactor followed by distillation, it finds its place only in few processes. Reason is, it also has one major disadvantage.

Disadvantage

In majority of the processes which involve reaction followed by distillation, optimum conditions for reaction and optimum conditions for distillation are different. Hence, to carry out both symultaneously in one equipment, one has to compromise in reaction or in distillation. In most of the cases, the compromise is in the distillation. In a few cases like in the process of MTBE, optimum conditions for the reaction and the same for distillation are more or less same. Hence, one can apply the concept of catalytic distillation in the process of MTBE. But in many cases, this combination is not economically feasible. For example, in the manufacturing process of acetic acid by carbonylation of methanol, first reaction is carried out in presence of homogeneous phase catalyst at 50 atm pressure and at 180°C temperature with conversion of methanol up to 50%. After the reaction, reactants, promoters and catalyst are separated from acetic acid by distillation at atmospheric or near atmospheric pressure. Hence, in this case one cannot apply the concept of reactive distillation because optimum conditions for the reaction and for the distillation are very much different.

8.7.1 Design Methods for Reaction Distillation System

Complex process design of reactive distillation system has received significant attention from many research scientists. As such the system involves simultaneous occurrence of distillation and reaction, high non-ideality of the mixtures in question and the formation of azeotropes. In developing a model of the reactive distillation system, problems are experienced due to simultaneous chemical and physical equilibriums and the types of variables involved in defining the hypothesis, general composition variables in molar units and the extents of reactions are the variables that are considered in modelling.

There are a number of methods, reported in literature for modelling reactive distillation system. A short method, presented by M. Carrera-Rodriguez *et al.*[33,34] is based on tray-to-tray calculations with a strategy to find reflux ratio and to locate the feed tray that minimises the total number of stages. The method is demonstrated for modelling synthesis of MTBE from *i*-butene and methanol in presence of an inert; *n*-butane.

Another method is based on Ponchon-Savarit diagram for design of a binary reactive distillation column. Developed by J. W. Lee *et al.*[4] it is more reliable as enthalpies are used in preparation of the diagram. Enthalpy values are reworked to take care of the heat of reaction. It is assumed that reaction takes place in the rectifying section

and hence the enthalpy ordinate for the rectifying section only is modified. With the assumption of no reaction in the stripping section, its enthalpy ordinate is not required to be modified.

Reader is advised to refer original articles for more details.

Kai Sundmacher and Achim Kienle have authored a book[35] on reactive distillation. It is an exhaustive volume on the subject covering design and practical aspects

8.7.2 Comparison of Reactive Distillation with Conventional Technology

Conventional technology involves two steps (Fig. 8.44).

Fig. 8.44 *Typical Conventional Process*

In the conventional process, first step is the reaction step. In this step, reactants are brought into contact with solid or liquid catalyst at appropriate temperature and pressure.

In second step, stream from the reactor is sent to separation section, where desired product is separated from unconverted reactants, catalyst, byproducts and/or inerts. In most of the cases, this separation is done by distillation.

When concept of reactive distillation (or catalytic distillation, if catalyst is in solid phase) is applied to such type of processes, flow sheet is changed. Typical flow sheet of catalytic distillation column is shown in Fig. 8.45.

Fig. 8.45 *Typical Flow Sheet of Catalytic Distillation*

In the middle section of catalytic distillation column, reaction and distillation take place simultaneously while upper and lower sections represent enriching and stripping sections of distillation or they are utilised only for separation.

The advantages of reactive distillation can be described best by considering its advantages over reaction step and distillation step of conventional process.

Advantages over reaction step

1. For equilibrium limiting reaction, continuous removal of one of the products at process conditions increases equilibrium conversion. Thus reactive distillation provides high conversion compared to conventional reaction step.
2. For exothermic or endothermic reaction, separate cooling or heating system is required for reactor in conventional process. In reactive distillation, separate heating or cooling system is not required for reaction zone. In reactive distillation, heat of reaction is utilised in distillation either for condensation or for boiling, thus achieving energy conservation.

Advantages over distillation step

Advantages related to reduction in capital cost

1. In conventional technology, separate equipment is required for distillation. Reactive distillation eliminates use of major process equipment and associated pump, piping and instrumentation.
2. Heat of reaction reduces either condensation duty or reboiling duty. Hence, it either reduces condenser size or reboiler size.
3. In reactive distillation, very less amount of uncoverted reactants are to be separated from products compared to distillation in conventional technology. Hence, reactive distillation requires lesser number of stages for same value of reflux or less amount of reflux compared to distillation of conventional technology for the same value of reflux ratio or it requires lesser size of distillation column compared to the same required for the distillation in conventional technology for the given production rate. Less amount of reflux also decreases sizes of condenser and reboiler.

Advantages related to reduction in operating cost

1. Heat of reaction reduces either condensation duty or reboiling heat duty. Hence, it reduces either load of cooling system or the same of heating system. Consequently, it reduces operating cost.
2. Operating cost required for recycling of unreacted reactants is reduced considerably.
3. In reactive distillation, amount of distillate is reduced considerably. Hence, for the same reflux ratio, lesser amount of reflux is required in reactive distillation compared to distillation column of conventional technology. It decreases condenser heat duty, reboiler heat duty and power required for pumping the reflux.

8.7.3 Different Ways by Which Reactive Distillation Can be Applied

There are three different ways by which Reactive Distillation can be applied:

1. Reactive distillation alone
2. Reactive distillation plus non-reactive distillation
3. Reaction alone plus reactive distillation plus non-reactive distillation

1. Reactive Distillation Alone: In this case, throughout the whole column, reaction and distillation take place simultaneously. At one of the ends, purified product is obtained. This is possible only for the systems for which pure product point lies within reaction space.

2. Reactive Distillation plus Non-reactive Distillation: There are many cases for which pure product point does not lie within reaction space. For such cases, non-reactive distillation section (either rectifying or stripping or both) is also required to achieve pure product and pure reactants.

3. Reaction alone plus Reactive Distillation Plus Non-reactive Distillation (Optional): If the reaction space required is considerably greater than distillation space, then this method is applied.

Sometimes it is not recommended to shift entire reaction space to reactive distillation unit. The reason may be one of the following:

(a) If the optimum reaction conditions and optimum distillation conditions are different, then it is better to improve only equilibrium conversion in reactive distillation, while major part of reaction (up to equilibrium conversion) can be carried out separately in another reactor.

(b) If the existing plant of conventional technology have more number of reactors in series than one may shift only last or a few of the last reactors to reactive distillation unit.

8.7.4 Various Contact Devices Used for Catalytic Distillation

In catalytic distillation, contact devices are required to improve mass transfer rate and to improve catalyst contact. Various types of contact devices used for catalytic distillation are as follows:

1. Tray column in which bed of catalyst is placed either in downcomers or above the trays.
2. Packed tower using random packing elements which contain the catalyst (with special coating) or which may be made of the catalyst.
3. Packed tower using ordered packing elements which contain catalyst particles. In orderly packing element, catalyst particles are sandwiched between two supporting materials. Two types of ordered pickings are used:

(a) Bale packing

(b) Structured packing

(a) Bale Packing: In bale packing (Fig. 8.46), catalyst particles are enclosed within several pockets in a fibre glass cloth belt.

The pockets are normally 2.5 to 5 cm wide with 0.625 to 1.25 cm web between them. Open ends of pockets are sewn closed. Belts are rolled up with alternating layers of stainless steel mesh which increases structural strength of cloth belt.

Bales constructed for commercial use are roughly cylindrical shape with 20 to 35 cm diameter and 50 cm height. Several bales then packed tightly into a column such that pockets remain vertical. Flow channels facilitate the contact between vapour and liquid. Ideally, contact between liquid reactants and catalyst is taking place in pockets and vapour-liquid contact takes place in flow channel. HETP as well as pressure drop relations for bale packings could be obtained from the supplier.

(b) Structured packing: KATMAX structural packing is developed by Koch Engineering Company, USA. From outside this packing looks like a brick with corrugated surface. In such type of packing, solid catalyst is held in a screen envelope.

Fig. 8.46 *Bale Packing*

The catalyst is sandwiched between the layers of screen. It is easily accessed by the liquid. Between two layers of catalyst (surrounded by screen), gaps are provided, known as flow channels which facilitate the flow of vapour. Inside the flow channel vapour makes the contact with liquid film.

8.7.5 Industrial Applications of Reactive Distillation

1. Manufacturing of ethyl acetate from ethanol
2. Manufacturing of methyl acetate from methanol
3. Manufacturing of butyl acetate from butanol
4. Manufacturing of MTBE (methyl tertiary butyl ether) and ETBE (ethyl tertiary butyl ether)
5. Selective hydrogenation of butadine of C_4 stream
6. Selective hydrogenation of pentadine of C_5 stream
7. Selective hydrogenation of hexadine of C_6 stream
8. Manufacturing of benzyl chloride from toluene
9. In benzene separation of gasoline

10. Manufacture of ethyl propionate from ethanol
11. Manufacture of amino resins (refer to Exercise 8.16)
12. Manufacture of monochloro acetic acid

Among these applications, manufacturing of ETBE by catalytic distillation is discussed in this chapter.

8.7.6 Manufacturing of ETBE

Ethyl tertiary butyl ether is used as octane number improver like MTBE.

Main relation

$$\underset{\textit{i}\text{–Butylene}}{(CH_3)_2C = CH_2} + \underset{\text{Ethanol}}{C_2H_5OH} \underset{9\ \text{atm}}{\overset{60 - 90°C}{\rightleftharpoons}} \underset{\text{ETBE}}{(CH_3)_3COC_2H_5}$$

Side reactions

(1) $$\underset{\textit{i}\text{-Butylene}}{(CH_3)_2C = CH_2} + \underset{\textit{i}\text{-Butylene}}{(CH_3)_2C = CH_2} \rightleftharpoons \underset{\text{Diisobutylene}}{(CH_3)_2C = (CH_2)_2}$$

(2) $$\underset{\textit{i}\text{–Butylene}}{(CH_3)_2C = CH_2} + \underset{\text{Water}}{H_2O} \rightleftharpoons \underset{\textit{i}\text{–Butyl alcohol}}{(CH_3)_3COH}$$

Conventional Process Refer to Fig. 8.47. Isobutylene rich stream is water washed in wet scrubber to remove trace contaminants that can deactivate the catalyst. Then this stream is mixed with rectified spirit and mixture is sent to isothermal, shell and tube type plug flow reactor. Ethanol is used as excess reactant. Second reactor is operated adiabatically. Product from second reactor is sent to distillation column. In first distillation column, the mixture of ethanol, water and isobutylene is separated from ETBE. Ethanol cannot be separated from the distillate of first column by common distillation because ethanol forms azeotrope with isobutylene as well as with water.

Further addition of water as solvent separates ethanol and water mixture from isobutylene in liquid-liquid extractor. Isobutylene rich stream is coming out from the top of extractor as light product or as raffinate. Ethanol-water stream or extract stream leaves from the bottom of the extractor. From the extract phase, azeotrope (95.6% ethanol and 4.4% water by mass) is separated by distillation and it is recycled back to the reactor.

Bottom product of first distillation column is ETBE-ethanol mixture from which ethanol is separated from ETBE by distillation.

Reactive Distillation Process[36] In reactive (catalytic) distillation technology (Fig. 8.48) only equilibrium conversion is increased in reactive distillation unit. Up to 80% conversion of isobutylene is achieved in a (conventional) separate isothermal shell and tube heat exchanger-type plug flow reactor. Hence, feed to reactive distillation column is rich in ETBE but still contains ethanol and isobutylene. Instead of rectified spirit, absolute alcohol is used as raw material with isobutylene.

Overhead product from reactive distillation column is non-reactive hydrocarbons (*n*-butane, *n*-butylene, etc.), isobutylene and ethanol, respectively.

Fig. 8.47 *Conventional Process of ETBE*

Fig. 8.48 *Reactive Distillation Process of ETBE*[36]

Reactive Distillation column has total ten packing sections. First and second packing sections use simple structured packing, act as rectification or enriching section. Third, fourth and fifth packing sections use orderly packing and act as reactive distillation zones. Sixth to tenth packing sections used simple structural packing which act as stripping section.

Typical data of this process are as follows.

1. Feed composition: 29.1% ETBE, 9.1% ethanol,
   73% isobutylene, 54.5% *n*–butylene (by mole)
2. Isobutylene conversion: 97.4%
3. Operating pressure = 900 kPa a
4. Reflux ratio = 5.0
5. Bottom composition = 96.5% ETBE, 2.8% ethanol
   0.7% butylene, 0.04% diisobutylene (by mole)
6. Distillate composition = 96.5% *n*-butylene, 1.7% isobutylene,
   0.7% ethanol (by mole)

8.7.7 Effect of Various Parameters on Reactive Distillation of ETBE

1. Effect of Feed Composition: Increase in isobutylene percentage in feed (isobutylene rich stream) results in

(a) Decrease in energy cost per kg of ETBE produced. As lesser energy is "wasted" in heating and cooling of inert compounds.

(b) Decrease in size and fixed cost of reactive distillation unit and associated equipments for the same production rate of ETBE.

(c) Increase in the reactant concentrations in reaction zone, increases reaction rate as well as equilibrium conversion.

$$f(X_e) = K \propto \left(\frac{p_t}{n_t}\right)^{\Delta n} \tag{8.124}$$

In this reaction, Δn = change in moles during reaction = 2 – 1 = 1. Hence, decrease in total number of moles of reaction system increases the value of equilibrium conversion X_e.

(d) Increase the temperature of reaction zone due to decrease in stabilising effect of inert components. Reaction is exothermic.

2. Effects of Excess Ethanol: Excess of ethanol is required to suppress rate of side reactions and to increase yield of ETBE. But, increase in excess of ethanol decreases the purity of ETBE as a bottom product of reactive distillation. It has been found that 4 to 7% excess ethanol is sufficient to produce high yield of ETBE without affecting the purity of ETBE as a bottom product from the reactive distillation unit.

3. Effect of Column Pressure: In conventional distillation, operating pressure is decided based on use of cooling water or cheaper cooling medium in overhead condenser. Sometimes, to avoid the thermal cracking of product or to alter the vapour-liquid equilibrium or to facilitate the use of cheaper heating medium, etc., conventional distillation is carried out in vacuum.

However, in reactive distillation, choice of operating pressure is more complicated. In reactive distillation pressure affects equilibrium conversion of reaction also.

In ETBE, synthesis moles are decreasing during the reaction, hence higher pressure is favourable as it increases equilibrium conversion. But, this is not always true, as increase in pressure increases temperature of reaction zone of reactive distillation unit. Since ETBE synthesis is exothermic reaction, increase in temperature decreases equilibrium conversion. It is found that best conversion is achieved at 400 to 500 kPa a. But considering distillation aspect, operating pressure is kept 700 to 900 kPa a.

4. Effect of Reactive Distillation Stages: Increase in length of reactor zone or increase in number of stages of reactive distillation zone improves the conversion, but at the same time decomposition of product starts in the lower stages, if the number of stages increased above the optimum number of stages.

5. Effect of Separation Stages: Ideally the rectification or enriching section of reactive distillation column for ETBE synthesis should (a) remove light inerts from reaction zone, (b) prevent loss of ether or alcohol in distillate, and (c) recycle unreacted reactants to the reaction zone. In actual practice, this is almost impossible to achieve as favourable conditions for the reaction cannot be changed. However, the loss of ETBE in distillate can be minimised without rejecting isobutylene from the column. More enriching stages are required to prevent loss of ethanol with distillate.

Ideally, stripping section should perform the following functions:

(a) Remove ETBE from the reaction zone to maintain favourable reaction conditions.
(b) Purify the ether (ETBE) product.
(c) Prevent loss of reactive isobutylene with ether (ETBE) product.
(d) Minimise ethanol loss with the ether product.

These functions are easily achievable at temperature and pressure of reaction conditions.

6. Effect of Reflux Ratio: Effect of increasing reflux ratio are as follows:

(a) The concentration of reactants in the distillate is reduced.
(b) The reaction zone temperature reduces which is favourable for exothermic reaction to increase equilibrium conversion.
(c) The concentration of ether in the reaction zone reduces. It reduces catalytic cracking of ether (ETBE).

All these three effects increase the conversion. However, higher reflux ratio increases tower diameter, condenser and reboiler sizes and energy requirements.

7. Effects of Feed Point: In ETBE synthesis, optimum feed point for reactive distillation column is just below the reactive distillation zone. In reaction zone, ETBE can decompose or it can react by cracking reaction in presence of catalyst. So to avoid the decomposition of ETBE in the feed (i.e., ETBE rich stream), it is fed just at the bottom of reaction zone. Feeding too far below the reaction zone reduces the stripping potential and increases energy requirement for the same height of stripping section.

8. Effects of Reboiler Duty: At some optimum value of reboiler duty, higher conversion (more than 95%) and higher purity (~90%) is achieved. If reboiler duty is too high or too low, conversion and product purity both are reduced.

Manufacture of ETBE was discussed as a case study of reactive distillation so that a process engineer can learn to analyse a system before actual process design is taken up.

8.7.8 Manufacture of Ethyl Propionate

Ethyl propionate can be synthesised by esterification of ethyl alcohol with propionic acid in a reaction distillation unit using cation exchange resin as catalyst. Bhanvase *et al*[37] have given pilot plant results for scale up and developing a process flow sheet for commercial scale plant.

8.8 AZEOTROPES AND SEPARATION THEREOF

In the previous sections, distillation of liquid mixtures was dealt which are amicable to ordinary fractional distillation. In these cases, VLE plot or an equilibrium curve of a binary is on one (upper) side of the diagonal line. However, when the equilibrium curve crosses the diagonal line, an azeotrope occurs. Thus at a given pressure, a mixture of specific composition boils at a constant temperature which produces vapour of the same composition. When this happens, the mixture cannot be separated and it is known as azeotropic mixture. When the vapour of the azeotropic mixture is condensed, liquid mixture so obtained decides the type of azeotrope. If the condensed liquid phase is homogeneous, it is called a *homogeneous azeotrope*. Ethanol-water forms homogeneous azeotrope at 101.325 kPa and 78.1°C having 95.6% ethanol (by mass). Figures 8.49 (a) and (b) and Figs. 8.50(a) and (b) refer to homogeneous azeotropes.

A heterogeneous azeotrope is one for which the overall liquid mixture forms two liquid phases. Mixture of *n*-butanol–water forms an azeotrope at 101.325 kPa and 92.2°C having 57.5% (by mass) or 24.8% (by mole) *n*-butanol in the overall liquid mixture. It separates in two layers. At 40°C, top layer contains 78.51% *n*-butanol while the bottom layer contains 6.60% *n*-butanol (by mass). Refluxing a specific layer provides a method to separate the components of the mixture. Figures 8.51 (a) and (b) refer to a heterogeneous azeotrope.

The temperature at which an azeotrope is distilled is important. A minimum boiling azeotrope is one for which the boiling temperature is less than that of pure components. Figures 8.49(a) and (b) and Figs. 8.51(a) and (b) are *t–x–y* and *y–x* diagrams of minimum boiling azeotropes. For example, at 101.325 kPa, *n*-butanol and water boil at 117.7°C and 100°C, respectively but their azeotrope boils at 92.2°C. In case of maximum boiling azeotrope, the boiling point of the azeotrope is higher than that of the pure component. Chloroform and acetone boil at 61.2°C and 56.4°C, respectively at 101.325 kPa. The mixture of these two compounds forms a maximum boiling azeotrope at 64.5°C, containing 65.5% (by mole) chloroform. Figures 8.50(a) and (b) are *t-x-y* and *y-x* diagrams of the system, comprising chloroform and acetone. Maximum boiling azeotropes are usually homogeneous. About 90% of the known azeotropes are of the minimum variety. Heterogeneous azeotropes are minimum boiling azeotropes. However, many homogeneous azeotropes are also minimum boiling azeotropes. Minimum boiling azeotropes have positive deviation from ideality. In other words, their partial pressures in vapour phases are greater than

the same given by Raoult's law. Maximum boiling azeotropes have negative deviation from ideality.

(a) *t-x-y* Diagram for Toluene-*n*-Butanol System

(b) *x-y* Diagram for Toluene-*n*-Butanol System

Fig. 8.49 *Homogeneous Minimum Boiling Azeotrope of Toluene-n-Butanol System*[38] *(Reproduced with the permission of Chemical Engineering by Access Intelligence, USA)*

Azeotropic data are well tabulated in literature. *Perry's Chemical Engineers' Handbook*[8] tabulate data for binary and ternary azeotropes of common interest. Extensive 3-volume compilation by J. Gmehling (Ref. 9) cover pratically all known azeotropes.

Fig. 8.50 *Homogeneous Maximum Boiling Azeotrope of Chloroform-Acetone System*[38] *(Reproduced with the permission of Chemical Engineering by Access Intelligence, USA)*

Property of some organic compounds to form azeotropes with water is advantageously used in many chemical reactions. Manufacture of butylated amino resins involve etherification reactions. Water is a product of reaction. One of the reactants is *n*–butanol which forms a heterogeneous azeotrope with water. While a batch of reaction is in progress, water is removed from the mass as an azeotrope.

The vapour of azeotropic composition is condensed which separates into two liquid layers. Top *n*-butanol rich layer is recycled to the reactor and bottom aqueous layer is removed. This way one of the products of reaction is removed from the reaction mass, thereby reaction proceeds in forward direction.

(a) *t-x-y* Diagram for *n*-Butanol-Water System

(b) *x-y* Diagram for *n*-Butanol-Water System

Fig. 8.51 *Heterogeneous Minimum Boiling Azeotrope of n-Butanol-Water System*[38]
(Reproduced with the permission of Chemical Engineering by Access Intelligence, USA)

In manufacture of alkyd resins also, water is a product of reaction. Toluene or xylene is added to the reaction mass and water is removed from the mass in form of a heterogeneous azeotrope. On condensation, two layers separate. Water is sparingly soluble in the hydrocarbon. Top hydrocarbon layer is recycled to the reactor.

In all esterification reactions, water is a byproduct. It is a common practice to remove water in the form of an azeotrope from top in a specially designed distillation column.

Removal of water in the form of an azeotrope has also expedited other operations, such as nitration of benzene and toluene. In this case, water formed in the nitration of the hydrocarbon is removed continuously, utilising hydrocarbon itself as an entrainer. Contrary to semi-continuous operation, mixed acid, consisting of nitric acid and sulphuric acid, is used for nitration in a batch reactor. This batch process suffers from a drawback of disposal of spent acid or recovery of acids from the spent acid is a problem. Bottom still acts as a reaction vessel in which nitric acid and excess hydrocarbon are initially charged. Nitrated hydrocarbon has higher boiling point and thus remains in the still. Azeotrope of hydrocarbon and water goes to the bottom of the dehydrating column. Optimum temperatures and concentrations can readily be maintained in the system with simple controls. Parameters, normally required to be controlled are feed rates of nitric acid and hydrocarbon, rate of supply of heat to respective heating units and rate of withdrawal of water at the top of the dehydrating column. Product is thus formed in single pass with almost 100% yield on both; nitric acid and hydrocarbon.

Similar azeotropic process is continuous production of acetamide. Continuous streams of ammonia and acetic acid are fed to a dehydrating column. Ammonium acetate, so formed is continuously dehydrated in azeotropic distillation system to give water and acetamide. Water, formed in both the reactions, goes overhead with the help of an entrainer. Acetamide with excess acetic acid flow out from the bottom of the column. This mixture is sent to second distillation column from which pure acetamide is withdrawn from the bottom. Acetic acid vapour from the column is recycled back to reaction-cum-azeotropic distillation column.

In general, following are the methods, used for separating homogeneous azeotropic mixtures:

1. As mentioned earlier, azeotrope is dependent on the operating pressure in most cases. If pressure is altered, it may be possible to avoid formation of the azeotrope thereby fractional distillation can be carried out. Table 8.50 gives azeotropic compositions of ethanol-water mixtures at different operating pressures.

Table 8.50 Effect of Pressures on Ethanol-Water Azeotropes[39]

Absolute Pressure		Concentration of Ethanol		Temperature °C
Torr	kPa	mole fraction	mass fraction	
80	10.666	1.000	1.000	30.30
95	12.666	0.987	0.995	33.35
130	17.332	0.967	0.987	39.30
198	26.398	0.934	0.973	47.63
300	39.997	0.925	0.969	56.50

(*Contd.*)

Table 8.50 (*Contd.*)

Absolute Pressure		Concentration of Ethanol		Temperature °C
Torr	kPa	mole fraction	mass fraction	
405	53.996	0.909	0.962	63.04
760	101.325	0.895	0.956	78.15
1075	143.322	0.889	0.953	87.10
1451	193.451	0.887	0.953	96.40
2585	344.638	0.882	0.950	112.60
5171	689.410	0.874	0.947	136.70

It can be seen from Table 8.50 that at pressure lower than 80 Torr, there does not exists an azeotrope. Similarly, for methanol-benzene mixture, mole fractions of methanol in azeotropic mixture at 200 Torr and at 760 Torr absolute pressures are 0.557 and 0.613, respectively.

A few azeotropes, however, do not change composition with the pressure. For example, *n*-hexane forms an azeotrope with ethanol[38], containing 21% ethanol (by mass) which boils at 58.7°C at 101.325 kPa. However, the composition of this azeotropic mixture is unchanged over the pressure range of 20 Torr to 10 000 Torr.

2. A popular method is to add specially chosen chemical to shift the azeotropic point to a more favourable position. When this chemical (also known as entrainer) appears in appreciable amounts at the top of the column, the operation is called *azeotropic distillation*. When the additional component (called solvent) appears mostly at the bottom of the column, the operation is called *extractive distillation*.
3. In distillation using ionic salt, the salt dissociates in the liquid mixture and alters the relative volatilities sufficiently that the separation becomes possible like fractional distillation.
4. Pressure-swing distillation is yet another method where a series of columns, operating at different pressures, are used to separate binary azeotropes which change appreciably in composition over a moderate pressure range or where a separating agent which forms a pressure-sensitive azeotrope is added.
5. Use other techniques like pervaporation or adsorption for separating a component (such as water) from the azeotrope. These techniques are discussed in Sec. 8.9.3.
6. Reactive distillation can be considered where the separating agent reacts preferentially and reversibly with one of the azeotropic constituents. The reaction product is then distilled from the non-reacting components. Dilute alcohol solutions or dilute aqueous acetic acid solution can be treated by this method to form esters in reactive distillation units.
7. Fixed adsorptive distillation method is yet another technique, found useful in separation of an azeotropic mixture. Mujiburohman *et al*[40] have developed a system consisting of two distillation columns with an interbed filled with an adsorbent, is installed. In this way the azeotropic point is avoided and purer products are obtained from two columns.

8.8.1 Azeotropic Distillation

Heterogeneous azeotropic distillation is a widely practised process for dehydration of number of compounds such as acetic acid, chloroform, ethanol, acetonitrile, etc. The technique involves addition of a third component in a homogeneous azeotrope, called an entrainer to form a minimum boiling heteroazeotrope with at least one of the components of the feed carrying water overhead and leaves near dry (pure) product in the bottom. The overhead vapours are condensed to two liquid phases; entrainer rich phase is refluxed while the aqueous phase is decanted.

Dehydration of ethanol-water mixture is carried out with the help of benzene as an entrainer. Ethanol and water forms azeotrope having 95.6% ethanol (by mass) at 78.15°C and 101.325 kPa. When benzene is added to the azeotrope (about 0.427 kg/kg azeotrope) it comes as ternary heterogeneous azeotrope at the top containing 74.1% benzene and 18.5% ethanol (by mass). Bottom product from the column is pure dry ethanol. Top azeotrope at 65°C is condensed which forms two layers. Top layer contains 90.9% benzene by mass (and practically no water). It is refluxed to the column. Bottom aqueous layer contains 53% ethanol and 11% benzene (by mass). Since this layer contains appreciable content of benzene, it can be distilled in another column to recover it in the form of ternary azeotrope. Water is removed from the bottom of the third column. Thus three columns are normally required for this distillation.

Process design of an azeotropic distillation system for separation of ethanol-water using trichloroethylene is given by Coates[38] and using benzene is given by Norman[41].

Another entrainer for ehtanol dehydration is *n*-pentane which is eco-friendly. The tower is operated at near 3.45 bar a. Total condenser operates at 3 bar a and a ternary azeotrope, containing 82.64% *n*-pentane and 12.33% ethanol (by mole) is formed. Condensate separates in two layers, top layer contains 87.9% *n*-pentane and 11.6% ethanol (by mole) and is refluxed to the column. Bottom aqueous layer contains 23.8% ethanol (by mole) and practically no *n*-pentane. This layer is distilled in second column to recover ethanol as azeotrope with water as top product at atmospheric pressure. In this case only two columns are required.

Another example of separation of homogeneous azeotrope is that of *iso*-propanol–water. It forms azeotrope at 80.4°C and 101.325 kPa. For their separation, cyclohexane or benzene is used as entrainer. Cho and Jeon[42] have given process design of heterogeneous azeotropic distillation system for dehydration of *iso*-propyl alcohol using benzene as the entrainer.

When the binary azeotrope is hetergeneous, separation of the components is possible without addition of an entrainer. Design of *n*-butanol–water distillation system is given by McKetta and Cunningham[43] in detail.

8.8.2 Extractive Distillation

Extractive distillation can be defined as distillation in the presence of a miscible, high boiling relatively non-volatile component (called the solvent) that forms no azeotropes with other components of the mixture. Solvent is continuously added near the top of the column so that an appreciable amount is present in the liquid phase throughout the column. The component having the greater volatility (not necessarily the lowest boiling point component) is taken overhead as a relatively pure distillate. The other component

leaves with the solvent via the column bottoms. The solvent is then separated from the bottoms in second distillation column. Selection of an extractive distillation solvent is the most important step in developing a successful extractive distillation sequence. Important selection criteria are given below.

1. It should enhance the relative volatility of the key component significantly.
2. Required solvent quantity to be added to the azeotropic mixture should not be excessive.
3. It should be soluble in the feed components.
4. It should be easily separable from the bottom product.
5. It should be relatively inexpensive and easily available.
6. It should be stable at the operating conditions of both the columns.
7. It should be non-reactive with the feed components.
8. It should have low latent heat of vaporisation.
9. It should be non-corrosive and non-toxic.
10. It should not form immiscible liquid mixtures at any point in the column.

Perrys' Chemical Engineers' Handbook[8] serves as a good reference for the solvent selection.

Cyclohexane is manufactured from benzene. The mixture from the reactor comes out with the desired product (cyclohexane) and also a significant amount of unreacted benzene which is to be recycled back to the reactor for economic reasons. Product mixture contains nearly 45% cyclohexane and balance benzene (by mole). It is desired to operate the distillation column at 150 kPa a as dictated by the process flow sheet. At this pressure, cyclohexane and benzene boil at 94.3°C and 93.5°C, respectively. Binary homogeneous azeotrope at this pressure is formed at 91°C, containing 45.5% cyclohexane (by mole) which is nearly the same composition of reactor exit product mix. In this case propylene glycol is selected as a solvent as benzene is highly soluble in the solvent. Further the solvent has high boiling point (200.4°C) at 150 kPa a and no new azeotropes are formed. Distillate from the column is 99.3% pure cyclohexane. Bottoms from the first column is fed to second column from which distillate is 85% pure benzene which is recycled back to the reactor. Bottoms from the second column is nearly pure solvent which is recycled to the first column.

Apart from organic solvents, inorganic salts such as potassium and sodium acetates, calcium chloride, calcium nitrate, caustic soda, etc., can also be used to break the azeotropes.

Another well-known example of extractive distillation is dehydration of nitric acid-water azeotrope (containing 64.1% HNO_3 by mass at 101.325 kPa a and 122°C). In this case sulphuric acid is used as solvent. Refer to Example 8.21.

Apart from above examples, extractive distillation finds many applications in the petroleum refinery. Pyrolysis gasoline byproduct from naphtha steam cracking contains a wealth of aromatics. Separation of aromatics, such as xylenes, is carried out by extractive distillation. Similarly, extractive distillation can be used to selectively extract the sulphur species from cracked gasoline to very low levels without losing its octane value.

Advantages of extractive distillation over azeotropic distillation are:

1. Choice of third component to be added is greater as selection of third component does not depend upon an incident of azeotrope formation.

2. Energy requirement is less as the third component or solvent is not completely vaporised in principle column or in auxiliary column. Only small fraction of solvent is vaporized in both columns as the same is obtained as bottom products. While entire entrainer is to be vaporized in azeotropic distillation in both columns as the same is coming out as one of the component of overhead vapour from both the columns. However, this advantage of extractive distillation is lost if volatilised component or the component to be taken out as top product along with entrainer which is the minor component of feed. Hence, in dehydration of ethanol from the azeotropic mixture (containing 4.4% by mass water), energy consumption of azeotropic distillation using *n*-pentane as entrainer is less as compared to the same by extractive distillation using ethylene glycol as solvent.

8.8.3 Pressure Swing Distillation

Although known from good old days, only now pressure swing distillation technique is gaining popularity for separation of a binary azeotrope over heterogeneous azeotropic distillation and extractive distillation. The chief reason for not finding many industrial applications till date was the lack of experimental VLE data for binary systems at different pressures. Two main advantages of pressure swing distillation are; energy saving by heat integration of the system and no need to add an entrainer or a solvent for the separation.

The technique is based on the principle that the composition of almost all azeotropes varies with the operating pressure. In industrially important water-solvent mixtures, the water content of the azeotrope increases with increasing pressure. This variation provides a technique for separation of the components. A shift in composition of azeotropic mixture with respect to pressure can be well understood by studying Figs. 8.52(a) and (b).

Acetonitrile—ACN (also called methyl cyanide; formula: C_2H_3N) forms a minimum boiling azeotrope with water at 1 atm and 77°C containing 83.5% by mass or 69.0% by mole ACN. This azeotrope composition shifts to 60.0% by mole ACN at 3.02 bar a which boils at 112.5°C. Using these data a two-column system can be designed to operate at two different pressures to separate ACN and water from the mixture. Feed with lower concentration than 69.0 mole % ACN can be fed to the first column operating at 1 atm. Distillate from the column with near azeotropic composition is the feed to the second column operating at 3.02 bar a. Bottom products from the first and second column are water and ACN, respectively. Distillate from the second column is recycled to the first column along with the fresh feed.

An important point to note is that in heterogeneous azeotropic distillation, bottom product from the first column is the solvent (low boiling component) while in pressure swing distillation, bottom product is water (high boiling component).

In pressure swing distillation since both the columns operate at different pressures, their heat exchangers can be readily thermally integrated. This will result in substantial savings in energy for distillation. However, such an integration results in complexity of the system which requires adequate process control and instrumentation. Also hazards of handling flammable (and toxic) solvents at high pressure must be given due considerations while designing such a system.

Fig. 8.52 *Homogeneous Minimum Boiling Azeotropes Acetonitrile-Water System*[44] *(Reproduced with the Permission of Dr. Repke)*

Both batch and continuous systems are developed. Jen-Uwe Repke *et al.*[45,46] have developed mathematical models and process control systems for ACN-water mixture.

Knapp and Doherty[47] have listed more than 35 pressure sensitive binaries which can be separated by pressure swing distillation technique.

Ethanol-water is also a pressure sensitive azeotrope (Ref. Table 8.50) but its separation by pressure swing distillation method is considered an uneconomical proposition. For separation of this azeotrope, Knapp and Doherty[47, 48] have suggested to add small amount of acetone in the dilute alcohol stream from a fermentor. The entrainer does not form a ternary azeotrope at atmospheric pressure but forms a ternary azeotrope at high pressure (say at 10 atm). On this basis a scheme is presented to operate first column at 1 atm and second column at 10 atm. Resulting pressure sequence yields dry ethanol from the bottom of the second (HP) column. Also thermal integration of both the columns result in nearly 53% energy saving over the conventional heterogeneous azeotropic distillation sequence.

Exercise 8.15 is yet another example of separation of tetrahydrofuran (THF) from aqueous THF solution using pressure swing distillation technique.

Example 8.18 Isopropyl alcohol (IPA) forms a minimum boiling azeotrope with water at 80.4°C and 101.325 kPa, having a composition of 31.47 mole % water. Vapour pressure of IPA can be calculated using Antoine equation[14].

$$\log p_v = 6.8651 - \frac{1359.50}{(T - 75.65)}$$

where, p_v = Vapour pressure, kPa

T = Saturation temperature, K

(a) A feed consisting of 50 mass % IPA under saturated liquid condition is to be distilled to give 67.5 mole % IPA as distillate (near azeotrope) and 0.1 mole % (0.0033 mass %) IPA in the bottoms. Using McCabe–Thiele technique and for $R = 3$, find the number of theoretical stages, required for the separation at 1 atmosphere and locate the feed introduction stage.

(b) Make tray-to-tray calculations for the desired separation using van Laar constants.

Solution

(a) Azeotrope contains 0.3147 mole fraction (0.121 mass fraction) water. To find the non-ideal behaviour of the IPA-water system, use of van Laar equation can be made. Assuming vapour fugacity (i.e., vapour escaping tendency) to be unity at low pressure.

Modified Raoult's law:

$$p.y_i = x_i \gamma_i p_{vi} \tag{8.3}$$

where, p = Total pressure of the system = 1 atm = 760 Torr

y_i = Mole fraction of ith component in vapour

x_i = Mole fraction of ith component in liquid

γ_i = Activity coefficient of ith component

p_{vi} = Vapour pressure of the ith component at the boiling point, Torr

If x_1 and x_2 are mole fractions of the two components in a binary liquid mixture,

$$\ln \gamma_1 = A \left[\frac{x_2}{\left(\frac{A}{B}\right) x_1 + x_2} \right]^2 \tag{8.125}$$

$$\ln \gamma_2 = B\left[\frac{x_1}{\left(\frac{B}{A}\right)x_2 + x_1}\right]^2 \tag{8.126}$$

Equations (8.125) and (8.126) are called *van Laar equations* and A and B are called *van Laar constants*. Let 1 refer to IPA, the more volatile component and, 2 refer to water.

Boiling point of the azeotrope, $t = 80.4°C$ or $T = 353.55$ K.

$$\log p_{v1} = 6.8651 - \frac{1359.50}{(353.55 - 75.65)} = 1.\ 973\ 05$$

$$p_{v1} = 93.984 \text{ kPa (vapour pressure of IPA at 80.4°C)} \equiv 704.94 \text{ Torr}$$

From steam tables, at $t = 80.4°C$, $p_{v2} = 48.14$ kPa = 361.08 Torr

At the azeotropic point,

$$\gamma_1 = 760/704.94 = 1.0781$$

$$\gamma_2 = 760/361.08 = 2.1048$$

$$\ln (1.0781) = A\left[\frac{0.3147}{\left(\frac{A}{B}\right)0.6853 + 0.3147}\right]^2$$

$$\ln (2.1048) = B\left[\frac{0.6853}{\left(\frac{B}{A}\right)0.3147 + 0.6853}\right]^2$$

Solving the equations by Mathcad,

$$A = 2.311\ 87 \text{ and } B = 1.107\ 77$$

These constants are themselves functions of the liquid compositions and temperature but their variations can be ignored for all practical purposes (for low heats of mixing) at a given total pressure and varying temperature conditions. Using these constants, van Laar equations can be used to develop the vapour-liquid equilibrium curve.

Consider $x_1 = 0.6$ (of IPA) and $x_2 = 0.4$ (of water) and calculate activity coefficients.

$$\ln \gamma_1 = 0.135\ 51 \text{ or } \gamma_1 = 1.145\ 12$$

$$\ln \gamma_2 = 0.636\ 31 \text{ or } \gamma_2 = 1.889\ 49$$

Assume boiling point of the mixture to be 81°C (or 354.15 K),

$$p_{v1} = 96.292 \text{ kPa} = 722.25 \text{ Torr}$$

$$p_{v2} = 49.311 \text{ kPa} = 369.86 \text{ Torr}$$

$$p = \Sigma x_i \cdot \gamma_i \cdot p_{vi}$$

$$= (0.6 \times 722.25 \times 1.145\ 12) + (0.4 \times 369.86 \times 1.889\ 49)$$

$$= 496.24 + 279.54 = 775.78 \text{ Torr} \neq 760 \text{ Torr}$$

Since total pressure does not equal to 760 Torr, assume new temperature equal to 80.5°C (353.65 K).

$$p_{v1} = 94.365 \text{ kPa} = 707.8 \text{ Torr}$$
$$p_{v2} = 48.336 \text{ kPa} = 362.55 \text{ Torr}$$
$$p = (0.6 \times 707.8 \times 1.145\ 12) + (0.4 \times 362.55 \times 1.889\ 49)$$
$$= 486.31 + 274.0 = 760.31$$
$$\simeq 760 \text{ Torr}$$

Mole fraction of IPA in vapour (corresponding to x_1), $y_1 = x_1 \cdot \gamma_1 \cdot p_{v1}$
$$= 0.6 \times 707.8 \times 1.145\ 12/760$$
$$= 0.64$$

In this manner, *x*-*y* data can be generated for the entire range which is tabulated in Table 8.51.

Table 8.51 Vapour-Liquid Equilibrium of IPA-Water at 1 atm (Based on van Laar Constants)

Mole fraction of IPA		Boiling point (*t*), °C
in Liquid, *x*	in Vapour, *y*	
0	0	100.0
0.02	0.2495	92.65
0.05	0.3993	87.4
0.075	0.4561	85.15
0.1	0.489	83.85
0.2	0.5371	82.0
0.3	0.5545	81.5
0.4	0.573	81.3
0.5	0.6	80.8
0.6	0.64	80.5
0.6853	0.6853	80.4 (azeotrope)
0.8	0.7682	80.6
0.9	0.87	81.15
0.95	0.93	81.65
1.0	1.0	82.5

Figure 8.53 is the plot of the vapour-liquid equilibrium of IPA-water system.

Feed (z_F) is marked at $x_1 = 0.2306$ (equivalent to 0.5 mass fraction) of IPA. Desired purities of distillate and bottoms are marked at $x_D = 0.675$ and $x_W = 0.001$. *q*-line is vertical as the feed is saturated liquid.

$$\frac{x_D}{(R_m + 1)} = 0.3537$$
$$R_m = 0.9084$$

In case of azeotropic distillation, other methods are available in literature[41] for calculation of minimum reflux ratio which are more reliable. Also actual reflux ratio is kept much higher than the minimum.

Fig. 8.53 *Azeotropic Distillation of Aqueous Isopropyl Alcohol*

Let $R = 3$ (3.3 times R_m)

$$\frac{x_D}{(R+1)} = \frac{0.675}{(3+1)} = 0.1688$$

For $R = 3$, from Fig. 8.53, total 10 theoretical stages are required for the separation and feed will be introduced at third stage from bottom.

(b) Tray-to tray Calculations:

Azeotropic distillation column design of IPA-water system can be carried out by numerical method, i.e., tray-to-tray calculations. Average latent heats of vaporisation of IPA and water are 40 093 and 41 081 kJ/kmol, respectively. Also column is assumed to operate at atmospheric pressure from top to bottom. Activity coefficients for each tray liquid mixture will be calculated using same van Laar constants, used for developing Table 8.51.

Basis: 100 kmol/h of feed consisting of 0.2306 mole fraction IPA.

With distillate, containing 0.675 mole fraction IPA and bottoms, containing 0.001 mole fraction IPA, material balance yields

$D = 34.065$ kmol/h and

$W = 65.935$ kmol/h

Reflux $R = L = 3D = 3 \times 65.935 = 102.195$ kmol/h

Calculations relate to stripping section.

Liquid flow in column $\bar{L} = L + F \cdot q = L + F$ $(q = 1)$

$= 102.195 + 100$

$= 202.195$ kmol/h

Vapour from the bottommost tray (No. 1),

$\bar{V}_1 = \bar{L} - W$

$= 202.195 - 65.935$

$= 136.26$ kmol/h

For $x_1 = 0.001$ (IPA) and $x_2 = 0.999$ (water)

$\gamma_2 = 9.996\,57$ and $\gamma_2 = 1.000\,00$

At $t_1 = 99.46°C$, $y_1 = 0.0191$ and $y_2 = 0.9809$ (Refer Table 8.51)

Latent heat of vapour leaving Tray-1

$\lambda_1 = 0.0191 \times 40\,093 + 0.9809 \times 41\,081$

$= 41\,062.3$ kJ/kmol

Table 8.52 Material Balance of Tray-1 (from Bottom)

Component	Bottom product, W kmol/h	Vapour, $\bar{V}_1$		Liquid flow from Tray-2, $\bar{L}_2$	
		mole fr.	kmol/h	kmol/h	mole fr.
IPA	0.066	0.0191	2.6	2.666	0.0132
Water	65.869	0.9809	133.66	199.529	0.9868
Total	65.965	1.0000	136.26	202.195	1.0000

With $x_1 = 0.0132$ and $x_2 = 0.9868$

$\gamma_1 = 8.917\,39$ and $\gamma_2 = 1.000\,82$

At $t_2 = 94.83°C$, $y_1 = 0.1904$ and $y_2 = 0.8096$

$\lambda_2 = 0.1904 \times 40\,093 + 0.8096 \times 41\,081$

$= 40\,901$ kJ/kmol

Adjust vapour flow from tray-2 with molar latent heats of vapour from tray-1 and tray-2.

$\bar{V}_2 = 136.26 \times 41\,062.3/40\,901$

$= 136.797$ kmol/h

Table 8.53 Material Balance of Tray-2 (from Bottom)

Component	$\bar{L}_2$ kmol/h	$\bar{V}_2$		$\bar{V}_1$	$\bar{L}_3$	
		mole fr.	kmol/h	kmol/h	kmol/h	mole fr.
IPA	2.666	0.1906	26.046	2.60	26.112	0.1288
Water	199.529	0.5696	110.751	133.66	176.520	0.8712
Total	202.195	1.0000	136.797	136.26	202.732	1.0000

With $x_1 = 0.1288$ and $x_2 = 0.8712$

$\gamma_1 = 3.858\ 09$ and $\gamma_2 = 1.063\ 53$

At $t_3 = 83.02$, $y_1 = 0.5121$ and $y_2 = 0.4879$

$\lambda_3 = 40\ 591.2$ kJ/kmol

Adjust vapour flow from tray-3 with molar latent heats of vapour from tray-2 and tray-3.

$$\bar{V}_3 = 136.797 \times 40\ 901/40\ 591.2$$
$$= 137.841 \text{ kmol/h}$$

Table 8.54 Material Balance of Tray-3 (without Fresh Feed)

Component	$\bar{L}_3$ kmol/h	$\bar{V}_3$		$\bar{V}_2$	$\bar{L}_4$	
		mole fr.	kmol/h	kmol/h	kmol/h	mole fr.
IPA	26.112	0.5121	70.588	26.046	65.654	0.3394
Water	176.629	0.4879	67.253	110.251	133.631	0.6606
Total	202.732	1.0000	137.841	136.797	202.285	1.0000

Composition of liquid mixture on tray-4 is 0.3394 mole fraction IPA which is higher than mole fraction 0.2306 of IPA in the fresh feed. Hence, feed is to be introduced on 3rd tray.

Table 8.55 Liquid Composition on Tray-4 after Addition of Fresh Feed on Tray–3

Component	$\bar{L}_4$ kmol/h	F kmol/h	L_4	
			kmol/h	mole fr.
IPA	68.654	23.06	45.594	0.4457
Water	133.631	76.94	56.691	0.5543
Total	202.285	100.00	102.285	1.0000

Tray-to-tray calculations for the rectification section can be carried out in analogous manner to the stripping section. These are summarised in Table 8.57.

Sample calculation for rectification section:

Table 8.56 Material Balance of Tray-4 (from Bottom)

Component	L_4 kmol/h	V_4		V_3	L_5	
		mole fr.	kmol/h	kmol/h	kmol/h	mole fr.
IPA	45.594	0.5837	80.628	70.588	55.634	0.5424
Water	56.691	0.4163	57.505	67.253	46.943	0.4536
Total	102.285	1.0000	138.133	137.841	102.577	1.0000

Further tray calculations of rectification section are summarised in Table 8.57. Total number of theoretical tray are calculated to be 23 with feed on 3rd tray from bottom.

Table 8.57 Summary of Tray-to-Tray Calculations for IPA-Water System at 101.325 kPa

Tray No. (from bottom)	Temperature, °C	Liquid flow rate, kmol/h	Vapour flow rate, kmol/h	Average molar latent heat of vaporisation λ, kJ/kmol	Liquid composition		Activity coefficients		Vapour composition	
					x_1(IPA)	$x_2(H_2O)$	γ_1(IPA)	$\gamma_2(H_2O)$	y_1(IPA)	$y_2(H_2O)$
1	2	3	4	5	6	7	8	9	10	11
Stripping Section:										
1	99.46	65.965	136.260	41 062.3	0.0010	0.9990	9.99657	1.00000	0.0191	0.9809
2	94.83	202.195	136.797	40 926.5	0.0103	0.9897	9.15797	1.00005	0.1565	0.8435
Saturated Fresh Feed		100.000			0.2306	0.7694				
Rectification Section										
3	83.02	202.732	137.841	40 591.2	0.1288	0.8712	3.85809	1.06353	0.5121	0.4871
4	80.96	102.285	138.133	40 505.3	0.4457	0.5543	1.38036	1.54487	0.5837	0.4163
5	80.62	102.577	138.480	40 462.8	0.5424	0.4576	1.21118	1.75378	0.6148	0.3849
6	80.52	102.924	138.483	40 461.9	0.5843	0.4157	1.16117	1.85170	0.6324	0.3678
7	80.47	102.927	138.526	40 449.2	0.608	0.392	1.13745	1.90897	0.6433	0.3568
8	80.44	102.970	138.585	40 432.1	0.622	0.378	1.12478	1.94343	0.6500	0.3499
9	80.44	103.029	138.465	40 449.8	0.6317	0.3683	1.11654	1.96757	0.6553	0.3451
10	80.422	102.909	138.527	40 431.2	0.6388	0.3612	1.11078	1.98537	0.6587	0.3413
11	80.415	102.971	138.546	40 425.8	0.6434	0.3566	1.10717	1.99697	0.6611	0.3388
12	80.413	102.990	138.545	40 426.2	0.6467	0.3533	1.10463	2.00532	0.6630	0.3371

(*Contd.*)

Table 8.57 (*Contd.*)

Tray No. (from bottom)	Temperature, °C	Liquid flow rate, kmol/h	Vapour flow rate, kmol/h	Average molar latent heat of vaporisation λ, kJ/kmol	Liquid composition		Activity coefficients		Vapour composition	
					x_1(IPA)	$x_2(H_2O)$	γ_1(IPA)	$\gamma_2(H_2O)$	y_1(IPA)	$y_2(H_2O)$
1	2	3	4	5	6	7	8	9	10	11
13	80.41	103.089	138.555	40 423.3	0.6486	0.3514	1.10318	2.01014	0.6640	0.3360
14	80.395	103.099	138.554	40 423.7	0.6499	0.3501	1.10318	2.01344	0.6649	0.3351
15	80.38	103.098	138.559	40 422.2	0.6511	0.3489	1.10318	2.01649	0.6657	0.3343
16	80.367	103.103	138.560	40 421.8	0.6522	0.3478	1.10318	2.01929	0.6665	0.3335
17	80.354	103.104	138.560	40 421.3	0.6533	0.3467	1.10318	2.02209	0.6672	0.3327
18	80.343	103.104	138.564	40 420.3	0.6542	0.3458	1.10318	2.02439	0.6679	0.3321
19	80.332	103.108	138.562	40 421.0	0.6552	0.3448	1.10318	2.02694	0.6686	0.3314
20	80.32	103.106	138.570	40 418.2	0.6561	0.3439	1.10318	2.02924	0.6692	0.3308
21	80.312	103.114	138.564	40 419.9	0.6569	0.3431	1.10318	2.03128	0.6698	0.3302
22	80.301	103.108	138.575	40 416.7	0.6577	0.3423	1.10318	2.03333	0.6703	0.3297
23	80.294	103.119	138.570	40 418.1	0.6584	0.3416	1.10318	2.03512	0.6708	0.3292

Note: It may be noted that in this method number of theoretical trays are worked out to be 23 (to achieve 67.08 mole % IPA in vapour) with fresh feed entry on third tray from the bottom. This number is much greater than that obtained by McCabe–Thiele diagram. It is therefore advisable to carry out tray to tray calculations for design of an azeotropic column with non-ideal behaviour. Further it is recommended to adjust pressure drop on each tray with the help of anticipated pressure drop across the tray. This will change total operating pressure on each tray.

With reflux flow of 102.195 kmol/h, difference in calculated in tray 23 is 0.924 kmol/h, i.e., 0.9%. This is quite low. However, if energy balance of each tray is also calculated, this could have been negligible.

This type of repetitive calculations can be conveniently carried out with a spreadsheet (such as Excell®) on the computer.

Example 8.19 Refer to Table 8.51 in Example 8.18. It can be seen that boiling point of 10 mole % IPA mixture is 83.85°C. This indicates that boiling point variation of only 1.35°C takes place from 0.1 mole fraction to the azeotropic point. Thus if the column is operated to separate up to 0.1 mole fraction, the column operation is nearly isothermal. In the overhead condenser, cooling medium at 72°C (max.) can be used while in the reboiler, heating medium at 95°C should be adequate.

For the above reason it is decided to operate the main column in the range of 0.1 mole fraction to 0.675 mole fraction IPA. It will be provided its own reboiler as shown in Fig. 8.54.

Fig. 8.54 *Azeotropic Distillation of Aqueous Isopropyl Alcohol (Conventional System)*

Liquid mixture, containing 0.1 mole fraction, is fed to another column in which open steam will be used for stripping to achieve 0.1 mole % (0.0033 mass %) IPA in the bottoms. Vapour from this column will be fed to the main column to supplement the heat.

For the new arrangement, rework the number of theoretical stages, required for both the columns for $R = 3$.

Solution

Note: It can be noted that cooling medium; water for vapour at 72°C is required in the overhead condenser while heating medium at 95°C is required in the reboiler. This suggests water as an auxiliary heat transfer medium in the heat pump (refer to para 6 of Sec. 8.9.1). Saturation pressure of water at 72°C is 0.34 bar a and at 95°C is 0.845 bar a. Compression ratio across the thermo-compressor will be just 2.485.

In the second column, since live steam is to be injected, its temperature can be just above 100°C. This permits use of flash steam at low pressure (may be at 105°C or 1.21 bar a) as live steam.

Use of live steam at low pressure and water as the auxiliary heat transfer medium in the heat pump will make distillation quite energy efficient. Thermal energy requirement will be considerably reduced with this arrangement.

From Fig. 8.55 number of theoretical stages required for the separation up to 10 mole %. IPA from 23.06% mole IPA (feed), $N = 8$.

Second column is an isothermal steam stripper. In stripping liquid, solution is contacted with vapour or gas in a countercurrent manner to strip off the most volatile component of liquid solution. Most volatile component of liquid solution is transferred to gas or vapour. Stripping is a reverse of gas absorption. For stripping, low pressure and high temperature are favourable.

Industrial strippers are of two types:

1. In first type of stripper, hot gas/steam (can be called stripping agent) is introduced at the bottom of stripper and the same is obtained from external source.
2. In second type, hot vapour is generated by partial vaporization of liquid solution in reboiler at bottom. This type of stripper is like a bottom section (stripping section) of distillation column. Feed solution is introduced from top and reboiler is provided at the bottom. Reflux is not provided at the top. If the component to be separated from feed is having very high relative volatility, then this type of stripper can be selected.

In this example, first type of stripper is selected. Stripping can be assumed as isothermal at 100°C.

Let, G_s = Molar flow rate of steam, kmol/h

L_s = Molar flow rate of water of feed, kmol/h

$$Y = \frac{y}{1-y} = \frac{\text{moles of IPA in vapour phase}}{\text{moles of steam in vapour phase}}$$

$$X = \frac{x}{1-x} = \frac{\text{moles of IPA in liquid phase}}{\text{moles of water in liquid phase}}$$

Equilibrium data can be approximated from Table 8.51.

x	y	X	Y
0	0	0	0
0.02	0.2495	0.0204	0.3324
0.05	0.3993	0.0526	0.6647
0.075	0.4561	0.081	0.8385
0.1	0.489	0.1111	0.957

Equilibrium curve is plotted in Fig. 8.55.

Fig. 8.55 *Stripping of Dilute IPA with Live Steam*

Minimum amount of steam required for desired separation can be obtained by following equation:

$$G_{sm}(Y_{1m} - Y_2) = L_s(X_1 - X_2) \tag{8.127}$$

where, Y_1 and X_1 represent the molar ratios at top of the stripper and Y_2, X_2 represent the molar ratios at the bottom of stripper.

Here equilibrium curve is concave upward, hence Y_{1m} is equilibrium value of Y corresponding to the value of X_F.

$$x_F = 0.1,\ X_F = \frac{x_F}{1 - x_F} = 0.1111$$

$$Y_{1m} = 0.957 \quad \text{(From Fig. 8.55)}$$

$$Y_2 = 0, \quad X_1 = X_F = 0.1111, \quad X_2 = \frac{0.001}{1 - 0.001} \cong 0.001$$

$$G_{sm} = \frac{\bar{L}_s(X_F - X_2)}{Y_{1m} - Y_2}$$

or $$\left(\frac{G_s}{\bar{L}_s}\right)_m = \frac{0.1111 - 0.001}{0.957 - 0} = 0.115$$

Let actual amount of steam, $G_s = 1.5\ G_{sm}$

$$\frac{G_s}{\bar{L}_s} = 1.5 \times 0.115 = 0.1725 = \frac{X_F - X_2}{Y_1 - Y_2}$$

$$0.1725 = \frac{0.1111 - 0.001}{Y_1 - 0}$$

$$Y_1 = 0.6383$$

Draw the operating line starting from (X_2, Y_2) to (X_1, Y_1). Staircase construction between equilibrium curve and operating line, starting from (X_1, Y_1) to (X_2, Y_2) gives number of theoretical stages required for the desired separation. From Fig. 8.55, number of theoretical stages required in the steam stripper; $N = 6$.

Example 8.20 Ethanol forms a minimum boiling azeotrope at 78.15°C and 101.325 kPa, with water having composition of 10.57 mole % water. Vapour pressure of ethanol can be calculated using Antoine equation[14].

$$\log p_v = 7.2371 - \frac{1592.90}{(T - 46.95)}$$

where, p_v = Vapour pressure, kPa
T = Saturation temperature, K

Extractive distillation technique is known for separation of an azeotrope using a solvent. In an extractive process, aqueous inorganic salt solution is used as a solvent to break the azeotrope. Lowering of vapour pressure is experienced when an inorganic salt is dissolved in water. Vapour pressures of aqueous calcium chloride solutions are given in Table 8.58.

Table 8.58 Vapour Pressure of Water over Aqueous Calcium Chloride Solution[49]

Temperature, °C	Calculated vapour pressure of water, Torr			
	Strength of $CaCl_2$ solution, mass %			
	0	20	25	35
40	55.3	47.9	41.5	30.1
70	233.7	197.7	179.6	136.4
90	525.9	448.4	408.7	318.0
100	760.0	660.6	593.9	466.1
110	1074.6	905.9	841.9	629.5
120	1489.2	—	—	886.6

It is proposed to use aqueous 25% $CaCl_2$ solution (by mass) as a solvent for dehydration of aqueous ethanol. Proposed flow diagram is shown in Fig. 8.55.

Fig. 8.56 *Extractive Distillation of Aqueous Ethanol with Aqueous Calcium Chloride Solution (25 mass %)*

Plot x-y diagrams of ethanol-water system at 101.325 kPa, with and without the solvent. Superimpose both the diagrams and study the effect of addition of aqueous $CaCl_2$ solution on the separation.

(a) A feed consisting of 50 mass % ethanol under saturated liquid conditions is to be distilled to give 99.8 mole % (99.92 mass %) ethanol as distillate and 0.1 mole % ethanol in bottoms in the extractive distillation column. Using McCabe–Thiele technique and for $R = 2\,R_m$, find the number of theoretical stages required for the separation at 101.325 kPa and locate the feed stage.

(b) If the distillate quality is restricted to 96.0 mole % (98.4 mass %) ethanol for the case (a), find the number of theoretical stages required for the separation and locate the feed stage.

Solution

Azeotrope contains 0.1057 mole fraction (0.0442 mass fraction) ethanol. Let suffix 1 represent ethanol and suffix 2 represent water.

At the boiling point of the azeotrope, $t = 78.15°C$ or $T = 351.3$ K,

$$\log p_{v1} = 7.2371 - \frac{1592.90}{(T - 46.95)} = 2.0033$$

$$p_{v1} = 100.763 \text{ kPa} = 755.78 \text{ Torr}$$

From steam tables, $p_{v2} = 43.93$ kPa = 329.50 Torr

$$\gamma_1 = 760/755.78 = 1.0056$$

$$\gamma_2 = 760/329.50 = 2.3065$$

van Laar equations:

$$\ln(1.0056) = A\left[\frac{0.1057}{\left(\frac{A}{B}\right)0.8943 + 0.1057}\right]^2$$

$$\ln(2.3065) = B\left[\frac{0.8943}{\left(\frac{B}{A}\right)0.1057 + 0.8943}\right]^2$$

Solving by Mathcad,

$$A = 1.950\,34 \text{ and } B = 0.9329$$

Using values of A and B, t-x-y data can be generated in an analogous manner to the IPA-water system (Example 8.18). When use of aqueous $CaCl_2$ solution (25 mass %) is considered, p_{v2} will be replaced by p'_{v2}, i.e., vapour pressure of water over the aqueous solution. For this purpose a graph of p'_{v2} vs temperature can be plotted and p'_{v2} values are read at the required temperature.

Sample calculations for $x_1 = 0.9$ and $x_2 = 0.1$:

$$\gamma_1 = 1.004\,98 \text{ and } \gamma_2 = 2.318\,96$$

Assume $t = 78.2°C$ or $T = 351.35$ K.

$$p_{v1} = 757.22 \text{ Torr}$$

$$p'_{v2} = 254.6 \text{ Torr against } p_{v2} = 330.15 \text{ Torr}$$

$$p = \Sigma x_i \cdot \gamma_i \cdot p_{vi}$$
$$= (0.9 \times 757.22 \times 1.004\,98) + (0.1 \times 254.6 \times 2.318\,96)$$
$$= 684.89 + 59.04 = 743.93 \text{ Torr} \neq 760 \text{ Torr}$$

Revise $t = 78.8°\text{C}$ or $T = 351.9$ K.

$$p_{v1} = 775.49 \text{ Torr}$$
$$p'_{v2} = 260.8 \text{ Torr}$$
$$p = 761.9 \text{ Torr} \approx 760 \text{ Torr}$$
$$y_1 = (0.9 \times 775.49 \times 1.004\,98)/760 = 0.923$$

Similarly *t-x-y* data are generated for ethanol-water-solvent system. Both *t-x-y* data are presented in Table 8.59 and Fig. 8.57(a).

Table 8.59 Vapour-Liquid Equilibrium for Ethanol-Water System at 1 atm

Without solvent, mole fraction ethanol			With solvent, mole fraction ethanol		
In Liquid	**In Vapour**	**Boiling point, °C**	**In Liquid**	**In Vapour**	**Boiling point, °C**
x	***y***	***t***	***x***	***y***	***t***
0	0	100.0	0	0	107.0
0.025	0.2503	92.8	0.025	0.298	97.7
0.05	0.365	88.8	0.05	0.4234	92.95
0.1	0.468	85.0	0.1	0.531	88.35
0.2	0.542	82.3	0.2	0.6026	85.1
0.3	0.578	81.2	0.3	0.638	83.75
0.4	0.6096	80.35	0.4	0.668	82.7
0.5	0.647	79.65	0.5	0.703	81.8
0.6	0.693	79.05	0.6	0.745	80.9
0.7	0.75	78.6	0.7	0.7945	80.05
0.8	0.817	78.25	0.8	0.8513	79.3
0.8943	0.8943	78.15 – azeotrope	—	—	—
0.96	0.953	78.1	0.9	0.921	78.8
1.0	1.0	78.1	1.0	1.0	78.1

Case (a)

Refer to Fig. 8.57(b), $x_D = 0.998$, $z_F = 0.2811$ (or 0.5 mass fraction) and $x_W = 0.001$

$$\frac{x_D}{R_m + 1} = 0.2005$$

$$R_m = 3.98$$

$$R = 2\,R_m = 2 \times 3.98 = 7.96$$

$$\frac{x_D}{R+1} = 0.1114$$

Total 29 theoretical stages are required for the separation and the feed will be introduced on 14th stage.

Fig. 8.57(a) *Vapour Liquid Equilibrium Curves for Ethanol Water System with and without Aqueous Calcium Chloride Solution (25 mass %)*

Case (b)

For $x_D = 0.96$, the graph [similar to Fig. 8.56(c)] was redrawn and following data were collected:

$$\frac{x_D}{R_m+1} = 0.2701$$

$$R_m = 2.554$$

$$R = 2 \times 2.554 = 5.108$$

$$\frac{x_D}{R+1} = 0.1572$$

With these values of R, the number of theoretical stages were calculated to be 17 and the feed will have to be introduced on 14th stage.

Required number of theoretical stages for both the cases were found using McCabe–Thiele diagrams [Figs. 8.56(b) and (c)]. However, it is recommended to recalculate the requirement by tray-to-tray calculations for actual process design.

Fig. 8.57(b) *Extractive Distillation with Aqueous Calcium Chloride Solution (25 mass %)*

Note: It may be noted that single stage extractive distillation (ED) column is adequate to produce ethanol with 99.8 mole % purity while in most ED systems, two columns are required for the same degree of separation. Boiling point elevation at atmospheric pressure (at the bottom of the column) is just 7°C. Also this ED system calls for 50% or less energy than the conventional ED columns.

Presence of high chlorides means corrosive nature of the aqueous system. The column, reboiler and concentrator should be built with material of construction, resistant to chlorides. Special stainless steel, such as Ti stabilised SS 316-L or high nickel alloy, could be used for such corrosive service. Circulation pumps of Alloy-20 could be used. However, cost of such materials of construction is higher than that of normally used materials. Nevertheless, overall operating cost of the ED system with aqueous $CaCl_2$ solution will be less than the conventional ED system.

Fig. 8.57(c) *Extractive Distillation with Aqueous Calcium Chloride Solution (25 mass %)*

Results of Case (a) and Case (b) indicate that the ED column will be more than 70% taller in Case (a). Also reflux ratio of Case (a) is nearly 55% more than Case (b). Both these factors call for higher investment and higher energy consumption for Case (a).

Distillate of Case (b) contains 4 mole % (1.6 mass % or 16 032 ppm or 12 650 mg/L) water. To dehydrate ethanol further, three options are shown in Fig. 8.56. In a conventional manner (Option I), distillate with 4 mole % water will be distilled in another column to yield the azeotrope at the top (to be recycled) and anhydrous ethanol at the bottom. In Option II, part of the vapour from the ED column will be fed to a pervaporation unit. Here again, contaminated water (with some ethanol), removed in the membrane unit, can be recycled to the feed of the ED column. Drying of the distillate from ED column by molecular sieves is shown as Option III. All three options are well proven in the industry and the selection should be made on the basis of minimum total operating cost. Please refer Section 8.9.3 for detailed discussions on various options.

Example 8.21 Nitric acid water mixture forms maximum boiling azeotrope at 1 atm a and 122°C, with a composition of 64.1% by mass of HNO_3. Distillate contains 99.9% HNO_3 (by mass). Separation is achieved from the azeotropic mixture by extractive distillation using concentrated Sulphuric acid (99% by mass) as solvent. Residue of this extractive distillation column is 80% by mass H_2SO_4 and remaining water. Extractive distillation is carried out in glass packed tower type distillation column at 180 Torr a pressure. Glass Raschig rings are used as a packing material. Determine height of packing required for the desired separation in this extractive distillation column.

Solution

Mole fraction of HNO_3 in feed,

$$x_F = \frac{(64.1/63)}{\left(\frac{64.1}{63}\right)+\left(\frac{35.9}{18}\right)} = 0.3378$$

$$x_D = \frac{(99.9/63)}{\left(\frac{99.9}{63}\right)+\left(\frac{0.1}{18}\right)} = 0.9965$$

Let $x_W = 0.001$

Here H_2SO_4 is not vaporised in distillation column and in reboiler. Hence, this distillation can be approximated as binary distillation and MeCabe–Thiele method can be applied. However, effect of addition of H_2SO_4 on vapour-liquid equilibrium data or on relative volatility of HNO_3–H_2O mixture must be considered.

Topmost temperature of column = Dew point temperature of overhead vapour
≅ Condensation temperature of pure HNO_3 vapour

From Table 3.16 of Ref. 8,

At 180 Torr a, condensation temperature of 100% HNO_3 is 48°C.

At 48°C, vapour pressure of water vapour over 80% H_2SO_4 (by mass) is 0.106×10^{-2} bar. (From Table 3.13 of Ref. 8).

Relative volatility at topmost temperature

$$\alpha = \frac{p_{vA}}{p_{vB}}$$

p_{vA} = Vapour pressure of 100% HNO_3 = 180 Torr

p_{vB} = Vapour pressure of water over 80% H_2SO_4
= 0.106×10^{-2} bar = 0.795 Torr

$$\alpha_1 = \frac{180}{0.795} = 226.4$$

Bottommost temperature = Bubble point of 80% H_2SO_4 ≅ 160°C
(From Table 3.15 of Ref. 8)

But at 160°C vapour pressure of HNO_3 is not available. Hence, here at intermediate temperature or at 90°C second value of α is determined.

At 90°C p_{vA} = 820 Torr (From Table 3.16 of Ref. 8)

$$p_{vB} = 0.0115 \text{ bar} = 8.6257 \text{ Torr} \quad \text{(From Table 3.13 of Ref. 8)}$$

$$\alpha_{av} = \frac{820}{8.6257} = 95.06$$

Considering this value as α_{av}, Table 8.60 is prepared.

Table 8.60 Vapour-Liquid Equilibrium Data for HNO_3–H_2SO_4 System

x	$y = \dfrac{\alpha_{av} x}{[1 + (\alpha_{av} - 1)\, x]}$	x	$y = \dfrac{\alpha_{av} x}{[1 + (\alpha_{av} - 1)\, x]}$
0.02	0.66	0.4	0.9845
0.04	0.7984	0.5	0.9896
0.06	0.8585	0.6	0.993
0.08	0.892	0.7	0.9955
0.1	0.9135	0.8	0.9973
0.2	0.9596	0.9	0.999
0.3	0.976	1.0	1.0

Equilibrium curve of y vs. x is shown in Fig. 8.58.

Fig. 8.58 *Extractive Distillation of Aqueous Nitric Acid*

Equation of q-line.

$$y = \frac{q}{q-1} x - \frac{z_F}{q-1}$$

$$q = \frac{H_G - H_F}{H_G - H_L}$$

Let feed is a liquid at atmospheric temperature = 30°C.

$$q = \frac{(H_G - H_L) + (H_L - H_F)}{(H_G - H_L)} = \frac{\lambda_F + C_{LF}\,(t_b - t)}{\lambda_F}$$

Assume that bubble point of the feed mixture at 180 Torr a = 85°C

At 85°C λ_N = 39 000 kJ/kmol (Ref. 50)

λ_W = 41 326 kJ/kmol (from steam tables)

$$\lambda_F = \sum \lambda_i\, x_i = 39\,000 \times 0.3378 + 41\,326 \times 0.6622$$

$$= 40\,540.3 \text{ kJ/kmol}$$

At 57.5°C C_{LN} = 110.88 kJ/(kmol · °C)

C_{LW} = 75.36 kJ/(kmol · °C)

$$C_{LF} = 110.88 \times 0.3378 + 75.36 \times 0.6622$$

$$= 87.36 \text{ kJ/(kmol} \cdot \text{K)}$$

$$q = \frac{40\,504.3 + 87.36\,(85 - 30)}{40\,540.3} = 1.1185$$

$$y = \frac{1.1185}{0.1185}\,x - \frac{0.3378}{0.1185}$$

$$= 9.439\,x - 2.85$$

For $y = 0.7, \quad x = 0.376$

$$\frac{x_D}{R_m + 1} = 0.98 \quad \text{(From Fig. 8.58)}$$

$$\frac{0.9965}{R_m + 1} = 0.98$$

$$R_m = 0.02$$

Let $R = 0.5$

$$\frac{x_D}{R + 1} = \frac{0.9965}{1.5} = 0.6643$$

From Fig. 8.58 $N' = 3$ for enrichment from x'_r to x_F

$$N = 3 + N_s$$

where, N_s = Nos. of theoretical stages required from 0.01 to 0.001

$$x_W = 0.001, \quad x'_r = 0.01$$

$$N_s = \frac{\log\left[\dfrac{(K'/S' - 1)\,(x'_r/x_W - 1)}{1/S'\,(K' - 1)} + 1\right]}{\log\,(K'/S')} + 1 \qquad (8.21)$$

$$K' = \text{slope of equilibrium curve} = \frac{0.49}{0.01} = 49$$

$$S' = \frac{\Delta y}{\Delta x} = \text{slope of stripping section operating line}$$

$$= \frac{0.4 - 0.01}{0.19 - 0.01} = 2.1667$$

$$N_s = \frac{\log(9.7812)}{\log(22.615)} + 1 = 1.73$$

Total number of theoretical stages required,

$$N = 3 + 1.73 = 4.73, \text{ say } 5$$

HETP for glass Raschig rings = 1 m

Total height of packing required = 5 m

Note: Although $R_m = 0.02$ only, R was selected 0.5, i.e., 25 times. This is because the system is highly exothermic and non-ideal. McCabe–Thiele method is not really suitable for such a non-ideal system.

8.9 ENERGY CONSERVATION IN DISTILLATION

It was stated earlier that distillation is the most widely used separation technique in chemical process industries but energy consumption in distillation is relatively high. Hence, saving of energy in distillation operation is important for lowering overall cost of process plant. This aspect is also becoming important in biotechnology routes.

For saving the energy in distillation systems there are two options:

1. Make the distillation more energy efficient; either by design or by operating the column more efficiently.
2. Replace the distillation partially or completely by a new separation technique.

8.9.1 Energy Efficient Distillation by Design

To decrease the energy consumption of distillation various design options can be considered. To name a few

1. Optimum design of the system
2. Heat integration
3. Use of high efficiency trays or high efficiency packings
4. Advanced process control
5. Use of thermally coupled distillation columns
6. Use of heat pumps (ejector/thermocompressor).

1. Optimum design of system: Process engineers have a general tendency to overdesign the system but at the expense of energy. For example, very high purities are specified for the distillate or bottom product which may not be warranted. At times, solvents are recycled in the system and very high purity may not be required. By designing the system for the desired purity only, reflux ratio can be trimmed thereby energy consumption can be reduced. In Sec. 8.8.2, a case of extractive distillation of cyclohexane and benzene mixture was discussed. As cyclohexane is a product, its purity of 99.3% was specified as a distillate from the first column. However, since benzene is a reactant which is to be recycled for further conversion to cyclohexane, its purity of 85% was specified as a distillate from the second column. If benzene purity of over 99% is specified, second column will be much taller with high reflux ratio, resulting in high energy consumption in the reboiler. Take another example of

ethanol-water mixture. When ethanol is a desired product for mixing with petrol, very high purity of ethanol will be needed.

For heterogeneous azeotropic distillation, first column is used as a concentrator to produce distillate of near azeotropic composition. Subsequently, two more columns are required for separation of the components. Cho and Jeon[42] have proved after rigorous simulation by varying design parameters that while separating aqueous IPA solution, the first column (the concentrator) should be designed to produce distillate containing 38.7 mole % IPA (and *not* the azeotrope) which should be subsequently processed in a two-column system to dehydrate IPA. This design is claimed to consume lowest energy for the entire distillation system.

Knapp and Doharty[47, 48] have proposed a two-column system (and not three column system) for concentrating dilute aqueous ethanol solution (containing just 4.2 mole % ethanol) from fermentor by adding approximately 0.043 kmol acetone per kmol feed and applying pressure swing distillation technique. This system is claimed to consume much less energy than conventional system.

Several such distillation case studies can be cited from the literature in which it has been demonstrated that by careful selection of process parameters at the design stage and the right technique, substantial energy savings can be achieved in operation. However, most energy efficient design could be expensive from equipment cost point of view. Nevertheless, energy saving outweighs capital cost in most cases.

2. Heat integration: Heat integration is low risk technology and it results in substantial energy saving. Reboiler of distillation column can act as a *cooler* for other unit. For example, in acetaldehyde plant, exhothermicity of the reaction is removed by circulating saturated liquid leaving from the bottom of distillation column in tubeside of shell and tube-type reactor. Liquid is vaporised and vapour is sent back to the distillation column. Thus reactor act as a reboiler for the distillation column. An additional reboiler is provided for the distillation column for supplying the balance heat (vapour) to the column.

Similarly, overhead condenser can act as a *heater* or *reboiler* for other unit. For example, in cryogenic distillation of air, overhead condenser of higher pressure distillation column also acts as a reboiler for the lower pressure distillation column. In such a heat exchanger, nitrogen condenses on one side and on other side, liquid oxygen boils.

Overhead vapour of distillation column can be partially used as heating medium of reboiler of other distillation column. This can be achieved conveniently with an ejector as discussed in Sec. 8.9.1 (f).

In Sec. 8.8.3, it was seen that pressure swing distillation technique offers an opportunity for thermal integration of condensers and reboilers of two columns as they operate at different temperatures.

Bottom product cooler can also act as feed preheater. Such an energy integration decreases the heat requirement of the preheater and also reduces the cooling load of the product cooler. It decreases heat required for reboiler and improves $(NPSH)_A$ of bottom product transfer pump.

3. Use of high efficiency trays or high efficiency packings: Number of equilibrium stages of existing distillation column can be increased by replacing sieve trays or bubble cap trays with high efficiency packings or trays. For the fixed degree of separation, increase in number of equilibrium stages reduces reflux ratio and consequently reduces reboiler energy. Other advantage of using high efficiency packings (such as structured packings) or high efficiency trays (such as modified sieve trays) is to increase in capacity of the distillation column. Disadvantage of this replacement is higher cost of structured packings or of high efficiency trays. Generally savings in energy cost outweigh the cost of high efficiency packings/trays.

Example[51]: Replacement of sieve trays of 400 mm diameter distillation column of styrene plant by a packed column, packed with structured packings, resulted in saving of energy consumption by 17.7%. This column was operating at its capacity of 24 500 kg/h of styrene. Its feed composition was 54% ethyl benzene and 46% styrene (by mole), distillate composition was 96.9% ethyl benzene and residue composition was 99.7% styrene (by mole). After replacement of sieve trays by a column with structured packings, reflux ratio was reduced from 7.3 to 5.8 and reboiler energy was reduced from 1605 kJ/kg of styrene to 1321 kJ/kg of styrene. In this modification, feed flow rate and purity of products were kept same.

Similarly replacement of conventional bubble cap trays by structured packing increases the capacity of the column or saves energy by decreasing the reflux ratio. However, structured packing cannot work efficiently at higher turndown ratio.

4. Advanced process control: Plant operators normally operate the distillation columns with a sufficient safety margin by achieving actual compositions of distillation products which are better than specifications. Operating a distillation column in this manner requires extra reflux and energy, but some margin is necessary so that the plant operator can cope with process upsets and other problems. The 'safety' margin is smaller in the plants which are properly designed and operated.

Installation of advanced process control systems based on accurate analytical sensors, reliable control loop hardware and instrumentation and good process models increases confidence in operators. In many instances, this added confidence translated to a reduced margin of 'safety', reflux ratio and reboiler energy. Nowadays, instrumentation and controls are given due weightage at the design stage itself. In fact data logging system provides useful information on optimising the column performance.

Example[51]: A conventional distillation column was used for separating a binary mixture of propane and proplyene. Feed composition was 73% propylene and 27% propane (by mole). Required purity of top product was 99.7 mole % propylene for which reflux ratio was 13.5. With a sufficient margin, conventional distillation column was operated with excess reflux ratio of 15.5 to get the purity of propylene of 99.9 mole % instead of the required purity of 99.7 mole %. After installation of distributed control system (DCS) on this column, more accurate control of the purity of propylene at 99.7 mole % could be achieved which reduced energy consumption up to 13.5%. However, from a practical standpoint, operators cannot control product purity precisely at required composition in the conventional system.

5. Use of thermally coupled distillation[52]: In the separation of multicomponent mixture (more than 2), thermally coupled distillation columns require less energy and fixed cost as compared to conventional distillation of multicomponent mixture. To understand this, consider the separation of a ternary mixture having components A, B and C by distillation. A is the most volatile component and C is the least volatile component. Component B is having intermediate volatility. Conventional methods for separating the ternary mixture by distillation is either via direct sequence or indirect sequence, as shown in Figs. 8.59(a) and (b).

Fig. 8.59 *Multicomponent Distillation in Sequence*

Fig. 8.60 *Thermally Coupled Distillation Columns*

For the given ternary mixture, selection between these two conventional arrangement of distillation column depends on composition of the ternary mixture, relative volatilities, latent heats of vaporisation, boiling points, heat sensitivities, etc. Compared to these conventional arrangements of distillation columns thermally coupled distillation columns require less energy consumption and fixed capital investment. For the separation of the same ternary mixture of A, B and C by thermally coupled distillation, two types of arrangement are possible, as shown in Figs. 8.60(a) and (b).

In thermally coupled side column rectifier, vapour stream is withdrawn from the stripping section of main column and is sent for rectification in side column where an overhead condenser is provided. Liquid stream from the bottom of side column is sent back to the main column.

In thermally coupled side column stripper, liquid stream from the enriching section the main column is withdrawn and is sent to side column for stripping where reboiler is provided at bottom. Vapour stream from side column is sent back to the main column.

Selection between these two arrangements depend on concentration profile of intermediate component B. If concentration of component B is maximum at any point of stripping section of the main or upstream column, then side column rectifier is selected. By withdrawing a side stream from the point at which the concentration of B is maximised, the distillation of B from either C or A is made much easier.

Side rectifiers and side strippers can be cost effective on any ternary separation where less than half of the feed leaves in middle product or where relatively low purity of middle product is acceptable. Compared to conventional column arrangements side stripper or side rectifier can reduce the energy consumption up to 50%. Saving in fixed cost is also achieved because thermally coupled distillation columns avoid the use of one condenser or reboiler and require reduced column diameter or fewer stages for at least one column. Concept of thermally coupled distillation is also applicable to any pseudoternary systems, i.e., multicomponent mixtures requiring separation into three products. Hence, it is applicable to the distillation of any mixture of more than two components.

Refer to Example 8.9 in which separation of fatty acids from a mixture is dealt. Such a system can be considered for thermal coupling. In this case thermally coupled side column is recommended.

In a petroleum refinery, this technique is extensively practised to produce different cuts (products like kerosene, naphtha, petrol, etc.) from crude by having number of side-stream rectifiers and strippers.

As an alternate to the thermally coupled columns, a novel idea in design of tray type distillation column is to have partitioned distillation column (Ref. 53) (also called a divided-wall column or Petyluk column) for multicomponent system. In such a column, a vertical baffle is placed (Fig. 8.61), which separates the feed location from that at which the intermediate boiling product is withdrawn. On the feed side of the partition, separation is achieved between the light (A) and heavy (C) fractions while the intermediate boiling fraction (B) is allowed to migrate to top and bottom sides of the partition. On the other side of the partition, the light fraction is washed

down, thereby intermediate boiling fraction (B) of desired purity is withdrawn. Such a column is larger in diameter than the otherwise required two columns but overall capital and operating costs could be significantly (up to 30%) lower. Process design of such columns is quite complex and is not available in open literature.

Fig. 8.61 *Partitioned Distillation Column*

6. Use of heat pumps in distillation: Steam ejectors have been used in limited distillation applications as inexpensive heat pumps. An ejector uses high pressure fluid to entrain low pressure fluid. Steam ejectors can be operated in different ways.

Simplest way to use an ejector is to operate with live steam. Figure 8.62 shows the proposed arrangement. Part of the top vapour is compressed in an ejector utilising live (fresh) steam as motive fluid. Two or more ejectors can be used in parallel to improve flexibility. Use of such an ejector system requires that (i) top product is water and (ii) temperature difference between the bottom and top of the column is low. Hydrogen peroxide-water distillation column can be fitted with a live steam ejector. This ejector system reduces steam and cooling water requirements for the distillation unit. It also reduces the load of vacuum pump. Refer to Example 11.1 for use of a thermocompressor in a triple-effect evapourator system. Similar arrangement is also recommended in distillation.

Figure 8.63 is a capacity ratio curve for the thermocompressor (ejector), operating with saturated steam at 20.7 bar g.

In Fig. 8.64, a system is shown in which top vapour product is used as a motive as well as entrained fluid. In this case, it should be possible to heat the top product to a substantially higher temperature and pressure than the column's operating conditions. Also the temperature difference between top and bottom of the column should be low. Ethyl benzene/styrene splitter can be fitted with such an ejector system. A 225×10^6 t/a styrene monomer plant when operated with such an ejector system reduced steam and cooling water requirements by 25% and 32%, respectively. Capital investment for

the modifications is expected to be repaid in less than 18 months. Acetic acid-water, ethylene glycol-water systems can also be operated in this manner.

Fig. 8.62 *Ejector System with Live Steam as the Driving Medium*[54]
(Reproduced with the Permission of Gul Publishing Co., USA)

Fig. 8.63 *Capacity Ratios of Steam Jet Thermocompressors Operating with Saturated 20.7 bar g Motive Steam*
(Courtesy: Mazda Ltd. Ahemedabad)

Fig. 8.64 *Ejector System with Use Top Product as the Driving Medium*[54]
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In a third alternative, bottom product can be used to generate motive fluid for the ejector (Fig. 8.65). Part of the top vapour is condensed in a heat exchanger with bottom product which operated under reduced pressure (say subatmospheric). High pressure bottom product vapour provides the driving medium for the ejector. For this system to operate satisfactorily, three conditions need be satisfied: (i) It should be possible to heat the bottom product to a substantially higher temperature and pressure than the operating conditions of the column, (ii) Top product is not suitable for compression with the ejector, and (iii) Temperature difference between bottom and top of the column is low. Alcohol (methanol or ethanol)-water distillation column can be fitted with such an ejector system.

Innovative flow sheets were presented as Figs. 8.62 8.64 and 8.65 for use of an ejector for energy conservation. Process engineer should therefore always look for innovative designs which are technically feasible and select the best option based on all aspects, including operational convenience.

It may be noted that in all the applications, it is necessary to have low temperature difference between top and bottom products of the column. Also the ejectors should be large in size so that with low differential pressure between motive and entrained fluids is permitted. In Figs. 8.62, 8.64 and 8.65, number of peripheral equipments are shown which require substantial capital expenditure. Because of these reasons, ejector installation is of lesser interest for the smaller columns. However, when large distillation column and substantial energy input are required, use of ejector(s) could prove economically viable (Ref. 54).

In Fig. 8.62, live steam is used for compressing the overhead vapours in an ejector to the reboiler for its use as heat transfer medium. As an alternate to the ejector, a vapour compressor (Ref. 55) can be used to compress the overhead vapours (Fig. 8.66). The compressor could be a twin lobe blower, a radial or an axial–flow turbocompressor

Fig. 8.65 *Ejector System with Bottom Product as the Driving Medium*[54]
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Fig. 8.66 *Distillation Columns with Direct Vapour Compressor*[55]
(Reproduced with the Permission of Chemical Engineering by Access Intelligence, USA)

or a screw compressor, driven directly or via gear box by an electric motor or a steam turbine. For overhead vapour having low molar mass (such as water), compression is rather restricted up to 10 bar. For high molar mass vapours, multistage compressors can be used to compress them up to 80 bar. For partial load operation, the compressor should have a variable speed drive or kick-back arrangement, performance of Vapour Recompression System (VRC) is measured by calculating the ratio of reboiler duty (kW) to the power consumption (kW) of the compressor. This ratio is called Coefficient of Performance (COP) and typical value ranges from 6 to 15. Although VRC system

can be used for any distillation column on technical ground, it is favoured in the following cases due to the economic factors:

1. When reboiler duty is high (> 2 MW), VRC system is promising.
2. Lower temperature rise across the compressor is preferred.
3. When chilled water or brine is used to condense overhead vapours in the condenser, use of VRC system is advantageous.
4. Columns that separate products with low separation factors require larger number of stages and high reflux ratio. Such columns are most suitable for adoption of VRC system. Separation of propylene-propane, ethylene-ethane, ethyl benzene-xylene, *iso*-propanol-water (refer to Example 8.18), etc., are the systems which can benefit significantly by adoption of the VRC system.

In a typical case, 60% (by mass) *iso*-propanol feed (balance water) is distilled to achieve azeotrope (87.5% *iso*-propanol) with bottom product containing 15% *iso*-propanol. When overhead azeotropic mixture is compressed and used as a heating medium in the reboiler, coefficient of performance in excess of 10 could be achieved.

Where low cost heat or free waste energy is available for use in the reboiler, VRC system is not recommended. Also when the column is operated under high vacuum (>710 Torr), VRC is not economical because compression ratio becomes high.

One more option for reducing energy consumption in distillation is to adopt heat pump with an auxiliary heat transfer medium (Ref. 56). In Fig. 8.67, distillation column with auxiliary heat transfer medium is shown. In the overhead condenser, vapours are condensed by evapourating the auxiliary medium. A compressor compresses auxiliary medium vapours. In the reboiler, auxiliary medium vapours are condensed and liquefied medium is recycled back to the overhead condenser through an expansion valve, thus completing the cycle.

Fig. 8.67 *Distillation Column Heat Pump with Auxiliary Heat Transfer Medium*[56]
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Typical auxiliary heat transfer media are refrigerants. Water (R*-718), ammonia (R–717), R–134a, R–22, etc., can be used for the purpose. Among these, water (R–718) has many advantages over others. Water is chemically and thermally stable. No negative environmental effect in case of leakage, no special material of construction is required, cheap, high heat transfer co-efficients and high latent heat of vaporization are chief factors that favour its selection. Performance of heat pump with auxiliary medium is measured by Rankin coefficient of performance. Generally, COP of water is higher than that of other refrigerants.

Considering capital investment for the auxiliary heat–transfer medium recycle system and larger condenser and reboiler (as compared to conventional system), heat pump system with auxiliary medium is cost effective when reboiler duty is high (say > 2 MW). In this case also when close boiling components are to be separated, the heat pump is economical. Separation of *p*-xylene and ethyl benzene mixture and styrene and ethyl benzene mixture can be good examples of the system. For a 225 000 t/a styrene monomer plant, COP of heat pump with water as auxiliary medium could be in excess of 5.5.

Example 8.23 A distillation column is to be designed for aromatics separation. Material balance around the column is fixed and compositions of various streams are given below.

Table 8.61 Composition of Distillation Column Streams

Component	Composition, mole %		
	Feed	Distillate	Bottoms
Benzene	2.2	22.8	0.
Toluene	7.4	72.2	0.5
Ethyl benzene	43.4	5.0	47.5
Styrene	47.0	0	52.0

Feed flow rate is 70 kmol/h as saturated liquid. Column must be operated under vacuum to avoid thermal cracking and polymerisation.

It is planned to use a heat pump (similar to one shown in Fig. 8.67) for the system. Refrigerant R–134a (1,1,1,2–tetrafluoroethane) is to be used as an auxiliary heat transfer medium. Bubble point of the distillate will be 12°C in the overhead condenser and accordingly operating pressure of the condenser will be fixed.

Determine the required number of theoretical stages for the desired separation. Also find the location of the feed stage. Based on number of theoretical stages, fix the height of packed bed, packed with fibreglass reinforced plastic (FRP) Pall rings of 25 mm size. Assume pressure drop in the column to be 0.04 atm and calculate the operating pressure of the reboiler. In the reboiler, compressed R–134a vapour will be condensed.

*ASHRAE standard designation of refrigerant.

Assume approach of 10°C in the condenser and the reboiler for effective heat transfer. Calculate heat duties of the condenser and reboiler and the circulation rate of the refrigerant. Consider ideal behaviour of all liquid and gaseous mixtures.

Solution

Bubble point of distillate: 12°C

Considering ideal VLE,

$$\Sigma x_i\, p_{vi} = p_t \tag{A}$$

$$x_{DB} = 0.228,\ x_{DT} = 0.722 \text{ and } x_{DEB} = 0.05$$

Table 8.62 Antoine Constants[14]

Component	Antoine Constants		
	A	***B***	***C***
Benzene	14.1603	2948.78	–44.5633
Toluene	14.2515	3242.38	–47.1806
Ethyl benzene	6.0821	1424.3	–59.95
Styrene	14.3284	3516.43	–56.1529

$$\ln p_v = \mathrm{A} - \frac{B}{(T + C)} \text{ for benzene, toluene and styrene} \tag{B}$$

and

$$\log p_v = \mathrm{A} - \frac{B}{(T + C)} \text{ for ethyl benzene} \tag{C}$$

where, p_v is in kPa and T is in K.

At $T = 12 + 273 = 285$ K,

$p_{vB} = 6.659$ kPa, $p_{vT} = 1.8547$ kPa and $p_{vEB} = 0.567$ kPa

Operating pressure of the condenser,

$$p_t = 0.228 \times 6.659 + 0.722 \times 1.8547 + 0.05 \times 0.567 = 2.8857 \text{ kPa}$$

Pressure drop in the column = 0.04 atm = 4.053 kPa

Operating pressure at the bottom of the column, $p_t' = 2.8857 + 4.053 = 6.9387$ kPa

This pressure (p_t') will fix the bubble point of the liquid in the reboiler.

$$\Sigma x_{wi}\, p_{vi} = p_t' \tag{D}$$

By trial and error, bubble point is to be fixed to satisfy Eq. (D).

At $t_B = 62.5$°C, $T_B = 335.5$ K

$p_{vB} = 55.9708$ kPa,

$p_{vT} = 20.2005$ kPa, $p_{vEB} = 8.1878$ kPa and $p_{vS} = 5.7$ kPa

$$\Sigma x_{Wi}\, p_{vi} = 0.005 \times 20.2005 + 0.475 \times 8.1878 + 0.52 \times 5.7$$

$$= 6.954, \text{ close enough to } 6.9387 \text{ kPa.}$$

In this example, toluene is the light key component and ethyl benzene is the heavy key component.

Table 8.63 Relative Volatilities

Component	$\alpha_{top} = \dfrac{p_{vi}}{p_{vhk}}$	$\alpha_{bottom} = \dfrac{p_{vi}}{p_{vhk}}$	$\alpha_{av} = (a_{top} \cdot a_{bottom})^{0.5}$
Benzene	11.744	6.836	8.96
Toluene (LK)	3.271	2.467	2.8407
Ethyl benzene (HK)	1	1	1
Styrene	0.625	0.6962	0.6596

FUG method

Minimum number of theoretical stages can be found, using Fenskey's equation:

$$N_m = \frac{\log\left[\left(\dfrac{x_{LK}}{x_{HK}}\right)_D \left(\dfrac{x_{HK}}{x_{LK}}\right)_B\right]}{\log \alpha_{LK}} \tag{8.26}$$

$$N_m = \frac{\log\left[\left(\dfrac{0.722}{0.05}\right)\left(\dfrac{0.475}{0.005}\right)\right]}{\log\,(2.8407)} = 6.919, \quad \text{say } 7$$

Underwood method

$$\sum \frac{\alpha_i x_{if}}{\alpha_i - \vartheta} = 1 - q \tag{8.15}$$

$$\frac{8.96 \times 0.022}{8.96 - \vartheta} + \frac{2.8407 \times 0.074}{2.8407 - \vartheta} + \frac{1 \times 0.434}{1 - \vartheta} + \frac{0.6596 \times 0.47}{0.6596 - \vartheta} = 0$$

Solving by Mathcad,

$\vartheta = 2.4052$

Also $\vartheta \in \{1, \ldots, 2.8407\}$

$\vartheta = R_m + 1$

$$\sum \frac{\alpha_i x_{iD}}{\alpha_i - \vartheta} = R_m + 1 \tag{8.14}$$

$$\frac{8.96 \times 0.228}{8.96 - \vartheta} + \frac{2.8407 \times 0.722}{2.8407 - \vartheta} + \frac{1 \times 0.05}{1 - \vartheta} = R_{\mathrm{m}} + 1$$

For $\vartheta = 2.4052, \quad R_m = 3.9856$

Gilliland correlation

$$f(N) = \frac{N - N_m}{N + 1} = 1 - \exp.\left[\left(\frac{1 + 54.4\,\psi}{11 + 117.2\,\psi}\right) \times \left(\frac{\psi - 1}{\psi^{0.5}}\right)\right] \tag{8.28}$$

where, $$\psi = \frac{R - R_m}{R + 1} \tag{8.29}$$

Table 8.64 Evaluation of Parameters

R	ψ	$f(N)$	N
4.5	0.093 53	0.5603	17
6	0.287 8	0.39	12
7	0.376 8	0.3288	10.76
8	0.446	0.2819	10
9	0.501 44	0.2483	9.53

Referring to Table 8.64, $R = 8$ is considered optimum and required number of theoretical stages are 10.

Feed tray location can be determined by Kirkbride equation,

$$\log\left(\frac{N_r}{N_s}\right) = 0.206 \log\left[\frac{W}{D}\left(\frac{x_{HK,F}}{x_{LK,F}}\right)\left(\frac{x_{LK,B}}{x_{HK,D}}\right)^2\right] \tag{8.30}$$

For determining W and D, material balance calculations are to be carried out.

Overall material balance:

$$F = D + W$$

$$70 = D + W \tag{E}$$

Benzene balance:

$$0.022 \times F = 0.228 \times D + 0 \times W$$

$$0.022\, F = 0.228\, D \tag{F}$$

Solving Eqs. (E) and (F),

$$D = 6.7544 \text{ kmol/h and } W = 63.2456 \text{ kmol/h}$$

$$\log\left(\frac{N_r}{N_s}\right) = 0.206 \log\left[\frac{63.2456}{6.7544} \times \frac{0.434}{0.074} \times \left(\frac{0.005}{0.05}\right)^2\right]$$

$$\frac{N_r}{N_s} = 0.8839$$

$$N_r + N_s = 10$$

$$N_s = 5.308. \quad \text{Let } N_s = 6$$

Therefore, sixth stage from bottom will be the feed stage. Take HETP = 0.5 m for FRP Pall rings. Thus total packed bed height works out to 5 m.

Pressure drop per stage is calculated to be 0.405 kPa which is reasonable.

Heat duty of condenser, ϕ_C

$$\phi_C = (R + 1)\, D\, \lambda_{av}$$

Using Watson equation, latent heats of vaporisation for the components of distillate are calculated as under at 12°C.

$$\lambda_B = 34\,252.6 \text{ kJ/kmol}, \quad \lambda_T = 38\,452.8 \text{ kJ/kmol and}$$

$$\lambda_{EB} = 42\,497.0 \text{ kJ/kmol}$$

$$\lambda_{av} = \Sigma\lambda_i \cdot y_i = 0.228 \times 34\,252.6 + 0.722 \times 38\,452.8 + 0.05 \times 42\,497.0$$
$$= 37\,697.3 \text{ kJ/kmol distillate}$$
$$\phi_C = (8 + 1) \times 6.7544 \times 37\,697.3 = 2291\,603.8 \text{ kJ/h} \equiv 636.56 \text{ kW}$$

Let $t = 12°\text{C}$ be the reference temperature

$$H_D = 0, \quad H_D \cdot D = 0$$

Refer to Table 6.29 of Chapter 6.

At $T = 335.5$ K

$$H_W = -17.712\,(335.5 - 285) + 1054.193 \times 10^{-3}\left(\frac{335.5^2 - 285^2}{2}\right)$$
$$- 1838.075 \times 10^{-6}\left(\frac{335.5^3 - 285^3}{3}\right)$$
$$+ 1694.332 \times 10^{-9}\left(\frac{335.5^4 - 285^4}{4}\right)$$
$$= 9240 \text{ kJ/kmol mixture}$$
$$\phi_W = H_W \cdot W = 9240 \times 63.2456 = 584\,389.3 \text{ kJ/h} \equiv 162.33 \text{ kW}$$

Feed is saturated and hence enthalpy of feed at its bubble point is to be calculated. At bubble point,

$$\sum \frac{x_i p_{vi}}{p_t''} = \Sigma y_i = 1$$

Since feed is nearly in the middle of the column, assume average pressure at the feed point

$$p_t'' = \frac{2.8857 + 6.9387}{2} = 4.9122 \text{ kPa}$$
$$0.022\, p_{vB} + 0.074\, p_{vT} + 0.434\, p_{vEB} + 0.47\, p_{vS} = 4.9122$$

This equation can be solved by trial-and-error calculations. At $t = 49°\text{C}$ (322 K),

$$p_{vB} = 34.18, \quad p_{vT} = 11.6264, \quad p_{vEB} = 4.435, \quad p_{vS} = 3, \text{ all in kPa}$$
$$\Sigma x_i\, p_{vi} = 4.947, \text{ close enough to } 4.9122 \text{ kPa}$$

Refer to Table 6.30 of Chapter 6.

$$H_F = -16.023\,(322 - 285) + 1030.422 \times 10^{-3}\left(\frac{322^2 - 285^2}{2}\right)$$
$$- 1809.476 \times 10^{-6}\left(\frac{322^3 - 285^3}{3}\right)$$
$$+ 1693.124 \times 10^{-9}\left(\frac{322^4 - 285^4}{4}\right)$$
$$= 6561.5 \text{ kJ/kmol feed}$$
$$\phi_F = H_F \cdot F = 6561.5 \times 70 = 459\,305 \text{ kJ/h} \equiv 127.6 \text{ kW}$$

Assume 5% heat loss to surrounding and no heat of mixing.

$$\phi_B = (\phi_C + H_D \cdot D + H_W \cdot W - H_F \cdot F)\ 1.05$$
$$= (636.56 + 0 + 162.33 - 127.6)\ 1.05$$
$$= 704.85 \text{ kW} \equiv 2537\ 460 \text{ kJ/h}$$

Approach for heat transfer in the overhead condenser and reboiler is 10°C. Since distillate bubble point is 12°C, auxiliary heat transfer medium (R–134a) should evaporate at 2°C. Similarly in reboiler, boiling takes place at 62.5°C and hence R–134a vapours are to be condensed at 72.5°C.

Table 8.65 Properties of R–134a[27]

Temperature °C	Pressure, bar a	Auxiliary heat transfer medium enthalpy, kJ/kg			Vapour, C_p/C_v
		Liquid	Latent	Vapour	
2.0	3.150 (satd.)	202.68	197.07	399.75	1.182
72.5	22.385 (satd.)	308.57	120.42	428.99	1.653
130.0	23.0 (sup.)	—	—	500.00	—

(Reference state: Enthalpy of saturated R–134a liquid at 0°C = 200 kJ/kg)

In the condenser, R–134a liquid from the reboiler (at 72.5°C) will be flashed to 2°C and vapours will be produced.

Heat, taken up in condenser,

$$H_1 = 399.75 - 308.57 = 91 \text{ kJ/kg}$$

$$\text{Evaporation rate of R–134a, } \dot{m}_R = \frac{\phi_C}{H_1}$$
$$= 2291\ 603.8/91 = 25\ 182.5 \text{ kg/h}$$

These vapours will be superheated by 10°C before taking to the compressor to avoid any liquid carryover. This can be done by exchanging heat with liquid from reboiler. However, for the present, this difference is neglected. Saturated vapour at 3.15 bar a will be compressed to 23 bar a.

Overall compression ratio = 23/3.15 = 7.302

Since this is high, it will be compressed in a two-stage reciprocating compressor.

Compression ratio in each stage = $\sqrt{7.302}$ = 2.702

Vapour will be compressed from 3.15 bar a to 8.511 bar a in the first stage, cooled suitably and compressed to 23.0 bar a in the second stage. Its temperature is expected to be 130°C.

$$\text{Heat, given up in the reboiler, } H_2 = 500 - 308.57$$
$$= 191.43 \text{ kJ/kg}$$

$$\text{Heat available, } \phi'_B = 25\ 182.5 \times 191.43 = 4820\ 686 \text{ kJ/h} \equiv 1339.1 \text{ kW}$$

Since ϕ'_B is much higher (by 90%) than ϕ_B, a trim condenser will be needed to take care of the excess heat load. This heat (low level) can be utilised to heat the feed or some other stream in the plant. In addition, during start-up, overhead condenser will not have any heat-load but reboiler will have to be commissioned. Therefore, an additional auxiliary reboiler will be needed with some other heat source, say with low

pressure steam for the start-up. It can be seen that adoption of heat pump system will thus call for a number of additional equipments.

Notes:

(i) Since required number of equipments are many more than for the conventional distillation system, initial investment will be substantially high.
(ii) Operating pressure of the reboiler shell will be 23 bar a. This will call for thick shell, calling for higher investment.
(iii) Adequate instrumentation and controls (I&C) will be needed for the refrigerant circulation system in addition to the I&C for distillation system, calling for additional investment.
(iv) Cost of Refrigerant R–134a is much higher than conventional utilities. Enough care is required to prevent its leakage from the circulation system.

Fixed and variable costs for driving the compressor and extra cost of the heat pump system should be carefully weighed against savings in normal utilities, used in the conventional distillation system. Also skilled personnel will be required for operating the integrated system.

Heat pumps save energy in distillation but at a definite cost. Careful economic evaluation is therefore necessary before its implementation.

8.9.2 Efficient Operation of Distillation Columns

Existing distillation columns can be operated in such a manner that substantial energy can be saved.

1. Many distillation columns are operated to excessively purify the products which might call for high reflux ratio and hence high reboiler duty. This should be avoided by having adequate I&C. Refer to example given in para 4 of Sec. 8.9.1.
2. Damaged or worn column internals can be costly. Over a period of operation, the column internals may get damaged. For example, in bubble cap columns, top nut may loosen and cap may open up. In floating valve trays, valves may fly off, in packed columns ceramic packings may crumble, scaling on trays may take place over a period of operation, etc., Due to similar reasons, the existing columns should be opened for inspection periodically and preventive maintenance should be carried out. Such preventive maintenance practice will pay rich dividends in terms of energy saving in operation.
3. Overhead condenser needs periodic cleaning. This will ensure condensation of overhead vapour at the right pressure and loss, if any, of vapour can be avoided.

8.9.3 Replace the Distillation Partially or Completely by New Separation Technique

Replacment of distillation by supercritical extraction or by reactive distillation can result in significant energy savings. The same is discussed in Secs. 7.7 and 8.7 of this text. Similarly, energy savings can be achieved by replacing distillation partially or completely by new separation techniques like membrane separation techniques (reverse

osmosis, pervaporation, membrane distillation, membrane reactor, etc.), molecular sieve adsorption, crystallisation, fixed adsorptive distillation, etc.

Examples: 1. Conventionally absolute alcohol (ethanol) is separated from azeotropic mixture of ethanol-water (95.6% ethanol, 4.4% water by mass) by azeotropic distillation using benzene or cyclohexane as an entrainer. This conventional method uses three distillation columns and requires very high energy. One of the new methods for separating the same mixture is by Pressure Swing Adsorption (PSA) which utilise 3 Å molecular sieves[51]. Molecular sieve adsorption consumes less energy and eliminates the use of the entrainer. Benzene is a proven carcinogen and therefore an air pollutant. For the production of 3400 kg/h of fuel–grade ethanol, installation of molecular sieve—pressure swing adsorption system instead of azeotropic distillation unit allows a reduction in energy consumption of boiler from 2257 to 147 kW. But this new technique requires additional electrical energy in the amount of about 100 kW to drive the vacuum pumps and other equipments for the PSA unit. The use of benzene or cyclohexane as entrainer is eliminated.

For the separation of same azeotropic mixture, pervaporation can also be used. For the production of fuel grade ethanol with the same rate; 3400 kg/h, installation of pervaporation allows a reduction in energy consumption of boiler from 2257 to 264 kW. But fresh electrical energy, equivalent to 150 kW is required to drive the vacuum pumps and other equipments for the pervaporation unit.

2. *Iso*-propyl–alcohol (IPA) is yet another application for which pervaporation can be used. IPA forms an azeotrope with water having composition of 87.9% (by mass) IPA. Dehydration of IPA azeotrope with zeolite membrane is commercially carried out.

Table 8.65 Comparison of Pervaporation and Adsorption with Distillation

Parameter	Distillation	Pervaporation	Adsorption
Capacity, m^3/d	3 to 30	0.5 to 30	10 to 30
Feed water content, %	5–95%	0.5 to 30	0.05 to 5
Product water content, mg/L	< 2000	< 1000	< 100
Operating cost	High	Low	Medium
Capital cost	Low	Medium	Medium
Floor space requirement	Large	Small	Large
Safety	Medium	High	Medium
Environment friendliness	Low	Excellent	Medium
Ease of installation	Medium	Easy	Medium
Operation	Skilled	Easy	Skilled
Turndown capability	Medium	Excellent	Poor
Start-up and shut-down time	Long	Short	Long
Maintenance requirements	High	Low	High
Availability/Uptime	Medium	High	Medium
Flexibility	Limited	Excellent	Medium

3. *Iso*-propyl alcohol (IPA) can be concentrated nearly pure IPA by dixed adsorptive distillation system[40].

4. Freeze crystallization is yet another technique which can be used for recovery of organic species, such as acetic acid, acrylic acid, etc., from very dilute solutions with 75 to 90% energy reduction as compared to conventional distillation. Heist[57] has given a process flow sheet for 95% recovery of acetic acid from 1% aqueous solution (by mass) by freeze crystallization. Normally, 1% solution could be considered as effluent with high non-biodegradable COD and its disposal will be a serious threat to the industry. Recovery will turn effluent disposal problem into an economical proposal by adoption of freeze crystallization.

EXERCISES

8.1 Fill the gaps in the following crossword puzzle:

Fig. 8.68 *Crossword Puzzle*[58]
(Reproduced with the Permission of Dr. Ming Tham of School of Chemical Engineering and Advanced Materials, University of Newcastle upon Tyne, UK)

KEYS:
Down:
1. A conduit that directs liquid from one tray to another.
4. Liquid carried by vapour up to the tray above.

6. This is determined by the amount of material going through the column.
9. Brought about by excessive vapour flow.
14. An alternative to the use of trays or plates.
15. Made-up of the two components.

Across:

2. A liquid mixture which when vaporised produces the same composition as the liquid.
3. A type of tray used in distillation/absorption.
5. An equipment used to cool vapours, coming off the top of the column.
7. Distillation operations require lots of this.
8. Operation performed by the top section of the column.
10. Product stream taken off the top of the column.
11. Liquid that is fed back to the top of the column.
12. An equipment used to raise vapour.
13. A mixture that is to be separated.
16. Expansion of liquid due to vapour or gas.
17. Operation performed by the bottom section of the column.
18. Ensures that there is sufficient liquid on a distillation tray.
19. Caused by low vapour flow.
20. Product stream taken off bottom of the column.

8.2 Refer to Examples 8.5 and 8.13. Considering the mixture to be an ideal one, find separate average volatilities for rectification section (α_{1avg}) and for stripping section (α_{2avg}) for the mixtures. Using these average volatility values, reflux ratio (R) as 4.2, same feed conditions, calculate number of theoretical tray requirement for achieving $x_W = 0.02$ by tray to tray calculations. Specify the feed tray position.

Hint: Use Eqs. (8.17) and (8.18) for evaluating liquid and vapour compositions on trays of both sections, considering $y_D = 0.9302$ and $x_W = 0.02$.

8.3 Distillation column is used to separate aqueous solution of dimethyl formamide (DMF). Determine the following based on given data:

(a) Minimum reflux ratio.
(b) Number of theoretical stages required for desired separation for $R = 2\ R_m$.
(c) Overhead condenser duty.
(d) Reboiler duty.

Data:

(i) Feed composition: 20% by mass DMF, Molar mass of DMF = 73.1 kg/kmol Feed is saturated liquid. Feed flow rate = 100 kmol/h
(ii) Distillate composition: 99% water (by mass).
(iii) Residue composition: 99% DMF (by mass).
(iv) Operating pressure at top: 650 Torr vacuum or 110 Torr a.
(v) For vapour pressure data refer Table 8.67.
(vi) Properties of DMF
Latent heat of vaporisation $\lambda = 576.85$ kJ/kg at the topmost temperature of distillation column
Specific heat $C_L = 2$ kJ/(kg · °C)

Table 8.67 Vapour Pressure Data of DMF and Water

Temperature °C	p_v of DMF Torr	p_v of water Torr
53.7	18	110
60	26	149.38
70	42	233.7
80	65	355.1
90	100	525.76
92	110	567
100	165	760

8.4 NRTL parameters for dimethyl formamide (1) and water (2) are as follows:

$$\tau_{12} = b_{12}/RT, \quad \tau_{21} = b_{21}/RT, \quad R = 8.314 \text{ kJ/(kmol}\cdot\text{K)}$$

$$b_{12} = -267.667, \quad b_{21} = 470.31$$

$$\ln \gamma_1 = x_2^2\left[\tau_{21}\left(\frac{G_{21}}{x_1 + x_2 G_{21}}\right)^2 + \frac{G_{12}\tau_{12}}{(x_2 + x_1 G_{12})^2}\right] \tag{8.127}$$

$$\text{In } \gamma_2 = x_1^2\left[\tau_{12}\left(\frac{G_{12}}{x_2 + x_1 G_{12}}\right)^2 + \frac{G_{21}\tau_{21}}{(x_1 + x_2 G_{21})^2}\right] \tag{8.128}$$

where, $G_{12} = \exp(-\alpha\, \tau_{12})$, $G_{21} = \exp(-\alpha\, \tau_{21})$

$\alpha = 0.277$

Solve Exercise 8.3 considering non-ideality of DMF-water system. To find the VLE data following equation can be used:

$$y = \frac{\gamma\, p_v}{p_t} x \tag{8.3}$$

y, x = Mole fraction of more volatile component (water) in vapour and liquid phase, respectively.

γ = Activity coefficient of water in solution.

p_t = Operating pressure = 110 Torr a

8.5 In the production of chloromethanes, a saturated vapour mixture consisting of 60% methyl chloride, 28% methylene dichloride, 9% chloroform and 3% carbon tetrachloride (by mass), is sent to distillation section for the separation of pure products. In first distillation column 99.9% (by mass) pure methyl chloride is separated as top product. Residue contains 0.2 kmol/h methyl chloride. Determine the following for this distillation column:

(a) Operating pressure of distillation column.
(b) Minimum reflux ratio by Underwood's method.
(c) For $R = 3\, R_m$ calculate the number of theoretical stages required for desired separation.
(d) Determine the tower diameter of distillation column, selecting sieve tray tower for the same.

Feed is a saturated vapour mixture and its flow rate is 5000 kg/h.

8.6 Refer to Example 8.16. Consider that total distillate at the end of distillation should contain 99% by mole (min.) methanol. Considering a constant reflux ratio (R) of 4,

same feed conditions and 6 hours of distillation period, calculate the quantities of distillate and residue with their compositions. Make calculations for $N = 2$ and 10.

8.7 Determine the minimum reflux ratio and number of theoretical stages required for reflux ratio, $R = 2\ R_m$, for the following given separation by

(a) Ponchon-Saverit method based on given H-x-y data and equilibrium x vs y data, given in Table 8.68.

(b) McCabe–Thiele method using phase equilibrium data x vs y, given in Table 8.68.

(c) McCabe–Thiele method by using vapour–liquid equilibrium data x vs y, which can be derived by using modified Raoult's law.

$$y_i\, p_t = \gamma_i\, p_{vi}\, x_i \tag{8.3}$$

where, γ_i = Activity coefficient can be determined by van Laar equations. van Laar constants $A = 1.978$, $B = 1.401$.

In Table 8.68, "1" stands for ethanol and "2" for benzene.

Data:

(i) Feed: 100 kmol/h contains 10% ethanol and 90% benzene (by mole) Feed is saturated liquid.

(ii) Distillate: It contains 44% ethanol (by mole).

(iii) Residue: 99.9% benzene (by mole)

Table 8.68 VLE Data and Enthalpy Data for Ethanol-Benzene System at 760 Torr

Mole fraction of ethanol		Temperature	Enthalpy of mixture, kJ/kmol	
x_1	y_1^*	t, °C	H_L	H_G
0	0	79.7	–15 870	19 090
0.01	0.075	78.0	–15 910	18 380
0.023	0.150	76.0	–16 120	17 580
0.045	0.217	74.0	–16 410	17 170
0.095	0.305	71.0	–17 170	16 540
0.200	0.395	69.0	–18 630	15 910
0.300	0.425	68.4	–20 050	15 740
0.447	0.447	68.2	–22 150	15 570

(Reference states: Enthalpies of ethanol and benzene in ideal gas state at 0 K = 0 kJ/kmol)

8.8 1000 kg of feed, containing 30% by mass ethylene glycol and 70% by mass water, is to be separated in batch distillation with rectification column. Bottom product must contain 98.8% by mass of ethylene glycol. Composition of distillate should remain constant throughout the batch distillation. Desired composition of distillate is 98% by mass water.

(a) Design the rectification column.

(b) Determine the operating (actual) reflux ratio vs time data.

(c) Compare the resulting data of this example (batch distillation with rectification with the resulting data of Example 8.14 (simple batch distillation).

Operating pressure in rectification column is 30.4 kPa a (maximum). Vapour liquid equilibrium data for ethylene glycol-water system at 30.4 kPa a are given in Example 8.14.

8.9 Refer to Exercise 10.5 relating to chlorination of benzene. At the end of reaction, the product mixture is expected to be 10.63 kmol and will have 13% benzene, 71.3% monochlorobenzene and 15.7% dischlorobenzene. It is desired to separate 99% pure benzene as distillate by batch distillation with rectification. Final residue will contain 1% benzene. Total time considered for distillation is 4 h. Determine the reflux ratio (R) vs time data (θ). All concentrations are mole %

Table 8.69 Antoine Equation Constants

Component	Antoine Constants			Normal boiling point at 101.325 kPa, °C
	A	*B*	*C*	
Benzene	4.725 83	1660.652	–1.641	80.15
Monochlorobenzene	4.110.83	1435.675	–55.124	131.75
Dichlorobenzene	4.122 90	1575.110	–64.637	174.06

Antoine equation: $\log p_v = A - \dfrac{B}{(T + C)}$

where, p_v = Vapour pressure, bar

T = Saturation temperature, K

8.10 Chloroform and methanol form a minimum boiling azeotrope at 101.325 kPa and 53.5°C containing 65.17 mole % chloroform. Using van Laar equations, generate *t-x-y* relationship and plot *y* vs. *x* diagram.

Table 8.70 Antoine Equation Constants[14]

Component	Antoine constants			Normal boiling point at 101.325 kPa, °C
	A	*B*	*C*	
Chloroform	14.5014	2938.55	–36.9972	61.15
Methanol	16.4948	3593.39	–35.2249	64.65

Antoine equation: $\ln p_v = A - \dfrac{B}{(T + C)}$

where, p_v = Vapour pressure, kPa

T = Saturation temperature, K

8.11 Formic acid and water form a maximum boiling azeotrope at 101.325 kPa and 107.1°C, containing 43.3 mole % water. Using van Laar equations generate *t-x-y* relationship and plot *y* vs. *x* diagram.

Table 8.71 Antoine Equation Constants[14]

Component	Antoine Constants			Normal boiling point at 101.325 kPa, °C
	A	*B*	*C*	
Formic acid	6.7067	1699.20	–12.45	100.8
Water	7.0436	1636.90	–48.25	100.0

Antoine equation: $\log p_v = A - \dfrac{B}{(T + C)}$

where, p_v = Vapour pressure, kPa
T = Saturation temperature, K

8.12 In Example 8.23, refrigerant R–134a was selected. Consider use of refrigerant R–22 (chlorodifluoromethane) **or** refrigerant R–717 (ammonia) as alternate auxiliary heat transfer mediums. Properties of R–22 and R–717 are given below.

Table 8.72 Properties of R–22[27]

Temperature °C	Pressure, bar a		Enthalpy, kJ/kg			Vapour C_p/C_v
			Liquid	Latent	Vapour	
2.0	5.315	(satd.)	202.36	202.65	405.01	1.296
72.5	31.51	(satd.)	296.44	118.15	414.59	2.147
130.0	32.0	—	—	—	478.7	—

(Reference state: Enthalpy of saturated liquid at 0°C = 200 kJ/kg)

Assume 10°C approach in the overhead condenser and reboiler for R–22.

Table 8.73 Properties of R–717[27]

Temperature °C	Pressure, bar a		Enthalpy, kJ/kg			Vapour C_p/C_v
			Liquid	Latent	Vapour	
7.0	5.54	(satd.)	232.55	1236.31	1468.86	1.415
72.5	35.06	(satd.)	558.85	923.10	1481.95	1.834
130.0	35.50	—	—	—	1692.34	—

(Reference state: Enthalpy of saturated ammonia liquid at 0°C = 200 kJ/kg)

Ammonia evaporator is normally a flooded one. Therefore, assume approach of 5°C in the overhead condenser and approach of 10°C in the reboiler.

Calculate required circulation rates of R–22 and R–717 for the same heat duties. Also review the performances of all three auxiliary heat transfer mediums and critically evaluate system requirements, including environment friendliness.

8.13 Extractive distillation of 50 mass % aqueous *iso*propyl alcohol (IPA) is to be carried out with the help of aqueous 35 mass % $CaCl_2$ solution. Vapour pressure data of aqueous calcium chloride solution are given in Table 8.58. Calculate *t-x-y* data for the system and plot the equilibrium (x, y) curve.

8.14 In a refinery, a gas stream, having composition 6.84% ethane, 40.81% propane, 9.70% *i*-butane and 42.65% *n*-butane (by mole), is to be fractionated at a rate of 300 kmol/h in a column to separate LPG fraction[59]. Feed gas is compressed, cooled and flashed in the fractionating column at 9.7 bar a and 45°C. Overhead vapours from the column are taken to partial condenser from which C_2 and C_3-fractions are separated in gaseous form. Composition of the gaseous stream from the condenser is 28.25% ethane, 68.55% propane, 1.6% *i*-butane and 1.6% *n*-butane (by mole). Flow rate of overhead vapour is 68.52 kmol/h. It leaves the condenser at 9.4 bar a and 20°C. Saturated liquid from the condenser at 20°C is recycled as reflux to the column at the rate of 1.5 kmol/kmol vapours separated. Pressure drop in the overhead

condenser may be considered negligible (< 10 kPa) for calculation purpose. Bottom residue (LPG fraction) is removed from the reboiler at 10 bar a. Use Fig. 6.20 for dew point and bubble point calculations.

(a) Make complete material balance calculations of the column.
(b) Determine minimum reflux ratio.
(c) Determine number of theoretical stages for the reflux ratio 1.5 kmol/kmol and the stage at which the feed should be introduced by FUG method.
(d) In Tables 8.74 and 8.75, enthalpies of liquid and gaseous hydrocarbons are given. Using these values, calculate the heat duties of the overhead condenser and the reboiler.
(e) Considering low temperature of the vapours (20°C), leaving the overhead condenser, use of ammonia (R-717) is proposed as an auxiliary heat transfer medium in the overhead condenser and the reboiler. Evaporation of ammonia at 15°C in the overhead condenser and condensation of ammonia at 65°C in the reboiler are fixed as design parameters. Consider conditions of ammonia at the compressor discharge as 30 bar a and 125°C. Properties of ammonia are given in Table 8.76. Calculate ammonia circulation rates, required in the overhead condenser and the reboiler. Comment on the results.

Table 8.74 Enthalpy of Liquid Hydrocarbons at Saturated Conditions[60] (in kJ/kmol)

Temperature, K	Ethane	Propane	*i*-Butane	*n*-Butane
290	304	1918	2521	2525
300	1840	3112	3939	3941
305.34[c]	4154			
310	—	4353	5397	5391
320	—	5650	6896	6878
330	—	7015	8442	8404

[c]Critical temperature of ethane
Reference state: Enthalpy of saturated liquid hydrocarbon at 0°C = 0 kJ/kmol

Table 8.75 Enthalpy of Gaseous Hydrocarbons[60] (in kJ/kmol)

Hydrocarbon	Temp., K	Pressure, bar a		
Ethane		**1**	**2**	**3**
	280	11 700	11 630	11 560
	300	12 730	12 680	12 620
	320	13 820	13 770	13 720
	330	14 380	14 330	14 280
Propane		**5**	**6**	**8**
	290	17 785	17 615	—
	300	18 585	18 425	18 105
	320	20 205	20 085	19 825
	340	21 885	21 785	21 565

(*Contd.*)

Table 8.75 (*Contd.*)

i-Butane		0.1	0.5	1.0
	280	21 935	21 835	21 695
	300	23 825	23 745	23 635
	320	25 825	25 755	25 665
	340	27 935	27 875	27 805

n-Butane		0.1	0.5	1.0	2.0	3.0
	280	23 545	23 425	22 845	—	—
	300	25 475	25 385	25 255	24 505	—
	305	—	—	—	—	25 245
	320	27 515	27 435	27 325	27 105	26 875
	340	29 655	29 575	29 485	29 305	29 105

(Reference state: Enthalpy of saturated liquid hydrocarbon at 0°C = 0 kJ/kmol)

Table 8.76 Properties of Ammonia (R-717)[26]

Temperature, °C	Pressure bar *a*	Condition	Enthalpy, kJ/kg		C_p/C_v
			Liquid	Vapour	
15	7.286	Saturated	270.2	1475.9	1.438
65	29.476	Saturated	518.6	1487.1	1.748
125	30.0	Superheated	—	1695.0	—

(Reference state: Enthalpy of saturated liquid ammonia at 0°C = 200 kJ/kg)

8.15 Tetrahydrofuran (also known as 1, 4-epoxy butane; formula: C_4H_8O) is widely used as a solvent and also as a reaction medium in chemical industry. Typical THF recovery method involves adsorption on activated carbon, followed by steam stripping of the bed. Resulting THF-water mixture is a dilute aqueous stream of THF which is required to be separated in pure components for reuse of THF.

THF forms a minimum boiling azeotrope at 63.5°C, containing 5.3% by mass of water at atmospheric pressure. For the separation of THF from the mixture, pressure swing distillation process[61] is used. Basic flow sheet of the process is shown in Fig. 8.69.

In the first column, THF-water mixture, containing 0.06 mole fraction THF is fed. Distillate D_1 from the top has near azeotropic composition (0.805 mole fraction THF). Reflux ratio of 0.26 kmol/kmol distillate is maintained in this column. Bottom product of the first column is pure water (in which THF content is 1×10^{-6} mole fraction).

Distillate D_1 is fed to the second column, operating at 7.908 bar a. At this pressure, THF-water mixture forms a minimum boiling azeotrope at 136°C, containing 12% by mass water. Distillate D_2 from the top has near azeotropic composition (0.65 mole fraction THF) and is recycled to the first column. Bottom product from the second column is pure THF (in which water content is 1×10^{-5} mole fraction). Reflux ratio in the second column is maintained at 0.65 kmol/kmol distillate.

Fig. 8.69 *Pressure Swing Distillation of THF - Water*

Note: All the concentrations are expressed as mole fractions.

Assume that van Laar correlations represent non-ideality of THF-water mixtures in both the columns. Carry out tray-to-tray calculations for both the columns and fix feed locations of F and D_1 for fresh feed rate of 1000 kmol/h.

Table 8.77 Properties of THF and Water

	THF				Water	
Normal boiling point, °C	66				100	
Freezing point, °C	–108.5				0	
Density, kg/L	0.889				1.0	
Antoine constants	Temperature range, °C					
	60–150	**60–70**	**70–80**	**80–90**	**90–100**	**100–150**
A	6.995 15	7.1365	7.1095	7.0883	7.0733	7.0436
B	1202.29	1695.2	1678.9	1665.9	1656.4	1636.9
C	226.25	–42.75	–44.15	–45.35	–46.25	–48.25
Average latent heat of vaporisation, λ, kJ/kmol		27 901			41 453	

Antoine equations: $\log p_v = A - \dfrac{B}{(t + C)}$ for THF

$$\log p_v' = A - \frac{B}{(T + C)} \text{ for water}$$

where, p_v = Vapour pressure of THF, Torr
p_v' = Vapour pressure water, kPa
t = Saturation temperature, °C
T = Saturation temperature, K

8.16 A plant employs a batch reactor with refluxing arrangement (with cooling water) to manufacture butylated amino resins. One of the products is melamine-formaldehyde (MF) resin. Melamine, aqueous formaldehyde (37% by mass) and *n*-butanol are the chief reactants. Phosphoric acid (98% pure, 0.5% by mass) is used as a catalyst. In a typical batch of 4 kL, average consumptions for a specific grade of MF resin are reported to be 0.6 t *n*-butanol, 0.4 t melamine and 0.3 t formaldehyde (100%) per t MF (100% strength) product. Etherification and condensation reactions take place in the reactor at atmospheric pressure and in the temperature range of 90 to 105°C. Stoichiometry of above reaction mechanism reveals that one mole of water is produced per one mole of melamine condensed (reacted) and one mole of *n*-butanol reacted.

Product water and water of aqueous formaldehyde solution are removed from the reactor in the form of an azeotrope with *n*-butanol. Condensed azeotrope separates in two layers; compositions of both the layers depend on the temperature. Butanol rich layer is recycled and aqueous layer at 40°C is collected in a separate tank for recovery of *n*-butanol by distillation. In the batch, 100% excess *n*-butanol is used for completion of reaction. At the end of reaction, excess *n*-butanol is recovered by distillation. Batch is normally completed in 8 to 10 h.

Product contains up to 1.25% formaldehyde and 2 to 2.5% *n*-butanol (by mass). Since the reaction takes longer time, substantial crosslinking takes place which is more than desired.

Reaction chemistry:

Total mass of reactants = 3n × 126.12 + a × 30.03 + b × 74.12

Fig. 8.70 *Typical Polymer Chain*

Molar mass of polymer = 324.3n + 30.03a + 56.16b + 18.02
Total water Produced = (3n + b –1) 18.02

It is decided to adopt a continuous system for production of MF resin as the product demand has shot up. For this purpose, process research was carried out in the laboratory. Use of strong cation exchange resin (up to 4% by mass), suitable for continuous operation at 140°C, was made as a catalyst instead of phosphoric acid. Results of the 20-litre flask showed that while consumptions of the reactants were nearly same as that of batch process (for the same grade), better quality product was produced in 3 h. MF resin so produced had less crosslinking and low formaldehyde (< 0.5%) content. Further experiments with a glass column, packed with glass Raschig rings, confirmed that 3 theoretical stages for stripping are required for achieving bottom product (MF resin) with less than 1% *n*-butanol (by mass). Data on *n*-butanol-water azeotrope are covered in Sec. 8.8. Heterogeneous liquid from overhead condenser separates in two layers. Mutual solubility data are given in Table 8.78.

Table 8.78 Mutual Solubility of Water-n-Butanol mixture[62]

Temperature in separator, °C	mass % *n*-Butanol	
	Upper layer	Lower layer
20	79.93	7.81
30	79.38	7.08
40	78.51	6.60
50	77.58	6.46
60	76.38	6.52
80	73.53	6.89

Consider: (i) Excess *n*-butanol will be just sufficient to remove water in the form of the azeotrope, and (ii) Residence time of reaction mass in the distillation column in contact with the resin will be 3 h.

Based on above information, develop a process flow diagram for the reactive distillation system for continuous production of MF resin along with desired instrument and control system with technical reasons. Consider whether pilot plant studies are required to implement the project commercially. Also study the advantages and disadvantages/limitations of proposed reactive distillation system over the batch system.

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9 Process Design of Absorbers

9.1 INTRODUCTION

Absorption is an important unit operation of chemical process industries. In absorption, a gas mixture (or a gas-vapour mixture) is contacted with a suitable liquid, called solvent, to separate one or more components of the gas mixture by dissolving them in the liquid. Hence, after the absorption, components of gas mixture are transferred to the solution. Dissolved component in the solution can be separated by distillation or stripping (reverse of absorption). For example, in the manufacturing of synthesis gas for ammonia from the gas mixture containing nitrogen, hydrogen, carbon dioxide and traces of other components, carbon dioxide is removed or separated by absorption in ethanol amine solution. Then carbon dioxide from the resulting solution is separated by stripping. "Absorber" and "scrubber" are sometimes used as synonyms. Truly speaking, if a component of the gas mixture, transferred to liquid, is a gas at operating conditions of mass transfer, then it is called absorption. If a component transferred is vapour at operating conditions then it is called scrubbing. Below critical temperature, a gas is known as vapour (which could be saturated or superheated). For example, separation of acetone vapour from air. Acetone vapour mixture in contact with water as solvent is called scrubbing. Operating conditions of this scrubber is atmospheric pressure and ambient temperature. Critical temperature of acetone (T_c = 508.1 K) is well above the ambient temperature. Absorption or scrubbing is chiefly carried out in a packed tower, although plate (tray) columns are not uncommon. Various types of equipment used as absorbers or scrubbers are:

1. Packed tower
2. Tray tower
3. Falling film absorber
4. Spray chamber
5. Venturi scrubber
6. Stirred tank

9.2 CRITERIA OF SELECTION

9.2.1 Use of Packed Tower

Packed towers are used for all mass transfer operations. In Chapter 7, utility of a packed tower for extraction was discussed and clarified that in extraction columns the mass transfer area is almost independent of the packing surface, but is directly linked to the drop phase hold-up in the tower. Contrary to this flow path process, a packed tower provides surface for mass transfer for distillation and absorption operations. Both random and structured packings are used for the latter operations. However, internals are some what different than those for extraction service. For example, disperser and coalescer play important role in extraction but they are not needed in other two operations. Instead, a demister pad at the top to catch entrainment is an important internal for two operations. In majority of absorption applications, packed towers are preferred possibly for the following reasons:

1. Tower diameter required for the majority of the applications is less than 0.75 m. Packed tower is generally cheaper than tray tower when tower diameter is less than 0.75 m.
2. Many absorption or scrubbing systems are corrosive systems. For corrosive systems plastic packed towers (for example FRP packed towers) with plastic packing are cheaper than metallic tray towers. For corrosive systems, use of graphite heat exchangers is increasingly considered in which gas and solvent flow in co-current fashion.
3. In majority of absorption with chemical reaction systems, overall rate of gas-liquid reaction is totally controlled by rate of mass transfer (i.e., rate of chemical reaction $>>$ rate of mass transfer). Packed tower provides higher rate of mass transfer due to higher interfacial area of contact compared to other equipments.
4. For the liquids having high foaming tendency and also for a heat sensitive liquid, packed tower is a better choice than tray tower.
5. For removing obnoxious gases from the vent gases or from the flue gases by absorption, available pressure of feed gas, fed to absorber or scrubber, is usually very low; close to atmospheric pressure. Hence, for such a case, pressure drop in absorber is the main criteria for selecting the equipment. Packed tower provides less pressure drop compared to a tray tower. Hence, for such applications, packed towers are preferred.

9.2.2 Use of Spray Tower

For removing sulphur dioxide (SO_2) from boiler flue gases in large scale plants, available pressure of flue gases is very low; close to atmospheric pressure. For this case, even packed tower is not suitable because pressure drop offered by packed tower is normally greater than 100 mm WC. Normally for such an application spray tower or spray chamber is used. Boiler flue gases are also contaminated by fine carbon particles, if coal is used as a fuel. This favours the selection of spray tower over packed tower. Alternatively, a venturi scrubber can be used which induces vacuum in the suction.

9.2.3 Use of Tray Tower

Three different types of tray tower were discussed in Section 8.4.6. These tray towers can also be used for absorption service.

Tray tower is selected as absorber in the following cases:

1. In case of exothermic absorption, it can be improved by providing external cooling during absorption. Cooling coils can be more readily built into tray towers and liquid can readily be removed from the trays to be passed through coolers and returned, than from packed towers. Take, for example, absorption of acetaldehyde from acetaldehyde vapour-gas mixture by using ethanol-water solution as solvent. This absorption is exothermic and hence tray tower with intermediate coolers is used as absorber. Similarly, absorption of formaldehyde in water is exothermic which can be conveniently carried out in a plate column. Refer to Example 9.6 and Figs. 4.2 and 4.8. Absorption of carbon dioxide in ammonia plant is preferably carried out in a tray column which is exothermic in nature, although packed columns are also in use.
2. For the absorption systems involving low liquid flow rate, tray towers are preferred to get the better gas-liquid contacting efficiency. Low liquid rate may lead to incomplete wetting of column packing in case of packed tower and can give lower contacting efficiency.
3. If required tower diameter is large, say more than 1.5 m, then uniform liquid distribution is difficult in a packed tower. Hence, in such a case, tray tower is preferred.
4. If solids are present in liquid or gas to a limited extent then tray towers are preferred because they permit easier cleaning. In practice, solids are getting accumulated in packed tower and result in increasing the pressure drop. For example, absorption of chlorine in aqueous caustic soda solution, fibre glass reinforced plastic (FRP) tray tower is preferred over FRP packed tower. Use of packed tower for this application restricts the conversion of NaOH to NaCl and NaOCl. Once the concentration of salts in the circulating solution increases above the limiting value then fine suspended solids may appear in the solution. Hence, with the packed tower, solution bled from the circulating system contains unconverted NaOH. Use of tray tower permits complete conversion of NaOH in the circulating solution.

High Speed Mass Transfer Trays For absorption service under high pressure, high speed mass transfer trays are developed by many suppliers. For example, dehydration of natural gas with ethylene glycol solution, sweetening of gases, rectification of hydrocarbon mixtures, removal of noxious gases by scrubbing, etc., require intense mass transfer with high liquid loading and high level of flexibility. For such applications, high speed trays are found economical as the tray column becomes compact in terms of diameter and height.

One such example of high speed tray is shown in Fig. 9.1.

Fig. 9.1 *High Speed Tray*
(Courtesy: HAT International Ltd., U. K.)

In this design, swirl-type cyclone elements are fixed into the opening of the plate. The element consists of a vertical cylinder with annular row of holes in the lower section. The axial swirler, firmly attached to the inside of the cylinder has inclined vanes in upper part. Gas from bottom induces liquid from the holes, travels through the swirl element and enters the cyclone separator where liquid separates from the froth mixture and falls back on the tray. Turbulence created in the swirl element can result in high mass transfer efficiency, reducing the tower diameter and column height. Needless to say, such trays offer high pressure drop as compared to conventional trays. Efficient separation of liquid also helps in reducing entrainment. Also sensitivity to weeping is reduced.

9.2.4 Use of Falling Film Absorber

For highly exothermic absorption, e.g., absorption of hydrogen chloride gas in water and absorption of ammonia gas in water, more efficient heat removal system is required to get the higher efficiency and higher concentration of solution. For such applications, falling film absorbers are used. This could be a shell and tube heat exchanger, in which absorption is carried out inside the tubes while cooling medium is circulated on shell side. In a heat exchanger, made of graphite blocks, absorption is carried out inside the vertical tube (path) while cooling medium is passed through horizontal holes (perpendicular to vertical tube path). Design of a falling film absorber is dealt later in this chapter (Example 9.5).

9.2.5 Use of Venturi Scrubbers

Venturi scrubbers are normally preferred for removing particulate matter from a gas stream as opposed to absorbing soluble vapour from vapour-gas mixture. Venturi scrubber provides higher gas-liquid contact or higher absorption efficiency but at the expense of relatively large liquid side pressure drop and consequently it requires higher power consumption.

9.2.6 Use of Stirred Tanks and Sparged Towers

Stirred tanks and sparged towers are preferred for slow gas-liquid reactions. Normally, in cases of absorption with chemical reaction, solvents or liquid reactants are selected in such a way that they offer fast or very fast chemical reaction with the solute gas. Hence, these equipments are normally not used as absorbers.

9.3 DESIGN OF PACKED TOWER-TYPE ABSORBER

9.3.1 Packings

Before attempting the process design of the packed tower, characteristics of various packings be understood. Packings can be divided into two major groups; random (or dumped) packing and structured packing. Random packings are either cylindrical or half cylinder (saddle) shaped. Some known packing shapes are shown in Fig. 9.2.

Fig. 9.2 *Random Packings*

Both rings and saddles provide surface area within the structure, resist the deformation and prevent from nesting when dumped. Within the packing types, packing elements are available in different sizes and material of construction. Elements fabricated from a given material look essentially same except their sizes. This implies that they are geometrically similar and differ only by a characteristic dimension; known as *nominal packing size* (δ_p). Mass transfer of a packing is significantly impacted by the specific area of the packing (a_t) which is inversely proportional to δ_p. Data on various random packings are given in Table 9.1. Packing factor F_p is the key to the pressure drop calculations and is determined experimental. For example, F_p for metal Raschig rings of 25 mm nominal size (δ_p) is 450 m^{-1} while that of 25 mm metal Pall rings is 157 m^{-1}, less than half. This is attributable to the openness of the Pall rings compared to Raschig rings. This can also be inferred from % void (ε) which is 86% for Raschig rings and 94% Pall rings. Correlations are reported in literature[4] for calculating a_t and F_p, knowing δ_p, but one should rely more on published data of the supplier or generalised data (similar to those given in Table 9.1).

Pall rings are the preferred and commonly used random packings but their cost per unit volume is high. Pall rings require minimum diameter and minimum height for the given absorption or distillation duty as compared to other types of random packings. Pall rings are available in metals and plastics. For high temperature services polyethylene or polypropylene Pall rings cannot be used. Plastic packings are also attacked by some organic solvents. For corrosive services ceramic Berl saddles are cheaper than metallic Pall rings. For example, in distillation of the mixture of phosphorous trichloride and phosphorous oxychloride, packed tower with ceramic packing is used. Ceramics are brittle at high temperature and have poor strength. For very high temperature and corrosive services glass packed columns with glass Raschig rings are preferred against packed column of special alloy. For example, in vacuum distillation (for extractive distillation of concentrated nitric acid using concentrated sulphuric acid as solvent) it is carried out in glass packed tower with glass Raschig rings. Raschig rings are the cheapest packing material but are less efficient. Raschig rings require the maximum diameter and height for the given absorption or distillation

Table 9.1 Characteristics of Dumped (Randomly Packed) Tower Packings[3]

Packing type	Nominal size δ_p, mm	Wall thickness, mm	Outside diameter & length, mm	Approximate No. of elements per m^3	Approximate mass per m^3, kg	Approximate surface area, a_t, m^2/m^3	Percent void space, ε	Packing factor F_p, m^{-1}
Berl saddles,	6	—	—	3.78×10^6	900	900	60	2950
ceramic	13	—	—	590 000	865	465	62	790
	25	—	—	77 000	720	250	68	360
	38	—	—	22 800	640	150	71	215
	50	—	—	8 800	625	105	72	150
Intalox saddles,	6	—	—	4.15×10^6	865	984	75	2380
ceramic	13	—	—	730 000	720	625	78	660
	25	—	—	84 000	705	255	77	320
	38	—	—	25 000	670	195	80	170
	50	—	—	9 400	760	118	79	130
	75	—	—	1 870	590	92	80	70
Intalox saddles,	(No. 25)	—	—	168 400	—	—	97	135
metal	(No. 40)	—	—	50 100	—	—	97	82
	(No. 50)	—	—	14 700	—	—	98	52
	(No. 70)	—	—	4 630			98	43
Intalox saddles,	25	—	—	55 800	76	206	91	105
plastic	50	—	—	7 760	64	108	93	69
(polypropylene)	75	—	—	1 520	60	88	94	50
Pall rings,	16	26 gauge	16	—	—	—	92	230
metal	25	24 gauge	25	49 600	480	205	94	157
	38	22 gauge	38	13 000	415	130	95	92
	50	20 gauge	50	6 040	385	115	96	66
	90		90	1170	270	92	97	53

(*Contd.*)

Table 9.1 (*Contd.*)

Packing type	Nominal size δ_p, mm	Wall thickness, mm	Outside diameter & length, mm	Approximate No. of elements per m^3	Approximate mass per m^3, kg	Approximate surface area, a_t, m^2/m^3	Percent void space, ε	Packing factor F_p, m^{-1}
Pall rings,	16	—	16	214 000	116	340	87	310
plastic	25	—	25	50 100	88	205	90	170
(polypropylene)	38	—	38	13 600	76	130	91	105
	50	—	50	6 360	72	100	92	82
	90	—	90	1 170	68	85	92	52
Raschig rings,	6	1.6	6	3.02×10^6	960	710	62	5250
ceramic	13	2.4	13	378 000	880	370	64	2000
	19	2.4	19	109 000	800	240	72	840
	25	3.2	25	47 700	670	190	74	510
	38	6.4	38	13 500	740	120	68	310
	50	6.4	50	5 800	660	92	74	215
	75	9.5	75	1 700	590	62	75	120
	100	9.5	100	700	580	46	80	
Raschig rings,	19	1.6	19	111 000	1500	245	80	730
Steel	25	1.6	25	46 300	1140	185	86	450
	38	1.6	38	14 100	785	130	90	270
	50	1.6	50	5 900	590	95	92	187
	75	1.6	75	1 800	400	66	95	105
Hy-Pac, steel	(No.1)		30	30 000	300	177	96	141
	(No.2)		60	3 780	225	95	97	59
Leva packing	(No.1)			34 000	270	164		
	(No.2)			10 500	210	118		
Low-density polyethylene Tellerettes	25			39 700	160	250	83	

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duty compared to other types of random packings. Hence, total fixed cost of packed column is higher. Diameter of tower is recommended to be more than 10 times the packing size. Increase in packing size decreases required tower diameter but increases height of packing. As increase in packing size reduces pressure drop per unit height of packing but decreases surface area of packing per unit volume and hence, decreases mass transfer area and rate of mass transfer. Increase in packing size increases the value of HETP or H_{OG}. In general increase in packing size reduces the cost of packing per unit volume.

The second category of packing refers to structured packing. It can be made of woven or solid material, usually thin metal sheet, which is typically corrugated and bundled into segments that are placed layer by layer into the column shell. In most cases, three types of structured packing are used. Type B (also known as sheet metal packing) is fabricated from corrugated sheets as explained above. Most B-type packing have a corrugation angle of 45°and a fold angle of 90°. Second category is Type BSH packing which combines the essential features and characteristics of sheet metal and wire gauze packing. In the BSH-packing surface structure consists of rhombic perforations with alternating burred-up. Third category is Type A which is basically Wire Gauze Packing. It is constructed of multiple layers of corrugated and pierced wire gauze sheets.

9.3.2 Process Design

Process design of packed tower type absorbers involve two major steps: (i) determination of tower diameter and (ii) determination of height of packing. Method for determining tower diameter is same for all types of packed tower absorbers. But methods for finding the height of packing are different for the following cases:

1. Physical absorption from dilute gas mixture in isothermal conditions.
2. Physical absorption from concentrated gas mixture in isothermal conditions.
3. Physical absorption in non-isothermal condition, for example, absorption of HCl or NH_3 in water.
4. Absorption with chemical reaction, for example, absorption of chlorine in sodium hydroxide solution.

Packed Tower Type Absorber for Physical Absorption Example of an absorber for physical absorption from dilute gas mixture in isothermal condition is the absorption of acetone vapour in water from air-acetone vapour mixture containing less acetone (say up to 6%). Step-wise design for this case is as follows.

1. From the given gas mixture data, find G_1, G_s, y_1 and Y_1.

where, G_1 = Molar flow rate of gas mixture, kmol/h
G_s = Molar flow rate of non-solute gas mixture, kmol/h
y_1 = Mole fraction of solute in feed gas

$$G_s = G_1 (1 - y_1) \tag{9.1}$$

$$Y_1 = \frac{y_1}{1 - y_1}$$

Y_1 is the ratio of the moles of solute gas to the same of non-solute gas for incoming gas,

Fig. 9.3 *Variables Used for Material Balance and Design*

$$G_1 = G_s(1 + Y_1) \tag{9.2}$$

Decide the degree of absorption to be carried out in absorber, if it is not specified. Find y_2, Y_2 and G_2.

where, y_2 = Mole faction of solute in outgoing gas mixture

$Y_2 = \dfrac{y_2}{1 - y_2}$, is the ratio of the moles of solute gas to non-solute gas for outgoing gas mixture

$$G_2 = G_s\,(1 + Y_2) \tag{9.3}$$

where, G_2 = Molar flow rate of outgoing gas mixture, kmol/h

If absorber is a pollution control tower then value of y_2 or Y_2 is decided by pollution control norms.

2. Select a suitable solvent. In the selection of solvent, the important properties to be considered are solubility of solute gas in the solvent, volatility, cost, corrosiveness, etc[1]. Solvent which reacts with the solute by chemical reaction provides high solubility. If recovery of solvent or solute gas is essential then chemical reaction should be reversible. For example, for the absorption of CO_2 gas, caustic soda solution provides higher solubility or absorptivity than ethanol-amine solution. But in actual application, ethanolamine solution is selected as solvent rather than caustic soda solution because CO_2 gas can be easily separated from ethanolamine solution at higher temperature and lower pressure in a stripping column while caustic soda solution does not easily release CO_2 gas in stripping operation.

3. Find the gas-liquid equilibrium data. Draw the equilibrium curve in terms of Y *vs* X.

4. Find the minimum amount of solvent required for the desired separation. Minimum amount of solvent is the maximum amount of solvent for which infinite number of equilibrium stages are required for the desired separation. In other words, it is the maximum amount of solvent for which desired degree of absorption is not possible. Minimum amount of solvent, in physical absorption is determined by graphical method.

(a) In this method, first draw the equilibrium curve Y vs X.

(b) Mark (X_2, Y_2) point, where, X_2 = ratio of the moles of solute to moles of solvent for *incoming solvent*, Y_2 = ratio of the moles of solute to non-solute gas for outgoing gas mixture.

(c) Draw the tangent starting from point (X_2, Y_2) and allow it to intersect with horizontal line from Y_1, if the equilibrium curve is concave upward [Fig. 9.4(a)]. But if the curve is concave downward as shown in Fig. 9.4(b), draw the line from (X_2, Y_2) and join it with the point of intersection of equilibrium curve and horizontal line from Y_1.

In any case, intersection point is represented as (X_{1m}, Y_1), where X_{1m} is the value of X_1 corresponding to minimum amount of solvent and X_1 is the ratio of the moles of solute to moles of solvent in outgoing solution.

Fig. 9.4 *Minimum Amount of Solvent*

(d) Write the overall solute balance equation

$$L_{sm}(X_{1m} - X_2) = G_s\,(Y_1 - Y_2) \tag{9.4}$$

substitute the value of X_{1m} obtained from the graph and find the value of L_{sm} from the above equation.

5. Decide the actual molar flow rate of solvent; L_s. Ideally it should be optimum. Optimum amount of solvent is the amount of solvent which gives minimum total cost of absorption. Total cost includes fixed cost, operating cost, interest on fixed capital, etc. At design stage, if it is not possible to find the optimum solvent flow rate, then actual flow rate L_s should be the greatest of the following values:

(a) $L_s = 1.5\,L_{sm}$ (9.5a)

(b) $L_s = \dfrac{1 \times \rho}{M_w}$ (9.5b)

where, M_W = Molar mass of solvent

Certain minimum flow rate is required to facilitate the use of a centrifugal pump.

(c) $L_s \geq$ MWR*

*Minimum wetting rate (MWR) is the liquid rate, required to form a liquid film over all the packing. No 100% reliable method is available to find MWR. Some designers[2] consider minimum liquid rate for any tower packing as 7340 kg/(h · m^2 of tower cross section). Packing wetting rates are related to packing material surface

Table 9.2 Minimum Wetting Rate for Different Packings

Surface	Minimum wetting rate (MWR), $m^3/(m^2 \cdot h)$
Unglazed ceramic	0.5
Glazed ceramic	2.0
Glass	2.5
Stainless steel	3.0
Carbon steel	0.7
Polypropylene	4.0
Polyvinyl chloride	3.5
Polytetrafluoroethylene (PTFE)	4.0

Morris and Jackson equation gives minimum solvent rate required in $m^3/(h \cdot m^2$ of cross section of tower) by following equation[2, 3]:

$$\text{MWR} = R \cdot a_t \tag{9.5c}$$

where, a_t = Packing surface area per unit volume, m^2/m^3.

Values of R in m^2/h for rings less than 75 mm size is 0.08 and for packing more than 75 mm size it is 0.12.

Kister[3,4] has suggested values of MWR in $m^3/(m^2 \cdot h)$, 1.26 to 5.4 for random packings and 0.252 to 0.504 for structured packing.

(d) For exothermic absorption L_S should be such that it gives per pass rise in temperature of liquid less than 10°C.

(e) Find the value of X_2 based on overall solute balance.

$$L_s(X_1 - X_2) = G_s(Y_1 - Y_2) \tag{9.6}$$

6. Find N_{OG} or N_{OL}.

where, N_{OG} = Number of overall gas phase transfer units

N_{OL} = Number of overall liquid phase transfer units

Height of packing required for the given absorption duty is determined by using one of the following equations:

$$Z = H_{OG} \cdot N_{OG} \tag{9.7}$$

or

$$Z = H_{OL} \cdot N_{OL} \tag{9.8}$$

If overall resistance to mass transfer is controlled by the gas film, then Eq. (9.7) is used to find the packing height Z for the desired absorption duty. If the gas is highly soluble in liquid (solvent), then major resistance to mass transfer is provided by the gas film. For example, HCl (hydrogen chloride gas) is highly soluble in water. Hence, in this absorption, overall resistance to mass transfer is governed by hydrogen chloride gas film resistance. Another example of gas-film controlled absorption is absorption of acetone vapour in water from air-acetone vapour mixture. In the same case, if the concentration of solute gas in gas mixture is less than 10% (by mole) then molar flow rate of gas can be assumed as constant and height of packing (Z), is given by following equation:

$$Z = H_{OG} \cdot N_{OG} = \frac{G_m}{K_G ap} \int_{y_2}^{y_1} \frac{dy}{y - y_e} \tag{9.9}$$

where,

$$H_{OG} = \frac{G_m}{K_G ap} \tag{9.10}$$

and

$$N_{OG} = \int_{y_2}^{y_1} \frac{dy}{y - y_e} \tag{9.11}$$

where, G_m = Molar flow rate of gas per unit cross-sectional area, kmol/(m^2 · s)
K_G = Overall gas phase mass transfer coefficient, kmol/(atm · m^2 · s)
a = Interfacial surface area per unit volume, m^2/m^3
p = Operating pressure, atm
H_{OG} = Height of an overall gas phase transfer units, m
N_{OG} = Number of an overall gas phase transfer units

Practically, it is not possible to find individual value of a. Hence majority of correlations are developed for $K_G a$.

N_{OG} can be determined by Eq. (9.11), for which area under the curve of $1/(y - y_e)$ vs y within limits from y_2 to y_1 is determined. In the special case, if equilibrium curve and operating line, both can be assumed as straight lines, N_{OG} can be calculated by the following equation[5]:

$$N_{OG} = \frac{1}{1 - \left(\frac{mG_m}{L_m}\right)} \ln\left[\left(1 - \frac{mG_m}{L_m}\right)\frac{y_1}{y_2} + m\frac{G_m}{L_m}\right] \tag{9.12}$$

where, G_m = Molar gas velocity, kmol/(m^2 · s)
L_m = Molar liquid velocity, kmol/(m^2 · s)

and m is the slope of the equilibrium line and G_m/L_m is the slope of the operating line.

If the overall resistance to mass transfer is controlled by liquid film then Eq. (9.8) is used to find the packing height. If the solubility of gas in liquid phase is very low then overall resistance to mass transfer is governed by liquid film resistance. For example, (i) absorption of carbon dioxide gas (CO_2) in water from CO_2 rich gas mixture and (ii) absorption of dimethyl ether in water. In such cases, normally a chemical reagent is added into liquid phase which reacts with solute gas and thereby increases the rate

of mass transfer dramatically. For example, for the absorption of CO_2, potassium carbonate (K_2CO_3) or ethanol amine is added into water to improve the rate of the absorption of carbon dioxide (CO_2). In the same case, if the concentration of solute gas in gas mixture fed to absorber is less than 10% (by mole) or for the dilute system, molar flow rate of liquid can be assumed as constant and height of packing required can be calculated by the following equations:

$$Z = H_{OL} \cdot N_{OL} \tag{9.8}$$

where,

$$H_{OL} = \frac{L_m}{K_{LC} a C_t} \tag{9.13}$$

and

$$N_{OL} = \int_{x_2}^{x_1} \frac{dx}{x_e - x} \tag{9.14}$$

where, L_m = Molar liquid flow rate per unit cross-sectional area, kmol/(m^2 · s)
N_{OL} = Number of overall liquid phase transfer units
H_{OL} = Height of an overall liquid phase transfer unit, m
K_{LC} = Overall liquid-phase mass transfer coefficient, m/s
a = Interfacial surface area per unit volume, m^2/m^3
C_t = Total molar concentration, kmol/m^3
x_1 = Mole fraction of solute in the liquid phase at the outlet from absorber
x_2 = Mole fraction of solute in the liquid phase at the inlet to the absorber
x_e = Equilibrium mole fraction of solute in liquid phase

Units of mass transfer coefficient depend on units of driving force. K_{LC} represents mass transfer based on concentration gradient.

Here again, a cannot measured experimentally and hence, value of $K_{LC}a$ are reported.

N_{OL} can be determined by Eq. (9.14), for which area under the curve of $\frac{1}{x_e - x}$ vs x within limits x_1 to x_2 should be determined.

Determination of Tower Diameter In a packed column as the throughput increases beyond the stable operating region, pressure drop begins to rise much more quickly and liquid entrainment increases from higher liquid holdup. As random packing is relatively open to cross-flow, liquid and vapour/gas tend to redistribute themselves. At very high throughput, there is sharp fallout in mass transfer. This point is the *flood point*.

For packed tower-type absorber, tower diameter is determined based on flooding velocity. Actual velocity of gas through packed tower is kept about 60 to 70% of flooding velocity. Recommended range of pressure drop for packed tower-type absorber is 15 to 50 mm water column per m of packing height depending on the application. Flooding velocity of gas for packed tower is defined as a velocity of gas through the packed tower at which pressure drop across the tower does not remain stable, even though liquid flow and gas flow through the tower are kept constant. It goes on increasing and tends to infinite. At the time of flooding, one of two conditions

may occur; (i) Liquid phase may occupy the entire cross section of tower. Continuous phase of liquid body rises in the column. The change in pressure drop is very high with only a slight change in gas rate. (ii) Phase inversion occurs and gas bubbles through the liquid. Pressure drop rises rapidly as phase inversion occurs. To find the tower diameter, at first the following factor (F_{LG}) is determined:

$$F_{LG} = \frac{L_w}{G_w}\sqrt{\frac{\rho_G}{\rho_L}} \tag{9.15}$$

where, L_w = Mass velocity of liquid, kg/(m^2 · s)
G_w = Mass velocity of gas, kg/(m^2 · s)
ρ_G = Density of gas, kg/m^3
ρ_L = Density of liquid, kg/m^3

Using Fig. 9.5, corresponding value of K at flooding (K_F) is determined. Actual value of K is determined as follows. This graph is known as Sherwood-Leva-Eckert correlation chart. Actual velocity of gas through the tower is kept in the range of 50 to 70% of the flooding velocity. Let the actual velocity of gas through tower be 66% of flooding velocity.

$$K = (0.66)^2 K_F \tag{9.16}$$

Corresponding value of pressure drop per m of packed height can be determined by using Fig. 9.5. Recommended range of pressure drop in atmospheric packed tower type absorber is 15 to 50 mm WC per m of packing. This chart (having log-log scales) is a good approximation of the pressure drop in packed beds but the values are correct within ±20%. This graph has limitations that it does not take into account of the packing geometry and is based on limited data source. Kister and Gill[4] later developed different charts for different packings on semilog scale. Y-axis is a linear scale in new charts which make interpolation easier. While X-axis factor is kept same as that given in Fig. 9.5. Y-axis factor includes packing factor. Massive data base was used to prepare the charts which give more accurate values of pressure drop and flood line is omitted from the graphs. Reader is advised to refer original article for these charts.

Billet and Schultes[6] have presented in a concise manner updated equations for calculating gas and liquid side mass transfer coefficients, pressure drops of dry and irrigated random and structured packings, their loading and flooding points as well as liquid hold-ups. Since their presentation is based on physical models with large experimental data bases, they are highly reliable in designing packed columns for mass transfer operations. The methods do not present graphical presentations such as that of Sherwood–Leva–Eckert but present empirical equations to be solved. However, the method involves elaborate and iterative calculations. Use of a computer is recommended for these repetitive calculations. Reader is advised to refer original article for more details.

K is a function of G_w.

$$K = \frac{G_w^2 F_p \psi \mu_L^{0.2}}{\rho_G \rho_L g} \tag{9.17}$$

where, K = Function of velocity of gas through tower
ρ_G, ρ_L = Density of gas and liquid, respectively, kg/m^3
G_w = Mass velocity of gas, kg/(m^2 · s)

$$G_w = \left(\frac{K \cdot \rho_G \rho_L g}{F_p \psi \mu_L^{0.2}} \right)^{1/2} \tag{9.18}$$

g = 9.81 m/s^2
F_p = Packing factor, m^{-1} (Ref: Table 9.2)
ψ = Ratio of density of water to density of liquid (inverse of specific gravity)
μ_L = Viscosity of liquid, m Pa · s (= cP)

Fig. 9.5 *Generalised Flooding and Pressure-Drop Correlation Chart for Packings*[3] *(Sherwood-Leva-Eckert Correlation Chart)*
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Determination of H_{OG} or H_{OL} Many different correlations are available to find out the height of transfer unit or to find out the mass transfer coefficients. But, these correlations are not as reliable as the same for finding the heat transfer coefficients. Hence, it is suggested to find out the height of transfer unit of mass transfer coefficient by using the correlation which is derived for the same or a similar system. But, if the correlation for the same or a similar system is not available, then, use more than one generalised correlations for finding the height of transfer unit (H_{OG} or H_{OL}) and the

maximum value obtained from the different correlation is considered for finding the height of packing. Two generalised correlations are given below.

1. Onda, Takeuchi and Okumoto Correlation[3]: Height of overall gas phase transfer unit H_{OG} and height of overall liquid phase transfer unit H_{OL} are related with H_G and H_L by following equation:

$$H_{OG} = H_G + m\frac{G_m}{L_m}H_L \tag{9.19}$$

$$H_{OL} = H_G + \frac{L_m}{mG_m}H_G \tag{9.20}$$

where, m is the slope of equilibrium line, G_m/L_m is the slope of operating line and G_m and L_m are the molar flow rates of gas and liquid, respectively per unit cross-sectional area of tower in kmol/(m^2 · s). Height of gas phase transfer unit (H_G) is determined by the following equation:

$$H_G = \frac{G_w}{K_G\, a_e\, pM_g} \tag{9.21}$$

and height of liquid phase transfer unit (H_L) is determined by the following equation:

$$H_L = \frac{L_w}{K_{LC}\, a_e\, \rho_L} \tag{9.22}$$

where, G_w = Superficial mass velocity of gas, kg/(s · m^2)
L_w = Superficial mass velocity of liquid, kg/(s · m^2)
p = Operating pressure, Pa
M_g = Molar mass of gas, kg/mol
ρ_L = Liquid density, kg/m^3
a_e = Effective area for mass transfer, m^2/m^3
K_G = Gas phase mass transfer coefficient, kmol/(m^2 · s · Pa)
K_{LC} = Liquid phase mass transfer coefficient, m/s

Effective area for mass transfer is obtained by the following equation:

$$\frac{a_e}{a_t} = 0.31\frac{\sigma_L^{0.5}}{Z^{0.4}}(C_{aL} \cdot Re_G)^{0.392} \tag{9.23}$$

where, a_e = Effective area for mass transfer, m^2/m^3
a_t = Total area of packing, m^2/m^3 (Ref. Table 9.1)
σ_L = Liquid surface tension, m N/m (= dyn/cm)
Z = Packed height, m
C_{aL} = Liquid capillary number = $\mu_L\, L_w/(\rho_L\, \sigma_L)$ (9.24)
(dimensionless group)
ρ_L = Density of liquid, kg/m^3
μ_L = Viscosity of liquid, mPa · s (= cP)

$$Re_G = \text{Gas Reynolds number} = \frac{6G_w}{a_t\,\mu_G} \tag{9.25}$$

μ_G = Gas viscosity Pa · s

Gas phase mass transfer coefficient is determined by the following equation:

$$\frac{K_G RT}{a_t D_G} = C_1 \left(\frac{G_w}{a_t \mu_G}\right)^{0.7} \left(\frac{\mu_G}{\rho_G D_G}\right)^{1/3} (a_t d_p)^{-2} \tag{9.26}$$

where, K_G = Gas phase mass transfer coefficient, kmol/(m^2 · s · Pa)
R = 8314 J/(kmol · K)
D_G = Gas phase diffusion coefficient, m^2/s
T = Operating temperature, K
ρ_G = Density of gas, kg/m^3
d_p = Packing size, m (derived from δ_p)
C_1 = 2 for random packings of size less than 12 mm
= 5.23 for random packings of size more than 12 mm

Liquid phase mass transfer coefficient is determined by the following equation:

$$K_{LC}\left(\frac{\rho_L}{\mu_L g}\right)^{1/3} = 0.0051\left(\frac{L_W}{a_w \mu_L}\right)^{2/3} \left(\frac{\mu_L}{\rho_L D_L}\right)^{-0.5} (a_t d_p)^{0.4} \tag{9.27}$$

where, μ_L = Liquid viscosity, kg/(m · s)
g = 9.81 m/s^2
L_W = Mass velocity of liquid, kg/(m^2 · s)
D_L = Liquid phase diffusion coefficient, m^2/s
d_p = Equivalent diameter of packing, m (derived from δ_p)
a_w = Wetted surface of packing, m^2/m^3
a_w is determined by following equation

$$\frac{a_w}{a_t} = 1 - \exp\left[-1.45\left(\frac{\sigma_C}{\sigma_L}\right)^{0.75} Re_L^{0.1} Fr_L^{-0.05} We_L^{0.2}\right] \tag{9.28}$$

where, σ_L = Surface tension of liquid, mN/m

Re_L = Liquid Reynolds number = $\dfrac{L_w}{a_t \mu_L}$ (dimensionless)

Fr_L = Froude number for liquid = $L_w^2 a_t/(\rho_L^2 g)$ (dimensionless)

We_L = Weber number for liquid = $L_w^2/(\rho_L \sigma_L a_t)$ (dimensionless)

σ_C = Critical surface tension of the packing material, mN/m
(Ref. Table 9.3)

Table 9.3 Critical Surface Tension of Packing Materials[3]

Material of Packing	σ_C, mN/m or dyn/cm
Carbon	56
Ceramic	61
Glass	73
Polyethylene	33
Polypropylene	33
Polyvinyl chloride	40
Steel	75

2. Cornell's method: In this method height of overall gas phase transfer unit H_{OG} or height of overall liquid phase transfer unit H_{OL} is calculated by the same equations Eqs. (9.19) and (9.20) that are used for Onda's method but H_G and H_L are determined by the following equations:

Height of gas phase transfer unit H_G is calculated by following equations.

For Raschig rings and Pall rings,

$$H_G = \frac{0.017\,\psi_c\,D^{1.24}Z^{0.33}Sc_G^{0.5}}{(L_w f_1 f_2 f_3)^{0.6}} \tag{9.29}$$

For saddle-type packings,

$$H_G = \frac{0.029\,\psi_c\,D^{1.11}Z^{0.33}Sc_G^{0.5}}{(L_w f_1 f_2 f_3)^{0.5}} \tag{9.30}$$

where H_G = Height of gas phase transfer unit, m

Sc_G = Gas phase Schmidt number = $\dfrac{\mu_G}{\rho_G D_G}$ (dimensionless) (9.31)

D_G = Gas phase diffusion coefficient, m^2/s

ρ_G = Density of gas, kg/m^3

μ_G = Viscosity of gas, kg/(m · s) or Pa · s

D = Column diameter, m

Z = Packed height, m

L_w = Mass velocity of liquid, $kg/(m^2 \cdot s)$

$f_1 = \left(\dfrac{\mu_1}{\mu_W}\right)^{0.16}$ with μ_W = 1.0 mPa · s

$f_2 = \left(\dfrac{\rho_W}{\rho_L}\right)^{1.25}$ with ρ_W = 1000 kg/m^3

$f_3 = \left(\dfrac{\sigma_W}{\sigma_L}\right)^{0.8}$ with σ_W = 72.8 mN/m

ψ_c = Correlation parameter which can be obtained from Fig. 9.6 for various types of random packings.

Height of liquid phase transfer unit is calculated by the following equation:

$$H_L = \frac{\phi_{CO}C}{3.28}\left(\frac{\mu_L}{\rho_L D_L}\right)^{0.5}\left(\frac{Z}{3.05}\right)^{0.1} \tag{9.32}$$

where, H_L = Height of liquid phase transfer unit, m

ϕ_{CO} = Correlation parameter for a given packing, m (can be obtained from Fig. 9.7)

C = Correlation factor for high gas rates (Refer to Fig. 9.8)

μ_L = Liquid viscosity, Pa · s

ρ_L = Liquid density, kg/m^3

D_L = Liquid diffusion coefficient, m^2/s

Z = Height of packing, m

In this method, if the column diameter (D) is greater than 0.6 m then take D = 0.6 m for the calculation of H_G.

Fig. 9.6 *Correlation Parameter ψ_C for Cornell's Method*[3]
(Reproduced with the Permission of McGraw-Hill Education, USA)

(a) Ceramic Raschig Rings

(b) Ceramic Berl Saddles

(c) Metallic Raschig Rings

(d) Metal Pall Rings

L_w = Liquid Rate, kg/(m^2 · s)

Fig. 9.7 *Correlation Parameter for Various Packings*[3]
(Reproduced with the Permission of McGraw-Hill Education, USA)

Fig. 9.8 *Flooding Correction Factor, C*[3]
(Reproduced with the Permission of McGraw-Hill Education, USA)

Design or Selection of Internals of Packed Tower Internals of packed tower are packings, packing support, liquid distributor, liquid redistributor, mist eliminator, hold-down plate, etc. Refer to Fig. 7.16 for packed tower details.

1. Packings: Suitable packing must be selected before the determination of tower diameter. Packings can be divided in two broad classes:

(a) Regular packings: Grids, structured packings, stacked rings, etc., are of this type. Grids are used for high gas rates as they offer very low pressure drop. They are used in cooling towers.

Structured packings (Refer Section 9.3.1) are widely used in chemical process industries. They are made from corrugated sheets with some perforations or wire mesh. They provide high surface area with high void fraction. HETP (Height Equivalent to Theoretical Plate) of structured packing is generally less than 0.5 m which could be less than half as compared to dumped Pall rings. They are produced by many manufacturers. They are preferred for difficult distillation operations, for high vacuum distillation, for high efficiency absorption with low allowable pressure drop and also for increasing the capacity of existing distillation column. Necessary data, correlations or graphs for calculating tower diameter can be obtained from the manufacturer.

(b) Random packings: Various types of random packings are covered in Section 9.3.1.

2. Liquid distributors: Various types of liquid distributors, used with packed tower, are shown in Fig. 9.9. Uniform initial distribution of liquid at the top of packed bed is essential for the efficient mass transfer operation (distillation or absorption).

Fig. 9.9 *Different Types of Liquid Distributors*

For small diameter packed columns (having diameter less than 0.3 m) single point distributor like one spray nozzle is adequate. For large diameter packed columns, multipoint distributors like perforated pipe distributor, trough-type distributor, orifice distributor, etc., are used. As per the Perry's Handbook[3], for each 194 cm^2 cross-sectional area one distribution point is required. As per the Eckert's recommendations, number of streams or number of distribution points required in packed tower are given in Table 9.4.

Table 9.4 Number of Distribution Points in Packed Tower[3]

Column diameter, m	No. of points/m^2 of cross-section
0.4	340
0.75	170
≥ 1.2	40

Perforated pipe distributors are preferred for column diameter ranging from 0.3 m to 1 m. It is used with clean liquids and offers minimum restriction to gas flow. They are preferred with reasonably constant flow rate.

Trough-type distributors are preferred for columns having diameter greater than or equal to 1.2 m. It can be used with liquids having solid in suspension. Trough-type distributors are fabricated from metal sheets, plastics or ceramics. This distributor consists of a series of troughs containing side notches. It provides good liquid distribution with a large free area for gas flow.

Orifice-type liquid distributors are also preferred for large diameter packed column. This type of distributor consists of flat perforated tray equipped with a number of risers or short stand pipes. Ideally, gas rises upward through risers or short stand pipes while liquid maintains the certain level over the perforated plate and flows down through perforations. The risers should be sized to give sufficient area for gas flow without creating a significant pressure drop. The holes (perforations) should be small enough to ensure that there is a level of liquid on the plate at the lowest liquid rate but large enough to prevent the distributor overflowing at the highest rate.

In **weir-type distributor** gas and liquid (both) are flowing through the same stand pipes. Notched weirs are provided in upper portion of stand pipes. Certain level of liquid is maintained over the tray and liquid flows over notched weirs and falling down through the same pipes from which gas rises upward. Weir-type distributor is preferred with fluctuating liquid flow rates.

3. Liquid redistributor: After travelling a certain distance in a packed tower, considerable fraction of liquid is migrated to the column wall and flows down in the vicinity of column wall while the gas rises upwards through the central portion. This phenomenon is called *channelling*. Liquid redistributor collects the liquid that has migrated to the column wall and redistributes it evenly over the next bed of the packing.

For small diameter column ($D \leq 0.6$ m) wall wiper-type liquid redistributor is preferred. It collects liquid from the column wall and redistributes it into the central portion. Sometimes packing support plate itself acts as liquid redistributor. Different types of liquid redistributors are shown in Fig. 9.10.

(a) Wiper redistributors (b) Bell-cap redistributor

Fig. 9.10 *Different Types of Liquid Redistributors*

4. Packing support: The function of packing support is to carry the load of wet packing bed without providing excessive restriction to gas and liquid flows. It also acts as distributor for both streams. Poor design of packing support provides higher pressure drop and can cause premature local flooding. Two types of packing supports are used (Fig. 9.11).

(a) Countercurrent type: Example; Mesh-type packing support
(b) Separate flow or gas injection type: Example; Cap-type packing support, beam-type gas injection support, etc.

Fig. 9.11 *Packing Supports*

In countercurrent-type packing support liquid and gas both are flowing through the same openings in countercurrent direction. Major open area of countercurrent-type packing support is occluded by packing pieces resting on it. Hence, this type of packing support provides the low value of net free area (50% or less). Column diameter is decided based on the characteristics of packing material. Berl saddles, Raschig rings, etc., provide the lesser free area for flow of gas, hence with such packings, counter current-type packing support can be used. But other packings like Pall rings, structured packings, etc., provide higher % free area (ε) or percentage void space for the flow of gas (85% or more). Hence, with these type of packings, counter flow-type packing support cannot be used. Gas injection-type packing support can be designed for free area up to 90% and because of their geometry there will have very little occlusion by the packing. In this type of packing support, separate flow passages or openings are provided for gas and liquid streams. Gas inlets are provided above the liquid level. With packing materials like Pall rings, structured packings, etc., gas injection-type packing support is recommended.

5. Hold-down plate: Hold-down plate is required to prevent damage to the packing which can result due to a breakdown in normal operating conditions. At high gas or vapour flow rates, packing at the top can be fluidised. This may result in breaking or deshaping of packing. Ceramic packings can be easily broken which may settle in voids of the packed bed and plug the flow channels. In case of metal packings, deshaping may take place and the deshaped pieces can plug the flow channels. In case of plastic packings, they may fly away with gas or vapour and seat at various locations in the column. Also plastic packing may break and seat randomly in the tower.

A typical hold-down plate is shown in Fig. 7.17.

Hold down plate, generally in the grid form or the screen form, can be used to prevent such fluidisation. While heavier hold down plates are used for ceramic and metal packings, lighter hold down plates of similar construction are used for packed towers with plastic packing. Hold down grids/screens have generally poor open area (< 50% of internal cross section of the tower). However, size of opening in the grid/plate are fixed in such a manner that fly off of the packing material can be prevented.

Example 9.1 Design a scrubber for absorbing acetone vapour from air–acetone vapour mixture by using pure water as solvent. The temperature in the scrubber is 25°C and scrubbing is isothermal. Operating pressure of scrubber is near atmospheric. A mixture of air with acetone vapour containing 6% by volume of acetone is passed through the scrubber. This mixture contains 1400 m^3/h of air. The scrubber is required to absorb 98% of the acetone.

Solution

Refer to Fig. 9.3.

Let G_s = Molar flow rate of air, kmol/h.

G_1 = Molar flow rate of incoming air-vapour mixture, kmol/h

y_1 = Mole fraction of solute (vapour) in incoming air-vapour mixture

$$Y_1 = \frac{y_1}{1 - y_1} \quad \frac{\text{kmol of vapour}}{\text{kmol of air}}$$

G_2 = Molar flow rate of outgoing air-vapour mixture, kmol/h
y_2 = Mole fraction of vapour in outgoing air-vapour mixture

$$Y_2 = \frac{y_2}{1 - y_2} \frac{\text{kmol of acetone}}{\text{kmol of air}}$$

x_2 = Mole fraction of solute in incoming solvent = 0

$$X_2 = \frac{x_2}{1 - x_2} = 0 \frac{\text{kmol of acetone}}{\text{kmol of water}}$$

$L_2 = L_s$ = Molar flow rate of solvent at top, entering the scrubber, kmol/h
L_1 = Molar flow rate of solution leaving the scrubber, kmol/h
x_1 = Mole fraction of acetone in liquid solution leaving the absorber

$$X_1 = \frac{x_1}{1 - x_1} \frac{\text{kmol of acetone}}{\text{kmol of water}}$$

Flow rate of air, q_s = 1400 m^3/h at 1 atm and 25°C

Density of air, $\rho_A = \dfrac{pM}{RT} = \dfrac{1 \times 29}{0.082\,06 \times (273 + 25)}$

$$\rho_A = 1.1859 \text{ kg/m}^3$$

$$G_s = \frac{1400 \times 1.1859}{29} = 57.25 \text{ kmol/h}$$

$$y_1 = 0.06 \text{ (mole \% = volume \%)}$$

$$Y_1 = \frac{y_1}{1 - y_1} = 0.063\,83 \frac{\text{kmol of acetone}}{\text{kmol of air}}$$

Scrubber is required to absorb 98% of acetone.

$$Y_2 = 0.02 \times Y_1 = 0.001\,277$$
$$X_2 = 0, \quad L_s = ?$$

L_s: To find the value of L_s, first the minimum amount of solvent required L_{sm} for the desired separation is to be determined.
Phase equilibrium equation

$$y = mx = \frac{p_v}{p_t} x$$

At 25°C, vapour pressure of acetone is given by

$$\ln p_v = 16.6513 - \frac{2940.46}{T - 34.93} \quad \text{(Antoine equation)}$$

where, p_v = Vapour pressure of acetone at 25°C, Torr
T = Temperature, K

$$\ln p_v = 16.6513 - \frac{2940.46}{(273 + 25) - 35.93}$$

$$p_v = 228.416 \text{ Torr}$$

$$m = \frac{228.416}{760} = 0.3$$

$$y = 0.3\,x \quad \text{and} \quad \frac{Y}{1+Y} = 0.3\,\frac{X}{1+X}$$

Table 9.5 Equilibrium Data Calculations

y	$Y = \dfrac{y}{1-y}$	$x = \dfrac{y}{m}$	$X = \dfrac{x}{1-x}$
0	0	0	0
0.0012	0.001 2014	0.004	0.004 016
0.01	0.010 1	0.033	0.034 12
0.02	0.020 4	0.067	0.071 8
0.03	0.030 9	0.1	0.111
0.04	0.041 67	0.133	0.153 4
0.05	0.052 63	0.167	0.2
0.06	0.063 83	0.2	0.25

Solute balance at minimum solvent rate

$$G_s(Y_1 - Y_2) = L_{sm}(X_{1m} - X_2)$$

$$57.22\,(0.063\,83 - 0.001\,277) = L_{sm}(X_{1m} - 0)$$

where, L_{sm} = Minimum amount of solvent required, kmol/h

X_{1m} = Value of X_1 corresponding to the minimum amount of solvent

Value of X_{1m} can be obtained from the graph. Here, the curve of Y vs X (equilibrium curve; Fig. 9.12) is concave downward, hence, the tangent is drawn from (X_2, Y_2) point and is allowed to intersect the horizontal line at Y_1.

Intersection point is (X_{1m}, Y_1)

$X_{1m} = 0.2304$ (from Fig. 9.12)

$57.22\,(0.063\,83 - 0.001\,277) = L_{sm}(0.2304 - 0)$

$L_{sm} = 15.535$ kmol/h

Actual amount of solvent should be the greatest of the following values:

1. $L_s = 1.5\,L_{sm} = 23.3$ kmol/h $\quad L_2 = 23.3 \times 18 = 419.4$ kg/h
2. $L_s = \dfrac{1 \times 1000}{18} = 55.55$ kmol/h, i.e. about 1 m^3/h (minimum flow rate required to facilitate the use of a centrifugal pump)
3. Let rise in temperature of solvent = 10°C per pass

   Heat of solution of acetone in water[3]

   $$\Delta H_s = -10\,467 \text{ kJ/kmol}$$

   Aceton absorbed = $G_S\,(Y_1 - Y_2)$

   = 3.58 kmol/h

   Heat generation on absorption, $\phi = 10\,467 \times 3.58$

   = 37 472 kJ/h

   ≡ 10.4089 kW

   $$\phi = \dot{m}_s\,C_L\,\Delta t$$

Fig. 9.12 *Determination of Minimum Amount of Solvent*

$$\dot{m}_s = 37\,472/(4.1868 \times 10)$$
$$= 895 \text{ kg/h}$$
$$L_{sm} = 895/18 = 49.722 \text{ kmol/h}$$

4. L_{sm} = Minimum wetting rate required to form a liquid film over all the packing But minimum wetting rate required can be calculated after finding the tower diameter. Tower diameter can be determined after deciding the actual amount of solvent. Hence, it requires trial and error calculations.

For the first trial let L_s = 55.55 kmol/h = 1000 kg/h; highest amongst first three.
Tower diameter required at top: Eq. (9.15):

$$L_w = \text{Mass velocity of liquid kg/(m}^2 \cdot \text{s)} = \frac{1000/3600}{(\pi/4)D^2}$$
$$= 0.2778/\left(\frac{\pi}{4}D^2\right)$$

Molar flow rate for gas-vapour mixture at the top

$$G_2 = G_s\,(1 + Y_2) = 57.25\,(1 + 0.001\,201\,44) = 57.3189 \text{ kmol/h}$$

Average molar mass of gas-vapour mixture at the top

$$M_{av} = \Sigma(M_i y_i)$$

$$M_{av} = 0.012 \times M \text{ of acetone} + 0.9988 \times M \text{ of air}$$

$$M_{av} = 0.0012 \times 58 + 0.9988 \times 29$$

$$M_{av} = 29.0348 \text{ kg/kmol}$$

$$G_w = \frac{(57.3189 \times 29.0348)/3600}{(\pi/4)D^2} = \frac{0.462}{(\pi/4)D^2} \text{ kg/(m}^2 \cdot \text{s)}$$

Density of vapour gas mixture at top

$$\rho_G = \frac{p M_{av}}{RT} = \frac{1 \times 29.0348}{0.082\,06 \times (273 + 25)} = 1.187 \text{ kg/m}^3$$

$$F_{LG} = \frac{L_w}{G_w}\sqrt{\frac{\rho_G}{\rho_L}} = \frac{0.2778}{0.462} \times \sqrt{\frac{1.187}{1000}} = 0.020\,72$$

From Fig. 9.5, $K_F = 0.2$

Let actual velocity of gas = 66% of folding velocity

$$\sqrt{\frac{K}{K_F}} \times 100 = 66\%$$

$$K = (0.66)^2 \times 0.2 = 0.087\,12$$

For $K = 0.087\,12$ and $F_{LG} = 0.020\,72$

Pressure drop = 60 mm H_2O/m of packing – acceptable (From Fig. 9.5)

Mass velocity of gas through tower

$$\psi = \frac{\text{Density of liquid}}{\text{Density of water}} = 1, \quad \rho_L = 1000 \text{ kg/m}^3$$

$$g = 9.81 \text{ m/s}^2$$

Viscosity of water at 20°C, $\mu_L = 1$ cP $= 10^{-3}$ kg/(m · s)

Select 25 mm polypropylene Pall rings as packing,

Packing factor $F_P = 170 \text{ m}^{-1}$ (Ref. Table 9.1)

$$G_w = \left(\frac{0.087\,12 \times 1.187 \times 1000 \times 9.81}{170 \times 1 \times 1^{0.2}}\right)^{1/2}$$

$$= 2.4428 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\text{Tower area required at top} = \frac{\text{Mass flow rate of gas-vapour mixture}}{G_w}$$

$$= \frac{(57.3189 \times 29.0348)/3600}{2.4428} = 0.1892 \text{ m}^2$$

Tower diameter required at top = 0.49 m ≈ 0.5 m ≡ 500 mm

$$\frac{\text{Tower diameter}}{\text{Packing size}} = \frac{500}{25} = 20 > 10 \quad \text{(satisfactory)}$$

$$\text{Liquid rate} = \frac{1}{\frac{\pi}{4}(0.50)^2} = 5.09\ \text{m}^3/(\text{m}^2 \cdot \text{h})$$

Hence, it is greater than 4 $m^3/(m^2 \cdot h)$ (minimum wetting rate required for polypropylene surface). Thus condition No. 4 (p. 680) is satisfied.

$$\text{Liquid rate} = \frac{1000}{\frac{\pi}{4}(0.50)^2} = 5093\ \text{kg}/(\text{h} \cdot \text{m}^2)$$

It is less than 7340 $kg/(h \cdot m^2)$, minimum wetting rate specified in literature (Ref. 2).
Based on Morris and Jackson's equation, minimum solvent required

$$\text{MWR} = R \cdot a_t \tag{9.5c}$$

For 25 mm polypropylene Pall rings $a_t = 205\ m^2/m^3$ (From Table 9.1)
For 25 mm polypropylene Pall rings $R = 0.08\ m^2/h$

$$L_{sm} = 0.08 \times 205 = 16.4\ \text{m}^3/(\text{m}^2 \cdot \text{h})$$

Considering the last method for predicting minimum wetting rate,
let L_s = 3500 kg/h = 194.4 kmol/h

At top $$F_{LG} = \frac{3500/3600}{0.462} \times \sqrt{\frac{1.187}{1000}} = 0.0725$$

From Fig. 9.5, $K_F = 0.17$
Let actual velocity = 66% of flooding velocity

$$K = (0.66)^2 \times 0.17 = 0.074$$

Pressure drop = 50 mm H_2O/m of packing
Mass velocity of gas through tower

$$G_w = \left(\frac{0.074 \times 1.187 \times 1000 \times 9.81}{170 \times 1 \times 1^{0.2}}\right)^{1/2}$$

$$= 2.2514\ \text{kg}/(\text{m}^2 \cdot \text{s})$$

Tower area required at top, $A_1 = \dfrac{0.462}{2.2514}$

$$= 0.2052\ \text{m}^2 = \left(\frac{\pi}{4}\right) D^2$$

$$D = 0.511\ \text{m} \approx 0.52\ \text{m}$$

$$\text{Liquid rate} = \frac{3500}{\frac{\pi}{4}(0.52)^2} = 16\,480.54\ \text{kg}/(\text{m}^2 \cdot \text{h}) \equiv 16.48\ \text{m}^3/(\text{m}^2 \cdot \text{h})$$

Hence, it satisfies the conditions.
Tower diameter required at the bottom:
Average molar mass of gas-vapour mixture

$$M_{\text{av}} = \Sigma M_i y_i = y_1 \times 58 + (l - y_1) \times 29 = 0.06 \times 58 + (1 - 0.06) \times 29$$

$$= 30.74\ \text{kg/kmol}$$

Density of gas-vapour mixture at bottom

$$\rho_{G1} = \frac{PM_{\text{av}}}{RT} = \frac{1 \times 30.74}{0.082\,06 \times (273 + 25)} = 1.257 \text{ kg/m}^3$$

For $L_s = 3500$ kg/h = 194.44 kmol/h

Acetone balance

$$G_s(Y_1 - Y_2) = L_s(X_1 - X_2)$$

$$57.25(0.063\,83 - 0.001\,277) = 194.44(X_1 - 0)$$

$$X_1 = 0.018\,42$$

$$x_1 = \frac{X_1}{1 + X_1} = 0.0181$$

Density of water = 1000 kg/m^3

Density of acetone = 790 kg/m^3

Average density of liquid solution leaving the scrubber

$$\rho_{L_1} = \frac{1}{\sum (w_i/\rho_i)}$$

$$w_1 = x_1 \times 58/[x_1 \times 58 + (1 - x_1) \times 18]$$

$$= \frac{0.0181 \times 58}{0.0181 \times 58 + 0.9819 \times 18} = 0.056 \quad \text{(acetone)}$$

$$\rho_{L_1} = \frac{1}{\dfrac{0.056}{790} + \dfrac{(1 - 0.056)}{1000}} = 985.33 \text{ kg/m}^3$$

$$L_1 = L_s(1 + X_1) = 194.44(1 + 0.018\,42) = 198.025 \text{ kmol/h}$$

Average molar mass of liquid solution

$$M_{\text{av}} = 0.0181 \times 58 + 0.9819 \times 18 = 18.724$$

$$L_{1W} = \frac{(198.02 \times 18.724)/3600}{(\pi/4)D^2} = \frac{1.03}{(\pi/4)D^2} \text{ kg/(m}^2 \cdot \text{s)}$$

$$G_1 = G_s(1 + Y_1) = 57.25(1 + 0.063\,83) = 60.904 \text{ kmol/h}$$

$$G_{1_w} = \frac{(60.904 \times 30.74)/3600}{(\pi/4)D^2} = \frac{0.52}{(\pi/4)D^2} \text{ kg/(m}^2 \cdot \text{s)}$$

At bottom, $$F_{LG} = \frac{L_{1_w}}{G_{1_w}} \sqrt{\frac{\rho_{G_1}}{\rho_{L_1}}} = \frac{1.03}{0.52} \times \sqrt{\frac{1.257}{985.33}} \tag{9.15}$$

$$= 0.0707$$

$$K_F = 0.175 \qquad \text{(From Fig. 9.5)}$$

Let actual velocity of gas = 66% of flooding velocity

$$K = (0.66)^2 \times 0.175 = 0.076\,23$$

Δp of packing $\cong$ 50 mm of H_2O/m of packing

Mass velocity of gas through tower

$$G_{1_w} = \left(\frac{K\,\rho_{G_1}\rho_{L_1}g}{F_p\,\psi\,\mu_L^{0.2}}\right)^{1/2} = \left(\frac{0.076\,23 \times 1.257 \times 985.33 \times 9.81}{170 \times \left(\frac{1000}{985.33}\right) \times 1^{0.2}}\right)^{1/2}$$

$$= 2.316 \text{ kg/(m}^2 \cdot \text{s)}$$

Tower area required at bottom $A = \dfrac{0.52}{2.316} = 0.2245 \text{ m}^2$

$$\frac{\pi}{4}D^2 = 0.2245 \text{ m}^2$$

$$D = 0.5346 \text{ m}$$

Thus the diameter of the entire tower, $D \approx 0.54$ m

N_{OG}: (Number of overall gas phase transfer units)

To calculate the N_{OG}, draw the operating line on the graph which starts from (X_2, Y_2) and terminates at (X_1, Y_1), i.e., starts from (0, 0.001 277) and terminates at (0.018 43, 0.06 383). (Ref. Fig. 9.12)

Given system is a dilute system ($x_1 < 0.1$). Here the portion of equilibrium curve under the operating line can be approximated as straight line. Hence, in the given case N_{OG} can be calculated by the following equation:

$$N_{OG} = \frac{1}{1-\left(\frac{m\,G_m}{L_m}\right)} \ln\left[\left(1-\frac{m\,G_m}{L_m}\right)\frac{y_1}{y_2} + \frac{m\,G_m}{L_m}\right] \tag{9.12}$$

$$\frac{m\,G_m}{L_m} = 0.3 \times \frac{\left(\frac{G_1+G_2}{2}\right)\Big/\left(\frac{\pi}{4}D^2\right)}{\left(\frac{L_1+L_2}{2}\right)\Big/\left(\frac{\pi}{4}D^2\right)} = 0.3 \times \frac{\left(\frac{60.904+57.3189}{2}\right)}{\left(\frac{198.025+194.45}{2}\right)}$$

$$= 0.090\,38$$

$$N_{OG} = \frac{1}{1-0.090\,38} \ln\left[(1-0.090\,38) \times \frac{0.06}{0.0012} + 0.090\,38\right]$$

$$= 4.1988$$

Here, acetone is highly soluble in water. Hence, liquid film resistance is negligible as compared to the gas film resistance. Hence, height of packing in the given case should be determined by equation,

$$Z = N_{OG} \cdot H_{OG} \tag{9.7}$$

H_{OG}: Height of Overall Gas Phase Transfer Unit

$$H_{OG} = H_G + m\frac{G_m}{L_m}H_L \tag{9.19}$$

Based on Onda, Takeuchi and Okumoto's method,

Height of gas phase transfer unit

$$H_G = \frac{G_w}{K_G a_e p\, M_g} \tag{9.21}$$

G_w = Superficial mass velocity of gas, kg/(m^2 · s)

$$= \frac{(G_{1w} + G_{2w})/2}{\frac{\pi}{4}(D_i^2)^2} = \frac{(0.462 + 0.52)/2}{\frac{\pi}{4}(0.540)^2} = 2.1439 \text{ kg/(m}^2 \cdot \text{s)}$$

Effective area of mass transfer a_e is determined by following equation:

$$\frac{a_e}{a_t} = 0.31\frac{\sigma_L^{0.5}}{Z^{0.4}}(C_{a_L} \cdot R_{eG})^{0.392} \tag{9.23}$$

a_t = 205 m^2/m^3

Surface tension data for acetone at the scrubbing temperature can be calculated by a group-contribution method, given in Ref. 3.

σ_L = Liquid surface tension in $\frac{\text{mN}}{\text{m}}$ or $\frac{\text{dyn}}{\text{cm}}$

$$x_i = x_{\text{av}} = \frac{x_1 + x_2}{2} = \frac{0.0181 + 0}{2} = 9.05 \times 10^{-3} = 0.0095$$

$[\mathcal{P}_i]$ for Acetone: $(CH_3—\overset{\overset{\displaystyle O}{||}}{C}—CH_3)$

$[\mathcal{P}_i] = 55.5 \times 2 + (9 + 22.3) = 141.7$ (From Table 3.343 of Ref. 3)

Surface tension of Acetone

$\sigma^{1/4} = [\mathcal{P}_i](\rho_L - \rho_G)$ (Ref. Eq. 3.151 of Ref. 3)

Density of pure acetone vapour, $\rho_G = \frac{1 \times 58}{0.082057 \times (273 + 25)} = 2.372$ kg/m^3

where, ρ_L, ρ_G are in mol/cm^3

$$\sigma^{1/4} = 141.7\left[\frac{(790 - 2.372) \times 10^{-3}}{58}\right]$$

$= 1.9242$

σ = 13.71 dyn/cm

Actual surface tension data are reported by Timmermans (Ref. 8).

σ = 24.5 dyn/cm at 25°C

Thus it can be seen that group-contribution method may give erroneous results. It is advised to use the actual value as far as possible.

For pure water, σ = 70 dyn/cm

For liquid mixture, using Eq. (7.37),

$$\sigma_L^{1/4} = \Sigma(\sigma_i^{1/4} x_i) = 24.5^{1/4} \times 0.009\,05 + (1 - 0.009\,05)70^{1/4}$$

σ_L = 69.58 dyn/cm or mN/m

For dilute aqueous solution, surface tension of water may be taken for calculations.

$$C_{aL} = \text{Liquid capillary number} = \frac{\mu_L L_W}{\rho_L \sigma_L} \tag{9.24}$$

$x_{av} = 0.009\ 05$

$W_{av} = (0.009\ 05 \times 58)/(0.009\ 05 \times 58 + (1 - 0.009\ 05) \times 18)$

$W_{av} = 0.028\ 58$ (average mass fraction)

$$\rho_L = \frac{1}{\Sigma(w_i/\rho_i)} = \frac{1}{\dfrac{0.028\ 58}{790} + \dfrac{(1 - 0.028\ 58)}{1000}} = 992.46 \text{ kg/m}^3$$

$\sigma_L = 69.58 \times 10^{-3}$ N/m

$$L_w = \frac{(L_{1w} + L_{2w})/2}{(\pi/4)(D_i^2)}$$

$$= \frac{(3500/3600 + 1.03)/2}{(\pi/4)(0.54)^2} = 4.3712 \text{ kg/(m}^2 \cdot \text{s)}$$

μ_L = Viscosity of liquid mixture in kg/(m · s)

$\ln \mu_{mix} = \Sigma(w_i \ln \mu_i)$ (Eq. 3.107 of Ref. 3)

Viscosity of acetone at 25°C, $\mu_{ac} = 0.33 \times 10^{-3}$ kg/(m · s)

Viscosity of water at 25°C, $\mu_w = 0.95$ cP $= 0.95 \times 10^{-3}$ kg/(m · s)

$\ln \mu_L = 0.028\ 58 \ln (0.33 \times 10^{-3}) + (1 - 0.028\ 58) \ln (0.95 \times 10^{-3})$

$\mu_L = 9.217 \times 10^{-4}$ kg/(m · s)

$$C_{aL} = \frac{9.217 \times 10^{-4} \times 4.3712}{992.46 \times 69.58 \times 10^{-3}}$$

$$= 5.834 \times 10^{-5}$$

Reynolds number:

$G_W = 2.1439$ kg/(m^2 · s), $a_t = 205$ m^2/m^3

Viscosity of gas mixture:

$$\mu_G = \sum_{i=1} \frac{(y_i \mu_i)}{\sum_{j=1} (y_i \Theta_{ij})} \tag{6.77}$$

i represents acetone while *j* represents air.

$$y_{av} = \frac{y_1 + y_2}{2} = \frac{0.06 + 0.0012}{2} = 0.0306$$

$$\Theta_{ij} = \frac{\left[1 + (\mu_i/\mu_j)^{1/2} (M_j/M_i)^{1/4}\right]^2}{\left[8(1 + M_i/M_j)\right]^{1/2}}$$

and $$\Theta_{ji} = \left(\frac{\mu_j}{\mu_i}\right)\left(\frac{M_i}{M_j}\right)\Theta_{ij}$$

Viscosity of acetone vapour, $\mu_{ac} = 750 \times 10^{-7}$ P $= 750 \times 10^{-5}$ cP
Viscosity of air $\mu_{air} = 1850 \times 10^{-7}$ P $= 1850 \times 10^{-5}$ cP

$$\Theta_{ij} = \frac{\left[1 + \left(\frac{750}{1850}\right)^{1/2}\left(\frac{29}{58}\right)^{1/4}\right]^2}{[8(1 + 58/29)]^{1/2}} = 0.4812$$

$$\Theta_{ji} = \left(\frac{1850}{750}\right)\left(\frac{58}{29}\right) \times 0.4812 = 2.374$$

$$\mu_G = \frac{0.0306 \times 750 \times 10^{-5} + (1 - 0.0306) \times 1850 \times 10^{-5}}{(1 - 0.0306) \times 0.4812 + 0.0306 \times 2.374}$$

$$= 0.033\,69 \text{ cP} \equiv 0.033\,69 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$Re_G = \frac{6G}{a_t \mu_G} = \frac{6 \times 2.1439}{205 \times 0.033\,69 \times 10^{-3}} = 1862$$

$$Z = N_{OG} \cdot H_{OG} = 4.1988\, H_{OG}$$

$$\frac{a_e}{a_t} = 0.31 \frac{\sigma_L^{0.5}}{Z^{0.4}} (C_{aL}\, Re_G)^{0.392} \tag{9.23}$$

$$\frac{a_e}{205} = 0.31 \frac{69.58^{0.5}}{Z^{0.4}} (5.834 \times 10^{-5} \times 1862)^{0.392}$$

$$= \frac{1.0832}{Z^{0.4}}$$

For the first trial calculations, let $H_{OG} = 0.6$ m
Approximate values of H_{OG} for random packings of size 25 mm is 0.3 to 0.6 m, for 38 mm size it is 0.5 to 0.75 m and for 50 mm size it is 0.6 to 1.0 m (Ref. 2).

$$Z = 4.1988 \times 0.6 = 2.52 \text{ m}$$

$$\frac{a_e}{205} = \frac{1.0832}{2.52^{0.4}} = 0.748\,42$$

$$a_e = 153.43 \text{ m}^2/\text{m}^3 \text{ of packing}$$

Gas phase transfer coefficient K_G is determined by Eq. (9.26).
where, $R = 8314$ J/(kmol · K)

T = operating temperature = 273 + 25 = 298 K

$$M_{av} = \Sigma y_i M_i = 0.0306 \times 58 + (1 - 0.0306) \times 29 = 29.8874$$

$$\rho_G = \frac{p\, M_{av}}{RT},$$

$$= \frac{1 \times 29.8874}{0.082\,057 \times 298} = 1.2222 \text{ kg/m}^3$$

D_G = Gas phase diffusion coefficient, m^2/s

From Table 3.318, of Ref. 3,
At 25°C diffusivity of acetone in air

$$D_G = 0.109 \text{ cm}^2/\text{s} = 0.109 \times 10^{-4} \text{ m}^2/\text{s}$$

$$D_G \propto T^{1.75}$$

$$D_G = 0.109 \times 10^{-4} \times \left(\frac{298}{273}\right)^{1.75} = 1.2706 \times 10^{-5} \text{ m}^2/\text{s}$$

$$C_1 = 5.23 \text{ for } d_p > 12 \text{ mm}$$

$$K_G \times \frac{8314 \times 298}{205 \times 1.2706 \times 10^{-5}} = 5.23\left(\frac{2.1439}{205 \times 0.033\,69 \times 10^{-3}}\right)^{0.7} \times \left(\frac{0.03369 \times 10^{-3}}{1.2222 \times 1.2706 \times 10^{-5}}\right)^{1/3} \times (205 \times 0.025)^{-2}$$

$$K_G \times 9.511\,82 \times 10^8 = 5.23 \times 55.509 \times 1.2945 \times 0.038\,07.$$

$$K_G = 1.504 \times 10^{-8} \text{ kmol}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$$

Height of gas phase transfer unit, Eq. (9.21):

G_W = Average superficial mass velocity of gas, kg/(m^2 · s)
$G_W = 2.1435$ kg/(m^2 · s)
$K_G = 1.504 \times 10^{-8}$ kmol/(m^2 · s · Pa)
$a_e = 153.43$ m^2/m^3
p = operating pressure = 101.325×10^3 Pa
M_g = average molar mass of gas = 29.8874 kg/mol

$$H_G = \frac{2.1439}{1.504 \times 10^{-8} \times 153.43 \times 101.325 \times 10^3 \times 29.8874}$$

$$= 0.3068 \text{ m}$$

Height of liquid phase transfer unit, Eq. (9.22)

L_W = Average mass velocity of liquid through tower

$$= \frac{[(3500/3600) + 1.03]/2}{\pi/4(0.54)^2} = \frac{8.7425}{2} = 4.3712 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$a_e = 153.41 \text{ m}^2/\text{m}^3, \quad \rho_L = 992.46 \text{ kg/m}^3$$

$$H_L = \frac{4.3712}{K_{LC} \times 153.41 \times 992.46} = \frac{2.871 \times 10^{-5}}{K_{LC}}$$

Liquid phase mass transfer coefficient K_{LC} can be determined by Eq. (9.27).

$\rho_L = 992.46$ kg/m^3, $\mu_L = 9.217 \times 10^{-4}$ kg/(m · s)
$L_W = 4.3712$ kg/(m^2 · s), $g = 9.81$ m/s^2, $a_t = 205$ m^2/m^3
$d_p = 0.025$ m
a_w = wetted surface of packing, m^2/m^3

$$\frac{a_w}{a_t} = 1 - \exp\left(-1.45\left(\frac{\sigma_C}{\sigma_L}\right)^{0.75} Re_L^{0.1} Fr_L^{-0.05} We_L^{0.2}\right) \tag{9.28}$$

For polypropylene packing, $\sigma_c = 33 \times 10^{-3}$ N/m (Table 9.3)

$\sigma_L = 69.58 \times 10^{-3}$ N/m

Liquid Reynolds number, $Re_L = \dfrac{L_W}{a_t \mu_L} = \dfrac{4.3712}{205 \times 9.217 \times 10^{-4}}$

$= 23.134$

Froude number of liquid, $Fr_L = L_W^2 a_t / (\rho_L^2 g)$

$$= \frac{4.3712^2 \times 205}{992.46^2 \times 9.81} = 4.0538 \times 10^{-4}$$

Weber number for liquid,

$$We_L = L_W^2 / (\rho_L \, \sigma_L \, a_t) = \frac{4.3712^2}{992.46 \times 69.58 \times 10^{-3} \times 205} = 1.35 \times 10^{-3}$$

$$\frac{a_w}{a_t} = 1 - \exp\left[-1.45\left(\frac{33}{69.58}\right)^{0.75} \times 23.134^{0.1} \times (4.0538 \times 10^{-4})^{-0.05} \times (1.35 \times 10^{-3})^{0.2}\right]$$

$$\frac{a_w}{a_t} = 1 - \exp(-0.4472)$$

$$\frac{a_w}{a_t} = 0.3606$$

$a_w = 0.3606 \times 205 = 73.923$ m^2/m^3

D_L = Diffusivity of acetone in water, m^2/s

D_L can be determined by Wilke and Chang equation.

$$D_L = \frac{(117.3 \times 10^{-18})(\phi_{as} M_s)^{0.5} T}{\mu \, V_A^{0.6}} \quad \text{(Equation 2.44, of Ref. 1)} \qquad (9.33)$$

where, V_A = Molar volume of solute at normal boiling point, m^3/kmol

ϕ_{as} = Association factor for solvent = 2.26 for water (a parameter)

M_s = Molar mass of solvent = 18 kg/kmol

μ = Solution viscosity = 9.217×10^{-4} kg/(m · s)

T = Temperature, K = 273 + 25 = 298 K

For calculating V_A, a group contribution method, given in Treybal is recommended. From Table 2.3 of Ref. 1

Table 9.6 Group Contributions for Diffnsivity of Acetone[1]

Atom	Volume, m^3/kmol	Total No. of atoms
C	0.0148	3
H	0.0037	6
O	0.0074	1

$$V_A = 0.0148 \times 3 + 0.0037 \times 6 + 0.0074 \times 1 = 0.074 \text{ m}^3/\text{kmol}$$

$$D_L = \frac{(117.3 \times 10^{-18}) \times (2.26 \times 18)^{0.5} \times 298}{9.217 \times 10^{-4} \times (0.074)^{0.6}} = 1.154 \times 10^{-9} \text{ m}^2/\text{s}$$

$$K_{LC}\left(\frac{992.46}{9.217 \times 10^{-4} \times 9.81}\right)^{1/3} = 0.0051\left(\frac{4.3712}{73.923 \times 9.217 \times 10^{-4}}\right)^{2/3}$$

$$\times \left(\frac{9.217 \times 10^{-4}}{992.46 \times 1.154 \times 10^{-9}}\right)^{-0.5} \times (205 \times 0.025)^{0.4}$$

$$K_{LC} \times 47.88 = 0.0051 \times 16.02 \times 0.03525 \times 1.9225$$

$$K_{LC} = 1.1564 \times 10^{-4} \text{ m/s}$$

$$H_L = \frac{2.871 \times 10^{-5}}{1.1564 \times 10^{-4}}$$

$$= 0.248 \text{ m}$$

$$H_{OG} = H_G + m\frac{G_m}{L_m}H_L \tag{9.19}$$

$$= 0.3068 + 0.09038 \times 0.248 = 0.329 \text{ m}$$

$$Z = N_{OG} \cdot H_{OG} = 4.1988 \times 0.329 = 1.38 \text{ m}$$

For second trial calculations, let $Z = 1.4$ m

$$a_e = \frac{1.0832 \times 205}{1.4^{0.4}} = 194 \text{ m}^2/\text{m}^3$$

$$H_G = 0.3068 \times \frac{153.43}{194} = 0.2426 \text{ m}$$

and $$H_L = 0.248 \times \frac{153.43}{194} = 0.196 \text{ m}$$

$$H_{OG} = 0.2426 + 0.090\,38 \times 0.196 = 0.2603 \text{ m}$$

$$Z = 4.1988 \times 0.2603 = 1.093 \text{ m} < 1.382 \text{ m}$$

H_{OG} for 25 mm size random packing ranges from 0.3 to 0.6 m.

Let $H_{OG} = 0.3$ m

$$Z = 0.3 \times 4.1986 = 1.26 \text{ m} \approx 1.3 \text{ m}$$

This is the value of packed bed height based on Onda, Takeuchi and Okumoto's method.

Cornell's method

For Pall rings, height of gas phase transfer unit [by Eq. (9.29)]

ψ_c = Correlation parameter = 60 m for metal Pall rings (Fig. 9.6)

For polypropylene Pall rings, data for ψ_c are not available. Hence, assume that the values of ψ_c for polypropylene Pall rings and ψ_c for metal Pall rings are same.

For PP Pall rings $\psi_c = 60$ m

$D = 0.54$ m, $Z = 1.3$ m (Assume for the first trial calculations)

$$Sc_G = \frac{\mu_G}{\rho_G D_G} = \frac{0.03369 \times 10^{-3}}{1.2222 \times 1.2706 \times 10^{-5}}$$

$$= 2.1695$$

$$f_1 = \left(\frac{\mu_L}{\mu_w}\right)^{0.16} \quad \text{with } \mu_w = 10^{-3}\ \text{Pa} \cdot \text{s}$$

$$= \left(\frac{9.217 \times 10^{-4}}{10^{-3}}\right)^{0.16} = 0.987$$

$$f_2 = \left(\frac{\rho_w}{\rho_L}\right)^{1.25} \quad \text{with } \rho_w = 1000\ \text{kg/m}^3$$

$$= \left(\frac{1000}{992.46}\right)^{1.25} = 1.01$$

$$f_3 = \left(\frac{\sigma_w}{\sigma_L}\right)^{0.8} \quad \text{where } \sigma_w = 72.8\ \text{mN/m}$$

$$= \left(\frac{72.8}{69.49}\right)^{0.8} = 1.038$$

$$L_w = \text{Liquid phase mass velocity} = 4.3712\ \text{kg/(m}^2 \cdot \text{s)}$$

$$H_G = \frac{0.017 \times 60 \times 0.54^{1.24} \times 1.3^{0.33} \times 2.1695^{0.5}}{(4.3712 \times 0.987 \times 1.01 \times 1.038)^{0.6}} = 0.3085\ \text{m}$$

Height of liquid phase transfer unit, can be determined by Eq. (9.31)

$$H_L = \frac{\phi_{CO} C}{3.28}\left(\frac{\mu_L}{\rho_L D_L}\right)^{0.5}\left(\frac{Z}{3.05}\right)^{0.15}$$

From Fig. 9.7, for $L = 4.3712$ kg/(m^2 · s),
Correlation parameter ϕ_{CO} for metal Pall rings = 0.05 m
Assume that ϕ_{CO} for PP Pall rings is ϕ_{CO} for *SS* Pall ring.

$$\phi_{CO} = 0.05\ \text{m}$$

$$C = 0.75 \text{ for 66\% Flooding} \quad \text{(From Fig. 9.8)}$$

$$H_L = \frac{0.05 \times 0.75}{3.28}\left(\frac{9.217 \times 10^{-4}}{992.46 \times 1.154 \times 10^{-9}}\right)^{0.5} \times \left(\frac{1.3}{3.05}\right)^{0.15}$$

$$= 0.2854\ \text{m}$$

$$H_{OG} = H_G + m\frac{G_m}{L_m}H_L = 0.3085 + 0.09038 \times 0.2854$$

$$= 0.334\ \text{m}$$

Height of packed bed, $Z = N_{OG} \cdot H_{OG}$

$$= 4.1988 \times 0.334 \text{ m} = 1.4 \text{ m}$$

For second trial calculations, let $Z = 1.4$ m

$$H_G = 0.3058 \times \left(\frac{1.4}{1.3}\right)^{0.33} = 0.3134 \text{ m}$$

$$H_L = 0.2854 \times \left(\frac{1.4}{1.3}\right)^{0.15} = 0.2856 \text{ m}$$

$$H_{OG} = 0.3134 + 0.090\,38 \times 0.2856 = 0.3392 \text{ m}$$

$$Z = 4.1988 \times 0.3392 = 1.424 \text{ m}$$

Let height of packed bed $Z = 1.5$ m (higher than the values of Z obtained based on Cornell's method as well as based on Onda, Takeuchi and Okumoto's method.)
No. of packing sections required = one

Liquid distributor

For clean solvent and for small diameter tower perforated pipe-type distributor is satisfactory. It is recommended that one liquid stream must be provided for each 194 cm^2 area (Ref. 3).
No. of holes (distribution points) required in pipe type distributor,

$$n_h = \frac{\text{Cross-sectional area of tower, cm}^2}{194}$$

$$n_h = \frac{(\pi/4)(0.54)^2 \times 10^4}{194} = 11.8, \text{ say } 12$$

Let the velocity of water through the pipe = 2 m/s

Flow rate of water (solvent) = 3500 kg/h

Volumetric flow rate of solvent, $q_v = \dfrac{3500}{1000} \times \dfrac{1}{3600} = 9.7222 \times 10^{-4}$ m^3/s

$$d = \sqrt{\frac{(9.722 \times 10^{-4}) \times 4}{2 \times \pi}} = 0.025 \text{ m}$$

$$= 25 \text{ mm} \quad \text{(size of pipe)}$$

Let the velocity of solvent through holes = 3 m/s

Let d_h = diameter of hole, m

$$12 \times \frac{\pi}{4} d_h^2 = \frac{9.7222 \times 10^{-4}}{3}$$

$$d_h = 0.006 \text{ m} \equiv 6 \text{ mm}$$

Resulting pipe-type liquid distributor is shown in Fig. 9.12.

Fig. 9.13 *Pipe-Type Liquid Distributor*

Packing support

Packing support should be selected such that flow area provided by packing support for the flow of gas should be greater than flow area (i.e., void) provided by packing material.

% Void space for 25 mm size polypropylene Pall rings = 90% (Ref. Table 9.1)

With Pall rings any gas-injection-type packing support can be used.

Let type of packing support: Cap-type packing support

Actual outer diameter of packing support is greater than 540 mm as some portion of packing support is sandwiched between two flanges.

Let h' = height of slot in riser

x = width of slot in riser

d = diameter of riser

n = number of risers

D_C = Inside diameter of column = 540 mm

If $d = D_C/6$, then nine number of risers can be provided on packing support.

$$= \frac{540}{6} = 90 \text{ mm}$$

Let, n_s = number of slots per riser

Total area of slots of risers = $n_s \cdot n\, h'\, x$

The distance between two adjacent slots is x mm

Let $x = 10$ mm

$$n_s = \frac{\pi d}{2x} = \frac{\pi \times 90}{2 \times 10} \cong 14$$

Total area of slots of risers = $14 \times 9 \times h' \times 10 \geq 0.9 \times \frac{\pi}{4}\,(540)^2$

$h' \geq 163.58$ mm
Consider $h' = 170$ mm
Let velocity of liquid through weep hole = 0.5 m/s
Let number of weeping holes = 34

$$34 \times \frac{\pi}{4} (d_w)^2 = 9.733 \times 10^{-4}/0.5$$

$d_w = 8.5$ mm ≈ 9 mm
Refer to Fig. 9.14 for packing support plate details.

Fig. 9.14 *Cap-Type Packing Support*

9.4 ABSORPTION WITH CHEMICAL REACTION

Many industrial absorption systems involve chemical reaction. Majority of pollution control towers are accompanied by chemical reaction. In absorption with chemical reaction, a chemical reagent is added in solvent which reacts with solute gas of feed gas mixture. If recovery of solute gas or solvent is essential then reaction should be reversible. Otherwise in most of the cases it is irreversible. For example, if CO_2 from the feed gas mixture is required to be separated in pure form then mono ethanol amine is selected as reagent as it reacts with CO_2 by reversible reaction. Otherwise NaOH can be selected as reagent if CO_2 recovery is not required.

Absorption with chemical reaction enhances the rate of absorption considerably. It converts the absorbed solute into other compound and hence, increases driving force for mass transfer. It also increases liquid phase mass transfer coefficient by a magnitude which contributes in increasing absorption rates.

Process design of packed tower type absorber involving chemical reaction includes following steps of calculations:

1. From the given feed gas flow rate and composition, Find G_1, G_s, G_{1w} and y_1. where,

   G_1 = Molar flow rate of feed gas mixture, kmol/h
   G_s = Molar flow rate of non-solute gas mixture, kmol/h
   y_1 = Mole fraction of solute in feed gas
   G_{1w} = Mass flow rate of feed gas mixture, kg/h

2. Fix the value of y_2, mole fraction of solute in exit gas. Decrease in the value of y_2 increases fixed cost and operating cost of absorber. In case of pollution control tower y_2 should be fixed based on the maximum permissible concentration in the vent gas, limited by pollution control norms.
3. Find the minimum amount of solvent L_{sm}, required for the desired separation. In this case L_{sm} is determined based on the stoichiometry of the chemical reaction.
4. Find the actual amount of solvent L_s. Actual amount of solvent L_s should be the greatest among the following values:
   (a) $L_s = 1.5\ L_{sm}$
   (b) $L_s \geq 1\ m^3/h$ to facilitate the use of centrifugal pump. Metering pump gives pulsating flow.
   (c) Actual wetting rate should be greater than minimum wetting rate required for packing (MWR). Value of MWR depends on type and MOC of packing. For example, MWR for polypropylene Pall ring is 4 $m^3/(h \cdot m^2)$.

   $$\text{Actual wetting rate} = \frac{L_{sw}/\rho_L}{\frac{\pi}{4} D_i^2} \tag{9.34}$$

   where, L_{sw} = Mass flow rate of solvent, kg/s
   ρ_L = Density of liquid solvent, kg/s
   D_i = Inside diameter of tower, m

   (d) L_s should be such that per pass rise in temperature of solvent is 5 to 10°C. Accompanying chemical reaction could be exhothermic in nature. Hence, if solvent is to be recirculated, a cooler is required in recirculation line of solvent to remove exhothermicity of reaction and to control the temperature of solvent.
5. Find the inside diameter of absorption tower. Method for finding tower diameter is same as that for physical absorption. Actual wetting rate can be calculated after finding D_i. Hence, for the first trial calculation L_s should be greatest of three values given by (a), (b) and (d). Value of (c) can be verified at the end of trial calculation. Thus calculations for finding L_s and D_i require trial-and-error calculations.
6. Find the number of overall gas phase transfer units.

$$N_{OG} = \int_{y_2}^{y_1} \frac{dy}{y - y_e} \tag{9.35a}$$

With irreversible reaction and excess of chemical reagent , $y_e = 0$.

$$N_{OG} = \ln\left(\frac{y_1}{y_2}\right) \tag{9.35b}$$

7. Find the height of overall gas phase transfer unit, H_{OG}.

$$H_{OG} = \frac{G_1 / \left(\frac{\pi}{4} D_i^2\right)}{K_G a} \tag{9.36}$$

where, H_{OG} = Height of overall gasphase transfer unit, m
G_1 = Molar flow rate of feed gas, kmol/s
D_i = Inside diameter of tower, m
$K_G a$ = Gas phase mass transfer coefficient, kmol/(h · m^3)

Table 9.7 Recommended Values of $K_G a$[3]

Gas phase reactant	Liquid phase reactant	$K_G a$, kmol/(h · m^3)
Cl_2	8% NaOH solution	272
SO_2	11% Na_2CO_3 solution	229
Br_2	5% NaOH solution	131
H_2S	4% NaOH solution	96
CO_2	4% NaOH solution	38
HCl*	H_2O	353
NH_3*	H_2O	337
HF*	H_2O	152
HCN*	H_2O	114
HCHO*	H_2O	114
HBr*	H_2O	59

*Physical absorption

These values of $K_G a$ are derived for operating conditions: 1 atm, 16–24°C, 38 mm Intelox saddles, Gas rate = 1.3 kg/(m^2 · s), Packed height = 3.05 m, Tower diameter = 0.76 m, Solute gas concentration = 0.5 to 1% (by mole)

Ideally these values of $K_G a$ can be used in the given case if

1. Solute concentration ≥ 0.5 %
2. Operating pressure ≥ 1 atm
3. Operating temperature ≤ 24°C
4. Tower diameter ≤ 0.76 m
5. Packing size ≤ 38 mm
6. Type of packing: Intelox saddles or better packings like Pall-rings, structured packing, etc.
7. Superficial mass flow rate of gas ≥ 1.3 kg/(s · m^2)

If one (or more) conditions are not fulfilled in the given case, then use of this value of $K_G a$ may result in underdesign of absorption unit.

$K_G a$ can also be derived from the performance data of commercial gas absorption tower working on the same system. Derived value of $K_G a$ from the performance of commercial tower can be used for the new design, if in the new case all conditions related to absorption are similar or better. Even today, most commercial gas absorption units are designed primarily on the basis of prior commercial experience.

8. Height of packing of packed tower

$$Z = N_{OG} \cdot H_{OG} \tag{9.38}$$

   where Z = Height of packing, m
9. Decide number of packing sections. Height of each packing section should be less than 3 m to avoid channelling.
10. Design the suitable type of cooler to remove the exothermic heat of absorption.
    If heat duty requirement is low, then a coil-type cooler can be inserted in the liquid pool of circulating solvent hold-up tank. For the higher heat duty a separate shell and tube-type heat exchanger can be selected and installed in recirculation line of solvent.
11. Design the suitable type of liquid distributor, liquid redistributors, packing support, etc.

Example 9.2 Design a scrubber for bromine for following service:
Feed gas to bromine scrubber = 25 kg/h of Br_2 and 250 kg/h of air (Here air is added for dilution.)

Design data of an existing bromine scrubber are available in literature as under.
Feed gas = 10 kg/h of Br_2 + 50 ft^3/min air measured at 1 atm and 25°C
Molar mass of Br_2 = 160
Concentration of Br_2 in outlet gas = 0.1 ppm
Gas temperature = Ambient
Type of scrubber = Counter flow packed bed
Column ID = 150 mm
Packed bed height = 2 m
No. of packing sections = 2
Type of packing = 16 mm (5/8 in) polypropylene Pall rings
Circulating liquid = 10% caustic soda solution
Pressure drop across packing = 45 mm WC
Capacity of circulation tank = 1200 L
Tank MOC = FRP
Solvent rate = 1 m^3/h
Gas pressure = Atmospheric
Type of fan: Induced draft fan 100 mm (4 in) WC pressure, 50 ft^3/min capacity

Solution

Feed gas flow rate = G_{1w} = 250 + 25 = 275 kg/h

$$G_1 = \frac{250}{29} + \frac{25}{160} = 8.777 \text{ kmol/h}$$

$$y_1 = \frac{(25/160)}{8.777} = 0.0178, \quad y_2 = 0.1 \times 10^{-6} = 10^{-7}$$

$$N_{OG} = \ln\left(\frac{y_1}{y_2}\right) = \ln\left(\frac{0.0178}{10^{-7}}\right) = 12.0895$$

Reaction between Br_2 and NaOH is instantaneous and irreversible. Hence $y_e = 0$
Minimum amount of solvent:
Reaction

$$2\ NaOH(aq) + Br_2(g) \longrightarrow NaOBr(aq) + NaBr(aq) + H_2O(l)$$

NaOH required to absorb 25 kg/h Br_2

$$= 25 \times \frac{80}{160} = 12.5 \text{ kg/h}$$

$$L_{sm} = \frac{12.5}{0.1} = 125 \text{ kg/h of 10\% NaOH solution}$$

Actual amount of solvent should be the greatest of the following:

1. $L_s = 1.5\ L_{sm} = 125 \times 1.5 = 187.5$ kg/h
2. To facilitate the use of a centrifugal pump for solvent.

   $L_s \geq 1\ m^3/h$

   $\dot{m}_s = 1\ m^3/h \times$ Density of 10% NaOH solution

   $= 1 \times 1100 = 1100$ kg/h
3. Rise in temperature of circulating solvent should be in the range of 5 to 10°C.

   Heat of reaction.

   $$\Delta H_r^o = \sum \nu_i\ \Delta H_{fi}^o$$

   $\Delta H_r^o = -285.83 - 334.3 - 361.67 - 2(-470.11) - 30.91$

   $\Delta H_r^o = -72.49$ kJ/mol $= \Delta H_r$

   Heat of absorption, $\phi_1 = 72.49 \times 1000 \times \frac{25}{160} = 11\ 326.6$ kJ/h

   $\equiv 3.13$ kW

   Let rise in temperature of solvent = 5°C

   $$L_{SW} = \frac{11\ 326.6}{C_L \Delta t} = \frac{11\ 326.6}{3.9 \times 5} = 580.85 \text{ kg/h}$$
4. Actual wetting rate should be greater than minimum wetting rate required.

   For the first trial calculation,

   Let $L_{sw} = 1100$ kg/h (= 1 m^3/h)

   $\rho_L = 1100$ kg/m^3

   $G_{1w} = 250 + 25 = 275$ kg/h

   Average molar mass of gas mixture at inlet

   $$M_{av} = \frac{275}{8.777} = 31.332$$

$$\rho_{G1} = \frac{pM_{\text{av}}}{RT} = \frac{1 \times 31.332}{0.082 \times (273 + 25)} = 1.282 \text{ kg/m}^3$$

Based on Eq. (9.15):

$$F_{LV} = \frac{1100}{275}\sqrt{\frac{1.282}{1100}} = 0.1365$$

For 45 mm WC /m of packing
$K = 0.045$ and $K_F = 0.15$ (From Fig. 9.5)

$$\%\text{ Flooding} = \sqrt{\frac{K}{K_f}} \times 100 = \sqrt{\frac{0.045}{0.15}} \times 100 = 55\%$$

Hence, it is in the range of 50 to 70%.
For 16 mm (5/8 in) polypropylene Pall rings, packing factor $F_p = 310 \text{ m}^{-1}$ (Ref. Table 9.1)
Mass velocity of gas through tower can be calculated using Eq. (9.18):

$$\psi = \frac{\rho_W}{\rho_L} = \frac{1000}{1100} = 0.909$$

$$\mu_L = 5 \text{ cP}$$

$$G_w = \left(\frac{0.045 \times 1.282 \times 1100 \times 9.81}{310 \times 0.909 \times 5^{0.2}}\right)^{0.5}$$

$$= 1.2654 \text{ kg/(m}^2\cdot\text{s)}$$

$$\frac{\pi}{4} D_i^2 = \frac{G_{1w}}{G_w} = \frac{(275/3600)}{1.2654} = 0.0604 \text{ m}^2$$

$$D_i = 0.277 \text{ m} \equiv 0.28 \text{ m}$$

$$\frac{D_i}{d_P} = \frac{0.28}{0.016} = 17.5 > 10$$

$$\text{Actual wetting rate} = \frac{1}{\frac{\pi}{4}(0.28)^2} = 16.24 \text{ m}^3/(\text{h}\cdot\text{m}^2)$$

MWR for polypropylene Pall ring = 4 $\text{m}^3/(\text{h} \cdot \text{m}^2)$
Hence, AWR > MWR (satisfactory)

Eq. (9.36) $$H_{OG} = \frac{8.777 / \left(\frac{\pi}{4}(0.28)^2\right)}{K_G a} = \frac{142.54}{K_G a}$$

For existing scrubber:
G_{1w} = 10 kg/h + 50 ft^3/min (≈ 85 m^3/h) at 1 atm and 25°C

Density of air

$$\rho_a = \frac{PM}{RT} = \frac{1 \times 29}{(273 + 25)} \times \frac{273}{1 \times 22.414}$$

$$= 1.185 \text{ kg/m}^3$$

Mass flow rate of air $= 85 \times 1.185 = 100.7$ kg/h

$$G_{1W} = 10 + 100.7 = 110.7 \text{ kg/h}$$

$$G_1 = \frac{100.7}{29} + \frac{10}{160} = 3.535 \text{ kmol/h}$$

$$y_2 = \frac{(10/1160)}{3.535} = 0.017\,68$$

$$y_2 = 0.1 \text{ ppm} = 0.1 \times 10^{-6}$$

$$N_{OG} = \ln\left(\frac{y_1}{y_2}\right) = \ln\left(\frac{0.017\,68}{0.1 \times 10^{-6}}\right) = 12.08$$

$$G_M = \frac{3.535}{\frac{\pi}{4}(0.15)^2} = 200 \text{ kmol/(m}^2 \cdot \text{h)}$$

$$H_{OG} = \frac{Z}{N_{OG}} = \frac{2}{12.082} = 0.1655 \text{ m}$$

$$K_G a = \frac{200}{0.1655} = 1208.7 \text{ kmol/(h} \cdot \text{m}^3)$$

For the new tower design

$$H_{OG} = \frac{142.54}{1208.7} = 0.118 \text{ m}$$

Height of packing $\quad Z = N_{OG} \times H_{OG}$

$$= 12.0895 \times 0.118 = 1.4265 \text{ m}$$

Actually increase in diameter decreases the value of $K_G a$. Hence, take actual as height of packing for new tower as 2 m.

Let number of packing section = 2

Consider 20% extra heat duty of cooler.

Heat duty of cooler

$$\phi_2 = 11\,326.6 \times 1.2 = 13\,600 \text{ kJ/h} \equiv 3.778 \text{ kW}$$

Rise in temperature of solvent

$$\Delta t = \frac{\phi}{C_L \times \dot{m}} = \frac{13\,600}{3.9 \times 1100} = 3.17°\text{C}$$

Coil can be immersed in liquid pool of circulation tank.

Let $\quad U = 350 \text{ W/(m}^2 \cdot \text{C)}$

Cooling water temperature:

Inlet temperature of cooling water = 32°C

Outlet temperature of cooling water = 34°C

Let $\Delta T_m = 7°C$

Hence, average temperature of solvent in absorber is 40°C (= 33 + 7).

Cooling water required

$$\dot{m}_a = \frac{13\,600}{4.1868 \times (34 - 32)} = 1624.15 \text{ kg/h} \equiv 1.624 \text{ m}^3\text{/h}$$

Let velocity of water through coil = 2 m/s

$$\frac{\pi}{4} d_{ci}^2 = \frac{(1.624/3600)}{2}$$

$$d_{ci} = 0.017 \text{ m} = 17 \text{ mm}$$

$$d_{co} = 22 \text{ mm} = \text{outside diameter of pipe}$$

Let helix diameter = 0.5 m

Heat transfer area per turn.

$$\text{HTA} = \pi \times 0.5 \times (\pi \times 0.022)$$

$$= 0.1086 \text{ m}^2$$

Area of coil required

$$A_r = \frac{(13\,600/3600) \times 1000}{350 \times 7} = 1.542 \text{ m}^2$$

$$\text{No. of turns of coil required} = \frac{1.542}{0.1086} = 14.2$$

Let number of turns = 15

Design of liquid distributor:

Pipe-type distributor may be selected.

Rate of solvent = 1 m^3/h

It is recommended that one distribution point must be provided for each 194 cm^2 area.

No. of holes (distribution points) required in pipe-type distributor

$$n_h = \frac{\left(\frac{\pi}{4}\right) D_i^2}{194} = \frac{\left(\frac{\pi}{4}\right) 28^2}{194} = \frac{615}{194} = 3.17$$

Let $n_h = 4$

Let velocity of solvent through pipe = 2 m/s

If d_i = ID of pipe, then

$$\frac{\pi}{4} d_i^2 = \frac{1/3600}{4 \times 3}$$

$$d_i = 0.0133 \text{ m} \equiv 13.3 \text{ mm}$$

Let velocity of solvent through hole = 3 m/s

If d_h = diameter of each hole, then

$$\frac{\pi}{4} d_h^2 = \frac{1/3600}{4 \times 3}$$

$$d_h = 5.42 \text{ mm}$$

Let $d_h = 5$ mm

Design of liquid redistributor:

Let 45° wiper-type liquid redistributors be employed.

Fig. 9.15 *Suggested Redistributor*

Design of packing support

Design criteria: Free area for the flow of gas provided by packing support should be greater than free area for the flow of gas provided by packing material.

% free space provided by 16 mm PP Pall ring = 87%

Let type of packing support = Cap type

Let diameter of rises or cap = $\frac{D_i}{6} = \frac{280}{6} = 46$ mm

Total number of caps = 9

Let width of slot $x = 5$ mm

No. of slots per cap = $\frac{\pi \times 46}{2x} = 14$

Let h' = height of slot

$$h' \times 9 \times 14 \times 5 \times \geq 0.87 \times \frac{\pi}{4}(280)^2$$

$$h' \geq 85 \text{ mm}$$

Let velocity of liquid through weeping hole = 0.5 m/s

Let number of holes = 12

$$\frac{\pi}{4} d_W^2 = \frac{1/3600}{12 \times 0.5}$$

$$d_W = 7.68 \times 10^{-3} \text{ m say 8 mm}$$

Fig. 9.16 *Suggested Cap-Type Packing Support Plate*

9.5 PROCESS DESIGN OF SPRAY CHAMBER OR SPRAY TOWER-TYPE ABSORBER[3, 9]

Spray absorbers are used for removing SO_2 (sulphur dioxide gas) form boiler flue gases. When very low pressure drop is essential and when incoming gas stream is contaminated by particulate matter, spray absorbers are preferred over the packed tower type absorber. In the large scale plant, boiler flue gases entering the absorber for the removal of SO_2 are available at very low pressure (100 to 200 mm WC) and with very high volumetric flow rate. These gases are also contaminated by particulate matters. If coal is used as a fuel then concentration of SO_2 and suspended particulate matter (SPM) in boiler flue gases are relatively higher and it favours selection of spray tower.

Spray tower facilitates the use of lime slurry as a solvent against caustic soda solution. Former is cheaper than the latter. Lime slurry as such cannot be considered in packed tower.

Spray chambers (absorbers) are having either circular or rectangular cross section. Normally there is no packing in the spray absorber.

They are of two types:

1. Vertical spray tower
2. Horizontal spray chamber

Liquid phase residence time in spray absorbers is in the range of 1 to 10 s which is very low. To compensate this, liquid phase resistance must be negligible. Hence, a chemical reagent is added in the liquid phase to get the higher rate of absorption.

9.5.1 Process Design of Vertical Spray Tower[3]

In vertical spray tower, gas stream is flowing vertically upward and liquid is sprayed downward in various sections within the tower as well as from the sides by spray nozzles.

Process design steps for vertical spray tower are as follows:

1. Determination of tower diameter: Maximum permissible gas velocity through vertical spray tower is 2.3 m/s. Actual velocity of gas through tower should be less than 2.3 m/s to avoid the excessive entertainment of liquid droplets in the exist gas. On fixing the actual velocity, tower diameter can be determined by following equation:

$$D = \sqrt{\frac{q_v \times 4}{u_g \times \pi}} \tag{9.37}$$

where, D = Tower diameter, m
q_v = Volumetric flow rate of gas, m^3/s
u_g = Velocity of gas through tower, m/s < 2.3 m/s

2. Determine the number of gas phase transfer units required for the desired separation by the following equation:

$$N_G = \frac{y_1 - y_2}{y_2} \tag{9.38}$$

where, N_G = Number of gas phase transfer units
y_1 = Gas phase solute mole fraction at inlet (at bottom)
y_2 = Gas phase solute mole fraction at outlet (at top)

Actually the correlation for N_G is

$$N_G = \int_{y_2}^{y_1} \frac{dy}{(y - y_e)} \tag{9.39}$$

Considering irreversible chemical reaction and use of excess reactant, $y_e = 0$. Also in vertical spray tower, gas tends to become completely backmixed by the action of liquid spray. Hence in spray section, concentration of solute is uniform and $y = y_2$ in Eq. (9.39).

3. Calculate the total volume of spray section by the following equation:

$$V = \frac{N_G \times G}{K_G a} \tag{9.40}$$

where, V = Volume of spray section, m^3
$K_G a$ = Overall volumetric gas phase mass transfer coefficient kmol/[(s · m^3) (mole fraction of solute in gas)]
G = Flow rate of gas phase, kmol/s

The best method for finding the $K_G a$ is from the similar type of commercial gas absorption unit noting that same value of $K_G a$ can be used only with same value of liquid flow rate to gas flow rate ratio.

9.5.2 Process Design of Horizontal Cross Flow Spray Chamber[9]

For the given absorption duty total volume required by horizontal cross-flow chamber is considerably less than that of a vertical spray tower. Horizontal chamber is having either rectangular or circular cross section. Gas passes horizontally through it and liquid is sprayed vertically downward perpendicular to the direction of gas flow. Process design steps for horizontal cross flow chamber are as follows.

1. Determination of duct area or diameter: Maximum permissible gas velocity through horizontal spray chamber is 7 m/s. On fixing the actual gas velocity through horizontal chamber, duct area or diameter can be determined.
2. Determine the number of gas phase transfer units, required for the desired separation by the following equation:

$$N_G = \ln\left(\frac{y_1}{y_2}\right) \tag{9.41}$$

   where, N_G = Number of gas phase transfer unit
   y_1 = Gas phase solute mole fraction at inlet
   y_2 = Gas phase solute mole fraction at outlet
3. Total volume of spray section is determined by the same equation that is used for vertical spray tower. (Eq. 9.40).

In a horizontal spray chamber, gas is well mixed radially by the action of liquid spray in a cross-current contact but not axially or in the direction of gas flow. Hence, concentration of solute in gas mixture is not uniform in the spray section.

Example 9.3 Design the spray chamber for the following duty:
Volumetric flow rate of boiler flue gas = 24 000 Sm^3/h
Pressure of gas = 150 mm WC (gauge)
Temperature = 80 to 90 °C
SO_2 concentration in boiler flue gas = 4000 ppm = 4000×10^{-6} kmol/kmol gas
Desired concentration of SO_2 in the outgoing gas
= 200 ppm = 200×10^{-6} kmol/kmol gas
Solvent = 1% lime solution

Solution

Type of scrubber: Spray chamber
Position: Horizontal or vertical
(a) Tower diameter
(i) For vertical spray chamber:
Volumetric flow rate of gas = 24 000 Sm^3/h
Volumetric flow rate of gas at actual operating condition

$$q_v = \frac{24\,000 \times 10\,333}{273} \times \frac{(273+90)}{(10\,333+150)}$$

$$= 31\,455.5 \text{ m}^3/\text{h} \equiv 8.7376 \text{ m}^3/\text{s}$$

Maximum permissible gas velocity through vertical spray chamber, $u_{g\,\max}$ = 2.3 m/s

Let actual velocity of gas through spray tower.

$$u_g = 66\% \text{ of } u_{g\,\max}$$

$$u_g = 0.66 \times 2.3 = 1.518 \text{ m/s}$$

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{u_g}$$

where, D_i = Inside diameter of spray tower

$$D_i = \sqrt{\frac{4 \times q_v}{\pi \times u_g}} = \sqrt{\frac{4 \times 8.7376}{\pi \times 1.518}} = 2.707 \text{ m}$$

Let $D_i = 2.71 \text{ m} \equiv 2710 \text{ mm}$

(ii) Horizontal cross-flow spray chamber:

Maximum permissible gas velocity through horizontal spray chamber $u_{g\,\max} = 7$ m/s

Let actual velocity of gas through horizontal spray chamber, $u_g = 66\%$ of $u_{g\,\max}$

Let cross-sectional area of horizontal spray chamber, $A = W^2$ for square duct

where, W = Width of chamber = Height of chamber

$$W^2 = \frac{q_v}{u_g} = \frac{8.7376}{4.62} = 1.8913$$

$$W = 1.375 \text{ m}$$

(b) Number of gas phase transfer units, N_G

(i) Number of gas phase transfer units required for desired separation, for vertical spray tower:

$$N_G = \frac{y_1 - y_2}{y_2} \tag{9.38}$$

$$= \frac{4000 \times 10^{-6} - 200 \times 10^{-6}}{200 \times 10^{-6}} = 19$$

(ii) For horizontal spray chamber:

$$N_G = \ln\left(\frac{y_1}{y_2}\right) = \ln\left(\frac{4000 \times 10^{-6}}{200 \times 10^{-6}}\right) = 3 \tag{9.41}$$

(c) Circulation rate of 1% lime solution

(i) For vertical spray chamber:

To decide the circulation rate and height of gas phase transfer unit duplication of similar/same type of commercial gas absorption unit is the best method.

From the existing vertical spray tower of one of the large scale industry, following data are available:

Flow rate of boiler flue gas = 12 000 Sm^3/h
Temperature of incoming gas = 80 to 90°C
SO_2 concentration in inlet stream = 2500 to 4000 ppm
SO_2 concentration in outlet stream = Less than 400 ppm
Circulation rate of 1% lime solution = 30 m^3/h
Tower diameter = 1.7 m

Height of spray zone = 3.5 m

Pressure drop = 100 mm WC (maximum)

Based on these data, circulation rate required for new tower $= 30 \times \dfrac{24\,000}{12\,000} = 60\ \text{m}^3/\text{h}$

(ii) For horizontal spray chamber:

For the trial run, let the circulation rate of 1% lime solution for horizontal spray chamber = 60 m^3/h

(d) Height of tower

(i) Vertical spray tower: Eq. (9.40)

For existing tower

$$N_G = \frac{4000 \times 10^{-6} - 400 \times 10^{-6}}{400 \times 10^{-6}} = 9$$

$$V = \frac{\pi}{4}(1.7)^2 \times 3.5 = 7.944\ \text{m}^3$$

$$G_m = \frac{12\,000}{22.414} = 535.38\ \text{kmol/h} \equiv 0.1487\ \text{kmol/s}$$

For existing tower, $K_G a = \dfrac{9 \times 0.1487}{7.944} = 0.1685\ \text{kmol/(m}^3 \cdot \text{s)}$

Volume of spray section required for new tower

$$V = \frac{N_G G_m}{K_G a} = \left(19 \times \frac{24\,000}{22.414} \times \frac{1}{3600}\right)/0.1685$$

$$= 33.54\ \text{m}^3 = (\pi/4) D_i^2 H$$

where, H = Height of spray section required

$$33.54 = (\pi/4) \times (2.7)^2 \times H$$

$$H = 5.86\ \text{m}$$

Let $H = 6$ m

(ii) For horizontal spray chamber:

Mass transfer data are available for vertical spray tower only. Assume the overall volumetric mass transfer coefficient $K_G a$ for cross flow arrangement in horizontal spray chamber (less than the same for vertical position), to be 0.1 kmol/($\text{m}^3 \cdot$ s).

$$V = \frac{N_G \times G_m}{K_G a} = \frac{3 \times 0.2974}{0.1} = 8.922$$

$$V = 8.922\ \text{m}^3 = W^2 L = (1.375)^2 L$$

Length of spray section required with horizontal position, $L = 4.72$ m

Let $L = 5$ m

(e) Approximate pressure drop calculations

(i) For vertical spray chamber:

Pressure drop $\Delta p \propto \dot{m}^{1.84} d^{-4.84} L$ [Ref. Eq. (5.5a)]

Pressure drop in actual vertical spray tower is 100 mm WC (maximum).

Pressure drop in proposed new vertical spray tower,

$$\Delta p = 100 \times \left(\frac{24}{12}\right)^{1.84} \times \left(\frac{2.71}{1.7}\right)^{-4.84} \times \left(\frac{6}{3.5}\right)$$

Δp = 64.24 mm WC (within permissible limit)

(ii) For horizontal spray chamber:

$$\Delta p = 100 \times \left(\frac{24}{12}\right)^{1.84} \times \left(\frac{1.375}{1.7}\right)^{-4.84} \times \left(\frac{5}{3.5}\right)$$

Δp = 1428 mm WC (very high)

To decrease the pressure drop, duct area must be increased.
Let W = 2 m and L = 2.5 m

$$\Delta p = 100 \times \left(\frac{24}{12}\right)^{1.84} \times \left(\frac{2}{1.7}\right)^{-4.84} \times \left(\frac{2.5}{3.5}\right)$$

= 116.45 mm WC (within permissible limit)

Note: For this approximate calculation, width or height of chamber W is considered as diameter.

Actually duct area (W^2) is higher than circular cross-sectional area $\left(\frac{\pi}{4}W^2\right)$ and hence it will offer less pressure drop than the circular cross section.
Volume of spray section, $V = (2)^2 \times 2.5 = 10\ m^3$ (revised)

(f) Make-up of lime solution

Lime required = Lime consumed in reaction + Drift loss
Liquid particles entrained in the outgoing gas.
Let drift loss = 0.2% of circulation rate (based on experience in cooling tower).

Drift loss = $\frac{0.2}{100} \times 60 = 0.12\ m^3/h \cong 120$ kg/h

Loss of lime as drift = 1.2 kg
Lime is consumed in reaction.

$$Ca(OH)_2 + SO_2 \rightarrow CaSO_3 + H_2O$$

SO_2 consumed in reaction = $\frac{24\,000}{22.414} \times (4000 - 200) \times 10^{-6} = 4.07$ kmol/h

$Ca(OH)_2$ consumed in reaction = 4.07 kmol/h = 4.07 × 74 = 301.18 kg/h

Lime required as $CaCO_3$ = $(301.18 + 1.2) \times \frac{100}{74} = 408.62$ kg/h

Loss of water = Drift loss + Evapouration rate + Blow down ≅ 1% of circulation rate
$CaCO_3$ addition required = 408 kg/h
Water addition required = 6 m^3/h = 6000 kg/h

Summary of results

Vertical spray tower	*Horizontal spray chamber*
1. Tower diameter = 2.71 m	1. Duct width and height = 2 m
2. Height of spray section = 6 m	2. Length of spray section = 2.5 m
3. Volume of spray section = 33.54 m^3	3. Volume of spray section = 10 m^3
4. Pressure drop = 64 mm WC	4. Pressure drop = 116.45 mm WC
5. Circulation rate = 60 m^3/h	5. Circulation rate = 60 m^3/h
6. Lime as $CaCO_3$ required = 408 kg/h	6. Lime as $CaCO_3$ required = 408 kg/h
7. Makeup water required = 6 m^3/h	7. Makeup water required = 6 m^3/h

Comment: From the resulting data, it is clear that horizontal spray chamber is a better option. But it is less common in operation as compared to vertical spray tower. Here mass transfer data used for calculation are available for vertical spray tower. Hence, design of the vertical spray tower is more reliable as compared to the horizontal spray chamber.

9.6 VENTURI SCRUBBER[3, 10]

Venturi scrubber is normally preferred for removing particulate matter from gas stream as opposed to absorbing soluble vapour. Efficient contact between liquid and gas is obtained in venturi scrubber. Gas is drawn into the throat of the venturi by a stream of absorbing liquid solvent sprayed into the convergent duct section. Compared to spray absorber, more efficient contact between gas and liquid is provided by the venturi scrubber but at the expense of relatively large pressure drop and a consequent higher power consumption. Spray absorber cannot remove very fine particles (below 10 μm size) while the venturi scrubber can remove the fine particles up to the size 2 μm effectively.

Venturi scrubber provides cocurrent contact (over all) between liquid and gas. The fractional solute removal for a dilute system in venturi scrubber is given by the following equation:

$$\text{Fractional solute removal} = \frac{y_1 - y_2}{y_1} = \frac{\eta(1 - m x_2/y_2)}{\left(1 + \dfrac{mG_M}{L_M}\right)} \tag{9.42}$$

where, y_1 = Mole fraction of solute in gas phase at outlet
y_2 = Mole fraction of solute in gas phase at inlet
m = Slope of equilibrium curve
x_2 = Mole fraction of solute in incoming solvent
G_M = Molar gas phase velocity, kmol/($m^2 \cdot s$)
L_M = Molar liquid phase velocity, kmol/($m^2 \cdot s$)
η = Mass transfer efficiency

$$\eta = 1 - e^{-N_G} \tag{9.43}$$

where, N_G = Number of overall gas-phase mass transfer units

$$= (K_G aRT/p_t)\theta_c \tag{9.44}$$

where, $K_G a$ = Overall volumetric gas-phase mass-transfer coefficient for dilute systems, kmol/(s · m^3)
R = Ideal gas constant = 8314 J/(kmol · K)
T = Operating temperature of gas, K
θ_c = Effective gas-liquid contact time, s
p_t = Operating pressure, Pa

The number of transfer units, N_G that can be achieved in venturi scrubber is in the range of 1 to 2 for physical absorption.

The liquid to gas ratio for venturi scrubber is in between 0.7 to 2.7 L/m^3. Throat velocity is kept in between 60 to 150 m/s. The estimated pressure drop or vacuum created by the venturi scrubber is given by following equation (Hesketh equation[9]).

$$\Delta p = 2.584 \times 10^{-3}\, v_G^2 \rho_G A_{th}^{0.133} (L'/G')^{0.78} \tag{9.45}$$

where, Δp = Venturi pressure drop from inlet duct to the outlet duct, cm WC
v_G = Gas velocity in the throat, m/s
ρ_G = Gas density, kg/m^3
A_{th} = Venturi throat area, cm^2
L'/G' = Liquid to gas ratio, L/m^3

Venturi scrubber can be selected as an absorber and placed before a packed tower type absorber in the following two cases:

1. If the feed gas to absorber is contaminated by fine solid particles.
2. If vent gases from the process are available at very low pressure. In this case, venturi scrubber is designed such that suction or negative pressure to be developed by the venturi scrubber is sufficient to overcome the pressure drop across the packed tower.

In both cases, venturi scrubber also decreases load of absorption of the packed tower as some absorption takes place in the venturi scrubber. Absorption equivalent to 1 to 2 theoretical stages in case of physical absorption and could be higher than two stages in case of absorption with chemical reaction.

Venturi scrubber itself acts as liquid jet ejector and creates negative suction or vacuum. Typical entrance cone angle is 21 ± 2° and exit cone angle is 5 to 15°. Throat length is equal to diameter of throat (d). Diameters of both cones at large ends (other ends) are equal to 3d.

Example 9.4 Predict the fractional solute removal and pressure drop in a venturi scrubber based on the following data:

Volumetric flow rate of boiler flue gas = 24 000 Nm^3/h
Discharge pressure of gas from venturi = Atmospheric
Temperature of gas = 80 to 90°C
SO_2 concentration in boiler flue gas = 4000 ppm (or mg/kg)
Solvent = 1% lime solution
Solvent to gas ratio = 1.4 L/m^3
Recommended throat velocity of gas phase = 100 m/s
Average molar mass of flue gas = 29.48 kg/kmol

Solution

Volumetric flow rate of gas at actual operating condition.

$$q_V = \frac{24\,000}{273} \times (273 + 90) = 31\,912.1 \text{ m}^3/\text{h}$$

$$\equiv 8.8645 \text{ m}^3/\text{s}$$

Gas velocity in throat = 100 m/s

Inside diameter of throat,

$$d_i = \sqrt{\frac{4 \times 8.8465}{\pi \times 100}} = 0.336 \text{ m}$$

Let angle of upstream cone = 21°

angle of downstream cone= 10°

Diameters of cones at large ends = 3 d_i = 1.008 m

Height of venturi = Height of upstream cone + Height of throat + Height of downstream cone

= [0.336/tan(21/2)] + 0.336 + [0.336/tan(10/2)]

= 5.99 m, say 6 m

Flow rate of solvent (1% lime solution) = 1.4 × 8.8645 = 12.41 L/s ≡ 44.676 m^3/h

Density of 1% lime solution = 1012.5 kg/m^3

Mass flow rate of solution = 45 234.5 kg/h

$$\dot{m}_L = 12.565 \text{ kg/s}$$

Molar flow rate of liquid, $L_M = \dfrac{12.565}{\left(\dfrac{1}{(0.99/18) + (0.01/74)}\right)} = 0.693$ kmol/s

Molar flow rate of gas, $G_M = \dfrac{p\,q_v}{RT} = \dfrac{1 \times 8.8645}{0.082 \times (273 + 90)}$

$= 0.2978$ kmol/s

Assuming $N_G = 1$ (safe value)

Mass transfer efficiency,

$$\eta = 1 - e^{-N_G}$$

$$\eta = 1 - e^{-N_E} = 1 - e^{-1} = 0.6321 \tag{9.43}$$

Fractional solute removal,

$$1 - \frac{y_2}{y_1} = E\frac{(1 - m\,x_2/y_2)}{\left(1 + m\dfrac{G'_M}{L'_M}\right)}$$

$$\frac{G'_M}{L'_M} = \frac{G_M/\text{Flow area}}{L_M/\text{Flow area}} = \frac{G_M}{L_M} = \frac{0.2978}{0.693} = 0.43$$

$$x_2 = 0, \qquad y_2 = ?$$

$$1 - \frac{y_2}{y_1} = \frac{E}{1 + m\dfrac{G'_M}{L'_M}} = \frac{0.6321}{1 + 0.43m}$$

$$y_1 = 4000 \times 10^{-6} = 0.004$$

Partial pressure of SO_2 in gas phase ≅ 0.004 × 760 = 3.04 Torr

Assume the average temperature of scrubber = 50°C

Equilibrium mass of SO_2 per 100 mass of H_2O = 0.035 (Table 3–144 of Ref. 3)

$$\text{Equilibrium mass fraction} = \frac{0.035}{100.035} \cong 3.5 \times 10^{-4}$$

$$\text{Equilibrium mole fraction} = \frac{3.5 \times 10^{-4}/64}{(3.5 \times 10^{-4}/64) + (1 - 3.5 \times 10^{-4})/18}$$

$$x_e = 9.846 \times 10^{-5}$$

$$y_e = 0.004$$

$$m = \frac{0.004}{9.846 \times 10^{-5}} = 40.63$$

Equilibrium constant for physical absorption at 50°C and 1 atm pressure, $m = 40.63$
For low concentration range m can be assumed constant.

$$1 - \frac{y_2}{y_1} = \frac{0.6321}{1 + 0.43 \times 40.63} = 0.034\ 22$$

$$\frac{y_2}{y_1} = 0.9658$$

$$y_2 = 0.003\ 86$$

Here, presence of 1% lime in solution or chemical reaction between lime and SO_2 is not considered. Chemical reaction enhances the rate and extent of absorption considerably. To find this, lab-scale or pilot plant scale data are required on the same system and also on the same type of absorber. Reliable theoretical equations are not available for the same.

Pressure drop by Eq. (9.45).

$$v_G = 100 \text{ m/s}$$

$$A_{\text{th}} = \text{Throat area} = \frac{\pi}{4}(33.6)^2 = 886.7 \text{ cm}^2$$

Density of gas $$\rho_G = \frac{PM}{RT} = \frac{1 \times 29.48}{0.082 \times (273 + 90)} = 0.99 \text{ kg/m}^3$$

$$\frac{L'}{G'} = 1.4 \frac{\text{L}}{\text{m}^3}$$

$$\Delta p = 82 \text{ cm WC} \equiv 820 \text{ mm WC}$$

Pressure drop provided by venturi scrubber (820 mm WC) is negative pressure or vacuum generated by the same.

9.7 PROCESS DESIGN OF FALLING FILM ABSORBER[11, 12]

Falling film absorber is a shell and tube heat exchanger [Fig. 9.17(a) and Fig. 10.12] in which absorption of gas in liquid is carried out inside the tubes while cooling medium like cooling water is circulated on the shell side. It is used for highly exothermic absorption like absorption of hydrogen chloride gas in water, absorption of ammonia gas in water, absorption of sulphur trioxide in alpha olefins, absorption of sulphur trioxide in methyl esters of saturated fatty acids, etc.

Use of graphite block-type heat exchanger is increasing for similar applications in which cocurrent absorption is carried out in vertical holes and cooling medium is circulated in horizontal holes. Schematic representation of block-type heat exchanger is given in Fig. 9.17(b).

(a) Graphite Shell and Tube-Type Heat Exchanger-cum-absorbers

(b) Graphite Block-Type Heat Exchanger-cum-absorbers (*Courtesy: Grephicarb Products, Ahmedabad*)

Fig. 9.17 *Graphite Heat Absorbers*

9.7.1 Advantages of Falling Film Absorber over Packed Tower-Type Adiabatic Absorber

1. Falling film absorber gives better quality of product solution. Concentration of HCl in product solution from falling film absorber can be 35% to 37% (by

mass), while the same from adiabatic packed tower ranges from 25% to 30%.

2. Absorption is more efficient in falling film absorber for the given absorption duty. Falling film absorber requires less space compared to adiabatic packed tower-type absorber.
3. Other advantages of falling film absorber are lower temperatures, lower pressure drops and higher turn down.

9.7.2 Disadvantages of Falling Film Absorber

1. More difficult to control and is sensitive to liquid and gas distribution problems.
2. It has greater tendency to absorb undesirable water soluble low boiling impurities.

9.7.3 Exothermic Absorption of Gases Like HCl and NH_3

In case of exothermic absorption of gas like HCl and NH_3 in water, there are two requirements:

1. Large liquid surface area to get the higher rate of mass transfer.
2. Rapid removal of the heat of absorption.

Both can be done most effectively by causing the liquid to flow down as a film inside of a vertical tube which is cooled on the outside.

Falling film absorber with countercurrent flow of the gas and liquid suffer from the limitation that flooding occurs when the gas velocity exceeds a limiting value of the order of 4.5 m/s for 25 mm OD tube. A sample calculation shows that at the usual ratio of liquid and gas flow rates, the liquid rate is barely sufficient to maintain a liquid film on the tube wall. Hence, countercurrent contact is not preferred. Cocurrent contact of gas and liquid allows the gas velocity up to 18 m/s.

For the absorption of hydrogen chloride gas falling film absorber (with cocurrent contact of gas and liquid) followed by tail gas scrubber is preferred if the concentration of hydrogen chloride gas in feed gas mixture is more than 40% (by mole). Nearly, 66 to 85% of absorption is carried out in falling film absorber while the balance is carried out in the tail gas scrubber. If the concentration of hydrogen chloride in feed gas mixture is less than 40% (by mole), then it is difficult to produce the product solution of commercial strength. With cocurrent contact, partial pressure of hydrogen chloride in exist gas is greater than or equal to exit gas equilibrium partial pressure of hydrogen chloride in product solution.

While most HCl gas will be scrubbed in these two scrubbers, local environmental standards could mandate an additional vent scrubber in which final wash of the outgoing gas mixture is carried out by spraying small quality of water. However, the bottom solution from the vent scrubber will contain some HCl and its disposal may require neutralisation with alkali.

Low concentration of HCl in exist gas gives low concentration of HCl in product solution. The arrangement of falling film absorber with tail gas scrubber is shown in Fig. 9.18.

Fig. 9.18 *Typical Falling Film Absorption System*

Feed gas and weak acid from the tail gas scrubber (5 to 10% HCl solution) is supplied at the top head of falling film absorber. Tubes are extended above the top tube sheet to facilitate the uniform distribution of liquid in each tube. Notches may be cut in the top ends of the tubes. Tubes are made from graphite, glass or tantalum. As per the recommendations tubes must be extended at least by 125 mm above the top tube sheet to minimise the effect of hydraulic gradient in the liquid. With cocurrent

contact in upper portion of tubes, rate of absorption is very high. It may lead to the overheating of tubes in upper portion. Overheating could break the glass tubes. To avoid this, inverted U in the discharge line of water is essential. Inside the tubes there is a two-phase flow of liquid and gas in which liquid forms the film over the inside periphery of tubes while gas flows in the central portion. Flow rates of liquid and gas should be so adjusted to get annular flow inside the tubes. Absorption of gas in liquid film generates the heat. Temperature of liquid film is controlled by cooling water circulated on shell side. In the bottom head, gas and product solution are separated. Exit gas is in the equilibrium of product solution at outlet condition. Hence, complete absorption of HCl with this arrangement is impossible. Remaining HCl is absorbed by contact with pure water in tail gas scrubber which is commonly used and is normally a packed tower-type absorber.

9.7.4 Process Design of Falling Film Absorber

Consider absorption of HCl in water.

1. Fix the inlet and outlet concentrations of aqueous hydrochloric acid. Also, fix the concentration of hydrogen chloride in the exist gas stream. Partial pressure of hydrogen chloride in the exist gas stream at bottom should be greater than vapour pressure of hydrogen chloride in product solution at outlet temperature. Based on this fact, outlet temperature and concentrations of liquid and gas streams can be fixed.
2. Determine the overall rate of absorption and based on the same, determine the total heat duty. Actually rate of absorption and hence, rate of heat transfer required are changing from point to point of the heat exchanger (i.e., falling film absorber)
3. Based on the heat balance, find the flow rate of cooling medium.
4. In the end, heat transfer area of falling film absorber must be determined:

$$A = N_t \, \pi \, d_o L = \frac{\phi}{U \, \Delta \mathcal{T}_m} \tag{6.17}$$

To facilitate two-phase flow, d_o (tube outside diameter) can be selected as 25.4 mm. Number of tubes must be decided based on following criteria:

1. For cocurrent contact velocity of feed gas mixture at inlet should be less than or equal to 18.3 m/s. To get the higher rate of mass transfer gas velocity at tube, inlet should be around 15 m/s as per the recommendations. (p. 409 of Ref. 11)
2. For efficient operation of falling film absorber certain minimum liquid flow rate is required to maintain a continuous film over the tube wall. Minimum required flow rate of liquid is 150 kg/(h · m of inside periphery of tubes).

Actually once velocity of gas is fixed, flow rate of liquid per unit periphery of tube is fixed by material balance and reverse is also true.

Tube length can be easily determined based on average value of overall heat transfer coefficient U and based on the overall values of heat duty (ϕ) and overall mean temperature difference ($\Delta \mathcal{T}_m$) (difference between temperature of liquid film and temperature of water). Actually rate of absorption, rate of heat transfer, overall heat transfer coefficient and temperature difference across the heat transfer wall are changing from point to point. Hence, for the precise calculations, heat exchanger

can be divided in different zones and for each zone, tube length is determined. Also for each zone, rate of absorption, heat duty and mean temperature difference are calculated. Overall coefficient U is assumed as constant. In trial-and-error calculations, film temperature is assumed. Based on that rate of absorption per unit area, heat duty per unit area and $\Delta\mathcal{T}_m$ are calculated. Check $\phi/A = U\Delta\mathcal{T}_m$. Rate of absorption of gas is given by equation:

Rate of absorption, $$N_A = K_G M(\overline{p}_{\text{AG}} - \overline{p}^*_{\text{AL}})\ \text{kg/(m}^2\cdot\text{s)} \tag{9.46}$$

where, K_G = Overall mass transfer coefficient based on the arithmetic mean partial pressure difference in kmol/(m^2 · atm · s)

M = Molar mass of gas absorbed kg/kmol = 36.5 for HCl or 17 for NH_3

$\overline{p}^*_{\text{AG}}$ = Partial pressure of solute gas in gas mixture, atm

$\overline{p}^*_{\text{AL}}$ = Equilibrium partial pressure of solute A at gas liquid interface, atm

Mass transfer coefficient for hydrogen chloride absorption in the tubular absorber is calculated by following equation[11]:

$$K_G = \frac{1.654\times10^{-5}}{M_{\text{av}}^{1.75}}\left(\frac{d_i G_t}{\mu}\right)\ \text{kmol/(m}^2\cdot\text{atm}\cdot\text{s)} \tag{9.47}$$

where, d_i = Inside diameter of tube, m

G_t = Mass velocity of gas mixture at inlet, kg/(m^2 · s)

μ = Viscosity of gas mixture kg/(m · s)

M_{av} = Average molar mass of gas mixture at inlet, kg/kmol

Heat transfer coefficient of liquid film can be calculated by McAdams, Drew and Bay's equation (Ref. 11).

$$h = 9136\times\tau^{1/3} \tag{9.48}$$

where, h = Tube side heat transfer coefficient, W/(m^2 · °C)

τ = Liquid flow rate per unit perimeter, kg/(m · s)

This correlation is valid for values of τ, ranging from 0.25 to 6.2 kg/(m · s).

Example 9.5 Gas mixture, obtained after the chlorination reaction and separation of organic vapours, contains 20% Cl_2 and 80% HCl by mole. From the mixture, hydrogen chloride gas is to be separated by absorption in water. For the absorption of HCl, falling film absorber (Fig. 9.19), followed by tail gas scrubber are used. Design the falling film absorber.

Flow rate of gas mixture = 2500 kg/h

Temperature of gas mixture = 10°C

% of HCl in the liquid stream leaving the tail gas scrubber and entering at the top of falling film absorber = 5% (by mass)

Desired concentration of HCl in product stream = 32% (by mass)

Operating pressure = 30 kPa g (at inlet)

Fig. 9.19 *Falling Film Absorption System*

Solution

Amount of product solution = q_m kg/h

HCl balance

$$2500 \times \left(\frac{0.8 \times 36.5}{0.8 \times 36.5 + 0.2 \times 71} \right) = q_m \times 0.32$$

$$2500 \times 0.6728 = q_m \times 0.32$$
$$q_m = 5256.25 \text{ kg/h}$$

Tail gas scrubber is operated in adiabatic manner. Hence, the total heat of solution of hydrogen chloride in water (which is highly exothermic) is removed in falling film absorber to maintain nearly isothermal condition. Heat duty of falling film absorber can be calculated based on heat of formation data of aqueous hydrochloric acid solution (Table 9.8).

Table 9.8 Heat of Formation of Aqueous Hydrochloric Acid Solution[13]

Formula	State	Heat of formation (ΔH_f^o) at K, 298 kJ/mol HCl	mass % HCl in aqueous solution
HCl 75 H_2O	aq	–165.72	2.63
HCl 50 H_2O	aq	–165.36	3.90
HCl 40 H_2O	aq	–165.10	4.82
HCl 30 H_2O	aq	–164.67	6.33
HCl 25 H_2O	aq	–164.34	7.50
HCl 20 H_2O	aq	–163.85	9.21
HCl 15 H_2O	aq	–163.03	11.9
HCl 10 H_2O	aq	–161.32	16.9
HCl 8 H_2O	aq	–160.00	20.2
HCl 6 H_2O	aq	–157.68	25.3
HCl 5 H_2O	aq	–155.77	28.9
HCl 4 H_2O	aq	–152.92	33.6
HCl 2 H_2O	aq	–140.96	50.3
HCl H_2O	aq	–121.55	67.0
HCl	gas	–92.31	100.0

Note: The number in the first column indicates the number of moles of water mixed with one mole of hydrogen chloride.

Heat of solution = (ΔH_f^o) of 32% solution – (ΔH_f^o) of HCl gas

Heat of formation of 32% HCl solution = –153.9 kJ/mol HCl (by interpolation)

Heat of solution = (–153.89) – (–92.31) = –61.58 kJ/mol HCl

$$\text{Moles of HCl absorbed} = \frac{2500 \times 0.6728}{36.5} = 46.082 \text{ kmol/h}$$

Heat duty of falling film absorber (including that of tail gas scrubber),

$$\phi_t = 61.58 \times 1000 \times 46.082 = 2837\,729.6 \text{ kJ/h} \equiv 788.26 \text{ KW}$$

(Actually in falling film absorber, concentration of HCl in aqueous solution is increased from 5% to 32% but the tail gas scrubber is operated in adiabatic manner hence heat of solution generated on increasing the concentration from 0 to 5% of HCl is to be added in heat duty of the falling film absorber.)

Temperature of 5% weak acid:
Heat of formation for 5% HCl solution = –165.05 kJ/mol (by interpolation)
Heat of solution = (–165.05) – (–92.31) = –72.74 kJ/mol
Moles of HCl absorbed in adiabatic absorber

$$= \left(\frac{46.082 \times 36.5}{0.32} - 46.082 \times 36.5\right) \times \frac{0.05}{0.95} \times \frac{1}{36.5}$$
$$= 5.154 \text{ kmol/h}$$
$$\equiv 188.121 \text{ kg/h}$$

Heat generated in adiabatic absorber

$$\phi_1 = 72.74 \times 1000 \times 5.154 = 374\,902 \text{ kJ/h} \equiv 104.14 \text{ kW}$$

Mass flow rate of pure water entering to adiabatic absorber = Mass flow rate of 32% solution × (1 – 0.32)

$$\dot{m}_w = 5256.35 \times (1 - 0.32) = 3574.25 \text{ kg/h}$$

Mass flow rate of 5% HCl solution

$$\dot{m}_{w1} = \frac{3574.25}{(1 - 0.05)} = 3762.37 \text{ kg/h}$$

Enthalpy of 5% solution = Enthalpy of pure water + Enthalpy of incoming gas mixture – Enthalpy of exist gas from adiabatic absorber + Heat of solution

Let reference temperature = 30°C
Here pure water enters at room temperature or at a temperature close to 30°C. Temperatures of incoming and exist gas mixture are also close to 30°C. Heat of solution must be determined at average temperature. Neglect the change in the value of heat of solution with temperature.
Heat capacity of 5% HCl solution = 4.19 kJ/(kg · °C)
Enthalpy of 5% HCl solution = $\dot{m}_{w1} \cdot C_L\,(t - 30) = \phi_1$

$$3762.37 \times 4.19(t - 30) = 374\,902$$
$$t = 53.78°\text{C}$$

Mass flow rate of cooling water:
Let inlet temperature of cooling water to absorber = 32°C and outlet temperature of cooling water from absorber = 38°C
Mass flow rate of cooling water,

$$\dot{m}_{cw} = \frac{\phi_t}{C_L \Delta t} = \frac{788.26}{4.1868 \times (38 - 32)} = 31.379 \text{ kg/s}$$
$$= 112\,964.4 \text{ kg/h} \approx 113 \text{ m}^3\text{/h}$$

Material of tube = Graphite
Outside diameter of tube d_o = 25 mm
Inside diameter of tube d_i = 20 mm
It is reported in literature that if the velocity of gas through the tubes is kept more than 15.24 m/s then higher tube side or film side heat transfer coefficient is obtained. There is a sharp increase in the tube side heat transfer coefficient when the velocity of gas at the tube inlet exceeds 15.24 m/s.

Let the velocity of gas at tube inlet = 16 m/s

Average molar mass of feed gas mixture at inlet

$$M_{av} = \Sigma(M_i \cdot y_i) = 0.2 \times 71 + 0.8 \times 36.5 = 43.4 \text{ kg/kmol}$$

Density of feed gas at inlet

$$\rho = \frac{pM}{RT} = \frac{(30 + 101.325) \times 43.4}{(273 + 10)} \times \frac{273}{101.325 \times 22.414}$$

$$\rho = 2.42 \text{ kg/m}^3$$

Volumetric flow rate of gas mixture at tube inlet, $q_v = \dfrac{2500}{2.42}$

$$= 1033 \text{ m}^3\text{/h}$$

Velocity of gas at tube inlet, $v = \dfrac{q_v}{N_t \times \dfrac{\pi}{4} d_i^2} = 16$

$$N_t = \frac{1033/3600}{16 \times \dfrac{\pi}{4}(0.02)^2} = 57$$

Tube side mass velocity, $G_t = u_t\,\rho = 16 \times 2.42 = 38.72 \text{ kg/(m}^2 \cdot \text{s)}$

Tube side heat transfer coefficient

Tube side or falling film side heat transfer coefficient

$$h_i = 9136 \times \tau^{1/3} \tag{9.48}$$

where, h_i is in W/(m^2 · °C)

Liquid flow rate at inlet = 3762.37 kg/s = q_{w_1}

$$\tau = \frac{q_{w1}}{N_t \pi\, d_i} \tag{9.49}$$

$$= \frac{3762.37/3600}{57 \times \pi \times 0.02}$$

$$\tau = 0.2918 \text{ kg/(m} \cdot \text{s) [= 1050.5 kg/(m} \cdot \text{h) > 150 kg/(m} \cdot \text{h)]}$$

$$h_i = 9136 \times (0.2918)^{1/3} = 6059.7 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Shell side heat transfer coefficient

Shell side flow area, $A_s = \dfrac{(P_t - d_o)B_s D_s}{P_t}$ (6.29)

P_t = Tube pitch = 1.25 d_o = 1.25 × 25 = 31.25 mm, d_o = 25 mm

Tube bundle diameter

$$D_b = d_o \left(\frac{N_t}{k_1}\right)^{1/n_1} \tag{6.1}$$

For P_t/d_o = 1.25, triangular pitch, 1-1 shell and tube fixed tube sheet heat exchanger, k_1 = 0.319, n_1 = 2.142 (From Table 6.2)

$$D_b = 25\left(\frac{57}{0.319}\right)^{\frac{1}{2.142}} = 281.4 \text{ mm}$$

$$D_s = D_b + 18.6 \text{ mm} = 300 \text{ mm}, \qquad \text{Let } B_s = 100 \text{ mm}$$

$$A_s = \frac{(31.25 - 25)}{31.25} \times 0.1 \times 0.3 = 0.006 \text{ m}^2$$

Shell side equivalent diameter

$$d_e = \frac{1.1}{d_o}(P_t^2 - 0.907 d_o^2) \tag{6.33}$$

$$= \frac{1.1}{25}(31.25^2 - 0.907 \times 25^2) = 18.026 \text{ mm}$$

Shell side mass velocity, $G_s = \dfrac{\dot{m}_{cw}}{A_s} = \dfrac{31.379}{0.006} = 5229.8 \text{ kg/(m}^2 \cdot \text{s)}$

Shell side velocity, $u_S = \dfrac{G_s}{\rho} = \dfrac{5229.8}{994.032} = 5.26 \text{ m/s} > 3 \text{ m/s (too high)}$

For the liquid flow on shell side, it is better to restrict the shell side velocity below 3 m/s to avoid the liquid induced vibrations.

Change the baffle spacing from 100 to 300 mm.

$$B_s = 300 \text{ mm}$$

$$A_s = \frac{31.25 - 25}{31.25} \times 0.3 \times 0.3 = 0.018 \text{ m}^2$$

Shell side mass velocity, $G_s = \dfrac{31.379}{0.018} = 1743.3 \text{ kg/(m}^2 \cdot \text{s)}$

$$u_s = \frac{G_s}{\rho} = \frac{1743.3}{994.032} = 1.7538 \text{ m/s}$$

Shell side Reynolds number, $Re = \dfrac{d_e G_s}{\mu} = \dfrac{0.018\,026 \times 1743.3}{(0.8 \times 10^{-3})} = 39\,280.9$

(Viscosity of water at 35°C, $\mu = 0.8$ cP)

Prandtl number for water at 35°C

$$Pr = \frac{C_p \mu}{k} = \frac{4.1868 \times (0.8 \times 10^{-3}) \times 10^3}{0.628} = 5.333$$

Evaluation of h_o (shell side heat transfer coefficient):

$$h_o = 0.36 \times \frac{0.628}{0.018\,026} \times (39\,280.9)^{0.55} \times (5.333)^{0.33} \times \left(\frac{\mu}{\mu_w}\right)^{0.14}$$

where, $\dfrac{\mu}{\mu_w} \cong 1$

$$h_o = 7329.3 \text{ W/(m}^2 \cdot \text{°C)}$$

Overall heat transfer coefficient:

Fouling coefficients, $h_{od} = h_{id} = 5000 \text{ W/m}^2 \cdot \text{°C)}$

Thermal conductivity of graphite = 150 W/(m · K) from Appendix of Ref. 14.

$$\frac{1}{U_o} = \frac{1}{7329.3} + \frac{1}{5000} + \frac{0.025 \ln\left(\frac{25}{20}\right)}{2 \times 150} + \frac{25}{20} \times \frac{1}{5000} + \frac{25}{20} \times \frac{1}{6059.7}$$

$$U_o = 1232.6 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

The temperature of the acid film on inside surface of tubes attains an equilibrium value at which the rate of heat transfer to the cooling water balances the rate of heat release due to absorption. This temperature is calculated by trial-and-error method for a series of points in the absorber.

Point 1: Bottommost point of tubes: At this point aqueous acid film concentration of liquid film is 32% (by mass). Liquid composition at point 1:

Table 9.9 Composition of Bottom Solution (Point 1)

Component	kg/h	mass %	kmol/h
HCl	1682.00	32	46.082
H_2O	3574.25	68	198.570
Total	5256.25	100	244.650

HCl amount in 5% acid stream = 188.12 kg/h

HCl balance around falling film absorber:

HCl in exist gas stream = HCl in inlet gas stream + HCl in 5% acid stream – HCl in product stream

HCl in exist gas stream = 2500 × 0.6728 + 188.12 – 1682 = 188.12 kg/h

Cl_2 in exist gas stream = 2500 (1 – 0.6728) = 818 kg/h

Table 9.10 Gas Composition at Point 1

Component	kg/h	mass %	kmol/h	mole %
HCl	188.12	18.7	5.154	30.91
Cl_2	818.00	81.3	11.520	69.09
Total	1006.12	100.0	16.674	100.00

Partial pressure of HCl gas in gas mixture at point 1

$$\begin{aligned}\bar{p}_{AG} &= p_t \times y_{\text{HCl}} \\ &= 131.325 \times 0.3091 \\ &= 40.6 \text{ kPa} \equiv 0.4007 \text{ atm}\end{aligned}$$

Equilibrium Partial Pressure of HCl ($\bar{p}^*_{AL}$)

Equilibrium Partial Pressure of HCl gas over aqueous solution of hydrochloric acid can be calculated by the following equation:

$$\log \bar{p}^*_{\text{AL}} = A - \frac{B}{T}$$

where, $\bar{p}^*_{\text{AL}}$ is in Torr and T is in K (Table 3.11 of Ref. 3)

Table 9.11 Antoine Equation Constants[3]

% HCl by mass	*A*	*B*
2	11.8037	4736
4	11.6400	4471
6	11.2144	4202
8	11.0406	4042
10	10.9311	3908
12	10.79	3765
14	10.6954	3636
16	10.6261	3516
18	10.4957	3376
20	10.3833	3245
22	10.3172	3125
24	10.2185	2995
26	10.1303	2870
28	10.0115	2732
30	9.8763	2593
32	9.7523	2457
34	9.6061	2316
36	9.5262	2279

Let the temperature of acid film at point 1 = 35.5°C. For 32% HCl solution, equilibrium partial pressure of HCl over solution,

$$\log \bar{p}^*_{\mathrm{AL}} = 9.7523 - \frac{2457}{(35.5 + 273)}$$

$$\bar{p}^*_{\mathrm{AL}} = 61.37 \text{ Torr} \equiv 0.08 \text{ atm}$$

Rate of Absorption, $N_A = K_G M\ (\bar{p}^*_{\mathrm{AG}} - \bar{p}^*_{\mathrm{AL}})$ kg/(m^2 · atm · s) (9.46)

where, M = Molar mass of HCl = 36.5 kg/mol

Mass transfer coefficient

$$K_G = \frac{1.654 \times 10^{-5}}{M_{\mathrm{av}}^{1.75}} \left(\frac{d_i G_t}{\mu} \right) \tag{9.47}$$

Average molar mass $M_{\mathrm{av}} = \Sigma(y_i M_i)$

$$M_{\mathrm{av}} = 0.3091 \times 36.5 + 0.6909 \times 71 = 60.336 \text{ kg/mol}$$

$$d_i = 0.02 \text{ m}, \quad G_t = \frac{q_m}{a_t} = \frac{(1006.12 / 3600)}{57 \times \frac{\pi}{4}(0.02)^2} = 15.607 \text{ kg/(m}^2 \cdot \text{s)}$$

Viscosity of gas mixture at low pressure can be calculated by the following equations:

$$\mu_{\text{mix}} = \sum_{i=1}^{n} \frac{y_i \mu_i}{\sum_{j=1} y_i \Theta_{ij}} \tag{6.77}$$

where, $$\Theta_{ij} = \frac{\left[1 + \left(\frac{\mu_i}{\mu_j}\right)^{1/2} \left(\frac{M_j}{M_i}\right)^{\frac{1}{4}}\right]^2}{[8(1 + M_i/M_j)]^{1/2}}$$

and $$\Theta_{ji} = \frac{\mu_i}{\mu_j}\left(\frac{M_i}{M_j}\right)\Theta_{ij}$$

At 35.5°C, $\mu_{\text{HCl}} = \mu_i = 0.014$ cP

$\mu_{\text{Cl}_2} = \mu_i = 0.014$ cP

$\mu_{\text{mix}} = 0.014 \text{ cP} = 0.014 \times 10^{-3}$ kg/(m · s)

$$K_G = \frac{1.654 \times 10^{-5}}{60.336^{1.75}}\left(\frac{0.02 \times 15.607}{0.014 \times 10^{-3}}\right) = 2.823 \times 10^{-4} \text{ kmol/(m}^2 \cdot \text{atm} \cdot \text{s)}$$

$$N_A = 2.823 \times 10^{-4} \times 36.5 \times (0.4007 - 0.08) = 3.3 \times 10^{-3} \text{ kg/(m}^2 \cdot \text{s)}$$

Heat of solution of 32% HCl

$$\Delta H_s = -61.58 \text{ kJ/mol} \equiv -61\ 580 \text{ kJ//kmol} \equiv -1687.12 \text{ kJ/kg}$$

Heat release due to absorption $= N_A \times (-\Delta H_s) = 3.3 \times 10^{-3} \times 1687.12$

$= 5.567$ kW/m^2

Rate of heat transfer at point 1 between acid film and cooling water

$$\frac{\phi}{A} = U\Delta t = 1232.6 \times (35.5 - 32)$$

$$= 4314.1 \text{ W/m}^2 \equiv 4.314 \text{ kW/m}^2$$

Heat release due to absorption $\neq U\Delta t$

Second trial

Let acid film temperature at point 1 = 36.5°C

Equilibrium partial pressure of HCl for 32% solution at 36.5°C:

$$\overline{p}_{AL}^{*} = 65.116 \text{ Torr} = 0.0857 \text{ atm}$$

Heat release due to absorption $= 2.8233 \times 10^{-4} \times 36.5\ (0.4007 - 0.0857)$

$= 3.25 \times 10^{-3}$ kg/(m^2 · s)

Heat release due absorption $= 3.25 \times 10^{-3} \times 1687.12 = 5.48$ kW/m^2

$$\frac{\phi}{A} = U\Delta t = 1232.6 \times (36.5 - 32)$$

$$= 5546.7 \text{ W/m}^2$$

$$= 5.546 \text{ kW/m}^2$$

Heat release due to absorption $\approx U\Delta t = q/A$

Hence, temperature of acid film at point 1 = 36.5°C

Point 2: At this point concentration of aqueous acid film is 26% (by mass).

Mass flow rate of 26% HCl solution = $\dfrac{5256.25 \times 0.68}{(1 - 0.26)}$ = 4830.07 kg/h

Amount of HCl absorbed in between points 1 and 2 = 5256.25 – 4830.07
= 426.18 kg/h

Composition of liquid mixture at point 2:

Table 9.12 Liquid Composition at Point 2

Component	kg/h	mass %
HCl	1255.82	26
Water	3574.25	74
Total	4830.07	100

Table 9.13 Composition of Gas Mixture at Point 2

Component	kg/h	kmol/h	mole %
HCl	614.3	16.83	59.365
Cl_2	818.0	11.52	40.635
Total	1432.3	28.35	100.000

For the first trial calculations let the temperature of 26% acid film at point 2 = 40°C

$$\log \overline{p}^*_{AL} = 10.1303 - \frac{2870}{(40 + 273)}$$

$$\overline{p}^*_{AL} = 9.14 \text{ Torr} = 0.012 \text{ atm}$$

$$\overline{p}_{AG} = y_{\text{HCl}} \times p_t = 0.593\,65 \times 131.325 = 77.96 \text{ kPa} \equiv 0.7694 \text{ atm}$$

Average molar mass of gas mixture

$$M_{\text{av}} = \Sigma(y_i M_i) = 0.593\,65 \times 36.5 + 0.403\,35 \times 71 = 50.306 \text{ kg/kmol}$$

Mass velocity of gas mixture

$$G_t = \frac{q_m}{a_t} = \frac{1432.3/3600}{57 \times \dfrac{\pi}{4}(0.02)^2} = 22.218 \text{ kg/(m}^2 \cdot \text{s)}$$

At point 2

At 40°C, $\mu_{Cl_2} = 0.0145$ cP, $\mu_{\text{HCl}} = 0.0145$ cP

$$K_G = \frac{1.654 \times 10^{-5}}{M_{\text{av}}^{1.75}}\left(\frac{d_i G_t}{\mu}\right) = \frac{1.654 \times 10^{-5}}{50.306^{1.75}} \times \left(\frac{0.02 \times 22.218}{0.0145 \times 10^{-3}}\right)$$

$$= 5.334 \times 10^{-4} \text{ kmol/(atm} \cdot \text{m}^2 \cdot \text{s)}$$

Rate of absorption, $N_A = K_G M(\overline{p}_{AG} - \overline{p}^*_{AL})$

$$N_A = 5.334 \times 10^{-4} \times 36.5 \times (0.7694 - 0.012) = 0.014\,746 \text{ kg/(m}^2 \cdot \text{s)}$$

Heat of solution for 26% HCl solution

$$\Delta H_s = (\Delta H_f^o)_{26\%\ HCl} - (\Delta H_f^o)_{HCl\ gas}$$
$$= (-157.3) - (92\ .31) = -65\ \text{kJ/mol}$$
$$\equiv -65\ 000\ \text{kJ/kmol} \equiv -1780.82\ \text{kJ/kg}$$

Heat release due to absorption (per unit area)

$$\phi = N_A \times (-\Delta H_s) = 0.014\ 746 \times 1780.82 = 26.26\ \text{kW/m}^2$$

Heat duty required in between 1 and 2

ϕ_{12} = Total heat duty of falling film absorber – Heat duty required to form 26% HCl solution

ϕ_{12} = 788.26 kW – $(-\Delta H_s)$ × kmol/h of HCl absorbed to form 26% solution

$$\phi_{12} = 788.26 - 65\ 000 \times \left(46.082 - \frac{426.18}{36.5}\right) \times \frac{1}{3600}$$
$$= 167.0435\ \text{kW} = q_{cw}\ C_L(t - 32)$$
$$167.0435 = 31.379 \times 4.1868 \times (t - 32)$$

Temperature of cooling water at point 2, t = 33.27°C

$$\frac{\phi}{A} = U\ \Delta t = 1232.6 \times (40 - 33.27) = 8295.4\ \text{W/m}^2 \equiv 8.295\ \text{kW/m}^2$$
$$= 8.295\ \text{kW/m}^2 \neq \text{Heat release due to absorption.}$$

Second trial

Let temperature of 26% acid film at point 2 = 54°C

Equilibrium partial pressure of HCl over solution

$$\log\ \overline{p}_{AL}^* = 10.1303 - \frac{2870}{(54 + 273)}$$
$$\overline{p}_{AL}^* = 22.57\ \text{Torr} \equiv 0.0297\ \text{atm}$$

At 54°C, $\mu_{HCl} = \mu_{Cl_2} = 0.015$ cP (Fig. 3.14 of Ref. 3)

$$K_G = 5.334 \times 10^{-4} \times \frac{0.0145}{0.0155}$$
$$= 4.9899 \times 10^{-4}\ \text{kmol/(m}^2 \cdot \text{atm} \cdot \text{s)}$$
$$N_A = 4.9899 \times 10^{-4} \times 36.5 \times (0.7694 - 0.0297)$$
$$= 0.013\ 47\ \text{kg/(m}^2 \cdot \text{s)}$$

Heat release due to absorption = 0.013 47 × 1780.82 = 23.9876 kW/m^2

$$\frac{\phi}{A} = U\ \Delta t = 1232.6 \times (54 - 33.27)$$
$$= 25\ 551.8\ \text{W/m}^2 = 25.55\ \text{kW/m}^2 \neq \text{Heat release}$$

Third trial

Let temperature of 26% Acid film = 52.5°C

$$\overline{p}_{AL}^* = 20.563\ \text{Torr} = 0.027\ \text{atm}$$
$$N_A = 4.9899 \times 10^{-4} \times 36.5\ (0.7694 - 0.027) = 0.013\ 52\ \text{kg/(m}^2 \cdot \text{s)}$$

Heat release due to absorption = 0.013 52 × 1780.82 = 24.076 kW/m^2

$$\frac{\phi}{A} = U\,\Delta t = 1232.6 \times (52.5 - 33.27) = 23\,703\ \text{W/m}^2 \equiv 23.7\ \text{kW/m}^2$$

$$\frac{\phi}{A} = U\,\Delta t \approx \text{Heat release due to absorption (close enough)}$$

Temperature of acid film = 52.5°C
Heat transfer between points 1 and 2:

$$\phi_{12} = 167.0435\ \text{kW}, \quad U_o = 1232.6\ \text{W/(m}^2 \cdot {}^\circ\text{C)}$$

$$\Delta\mathcal{T}_{\ln} = \frac{(52.5 - 33.27) - (36.5 - 32)}{\ln\left(\dfrac{52.5 - 33.27}{36.5 - 32}\right)} = 10.142^\circ\text{C}$$

$$\text{Area required } A_{12} = \frac{\phi_{12}}{U_o \Delta\mathcal{T}_{\ln}} = \frac{167.0435 \times 10^3}{1232.6 \times 10.142} = 13.362\ \text{m}^2$$

$$N_t\,\pi\,d_o\,L_{12} = A_{12} = 13.362\ \text{m}^2 \text{ or } L_{12} = \frac{13.362}{57 \times \pi \times 0.025} = 2.984\ \text{m}$$

Point 3: At this point aqueous acid film contains 20% by mass HCl.
Let liquid film temperature at point 3 = 68°C

Table 9.14 Liquid Composition at Point 3

Component	kg/h	mass %	kmol/h
HCl	893.56	20	24.48
H_2O	3574.25	80	198.57
Total	4467.81	100	223.05

Table 9.15 Gas Composition at Point 3

Component	kg/h	kmol/h	mole %
HCl	976.56	26.755	69.9
Cl_2	818.00	11.52	30.1
Total	1794.56	38.275	100.0

Equilibrium partial pressure of HCl over 20% HCl solution:

$$\log \overline{p}^*_{AL} = 10.3883 - \frac{3245}{T}$$

$$= 10.3833 - \frac{3245}{(273 + 68)}$$

$$\overline{p}^*_{AL} = 7.365\ \text{Torr} \equiv 0.0097\ \text{atm}$$

$$\overline{p}_{AL} = p_t \times y_{\text{HCl}} = 131.325 \times 0.699 = 91.8\ \text{kPa} \equiv 0.906\ \text{atm}$$

Average molar mass of gas mixture,

$$M_{av} = \Sigma(M_i y_i) = 0.699 \times 36.5 + 0.301 \times 71 = 46.885$$

Mass velocity, $G_t = \dfrac{1794.56/3600}{57 \times (\pi/4)(0.02)^2} = 27.8375 \text{ kg/(m}^2 \cdot \text{s)}$

At 68°C, $\mu = \mu_{HCl} \cong \mu_{Cl_2} = 0.0162$ cP

$$K_G = \frac{1.654 \times 10^{-5}}{M_{av}^{1.75}}\left(\frac{d_i G_t}{\mu}\right) = \frac{1.654 \times 10^{-5}}{46.885^{1.75}} \times \left(\frac{0.02 \times 27.8375}{0.0162 \times 10^{-3}}\right)$$

$$K_G = 6.766 \times 10^{-4} \text{ kmol/(atm} \cdot \text{m}^2 \cdot \text{s)}$$

Rate of absorption,

$$N_A = K_G M(\overline{p}_{AG} - \overline{p}^*_{AL})$$
$$= 6.766 \times 10^{-4} \times 36.5 \times (0.906 - 0.0097)$$
$$= 0.022\,14 \text{ kg/(m}^2 \cdot \text{s)}$$

Heat of solution of 20% HCl solution $\Delta H_s = (-160.08) - (-92.31)$

$$\Delta H_s = -67.77 \text{ kJ/mol} \equiv -67\,770 \text{ kJ/mol} \equiv -1856.7 \text{ kJ/kg}$$

Rate of heat generated due to absorption $= N_A \times (-\Delta H_s)$

$$= 0.022\,14 \times 1856.7 = 41.1073 \text{ kW/m}^2$$

Amount of HCl absorbed in between point 3 and 2 = 362.26 kg/h

Heat duty required in between points 2 and 3

$$\phi_{23} = \phi_t - \phi_{12} - \text{Heat required to form 20\% HCl solution}$$

Heat required to form 20% HCl solution

$$= -\Delta H_s \times 893.56 \text{ kg/h}$$
$$= 1856.7 \times 893.56 = 1659\,072.9 \text{ kJ/h} \equiv 460.85 \text{ kW}$$

$$\phi_{23} = 788.26 - 167.044 - 460.85 = 160.366 \text{ kW}$$
$$= 160.336 \text{ kW} = \dot{m}_{cw} \cdot C_L\,(t - 33.27)$$
$$160.336 = 31.379 \times 4.1868 \times (t - 33.27)$$
$$t = 34.49°\text{C} = \text{Temperature of cooling water at point 3}$$

$$\frac{\phi}{A} = U\Delta t = 1232.6 \times (68 - 34.49) = 41\,304 \text{ W/m}^2 \equiv 41.304 \text{ kW/m}^2$$

= Rate of heat generated due to absorption

Temperature of acid film at point 3 = 68°C

$$\phi_{23} = 160.336 \text{ kW}, \qquad U = 1232.6 \text{ W/(m}^2 \cdot °\text{C)}$$

$$\Delta T_{lm} = \frac{(68 - 34.49) - (52.5 - 33.27)}{\ln\left(\dfrac{68 - 34.49}{52.5 - 33.27}\right)} = 25.71°\text{C}$$

$$A_{23} = \frac{\phi_{23}}{U\Delta_{lm}} = \frac{160.336 \times 10^3}{1232.6 \times 25.71} = 5.0595 \text{ m}^2 = N_t\,\pi\,d_o\,L_{23}$$

$$57 \times \pi \times 0.025 \times L_{23} = 5.0595 \text{ m}^2$$
$$L_{23} = 1.13 \text{ m}$$

Point 4: Assume that at this point aqueous acid contains 12% HCl.

Table 9.16 Liquid Composition at Point 4

Component	kg/h	mass %
HCl	487.4	12
H_2O	3574.25	88
Total	4061.65	100

Table 9.17 Gas Mixture Composition at Point 4

Component	kg/h	mass %	kmol/h	mole %
Cl_2	818	37.17	11.52	23.32
HCl	1382.72	62.83	37.883	76.68
Total	2200.72	100.00	49.403	100.00

Let temperature of acid film at point 4 = 72°C
Equilibrium partial pressure of HCl over 12% HCl solution

$$\log \bar{p}^*_{AL} = 10.79 - \frac{3765}{T} = 10.79 - \frac{3765}{(273+72)}$$

$$= 0.753 \text{ Torr} \equiv 9.9 \times 10^{-4} \text{ atm}$$

$$\bar{p}_{AG} = y_{\text{HCl}} \times p_t = 0.7668 \times 131.325$$

$$= 100.7 \text{ kPa} \equiv 0.9938 \text{ atm}$$

$$M_{\text{av}} = \Sigma(y_i M_i) = 0.7668 \times 36.5 + 0.2332 \times 71 = 44.55 \text{ kg/mol}$$

$$G_t = \left(\frac{2200.72/3600}{57 \times (\pi/4)(0.02)^2}\right) = 34.138 \text{ kg/(m}^2 \cdot \text{s)}$$

Mass transfer coefficient
At 72°C, $\mu = \mu_{\text{HCl}} = \mu_{\text{Cl}_2} = 0.0163$ cP

$$K_G = \frac{1.654 \times 10^{-5}}{44.55^{1.75}} \times \left(\frac{0.02 \times 34.138}{0.0163 \times 10^{-3}}\right)$$

$$= 9.0185 \times 10^{-4} \text{ kmol/(m}^2 \cdot \text{atm} \cdot \text{s)}$$

Rate of absorption

$$N_A = K_G M \ (\bar{p}_{AG} - \bar{p}^*_{AL})$$

$$= 9.0185 \times 10^{-4} \times 36.5 \ (0.9938 - 9.9 \times 10^{-4})$$

$$= 0.032\,68 \text{ kg/(m}^2 \cdot \text{s)}$$

Heat of formation of 12% HCl solution $\Delta H^{\text{o}}_{\text{f}} = -163$ kJ/mol
Heat of solution of 12% HCl solution

$$\Delta H_s = (\Delta \text{H}^{\text{o}}_{\text{f}})_{12\% \text{ HCl}} - (\Delta \text{H}^{\text{o}}_{\text{f}})_{\text{HCl gas}}$$

$$= -163 - (-92.31) = -70.69 \text{ kJ/mol}$$

$$\equiv -70\,690 \text{ kJ/mol} \equiv -1936.7 \text{ kJ/kg}$$

Heat release due to absorption

$$= 0.032\,68 \times 1936.7 = 63.29 \text{ kW/m}^2$$

Heat duty required in between points 3 and 4

$$\phi_{34} = \phi_t - \phi_{12} - \phi_{23} - \text{Heat duty required to form 12\% HCl solution}$$

$$\phi_{34} = 788.26 - 167.044 - 160.366 - (1936.7 \times 487.4) \times \frac{1}{3600}$$

$$= 198.64 \text{ kW} = q_{cw}\, C_l\, \Delta t_{34} = 31.379 \times 4.1868 \times (t - 34.49)$$

$$t = 36°\text{C} = \text{Temperature of cooling water at point 4}$$

$$\frac{\phi}{A} = U\,\Delta t = 1232.6 \times (72 - 36) = 44\,374 \text{ W/m}^2 \equiv 44.374 \text{ kW/m}^2$$

Hence $U\Delta t \neq$ heat release due to absorption

Second trial

Let film temperature of 12% HCl solution = 85°C

$$\log \overline{p}^*_{AL} = 10.79 - \frac{3765}{(85 + 273)}$$

$$\overline{p}^*_{AL} = 1.876 \text{ Torr} \equiv 0.0025 \text{ atm}$$

Viscosity of gas mixture $\mu = 0.017$ cP

$$K_G = 9.0185 \times 10^{-4} \times \frac{0.0163}{0.017}$$

$$= 8.647 \times 10^{-4} \text{ kmol/(m}^2 \cdot \text{atm} \cdot \text{s)}$$

$$N_A = K_G M(\overline{p}_{AG} - \overline{p}^*_{AL}) = 8.64\text{ss}7 \times 10^{-4} \times 36.5 \times (0.9938 - 0.0025)$$

$$= 0.031\,29 \text{ kg/(m}^2 \cdot \text{s)}$$

Heat release due to absorption $= N_A \times (-\Delta H_s) = 0.031\,29 \times 1936.7 = 60.6 \text{ kW/m}^2$

$$\frac{\phi}{A} = U\Delta t = 1232.6 \times (85 - 36) = 60\,397 \text{ W/m}^2 = 60.397 \text{ kW/m}^2$$

$$\neq \text{Heat release due to absorption}$$

Third trial

Let film temperature of 12% HCl solution = 85.1°C

$$\overline{p}^*_{AL} = 1.889 \text{ Torr} = 0.0025 \text{ atm}$$

$$N_A = 8.647 \times 10^{-4} \times 36.5 \times (0.9938 - 0.0025) = 0.031\,287 \text{ kg/(m}^2 \cdot \text{s)}$$

Heat release due to absorption $= 0.031\,287 \times 1936.7 = 60.59 \text{ kW/m}^2$

$$\frac{\phi}{A} = U\,\Delta t = 1232.6 \times (85.1 - 36) = 60\,521 \text{ W/m}^2 \equiv 60.52 \text{ kW/m}^2$$

$$\frac{\phi}{A} = U\,\Delta t = \text{Heat release due to absorption}$$

$$\phi_{34} = 198.64 \text{ kW}, \; U_o = 1232.6 \text{ W/m}^2$$

$$\Delta\mathcal{T}_{\ln} = \frac{(85.1 - 36) - (68 - 34.49)}{\ln\left(\dfrac{85.1 - 36}{68 - 34.49}\right)} = 40.81°\text{C}$$

$$A_{34} = \frac{\phi_{34}}{U_o \Delta\mathcal{T}_{\ln}} = \frac{198.64 \times 10^3}{1232.6 \times 40.81} = 3.949 \text{ m}^2 = N_t\, \pi\, d_o\, L_{34}$$

$$L_{34} = \frac{3.949}{57 \times \pi \times 0.025} = 0.88 \text{ m}$$

Point 5: At this point concentration of aqueous acid film is 5% HCl (by mass).

Table 9.18 Liquid Composition at Point 5

Component	kg/h	mass %
HCl	188.12	5
H_2O	3574.25	95
Total	3762.37	100

Let film temperature of 5% HCl solution = 97.5°C
For 5% HCl solution

$$\log\ \overline{p}^*_{AL} = 11.4272 - \frac{4336.5}{T}$$

$$\overline{p}^*_{AL} = 0.5281 \text{ Torr} = 6.95 \times 10^{-4} \text{ atm}$$

$$\overline{p}_{AG} = 0.8 \times 131.325 = 105.06 \text{ kPa} \equiv 1.0369 \text{ atm}$$

$$M_{\text{av}} = 0.8 \times 36.5 + 0.2 \times 71 = 43.4 \text{ kg/kmol}$$

$$G_t = \frac{\left(\dfrac{2500}{3600}\right)}{57 \times \dfrac{\pi}{4}(0.02)^2} = 38.78 \text{ kg/(m}^2 \cdot \text{s)}$$

Viscosity of gas mixture at 97.5°C, $\mu = 0.018$ cP

$$K_G = \frac{1.654 \times 10^{-5}}{43.4^{1.75}}\left(\frac{0.02 \times 38.78}{0.018 \times 10^{-3}}\right) = 9.7116 \times 10^{-4} \text{ kmol/(atm} \cdot \text{m}^2 \cdot \text{s)}$$

$$N_A = 9.7116 \times 10^{-4} \times 36.5 \times (1.0369 - 6.95 \times 10^{-4}) = 0.036\,73 \text{ kg/(m}^2 \cdot \text{s)}$$

Rate of heat generated due to absorption = $N_A \times (-\Delta H_s)$

$$= 0.036\,73 \times \left(72.74 \times 1000 \times \frac{1}{36.5}\right)$$

$$= 73.2 \text{ kW/m}^2$$

At point 5

$$\frac{\phi}{A} = U\Delta t = 1232.6\ (97.5 - 38) = 73\,340 \text{ W/m}^2 \equiv 73.34 \text{ kW/m}^2$$

$\cong$ Heat release due to absorption

Table 9.19 Gas Composition at Point 5

Component	kg/h	kmol/h	mole %
HCl	1682	46.082	80
Cl_2	818	11.521	20
Total	2500	57.603	100

Equilibrium acid film temperature at point 5 = 97.5°C

$$\phi_{45} = 788.26 - 167.044 - 160.366 - 198.64$$

$$\phi_{45} = 262.2 \text{ kW}, \quad U = 1232.6 \text{ W/(m}^2 \cdot \text{°C)}$$

$$\Delta T_{lm} = \frac{(97.5 - 38) - (85.1 - 36)}{\ln\left(\dfrac{97.5 - 38}{85.1 - 36}\right)} = 54.134\text{°C}$$

$$A_{45} = \frac{\phi_{45}}{U_o \Delta T_{lm}} = \frac{262.2 \times 10^3}{1232.6 \times 54.134} = 3.9295 \text{ m}^2$$

$$L_{45} = \frac{A_{45}}{N_t \pi\, d_o} = \frac{3.9295}{57 \times \pi \times 0.025} = 0.878 \text{ m}$$

Aqueous 5% HCl solution enters the falling film absorber at 53.78°C. Hence, it will absorb the heat to reach equilibrium temperature (97.5°C). Actually this direct heating of liquid film will reduce some heat duty of heat exchanger. So, further addition in heat transfer area is not required.

Table 9.20 Summary of Zonewise Calculations for Falling Film Absorber

Point	% HCl (mass)	Water, kg/s	Temperature of acid film, °C	Temperature of water °C	Heat Transfer Area, m^2	Length from, bottom, m
1 (bottom)	32	31.379	36.5	32		
2	26	31.379	52.5	33.27	13.362	2.984
3	20	31.379	68	34.49	5.059	1.13
4	12	31.379	85.1	36	3.949	0.95
5 (top)	5	31.379	97.5	38	3.9295	0.88
Total					26.300	5.944

Normally 10% excess area is provided.
Desired HTA = 26.3 × 1.1 = 28.93 m^2
Select two heat exchangers with equal HTA in series.
HTA per heat exchanger = 28.93/2 = 14.465 m^2
Let tube length be L.

$$57 \times L \times \pi \times 0.025 = 14.465$$

$$L = 3.23 \text{ m}$$

Carbate tube length equal to 3 m is standard. To provide sufficient area, provide 58 tubes instead of 57. This will mean 14.137 m^2 HTA. Excess area with two heat exchangers will be 4.0%.

Comment: For the given case, falling film absorber with glass tubes can be considered as another option. But glass falling film absorber with once through operation for the given case cannot be used. Conductive coefficient, offered by glass tubes, is very low hence, it will control the value of overall coefficient. For absorption of HCl in water with once through operation in glass tube heat exchanger, temperature of acid film in upper portion of absorber reaches boiling temperature. However, glass falling film absorber can be used for the given case with only recirculation of hydrochloric acid solution. Further to reduce the acid film temperature, gas feed can be diluted by air. Recirculation of hydrochloric acid solution or addition of air in gas feed decreases acid film temperatures but increases heat transfer area required for the falling film absorber.

9.8 PROCESS DESIGN OF SIEVE PLATE ABSORBER

Various process design aspects of a sieve-plate column were covered in Sec. 8.4.7.1. An example of the sieve-plate column for absorption is given below:

Example 9.6 Design a crossflow-type sieve plate absorber for the absorption of formaldehyde from the Reactor Exit Gas Mixture by water to make 37% (by mass) of formaldehyde solution in water. Material balance data around absorber of formaldehyde plant (Via Formox process) are given as follows.

Reactor exit gas is at 110°C (from recuperative heat exchanger) and at 135 kPa a pressure.

Enthalpies of reactor exit gas mixture, absorber outlet gas mixture, bottom product solution and fresh water with reference to 298.15 K (25°C) are 1141.77 kW, 295.38 kW, 216.35 and 32.17 kW, respectively. Exothermic heat of solution of formaldehyde gas in water is substantially independent of concentration and has a value of 62.75 kJ/mol HCHO at 298.15 K up to a concentration of about 40% (by mass) of HCHO. Fresh water enters at 30°C and product solution leaves the absorber at 50°C. Temperature of exit gas from absorber is 50°C (Ref. 13).

Table 9.21 Stream Compositions across Absorber[13]

Component	Reactor Exit Gas		Absorber Outlet Gas		Bottom Product	
	kg/h	mass %	kg/h	mass %	kg/h	mass %
CH_3OH	40	0.09	nil	—	40.0	0.44
O_2	6 806.8	15.81	6 806.8	17.21		
N_2	29 626.8	68.81	29 626.8	74.9		
H_2O	2 766.0	6.42	2 649.0	6.7	5649.1	62.56
HCHO	3 341.3	7.76	nil	—	3341.3	37.00
CO_2	386.6	0.9	386.6	0.98		
CO	27.7	0.07	27.7	0.07		
H_2	4.0	0.01	4.0	0.01		
CH_4	9.9	0.02	9.9	0.02		
$(CH_3)_2O$	45.5	0.11	45.5	0.11		
Total	43 054.6	100.00	39 556.3	100.00	9030.4	100.00

Solution

Total moles of formaldehyde absorbed = $\dfrac{3341.3}{30}$ = 111.376 kmol/h

Heat of solution = 62.75 kJ/mol

Total heat evolved by absorption

$$= 111.376 \times 62.75 \times 1000 = 6988\ 844 \text{ kJ/h} \equiv 1941.35 \text{ kW}$$

This exothermic physical absorption is carried out either in tray tower or packed tower in actual practice. In packed tower or tray tower, recirculation streams are provided and these recirculation streams are passing through external coolers. Recirculation streams are provided to control the temperature of absorption. To achieve the complete absorption, final contact of exit gas is with fresh water, which is fed from top.

In tray tower, intercoolers are provided. These intercoolers may be shell and tube type or plate type or spiral heat exchangers. Here tray tower with sieve trays is designed.

Total heat to be removed by coolers

= Total enthalpy in + Heat generated due to absorption – Total enthalpy out

= (1141.77 + 32.17) + 1941.35 – (295.38 + 216.35) = 2603.56 kW

Flow rate of fresh water fed at the top of tower = 5649.1 + 2649 – 2766 = 5532.1 kg/h

Absorption of formaldehyde in water is highly exothermic but it can be carried out approximately in isothermal condition by using intercoolers and providing recirculation streams.

Let the average temperature of absorption in the entire tower is 45°C.

Consider the topmost section of tower [Refer to Figs. (4.2) and (4.8)]. To achieve the complete absorption of formaldehyde, gas mixture must be finally contacted with pure water. Recirculation cannot be provided in the topmost section. If dilute solution of formaldehyde is fed from the top with fresh water, then it limits the concentration of formaldehyde in the exist gas.

Assuming that the heat of solution is utilised only in increasing the temperature of formaldehyde solution. Fix the temperature of formaldehyde solutions leaving the topmost section as 60°C.

Enthalpy of solution leaving the top most (first) section – Enthalpy of fresh water

= Heat of solution × Formaldehyde absorbed in top section

$$\frac{\left(\dfrac{5532.1}{3600}\right)}{\left(1-\dfrac{w_1}{100}\right)} C_L(60-25) - \frac{(5532.1 \times 4.1868(30-25))}{3600}$$

$$= \frac{62.75 \times 1000}{30 \times 3600}\left(\frac{5532.1}{\left(1-\dfrac{w_1}{100}\right)} - 5532.1\right) \qquad \text{(a)}$$

where, w_1 = Mass % of formaldehyde in solution leaving the first section

Specific heat capacity (C_L) of formaldehyde solution is given by following equation[15]:

$$C_L = 4.1868\ [1 - 0.005\ 34\ w + (0.004 + 0.000\ 01w)\ t] \qquad \text{(b)}$$

where, C_L is in kJ/(kg · °C), w is mass %, t is temperature in °C.

Here, $t = \dfrac{60+25}{2} = 42.5°\text{C}$ and $w = w_1$

$$C_L = 4.9 - 0.0206\, w_1 \qquad \text{(c)}$$

From (a) and (c),

$$\frac{1.5367}{(1-0.01\,w_1)}\,(4.9 - 0.0206\,w_1)\,(60-25) - 32.17$$

$$= 0.581 \times 5532.1\left(\frac{1}{(1-0.01\,w_1)} - 1\right)$$

$$\frac{53.7845}{(1-0.01\,w_1)}\,(4.9 - 0.0206\,w_1) - 32.17$$

$$= 3214.15\left(\frac{1}{(1-0.01\,w_1)} - 1\right)$$

By trial-and-error calculations

For $w_1 = 7.0$, LHS ≅ RHS = 242 kW

Hence, mass % of formaldehyde in solution leaving the topmost (first) section = 7.0%
This solution is leaving at 60°C.
Provide the first intercooler to cool this solution from 60°C to 40°C by cooling water.
Heat duty of first intercooler.

$$\phi_I = \dot{m}\,C_L(60-40)$$

$$\dot{m} = \frac{5532.1}{1-(0.01\times 7.0)} = 5948.5 \text{ kg/h} \equiv 1.6524 \text{ kg/s}$$

$$C_L = 4.1868\left[(1 - 0.005\,34\times 7.0) + (0.004 + 0.000\,01\times 7)\left(\frac{40+60}{2}\right)\right]$$

$$C_L = 4.88 \text{ kJ/(kg}\cdot°\text{C)}$$

$$\phi_1 = 1.6524 \times 4.88\,(60-40) = 161.27 \text{ kW}$$

Stream leaves the first intercooler and enters at the top of second section which is 7% (by mass) formaldehyde solution at 40°C.
This stream is mixed with recirculated stream as shown in Figs. (4.2) and (4.8).
Let recirulation ratio = R
Mass flow rate of recirculated stream = $R \times 1.6524$
Total mass flow rate of formaldehyde solution feed to the top of the second section
$= (R+1) \times 1.6524.$
% by mass of formaldehyde in liquid solution leaving the second section = 37%
Mass flow rate of formaldehyde solution leaving the second section

$$= R \times 1.6524 + \frac{\left(\dfrac{5532.1}{3600}\right)}{\left[1 - \left(\dfrac{37}{100}\right)\right]} = (R \times 1.6524 + 2.4392) \text{ kg/s}$$

This stream is leaving at 50°C.
Enthalpy balance around second section:
Enthalpy of solution leaving the second section – Enthalpy of recirculated stream – Enthalpy of solution coming from 1st section = Heat of solution × Formaldehyde absorbed in second section.

$$(1.6524 \times R + 2.4392)\, C_L\,(50 - 25) - 1.6524\, R\, C'_L \times (40 - 25) - 1.6524\, C''_L \times (40 - 25)$$

$$= \frac{62.75 \times 1000}{30}(2.4392 - 1.6524) \qquad \text{(I)}$$

where, $$C_L = 4.1868\left[1 - 0.005\,34 \times 37 + (0.004 + 0.000\,01 \times 37)\left(\frac{50 + 25}{2}\right)\right]$$

$$C_L = 4.0457 \text{ kJ/(kg} \cdot \text{°C)} \qquad \text{(II)}$$

$$C'_L = 4.1868\left[1 - 0.005\,34 \times 37 + (0.004 + 0.000\,01 \times 37)\left(\frac{40 + 25}{2}\right)\right]$$

$$C'_L = 3.9542 \text{ kJ/(kg} \cdot \text{°C)} \qquad \text{(III)}$$

$$C''_L = 4.1868\left[1 - 0.005\,34 \times 7 + (0.004 + 0.000\,01 \times 7)\left(\frac{40 + 25}{2}\right)\right]$$

$$C''_L = 4.5841 \text{ kJ/(kg} \cdot \text{°C)} \qquad \text{(IV)}$$

From (I), (II), (III) and (IV)

$$(1.6524 \times R + 2.4392) \times 4.0457\,(50 - 25)$$
$$= 1.6524\, R \times 3.9542\,(40 - 25)$$
$$- 1.6524 \times 4.5841\,(40 - 25)$$
$$= \frac{62.75 \times 1000}{30}(2.4392 - 1.6524)$$

$$69.119\, R + 133.085 = 1645.723$$
$$R = 21.8845$$

Recirculated stream back to the top of second section = 21.8845 × 1.6524
= 36.16 kg/s

Second intercooler will cool this stream from 50°C to 40°C.
Heat duty of second intercooler,

$$\phi_2 = \dot{m}C_L(50 - 40) = 36.16 \times C_L\,(50 - 40)$$

$$C_L = 4.1868\left[1 - 0.005\,34 \times 37 + (0.004 + 0.000\,01 \times 37)\left(\frac{50 + 40}{2}\right)\right]$$

$$C_L = 4.1829 \text{ kJ/(kg} \cdot \text{°C)}$$
$$\phi_2 = 1512.5 \text{ kW}$$

Let cooling water enters to this cooler at 32°C and leave at 37°C. Mass flow rate of cooling water required.

$$\dot{m}_w = \frac{1512.5}{4.1868 \times (37 - 32)} = 72.25 \text{ kg/s}$$

Flow rate of cooling water required = 72.25 × 3600 = 260 100 kg/h ≡ 260 m^3/h.
It is planned to use the same stream of cooling water at 37°C as a cooling medium for both, top section intercooler and inlet gas cooler on parallel basis. Let the outlet temperature of cooling water from top section intercooler is equal to 39°C.

$$\phi_1 = 161.27 \text{ kW} = \dot{m}_1 \times 4.1868 \times (39 - 37)$$
$$\dot{m}_1 = 19.26 \text{ kg/s}$$

Remaining, $\dot{m}_2 = 72.25 - 19.26 = 52.99$ kg/s will be used for the gas cooler.

Calculations for heat duty of gas cooler

Reactor exist gas is at 110°C and at 135 kPa pressure. This gas mixture is cooled from 110°C to 55°C in cooler.

Dew point temperature calculation

Mass % of water in Reactor exist gas = 6.42%

Molar flow rate of reactor exit gas

$$= \frac{40}{32} + \frac{6806.8}{32} + \frac{29626.8}{28} + \frac{2766}{18} + \frac{3341.3}{30} + \frac{386.6}{44} + \frac{27.7}{28} + \frac{4}{2} + \frac{9.9}{16} + \frac{45.5}{46}$$
$$= 1550.49 \text{ kmol/h}$$

Moisture (H_2O) content of gas mixture

$$= \frac{2766/18}{1550.49 - \left(\frac{2766}{18}\right)} = 0.110\,01 \ \frac{\text{kmol } H_2O}{\text{kmol of dry gas}}$$

At dew point

$$\frac{p_v}{p_t - p_v} = 0.110\,01$$
$$\frac{p_v}{135 - p_v} = 0.110\,01$$
$$1.110\,01\, p_v = 135 \times 0.110\,01 = 14.851\,35$$
$$p_v = 13.379 \text{ kPa}$$

At 51.6°C, vapour pressure of water is 13.379 kPa (Ref. 13). Hence, dew point temperature of gas mixture is 51.6°C. Therefore on cooling the gas mixture from 110°C to 55°C, no condensation is expected.

Heat duty of cooler,

$$\phi_3 = \int_{(273.15+55)}^{(273.15+110)} C^0_{mpi} dT$$

$$\phi_3 = \Sigma a_i (383.15 - 328.15) + \frac{\Sigma b_i (383.15^2 - 328.15^2)}{2}$$
$$+ \frac{\Sigma c_i (383.15^3 - 328.15^3)}{3} + \frac{\Sigma d_i (383.15^4 - 328.15^4)}{4}$$

Σa_i, Σb_i, Σc_i and Σd_i are taken from Ref. 13.

$$\phi_3 = 47\,588.5\,(383.15 - 328.15) - \frac{2267.1 \times 10^{-3}}{2}(383.15^2 - 328.15^2)$$

$$+ \frac{15\,211.7 \times 10^{-6}}{3}(383.15^3 - 328.15^3) - \frac{5816.3 \times 10^{-9}}{4}(383.15^4 - 328.15^4)$$

$$= 2617\,367.5 - 44\,346.20 + 106\,035.4 - 14\,476.6$$

$$= 2664\,580.1 \text{ kJ/h} \equiv 740.16 \text{ kW}$$

Total heat removed by two intercoolers and one gas cooler

$$= \phi_1 + \phi_2 + \phi_3$$

$$= 161.27 + 1512.5 + 740.16 = 2413.93 \text{ kW}$$

$$\phi_3 = \dot{m} \times 4.1868\,(t_o - 37) = 740.16$$

$$= 52.99 \times 4.1868\,(t_o - 37) = 740.16$$

$$t_o = 40.336°\text{C}$$

Calculations for number of trays:

(b) Top section

Flow rate of fresh water at top, $L_{2w} = 5532.1$ kg/h

$$L_2 = L_s = \frac{5532.1}{18} = 307.34 \text{ kmol/h}$$

$$x_2 = 0, \quad X_2 = \frac{x_2}{1 - x_2} = 0$$

Mass flow rate of exist gas at top = 39 556.3 kg/h

$$G_{2w} = 39\,556.3 \text{ kg/h}$$

Molar flow rate of gas mixture at the outlet of absorber,

$$\dot{m}_2 = \frac{6806.8}{32} + \frac{29\,626.8}{28} + \frac{2649}{18} + \frac{386.6}{44} + \frac{27.7}{28} + \frac{4}{2} + \frac{9.9}{16} + \frac{45.5}{46}$$

$$= 1431.36 \text{ kmol/h}$$

Let concentration of formaldehyde in outgoing gas mixture = 120 ppm

$$y_2 = 120 \times 10^{-6} = 0.000\,12$$

$$Y_2 = \frac{y_2}{1 - y_2} = 0.000\,12$$

Let x'_2 = Mole fraction of formaldehyde in solution entering to the second section.

y'_2 = Mole fraction of formaldehyde in gas mixture leaving the second section.

$\dot{n}_F$ = Moles of formaldehyde in liquid solution entering to second section

$$= \frac{42.525 \times 0.37 + 1.6524 \times 0.07}{30}$$

$$= 0.528\,33 \text{ kmol/s}$$

Moles of water in the same solution

$$\dot{n}_w = \frac{42.525\,(1-0.37)+1.6524\,(1-0.07)}{18}$$

$$= 1.5737 \text{ kmol/s}$$

$$x'_2 = \frac{0.528\,33}{0.528\,33+1.5737} = 0.251\,34$$

$$X'_2 = \frac{x'_2}{1-x'_2} = 0.335\,72$$

Formaldehyde absorbed in top section

$$G_S(Y'_2 - Y_2) = \frac{\left(\dfrac{5532.1}{1-0.07} - 5532.1\right)}{30}$$

$$G_S(Y'_2 - Y_2) = 13.88 \text{ kmol/h}$$

$$G_S = G_2(1-y_2) = 1431.19 \text{ kmol/h}$$

$$1431.19\,(Y'_2 - 0.000\,12) = 13.88$$

$$Y'_2 = 0.009\,818$$

$$y'_2 = \frac{Y'_2}{1+Y'_2} = 0.009\,72$$

At the base of tower

$$G_1 = 1550.49 \text{ kmol/h}$$

Mole fraction of formaldehyde in gas mixture

$$y_1 = \frac{\dfrac{3341.3}{30}}{1550.49} = 0.071\,83$$

$$Y_1 = \frac{y_1}{1-y_1} = 0.0774$$

Flow rate of liquid solution leaving the tower

$$L_{1w} = R \times 1.6524 \times 3600 + 9030.4 \quad (R = 21.8845)$$

$$L_{1w} = 139\,213 \text{ kg/h}$$

Note: From this stream 9030.4 kg/h is separated as the product stream and the remaining is recirculated back.

Average molar mass of product solution

$$M_{av} = \frac{100}{\dfrac{37}{30} + \dfrac{62.56}{18} + \dfrac{0.44}{32}} = 21.175 \text{ kg/mol}$$

$$L_1 = \frac{139\,213}{21.175} = 6574.4 \text{ kmol/h}$$

Mole fraction of formaldehyde in this solution.

$$x_1 = \frac{\left(\dfrac{139\,213 \times 0.37}{30}\right)}{6574.4} = 0.261\,16$$

$$X_1 = \frac{x_1}{1 - x_1} = 0.353\,47$$

Mass % of formaldehyde at the top of second section, w_2

$$= \frac{x_2' \times 30}{x_2' \times 30 + (1 - x_2') \times 18} \times 100$$

$$= \frac{0.25134 \times 30 \times 100}{0.251\,34 \times 30 + (1 - 0.251\,34) \times 18}$$

$$w_2 = 35.878\%$$

Average mass % of formaldehyde in second section $= \dfrac{35.878 + 37}{2} = 36.44\%$

Average temperature of absorption $= 45°C$

Partial pressure of formaldehyde in atmospheric air over formaldehyde solution is given by Lacy's empirical equation (Ref. 15).

$$\text{Log}\ \bar{p}_v = 9.942 - 0.953\ (0.488)^{w/10} - 2905/T$$

At $T = 45 + 273 = 318$ K and $w = 36.44\%$

$$\bar{p}_v = 5.46 \text{ Torr}$$

At atmospheric pressure,

equilibrium constant, $m = \dfrac{5.46/760}{(36.44/30)/(36.44/30 + (100 - 36.44)/18)}$

$= 0.028$ at atmospheric pressure

Operating pressure in the given absorber is close to atmospheric, hence, the average value of m for second section is 0.028.

$$\frac{Y}{1+Y} = 0.028\ \frac{X}{1+X}$$

Y	0	0.005	0.01	0.015	0.02	0.025	0.028
X	0	0.216	0.547	1.1177	2.336	6.76	∞

Average mass % of formaldehyde for top section, $w = \dfrac{7}{2} = 3.5\%$

Average temperature of absorption $= \dfrac{30 + 60}{2} = 45°C$

$$\log \bar{p}_v = 9.942 - 0.953\ (0.488)^{\frac{3.5}{10}} - 2905/T$$

$$\bar{p}_v = 1.1625 \text{ Torr} \quad [\text{for } T = (273 + 45) \text{ K}]$$

$$m = \frac{(1.1625/760)}{(3.5/30)/([(3.5/30) + (96.5/18)]} = 0.071\,82$$

$$\frac{Y}{1+Y} = 0.071\,82\,\frac{X}{1+X}$$

Equilibrium data for top section:

Y	0	0.001	0.002	0.003	0.004	0.006	0.008	0.01
X	0	0.014	0.0286	0.0434	0.0587	0.09	0.1242	0.16

For first section operating line starts from (0, 0.000 12) to (X, 0.009 817).

where $X = \dfrac{x}{1-x}$ and $x = \dfrac{\frac{7}{30}}{\frac{7}{30}+\frac{93}{18}} = 0.0432$

$X = 0.045\,16$

Fig. 9.20 *Theoretical Stage Calculations for Top Section of Formaldehyde Absorber*

Number of theoretical stages required in top section is $n_1 = 3.73$
For second section, operating line starts from (0.335 72, 0.009 818) and terminates at (0.353 47, 0.0774).
Number of theoretical stages required in bottom section is $n_2 < 1$ but assumed as 1.
Total number of theoretical stages required for the desired separation, $n_T = 4.7304$

Fig. 9.21 *Theoretical Stage Calculations for Bottom Section of Formaldehyde Absorber*

Refer to Fig. 4.8 for two sections of the tray tower.

Tower diameter calculations

At the base of second section

$$G_{1w} = 43\,054.6 \text{ kg/h}, \quad L_{1w} = 139\,213 \text{ kg/h}$$

Average molar mass of gas mixture that enters the tower,

$$M_{\text{av}} = \frac{43\,054.6 \text{ kg/h}}{1550.49 \text{ kmol/h}} = 27.768 \text{ kg/kmol}$$

Liquid vapour flow factor F_{LV} for sieve tray tower

$$F_{LV} = \frac{L_w}{G_w}\sqrt{\frac{\rho_v}{\rho_L}} \tag{8.66}$$

$$L_w = L_{1w} = 139\ 213 \text{ kg/h}$$
$$G_w = G_{1w} = 43\ 054.6 \text{ kg/h}$$
$$\rho_V = \frac{p\,M_{\text{av}}}{RT} = \frac{135 \times 27.768}{8.3145 \times (273 + 55)} = 1.375 \text{ kg/m}^3$$

(Density of water at 50°C, $\rho_w = 988$ kg/m^3, $\rho_{Fo} = 815$ kg/m^3, $\rho_{me} = 780$ kg/m^3)

$$\rho_L = \frac{1}{\Sigma \dfrac{w_i}{\rho_{Li}}} = \frac{1}{\dfrac{0.37}{815} + \dfrac{0.6256}{988} + \dfrac{0.0004}{780}}$$
$$= 919.37 \text{ kg/m}^3$$

$$\text{Volumetric flow rate of liquid} = \frac{139\ 213/3600}{919.37}$$
$$= 0.042 \text{ m}^3\text{/s}$$

Select two pass sieve tray (Ref. Table 8.33) as liquid flow rate is high.

$$F_{LV} = \frac{139\ 213/2}{43\ 054.6}\sqrt{\frac{1.375}{919.37}} = 0.0625$$

For tray spacing, $S = 0.45$ m
From Fig. 8.20, $C_f = 0.079$
Flooding velocity

$$v_F = C_f \left(\frac{\sigma_L}{0.02}\right)^{0.2} \left(\frac{\rho_L - \rho_V}{\rho_V}\right)^{0.5} \tag{8.65}$$

σ_L = Surface tension of liquid, N/m

$$\sigma_{\text{Fo}}^{1/4} = [\mathcal{P}_i]\ (\rho'_L - \rho'_V)\,; \quad \rho' \text{ is in kmol/m}^3$$

$[\mathcal{P}_i]$ for HCHO = 15.5 + 66 = 81.5
(Table 3–343 of Ref. 3)

$$\rho_V = \frac{pM}{RT} = \frac{135 \times 30}{8.3145 \times (273 + 50)} = 1.5 \text{ kg/m}^3$$

$$\sigma_{\text{Fo}}^{1/4} = 81.5\left(\frac{(815 - 1.5) \times 10^{-3}}{30}\right) = 2.21$$

$$\sigma_{\text{Fo}} = 23.85\ \frac{\text{dyn}}{\text{cm}} = 23.85 \times 10^{-3} \text{ N/m}$$
$$\sigma_L^{1/4} = \Sigma\, \sigma_i^{1/4}\, x_i$$
$$\sigma_L^{1/4} = 0.26 \times (23.85 \times 10^{-3})^{1/4} + 0.74 \times (70 \times 10^{-3})^{1/4} = 54.3 \times 10^{-3} \text{ N/m}$$
$$v_F = 0.079\left(\frac{0.0543}{0.02}\right)^{0.2} \left(\frac{919.37 - 1.375}{1.375}\right)^{0.5}$$
$$= 2.5257 \text{ m/s}$$

Let actual velocity of gas-vapour mixture through tower

$$v_1 = 0.66\ v_F = 1.667 \text{ m/s}$$

Volumetric flow rate at bottom (of gas mixture)

$$q_{V1} = \frac{G_{1w}}{\rho_V} = \frac{43\,054.6/3600}{1.375} = 8.7 \text{ m}^3/\text{s}$$

Net area of tray required at bottom

$$A_n = \frac{q_{v1}}{v} = \frac{8.7}{1.667} = 5.219 \text{ m}^2$$

Let downcomer area, $A_d = 0.1\ A_c$ per downcomer

where, A_c = Inside cross-sectional area of tower

$$A_n = A_a - A_d = A_c - 0.2\ A_c = 0.8\ A_a$$

$$0.8\ A_c = 5.219 \text{ m}^2$$

$$A_c = 6.524 \text{ m}^2 = \left(\frac{\pi}{4}\right) D_i^2$$

$$D_i = 2.88 \text{ m}$$

Tower diameter required at the base of top section:

At the base of top section.

$$L_w = 1.6524 \text{ kg/s}$$

Gas flow rate at the base of first section

$$G_w = \text{Gas flow rate at top of first section} + L_w - \text{Solvent flow rate at top}$$

$$G_w = 39\,556.3 + 1.6524 \times 3600 - 5532.1$$

$$G_w = 39\,972.84 \text{ kg/h} \equiv 11.1036 \text{ kg/s}$$

Molar flow rate of gas mixture at the entrance of first section,

$$G = G_s\,(1 + Y'_2) = 1431.36\,(1 + 0.009\,818)$$

$$G = 1445.41 \text{ kmol/h}$$

$$M_{av} = \frac{39\,972.84}{1445.41} = 27.655 \text{ kg/kmol}$$

$$\rho_V = \frac{p\,M_{\text{av}}}{RT} \cong \frac{135 \times 27.655}{8.3145 \times (60 + 273)} = 1.35 \text{ kg/m}^3$$

$$\rho_L = \frac{1}{\Sigma \dfrac{w_i}{\rho_{Li}}} = \frac{1}{\dfrac{0.07}{815} + \dfrac{0.93}{983.2}} = 969.2 \text{ kg/m}^3$$

(Density of water at 60°C = 983.2 kg/m^3)

$$F_{LV} = \frac{L_w}{G_w}\sqrt{\frac{\rho_v}{\rho_L}} = \frac{1.6524}{11.1036}\sqrt{\frac{1.35}{969.2}} = 5.554 \times 10^{-3}$$

for tray spacing $S = 0.45$ m

from Fig. 8.20, $C_f = 0.08$

Surface tension of 7% formaldehyde solution.

$$\sigma_L^{1/4} = \Sigma \sigma_i^{1/4} x_i$$

$$x = \frac{7/30}{7/30 + 93/18} = 0.0432$$

$$\sigma_L^{1/4} = 0.0432 \times (23.843 \times 10^{-3})^{1/4} + (1 - 0.0432) \times (70 \times 10^{-3})^{1/4}$$

$$\sigma_L = 0.068 \text{ N/m}$$

Flooding Velocity

$$v_F = C_F \left(\frac{\sigma_L}{0.02}\right)^{0.2} \left(\frac{\rho_L - \rho_V}{\rho_V}\right)^{0.5}$$

$$= 0.08\left(\frac{0.068}{0.02}\right)^{0.2} \left(\frac{969.2 - 1.35}{1.35}\right)^{0.5} = 2.736 \text{ m/s}$$

For 66% flooding,

$$v = 0.66 \times 2.736 = 1.8 \text{ m/s}$$

Volumetric flow rate of gas mixture at the base of first section.

$$q_V = \frac{11.1036}{1.35} = 8.225 \text{ m}^3\text{/s}$$

Net area of tray required,

$$A_n = \frac{q_v}{v} = \frac{8.225}{1.8} = 4.57 \text{ m}^2$$

Let downcomer area = 12% of column area (A_c)

$$A_c = \frac{4.57}{0.88} = 5.19 \text{ m}^2 = \frac{\pi}{4} D_i^2$$

$$D_i = \sqrt{\frac{4 \times 5.19}{\pi}} = 2.57 \text{ m}$$

Checking of weeping for bottom section:

The minimum design vapour velocity through holes to avoid weeping is given by following equation:

$$u_h = \frac{K - 0.9(25.4 - d_h)}{\sqrt{\rho_G}} \tag{9.70}$$

u_h = Minimum gas velocity through holes, m/s
K = Constant can be obtained from Fig. 9.19
d_h = hole diameter, mm

Let $d_h = 5$ mm, $\rho_G = 1.375$ kg/m^3

Height of weir, $h_w = 50$ mm

Height of liquid crest over the weir

$$h_{ow} = 750\left[\frac{L_w}{\rho_L l_w}\right]^{\frac{2}{3}} \tag{9.69}$$

L_w = Liquid flow rate, kg/s
L_w = 139 213 kg/h = 38.67 kg/s
ρ_L = 919.37 kg/m^3
l_w = Length of weir, m

For $\frac{A_d}{A_c} = 0.1 \quad \frac{l_w}{D_i} = 0.727$ (Ref. Table 8.34)

$$l_w = 0.727 \times 2.88 \text{ m} = 2.0938 \text{ m}$$

$$h_{ow} = 750\left[\frac{(38.67/2)}{919.37 \times 2.0938}\right]^{2/3} = 34.9 \text{ mm}$$

$$h_L = h_w + h_{ow} = 84.9 \text{ mm}, \quad K = 30.8$$

$$u_h = \frac{30.8 - 0.9(25.4 - 5)}{\sqrt{1.375}}$$

$$= 10.61 \text{ m/s}$$

Maximum hole area required

$$A_{hm} = \frac{8.7}{10.61} = 0.82 \text{ m}^2$$

$$\frac{A_{hm}}{A_c} = \frac{0.82}{6.524} = 0.1257$$

Let $A_h = 0.1\, A_{c1} = 0.391\,44 \text{ m}^2$

Actual velocity, of gas through holes, $v_h = 22.22$ m/s > 10.61 m/s

Hence, weeping will not take place.

A check by Eq. (8.72) would confirm no weeping.

$$Fr_L = \left[\frac{1.375\,(22.22)^2}{(919.37 \times 9.81 \times 0.0849)}\right]^{0.5}$$

$$= 0.9416 > 0.5$$

Checking of weeping for top section:

For the trays of top section, let hole diameter, $d_h = 5$ mm

Height of weir, $h_w = 50$ mm

Height of liquid crest over the weir

$$h_{ow} = 750\left[\frac{L_w}{\rho_L l_w}\right]^{2/3} \tag{8.69}$$

L_w = Liquid flow rate = 1.6524 kg/s

$\rho_L = 969.2 \text{ kg/m}^3$

For $\frac{A_d}{A_c} = 0.12 \Rightarrow \frac{l_w}{D_i} = 0.77$

Length of weir,

$$l_w = 0.77 \times 2.57 = 1.979 \text{ m}$$

$$h_{ow} = 750\left[\frac{1.6524}{969.2 \times 1.979}\right]^{2/3}$$

$$h_{ow} = 6.79 \text{ mm}$$

$$h_L = h_w + h_{ow} = 56.79 \text{ mm}, \quad K = 30.2$$

Minimum vapour velocity required through holes to avoid weeping

$$u_h = \frac{K - 0.9(25.4 - d_h)}{\sqrt{\rho_G}} \tag{8.71}$$

$$= \frac{30.2 - 0.9(25.4 - 5)}{\sqrt{1.35}}$$

$$= 10.19 \text{ m/s}$$

Maximum hole area which can be provided,

$$A_{hm} = \frac{8.225}{10.19} = 0.8072 \text{ m}^2$$

$$\frac{A_{hm}}{A_c} = \frac{0.8072 \text{ m}^2}{\frac{\pi}{4}(2.57)^2} = 0.1556$$

Let $\dfrac{A_h}{A_c} = 0.1$

$$A_h = 0.1A_c = 0.518\,75 \text{ m}^2$$

Actual velocity of gas through holes,

$$v_h = \frac{8.225}{0.518\,75} = 15.855 \text{ m/s} > 10.10 \text{ m/s}$$

Hence, weeping will not take place.

Based on Eq. (8.72),

$$Fr_L = \left[\frac{1.375\,(15.855)^2}{(969.2 \times 9.81 \times 0.056\,79)}\right]^{0.5}$$

$$= 0.8001 > 0.5$$

In this tray tower both sections are equally sensitive to weeping.

Checking of downcomer flooding for the bottom section:

Clear liquid back-up in downcomer,

$$h_b = h_w + h_{ow} + h_t + h_{dc} \tag{8.83}$$

where, h_t = Total tray pressure drop, mm LC

h_{dc} = head loss in downcomer, mm LC

$$h_t = h_d + (h_w + h_{ow}) + h_r \tag{8.75}$$

where, h_d = Dry tray pressure drop, mm LC

h_r = Residual pressure drop, mm LC

$$h_d = 51\left(\frac{v_h}{C_o}\right)^2 \frac{\rho_G}{\rho_L} \tag{8.76}$$

where, C_o = Orifice coefficient can be determined by using Fig. 8.24

Let plate thickness = 5 mm,

$$\frac{\text{Plate thickness}}{\text{hole diameter}} = \frac{5}{5} = 1$$

$$\frac{A_h}{A_p} \cong \frac{A_h}{A_a} = \frac{A_h}{A_c - 4A_d} = \frac{A_h}{A_c - 4 \times 0.1\, A_c}$$

$$\frac{A_n}{A_p} = 0.1$$

$$C_o = 0.84 \quad \text{(From Fig. 8.24)}$$

$$h_d = 51 \times \left(\frac{22.22}{0.84}\right)^2 \times \frac{1.375}{919.37} = 53.37 \text{ mm LC}$$

Residual pressure drop,

$$h_r = \frac{12.5 \times 10^3}{\rho_L} = \frac{12.5 \times 10^3}{919.37} = 13.6 \text{ mm LC}$$

$$h_t = 53.37 + (50 + 34.9) + 13.6 = 151.87 \text{ mm LC}$$
$$= 50 + 34.9 + 151.87 + h_{dc}$$

head loss in downcomer,

$$h_{dc} = 166\left[\frac{L_{wd}}{\rho_L A_m}\right]^2 \tag{8.80}$$

L_{wd} = Liquid flow rate in downcomer, kg/s

$A_m = A_d$ or A_{ap} whichever is smaller

$A_{ap} = h_{ap} l_w$

h_{ap} = Clearance between bottom edge of downcomer apron and tray

$h_{ap} = h_w - (5 \text{ to } 10 \text{ mm})$

$$h_{dc} = 166\left(\frac{(38.67/2)}{919.37 \times A_m}\right)^2$$

$A_m = A_d\ (= 0.6524 \text{ m}^2)$ or $A_{ap} = 0.04 \times 2.0938 = 0.083\,75 \text{ m}^2$

$A_m = 0.083\,75 \text{ m}^2$

$$h_{dc} = 166\left(\frac{(38.67/2)}{919.37 \times 0.089\,38}\right)^2$$

$h_{dc} = 10.46$ mm

$h_b = 50 + 34.9 + 151.87 + 10.46$

$= 247.23$ mm

$S + h_w = 450 \text{ mm} + 50 \text{ mm} = 500 \text{ mm}$

$h_b < 0.5\,(S + h_w)$

Hence, downcomer flooding will not take place in bottom section.

Downcomer residence time,

$$\theta_r = \frac{A_d h_b \rho_L}{L_{wd}}$$

$$= \frac{(2 \times 0.1 \times 6.524) \times 0.247}{38.67} \times 919.37$$

$$= 7.66 \text{ s} > 3 \text{ s (satisfactory)}$$

Checking of downcomer flooding in top section:
Dry plate pressure drop,

$$h_d = 51\left(\frac{v_h}{C_o}\right)^2 \frac{\rho_G}{\rho_L} \tag{8.76}$$

$$\frac{\text{Plate thickness}}{\text{Hole diameter}} = \frac{5}{5} = 1$$

$$\frac{A_h}{A_p} = \frac{A_h}{0.76\, A_c} = \frac{0.1}{0.76} = 0.1315$$

$$C_o = 0.87 \quad \text{(From Fig. 8.24)}$$

$$h_d = 51\left[\frac{15.855}{0.87}\right]^2 \frac{1.35}{969.2} = 23.6 \text{ mm}$$

Residual pressure drop,

$$h_r = \frac{12.5 \times 10^3}{\rho_L} = \frac{12.5 \times 10^3}{969.2} = 12.9 \text{ mm}$$

$$h_t = 23.6 + 50 + 6.79 + 12.9 = 93.3 \text{ mm}$$

$$l_w = 1.979 \text{ m}, \qquad A_{ap} = 0.045 \times 1.979 = 0.089 \text{ m}^2$$

Head loss in downcomer,

$$h_{dc} = 166\left[\frac{L_{wd}}{\rho_L A_m}\right]^2$$

$$= 166\left[\frac{1.6524}{969.2 \times 0.089}\right]^2$$

$$h_{dc} = 0.06 \text{ mm}$$

$$h_b = 50 + 6.79 + 93.35 + 0.06 \text{ mm} = 150.2 \text{ mm}$$

$$S + h_w = 450 + 50 = 500 \text{ mm}$$

$$h_b < 0.5\,(S + h_w)$$

Hence, downcomer flooding will not take place.

Downcomer residence time

$$\theta_r = \frac{A_d h_b \rho_L}{L_{wd}} = \frac{0.12\left(\frac{\pi}{4}\right)2.57^2 \times 0.15 \times 969.2}{1.6524}$$

$$= 54.77 \text{ s (satisfactory)}$$

Tray efficiency

Overall tray efficiency based on O'Connell correlation for the absorption in tray tower is given in Fig. 14.7, *Perry's Chemical Engineers' Handbook*, 7th Ed., 1998.

The O'Connell parameter for top section

$$\frac{\rho_L}{K M_L \mu_L} = \frac{969.2}{K M_L \mu_L}$$

where, K = Equilibrium constant

M_L = Average molar mass of liquid solution

μ_L = Viscosity of liquid solution

For top section

$$K = 0.071\,82$$

$$M_L = \frac{0.0432}{2} \times 30 + \left(1 - \frac{0.0432}{2}\right) \times 18 = 18.26$$

$$\mu = \text{Viscosity of liquid solution} = 10^{-3} \text{ Pa} \cdot \text{s}$$

$$\frac{\rho_L}{K M \mu_L} = \frac{969.2}{0.071\,82 \times 18.26 \times 10^{-3}} = 739\,038.8 \text{ kmol/(m}^3 \cdot \text{Pa} \cdot \text{s)}$$

$$= \frac{739\,038.8}{1.6 \times 10^4} = 46.2 \text{ lb mole /(ft}^3 \cdot \text{cP)}$$

Plate efficiency, η is more than 60% (from Fig. 14.7, *Perry's Chemical Engineers Handbook*, 7th Ed.)

For bottom section

$$M_L = 0.2563 \times 30 + (1 - 0.2563) \times 18 = 21.0756$$

$$\frac{\rho_L}{K M_L \mu_L} = \frac{919.37}{0.028 \times 21.0756 \times 1.2 \times 10^{-3}} = 1298\,288.2 \text{ kmol/(m}^3 \cdot \text{Pa} \cdot \text{s)}$$

$$= 81.143 \quad \text{lb mole/(ft}^3 \cdot \text{cP)}$$

Plate efficiency, η is more than 60%.

(From Fig. 14.7, *Perry's Chemical Engineers Handbook*, 7th Ed.)

However, plate efficiencies of commercial hydrocarbon absorbers are ranging from 15 to 55%.

Let tray efficiencies be 0.34 for top section and 0.33 for bottom section.

Hence, actual number of sieve trays in top section will be 11 and in bottom section will be 3. Total number of sieve trays required is 14.

Resulting data

Inside diameter of tower = 2570 mm

No. of trays in top section = 11

No. of trays in bottom section = 3

Flow rate of liquid (37% formaldehyde) solution recirculated and fed to the top of bottom section = 153 239 kg/h

Details of the sieve trays in both sections are shown in Fig. 9.22 and Fig. 9.23.

All Dimensions are in mm (NTS).

Fig. 9.22 *Single Pass Cross-flow Sieve Tray for Top Section*

All Dimensions are in mm (NTS).

Fig. 9.23 *Double Pass Cross-flow Sieve Tray for Bottom Section*

Figure 4.9 is the P&I diagram of the entire formaldehyde manufacturing. The diagram shows four coolers in which cooling water is required. There are two alternative cooling water circuits considered for design in Fig. 9.24.

Fig. 9.24 *Alternative Cooling Water Circuits*

Alternative I: Solution Cooler (HE-3) has the highest cooling load of 1512.5 kW. Cooling water at the rate of 260.0 m^3/h is supplied at 32°C to this cooler. Cooling water leaves the HE-3 at 37°C. Cooling water stream at 37°C is now split in three streams. One stream will go to Gas Cooler (HE-2) to cater cooling load of 161.27 kW. Its flow rate can be calculated to be 69.33 m^3/h for a temperature rise of 2°C. Balance stream (190.76 m^3/h) will flow through Solution Cooler (HE-4) to cater cooling load of 740.16 kW. A small stream is connected to Sample Cooler (SC-1) which is used only when sample is drawn. Thus overall rise of 7.98°C in cooling water temperature will be realised.

Alternative II: In this alternative, it is considered that all coolers will be supplied cooling water at 32°C. Increase of 5°C is considered in each cooler. This will mean total cooling water flow of 415.12 m^3/h.

It can be seen that total cooling water flow defers in both the alternatives although total cooling load is same (2413.93 kW). While procuring cooling tower for the plant, specifications will have different cooling water flow rates with different temperature ranges. Although in both alternatives, it will be called the tower to cater 2413.93 kW (686.85 TR).

In cooling tower design wet bulb temperature of ambient air is an important consideration. Cold water temperature is limited by approach to the wet bulb temperature. Cooling tower with a range of 7.98°C and flow rate 260.0 m^3/h will require lesser floor space than the cooling tower with a range of 5°C and flow rate of 415.12 m^3/h. Hence, cost of cooling tower with a cooling range of 7.98°C will be lower. Also, cost of pumping cooling water will be lower.

Above discussion should not mislead to assume that coolers should always be designed for series cooling as heat transfer area requirement of the coolers also will have to be checked vis-à-vis cooling tower design. In general, a cooling range of 8 to 10°C and approach of 5°C to design wet bulb temperature of ambient air can be considered optimal for the design of cooling water system.

EXERCISES

9.1 Design a scrubber for absorbing dimethyl ether (DME) vapour from nitrogen-dimethyl ether vapour mixture. The temperature in the scrubber is 20°C and scrubbing is approximated as isothermal. Operating pressure of scrubber is atmospheric. A mixture of nitrogen—DME vapour contains 10% DME (by volume) and its flow rate is 60 kmol/h. The scrubber is required to absorb 98% of the DME. Molar mass of DME is 46 kg/kmol. Cold water is used as solvent.

9.2 Gas mixture at the rate of 100 kmol/h, obtained from chlorination process, contains 20% hydrogen chloride gas and 80% chlorine (by mole). From the mixture, hydrogen chloride gas is to be separated by absorption in water. For the absorption of HCl, FRP (fibre glass reinforced plastic) packed tower is used. Product hydrochloric acid solution, leaving the packed tower, must contain 30% HCl (by mass). Design the FRP packed tower.

9.3 Falling film absorber is used in ammonia-water absorption refrigeration plant[12]. Refrigeration duty of this plant is 200 kW (56.87 TR). Flow rate of ammonia vapour, entering to falling film absorber is 0.1935 kg/s. It is absorbed by weak

aqueous solution of ammonia having 30% strength (by mass). Product strong solution of ammonia leaving from the bottom of absorber is having 36.5% strength. Average value of heat of absorption of ammonia for the required concentration change is 2046.6 kJ/kg. Design the falling film absorber. Temperature of ammonia gas at inlet to absorber is –10°C.

Operating pressure = 280 kPa a

Assume that Eq. (9.46), Eq. (9.47) and Eq. (9.48) are also valid for the absorption of ammonia in water (in falling film absorption) to calculate mass transfer coefficient and heat transfer coefficient, respectively.

9.4 Venturi scrubber is planned for absorbing NO_x gases from the exist gas stream of nitric acid plant by using 10% NaOH solution (by mass). Exist gas mixture, leaving the absorber of nitric acid plant, is having the following composition (Ref. Table 2.9 of Chapter 2).

Component	kmol/h
N_2	1275.097
O_2	61.537
Ar	16.330
NO	2.720
N_2O	2.190
H_2O	169.191
Total	1527.065

Discharge pressure of gas from venturi = atmospheric

Solvent to gas ratio = 1.5 L/m^3

Temperature of gas mixture, entering the venturi scrubber = 50°C

Determine: (a) throat diameter of venturi scrubber,
(b) % removal of NO_x gases,
(c) consumption of NaOH, and
(d) pressure drop in venturi scrubber.

9.5 Design a sour-water stripper based on following information[16]:

Feed liquid mixture flow rate = 50 000 kg/h

% of H_2S in feed = 0.73% (by mass)

% of NH_3 in feed = 0.556% (by mass)

% of H_2O in feed = 98.714% (by mass)

Feed condition = Saturated liquid at operating condition

Stripping steam rate = 5100 kg/h

Operating pressure at bottom = 2.4 atm a

Concentration of H_2S and NH_3 in bottom stream = 5 ppm and 20 ppm, respectively

Operating temperature at bottom = 126°C

Steam is generated by using reboiler at bottom. Stripped overheads are to be condensed in overhead condenser. Condensate and tail gas from this condenser are separated in a reflux drum. Liquid phase from drum is recycled back to top tray. Tail gas will be sent to sulphur recovery plant.

9.6 From a 10 mole % NH_3 in NH_3-air mixture, nearly 100% of the ammonia is to be removed by countercurrent scrubbing at 1 atm and 20°C. Consider concentration of ammonia from scrubber to be 20 ppm. Entering gas rate is 3600 kg/(h · m^2). Calculate the following for both solvents, water and 0.1 N H_2SO_4 solution.

For NH_3-H_2O system, *Henery's* law constant at 30°C, m = 0.85 (Ref. 1).

(a) Minimum amount of solvent
(b) Actual amount of solvent
(c) The number of overall gas transfer units, N_{OG}

(d) H_{OG} based on $K_G a$[17].

$K_G a = 78.1$ kmol/(h · m^3 · atm) for 0.1 N H_2SO_4 solution

(e) Column height

9.7 Enrichment of a lean natural gas stream is carried out by absorbing CO_2 and H_2S from a gas stream containing 45% CO_2, 3% H_2S and 52% CH_4 (by mole). A packed tower with 25 mm polypropylene Pall rings is considered for absorption of acidic gases in 10% (by mass) aqueous NaOH solution. Feed gas is introduced at a rate of 900 Nm^3/h at 1.2 bar a and 40°C. Fresh aqueous solution is introduced at 35°C. Desired purity of gas stream is 100 mg/L acid.

Design packed tower system with liquid circulation requirement. Calculate heat duty of heat exchanger(s), installed in the recirculation system.

Stoichiometry:

$2\ NaOH(aq) + CO_2(g) = Na_2CO_3(ag) + H_2O(l)$ $\Delta H^\circ_{r1} = -104.67$ kJ/mol

$2\ NaOH(aq) + H_2S(g) = Na_2S(aq) + 2\ H_2O(l)$ $\Delta H^\circ_{r2} = -52.73$ kJ/mol

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10 Process Design of Reactors

10.1 INTRODUCTION

Reactor is the most important equipment of any chemical plant. Minor change or improvement in the reactor design or in its performance greatly affects the process and mechanical design of other equipments. Sometimes it may even change the entire flow sheet. For example, in the process of manufacturing of monochloroacetic acid, reaction is carried out between acetic acid and chlorine. Conversion of acetic acid is about 90%. Product mass contains a mixture of monochloroacetic acid, dichloroacetic acid and unconverted acetic acid from which monochloroacetic acid is separated by crystallization. But, if in the same reactor, conversion of acetic acid is restricted to 50%, then dichloroacetic acid is not formed in noticeable quantity and the product mixture contains mainly acetic acid and monochloroacetic acid from which monochloroacetic acid can be separated by vacuum distillation. Thus, change in conversion of reactant in the reactor changes the entire flow sheet. Hence, for any chemical process, development in the reaction kinetics or reactor design is important in research. Major area of the research is the development of a catalyst for the reaction. By developing a new catalyst, entirely a new process route can be developed. Final goal of the research for all the reactions and reactors is to carry out the reaction at atmospheric pressure and at ambient temperature with sufficiently high rate of reaction, 100% conversion and 100% yield. At the same time, heating or cooling medium requirement to run the reaction should be minimum. To achieve this goal one can take inspiration from natural reactor like human body. In the human body, thousands of reactions are running at about 37°C temperature and at a pressure close to one atmosphere. For the process design of the reactor, knowledge of various subjects of chemical engineering is required like of chemical reaction engineering, thermodynamics, stoichiometry, heat transfer operation, mass transfer operation, fluid flow operation, chemistry, etc. Process design of various types of reactors are outlined in the subsequent sections.

10.2 DIFFERENT TYPES OF REACTORS

Several types of reactors are in use in industry. Basically they fall into two categories; batch and continuous. In each category, different designs are available. In fact, development of a reactor can be an innovative idea and a process engineer can use his skills for such a development.

In Fig. 10.1, different types of reactors are represented schematically. Designs date back to a simple batch reactor to modern continuous catalytic reactors such as ammonia converter. Designs also vary with the type of reactions; such as homogenous, heterogeneous, run-away reactions, etc.

In many cases, a number of reactors are used in series. Continuous stirred tank reactors [Fig. 10.1(c)] can be cited as an example. In some cases, number of parallel reactors are used. For example, in reforming of hydrocarbons (such as naphtha or natural gas), several hundred tubular reactors are used in parallel in which catalyst is packed in different shapes and sizes. Selection is made on the basis of degree of conversion, ease of heat removal/addition, safety and other considerations.

10.3 BATCH REACTORS[1, 2, 3]

In a batch reactor, all the reactants are initially charged into the reactor. In most cases, then they are allowed to react for certain period of time. The resultant mixture is then discharged. In any batch reactor, composition of reaction mass changes with time, hence, it is called an *unsteady state reactor*.

In an ideal batch reactor at any instant, composition throughout the reactor is uniform while the same is not true for a non-ideal batch reactor. Size of batch reactor does not directly depend on the reaction time but it depends on volume of material charged to the reactor. For small capacity plant having production capacity less than 5 tonnes per day, batch process with a batch reactor is more economical than continuous process (continuous reactor).

In a batch reactor, variety of agitators are used. Each one has its specific advantages and limitations. Figure 10.2 gives different types of mixing equipment, used in chemical industry. Selection of an agitator plays an important role in achieving desired conversion in the reactors. Selection criteria for the different types of agitators are discussed in Sec. 10.6. In majority of reactors, turbine-type agitators are used. While for simple mixing, flat blade turbine agitator is used, pitched blade turbine agitator is preferred for a slurry. For viscous liquids (such as manufacture of amino/alkyl resins), anchor/paddle type agitators of different designs are used. For homogenising applications, such as milk powder dissolution in water, degumming of edible oils (with phosphoric acid), etc., shear mixer is an ideal choice but is a costly equipment. For gas-liquid reactions gas induction-type agitator [Fig. 10.1(k)] and jet loop reactor [Fig. 10.1(i) or (j)] are preferred. Design of agitation equipment is discussed at length in literature. Reference (3) gives a good treatise on the subject.

10.3.1 Industrial Examples of Batch Reactor

1. Synthesis of dyes, e.g., sulphonation of naphthalene gives β-sulphonate
2. Benzene sulphonic acid from benzene with oleum
3. Nitrobenzene from benzene with mixed acid
4. Synthesis of polyurethane from toluene diisocyanate and polyglycol
5. Synthesis of cypermathrin
6. Batch fermenter, e.g., fermentation of molasses to ethanol
7. Manufacture of active pharmaceutical ingredients (API)
8. Manufacture of amino and alkyd resins, etc.

(a) Batch Reactor

(b) Typical Batch Loop Reactor

(c) Battery of Stirred Tank Reactors

(d) Longitudinal Tubular Reactor

(e) Heterogeneous Continuous Reactor

Fig. 10.1 (*Contd.*)

(f) Baffle Tank Reactor

(g) Longitudinal Catalytic Fixed Bed Reactor

(h) Fluid-Bed Catalytic Reactor

(i) Jet Reactor

(j) Monolith Loop Reactor

Fig. 10.1 (*Contd.*)

(k) Internal Monolith Loop Reactor

Fig. 10.1 *Different Types of Reactors*

Fig. 10.2 (*Contd.*)

Fig. 10.2 *Different Types of Agitators*

10.3.2 Advantages of Batch Reactors over Continuous Flow Reactors

1. For small scale production, batch process with a batch reactor is more economical than continuous process with a continuous reactor.
2. Batch reactors provide more flexibility. For example, it is readily possible to increase the reaction time in the batch reactor while the same is difficult in continuous flow reactor without sacrificing the production rate.
3. Some process steps (upstream or downstream processing) of the reaction can also be performed in the reactor.
4. Batch reactors require less and relatively simple or cheaper instruments while continuous flow reactors require more sophisticated and costlier instruments.

10.3.3 Disadvantages of Batch Reactors over Continuous Flow Reactors

1. High manpower requirement.
2. Time is wasted in between two batches.
3. Temperature control is poor, especially for highly endothermic or exothermic reaction. While starting reaction in a batch reactor, rate of reaction is high and hence, heat duty is substantially high.
   Some of these aspects were discussed in Chapter 3.
4. Quality of the product may vary from a batch to batch.

Performance equation of ideal batch reactor is $\theta = N_{Ao} \int_{o}^{X_A} \frac{dX_A}{(-r_A)V}$ (10.1)

where, N_{Ao} = Initial amount of limiting reactant, kmol
θ = Time required to achieve the conversion X_A, s
X_A = Fractional conversion of limiting reactant A
$-r_A$ = Rate of reaction kmol/(m^3 · s)
V = Working volume of reactor, m^3

For the constant volume reaction system,

$$\theta = \frac{N_{Ao}}{V} \int_{o}^{X_A} \frac{dX_A}{-r_A} = C_{Ao} \int_{o}^{X_A} \frac{dX_A}{-r_A} \quad (10.2)$$

where, C_{Ao} = Initial concentration of limiting reactant A, kmol/m^3

Example 10.1 Butyl acetate formation is carried out in a batch reactor at 90°C with sulphuric acid as a homogeneous catalyst. The feed contained 4.97 moles of *n*-butanol per mole of acetic acid and the catalyst concentration is 0.032% by mass as H_2SO_4.

Rate equation for this reaction is $-r_A = kC_A^2$.

where, C_A = Concentration of acetic acid in mol/cm^3
k = 17.4 cm^3/(mol · min) (Ref. 4)

Density of the reaction mixture at 90°C can be assumed constant and equal to 0.75 g/cm^3.

(a) Calculate the time required to obtain conversion of 50%.
(b) Determine the size of reactor and the mass of reactants that must be charged to the reactor in order to produce ester at the average rate of 100 kg/h. Conversion is 50%. Reactor will be shut down for 30 min between batches for removal of product, cleaning and start-up. Assume that batch reactor is ideal. Also decide height and diameter of the reactor.
(c) Determine the heat duty required and heat transfer area required for starting conditions of the reaction.

Solution

(a) For ideal batch reactor, time required to obtain the conversion X_A can be obtained using Eq. (10.1).
Given reaction is a liquid phase reaction in homogenous phase hence working volume can be assumed constant.

$$\theta = \frac{N_{Ao}}{V}\int_o^{X_A}\frac{dX_A}{kC_A^2} = C_{Ao}\int_o^{X_A}\frac{dX_A}{kC_{Ao}^2(1-X_A)^2}$$

$$\theta = \frac{1}{kC_{Ao}}\left[\frac{1}{1-X_A}\right]_0^{0.5} = \frac{1}{kC_{Ao}}\left(\frac{1}{(1-0.5)} - \frac{1}{(1-0)}\right)$$

$$\theta = \frac{1}{kC_{Ao}}(2-1) = \frac{1}{kC_{Ao}}$$

$$C_{Ao} = \frac{\text{Moles of acetic acid}}{\text{Total volume}}$$

$$= \frac{\text{Moles of acetic acid}}{\text{Total moles}} \times \frac{\text{Total moles}}{\text{Total volume}}$$

$$C_{Ao} = \frac{x_A^o \times \rho_{\text{mix}}}{M_{\text{av}}}$$

$$= \frac{(1/5.97)}{\left(\frac{1}{5.97}\times 60\right) + \left(\frac{4.97}{5.97}\times 74\right)} \times 0.75 = 0.0018 \text{ mol/cm}^3$$

$$\text{Mole ratio in feed} = \frac{\text{1 mole of acetic acid}}{\text{4.97 mole of butyl alcohol}}$$

$$x_A^\circ = 1/5.97$$

$$M_{\text{av}} = \left(\frac{1}{5.97}\times 60\right) + \left(\frac{4.97}{5.97}\times 74\right) = 71.655$$

where M of acetic acid = 60 and M of butyl alcohol = 74

$$k = 17.4 \text{ cm}^3/(\text{mol}\cdot\text{min})$$

$$\theta = \frac{1}{17.4\times 0.0018} = 32 \text{ min or } 0.53 \text{ h}$$

Time required to obtain 50% conversion is 32 min.

(b) Working volume of reactor = $\dfrac{\text{Mass of liquid (raw material) charged}}{\text{Density of liquid mixture charged}}$

Assume that volume of liquid mass inside the reactor will remain same during the reaction (or change in liquid volume during the reaction is negligible).
For 100 kg/h ester production with 50% conversion,
Molar mass of butyl acetate ($CH_3COOC_4H_9$) = 6 × 12 + 2 × 16 + 1 × 12 = 116
Mass of acetic acid required per hour

$$= \frac{100 \text{ kg/h}}{\text{Molar mass of ester}} \times \frac{\text{Molar mass of acetic acid}}{0.5}$$

$$= \frac{100}{116} \times \frac{60}{0.5} = 103.45 \text{ kg/h}$$

Mass of acetic acid required per batch

$$m_A = 103.45 \times \frac{(32 + 30)}{60} \quad \text{(Time between batches = 30 min)}$$

$$= 106.9 \text{ kg/batch}$$

Reaction

$$CH_3COOH(l) + C_4H_9OH(l) \rightleftharpoons CH_3COOC_4H_9(l) + H_2O(l)$$

Reaction is carried out at 90°C and with 50% conversion.
Mass of butanol required per batch,

$$m_B = \frac{106.9}{60} \times 74 \times 4.97 = 655.26 \text{ kg/batch}$$

Mass of H_2SO_4 required per batch

$$m_s = \frac{106.9 + 655.26}{(1 - 0.000\,32)} \times 0.000\,32 = 0.244 \text{ kg/batch}$$

Total mass of raw materials = 106.9 + 655.26 + 0.244 = 762.404 kg

$$V_{\text{working}} = \frac{762.404}{0.75 \times 1000} = 1.0165 \text{ m}^3$$

Let $\dfrac{h}{D_i} = 1$,

where, h = Depth of liquid in reactor shell, m
D_i = Diameter of vessel (inside diameter), m

Let the type of bottom head = Torispherical
Inside volume of Torispherical head,

$$V = 0.084\,672\, D_i^3 + \frac{\pi}{4} D_i^2 S_F \quad \text{(Ref. 2)} \tag{10.3}$$

where, D_i = Inside diameter of reactor, m
V = Inside volume, m^3
S_F = Straight flange, m

$$V_{\text{working}} = \frac{\pi}{4} D_i^2 h + 0.084\,672\ D_i^3 + \frac{\pi}{4} D_i^2 S_F = 1.0165 \text{ m}^3$$

Let $\quad S_F = 1.5 \text{ in} = 0.0381 \text{ m and } \dfrac{h}{D_i} = 1$

$$1.0165 = \frac{\pi}{4} D_i^3 + 0.084\,672\, D_i^3 + 0.029\,92 \times D_i^2$$

$$1.0165 = 0.870\,007\, D_i^3 + 0.029\,92\, D_i^2$$

$$D_i = 1.042 \text{ m} = h$$

Conside provision of 20% extra space for vapour-liquid disengagement, then actual height of shell of reactor $H = 1.2$ m.

(c) To determine heat duty required at starting condition of reactor, heat of reaction ΔH_r is required.

Table 10.1 Standard Heat of Formation and Heat Capacity Data

Compound	ΔH_f^o, kJ/mol at 25°C	C_{Li}, kJ/(kmol · °C) at 57.5°C
Acetic acid	–484.2	129.67
Butanol	–327.3	215.41
Water	–285.83	75.36
Butyl acetate	–522.766	267.12

$$\Delta H_r^o = (-522.766 - 285.83) - (-327.3 - 484.2)$$
$$= 2.904 \text{ kJ/mol (at 25°C)} \qquad \text{(endothermic)}$$

In a reactor, when reaction starts, both reactants and products are at the reaction temperature.

Heat of reaction at reaction temperature (90°C)

$$\Delta H_r = \Delta H_r^o + (\Sigma v_i\, C_{Li}\, \Delta t)/1000$$

where, $\quad v_i$ = Stoichiometric coefficient = 1 for all reactants

Assuming heat capacities to be nearly constant up to 90°C,

$$\Delta H_r = 2.904 + (267.12 + 75.26 - 215.41 - 129.67) \times \frac{(90 - 25)}{1000}$$

$$= 2.904 - 0.1755 = 2.7285 \text{ kJ/mol} \quad \text{(endothermic)}$$

Heat required at the starting of reaction

$\phi = \Delta H_r \times 1000 \times$ kmol/h of acetic acid consumed

+ Heat required to remove the water continuously from the reaction mass of ternary azeotrope

$$\phi = \left(2.7285 \times 1000 \times \left(\frac{-dN_A}{d\theta}\right) + \phi_v\right) \times 1.05 \quad \text{(Considering 5\% heat loss)}$$

Water will leave the reaction mass either as binary azeotrope with butanol (normal boiling point = 92.2°C) or as ternary azeotrope with butyl acetate and butanol (normal boiling point = 89.4°C). Boiling point of ternary azeotrope is less than binary azeotrope.

Composition of ternary azeotrope:
60.86% butyl acetate, 10.47% butanol, 28.92% water (by mass).

$$\text{Ratio of } \frac{\text{Butyl acetate formed}}{\text{Water formed}} = \frac{116}{18} = 6.44 > \frac{60.86}{28.92} \ (= 2.1)$$

Hence, it will be logical to assume that water will leave as ternary azeotrope.
Rate of reaction at the starting of reaction

$$-r_A = kC_{Ao}^2 \quad (C_A = C_{Ao} \text{ when } \theta = 0)$$

or

$$-\frac{dC_A}{d\theta} = 17.4 \ \frac{\text{cm}^3}{\text{mol} \cdot \text{min}} \times (0.0018)^2 \left(\frac{\text{mol}}{\text{cm}^3}\right)^2$$

Moles of acetic acid consumed per unit volume of reactor

$$= \frac{-dC_A}{d\theta} = 5.6376 \times 10^{-5} \ \frac{\text{mol}}{(\text{cm}^3 \cdot \text{min})}$$

$$\text{Moles of acetic acid consumed} = \frac{-dN_A}{d\theta} = 5.6376 \times 10^{-5} \ \frac{\text{mol}}{(\text{cm}^3 \cdot \text{min})} \times V$$

$$\frac{-dN_A}{d\theta} = 5.6376 \times 10^{-5} \times 1.0165 \times 10^6 \times 60 \times \frac{1}{10^3}$$

$$= 3.438\ 37 \text{ kmol/h (when } \theta = 0 \text{ or at starting of reaction)}$$

Water formation rate = 3.438 37 kmol/h = 61.89 kg/h
Total flow rate of ternary azeotrope in starting of reaction

$$= \frac{61.89}{0.2892} = 214 \text{ kg/h}$$

It will contain 130.24 kg/h of butyl acetate, 22.4 kg/h of butanol and 61.89 kg/h of water.
ϕ_v at starting of reaction = $\Sigma m_i \lambda_i$

Table 10.2 Latent Heat of Vaporization Data[2]

Component	Latent heat of vaporization at 89.4°C (λ), kJ/kg
Butanol	625.39
Butyl acetate	330.2
Water	2284

$$\phi_v = 130.24 \times 330.2 + 22.4 \times 625.39 + 61.89 \times 2284$$
$$= 198\ 370.7 \text{ kJ/h} \equiv 55.103 \text{ kW}$$
$$\phi = (2.7285 \times 1000 \times 3.43837 + \phi_v) \times 1.05 = 218\ 139.9 \text{ kJ/h}$$
$$\equiv 60.5944 \text{ kW}$$

As a heating medium saturated steam is used.
Let saturated steam pressure = 0.2 MPa a
Saturation temperature = 120.23°C
Latent heat of vaporization of steam, λ_s = 2201.6 kJ/kg

Steam required $\dot{m}_s = \dfrac{\phi}{\lambda_s} = \dfrac{60.5944}{2201.6} = 0.0275 \text{ kg/s} \equiv 99.08 \text{ kg/h}$

Saturated steam side heat transfer coefficient $h_o' = 6000 \text{ W/(m}^2 \cdot \text{°C)}$ (assumed)

Inside heat transfer coefficient, h_i:

It is difficult to find the suitable correlation for the reacting mass side heat transfer coefficient as the reaction is taking place. h_i can be governed by boiling coefficient or by convective film coefficient. If flat blade disc turbine agitator is used to improve the reaction rate and to improve the convective film heat transfer coefficient, then

$$h_i = h_{nB} \text{ or } h_{iC} \text{ whichever is less}$$

where, h_{iC} = Convective film coefficient, W/(m^2 · °C)

h_{nB} = Nucleate boiling coefficient, W/(m^2 · °C)

h_{ic}

Normally, tip velocity of turbine agitator is in the range of 150 to 250 m/min.

To calculate the convective film coefficient, let the tip velocity of turbine agitator

$$v = 200 \text{ m/min} = \pi D_a n$$

where, D_a = Diameter of agitator

n = Rotational speed of agitator in revolutions per min

For flat blade disc turbine

$$D_a = \frac{\text{shell ID}}{3} = \frac{1.042}{3} = 0.347 \text{ m}$$

$$n = \frac{200}{\pi \times 0.347} = 183.5 \cong 184 \text{ rpm}$$

For flat blade disc turbine, baffled vessel for $Re > 400$ (Ref. 5)

$$\frac{h_i D_a}{k} = 0.74\, Re^{0.67}\, p_r^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{10.4}$$

where, $$Re = \frac{n D_a^2 \rho}{\mu} \tag{10.5}$$

Table 10.3 Composition of Reaction Mass at the Starting of Reaction

Component	Mass fractions at $\theta = 0$
Acetic acid	0.1405
Butanol	0.8595
Total	1.0000

$$\rho_{\text{mix}} = 750 \text{ kg/m}^3$$

Viscosity of organic liquid mixture

$$\frac{1}{\mu_{\text{mix}}} = \frac{w_1}{\mu_1} + \frac{w_2}{\mu_2} = \frac{0.1405}{\mu_{AA}} + \frac{0.8595}{\mu_{Bu}} \tag{7.31}$$

$$\mu_{Bu} = 0.65 \text{ mPa} \cdot \text{s at 90°C}$$

$$\mu_{AA} = 0.53 \text{ mPa} \cdot \text{s at 90°C}$$

$$\mu_{\text{mix}} = 0.63 \text{ mPa} \cdot \text{s}$$

Reynolds number, $$Re = \frac{nD_a^2 \rho_{\text{mix}}}{\mu_{\text{mix}}} \frac{(184/60)\, 0.347^2 \times 750}{0.63 \times 10^{-3}} = 439\ 588$$

$$Pr = \frac{C_{p\,\text{mix}} \cdot \mu_{\text{mix}}}{k_{\text{mix}}} \tag{10.6}$$

Table 10.4 Thermal Properties of Acetic Acid and Butanol

Component	C_{pi}, kJ/(kg · °C)	k, W/(m · °C)
Acetic acid	2.1855	0.1557
Butanol	2.8763	0.164

$$C_{p\,\text{mix}} = \Sigma C_{pi}\, w_i = 2.1855 \times 0.1405 + 2.8763 \times 0.8595 = 2.78 \text{ kJ/(kg} \cdot \text{°C)}$$

Thermal conductivity of liquid mixture can be calculated by Li method. (Equations 3.127 to 3.130 of Ref. 2)

$$k_{\text{mix}} = \phi_1^2\, k_{L1} + 2\phi_1\phi_2\, k_{12} + \phi_2^2\, k_{L2} \tag{10.7}$$

In Eq. (10.7), ϕ_1 and ϕ_2 are parameters and do not represent heat loads.

$$k_{L1} = 0.1557 \text{ W/(m} \cdot \text{°C)} \quad k_{L2} = 0.164 \text{ W/(m} \cdot \text{°C)}$$

$$k_{12} = 2(k_{L1}^{-1} + k_{L2}^{-1})^{-1} \tag{10.8}$$

$$= 0.159\ 74 \text{ W/(m} \cdot \text{°C)}$$

$$\phi_1 = \frac{x_1 V_1}{x_1 V_1 + x_2 V_2} \quad \text{and} \quad \phi_2 = \frac{x_2 V_2}{x_1 V_1 + x_2 V_2} \tag{10.9}$$

$$x_1 = \text{Mole fraction of acetic acid} = \frac{\left(\dfrac{0.1405}{60}\right)}{\left(\dfrac{0.1405}{60}\right) + \left(\dfrac{0.8595}{74}\right)} = 0.1678$$

$x_2 = 1 - 0.1678 = 0.8322 =$ Mole fraction of butanol

$V_1 =$ Molar volume of pure acetic acid

$$= \frac{M}{\rho} = \frac{60}{1048} = 0.057\ 25 \text{ m}^3\text{/kmol}$$

$V_2 =$ Molar volume of pure butanol

$$= \frac{M}{\rho} = \frac{74}{859.5} = 0.086 \text{ m}^3\text{/kmol}$$

$$\phi_1 = \frac{0.1678 \times 0.057\ 25}{0.1678 \times 0.057\ 25 + 0.8322 \times 0.086} = 0.118\ 34$$

$$\phi_2 = 0.881\ 66$$

$$k_{\text{mix}} = 0.118\ 34^2 \times 0.1557 + 2 \times 0.118\ 34 \times 0.881\ 66 \times 0.159\ 74 + 0.881\ 66^2 \times 0.164$$

$$= 0.163 \text{ W/(m} \cdot \text{°C)}$$

$$Pr = \frac{2.78 \times 0.63 \times 10^{-3} \times 10^3}{0.163} = 10.744$$

Using Eq. (10.4): $h_i = 0.74 \times \dfrac{0.163}{0.347} \times (439\,588)^{0.67} \times (10.744)^{0.33} \times 1$

$$= 4594.25 \text{ W/(m}^2 \cdot \text{°C)} = h_{nC}$$

Nucleate boiling coefficient for the boiling of azeotropic mixture can be determined by Mostinski's equation:

$$h_{nB} = 0.104\, p_c^{0.69} \left(\frac{\phi}{A}\right)^{0.7} \left[1.8\left(\frac{p}{p_c}\right)^{0.17} + 4\left(\frac{p}{p_c}\right)^{1.2} + 10\left(\frac{p}{p_c}\right)^{10}\right] \quad (6.82)$$

It is valid for single component boiling. But it can be used for close boiling range and azeotropic mixtures.

$$\phi = 60.5944 \times 10^3 \text{ W}$$

For nucleate boiling $\Delta T_m = 30.83$°C (as decided earlier)

Heat transfer area provided with full height covered jacket,

$$A_{\text{opr}} \cong \pi D_o h \cong \pi \times 1.0547 \times 1.042 = 3.4526 \text{ m}^2$$

(Assuming shell thickness, $t_s = 6.35$ mm)

p = operating pressure = 0.9869 bar

p_c = critical pressure, bar

Pseudo-Critical pressure of mixture $p_{cm} = \Sigma p_{ci}\, y_i$ (Kay's rule)

Table 10.5 Critical Pressure of Azeotropic Mixture

Component of Azeotropic mixture	Mass fraction	Mole fraction	Molar mass	Critical pressure, p_c, bar
Butyl acetate	0.6086	0.23	116	31.4
Butanol	0.1047	0.06	74	44.2
Water	0.2892	0.71	18	220.5
Total	1.0000	1.00		

$$p_{cm} = 0.23 \times 31.4 + 0.06 \times 44.2 + 0.71 \times 220.5 = 166.43 \text{ bar}$$

$$p_r = \frac{p}{p_c} = \frac{0.9869}{166.43} = 5.9298 \times 10^{-3}$$

$$h_{nB} = 0.104\,(166.43)^{0.69} \left(\frac{60.5944 \times 10^3}{3.4526}\right)^{0.7} (1.8 p_r^{0.17} + 4 p_r^{1.2} + 10 p_r^{10})$$

$$h_{nB} = 2524.95 \text{ W/(m}^2 \cdot \text{°C)}$$

Convective film coefficient, $h_i = 4594.25$ W/(m^2 · °C) > h_{nB}

Since nucleate boiling coefficient is less than the convective coefficient, it is taken for heat transfer area calculations.

$$h_i = 2524.95 \text{ W/(m}^2 \cdot \text{°C)}$$

Jacket side or saturated steam side heat transfer coefficient,

$$h'_o = 6000 \text{ W/(m}^2 \cdot \text{°C)}$$

Thermal conductivity of reactor shell material (SS-304) = 16 W/(m · °C)

Let thickness of reactor shell, t_s = 6.35 mm (Assumed but it should be decided based on mechanical design).

Overall heat transfer coefficient: Eq. (6.46)

$$\frac{1}{U_o} = \frac{1}{h'_o} + \frac{D_o \ln\left(\frac{D_o}{D_i}\right)}{2k_w} + \frac{D_o}{D_i} \times \frac{1}{h_i} + \frac{D_o}{D_i} \times \frac{1}{h_{id}}$$

$$D_i = 1.042 \text{ m}, \quad D_o = D_i + 2t_s = 1.042 + 2 \times 0.006\,35 = 1.0547 \text{ m}$$

$$\frac{1}{U_o} = \frac{1}{6000} + \frac{1.547 \ln\left(\frac{1.0547}{1.042}\right)}{2 \times 16} + \frac{1.0547}{1.042} \times \frac{1}{2524.95} + \frac{1.0547}{1.042} \times \frac{1}{5000}$$

$$U_o = 737.66 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat duty, $\phi_t = 60.5944 \times 10^3$ W, $\Delta T_m = 30.23°C$

Heat transfer area required

$$A = \frac{\phi_t}{U_o \Delta T_m} = \frac{60.5944 \times 10^3}{737.66 \times 30.23} = 2.7173 \text{ m}^2$$

Outside heat transfer area provided with full height covered jacket

$$A_{o\text{av}} \cong 3.4526 \text{ m}^2$$

Excess heat transfer area = 27.06%

Thus available heat transfer area is sufficient.

10.4 CONTINUOUS FLOW REACTORS

All continuous flow reactors are steady state flow reactors. In a steady state flow reactor, composition at any point is unchanged with time.

For continuous flow reactors, in-line mixers are used. In-line shear mixers are developed for continuous degumming of edible oils. In-line static mixers are effective in achieving desired mixing in continuous systems. In continuous nitration, mixing of a reactant with mixed acid is very effective through a static mixer. Similarly, mixing of natural gas/naphtha vapour with steam prior to reforming can be carried out in an in-line static mixer. For removal of free fatty acids from edible/non-edible oils with caustic soda solution, a static mixer is used in a continuous plant. In Fig. 10.3, different designs, of static mixers are shown.

Continuous flow reactors are used for achieving high throughputs. Also quality of product can be maintained at desired level once the operating conditions are stabilised.

There are two ideal steady state flow reactors:

(a) Ideal plug flow reactor (b) Ideal mixed flow reactor

10.4.1 Ideal Plug Flow Reactor[1, 2, 3]

It is also known as ideal tubular, unmixed flow, slug flow or piston flow reactor. Ideal plug flow reactor has flat velocity profile. In this type of reactor, lateral or radial mixing

is permitted, but axial mixing is not permitted. Hence, in ideal PFR, the residence time in the reactor is same for all elements or particles of fluid.

Fig. 10.3 *Different Types of Static Mixers*

Ideal PFR is defined by two criteria:

1. Over any cross section of ideal PFR, the mass flow rate, velocity and the fluid properties like pressure, temperature and composition are uniform and the same do not change with time.
2. There is negligible diffusion relative to bulk flow or no element of fluid overtaking or mixing with any other element ahead or behind.

In ideal PFR, composition at any point is not changing with time but it changes with length or axial distance in reactor.

Fig. 10.4 *Plug Flow Reactors*

Industrial plug flow reactors can be fixed bed catalytic reactor, packed tower-type reactor, tubular reactor with very high length to diameter ratio and zigzag path, shell

and tube heat exchanger, double pipe heat exchanger, sieve tray tower, etc. Working volume of ideal PFR can be determined by using the following equation:

$$\frac{V}{F_{Ao}} = \int_{o}^{X_A} \frac{dX_A}{-r_A} \tag{10.10}$$

where, V = Working volume of ideal PFR, m^3

F_{Ao} = Molar flow rate of limiting reactant A at inlet, kmol/s

X_A = Fractional conversion of liming reactant required

$-r_A = \frac{dC_A}{d\theta}$ = Rate of chemical reaction, kmol/($m^3 \cdot s$)

Residence time required in ideal PFR for the desired extent of given reaction can be determined by the following equation:

$$\theta = C_{Ao}\frac{V}{F_{Ao}} = \frac{V}{V_o} = C_{Ao}\int_{o}^{X_A} \frac{dX_A}{-r_A} \tag{10.11}$$

where, θ = Residence time required, s

V_o = Volumetric flow rate of reacting fluid at inlet, m^3/s

Advantages and disadvantages of plug flow reactors over mixed flow reactors

Advantages

1. For any positive order reaction, plug flow reactor require minimum volume among all types of continuous flow reactors. Volume required by ideal PFR is very much less than the same by ideal mixed flow reactor, particularly for a higher order reaction with higher desired conversion.
2. Plug flow reactor does not use moving mechanical parts and hence it is suitable for high pressure reaction system, corrosive reaction system and for the reaction system which involves the use of a toxic gas.

Disadvantages

1. Cost per unit volume of plug flow reactor is higher than the same of mixed flow reactor.
2. It requires high investment cost for lower conversion and lower order reaction.

Industrial examples of plug flow reactors

1. Conversion of ethylene to low density polythylene (LDPE).
2. Hydrolysis of ethylene oxide to ethylene glycol.
3. Fixed bed catalytic reaction.
   Some examples in this category are as follows:
   (a) Conversion of NH_3 from a mixture of N_2 and H_2.
   (b) Methanol synthesis reaction from a mixture of CO, CO_2 and H_2.
   (c) Steam reforming reaction.
   (d) Air oxidation of methanol to formaldehyde.

(e) Oxidation of SO_2 to SO_3 in sulphuric acid process.
(f) Oxidation of ethylene to ethylene oxide.

4. Olefins production by steam cracking, e.g., ethylene.
5. Conversion of ammonium carbamate to urea in an autoclave.
6. Splitting of edible oil to fatty acids.
7. Trickel bed reactors; Examples in this category are:
   (a) Hydrocracking of heavy gas oil.
   (b) Hydrodenitrogenation of a lube oil distillate.

10.4.2 Ideal Mixed Flow Reactor

It is also known as the back mix reactor (BMR), ideal continuous stirred tank reactor or as constant flow stirred tank reactor. In ideal mixed flow reactor (MFR), composition within the reactor is uniform throughout and does not change with time. Hence, in ideal MFR (or in ideal BMR or CSTR), exit stream from the reactor has the same composition as the fluid within the reactor.

Among all types of continuous flow reactors, ideal mixed flow reactor requires maximum volume for the given positive order reaction. So, volume required by ideal mixed flow reactor (V_m) is greater than volume required by ideal plug flow reactor (V_p). Ratio V_m/V_p increases with increase in order of reaction and also with increase in required conversion.

In an ideal CSTR, once the reactants enter into the reaction mass, they immediately (practically within no time) achieve final required composition. This is not due to very fast reaction but this is merely due to the dilution of reactants by the products. Actually, the rate of reaction is minimum in ideal CSTR, compared to all types of continuous flow reactors as it provides minimum concentration of reactants during reaction.

In an ideal CSTR, not only composition but temperature is also uniform throughout. Temperature of product stream is same as that within the reactor.

Industrial mixed flow reactors can be

(a) Agitated vessel-type reactor.
(b) Sparged vessel (including gas induced agitator type) or bubble column-type reactor.
(c) Loop reactor (with recirculation, recirculating pump and external heat exchanger).
(d) Jet reactor with recirculation through an ejector.

Volume required by ideal mixed flow reactor is determined by the following equation:

$$\frac{V}{F_{Ao}} = \frac{X_A}{-r_A} \tag{10.12}$$

Residence time required in ideal mixed flow reactor for the desired extent of given reaction is determined by the following equation [modified form of Eq. (10.2)].

$$\theta = C_{Ao}\frac{V}{F_{Ao}} = \frac{V}{V_o} = C_{Ao}\frac{X_A}{-r_A} \tag{10.13}$$

where, θ = Residence time required, s
C_{Ao} = Initial concentration of limiting reactant A, kmol/m^3
V = Working volume of reactor, m^3
V_o = Volumetric flow rate of reacting fluid at inlet, m^3/s
X_A = Fractional conversion of limiting reactant A

Advantages of ideal mixed flow reactor

1. Good mixing and lower rate of reaction (lower rate of heat generation in case of exothermic reaction) makes the temperature control easier. In the entrance portion of plug flow reactor and in starting period of batch reaction, rate of reaction is very high and hence, temperature control is difficult. Agitated vessel-type ideal mixed flow reactor also provides higher heat transfer coefficient compared to packed tower-type or sieve tray-type plug flow reactor which are normally operated in adiabatic manner.
2. Ideal mixed flow reactor may require less fixed cost compared to ideal plug flow reactor for lower order reaction with lower equilibrium conversion because cost per unit volume of mixed flow reactor is less than the same of plug flow reactor.
3. Polymerisation reactions can be better controlled in ideal CSTR compared to the same in ideal PFR (e.g., desired molar mass of polymer can be easily achieved).
4. Fine catalyst particles can be effectively suspended throughout the liquid reaction system with agitated vessel-type mixed flow reactor.
5. For highly viscous fluid or slurry solution, agitated vessel-type mixed flow reactor provides higher heat transfer coefficient and mass transfer coefficient, compared to plug flow reactor.

Disadvantages of ideal mixed flow reactor

1. Ideal mixed flow reactor provides the minimum rate of reaction among all types of continuous flow reactors. Hence, it requires maximum volume among all types of continuous reactors for any given positive order reaction and required conversion. For the higher order reaction with high conversion; e.g., second order reaction with 90% conversion, volume required by ideal mixed flow reactor is many times higher than that required for ideal PFR.
2. Changeover to other product is difficult compared to stirred tank batch reactor.

Industrial examples of mixed flow reactors

1. Carbonylation of methanol gives acetic acid in presence of liquid phase catalyst.
2. Oxidation of acetaldehyde to acetic acid in presence of fine $KMnO_4$ particles as catalyst.
3. Syrene to polystyrene by solution polymerisation.
4. Slurry polymerisation of propylene to polypropylene.
5. Chlorination of benzene to chlorobenzene by continuous process.
6. Oxidation of toluene to benzoic acid.
7. Vinyl chloride to polyvinyl chloride by emulsion and suspension polymerisation.

Example 10.2 A homogeneous liquid phase reaction

$$A \rightarrow R$$

takes place with 50% conversion in a mixed flow reactor. Its rate equation is

$$-r_A = \frac{dC_A}{d\theta} = kC_A^2$$

(a) What will be the conversion if this reactor is replaced by the one, six times larger reactor and all else remaining unchanged?
(b) What will be the conversion if the original reactor is replaced by a plug flow reactor of equal size and all else remaining unchanged?

Solution

Performance equation of ideal mixed flow reactor is

$$\frac{V}{F_{Ao}} = \frac{X_A}{-r_A} \tag{10.11}$$

$$\frac{V}{F_{Ao}} = \frac{X_A}{kC_A^2} = \frac{X_A}{kC_{Ao}^2(1-X_A)^2} = \frac{0.5}{kC_{Ao}^2(1-0.5)^2} = \frac{2}{kC_{Ao}^2}$$

(a) For $V' = 6V$ (where V' = volume of larger reactor)

$$\frac{V}{F_{Ao}} = \frac{X_A}{-r_A} = \frac{X_A}{kC_A^2} = \frac{X_A}{kC_{Ao}^2(1-X_A)^2} \tag{1}$$

Also $$\frac{V'}{F_{Ao}} = 6\frac{V}{F_{Ao}} = 6 \times \frac{2}{kC_{Ao}^2} = \frac{12}{kC_{Ao}^2} \tag{2}$$

From Eqs. (1) and (2), $$\frac{X_A}{kC_{Ao}^2(1-X_A)^2} = \frac{12}{kC_{Ao}^2}$$

$$12(1-X_A)^2 = X_A$$

$$X_A^2 - 2.083\,33\,X_A + 1 = 0$$

$$X_A = 1.333 \text{ or } X_A = 0.75$$

X_A cannot be greater than 1; hence $X_A = 0.75$

Hence, six times larger ideal mixed flow reactor will give 75% conversion.

(b) $V_{\text{mixed}} = V_{\text{plug}} = V$

Performance equation of ideal plug flow reactor

$$\frac{V}{F_{Ao}} = \int_o^{X_A} \frac{dX_A}{-r_A} = \int_o^{X_{Ap}} \frac{dX_A}{kC_{Ao}^2(1-X_{AP})^2} \tag{10.9}$$

where, X_{AP} = Fractional conversion of reactant A in ideal plug flow reactor

Let $p = 1 - X_{AP}$, hence, $\dfrac{dp}{dX_{AP}} = -1$

$$\frac{V}{F_{Ao}} = \frac{1}{kC_{Ao}^2}\int_1^p \frac{dp}{p^2} = -\frac{1}{kC_{Ao}^2}\left[\frac{p^{-2+1}}{-2+1}\right]_1^p = \frac{1}{kC_{Ao}^2} \times \frac{1-p}{p}$$

$$\frac{V}{F_{Ao}} = \frac{1}{kC_{Ao}^2}\frac{X_{AP}}{1-X_{AP}}$$

For ideal mixed flow reactor $$\frac{V}{F_{Ao}} = \frac{2}{kC_{Ao}^2}$$

$$V = \frac{2F_{Ao}}{kC_{Ao}^2} = V_{\text{mixed}} = V_{\text{plug}}$$

$$\frac{2F_{Ao}}{kC_{Ao}^2} \times \frac{1}{F_{Ao}} = \frac{1}{kC_{Ao}^2} \frac{X_{AP}}{1 - X_{AP}}$$

$$2 - 2X_{AP} = X_{AP}$$

$$X_{AP} = 2/3 = 0.6667$$

Hence, if the original ideal mixed flow reactor (giving 50% conversion) is replaced by an ideal plug flow reactor of equal size, then conversion will be 66.67%.

Example 10.3 The following data have been obtained on the decomposition of gaseous reactant A in a constant volume batch reactor at 100°C.

Table 10.6 Time vs Pressure Data of Decomposition of A

θ, s	p_A, atm	θ, s	p_A, atm
0	1	140	0.25
20	0.8	200	0.14
40	0.68	260	0.08
60	0.56	330	0.04
80	0.45	420	0.02
100	0.37		

The stoichiometry of the reaction is 2 A = R + S.

(a) What size of plug flow reactor (in litres) operating at 100°C and 1 atm can treat 100 moles of A/h in a feed consisting of 20% inerts to obtain 95% conversion of A?

(b) What conversion of A can be expected in mixed flow reactor of volume $V = 208$ L, for the same identical feed and identical operating conditions?

Solution

First rate equation, based on constant volume batch reactor data, must be determined. From the data, at $\theta = 420$ s, p_A is 0.02 atm means it is reduced to a value very close to zero. Hence, given reaction is irreversible reaction,

Assuming first order irreversible reaction,

$$-r_A = \frac{-dC_A}{d\theta} = kC_A$$

$$p_A = C_A RT$$

$$-\frac{1}{RT}\frac{dp_A}{d\theta} = k\frac{p_A}{RT}$$

$$-\frac{dp_A}{d\theta} = kp_A$$

$$\int_{p_{Ao}}^{p_A} -\frac{dp_A}{p_A} = k\int_{o}^{\theta} d\theta$$

$$-\ln\left(\frac{p_A}{p_{Ao}}\right) = k\theta$$

From the given data, following table can be prepared:

Table 10.7 Tabulation for Calculation of Integral

θ, s	p_A, atm	$-\ln\left(\frac{p_A}{p_{Ao}}\right)$
0	1.00	0.0000
20	0.80	0.2230
40	0.68	0.3856
60	0.56	0.5800
80	0.45	0.7980
100	0.37	0.9940
140	0.25	1.3860
200	0.14	1.9660
260	0.08	2.5260
330	0.04	3.2200
420	0.02	3.9100

The graph of $-\ln\left(\frac{p_A}{p_{Ao}}\right)$ vs θ (Fig. 10.5) is a straight line passing through origin.

Hence, given reaction is irreversible reaction and is of first order.

$$k = 0.009\ 76\ \text{s}^{-1}$$

$$-r_A = 0.009\ 76\ C_A\ \text{mol/(L} \cdot \text{s)} \equiv 35.136\ C_A\ \text{mol/(L} \cdot \text{h)}$$

(a) Performance equation of ideal plug flow reactor,

$$\theta = C_{Ao}\int_{o}^{X_A} \frac{dX_A}{-r_A}$$

Reaction 2 A → R + S, with 20% inert:

$$\varepsilon_A = \frac{V_{X_A=1} - V_{X_A=0}}{V_{X_A=0}}$$

$\varepsilon_A = 0$, as total number of moles of reaction system is not changing

$$\theta = C_{Ao}\int_{o}^{X_A} \frac{dX_A}{kC_{Ao}(1-X_A)} = \frac{1}{k}\int_{o}^{X_A} \frac{dX_A}{(1-X_A)}$$

Fig. 10.5 *Determination of Order of Reaction*

$$-\ln(1 - X_A) = k\theta$$

$$\theta = -\frac{1}{k}\ln(1 - X_A) = -\frac{1}{0.009\ 76}\ \ln(1 - 0.95) = 307 \text{ s}$$

$$\theta = C_{Ao}\frac{V}{F_{Ao}} = 307 \text{ s}$$

Working volume of reactor, $V = \theta\dfrac{F_{Ao}}{C_{Ao}} = 307\dfrac{F_{Ao}}{C_{Ao}}$

$$C_{Ao} = \frac{p_{Ao}}{RT}, \quad p_{Ao} = p_t \times 0.8 = 0.8 \text{ atm (20\% inerts)}$$

$$C_{Ao} = \frac{0.8}{(273+100)\times 0.082} = 0.026\,16 \text{ mol/L}$$

$$F_{Ao} = 100 \text{ mol/h}$$

$$V = \frac{307\times 100\times (1/3600)}{0.026\,16} = 326 \text{ L}$$

(b) For ideal mixed flow reactor,

$$\frac{V}{F_{Ao}} = \frac{X_A}{-r_A} = \frac{X_A}{kC_{Ao}(1-X_A)}$$

$$\frac{X_A}{1-X_A} = \frac{kC_{Ao}V}{F_{Ao}} = \frac{0.009\,76\times 0.026\,16\times 208}{100\times\left(\frac{1}{3600}\right)}$$

$$\frac{X}{1-X_A} = 1.912$$

$$X_A = 0.6565$$

65.65% conversion of reactant A can be expected in ideal mixed flow reactor.

Example 10.4 It has been reported that the reaction

$$\begin{array}{c} CH_2OH \\ | \\ CH_2Cl \\ \text{A} \\ \text{Ethylene Chlorohydrin} \end{array} + \begin{array}{c} NaHCO_3 \\ \\ \\ \text{B} \\ \text{Sodium Bicarbonate} \end{array} \rightarrow \begin{array}{c} CH_2OH \\ | \\ CH_2OH \\ \\ \text{Ethylene Glycol} \end{array} + \begin{array}{c} NaCl \\ \\ \\ \\ \text{Salt} \end{array} + \begin{array}{c} CO_2 \\ \\ \\ \\ \text{Carbon Dioxide} \end{array}$$

is elementary with rate constant k = 5.2 L/(mol · h) at 82°C. On the basis of this information it is intended to construct a pilot plant to determine the economical feasibility of producing ethylene glycol from two available feeds, a 15 mass % aqueous solution of sodium bicarbonate and a 30 mass % aqueous solution of ethylene chlorohydrin.

(a) What volume of tubular (plug flow) reactor will produce 20 kg/h ethylene glycol at 95% conversion of an equimolar feed produced by intimately mixing appropriate quantities of the two feed streams?

(b) What size of mixed reactor is needed for the same feed, conversion and production rate as in Part (a)?

Assume all operations at 82°C at which specific gravity of the mixed reacting fluid is 1.02.

Solution

(a) Performance equation of ideal tubular reactor

$$\frac{V_p}{F_{Ao}} = \int_o^{X_A} \frac{dX_A}{-r_A} \tag{10.9}$$

Here given reaction is elementary reaction. Hence, rate equation is corresponding to stoichiometric equation.

$$-r_A = kC_A\,C_B$$

for equimolar feed, $C_{Ao} = C_{Bo}$ and $C_A = C_B$

$$-r_A = kC_A^2$$

$$\frac{V_p}{F_{Ao}} = \int_o^{X_A} \frac{dX_A}{kC_A^2} = \int_o^{X_A} \frac{dX_A}{kC_{Ao}^2(1-X_A)^2}$$

$$kC_{Ao}^2 \frac{V_p}{F_{Ao}} = \int_o^{X_A} \frac{dX_A}{(1-X_A)^2} = \left[\frac{1}{1-X_A}\right]_0^{X_A} = \frac{1}{1-X_A} - 1 = \frac{X_A}{1-X_A}$$

$$5.2 \times C_{Ao}^2 \frac{V_P}{F_{Ao}} = \frac{0.95}{1-0.95}$$

For equimolar feed and based on stoichiometric equation,

$F_{Ao} \times 0.95$ = Moles of ethylene glycol produced (for 95% conversion)

$$F_{Ao} = \frac{20 \text{ kg/h}}{0.95 \times \text{Molar mass of ethylene glycol}}$$

$$= \frac{20}{0.95 \times 62} = 0.3396 \text{ kmol/h}$$

Total Mass of Feed:

$\dot{m}_F$ = Mass flow rate of ethylene chlorohydrin solution + Mass flow rate of sodium bicarbonate solution

= $(0.3396 \times M_{EC}$ + Mass of associated water) + $(0.3396 \times M_{SB}$ + mass of associated water)

where, M_{EC} = Molar mass of ethylene chlorohydrin = 80.5 kg/kmol

M_{SB} = Molar mass of sodium bicarbonate = 84 kg/kmol

$$\dot{m}_F = \left(0.3396 \times 80.5 + \frac{0.3396 \times 80.5}{0.3} \times 0.7\right)$$

$$+ \left(0.3396 \times 84 + \frac{(0.3396 \times 84)}{0.15} \times 0.85\right)$$

= (27.3378 + 63.7882) + (28.5264 + 161.6496)

= 281.302 kg/h

Density of reacting fluid = 1020 kg/m^3

$$V_o = \frac{281.302}{1020} = 0.2758 \text{ m}^3\text{/h}$$

$$C_{Ao} = \frac{F_{Ao}}{V_o} = \frac{0.3396}{0.2758} = 1.2313 \text{ kmol/m}^3$$

$$k = 5.2 \text{ L/(mol} \cdot \text{h)} = 5.2 \text{ m}^3\text{/(kmol} \cdot \text{h)}$$

$$5.2 \times C_{Ao}^2 \times \frac{V}{F_{Ao}} = \frac{0.95}{1-0.95}$$

$$5.2 \times \frac{(1.2313)^2}{0.3396} \times V_p = \frac{0.95}{1-0.95}$$

$$V_p = 0.818\,45 \text{ m}^3 \equiv 818.45 \text{ L}$$

(b) For ideal mixed flow reactor

$$\frac{V_m}{F_{Ao}} = \frac{X_A}{-r_A} = \frac{X_A}{kC_{Ao}^2(1-X_A)^2}$$

$$V_m = \frac{F_{Ao}}{kC_{Ao}^2}\frac{X_A}{(1-X_A)^2}$$

$$V_m = \frac{0.3396}{5.2\times(1.2313)^2}\times\frac{0.95}{(1-0.95)^2}$$

$$V_m = 16.369\ \text{m}^3 \equiv 16\ 369\ \text{L}$$

Note: (i) In general, for any positive order reaction, V_m is greater than V_p.
(ii) Ratio V_m/V_p increases with increase in the order of reaction and increase in conversion.

10.5 DEGREE OF COMPLETION OF REACTION

Chemical engineers are familiar with degree of conversion of a reaction which is well defined as the ratio of moles of limiting reactant consumed to that in the feed. However, it is not possible to define the degree of conversion of reaction(s) in all cases. Chief reason for not able to define the conversion clearly is (known or unknown) composition of feed in which more than one reactant react simultaneously (i.e., parallel reactions) and degree of completion of each reaction is unknown. Under these circumstances, degree of completion of reaction is defined in several ways.

Consider a classical reforming reaction of hydrocarbons with steam. There are a number of hydrocarbons present in the feed. They react simultaneously with steam and produce carbon monoxide, carbon dioxide and hydrogen.

$$C_6H_{14} + 6\ H_2O \rightleftharpoons 6\ CO + 13\ H_2$$
$$C_5H_{12} + 5\ H_2O \rightleftharpoons 5\ CO + 11\ H_2$$

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— — — — — — — — — —

$$C_2H_6 + 2\ H_2O \rightleftharpoons 2\ CO + 5\ H_2$$
$$CH_4 + H_2O \rightleftharpoons CO + 3\ H_2$$
$$CO + H_2O \rightleftharpoons CO_2 + H_2$$

Study of equilibrium conversions of competing reactions reveal that while most hydrocarbons reform to hydrogen, methane is not always fully reformed. Thus, degree of completion of the reforming reaction is judged by methane slip (i.e., mole % methane in outgoing gas mixture from the reformer).

Edible oils are hydrogenated to produce hydrogenated fat. Basically, edible oil is a mixture of (mostly) triglycerides of fatty acids. Each edible oil lot has different composition of variety of fatty acids. It is not possible to know the exact composition in terms of fatty acids for each batch of oil. These fatty acids may be saturated or unsaturated. The degree of unsaturation is measured by titration with potassium iodide and is known as *iodine number*. Higher the iodine number, higher is the unsaturation. When hydrogenated, double and triple bonds open up and saturation takes place with hydrogen. For this hydrogenation reaction, degree of completion is measured by reduction in the iodine value of the oil.

Alkyd resins are produced by reacting an organic acid such as phthalic anhydride, benzoic acid, etc., with glycerine or other polyols. During the course of esterification reaction, acid is consumed. Degree of completion of this process is measured by the final acid value (refer Exercise 2.9) of the product.

In demineralisation of water, cations are removed in the cation exchanger while anions are removed in the anion exchanger. While bi- and tri-valents are removed on a priority, monovalent ions are not easily removed. Hence, degree of demineralisation (i.e., degree of completion of each ion exchanger) is measured by sodium and chloride slips, respectively.

Several other examples can be cited for the degree of completion.

It can, therefore, be visualised that the degree of completion of reaction is defined in terms of a measurable parameter.

These reactors are designed for desired completion of reaction. For such reactions, rate of reaction need not be defined as for pure chemical reaction (see Example 10.5). It is normally expressed in terms of measurable index with respect to other parameters.

Rate of reaction of manufacture of alkyd resin can be expressed in terms of disappearance of acid value. Typical kinetic expression could be

$$AV = X\theta + Y$$

where, AV = Acid value after time θ

θ = Time

X and Y are constants. These could be determined by laboratory and pilot plant studies.

In case of hydrocarbon reforming, methane slip is dependent of pressure and temperature. It may be defined in an empirical form in terms of pressure and temperature.

$$C_{CH_4} = X\,p^a\,T^b + Y$$

where, C_{CH_4} = Concentration of methane (mole %) at exit

p = Operating absolute pressure

T = Operating absolute temperature

X, a, b and Y are constants

Alternately, degree of approach is used to define completion of reforming reaction. Based on actual methane slip and pressure, equilibrium temperature is found from the standard chart. Difference between actual operating temperature and equilibrium temperature (read from the graph) is defined as the approach.

While designing a chemical reaction system, degree of conversion is used for a well-defined reaction, if known. In case of ammonia manufacture, conversion per pass is commonly expressed as the reaction is well defined. For all other systems, desired degree of completion is defined and the system is designed accordingly.

10.6 MIXING FOR DIFFERENT TYPE OF REACTION SYSTEMS[6]

For the better mixing or better mass transfer rate, an agitator, a circulating pump, etc., are used as mixing tools. Some new techniques are also available for the same. Selection of agitator for the given reaction system depends on the viscosity of reaction mixture, homogeneity of the reaction system, desired duty, etc.

Following are the guidelines for the selection of agitator (or other technique) for the different types of reaction systems:

1. **Homogeneous Liquid Phase Reaction System:** If the viscosity of homogeneous liquid phase system is less than 500 mPa · s (or cP) then flat blade turbine stirrer (with four or six flat blades on disc as shown in Fig. 10.2(v)) or Pfaudler impeller (as shown in Fig. 10.2(ix)) can be selected. They are used with side baffles. These agitators create the currents mainly in radial and tangential directions. Use of side baffles are required to avoid the vortex formation. Pitched blade turbine with 45° blades and with side baffles can also be selected for this application. This agitator creates the currents in axial, tangential and radial directions, it consumes less power but provides lesser heat transfer coefficient for jacketed heat transfer as compared to flat blade turbine agitator. The latter creates more currents towards the heat transfer wall.

   If the viscosity is in the range of 500 to 5000 mPa · s, cross beam, grid and blade stirrers (Ref. Fig. 10.2(xiv), (xvii) and (xviii)) are suitable for homogeneous liquids. For the lower viscosity (nearer to 500 mPa · s), side baffles are used with these types of agitators. For the higher viscosity (nearer to 5000 mPa · s), side baffles are not required.

   For highly viscous liquid (viscosity, $\mu > 5000$ mPa · s), anchor stirrer (Ref. Fig. 10.2(xi)) is selected. Normally, the diameter of anchor agitator is 90% or more of the inside diameter of tank. It rotates with keeping a close clearance with inside surface of shell. It removes the sticky material from the heat transfer surface and thereby improves heat transfer coefficient. But this agitator provides poor mixing. Anchor agitator can be used in conjunction with a higher speed paddle or other agitator to improve the mixing. Other agitator is usually turning in the opposite direction. Miscible liquids are often simply mixed in pipelines by using a static mixer. The deflection elements in static mixer divide the stream into two streams and turn each through 180°, so after passing through N elements the stream has been blended 2^N times.

2. **Gas-Liquid Reaction:**
   (a) Gas itself creates the axial currents. Hence, for the gas-liquid reaction system suitable agitator is that which creates the current in tangential and radial directions. Hence, conventional flat blade turbine agitator (as shown in Fig. 10.2(ii) and 10.2(v)) is used for such applications in which separate sparger is provided.
   (b) Gas induction-type hollow agitator is new innovation for this application. Special type of impeller (as shown in Fig. 10.1(k) and Fig. 10.17) is attached with hollow shaft. In the upper part of the hollow shaft, windows are provided for gas suction. Gas enters from these windows and discharges through the lowest part of the impeller. The agitator operates on the principle of water jet ejector. The suction so generated blows the stirrer edges during the rotation and hence, gas enters through windows and discharges from the bottom of impeller to liquid pool. A specially designed impeller vigorously disperses the gas bubbles and creates a mixture akin to a boiling liquid. Gas bubbles react with liquid as they rise. Unreacted gas is reinduced into the liquid through the windows. Recirculation of gas is important because bubbling of gas only once through the liquid does not use it completely. It

offers the following advantages:

(i) It provides vigorous gas-liquid mixing.

(ii) It substantially increases gas-liquid interfacial area of contact and enhances gas-liquid mass transfer rate.

(iii) It reduces reaction time considerably for the gas-liquid reaction in which overall reaction rate is governed by rate of mass transfer.

(iv) It provides very high vessel side (i.e., inside) coefficient which approaches a boiling coefficient.

(v) It is also the best choice for the gas-liquid reaction with suspended solid catalyst. For example, hydrogenation in presence of suspended Reiny Ni catalyst.

It is used for hydrogenation, alkylation, ozonisation, oxidation, amination, etc., reactions.

(c) Jet reactor is a new design of reactor which can be used to achieve the excellent gas-liquid mixing. As shown in Fig. 10.1(i), jet reactor consists of a reaction autoclave, a circulation pump, an external heat exchanger and a venturi-type ejector. Jet reactors are available in the capacities from 0.02 m^3 to 100 m^3, operating pressure up to 200 bar, operating temperature up to 350°C and in a variety of materials of constructions like stainless steel, Hastelloy, Monel, etc. This reactor can be used for the viscosity of reaction mixture up to 500 mPa · s and for gas-liquid reaction with suspended solid particles (solid catatyst load should be less than 10% by mass).

Following are the advantages of jet reactor over agitated vessel-type reactor:

(i) Length-to-diameter ratio of jet reactor is higher than the same of agitated vessel. Hence, jet reactor requires less cost particularly for high pressure reactions.

(ii) The external heat exchanger (instead of internal coil or jacket) can be built as large as needed and is not limited by the reactor geometry. Sufficient heat transfer area could be made available for accurate temperature control even if the reactor is operated with reduced working volume.

(iii) The maximum power input per unit volume is often a limiting factor, especially for large reactors with an agitator. Since there is no agitator in the jet reactor, this limitation does not exist.

The circulation pump can provide very high power per m^3 of working volume if it is required to achieve the desired mass transfer rate.

(iv) The down flow jet ejector forms fine gas bubbles in the liquid and creates high mass transfer rates.

Jet reactor is used for hydrogenation, alkylation, carbonylation oxidation, halogenation, amination, phosgenation, etc., reactions. Jet reactor is a type of loop reactor.

(d) Another loop reactor is a monolith catalytic reactor [Fig. 10.1(j)]. Monolithic structure of the base carrier is impregnated with a nobel metal (such as platinum, palladium, etc.). Liquid is circulated through a pump and passed through the catalyst with induced gas. Originally developed for emission

control from auto vehicles (for catalytic oxidation of carbon monoxide), monolithic structure is found equally effective for chemical reactors. Monolithic structure [Fig. 10.14(g)] provides large surface area and hence, low concentration of catalyst (0.5 to 1%) on the structure is sufficient for accelerating the reaction. The design is claimed to be highly effective in hydrogenating a nitro compound to an amine.

(e) Recent innovation for gas-liquid reaction is to convert the heterogeneous gas-liquid reaction into single homogeneous phase; supercritical phase, by changing the operating conditions. Foreign substance (such as carbon dioxide, propane, etc.) is added to the reaction system to get the homogeneous supercritical phase.

Examples

(i) Hydrogenation of oleochemicals at supercritical single phase conditions: In this case, propane is added to reaction system. Supercritical propane dissolves both hydrogen and oil and creates the single homogeneous supercritical phase. This supercritical phase is contacted with catalyst and it gives very high overall rate of reaction and higher selectivity. To create the necessary single phase (supercritical phase) conditions, operating pressure is kept near 150 bar, temperature of 280°C and propane is added to the extent of 5 to 6 times product mass. Hydrogenated product will have less of *trans*-isomer and less free fatty acids.

(ii) Oxidation of waste water at supercritical phase conditions[7]*:* It is carried out at above 374°C and 22.1 MPa a pressure. It is carried out between supercritical wastewater and oxygen. Enhanced solubility of oxygen in supercritical water eliminates mass transfer resistance and provides very high mass transfer rate. Residence time required in reactor is only 60 seconds for 99.98% COD reduction.

3. **For gas-liquid reaction with suspended solid particles:** Simple loop reactor or jet reactor are also valid for gas-liquid reaction with suspended solid particles, except with static mixer. Oxidation of acetaldehyde to acetic acid in presence of potassium permanganate can be classified in this category.
4. **For liquid-liquid reaction:** Such type of reaction mixture is a mixture of two immiscible liquids, e.g., nitration of benzene using mixed acid, reaction of phenol with an alkali, sulphonation of benzene with oleum, etc.

For better mixing of two immiscible liquids, axial currents are more important. If the viscosity of liquid-liquid mixture is less than 500 mPa · s, then pitched blade turbine or propeller are more effective. For smaller vessel propeller (Fig. 10.2(i)) and for larger vessel pitched blade turbine (Fig. 10.2(iv)) is suitable. For higher viscosity ($\mu > 400$ mPa · s), agitator which contains cross beam with inclined blades (Fig. 10.2(xiv)), can be selected for such a liquid-liquid reaction system.

For nitration reaction, a loop reactor with a static mixer can be used. Mixed acid can be fed into the static mixer (at a controlled rate). Heat exchanger, located on downstream of the static mixer can remove exothermic heat of reaction effectively. It is claimed that in such a system chances of side reaction (such as dinitro formation) are minimal.

5. **For liquid-solid reaction:** In this case, entire surface of solid particles must be accessible to the liquid. Hence, all solid particles must be suspended in the liquid. For the suspension of solids, axial currents are important. Hence, for the lower viscosity of slurry, 45° pitched blade turbine or propeller can be selected. For more viscous slurry (500 mPa · s < μ < 5000 mPa · s) cross beam with inclined blades and for highly viscous slurry, helical ribbon (Fig. 10.2(xvi)) can be selected. If solids are sticky and heat transfer by jacket is important, then anchor agitator in conjunction with off-centre propeller or pitched blade turbine is preferred.

Following guidelines can be followed in scale up of agitated vessel type batch reactor.

1. Large scale and lab scale batch reactors should be geometrically similar. Type of agitators and Shape factors of both reactors should be same. h/D, D_a/D, D_a/E, J/D, etc. of both reactors should be same. Where h, D, D_a, E and J are height of liquid in vessel, diameter of vessel, diameter of agitator, clearance between agitator and bottom surface of vessel and width of jacket, respectively.
2. For homogeneous liquid phase reaction tip velocities of both reactors should be same or $n_1D_{a1} = n_2D_{a2}$. Where n_1 and n_2 are speeds of smaller and larger sized agitators in rpm and D_{a1} and are D_{a2} diameters of smaller and larger sized agitators. For gas-liquid reactions and for liquid-liquid reactors, power required per unit volume should be same in both reactors or $n_1D_{a1}^{0.66} = n_2D_{a2}^{0.66}$
   For slurry reactions, for both reactors $n_1D_{a1}^{0.75} = n_2D_{a2}^{0.75}$

Example 10.5 Hydrogenation of edible oil is carried out to produce 'Vanaspati' (hydrogenated fat) in presence of nickel catalyst in a batch reactor. In the standard age-old process, edible oil is hydrogenated at about 2 bar g and 140–165°C in 8 to 10 hours (excluding heating/cooling). During this period, iodine value of the mass is reduced, say from 128 to 68. Final mass has a melting (slip) point of 39°C. The batch reactor [Fig. 10.1(a)] has a jacket for heating the initial charge with circulating hot oil. Cooling requirements are met by passing cooling water in internal coils.

In a newly developed Jet Reactor [Fig. 10.1(i)], it is planned to complete the reaction in five hours by improving mass transfer in the reactor and cooling the mass in external heat exchanger, thereby maintaining near isothermal conditions. Figure 10.6 shows the suggested scheme.

Soybean oil, having iodine value (IV) of 128 is to be hydrogenated in the jet reactor at 5 bar g and 165°C. Initially the charge is heated from 30°C to 140°C with the circulating hot oil in external heat exchanger. Hydrogen is introduced in hot soybean oil and pressure is maintained in the reactor at 5 bar g. Reaction is exothermic and the temperature of mass increases. Cold oil flow in the external heat exchanger controls the temperature at 165°C as per the requirement. IV reduction is desired up to 68 when the reaction is considered over. Thereafter, hydrogenated mass is cooled to 60°C in about 1.5 h before it is discharged to filter. 150 kg spent nickel catalyst is charged with soybean oil while fresh 5 to 10 kg nickel catalyst is charged at intervals in the reactor under pressure. A bleed is maintained from the system to purge out water vapour and non-condensables. Design the jet reactor for the following duty:

(i) Charge = 10 t soybean oil with 128 IV
(ii) Average molar mass of soybean oil = 278.0
(iii) Average chain length of fatty acids = 17.78

Fig. 10.6 *Jet Reactor System for Hydrogenation of Edible Oil*

(iv) Product specifications: 68 IV, 39°C melting point (max.)
Assume linear drop of IV in five hours.
(v) Average exothermic heat of reaction = 7.1 kJ/(kg IV reduction)
(vi) Hydrogen feed rate = 110 to 125 Nm^3/h
Bleed rate = 1 to 2 Nm^3/h
(vii) Thermic fluid or oil is used as both, heating medium in starting of reaction and cooling medium during the reaction.
(viii) Cooling water is available at 2 bar g and 32°C. A rise of 5°C is permitted. Cooling water is used for cooling the oil from 80°C to 70°C in oil cooler (HE-2) of oil cycle.
(ix) Assume the following properties of fluids for the design.

Table 10.8 Average Properties of Edible Oil and Circulating Oil

Property	Soybean oil or Hardened fat	Circulating oil (thermic fluid)
Density, kg/L	0.825	0.71
Specific heat, kJ/(kg · °C)	2.56	2.95
Viscosity, mPa · s	2.0	0.5
Thermal conductivity, W/(m · °C)	0.16	0.1

Solution

Volume of liquid inside the jet reactor, $V_L = \dfrac{10\,000}{0.825} = 12\,121.2 \text{ L} \equiv 12.12 \text{ m}^3$

$$V_L = \frac{\pi}{4} D_i^2 h_L + \text{Inside volume of bottom head}$$

where, D_i = Inside diameter of jet reactor, m
h_L = Height of liquid inside the shell of jet reactor, m
Let $h_L = 1.5\, D_i$
Type of bottom head = Torispherical

Inside volume of torispherical head = $0.084\,672\, D_i^3 + \dfrac{\pi}{4} D_i^2 S_F$

Let $S_F = 1.5 \text{ in} = 0.0381 \text{ m}$

$$12.12 = \frac{\pi}{4} D_i^2 (1.5\, D_i) + 0.084\,672\, D_i^3 + \frac{\pi}{4} \times 0.0381\, D_i^2$$

$$12.12 = 1.26\,28\, D_i^3 + 0.029\,92\, D_i^2$$

$D_i = 2.115$ m
$h_L = 1.5 \times 2.115 = 3.1725$ m

Let total height of shell of reactor, $H = 2 \times 2.115 = 4.23$ m
Design of external heat exchanger:
Type of heat exchanger: BEM-type shell and tube heat exchanger.
Heat duty of HE-1 for cooling period:

$$\phi_C = \text{Average heat of reaction} \times \text{IV reduction} \times \frac{\text{kg of reaction mass}}{\text{Reaction time}}$$

$$\phi_C = 7.1 \times (128 - 68) \times \frac{10\,000}{5} = 852\,000 \text{ kJ/h} \equiv 236.67 \text{ kW}$$

Let circulation rate of soybean oil = 71 m^3/h ≡ 58 575 kg/h
This assumption means entire volume gets recirculated in 8.45 min.

$$\dot{m} = 58\,575/3600 = 16.27 \text{ kg/s}, \; C_L = 2.56 \text{ kJ/(kg} \cdot {}^\circ\text{C)}$$

$$\Delta\mathcal{T} = 5.682^\circ\text{C}$$

Hence, in heat exchanger, temperature of circulating stream of reacting mass is to be reduced from 165°C to 159.318°C. It is cooled by thermic fluid entering at 70°C and leaving at 80°C.
Mass flow rate of cooling oil $\dot{m}_o$:

$$\dot{m}_o = \frac{236.67}{2.95\,(80 - 70)} = 8.023 \text{ kg/s} \equiv 28\,883 \text{ kg/h} \equiv 40.68 \text{ m}^3\text{/h}$$

Mean temperature difference:

$$\Delta\mathcal{T}_m = \Delta\mathcal{T}_{lm} \times F_t$$

$$\Delta\mathcal{T}_{lm} = \frac{(165 - 80) - (159.318 - 70)}{\ln\,[(165 - 80)/(159.318 - 70)]}$$

$$= 87.141^\circ\text{C}$$

For 1-1 heat exchanger, $F_t = 1$.
Hence, $\Delta\mathcal{T}_m = 87.141^\circ$C

Allocating reacting oil stream on tube side and thermic fluid (cooling medium) on shell side.

Calculations of h_i: Let tube OD, $d_o = 25.4$ mm

For 16 BWG tube, tube ID, $d_i = 22.098$ mm (Table 11–2 of Ref. 2)
Let tube side velocity, $u_t = 1.5$ m/s

$$u_t = G_t/\rho$$

$$G_t = u_t\rho = 1.5 \times 0.825 \times 10^3 = 1237.5 \text{ kg/(m}^2 \cdot \text{s)}$$

$$G_t = \frac{\dot{m}}{a_t}$$

$$a_t = \frac{\dot{m}}{G_t} = \frac{58\,575/3600}{1237.5} = 0.013\,148 \text{ m}^2$$

$$a_t = \frac{N_t}{N_p} \times \frac{\pi}{4}\, d_i^2$$

where, N_t = Total number of tubes, N_P = number of tube side passes = 1

$$N_t = \frac{0.013\,148}{\frac{\pi}{4} \times (0.022\,098)^2} \approx 34$$

$$Re_t = \frac{d_t G_t}{\mu} = \frac{0.022\,098 \times 1237.5}{2 \times 10^{-3}} = 13\,673$$

$$Pr = \frac{C_p \mu}{k} = \frac{2.56 \times 2 \times 10^{-3} \times 10^3}{0.16} = 32$$

Dittus–Boelter's equation

$$\frac{h_i d_i}{k_f} = 0.023\, Re^{0.8}\, Pr^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.19}$$

$$h_i = 0.023 \times \frac{0.16}{0.022\,098} \times (13\,673)^{0.8} \times (32)^{0.33}$$

$$h_i = 1063.87 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Calculations of h_o:

For 25.4 mm (1 in) OD and 31.75 (1.25 in.) triangular pitch, from Table 6.1 (f), for $N_t = 24$, BEM-type 1.1 heat exchanger:

Shell ID, $D_i = 203$ mm

Let baffle spacing, $B_s = 150$ mm

Type of baffle = 25% cut segmental

Shell side flow area

$$A_s = \frac{(P_t - d_o)}{p_t} \times D_s \times B_s \tag{6.33}$$

$$A_s = \frac{(31.75 - 25.4)}{31.75} \times 0.254 \times 0.15 = 7.62 \times 10^{-3} \text{ m}^2$$

Shell side mass velocity, $G_s = \dfrac{\dot{m}_s}{A_s}$ (6.34)

$$G_s = \frac{8.023}{7.62 \times 10^{-5}} = 1052.89 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_s = \frac{G_s}{\rho_o} = \frac{1052.89}{710} = 1.483 \text{ m/s}$$

Shell side equivalent diameter, d_e:

$$d_e = \frac{1.1}{d_o}\,(P_t^2 - 0.907\, d_o^{\,2}) \tag{6.37}$$

$$= \frac{1.1}{25.4}\,(31.75^2 - 0.907 \times 25.4^2) = 18.3147 \text{ mm}$$

Shell side Reynolds number:

$$Re = \frac{d_e G_s}{\mu_o} = \frac{0.018\,3147 \times 1052.89}{0.5 \times 10^{-3}} = 38\,566.7$$

Prandtl number:

$$Pr = \frac{C_{po}\mu_o}{k_o} = \frac{2.95 \times 0.5 \times 10^{-3} \times 10^3}{0.1} = 14.75$$

$$\frac{h_o d_e}{K_o} = 0.36\, Re^{\,0.55}\, Pr^{\,0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.39}$$

$$h_o = \frac{0.36 \times 0.1}{0.018\,3147} \times (38\,566.7)^{0.55} \times (14.75)^{0.33}$$

$$h_o = 1590.82 \text{ W/(m}^2 \cdot \text{°C)}$$

Calculations of overall heat transfer coefficient, U_o:

Take thermic fluid (oil) side fouling coefficient, $h_{od} = 5000$ W/(m^2 · °C) and soybean oil side fouling coefficient, $h_{id} = 3000$ W/(m^2 · °C) (Ref.: Table 6.9)

Tube material = SS 316

Thermal conductivity of tube material, $k_w = 16.26$ W/(m^2 · °C) (Table 3–322, of Ref. 2)

$$\frac{1}{U_o} = \frac{1}{1590.82} + \frac{1}{5000} + \frac{0.0254 \ln(25.4/22.098)}{2 \times 16.26} + \frac{25.4}{22.098} \times \frac{1}{3000} + \frac{25.4}{22.098} \times \frac{1}{1063.87}$$

$$U_o = 416.5 \text{ W/(m}^2 \cdot \text{°C)}$$

Heat transfer area:

Heat transfer area required, $A_r = \dfrac{\phi_c}{U_o \Delta T_m} = \dfrac{236.67 \times 10^3}{416.5 \times 87.141} = 6.52 \text{ m}^2$

Length of tubes required, $L_r = \dfrac{A_r}{N_t \pi d_o} = \dfrac{6.52}{34 \times \pi \times 0.0254} = 2.403 \text{ m}$

Let tube length $L = 3$ m

Heat transfer area provided, $A = N_t\, \pi\, d_o\, L$

$A = 34 \times \pi \times 0.0254 \times 3 = 8.139 \text{ m}^2$

$$\% \text{ excess heat transfer area} = \left(\frac{8.139 - 6.52}{6.52}\right) \times 100 = 24.83\ \% \text{ (satisfactory)}$$

Tube side pressure drop, Δp_t:

For $Re = 13\,673.14$, $J_f = 0.0045$ (from Fig. (6.13))

$$\Delta p_t = 1\left(8 \times 0.0045\left(\frac{3}{0.022\,098}\right) \times 1 + 2.5\right)\left(\frac{825 \times 1.5^2}{2}\right)$$

$$= 6856.4 \text{ Pa} \equiv 6.856 \text{ kPa (acceptable)}$$

Shell side pressure drop, Δp_s:

For $R_e = 38\,566.73$ and 25% cut segmental baffles

$J_f = 0.04$ (From Fig. 6.15)

$$\Delta p_s = 8 \times 0.04 \times \left(\frac{254}{18.3147}\right)\left(\frac{3}{0.15}\right)\left(\frac{710 \times 1.483^2}{2}\right) = 69\,299 \text{ Pa} = 69.3 \text{ kPa}$$

$\Delta p_s = 69.3$ kPa which is (optimum) pressure drop < $\Delta p_{s\,\max} = 70$ kPa (Ref. Table 6.8)

Resulting data for heat exchanger (HE-1) are presented in TEMA format in Fig. 10.7.

HEAT EXCHANGER SPECIFICATION SHEET
(TEMA FORMAT)

Customer : ABC Vanspati Ltd.			Job No./Reference No.	
Address :			Date :	Rev. : 0
Plant Location :			Item No.: HE-1	
Service of Unit : Soybean Oil Cooler				
Size : 262 mm x 3.6 m Type : BEM Horizontal/Vertical : Vertical				
HTA/Shell : 8.139 m² Shell/Unit : One			Shell Gross Area : 8.139 m²	

PERFORMANCE OF ONE UNIT

Fluid Allocation :		Shell Side		Tube Side	
Fluid Name		Thermic Fluid		Soybean Oil	
Fluid Quantity Total	kg/h	28 883		58 575	
Vapour (In/Out)					
Liquid		28 883	28 883	58 575	58 575
Steam					
Water					
Noncondensables					
Temperature (In/Out)	°C	70	80	165	159.318
Specific Gravity		0.71	0.71	0.825	0.825
Viscosity, Liquid	cP	0.5	0.5	2	2
Molar Mass, Liquid	kg/kmol	278.0			
Molar Mass, Noncondensables	kg/kmol				
Specific Heat	kJ/(kg ·°C)	2.95	2.95	2.56	2.56
Thermal Conductivity	W/(m · °C)	0.1	0.1	0.16	0.16
Latent Heat Vaporisation	kJ/kg				
Inlet Pressure	kPa a				
Velocity	m/s	1.483		1.5	
Pressure Drop, Allow./Cal.	kPa	70/69.3		70/6.856	
Fouling Resistance (min.)	(m²·°C)/W	0.0002		0.000 333	
Heat Exchanged		236.67 kW		MTD (Corrected)	87.141°C
Transfer Rate, Service 333.7	Dirty 416.5	Clean 550.1			W/(m²·°C)

CONSTRUCTION OF ONE SHELL

		Shell Side	Tube Side
Design/Test Pressure	kPa g	600	300
Design Temperature	°C	130	225
Number of Passes/Shell		One	One
Corrosion Allowance	mm	Nil	Nil
Constructions Size & Rating	In	100 mm NB/300 #	125 mm NB/150 #
	Out	100 mm NB/300 #	125 mm NB/150 #
	Intermediate		

Sketch (Bundle/Nozzle Orientation)

Tube No. 34 OD 25.4 mm Thickenss(min./avg.) 1.651 mm Length 3000 mm Pitch			
Tube Type Standard	Material SS 316, SA 312 TP 316		
Shell 254 mm ID 262 mm		Shell Cover (Integral) (Removable)	
Channel or Bonnet SS 316, SA 240 Gr. M		Channel Cover Type : Torrispherical, SA 240 Gr. M	
Tube Sheet - Stationary SS 316, SA 240 Gr. M		Tube Sheet Floating	
Floating Head Cover		Impingement Protection	
Baffles - Cross Segmental Type 25% Cut (Diameter/~~Area~~) Spacing (C/C) 150 mm Inlet 200 mm			
Baffles - Long		Seal Type	
Supports - Tubes	U - Bend	Type	
Bypass Seal Arrangement		Tube to Tube Sheet Joint Expanded Type	
Expansion Joint		Type	
pV^2 - Inlet Nozzle	Bundle Entrance		Bundle Exit
Gaskets - Shell Side PTFE		Tube Side PTFE	
Floating head			
Code Requirements TEMA & ASME Code Sec. VIII, Div. - I		TEMA Class R	
Weight/Shell			
Remarks Use of seamless tubes is recommended.			
Prepared By :		Checked By :	

Fig. 10.7 *Soybean Oil Cooler Specifications*

Heating before starting of reaction:

Let time required for heating the soybean oil from 30°C to 140°C,

$\theta = 2\text{ h} = 3600 \times 2 = 7200\text{ s}$

The same thermic fluid (oil) will be used as heating medium. Let t_1 is the inlet temperature of hot oil (thermic fluid) to heat exchanger in heating period. Both side flow rates (shell side and tube side) for heating period can be kept same as that for the cooling period.

For heating in batch reactor by circulation through external exchanger.

$$\ln\left(\frac{t_1 - t_1'}{t_1 - t_2'}\right) = \frac{\dot{m}C_{LH}}{mC_{LC}}\left(\frac{K_2 - 1}{K_2}\right)\theta \tag{10.14}$$

(Eq. (10–147d) of Ref. 2)

t_1 = temperature of heating medium at inlet, °C

t_1' = temperature of cold fluid at the beginning of heating period, °C

t_2' = temperature of cold fluid at the end of heating period, °C

$\dot{m}$ = flow rate of hot fluid through external heat exchanger, kg/s

θ = time of heating period, s

m = mass of cold fluid in batch reactor, kg

$$K_2 = e^{(UA/\dot{m}C_{LH})} \tag{10.15}$$

U = Overall heat transfer coefficient, W/(m^2 · °C)

A = heat transfer area, m^2

C_{LH}, C_{LC} = specific heats of hot and cold fluids, kJ/(kg · °C)

$t_1' = 30$°C, $t_2' = 140$°C, $\dot{m} = 16.27$ kg/s, $C_{LH} = 2950$ J/(kg · °C)

$A = 8.139$ m^2, $m = 10\,000$ kg, $\theta = 7200$ s

$U = 400$ W/(m^2 · °C) (approximately same as that for the cooling period)

$C_{LC} = 2560$ J/(kg · °C)

Here, flow rates of oils are same as that for the cooling period. However, small change in the value of U can be expected due to the change in the values of thermal properties of both oils with temperature.

$$\frac{UA}{\dot{m}C_{LH}} = \frac{400 \times 8.139}{16.27 \times 2950} = 0.067\,83$$

$$K_2 = e^{(UA/\dot{m}C_{LH})} = 1.07$$

$$\ln\left(\frac{t_1 - 30}{t_1 - 140}\right) = \frac{16.27 \times 2950}{10\,000 \times 2560}\left(\frac{1.07 - 1}{1.07}\right) \times 7200 = 0.8831$$

$$\frac{t_1 - 30}{t_1 - 140} = e^{0.8831} = 2.4184$$

$$t_1 = 217.55\text{°C} \quad \text{(minimum)}$$

Let inlet temperature of hot oil, $t_1 = 230$°C

Outlet temperature of hot oil, $t_2 = 220$°C

Design of cooler (HE-2) of oil cycle:

Type of heat exchanger: BEM type, Fixed tube sheet

Tube side fluid: Cooling water

Shell side fluid: Oil (thermic fluid)
Cooling water inlet temperature = 32°C
Cooling water outlet temperature = 37°C
Heat duty, ϕ = heat duty of external heat exchanger during cooling period = 236.67 kW
Cooling water flow rate,

$$236.67 \times 10^3 = \dot{m} C_L \Delta t = \dot{m} \times (4.1868 \times 10^3)(37 - 32)$$

$$\dot{m} = 11.3055 \text{ kg/s} \equiv 40\,700 \text{ kg/h} \equiv 40.7 \text{ m}^3\text{/h}$$

Mean temperature difference:

$$\Delta T_m = \Delta T_{lm} \times F_t$$

$$\Delta T_{lm} = \frac{(80 - 37) - (70 - 32)}{\ln[(80 - 37)/(70 - 32)]} = 40.4485 \text{ °C}$$

Let number of tube side passes = two

$$R = \frac{t_1 - t_2}{t_2' - t_1'}, \quad S = \frac{t_2 - t_1}{t_1' - t_1'}$$

$$R = \frac{80 - 70}{37 - 32} = 2, \quad S = \frac{37 - 32}{80 - 32} = 0.1042$$

$$F_t = 0.99 \quad \text{(From Fig. 6.11)}$$

$$\Delta T_m = 40.4485 \times 0.99 = 40.044 \text{ °C}$$

Evaluation of tube side heat transfer coefficient, h_i:
Let tube side velocity, u_t = 1.5 m/s
Density of water at 34.5°C = 994.202 kg/m^3

$$\text{Volumetric flow rate of water} = \frac{40\,700./3600}{994.202} = 0.011\,37 \text{ m}^3\text{/s}$$

$$u_t = \frac{0.01137}{a_t} = 1.5 \text{ m/s}$$

$$\text{Hence, tube side flow area, } a_t = 7.58 \times 10^{-3} \text{ m}^2 = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2$$

$N_p = 2$ (selected)

Let $d_o = 19.05$ mm, $d_i = 15.748$ mm

$$7.58 \times 10^{-3} = \frac{N_t}{2} \times \frac{\pi}{4} (0.015\,748)^2$$

$$N_t = 78$$

From Table 6.1 (d), for 25.4 mm triangular pitch, $N_p = 2$, Shell ID = 305 mm

$$Re = \frac{d_i u_t \rho}{\mu} = \frac{0.015\,748 \times 1.5 \times 994.202}{0.73 \times 10^{-3}}$$

$$= 32\,171.3$$

$$Pr = \frac{C_L \mu}{k} = \frac{4.1868 \times 0.73 \times 10^{-3} \times 10^3}{0.628} = 4.867$$

(Viscosity of water, μ = 0.73 cP, Thermal conductivity of water, k = 0.628 W/(m · °C)

$$\frac{h_i d_i}{k} = 0.023\, Re^{0.8}\, Pr^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.19}$$

$$h_i = 0.023 \times \frac{0.628}{0.015\,748} \times 32\,171.3^{0.8} \times 4.867^{0.33}$$

$$= 6240.7 \text{ W/(m}^2 \cdot \text{°C)}$$

Evaluation of shell side heat transfer coefficient, h_o:
Shell side flow area,

$$A_s = \frac{P_t - d_o}{P_t} \times D_s \times B_s \tag{6.33}$$

$$P_t = 25.4 \text{ mm}, \quad d_o = 19.05 \text{ mm}, \quad D_s = 305 \text{ mm}$$

Let $B_s = 125$ mm

$$A_s = \frac{25.4 - 19.05}{25.4} \times 0.305 \times 0.125 = 9.531\,25 \times 10^{-3} \text{ m}^2$$

Shell side mass velocity, $G_s = \dfrac{\dot{m}_o}{A_s}$ (6.34)

$$G_s = \frac{8.023}{9.531\,25 \times 10^{-3}} = 841.757 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_s = (G_s/\rho_o) = (841.757/710) = 1.1856 \text{ m/s} \tag{6.35}$$

Shell side equivalent diameter,

$$d_e = \frac{1.1}{d_o}\,(P_t^2 - 0.907\, d_o^2) \tag{6.37}$$

$$= \frac{1.1}{19.05}\,(25.4^2 - 0.907 \times 19.05^2)$$

$$d_e = 18.25 \text{ mm}$$

$$Re = \frac{d_e G_s}{\mu} = \frac{0.018\,25 \times 841.757}{0.5 \times 10^{-3}} = 30\,724$$

$$Pr = 14.75$$

$$\frac{h_o d_e}{k} = 0.36\, R_e^{0.55}\, Pr^{0.33} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{6.39}$$

$$h_o = \frac{0.36 \times 0.1}{0.01825} \times (30\,724)^{0.55} \times (14.75)^{0.33}$$

$$= 1408.8 \text{ W/(m}^2 \cdot \text{°C)}$$

Evaluation of overall heat transfer coefficient, U_o:
Thermic fluid (oil) side fouling coefficient, $h_{od} = 5000$ W/(m^2 · °C)
Tube material = mild steel

$$k_W = 50 \text{ W/(m} \cdot \text{°C)}$$

$$\frac{1}{U_o} = \frac{1}{1408.8} + \frac{1}{5000} + \frac{0.019\,05 \ln(19.05/15.748)}{2 \times 50}$$

$$+ \frac{19.05}{15.748} \times \frac{1}{5000} + \frac{19.05}{15.748} \times \frac{1}{6240.7}$$

$$U_o = 723.66 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Heat transfer area required,

$$A_r = \frac{\phi}{U_o \Delta T_m} = \frac{236.67 \times 10^3}{723.66 \times 40.044} = 8.167 \text{ m}^2$$

Tube length required, $L_r = \dfrac{8.167}{78 \times \pi \times 0.019\,05} = 1.7495$ m

Let tube length, $L = 2$ m

Heat transfer area, $A = N_t\, \pi\, d_o\, L = 78 \times \pi \times 0.019\,05 \times 2 = 9.336 \text{ m}^2$

% excess heat transfer area = $\dfrac{9.336 - 8.167}{8.167} \times 100 = 14.31\%$ (adequate)

Tube side pressure drop, Δp_t:

For $Re = 32\,171.3$, $J_f = 3.5 \times 10^{-3}$ from Fig. 6.13

$$\Delta p_t = 2 \times \left(8 \times 3.5 \times 10^{-3} \left(\frac{2000}{15.748}\right) \times 1 + 2.5\right)\left(\frac{994.202 \times 1.5^2}{2}\right)$$

$$\Delta p_t = 13\,547 \text{ Pa} \equiv 13.547 \text{ kPa (acceptable)}$$

Shell side pressure drop, ΔP_s:

For $Re = 30\,724$, $J_f = 0.041$ from Fig. 6.15 for 25% baffle cut

$$\Delta p_s = 8 \times 0.041 \left(\frac{305}{18.25}\right)\left(\frac{2000}{125}\right)\frac{710 \times 1.203^2}{2} \times 1$$

$$= 45\,060 \text{ Pa} \equiv 45.06 \text{ kPa (acceptable)}$$

Resulting data for oil cooler (HE-2) of oil cycle are presented in TEMA format as Fig. 10.8.

Notes: (i) Circulation rate of soybean oil is fixed at 71 m^3/h. However, differential pressure (DPIC) is unknown. It will depend on desired hydrogen circulation rate in the reactor. To begin with $\Delta p = 3$ to 4 bar may be fixed and by actual experience this can be optimised. Reactor pressure and differential pressure together will decide discharge pressure and power requirement of the gear pump.

(ii) Jet reactor shown in Fig. 10.6 is basically a slurry reactor in which a small amount of fine nickel catalyst particles are in motion. Such a reactor can also be called three-phase fluidised bed reactor or a suspended bed reactor. Such a reactor has advantages of high capacity to provide good temperature control, adoptability to batch processing and potentially high rate of reaction per unit volume of reactor (depending on the catalyst activity). However, its disadvantages are the residence time distribution close to that in a CSTR, difficulty of removal of fine catalyst particles by filtration and possibly allow more side reactions due to high ratio

HEAT EXCHANGER SPECIFICATION SHEET
(TEMA FORMAT)

Customer : ABC Vanspati Ltd.			Job No./Reference No.	
Address :			Date :	Rev. : 0
Plant Location :			Item No.: HE-2	
Service of Unit : Oil (Thermic Fluid) Cooler				
Size : 315 mm x 2.6 m Type : BEM Horizontal/Vertical : Horizontal				
HTA/Shell : 9.336 m^2 Shell/Unit : One			Shell Gross Area : 9.336 m^2	
PERFORMANCE OF ONE UNIT				
Fluid Allocation :	Shell Side		Tube Side	
Fluid Name	Thermic Fluid		Soybean Oil	
Fluid Quantity Total kg/h	28 883		40 700	
Vapour (In/Out)				
Liquid	28 883	28 883	40 700	40 700
Steam				
Water				
Noncondensables				
Temperature (In/Out) °C	80	70	32	37
Specific Gravity	0.71	0.71	1	1
Viscosity, Liquid cP	0.5	0.5	0.73	0.73
Molar Mass, Liquid kg/kmol	18			
Molar Mass, Noncondensables kg/kmol				
Specific Heat kJ/(kg ·°C)	2.95	2.95	4.1868	4.1868
Thermal Conductivity W/(m · °C)	0.1	0.1	0.628	0.628
Latent Heat Vaporisation kJ/kg				
Inlet Pressure kPa a				
Velocity m/s	1.1856		1.5	
Pressure Drop, Allow./Cal. kPa	70/45.06		70/13.547	
Fouling Resistance (min.) $(m^2 \cdot °C)/W$	0.0002		0.0002	
Heat Exchanged	236.67 kW		MTD (Corrected)	40.044 °C
Transfer Rate, Service 633.06 Dirty 723.66 Clean 1063.91				$W/(m^2 \cdot °C)$

CONSTRUCTION OF ONE SHELL				Sketch (Bundle/Nozzle Orientation)
		Shell Side	Tube Side	
Design/Test Pressure kPa g		600	600	
Design Temperature °C		150	50	
Number of Passes/Shell		One	Two	
Corrosion Allowance mm		2	2	
Constructions Size & Rating	In	100 mm NB/300 #	100 mm NB/150 #	
	Out	100 mm NB/300 #	100 mm NB/150 #	
	Intermediate			

Tube No. 78 OD 19.05 mm Thickenss(min./avg.) 14 mm Length 2000 mm Pitch ☑ □ ◇			
Tube Type Standard	Material CS SA 192		
Shell 305 mm ID 315 mm		Shell Cover (Integral) (Removable)	
Channel or Bonnet CS SA 516 Gr. 70		Channel Cover Type : Torrispherical	
Tube Sheet - Stationary CS SA 516 Gr. 70		Tube Sheet Floating	
Floating Head Cover		Impingement Protection	
Baffles - Cross Segmental Type 25% Cut (Diameter/~~Area~~) Spacing (C/C) 150 mm Inlet 200 mm			
Baffles - Long		Seal Type	
Supports - Tubes	U - Bend	Type	
Bypass Seal Arrangement		Tube to Tube Sheet Joint Expanded Type	
Expansion Joint		Type	
pV^2 - Inlet Nozzle	Bundle Entrance		Bundle Exit
Gaskets - Shell Side PTFE		Tube Side PTFE	
Floating head			
Code Requirements TEMA & ASME Code Sec. VIII, Div. - I		TEMA Class R	
Weight/Shell			
Remarks Thermal expansion analysis of HE is recommended and expansion joint on the shell may be provided, if required.			
Prepared By :		Checked By :	

Fig. 10.8 *Thermic Fluid Cooler Specifications*

of liquid to solid. For example, during hydrogenation of edible oil by the slurry process, hydrogenated fat might contain high trans fatty acids (due to isomerisation) although the edible oil may hardly contain any trans fatty acid. At times, cost of filtration system may be substantially high. In contrast, fixed bed catalyst reactor results in less side reactions and freedom from difficulty of filtration.

10.7 HEAT TRANSFER IN BATCH REACTORS

In earlier Examples 10.1 and 10.5, diameter and height of batch reactors were fixed by calculations. However, heat transfer in batch reactors is effected in many ways.

If jacket side fluid is cooling water, chilled water, brine, hot oil, etc., or if convective heat transfer through the jacket is required then spiral baffled jacket is preferred. Use of spirals in the plain jacket decreases flow area considerably, increases velocity and *Re* of jacket side fluid and consequently increases jacket side heat transfer coefficient considerably. In this case, flow area for jacket side fluid is area between two successive metal strips which forms the spiral. (Refer Fig. 10.9(c)). Spirals also act as stiffeners and decrease thickness required by the shell to withstand external pressure and thereby decrease the conductive resistance of the shell.

For the exothermic reaction (e.g., chlorination of acetic acid) saturated steam is fed into the jacket to initiate the reaction but then after cooling water is circulated through the same jacket to remove the heat of reaction. For such a case plain jacket is selected as it is preferred for saturated steam. Plain jacket avoids the accumulation of condensate over heat transfer surface. For cooling water circulation or hot oil circulation spiral jacket, channel jacket, limpet (half) coil, etc., provide considerably higher heat transfer coefficient as compared to plain jacket. Refer to Fig. 10.9 for common arrangement of jackets, limpet coil, and internal coils.

(a) Conventional Jacket (b) Conventional Jacket

(c) Spiral Baffled Jacket

(d) Jacket with Limpet Coil

(e) Internal Helical Coil

(f) Vessel with Helical Coils as Baffles

Fig. 10.9 *Common Arrangements of Heat Transfer Jackets and Internal Coils*

10.8 BUBBLE COLUMN REACTOR[8, 9]

In a bubble column reactor (Fig. 10.10), gas is dispersed in liquid phase at the bottom of column by a suitable distributor. In most of the cases this distributor is a sparger. Bubble column can be operated in a semibatch, countercurrent or cocurrent manner. Bubble columns are used as reactors, absorbers, and strippers.

Bubble column reactor is preferred for slow or very slow gas–liquid reactions. As overall rate of this reaction is governed by rate of chemical reaction, the rate of consumption of limiting reactant in chemical reaction ($-dN_A/d\theta$) is directly proportional to V_l (volume of liquid phase inside the reactor or liquid holdup). Rate of consumption of limiting reactant in reaction ($-dN_A/d\theta$), in case of slow or very slow reaction, does not depend on interfacial surface area of contact between gas and liquid phase.

Fig. 10.10 *Bubble Column Reactor*

Bubble column reactor does not use any moving part like agitator. Construction of this reactor is simple and inexpensive.

10.8.1 Various Factors Affecting the Performance of Bubble Column Reactor

1. Superficial gas velocity: Increase in superficial gas velocity increases gas holdup, the effective interfacial area and the overall mass transfer rate. Increase in superficial gas velocity decreases the size of bubbles hence, increases the interfacial area of contact between gas and liquid. Effect of superficial gas velocity is negligible on mass transfer coefficient (K_L) but significant on mass transfer area (a). For chemical reaction controlled gas–liquid reaction (slow or very slow gas–liquid reaction) also, certain minimum K_La must be achieved to overcome the effect of mass transfer. It is recommended to keep superficial velocity of gas less than or equal to 10 m/s.

2. Properties of gas phase and liquid phase: Physical properties of gas phase have practically no effect on the performance of the column. However, the physical properties of liquid phase like surface tension, viscosity, etc., have a profound effect on the performance of column. Increase in the viscosity of liquid phase or decrease in the surface tension increases the effective interfacial area and hence increases mass transfer rate. Presence of electrolyte in the liquid phase effects greatly the performance. The electrolyte solution gives smaller bubble size and consequently higher effective interfacial area and higher rate of mass transfer.

3. Back mixing: Based on a study, it is found that gas flow in bubble column operates in plug flow manner without any back mixing while considerable amount of back mixing in the liquid phase is observed.

Back mixing in liquid phase decreases the concentration of liquid reactant and hence the rate of chemical reaction. Consequently, it decreases the overall rate of reaction. Use of packings, trays or baffles reduces liquid back mixing and thereby reduces dilution of reactants by products. Hence, they provide the higher concentration of reactant and hence the higher rate of chemical reaction; $-r_A = (-1/V_l)\,(-dN_A/d\theta)$. But they decrease the liquid hold-up (V_l). Hence, combined effect on rate of consumption of limiting reactant in reaction ($-dN_A/d\theta$) must be checked before using any internal in the bubble column. In case of highly exothermic gas–liquid reaction, back mixing in liquid phase is desirable to control the exothermicity of reaction or to control the temperature of reaction. In such a case, placing packings to improve the overall rate makes the temperature control very difficult because it increases the heat duty required and decreases heat transfer coefficient.

4. Mode of operation: For very slow gas–liquid reaction, semi-batch operation is preferred in which liquid is charged to the reactor in batchwise manner while gas is continuously passed through the column. For moderately fast reaction, continuous operation is preferred. Both countercurrent and cocurrent operation is used. But cocurrent is more common. In countercurrent contact, velocities of gas and liquid through the tower are limited by flooding conditions while cocurrent contact permits the higher velocities of gas and liquid through the tower compared to countercurrent contact. Also, concentration of reactants, rate of chemical reaction and overall rate of reaction do not depend on the mode of operation (whether it is cocurrent or countercurrent) becuase bubble column reactors are ideal mixed flow reactors.

If superficial liquid velocity is greater than 30 cm/s and superficial gas velocity is less than 1 to 3 cm/s then it is better to use cocurrent down flow column. (Ref. 8)

5. Gas expansion and shrinkage: Inside the bubble column, there may be a dramatic increase in superficial velocity of gas from gas inlet to outlet and is called *gas expansion*. Decrease in superficial velocity of gas from inlet to outlet is called *gas shrinkage*.

Example of gas expansion:

Chlorination of benzene gives chlorobenzene and hydrogen chloride gas. In this case theoretically total number of moles of gas phase remains constant and hence, flow rate of gas and superficial velocity of gas remain unchanged. But actually this reaction is exothermic, resulting in vaporisation of organic liquids. Hydrogen chloride and unconverted chlorine, leaving from the top, are saturated with organic vapours at the

outlet conditions. In addition to that hydrostatic head of liquid decreases in upper part of column. Because of these combined effects, there is a considerable increase in superficial velocity of gas from inlet to outlet.

Example of gas shrinkage:
Alkylation of benzene with ethylene in presence of $AlCl_3$ (aluminium chloride) solution as catalyst. Because of the consumption of gas (ethylene) in the reaction, there is a substantial decrease in superficial velocity of gas in upper part of column.

Since superficial gas velocity affects overall reaction rate, mass transfer rate and heat transfer rate. This factor is important and should be carefully considered in designing of bubble column reactor.

6. Pulsation: The performance of bubble column can be improved by pulsations. For very low superficial velocity of gas (0.8 to 2.4 cm/s) the value of $K_L a$ can be increased by factor as much as 3 by pulsation.

7. Addition of packing or packed bubble column: Adding packing reduces axial mixing of liquid. Hence, packed bubble column is used where liquid back mixing is undesirable. For gas–liquid reaction in which substantial gas shrinkage is taking place in the reactor due to reaction, this results in very low superficial gas velocity which in turn results in poor rate of mass transfer in the upper part of reactor. Hence, in such a case placing of packings in upper part is beneficial. Packing increases effective interfacial area and gas hold-up to an extent. Hence, it provides higher rate of mass transfer. Packed bubble column is not used if fine solid particles are present in the system as catalyst or as reactant. Packing is also not preferred for highly exothermic or endothermic reaction as packed tower provides poor heat transfer coefficient.

10.8.2 Industrial Examples of Bubble Column Reactor

1. Production of protein from methanol is carried out in a fermentor which is a bubble column reactor. Largest size of bubble column reactor having capacity of 3000 m^3 is used for this application (Ref. 3).
2. Absorption of carbon dioxide in ammoniated brine for the manufacture of soda ash.
3. Liquid phase air oxidation of a variety of organic compounds. For example, (a) Air oxidation of acetaldehyde in presence of fine $KMnO_4$ particles as catalyst, and (b) Air oxidation of *p*–nitrotoluene sulphonic acid.
4. Air oxidation of black liquor containing Na_2S (sodium sulphide) in pulp (in a paper mill)
5. Air oxidation of ammonium sulphide $[(NH_4)_2S]$
6. Liquid phase chlorination of variety of organic compounds. For example, (a) Chlorination of benzene to chlorobenzene, and (b) Chlorination of acetic acid to monochloroacetic acid, etc.
7. (a) Carbonylation of methanol to acetic acid.
   (b) Carbonylation of ethanol to propionic acid.
8. Liquid phase oxychlorination of ethylene for the production of vinyl chloride.
9. Hydration of propylene with sulphuric acid for the production of vinyl chloride.
10. Reaction between ethylene (C_2H_4) and liquid hydrogen fluoride for the manufacture of difluoroethane.

10.8.3 Advantages and Disadvantages of Bubble Column Reactor Over Stirred Tank Reactor (Agitated Vessel-type Reactor)

Advantages

1. Sealing problem is negligible in bubble column while the same is severe in agitated vessel-type reactor. Sealing of the shaft of an agitator for high pressure reactor is not only an initial design problem but also a continuing maintenance problem. This problem is very important for highly toxic, high pressure reaction system, e.g., ozonation reaction. Above 40 atm operating pressure, agitated vessel-type reactor is not recommended for use.
2. Bubble column reactor provides more liquid hold-up than agitated vessel-type reactor. Hence, it requires less volume for chemical reaction controlled gas-liquid reaction (for slow and very slow gas-liquid reaction).
3. Bubble column reactor requires less maintenance as compared to agitated vessel-type reactor.
4. Bubble column reactor requires less power consumption. Because of the agitator, power consumption is higher with agitated vessel-type reactor.
5. It requires lesser floor space.

Disadvantages

1. Agitated vessel-type reactor provides higher mass transfer coefficient. Hence, for mass transfer controlled gas-liquid reaction, agitated vessel-type reactor requires lesser volume for the given extent of reaction. It may require lesser fixed capital investment than bubble column reactor for such applications.
2. Agitated vessel-type reactor provides higher heat transfer coefficient compared to bubble column reactor. Hence, temperature control is easier with agitated vessel-type reactor than the same for bubble column reactor.
3. Higher interfacial area is obtained with agitated vessel-type reactor. Agitator produces bubbles in smaller sizes and thereby increases interfacial area of contact. Higher interfacial area provides higher mass transfer rate.
4. Bubble column reactor requires more height.

10.8.4 Criteria of Selection for Different Types of Gas–Liquid Reactors

Table 10.9 Typical Ratios for Gas-Liquid Reactors (Ref. 1)

Type of reactor	S/V_l	S/V_R	V_l/V_R	V_l/V_{film}
Packed column	1200	100	0.08	10 to 100
Tray tower	1000	150	0.15	40 to 100
Agitated vessel	200	200	0.9	150 to 800
Bubble column	20	20	0.98	4000 to 10 000

where, S = Interfacial surface area, m^2
V_l = Volume of liquid, m^3
V_R = Volume of reactor, m^3

V_{film} = Volume of liquid film, m^3

Overall rate of any gas-liquid reaction is a function of rate of chemical reaction and rate of mass transfer.

For very fast reaction overall rate of reaction is governed by rate of mass transfer, i.e., rate of chemical reaction >> rate of mass transfer, or

Overall rate of reaction ≈ Rate of mass transfer.

To get the higher rate of mass transfer, packed tower is the best choice. Packed tower provides higher mass transfer coefficient as well as higher interfacial area of contact. But packed bed provides poor heat transfer coefficient.

For slow and very slow gas liquid reaction overall rate is governed by rate of chemical reaction. Rate of consumption of reactant in a chemical reaction does not depend on interfacial area of contact (S), but it depends on liquid hold-up

$$V_l\left(\frac{-dN_A}{d\theta}\right) = V_l k\, f(C) \tag{10.16}$$

Bubble column reactor provides maximum liquid hold-up. V_l/V_R ratio is maximum for bubble column reactor while the same is very low for packed bed reactor. Bubble column reactor provides low heat transfer coefficient.

For the gas-liquid reactions having intermediate rates, overall rate of reaction depends on both; rate of chemical reaction and rate of mass transfer. Hence, for these reactions agitated vessel-type reactor is the best choice because it provides higher S/V_R ratio and higher V_l/V_R ratio. It provides higher heat transfer coefficient but it consumes more power. For high pressure gas-liquid reaction if gaseous reactant is toxic then bubble column reactor is preferred against agitated vessel-type reactor because of the sealing problem. For example, carbonylation of methanol gives acetic acid. It is an intermediate gas-liquid reaction. Agitated vessel-type reactor is the best choice. But, operating pressure of reactor is 50 atm and carbon monoxide is a toxic gas. So, to avoid the use of agitator shaft sealing, for the same reaction, bubble column reactor is preferred.

For highly exothermic gas-liquid reaction, loop reactor or jet reactor (with recirculation, recirculating pump and external heat exchanger) can be considered. Loop reactor provides higher heat transfer coefficient and hence better temperature control than bubble column reactor. But, it consumes more power. For hydrogenation of edible and non-edible oils, loop reactor or jet reactor or gas induced agitated reactor is found to be an excellent choice as it gives desired product pattern in less time than an agitated-type batch reactor with sparger mechanism.

10.8.5 Process Design of Bubble Column Reactor

It can be divided in the following steps:

1. Find the working volume of reactor. Working volume of reactor means volume of liquid in the reactor in running condition which also includes gas hold-up.

   Bubble column reactors are selected for slow or very slow gas-liquid reaction for which overall rate of reaction is totally controlled by the rate of chemical reaction. Hence, with most of the bubble column reactors (bubble column reactors are used for different applications), it is very easy to overcome the effect of mass transfer. Superficial velocity of gas in bubble column reactor

is fixed in such a way that effect of $K_L a$ or mass transfer is eliminated. If the superficial velocity of the gas is fixed above the limiting value S_{gm} then overall rate of gas-liquid reaction is equal to the rate of chemical reaction. S_{gm} is the minimum superficial velocity of gas required to overcome the effect of mass transfer.

For example, for the reaction:

A(g) + b B(l) → Product

If $S_g >> S_{gm}$

$$\text{Overall rate} = -r_{Al} = \frac{1}{V_l}\frac{dN_A}{d\theta} = k\, C_A\, C_B \tag{10.17}$$

In bubble column reactor, liquid is flowing in mixed flow manner. Hence, composition of reactants in liquid phase is almost uniform throughout and working volume of reactor can be determined by using the performance equation of ideal mixed flow reactor.

$$\frac{V_l}{F_{Ao}} = \frac{X_A}{-r_{Al}} \tag{10.12}$$

If for the given gas-liquid reaction it is not possible to overcome the effect of mass transfer, then form of overall rate of gas-liquid reaction must be developed. It can be developed by laboratory scale and pilot plant scale experiments. Different forms of rate equation are assumed and then verified by experimental data. In such a case overall rate is the function of both mass transfer coefficient ($K_L a$) and reaction rate constant k.

For example, for the gas-liquid reaction

A(g) + B(l) → Product,

following form of rate equation can be assumed for verification:

$$-r_A = \frac{1}{V_l}\frac{dN_A}{d\theta} = \frac{p_A}{\left[\dfrac{H_A}{K_L a} + \dfrac{H_A}{kC_B}\right]} = \frac{C_A}{\left[\dfrac{1}{K_L a} + \dfrac{1}{kC_B}\right]} \quad \text{(Typical)} \tag{10.18}$$

where, $K_L a$ = Mass transfer coefficient

a = Interfacial area per unit volume

k = Reaction rate constant

H_A = Henry's law constant

p_A = Partial pressure of reactant A in gas phase

C_A = Concentration of reactant A in liquid phase

In such a case also, since in bubble column reactor liquid phase is flowing in mixed flow manner, working volume V_l can be determined by the following equation:

$$\frac{V_l}{F_{Ao}} = \frac{X_A}{-r_A} \tag{10.12}$$

where, $-r_A$ = Overall rate of reaction = $f(K_L a, k)$

2. Find or fix the value of superficial velocity of gas. Superficial velocity of gas in bubble column reactor should be such that overall rate of reaction becomes

independent of mass transfer coefficient $K_L a$. Minimum superficial velocity of gas required to overcome the effect of mass transfer should be determined by actual experiment in small scale (labscale or pilot plant reactor). Many different correlations are available which relate the superficial velocity of gas and mass transfer coefficients; $K_L a$. However, they are not reliable and change from system to system. If it is not possible to overcome the effect of mass transfer, then correlation of $K_L a$ must be developed in small scale reactor for the given system and the same can be used to determine the diameter of a commercial reactor. In scale-up, influence of wall effect (which is significant in small scale reactor while it is negligible in commercial scale reactor) must be considered.

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{S_g} \tag{10.19}$$

where, q_v = Volumetric flow rate of gas, m^3/s
D_i = Inside diameter of reactor, m
S_g = Superficial velocity of gas, m/s

Based on the value of D_i, height of reactor H, can be determined.

3. Calculate the heat transfer area required. Heat duty required for bubble column reactor can be determined from the heat of reaction ΔH_r at the reaction temperature.

$$\phi_t = \Delta H_r \text{ kJ/mol} \times \frac{1000 \text{ mol}}{1 \text{ kmol}} \times \frac{\text{kmol}}{\text{h}} \text{ of limiting reactant consumed} \pm \phi'$$

where, ϕ' = Heat utilised for other purpose

$$h_i = 21\,766.5\ S_g^{0.22} \left(\frac{Pr_w}{Pr_L}\right)^{1/2} \tag{10.20}$$

where, h_i = Reacting fluid side heat transfer coefficient, $W/(m^2 \cdot C)$
S_g = Superficial velocity of gas, m/s
Pr_w = Prandtl number of water at room temperature
Pr_L = Prandtl number of liquid phase of reactor at reaction conditions.

4. Sparger design is not important if superficial gas velocity is more than or equal to 10 cm/s.

Example 10.6 In the continuous process for the manufacturing of monochloroacetic acid (MCA) conversion of acetic acid is restricted to 50% to avoid the formation of dichloroacetic acid. Reaction is carried out in a bubble column reactor.

Determine the following:

(a) Working volume of reactor
(b) Diameter of reactor
(c) Height of liquid inside the reactor during reaction
(d) Heat duty of overhead condenser in which exist gas-vapour mixture is cooled down to 40°C by cooling with water
(e) Heat transfer area required for bubble column reactor

Data

(i) Reaction: $CH_3COOH(l) + Cl_2(g) \xrightarrow[100°C]{} CH_2ClCOOH(l) + HCl(g)$

(ii) Heat of reaction at reaction temperature, i.e., at 100°C

$\Delta H_r = -87.92$ kJ/mol (Exothermic)

(iii) Chlorine supply rate is 20% higher than that consumed in reaction.

(iv) Cooling water is available in plant at 32°C.

(v) Mass transfer coefficient data

$S_g = 1$ to 30 cm/s $\quad K_L a = 0.25 \times 10^{-2}$ to 0.4 s^{-1}

(vi) Rate of chemical reaction

$$-r_A = \frac{1}{V_l}\frac{dN_A}{d\theta} = kC_A, \quad k = 2.777 \times 10^{-5}\ s^{-1}$$

Density of acetic acid, $\rho = 1048$ kg/m^3

(vii) Production rate of monochloroacetic acid = 1 t/h

(viii) Operating pressure in reactor = 0.1 atm g

Solution

(a) Working volume of reactor:

Bubble column reactor can be assumed as ideal mixed flow reactor.

$$\frac{V_1}{F_{Ao}} = \frac{X_A}{-r_A} \tag{10.12}$$

$$\frac{V_1}{F_{Ao}} = \frac{X_A}{kC_A} = \frac{X_A}{kC_{Ao}(1 - X_A)} = \frac{1}{kC_{Ao}} \times \frac{0.5}{(1 - 0.5)}$$

$$V_l = \left(\frac{F_{Ao}}{C_{Ao}}\right) \cdot \frac{1}{k} = \frac{V_o}{k}$$

where, V_o = Volumetric flow rate of acetic acid at inlet

Stoichiometric equation:

	$CH_3COOH(l)$	+	$Cl_2(g)$	=	$CH_2ClCOOH(l)$	+	$HCl(g)$
Molar mass (M)	60		71		94.5		36.5

Conversion of acetic acid is restricted to 50% to avoid the DCA formation.

Acetic acid required to produce 1 t/h of MCA:

$$\dot{m}_{AA} = 1000 \times \frac{60}{94.5} \times \frac{1}{0.5}$$

$$= 1269.84 \text{ kg/h}$$

$$V_o = \frac{1269.84}{1048} = 1.2117 \text{ m}^3/\text{h}$$

$$V_o = 3.3658 \times 10^{-4} \text{ m}^3/\text{s}$$

$$V_l = \frac{V_o}{k} = \frac{3.3658 \times 10^{-4}}{2.777 \times 10^{-5}} = 12.12 \text{ m}^3$$

Working volume of reactor, $V_l = 12.12$ m^3 Ans. (a)

(b) For superficial velocity of gas

$$S_g \in \{1 \text{ cm/s}, \ldots, 30 \text{ cm/s}\}, \quad K_L a >> k \quad \text{(as given in data)}$$

Let $S_g = 10$ cm/s

Overall rate of reaction = Rate of chemical reaction

Mass flow rate of chlorine at inlet

$$\dot{m}_{Cl_2} = 1.2 \times \frac{71}{94.5} \times 1000$$

$$\dot{m}_{Cl_2} = 901.59 \text{ kg/h}$$

Density of chlorine gas

$$\rho_{Cl_2} = \frac{pM}{RT} = \frac{1.1 \times 71}{(273+100)} \times \frac{273}{1 \times 22.414} = 2.55 \text{ kg/m}^3$$

Volumetric flow rate of chlorine = $\frac{901.59}{2.55} = 353.56 \text{ m}^3\text{/h} \equiv 0.098\,21 \text{ m}^3\text{/s}$

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{S_g} \tag{10.19}$$

$$\frac{\pi}{4} D_i^2 = \frac{0.098\,21}{10 \times \left(\frac{1}{100}\right)} = 0.9821 \text{ m}^2$$

$$D_i = 1.118 \text{ m}$$ Ans. (b)

(c) Let h_L = Height of liquid during reaction

$$V_l = \frac{\pi}{4} D_i^2 h_L$$

$$12.12 = \frac{\pi}{4} (1.118)^2 \times h_L$$

$$h_L = 12.346 \text{ m}$$

Minimum distance required to facilitate gas-liquid separation is recommended to be D_i.

Let H = height of bubble column reactor = $h_L + D_i$

$H \cong 12.346 + 1.118 = 13.464$ m

Let $H = 14$ m Ans. (c)

(d) Let ϕ_c = Heat duty of overhead condenser

In overhead condenser gas-vapour mixture is cooled down to 40°C.

Let $\dot{n}_t$ = Total molar flow rate of gas-vapour mixture leaving the reactor.

Exit gas from the reactor is saturated with liquid vapour at outlet conditions of the reactor. Molar flow rate of chlorine at outlet of reactor.

$$\dot{n}_{Cl_2} = 0.2 \times \frac{1000}{94.5} = 2.116 \text{ kmol/h}$$

Molar flow rate of hydrogen chloride gas at outlet of reactor

$$\dot{n}_{HCl} = \frac{1000}{94.5} = 10.582 \text{ kmol/h}$$

$$\dot{n}_t = \dot{n}_{Cl_2} + \dot{n}_{HCl} + \dot{n}_{AA} + \dot{n}_{MCA}$$

and $$p_t = \overline{p}_{Cl_2} + \overline{p}_{HCl} + \overline{p}_{AA} + \overline{p}_{MCA}$$

$$p_t = \overline{p}_{Cl_2} + \overline{p}_{HCl} + p_{vAA} \times x_{AA} + x_{MCA} \times p_{vMCA}$$

At 100°C vapour pressure of acetic acid $p_{vAA} = 400$ Torr

At 100°C vapour pressure of MCA, $p_{VMCA} = 30$ Torr

$$p_t = 1.1 \times 760 = \overline{p}_{Cl_2} + \overline{p}_{HCl} + 400 \times 0.5 + 30 \times 0.5 = 836 \text{ Torr}$$

$$\overline{p}_{Cl_2} + \overline{p}_{HCl} = 621 \text{ Torr}$$

$$(\overline{p}_{Cl_2} + \overline{p}_{HCl})/p_t = (\dot{n}_{HCl} + \dot{n}_{Cl_2})/\dot{n}_t$$

$$\frac{621}{836} = \frac{10.582 + 2.1164}{\dot{n}_t}$$

$$\dot{n}_t = 17.095 \text{ kmol/h}$$

At 100°C or in the exit gas-vapour mixture from the reactor

$$\dot{n}_{AA} = \frac{400 \times 0.5}{836} \times 17.095 = 4.09 \text{ kmol/h}$$

$$\dot{n}_{MCA} = \frac{30 \times 0.5}{836} \times 17.095 = 0.307 \text{ kmol/h}$$

This gas-vapour mixture is cooled to 40°C by cooling water in the overhead condenser. At outlet of the overhead condenser, gas mixture is saturated with acetic acid and MCA vapour.

To find out the composition of gas-vapour mixture at the outlet of overhead condenser, for the first trial calculations assume that almost total condensation is taking place in condenser.

Condensate composition at outlet of condenser

$$x_{AA} = \frac{4.09}{4.09 + 0.307} = 0.93$$

$$x_{MCA} = 0.07$$

At 40°C, $$p'_t = p_t - \Delta p_{HE}$$

$$= 1.1 - 0.04 = 1.06 \text{ atm}$$

The pressure drop in heat exchanger for gas-vapour mixture is assumed to be 0.04 atm.

At 40°C or at outlet of overhead condenser

$$p'_t = 1.06 \times 760 = \overline{p}_{HCl} + \overline{p}_{Cl_2} + 0.93 \times p_{vAA} + 0.07 \times p_{vMCA}$$

At 40°C, $p_{vAA} = 38$ Torr, $p_{vMCA} = 1$ Torr

$$805.6 = \overline{p}_{HCl} + \overline{p}_{Cl_2} + 0.93 \times 38 + 0.07 \times 1$$

$$\overline{p}_{HCl} + \overline{p}_{Cl_2} = 770.19 \text{ Torr}$$

$$\frac{\overline{p}_{HCl} + \overline{p}_{Cl_2}}{p'_t} = \frac{10.582 + 2.1164}{\dot{n}_t} = \frac{770.19}{805.6}$$

$$\dot{n}_t = 13.28 \text{ kmol/h}$$

At 40°C, $P_{vAA} = 38$ Torr, $p_{vMCA} = 1$ Torr

$$\dot{n}_{AA} = \frac{0.93 \times 38 \times 13.28}{805.6} = 0.5826 \text{ kmol/h}$$

$$\dot{n}_{MCA} = \frac{0.07 \times 1}{805.6} \times 13.28 = 1.154 \times 10^{-3} \text{ kmol/h}$$

For second trial calculations, composition of condensate at outlet of overhead condenser is calculated.

$$x_{AA} = \frac{4.09 - 0.5826}{(4.09 - 0.5826) + (0.307 - 1.154 \times 10^{-3})} = 0.9198$$

$$x_{MCA} = 1 - x_{AA} = 0.0802$$

$$p_t = 805.6 = \overline{p}_{HCl} + \overline{p}_{Cl_2} + 0.9198 \times p_{vAA} + 0.0802 \times p_{vMCA}$$

$$805.6 = \overline{p}_{HCl} + \overline{p}_{Cl_2} + 0.9198 \times 38 + 0.0802 \times 1$$

$$\overline{p}_{HCl} + \overline{p}_{Cl_2} = 770.5674 \text{ Torr}$$

$$\frac{\overline{p}_{HCl} + \overline{p}_{Cl_2}}{p_t} = 0.9565 = \frac{10.582 + 2.1164}{\dot{n}_t}$$

$$\dot{n}_t = 13.276 \text{ kmol/h}$$

$$\dot{n}_{AA} = \frac{0.9198 \times 38 \times 13.276}{805.6} = 0.576 \text{ kmol/h}$$

$$\dot{n}_{MCA} = \frac{0.0802 \times 1}{805.6} \times 13.276 = 1.32 \times 10^{-3} \text{ kmol/h}$$

For the new values of $\dot{n}_{AA}$ and $\dot{n}_{MCA}$

$$x_{AA} = 0.919\,97, \quad x_{MCA} = 0.08 \quad \text{or} \quad x_{AA} = 0.92, \quad x_{MCA} = 0.08$$

These values are very close to previous values, Hence, third trial is not required.
Mass of acetic acid condensed = $(4.09 - 0.576) \times 60 = 210.84$ kg/h
Mass of MCA condensed = $(0.307 - 1.32 \times 10^{-3}) \times 94.5 = 28.887$ kg/h
Heat duty of overhead condenser

ϕ_{tc} = Sensible heat transfer of gas-vapour mixture + Latent heat transfer for condensation of vapours + Subcooling of condensate (Subcooling of condensate from inlet to outlet temperature is necessary in multicomponent condensation.)

$$\phi_{tc} = (\dot{m}_{Cl_2} Cp_{Cl_2} + \dot{m}_{HCl} Cp_{HCl} + \dot{m}'_{AA} Cp_{AA} + \dot{m}'_{MCA} Cp_{MCA})\Delta\mathcal{T}$$
$$+ \dot{m}_{AA} \lambda_{AA} + \dot{m}_{MCA} \lambda_{MCA} + (m''_{AA} C_{LAA} + m''_{MCA} C_{LMCA})\Delta\mathcal{T}$$

where, $\dot{m}_{Cl_2} = 2.1164 \times 71 = 150.26$ kg/h

$\dot{m}_{HCl} = 10.582 \times 36.5 = 386.24$ kg/h

$$\dot{m}'_{AA} = \frac{4.09 + 0.576}{2} \times 60 = 139.98 \text{ kg/h}$$

$$\dot{m}'_{MCA} = \frac{0.307 + 1.32 \times 10^{-3}}{2} \times 94.5 = 14.568 \text{ kg/h}$$

Table 10.10 Specific Heat of Gases/Vapours and Liquids at 70°C

Component	C_{pi}, kJ/(kg · °C)	C_{Li}, kJ/(kg · °C)
Chlorine	0.5024	—
Hydrogen chloride	0.7955	—
Acetic acid	1.2267	2.22
MCA	1.0467	1.9

$$\dot{m}_{AA} = 210.84 \text{ kg/h}, \quad \dot{m}_{MCA} = 28.887 \text{ kg/h}$$

$$\lambda_{AA} = 110 \text{ kcal/kg} = 460.55 \text{ kJ/kg at 70°C temperature}$$

$$\lambda_{MCA} = 85 \text{ kcal/kg} = 355.88 \text{ kJ/kg at 70°C temperature}$$

$$\dot{m}''_{AA} = \frac{0 + 210.84}{2} = 105.42 \text{ kg/h}$$

$$\dot{m}'_{MCA} = \frac{0 + 28.887}{2} = 14.44 \text{ kg/h}$$

$$\phi_{tc} = (150.26 \times 0.5024 + 386.24 \times 0.7955 + 139.98 \times 1.2267 + 14.568 \times 1.0467) \times (100 - 40) + 210.84 \times 460.55 + 28.887 \times 355.88 + (105.42 \times 2.22 + 14.44 \times 1.9) \times (100 - 40)$$

$$= 157\,253.15 \text{ kJ/h} \equiv 43.6814 \text{ kW}$$ Ans. (d)

(e) Heat transfer area required for bubble column reactor:

Energy balance around reactor

Let 25°C be the reference temperature and liquid feed is at 25°C.

Heat must be removed by cooling medium circulated through the jacket around reactor

$\phi_t =$ Heat produced during reaction – Heat removed in overhead condenser – Enthalpy of product stream + Enthalpy of feed stream

$$= 87.92 \frac{\text{kJ}}{\text{mol}} \times 1000 \frac{\text{mol}}{\text{kmol}} \times \frac{\text{kmol}}{\text{h}} \text{ of Acetic acid consumed} - 157\,253.15 - [(1000 \times 1.9) + (634.92 \times 2.2)]\,(100 - 25)$$

$$= 87.92 \times 1000 \times 10.582 - 157\,253.15 - 248\,214.18$$

$$= 524\,903 \text{ kJ/h} \equiv 145.806 \text{ kW}$$

Let outlet temperature of cooling water from heat exchanger = 40°C

Mass flow rate of cooling water, $\dot{m}_w = \dfrac{524\,903}{4.1868 \times 8}$

$$= 15\,671 \text{ kg/h} \equiv 15.7 \text{ m}^3\text{/h}$$

If cooling water is circulated through plain jacket then jacket side heat transfer coefficient can be calculated by considering plain jacket as outside pipe of double pipe heat exchanger.

h_o:

where, d_e = Equivalent diameter = $4 \times r_H$ (10.21)

$$r_H = \frac{\text{Cross-sectional area}}{\text{Wetted perimeter}}$$

$$r_H = \frac{(\pi/4)(d_2^2 - d_1^2)}{\pi(d_2 + d_1)} = \frac{d_2 - d_1}{4} \quad (10.22)$$

where, d_2 = Inside diameter of jacket, m

d_1 = Outside diameter of reactor shell, m

Let $d_2 - d_1 = 100$ mm

$d_e = 4r_H = d_2 - d_1 = 0.10$ m

$$G = \frac{15\,671/3600}{(\pi/4)(d_2^2 - d_1^2)}$$

d_1 = Reactor inside diameter + 2 × Thickness of shell (t_s)

Let thickness of reactor shell, t_s = 8 mm (assumption)

This thickness is actually determined based on mechanical design of shell.

$d_1 = 1118 + 2 \times 8 = 1134$

$d_2 = 1134 + 100 = 1234$ mm

$$G = \frac{15\,671/3600}{(\pi/4)(1.234^2 - 1.134^2)} = 23.45 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{0.1 \times 23.45}{0.72 \times 10^{-3}} = 3256.9$$

(Viscosity of water at 36°C = 0.72 mPa · s)

$$Pr = \frac{C_L \mu}{k} = \frac{4.1868 \times (0.72 \times 10^{-3})\,10^3}{0.6228}$$

$$= 4.84$$

$$h_o = 0.023 \times \frac{0.6228}{0.1} \times (3256.9)^{0.8} \times (4.84)^{1/3}$$

$$h_o = 156.53 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Value of h_o is very low. To improve or to increase its value, let outlet temperature of cooling water = 34°C

$$\dot{m}_w = \frac{524\,903}{4.1868 \times 2} = 62\,685 \text{ kg/h}$$

$$G = 23.45 \times \frac{62\,685}{15\,671} = 93.65 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = 13\,006.9$$

$$h_o = 156.53 \times \left(\frac{13\,006.9}{3256.9}\right)^{0.8} = 473.9 \text{ W/(m}^2 \cdot \text{°C)}$$

h_i: Eq. (10.20)

where, S_g = Superficial velocity of gas, m/s

S_g = 10 cm/s = 0.1 m/s

where, C_L, μ_L and k_L are the properties of liquid phase of reactor at 100°C (reaction temperature).

Table 10.11 Properties at 100°C

	Acetic acid	MCA	Water
C_L, kJ/(kg · °C)	2.3	1.884	4.1868
μ, mPa · s	0.49	0.55	0.28
k, W/(m · °C)	0.173	0.143	0.68

$$C_{L\,\text{mix}} = \Sigma\, w_i\, C_{Li}$$

$$w_{\text{AA}} = \frac{0.5 \times 60}{(0.5 \times 60) + (0.5 \times 94.5)} = 0.3883$$

$$w_{\text{MCA}} = 1 - w_{\text{AA}} = 0.6117$$

$$C_{L\text{mix}} = \Sigma\, C_{Li}\, w_i = 0.6117 \times 1.884 + 0.3883 \times 2.3 = 2.046 \text{ kJ/(kg} \cdot \text{°C)}$$

$$\frac{1}{\mu_L} = \frac{1}{\mu_{\text{mix}}} = \frac{w_{\text{AA}}}{\mu_{\text{AA}}} + \frac{w_{\text{MCA}}}{\mu_{\text{MCA}}} = \frac{0.6117}{0.55} + \frac{0.3883}{0.49}$$

$$\mu_L = 0.525 \text{ mPa} \cdot \text{s}$$

$$k_L = \Sigma\, k_i\, w_i = 0.3883 \times 0.173 + 0.6117 \times 0.143$$

$$= 0.1546 \text{ W/(m} \cdot \text{°C)}$$

$$Pr_L = \frac{2.046 \times 0.525 \times 10^{-3}}{0.1546} \times \frac{1000}{1}$$

$$= 6.9479$$

Prandtl number of water at 100°C:

$$Pr_W = \frac{4.1868 \times 0.28 \times 10^{-3}}{0.68} \times \frac{10^3}{1}$$

$$= 1.724$$

$$h_i = 21\,766.5 \times (0.1)^{0.22} \times \left(\frac{1.724}{6.9479}\right)^{1/2}$$

$$= 6533.27 \text{ W/(m}^2 \cdot \text{°C)}$$

For MCA reactor, lead lined vessel is normally used.

Let thickness of lead lining = 5 mm

d_o' = OD of lead lining = 1.118 m

d_i' = ID of lead lining = 1.108 m

At 100°C, $k_{\text{lead}} = 34$ W/(m · °C),

$k_{\text{steel}} = 45$ W/(m · °C)

Conductive resistance offered by lead lining

$$= \frac{d_o}{d'_{om}} \times \frac{\Delta x}{k_{\text{lead}}} = \frac{d_o}{(d'_o - d'_i)} \times \ln\left(\frac{d'_o}{d'_i}\right) \times \frac{(d'_o - d'_i)/2}{k_{\text{lead}}}$$

$$= \frac{d_o \ln (d'_o/d'_i)}{2\, k_{\text{lead}}} \tag{10.23}$$

Fig. 10.11 *Proposed MCA Reactor Design*

Overall heat transfer coefficient U_o is calculated by Eq. (6.46).

Let fouling coefficients

$$h_{od} = h_{id} = 5000 \text{ W/(m}^2 \cdot \text{°C)}$$

$$\frac{1}{U_o} = \frac{1}{473.9} + \frac{1}{5000} + \frac{1.134 \ln\left(\frac{1.134}{1.118}\right)}{2 \times 45} + \frac{1.134 \ln\left(\frac{1.118}{1.108}\right)}{2 \times 34}$$

$$+ \frac{1.134}{1.108} \times \frac{1}{6533.27} + \frac{1.134}{1.108} \times \frac{1}{5000} = 333.3 \text{ W/(m}^2 \cdot \text{°C)}$$

$$\Delta\mathcal{T}_m = \Delta\mathcal{T}_{lm} = \frac{(100 - 32) - (100 - 34)}{\ln\,[(100 - 32)/(100 - 34)]} = 66.995 \text{ °C}$$

$$A_{o\text{req}} = \frac{\phi_t}{U_o \Delta \mathcal{T}_m} = \frac{524\,903 \times \left(\frac{1}{3600}\right) \times \left(\frac{1000}{1}\right)}{333.3 \times 66.995} = 6.53 \text{ m}^2$$

Let $A_{o\text{avai}} = 6.53 \times 1.2 = 7.84 \text{ m}^2$ (Area provided)

$= \pi d_o L' = \pi \times (1.134)\, L'$

where, L' = Length of cylinder that must be covered by jacket = 2.2 m

Note: However, entire height of liquid pool (h_L = 12.346 m) must be covered by plain jacket as shown in Fig. 10.11 to keep the uniform temperature of entire liquid pool. Reaction temperature can be controlled by controlling the flow rate of cooling water as shown in Fig. 10.11.

10.9 THIN FILM ABSORBER CUM REACTOR

A thin film absorber can be used as a continuous reactor for gas-liquid reaction. As shown in Fig. 10.12, it is basically a shell and tube heat exchanger. Gas and liquid enter at top and traveling cocurrent manner. Liquid is introduced as a thin film (2–3 mm thick) in the tubes (all around periphery) while gas flows in the middle. Product mass leaves from bottom. Unreacted gas, if any, escapes from a separate nozzle from bottom.

Thin film absorber cum reactor is strongly recommended for exothermic reactions in which reaction rate is high and the reaction is controlled by heat removal. Cooling medium (cooling water, chilled water, brine, etc.) can be used in the shell to remove heat of reaction.

Absorption of hydrogen chloride in an alkali, sulphonation of organic compounds with sulphur trioxide gas, ozonation, etc., can be carried out in such reactors.

10.9.1 Thin Film Absorber-cum-Reactor for Sulphonation

Use of sulphur trioxide (SO_3) as a sulphonating agent in place of oleum offers several advantages. It reduces reaction time from few hours to few seconds or minutes and avoids the problem of the disposal of spent acid. Thus it forms a part of green engineering. One of the most widely used systems for reacting sulphur trioxide with organic compound is a tubular thin film sulphonation reactor. The sulphonation reaction with SO_3 as agent is instantaneous and exothermic. Heat of reaction is ΔH_r = –2094 kJ/kg of SO_3 reacted for most aromatic compounds. Liquid SO_3 cannot be added directly to the organic material to be sulphonated because of safety and possible product degradation due to high exothermic heat of reaction. Hence SO_3 is vaporised and diluted with dry air/nitrogen.

In thin film sulphonation reactor SO_3-air mixture is contacted with organic liquid inside the tubes in cocurrent manner. Annular flow should be obtained inside the tube in which liquid forms a thin film on inside of the tube wall and gas mixture travels down the centre of the tube. Cooling water or chilled water is circulated on shell side as a cooling medium.

Fig. 10.12 *Falling Film Absorber-cum-Reactor*

Example 10.7 Design a thin film sulphonation reactor for the sulphonation of dodecylbenzene (also known as linear alkylbenzene) with sulphur trioxide based on following data:

(i) Heat of reaction: $\Delta H_r = -2094$ kJ/kg of SO_3

(ii) Residence time of organic liquid in reactor, $\theta = 10$ s

(iii) Dodecylbenzene feed rate = 1000 kg/h

(iv) Liquid film temperatures:
At inlet = 60°C, at outlet = 50°C

(v) Liquid flow rate inside the tubes
$= 6.25 \times 10^{-5} = m^3/(s \cdot m \text{ of periphery})$
Volumetric fraction of liquid film = 0.052

(vi) SO_3/Dodecylbenzene mole ratio = 1.05
(vii) Density of dodecylbenzene liquid at 50°C = 800 kg/m^3
(viii) SO_3 in air – SO_3 mixture = 6% (by mole)
(ix) Reaction pressure = 140 kPa a
(x) Material of construction of reactor = SS 316
(xi) Cooling water supply temperature, $t_1' = 32°C$
(xii) Percent conversion of dodecylbenzene = 100% in single pass

Stoichiometry:

	N-Dodecylbenzene (Linear alkybenzene)	Sulphur trioxide	Dodecyl benzene sulphonic acid
Molar mass (M):	246.43	80.06	326.49

Solution

Mass flow rate of organic liquid, $\dot{m}_1 = 1000$ kg/h

Volumetric flow rate of liquid, $q_{v_1} = \dfrac{1000}{800} = 1.25 \text{ m}^3/\text{h}$

Residence time of organic liquid in reactor = 10 s
Liquid flow rate inside the tubes = 6.25×10^{-5} m^3/(s · m of periphery)

$$6.25 \times 10^{-5} \times N_t \times \pi\, d_i = \frac{1.25}{3600}$$

Let tube OD, d_o = 10.05 mm, tube ID, d_i = 15.748 mm (16 BWG)

$$6.25 \times 10^{-5} \times N_i \times \pi \times 0.015\,748 = \frac{1.25}{3600}$$

$$N_t \approx 122$$

Volume fraction of liquid film = 0.052

Residence time of liquid film in reactor, $\theta = \dfrac{\text{Volume of reactor} \times 0.052}{\text{Volumetric flow rate of liquid}}$

$$10 = \frac{N_t \times \dfrac{\pi}{4} d_i^2\, L \times 0.052}{(1.25/3600)} = \frac{122 \times \dfrac{\pi}{4} (0.015\,748)^2 \times L \times 0.052}{(1.25/3600)}$$

$$L = 2.8 \text{ m}$$

Let length of tube, L' = 3.048 m (10 ft)

Heat transfer area provided by shell and tube heat exchanger or reactor:

$$A_{pr} = N_t \pi d_o L' = 122 \times \pi \times 0.019\,05 \times 3.048$$
$$= 22.254 \text{ m}^2$$

Heat duty

$$\phi_R = 2094 \times \frac{\text{kg of } SO_3 \text{ consumed}}{\text{h}}$$

$$SO_3 \text{ consumed in reaction} = \frac{1000}{246} \times 80 = 325.2 \text{ kg/h}$$

$$\phi_R = 2094 \times 325.2 = 680\,968.8 \text{ kJ/h}$$
$$\equiv 189.16 \text{ kW}$$

For triangular pitch arrangement $P_t = 23.8125$ mm (= 1.25 d_o)
Tube bundle diameter by Eq. (6.1):

$$k_1 = 0.319, \quad n_1 = 2.142 \quad \text{(From Table 6.2)}$$

$$D_b = 19.05 \left(\frac{122}{0.319}\right)^{\frac{1}{2.142}}$$
$$= 306 \text{ mm}$$
$$D_i = \text{shell ID} = D_b + C = 306 + 14 = 320 \text{ mm}$$

Let baffle spacing, $B_s = 100$ mm
Type of baffle = 25% cut segmental
Cooling medium = cooling water
Let outlet temperature of cooling water from reactor, $t_2' = 40°C$
Mass flow rate of cooling water,

$$\dot{m}_W = \frac{\phi_R}{C_L\ \Delta t'} = \frac{189.16}{4.1868 \times (40 - 32)}$$

$$\dot{m}_W = 5.6475 \text{ kg/s} \equiv 20.33 \text{ m}^3\text{/h}$$

Calculations for h_i or h_L:
Tube side heat transfer coefficient[10] can be calculated using Eq. (10.24).

$$h_L = \left(\frac{k_L}{d_i}\right) \times 0.06 \left(\frac{\rho_L}{\rho_G}\right)^{0.28} \left(\frac{d_i\ G_t\ w_g}{\mu_L}\right)^{0.87} (p_{r_L})^{0.4}$$

where,

k_L = Average thermal conductivity of liquid in the film, W/(m · °C)
ρ_L = Average density of liquid in film, kg/m^3
ρ_G = Average density of total gas stream, kg/m^3
μ_L = Average viscosity of liquid in film, kg/(m · s)
G_t = Total gas and liquid mass velocity, kg/(m^2 · s)
w_g = Mass fraction of gas

Viscosity of dodecyl benzene at 55°C,

$$\mu_1 = 0.010\ 3174 \exp\,[1934.3/(t + 273.15]\ \text{cP}$$
$$= 3.746 \text{ cP}$$

Viscosity of liquid dodecyl benzene sulphonic acid at 55°C,

$$\mu_2 = 0.022\ 9282 \exp\,[6643.4/(t + 273.15] \times 10^{-6} \text{ cP}$$
$$= 14.21 \text{ cP}$$

Assume average mole fraction of alkyl benzene = 0.5 in the absorber cum reactor.

Average mass fraction of alkyl benzene

$$w_{AB} = \frac{0.5 \times 246}{(0.5 \times 246) + (0.5 \times 326)} = 0.43$$

$$\ln \mu_L = w_{AB} \ln \mu_1 + (1 - w_{AB}) \ln \mu_2$$

$$\ln \mu_L = 0.43 \ln (3.746) + (1 - 0.43) \ln (14.21)$$

$$\mu_L = 8 \text{ cP or mPa} \cdot \text{s}$$

Thermal conductivity of organic liquid, $k_L = 0.15$ W/(m · °C)

Specific heat of liquid, $C_L = 2$ kJ/(kg · °C)

Prandtl Number for liquid

$$Pr_L = \frac{C_L \mu_L}{k_L} = \frac{2 \times 10^3 \times 8 \times 10^{-3}}{0.15}$$

$$= 106.67$$

Average flow rate of liquid in absorber

$$\dot{m}_{av} = \frac{1000 + \left[\left(\frac{1000}{246}\right) \times 326\right]}{2} = 1162.6 \text{ kg/h}$$

$$SO_3 \text{ at inlet} = \frac{1000}{246} \times 1.05 = 4.268 \text{ kmol/h}$$

$$SO_3 \text{ at outlet} = \frac{1000}{246} \times 0.05 = 0.203 \text{ kmol/h}$$

$$\text{Air flow rate} = \frac{4.268 \times 0.94}{0.06} = 66.865 \text{ kmol/h}$$

Average flow rate of gas mixture, $\dot{m}_2$

$$= 66.865 \times 29 + \left(\frac{4.268 + 0.203}{2}\right) \times 80$$

$$= 2117.9 \text{ kg/h}$$

$$\text{Mass fraction of gas} = \frac{2117.9}{(2117.9 + 1162.6)} = 0.6456$$

Density of gas mixture $\rho_G = \dfrac{PM_{av}}{RT}$

$$M_{av} = \frac{2117.9}{(66.865 + 2.2355)} = 30.65 \text{ kg/kmol}$$

$$\rho_G = \frac{140 \times 30.65}{(273 + 45)} \times \frac{273}{101.325 \times 22.414} = 1.622 \text{ kg/m}^3$$

$$G_t = \frac{(2117.9 + 1162.6)/3600}{a_t}$$

$$a_t = \frac{N_t}{N_P} \times \frac{\pi}{4} d_i^2 = \frac{122}{1} \times \frac{\pi}{4} (0.015\,748)^2$$

$$a_t = 0.023\,76 \text{ m}^2$$

$$G_t = \frac{0.91\,125}{0.023\,76 \text{ m}^2} = 38.352 \text{ kg/(m}^2 \cdot \text{s)}$$

Eq. (10.24): $$h_L = \frac{0.15}{0.015\,748} \times 0.06 \left(\frac{800}{1.622}\right)^{0.28} \left(\frac{0.015\,748 \times 38.352 \times 0.6456}{8 \times 10^{-3}}\right)^{0.87}$$

$$\times (106.67)^{0.4}$$

$$h_i = h_L = 617.66 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Calculations of h_o

Shell side flow area, Eq. (6.33)

$$A_s = \left(\frac{23.8125 - 19.05}{23.8125}\right) \times 0.32 \times 0.1$$

$$= 0.0064 \text{ m}^2$$

Shell side mass velocity,

$$G_s = \frac{\dot{m}_W}{A_s} = \frac{5.6475}{0.0064} = 882.42 \text{ kg/(m}^2 \cdot \text{s)}$$

Shell side velocity

$$u_s = \frac{882.42}{993} = 0.8886 \text{ m/s}$$

Shell side equivalent diameter; Eq. (6.37):

$$d_e = \frac{1.1}{d_o} (P_t^2 - 0.907\, d_o^2)$$

$$= \frac{1.1}{19.05} (23.8125^2 - 0.907 \times 19.05^2)$$

$$= 13.736 \text{ mm}$$

$$Re_o = \frac{d_e G_s}{\mu_W} = \frac{0.013\,736 \times 882.42}{0.72 \times 10^{-3}} = 16\,834.6$$

$$Pr_o = \frac{C_L\, \mu}{k} = \frac{4.1868 \times 10^3 \times 0.72 \times 10^{-3}}{0.63} = 4.7849$$

$$\frac{h_o d_e}{k} = 0.36\, Re_o^{\,0.55}\, Pr_o^{\,0.33} \left(\frac{\mu}{\mu_W}\right)^{0.14} \tag{6.39}$$

$$h_o = 0.36 \times \frac{0.63}{0.013\,736} \times (16\,834.6)^{0.55} \times (4.7489)^{0.33} \times 1$$

$$h_o = 5827.33 \text{ W/(m}^2 \cdot \text{i)}$$

Overall heat transfer coefficient: Eq. (6.46)

$$\frac{1}{U_o} = \frac{1}{5827.33} + \frac{1}{10\,000} + \frac{0.019\,05 \ln\left(\frac{19.05}{15.748}\right)}{2 \times 16.3} + \frac{19.05}{15.748}\left(\frac{1}{617.66} + \frac{1}{10\,000}\right)$$

$$U_o = 406.1 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Mean temperature difference $\Delta\mathcal{T}_m = F_t \times \Delta\mathcal{T}_m$

$F_t = 1$ for 1–1 shell and tube heat exchanger

$$\Delta\mathcal{T}_{\text{lm}} = \frac{(50-32)-(60-40)}{\ln\left(\frac{50-32}{60-40}\right)} = 18.98^\circ\text{C} = \Delta\mathcal{T}_m$$

Heat transfer area required

$$A_r = \frac{\phi_R}{U_0\ \Delta\mathcal{T}_m} = \frac{189.16 \times 10^3}{406.1 \times 18.98} = 24.54 \text{ m}^2$$

Area provided $A_{pr} = 22.254 \text{ m}^2 < A_r$

Revised tube length, $L = 12 \text{ ft} = 3.6576 \text{ m}$

$A_{pr} = 122 \times \pi \times 0.019\,05 \times 3.6576 = 26.71 \text{ m}^2$

Hence excess heat transfer area

$$= \left(\frac{26.71}{24.54} - 1\right) \times 100$$

$$= 8.84\ \%\ \text{(adequate)}$$

Note: Sulphonation of aromatic compounds is a very well-known unit operation in chemical industry. Either sulphuric acid (H_2SO_4) or sulphur trioxide (SO_3) is used as a sulphonating agent. When SO_3 (liquid or gas) is used, it is observed that its diffusion into the aromatic compound and the rate of reaction are quite high. Since the exothermic heat of reaction is very high, the design problem gets centred to effective design for heat transfer so that heat is removed from the reaction mass at a rate equal to the product formation rate. If this heat transfer rate is not effective, it is observed that side reactions take place and eventually charing may take place.

To control the heat transfer, various solvents are being used. In one case, sulphuric acid itself acts as a solvent. For the purpose, oleum of different strengths is used by the industry, among which oleum of 30% strength is common. In a jacketed batch reactor, aromatic compound is charged and oleum is added at a controlled rate. Cooling medium (mostly cooling water) is circulated in the jacket to cool the reaction mass. Presence of sulphuric acid provides dilution effect thereby temperature control becomes easier. With continuous stirring, oleum addition is carried out. The product mass is tested and filtered through a filter. Mother liquor

from the filter is contaminated sulphuric acid. Excess sulphuric acid (with wash water) is filtered out. Thus highly corrosive effluent generates during this operation which is contaminated with organic impurities. Disposal of this waste acid is a serious problem. It is either sold to some other manufacturers who can use for the manufacture of $FeSO_4$, $Al_2(SO_4)_3$, etc., or the spent acid is neutralised with lime solution and disposed as $CaSO_4$ after leaching and drying. Final leached out effluent from neutralisation is highly contaminated with organic impurities which can be measured in terms of COD. Disposal of leached liquor is not an easy task and calls for its extensive treatments, depending on local stipulations by the pollution control authority. Apart from the waste disposal problems, it is observed that if addition of oleum is not controlled effectively or temperature is not controlled, byproducts do form and overall recovery of the product is rather poor.

Use of a falling film absorber for the sulphonation reaction could be a relief. As seen in this example, heat can be easily controlled by circulating the cooling medium outside of the tubes. SO_3 can be relatively easily generated by gentle heating of liquefied SO_3 in a separate vessel and passed in the absorber. As addition of 100% SO_3 is not desired, normally a mixture of SO_3 and air or nitrogen is preferred and concentration of SO_3 in the mixture is controlled in the range of 5 to 8 mole %. Reaction in this absorber produces the product with less byproducts. Only constraint in use of an absorber for this reaction is that the aromatic compound is required to be introduced as a solution (liquid) and hence, if the reactant is a solid, a suitable solvent will have to be found which has good solubility for the reactant as well as for the products and it does not react with SO_3. Thus use of falling film absorber acts as means for minimising the waste generation and forms a part of Green Engineering practice. Several solvents are in use, e.g., *n*-hexane, carbon tetrachloride, methanol, ethyl acetate, pyridine, etc., for the purpose. For selection of a solvent and also developing a film coefficient correlation [such as Eq. (10.24)], laboratory and pilot experiments are needed. Such experiments can also provide useful information on various other design parameters such as desired liquid film thickness in the tubes, optimum concentration of SO_3 in the ingoing gas mixture, minimum reaction path (length) required, etc. It is thus possible to develop a continuous or semi-continuous plant with a falling film absorber for such a sulphonation unit operation without appreciable waste generation.

10.10 CATALYSIS

Catalysis plays an important role in reaction engineering. A preamble on catalysts is therefore a necessity to process engineers who design catalytic reactors for the process industry.

It is a known fact that a catalyst is a substance which increases the rate of a reaction without being consumed in it. Catalysis can be described as being homogeneous or heterogeneous. Heterogeneous catalysis involves the use of a catalyst in a different phase from the reactants. Example is the use of a solid catalyst with the liquid or gaseous reactants. Solid catalyst can be in the suspended form (as a slurry) in liquid

or in the fixed bed form on which liquid or gas can react. In Example 10.5, nickel in slurry form was used as a catalyst.

In homogeneous catalysis, catalyst is in the same phase as that of reactant. Commenly cited example in this category is the use of NaI or MnO_2 as a catalyst for decomposition of H_2O_2. Acid (such as sulphuric acid, phosphoric cid, etc.) catalyzed reactions are well known in process industry. Manufacture of amino resins, esterification reactions and other examples can be cited in this category.

Enzymes are organic catalysts with high molar mass. In human body, sugar (glucose) is decomposed at 37°C to produce carbon dioxide and water which releases energy with the help of a biological catalyst (a protein). In a similar manner, enzymes allow reactions to take place under ordinary conditions (say atmospheric pressure and ambient temperature). Esterification of acetic acid and butyl alcohol, fermentation of molasses, manufacture of many antibiotics (such as penicillin), etc., are examples of enzymatic reactions.

The fundamental mechanism of catalysed reaction relates to activation energy requirement of a chemical reaction. Barring some spontaneous reactions, most reactions need activation energy (E_a) for initiation. This energy can be supplied in various forms; such as heat, electricity, ultraviolet light, friction, vibration, etc. A catalyst lowers the activation energy required for the reaction. It is known that most chemical reactions are exothermic (release heat to environment) while some reactions are endothermic in nature and energy is supplied to the system. Potential energy diagrams for both types of reactions are given in Fig. 10.13.

Fig. 10.13 *Potential Energy Diagrams for Chemical Reactions*

It can be observed in Fig. 10.13 that the catalyst has no effect on relative (potential) energies of the reactants and products or it does not change equilibrium constant (K_c). Role of a catalyst is thus to speed up the reaction allowing equilibrium to be achieved faster. Catalyst activity is thus judged by order of reduction in the activation energy.

With above introduction to the subject, focus will be on heterogeneous catalysis. Solid catalysts are used in the process industry in different shapes and materials. Figure 10.14 gives different shapes in which solid catalysts are available.

Fig. 10.14 *Some Shapes of Solid Catalysts*

Ceramic support materials are well known for impregnation of noble metals (Pt, Pd, Rh). Supports can take shape of Raschig and other ring forms, monolith (honeycomb) structures, etc., so that large surface area is available for reaction cites with high void for low pressure drop.

Zeolite is a class of materials which are made up of oxides of aluminium and silicon. These materials are excellent for ion exchange applications, offering themselves as catalysts for many reactions.

In a few applications, catalyst can be in powder form. Catalysts in wire gauge or wire net form are also used in the industry.

10.10.1 Examples of Solid Catalysed Reactions

1. Reforming of natural gas or naphtha with steam. Raschig rings, partition rings or wagon wheels, impregnated with nickel, are commonly used for this application.
2. Shift conversion, ammonia/methanol synthesis, oxidation/hydrogenation reactions are carried out in catalytically packed bed reactors. Pellets, irregular shapes and extrudates are some forms on which these reactions are carried out.

3. Oxidation of ammonia to nitric acid is carried out on Pt/Rh wire net.
4. Palladium impregnated monolith catalysts are commonly used for hydrogenation applications. For example, aromatic nitro compounds are hydrogenated to corresponding amino compounds by hydrogenation.
5. Oxidation of toluene to benzaldehyde and subsequently to benzoic acid can be carried out on a bed, packed with solid catalyst.
6. Reactive distillation unit can be designed for esterification reaction in which a packed bed of strong cation exchange resin or zeolith material can be used for the reaction.
7. In Bechamp process, nitro aromatic compounds are reduced to corresponding amino compounds using iron powder as a catalyst.

10.10.2 Catalysis – A key to Waste Minimisation

Waste is generated in chemical synthesis on two accounts; one by formation of byproducts and another due to use of excess reactants that are not always recovered fully for recycle. Solvents are used in many reactions which also add to the problem as solvents are not fully recovered on completion of the reaction in most cases. An examples can be cited to the generation of the waste.

Consider reduction of *para*- or *ortho*-nitro toluene to corresponding amines. Classically, this process is carried out in water as a solvent. To this, fine iron powder is added and a small amount of acid (say HCOOH) is also added. At least 10 to 20% excess iron powder is added to ensure completion of reaction and also to account for purity of the powder. Reaction mass is continuously stirred and heated to about 80°C at atmospheric pressure. After a few hours, the reaction is considered completed (based on thin layer chromatographic results – TLC). Stoichiometry of the reaction can be written as under.

	ONT	Iron	Water	OT	Iron Hydroxides	
M	4 × 139.11	9 × 55.85	16 × 18.02	4 × 109.13	3 × 89.86	6 × 106.87

After the completion of the reaction, stirring is stopped and mass is allowed to separate in two layers. Organic (top) layer is decanted to another vessel. Vacuum distillation is carried out at about 10 Torr a to recover the amine product. Bottom aqueous layer is filtered and recycled as solvent for the next batch. However, every time recycle may not be possible and it may become necessary to discard contaminated aqueous layer (having high chemical oxygen demand – COD) after a few cycles. Solids are removed from filter which is a mixture of iron oxides, unreacted iron and other impurities. On drying (in open sky) the waste has to be discarded.

In a typical run, a batch is charged with 700 kg of *ortho*-nitro toluene, 800 kg iron powder (90% Fe), 6 kg acid (as catalyst) and 1000 kg water. Stoichiometrically, the reaction should produce 547 kg *ortho*-amino toluene (also known as *ortho*-toluidine) but, in actual practice, approximately 500 kg *ortho*-amino toluene is produced for

the same input of the reactant. Wet solid waste generation (on filtration) is more than 1200 kg which may reduce to about 1000 kg on drying. This solid waste is contaminated with organic impurities and cannot be used as such. Some attempts are made to convert this mass into a usable product as a red oxide for mixing with paints but they have met with limited success. Some cement manufacturers use this waste in formation of clinker. If no disposal is realised, solid waste must be buried as a hazardous waste. Considering 90% purity of iron powder, excess reactant is 11.8%. It is reported that byproduct formation is quite low.

Above example clearly gives a picture of waste generation; both solid and liquid. Also actual product realisation is 92%.

The key to waste minimisation is selectivity in organic synthesis. Unfortunately, the standard definition of selectivity is the yield of product formed divided by the amount of substrate converted. Hence, it is necessary to consider the overall stoichiometry of the process by examining the atom utilisation. The atom utilisation or atom selectivity is defined as the ratio of the molar mass of the desired product to the sum of the molar mass of all materials produced in the process. It is an useful tool for quickly evaluating the amount of waste generated in alternative routes to a particular product. Alternative processes to the particular product are compared on the basis of their atom utilisations and *E* factor (kg byproduct per kg product). In order to calculate the *E* factor, the amount of solvent consumed is also taken into account. A further refinement could also take the energy consumption of alternative routes into consideration.

The next level of sophistication is to design catalytic processes which lend themselves to facilitate recovery and recycling of the catalyst. In the foregoing case, catalytic hydrogenation processes are developed. Stoichiometry of the reaction can be written as given below

	ONT	Hydrogen	OT	Water
M	139.11	3 × 2.02	109.13	2 × 18.02

In one alternate, Rainey nickel is used as a slurry catalyst. Its presence in the amount of say 5% is required and nearly 100% conversion with nearly 100% yield are achieved. In this case, solid waste generation could be just 45 kg per batch. Product realisation could be 98%. Spent nickel can be processed to recover its metal value to produce fresh catalyst.

In another alternative, monolith catalyst, coated with palladium, can be considered. Refer Fig. 10.1(j) or 10.1(k) in which an arrangement is represented to hydrogenate nitro aromatic compound. In this alternative, there is no waste generation and conversion/yield can be nearly 100%. Same vessel can be utilised to distils out the water (byproduct), leaving behind the product. Such an arrangement also calls for less energy than that required in the classical process. This process can be considered as a part of cleaner production and forms a part of Green Engineering.

Several similar cases can be cited to show that catalytic processes reduce waste generation and increase productivity.

Hence, it can be concluded that catalysis is the key to waste minimisation.

10.11 DESIGN OF FIXED CATALYST BED REACTORS FOR GASEOUS REACTIONS[10, 11, 12, 2]

In many syntheses, fixed bed catalytic reactors are used. Water shift reaction to produce hydrogen by reaction of carbon monoxide with steam is carried out in a fixed catalyst bed. However, this reaction is exothermic with practically no effect of pressure on the reaction rate. At high temperature, the rate of reaction is relatively high but equilibrium conversion is low. In such a situation, a battery of reactors in series is used. High temperature, medium temperature, and low temperature shift converters are common in syngas production from hydrocarbons. Each reactor is packed with different types of catalysts. Exit gas stream from each converter is passed through heat exchangers to recover the heat and to lower the gas temperature, entering the next reactor. In this way, advantages of high rate of reaction at higher temperature and higher equilibrium conversion at low temperature are achieved. In a typical natural gas based plant, carbon monoxide content in the reformer outlet gas is about 15% (v/v) which is reduced to 0.2% (v/v) in two or three different axial flow converters. Generally, these converters are designed for low to moderate pressures.

For certain exothermic gaseous reactions, rate of reaction at low temperature is so low that required volume of catalyst works out to be very large. Cost of such a large reactor could be prohibitive, more so for high pressure reaction. Hence certain exothermic gaseous reactions are carried out at high temperature. Examples are ammonia synthesis, methanol synthesis, dissociation of nitrous oxide to nitrogen, etc. While designing such converters, process engineer can consider different options. In one option, reaction is carried out in multiple reactors having the same or different catalysts. Exit gas from each reactor is cooled in an external heat exchanger. Partially converted gas mixture from the heat exchanger enters the next reactor and so on as shown in Fig. 10.15.

Note:
Catalysts in R-I, R-II and R-III could be same or of different qualities and quantities.

Fig. 10.15 *Multiple Catalytic Reactors with External Heat Exchangers*

Cost of number of reactors and heat exchangers is becoming prohibitive in many cases. Also heat recovery from the exit gas mixture from a reactor may not be possible in each case.

Quench bed reactors are developed for carrying out exothermic reactions at high temperature. Figures 10.16(a) and (b) depict such designs. Entire reactor is one vessel in which there are a number of catalyst beds (usually 3 or 4).

A known portion of incoming gas mixture (stream F_1 which is more than 50% of total feed) is introduced at the bottom at relatively low temperature. It passes through the annulus around catalyst baskets, thereby keeping the shell of the reactor cool and also gets some heat from the reacting gas mixture. F_1 stream enters the shell and tube heat exchanger, located at top and gets heated by outgoing gas mixture. Hot F_1 stream enters first catalyst bed and partial conversion takes place thereby the gas mixture is heated up. Portion of the main gas stream is so controlled that temperature of first bed exit gas stream is controlled at predefined value. Partially reacted gas mixture from first bed is cooled by quenching by a definite proportion of the fresh gas mixture (first quench). Mixing of both gas mixtures results in lowering of the temperature of the gas mixture, entering second catalyst bed. In this manner, temperature of gas mixture, entering other beds is controlled by quenching, thereby, conversion in each bed is increased. Gas mixture from final catalyst bed is taken out through a central pipe and taken to the heat exchanger for exchanging heat with incoming gas mixture. Overall effect is to get definite conversion per pass through the converter without exchanging heat in external heat exchangers.

Since number of moles reduce during syntheses of ammonia and methanol, high pressure is employed. Quench bed reactor for such syntheses is a multi-layer wall vessel. As explained earlier, wall temperature of the reactor is kept low by the flow of ingoing gas mixture in the annulus. This is advantageous as at low temperature, lower wall thickness is required.

In Fig. 10.16(a), gas flow through the catalyst bed is axial while in Fig. 10.16(b), it is radial. Design, shown in Fig. 10.16(b), results in lower pressure drop in the gas mixture, resulting in energy saving. Volume of catalyst in each bed differs. Volume of fourth bed is highest while that of first bed is lowest. Volumes are decided by reaction rate, catalyst activity and safety margin against possible deactivation due to catalyst poisons.

While dissociating nitrous oxide to nitrogen and oxygen, increase in moles takes place. According to Le Chatélier's principle, low pressure favours the reaction. Therefore, a quench bed reactor with multiple catalyst beds for dissociation of nitrous oxide is designed for low pressure.

Design of fixed catalyst bed reactors for gaseous reactions thus can be innovative. Process Engineer can design an efficient system with best heat recovery options. Some other designs of industrial fixed bed catalytic reactors are as follows.

(i) Packed bed reactor, (ii) shell and tube heat exchanger in which catalysts are placed inside the tubes, (iii) annulus flow-type reactor in which catalyst are placed inside the inner tube. (Feed gas first pass through the annulus and then enter the inner tube. Product gases are taken out from the bottom of inner tube), (iv) reactor in which catalyst can be in form of mesh. Mesh of catalyst may be placed inside tower. Feed gas mixture

Fig. 10.16 *Multiple Quench Bed Reactor*

is introduced from the top of tower. Product gases leave from the bottom of catalyst mesh. (For example, ammonia oxidation to nitric acid.) In some cases catalyst mesh may be wound around the tubes of shell and tube heat exchanger. (For example, reactor of acetaldehyde plant.) In partial oxidation of ethanol vapour, silver catalyst in the form of mesh is wrapped around the tubes of shell and tube heat exchanger.

10.11.1 Industrial Examples of Fixed Bed Catalytic Reactor

	Product	*Major reaction step*	*Catalyst*
1.	Ammonia	$N_2 + 3\ H_2 \rightleftharpoons 2\ NH_3$	FeO/Fe_2O_3, promoted by Al_2O_3 and K_2O
2.	Nitric acid	$4\ NH_3 + 5\ O_2 \rightarrow 4\ NO + 5\ H_2O$ $2\ NO + O_2 \rightarrow 2\ NO_2$ $3\ NO_2 + H_2O \rightarrow 2\ HNO_3 + NO$	Pt and Rh
3.	Sulphur trioxide (Sulphuric acid plant)	$SO_2 + 1/2\ O_2 \rightarrow SO_3$	$V_2O_5 + K_2O$
4.	Methanol	$CO + 2\ H_2 \rightarrow CH_3OH$	CuO
5.	Formaldehyde	$CH_3OH + 1/2\ O_2 \rightarrow HCHO + H_2O$	Ag
6.	Aniline	$C_6H_5OH + NH_3 \rightarrow C_6H_5NH_2 + CO_2 + H_2O$	Ni Supported on Al_2O_3
7.	Disintegration of nitrous oxide	$N_2O \rightarrow N_2 + 1/2\ O_2$	Transition metal oxides
8.	Ethyl benzene	$C_6H_6 + C_2H_4 \rightarrow C_6H_5CH_2CH_3$	Zeolite

Fixed bed catalytic reactors are operated in both adiabatic and non-adiabatic manner. If the heat of reaction is small then adiabatic packed bed-type reactor is used as fixed bed reactor. In such a type of reactor, there is no provision for heat transfer.

But the reactions with a large heat of reaction as well as reactions that are extremely temperature sensitive are carried out in non-adiabatic reactors in which indirect heat exchange takes place by circulating heat transfer medium integrated in the fixed bed. With large heat of reaction, shell and tube-type design is preferred in which catalyst is inside the tubes and heat transfer medium is circulated on shell side. Alternatively, multiple quench bed reactor can be used (e.g., ammonia synthesis). When high pressure and high temperature reaction is carried out with indirect heat exchangers, conversion per pass can be near equilibrium. However, in multiple quench bed reactor, temperature of final exit stream controls the conversion per pass.

The heart of a fixed-bed reactor and the site of the chemical reaction is the catalyst. The overall reaction taking place on catalyst surface can be theoretically divided into the following five separate steps (a well accepted mechanism in chemical reaction engineering):

1. Diffusion of the reactants from the gas space through the outer gas particle boundary larger, macropores and micropores.

2. Chemisorption on active centres
3. Surface reactions
4. Desorption of the products
5. Back-diffusion of the products into the gas space. Also deactivation of catalyst due to fouling, poisoning and elevation in temperature must be considered.

In the development of rate equation only a few of these five steps are considered which control the overall rate; other steps may be either neglected or combined. These steps do not necessarily proceed in series or in parallel, frequently making it impossible to combine them by simple means.

For different type of solid catalysed reactions and for different mechanisms, rate equations are given in Table 4.8 of Ref. 2.

For example, if the chemical reaction is $A \rightleftharpoons R$

Assumed mechanism for this reaction is

$A + S \rightleftharpoons AS$ (Absorption)

$AS \rightleftharpoons RS$ (Reaction)

$RS \rightleftharpoons R + S$ (Desorption)

If desorption of R controls the overall rate, then rate equation is

$$r_R = \frac{k\left(C_A - \dfrac{C_R}{K}\right)}{1 + k_A C_A} \tag{10.25}$$

where, r_R = Rate of formation of product R (overall)
k = Overall reaction rate constant
k_A = Reaction rate constant for forward reaction
K = Overall equilibrium constant for the chemical reaction
C_A, C_R = Concentrations of A and R, respectively.

All fixed bed catalytic reactors are assumed to behave like ideal plug flow reactor. Equation used for the design or sizing of fixed bed reactor is

$$\frac{W}{F_{Ao}} = \int_o^{X_A} \frac{d\,X_A}{-r_A'} \tag{10.12}$$

where, W = Mass of catalyst, kg
F_{Ao} = Molar flow rate of limiting reactant in feed, kmol/s
X_A = Fractional conversion of limiting reactant A

or $$X_A = \frac{\text{Moles of } A \text{ reacted}}{\text{Moles of } A \text{ in feed}}$$

$-r_A'$ = Overall rate of reaction in $\dfrac{\text{Moles of } A \text{ reacted}}{\text{S} \cdot \text{kg of catalyst}}$

S = Surface area of catalyst, m^2/kg

Most difficult part is the development of reliable expression of overall rate of reaction. It also requires detailed kinetic data.

Rearranging Eq. (10.12),

$$\frac{dX_A}{d(W/F_{AO})} = -r'_A \tag{10.26}$$

For a real reactor with packed catalyst bed, Eq. (10.26) is modified as under.

$$\frac{dX_A}{d(W/F_{AO})} = -r'_A \cdot \eta \cdot \zeta \tag{10.27}$$

where η represents catalyst effectiveness while ζ represents deactivation function. Both these factors are derived from actual experience or pilot plant studies for a reasonable long period.

10.11.2 Scale-up Method

Any type of commercial scale reactor cannot be designed totally based on theoretical equations. It requires at least laboratory and/or pilot plant data of the reactions involved. A satisfactory scale-up procedure requires a stepwise empirical approach in which the size of the reactor is increased successively. Basically the rate of chemical reaction is independent of the size and structure of a reactor. But the overall rate of reaction is influenced by rate of mass transfer and rate of heat transfer which are usually controlled by the size and structure of the reactor. For example, due to significant wall effect smaller size reactor provides higher value of mass transfer coefficient compared to larger size reactor. Shell and tube-type construction provides higher heat transfer coefficient, compared to jacketted tower-type structure. Thus a chemical reaction is indirectly affected by reactor type and scale. Based on only lab scale data, it is not possible to predict the reliable values of overall rate of reaction, reaction time, and product composition for commercial scale reactor.

Example 10.8 Design the fixed bed catalytic reactor for 1000 t/a diethylbenzene plant based on the following data obtained on a pilot plant reactor:

Data of pilot plant reactor[14]

(i) Feed composition

Component	Mass %
Ethyl benzene	89
Ethanol	11

Feed flow rate in pilot plant reactor = 100 kg/h, Feed temperature = 375°C

(ii) Chemical reaction

(a) Main reaction

$$\underset{\text{Ethyl benzene}}{C_6H_5C_2H_5(g)} + \underset{\text{Ethanol}}{C_2H_5OH(g)} \rightleftharpoons \underset{\text{Diethyl benzene}}{C_6H_4\text{–}(C_2H_5)_2(g)} + \underset{\text{Water}}{H_2O(g)}$$

Reaction temperature = 375° C

(b) Side reactions are taking place in the reactor.

(iii) Product composition:

Component	mass %
Diethyl benzene	16
Benzene	6
Ethyl benzene	74
Water	4

Product rate is approximately equal to the feed rate.

(iv) Catalyst: Zeolite with binders

Density of catalyst particle: 630 kg/m^3

Catalyst porosity: 0.3

Form of catalyst: 1.5 mm extrudates of 4 to 6 mm length

(v) Type of reactor: Shell and tube heat exchanger type in which catalyst is placed inside the tube.

(vi) Reaction is endothermic and heat of reation is 29 kJ/mole ethanol. It is carried out in isothermal manner. Dowtherm A is to be circulated in liquid form on shell side to maintain the isothermal condition.

(vii) $$\frac{m_W}{\dot{m}_F} = 17.3 \frac{\text{kg of catalyst}}{\text{kg of ethanol in feed/hour}}$$

(viii) Superficial mass velocity of feed gas

$G = 0.55$ kg/(m$^2 \cdot$ s)

Feed composition, feed temperature, reaction temperature, operating pressure, catalyst, type of reactor, etc., are same in pilot plant, and commercial reactor.

Solution

Capacity of plant = 1000 t/a of diethyl benzene (DEB)
Let number of working days per annum = 330

$$\text{Production rate of DEB} = \frac{1000 \times 1000}{330 \times 24} = 126.26 \text{ kg/h}$$

Let design production rate of DEB = 150 kg/h

Table 10.12 Product Composition for Commercial Scale Plant

Component	mass %	kg/h
DEB	16	150.00
Benzene	6	56.25
Ethyl benzene	74	693.75
Water	4	37.50
Total	100	937.50

Feed rate ≅ Product rate

Table 10.13 Feed Composition for Commercial Scale Plant

Component	mass %	kg/h
Ethyl benzene	89	834.375
Ethanol	11	103.125
Total	100	937.500

Ethanol is the limiting reactant. It does not appear in the product mixture which means conversion based on ethanol is 100%.

Mass of catalyst required in commercial scale plant = 17.3 × 103.125 = 1784 kg (min.)

$$\text{Volume of solid catalyst} = \frac{\text{Mass of catalyst}}{\text{Density of catalyst}}$$

$$= \frac{1784}{630} = 2.8317 \text{ m}^3$$

Porosity of catalyst bed = 0.3

Bulk volume occupied by the catalyst = 2.8317/0.7 = 4.045 m^3

Superficial mass velocity of feed gas G = 0.55 kg/($m^2 \cdot$ s)

$$\text{Total cross-sectional area of catalyst (or of) tubes} = \frac{(937.5/3600)}{0.55} = 0.4735 \text{ m}^2$$

Select 50 mm NB tubes of 14 BWG (3.76 mm) thickness.

MOC of tube = SS 316

Tube OD = 50.8 mm, Tube ID = 43.28 mm

$$\text{Total number of tubes required } n_t = \frac{0.4735}{(\pi/4)\,(0.043\,28)^2} = 322$$

Length of tubes required

$$L = \frac{\text{Net volume of catalyst}}{n_t \times (\pi/4)\, d_i^2} = \frac{4.045}{322 \times (\pi/4)(0.043\,28)^2}$$

$$= 8.539 \text{ m}$$

Select the tubes of 10 m length. Above calculations have fixed the value of heat transfer area available for heat transfer. Temperatures and flow rate of Dowtherm A should be fixed to satisfy the heat transfer requirement.

$$A_{av} = N_t\, \pi\, d_o\, L$$
$$= 322 \times \pi \times 0.0508 \times 10$$
$$= 513.89 \text{ m}^2$$

Heat duty, $\theta_t = \Delta H_r \times \text{kmol/h of ethanol consumption} = 29 \times 1000 \times \dfrac{103.125}{46}$

$$= 65\,013.6 \text{ kJ/h} \equiv 18.06 \text{ kW}$$

Calculations of fixed bed side film coefficient, h_i:

d_p = equivalent diameter of catalyst cylindrical particle, m

$$\frac{\pi d_p^3}{6} = \frac{\pi}{4}(1.5)^2 \times 5 \times 10^{-9}$$

$$d_p = 0.002\,565 \text{ m}$$

$$\frac{d_p}{d_t} = \frac{0.002\,565}{0.043\,28} = 0.059 < 0.35$$

$$\frac{h_i d_t}{k} = 0.813\ e^{(-6d_p/d_t)}(d_p G/\mu)^{0.9} \tag{10.28}$$

$$G = 0.55 \text{ kg/(m}^2 \cdot \text{s)}$$

$$k = 0.04 \text{ W/(m} \cdot \text{K)}$$

$$\mu = 0.015 \text{ mPa} \cdot \text{s} = 0.015 \times 10^{-3} \text{ Pa} \cdot \text{s}$$

$$h_i = 0.813 \times \left(\frac{0.04}{0.043\,28}\right) e^{(-6 \times 0.059)} \times \left(\frac{0.002\,565 \times 0.55}{0.015 \times 10^{-3}}\right)^{0.9}$$

$$= 31.49 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

For heat transfer in packed beds[2], heat transfer coefficient for the fluid passing through the packed bed can be calculated by Eq. (10.28) or Eq. (10.29). Lesser of the two is selected.

$$\frac{h_i d_p}{k} = 3.6\left(\frac{d_p G}{\mu\,\varepsilon}\right)^{0.365} \tag{10.29}$$

$$\frac{h_i \times 0.002\,565}{0.04} = 3.6\left(\frac{0.002\,565 \times 0.55}{0.015 \times 10^{-3} \times 0.3}\right)^{0.365}$$

$$h_i = 457.51 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Let $h_i = 31.49 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$ (Lesser of two values)

Shell side heat transfer coefficient, h_o:

Let tube pitch, $P_t = 1.25\ d_o = 1.25 \times 0.0508 = 0.0635$ m

Type of tube arrangement = Equilateral triangular

Equivalent diameter, $d_e = \dfrac{1.1}{d_o}(P_t^2 - 0.907\,d_o^2)$

$$= \frac{1.1}{0.0508}(0.0635^2 - 0.907 \times 0.0508^2) = 0.036\,63 \text{ m}$$

Shell side mass flow rate, $\dot{m}$:

$$\phi_t = \dot{m}\,C_L\,\Delta t$$

Table 10.14 Properties of Dowtherm A at 380°C[15]

Property	Value
C_L	2.5832 kJ/(kg · °C)
μ	0.135 mPa · s
k	0.098 W/(m · °C)

Let $\Delta t = 2°C$ for Dowtherm–A

$$\dot{m} = \frac{18.06}{(2.5832 \times 2)} = 3.4957 \text{ kg/s}$$

Density of Dowtherm A at 380°C, $\rho_l = 0.709$ kg/L

Circulation rate, $q_v = \frac{3.4957}{0.709} \times \frac{3600}{1000} = 17.775 \text{ m}^3/\text{h}$

Shell side flow area, A_s can be calculated using Eq. (6.33).
Shell inside diameter, D_s

$$D_b = d_o \left(\frac{N_t}{K_1}\right)^{\frac{1}{n_1}} = 50.8 \left(\frac{322}{0.319}\right)^{\frac{1}{2.285}} \tag{6.1}$$

($k_1 = 0.319$ and $n_1 = 2.285$ from Table 6.2)

$D_b = 1048.46$ mm

Let $D_i = 1065$ mm

Baffle spacing, $B_s = 0.4\, D_i = 426$ mm

$$A_s = \frac{(0.0635 - 0.0508)}{0.0635} \times 1.048\,46 \times 0.426 = 0.0893 \text{ m}^2$$

$$G_s = \frac{\dot{m}}{A_S} = 39.1456 \text{ kg/(m}^2 \cdot \text{s)}$$

Reynolds number, Re:

$$Re = \frac{d_e G_S}{\mu} = \frac{0.036\,63 \times 39.1456}{0.135 \times 10^{-3}} = 10\,621.5$$

Prandtl number, Pr:

$$Pr = \frac{C_L \mu}{k} = \frac{2.5832 \times 0.135 \times 10^{-3} \times 10^3}{0.098} = 3.558$$

From Fig. 6.14, $J_h = 0.0058$ for 25% baffle cut
Using Eq. (6.40),

$$\frac{h_o \times 0.036\,63}{0.098} = 0.0058 \times 10621.5 \times 3.558^{0.33}$$

$$h_o = 250.55 \text{ W/(m}^2 \cdot °\text{C)}$$

Overall heat transfer coefficient, U_o:

$$h_{od} = h_{id} = 5000 \text{ W/(m}^2 \cdot °\text{C)}$$

$$\frac{1}{U_o} = \frac{1}{250.55} + \frac{1}{5000} + \frac{0.0508 \ln (50.8/43.28)}{2 \times 16} + \frac{50.8}{43.28} \times \frac{1}{5000} + \frac{50.8}{43.28} \times \frac{1}{31.49}$$

$$U_o = 23.8356 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$
$$\phi_t = U_o A \, \Delta\mathcal{T}_{\ln} = 18.06 \times 10^3 = 23.8356 \times 513.89 \times \Delta\mathcal{T}_{\ln}$$
$$\Delta\mathcal{T}_{\ln} = 1.474^\circ\text{C}$$

Very low temperature difference is required for the required duty of heat transfer. Hence, area available is adequate for heat transfer. Dowtherm-A will be circulated under pressure above its saturation pressure (8.21 bar a) corresponding to operating temperature of 380°C. This means shell will be under pressure.

Note: Empty volume of catalyst tubes works out to be 4.7272 m^3 (based on 10 m tube length) which is 17.11% in excess over the bulk volume of the catalyst (4.045 m^3). In reality, manufacturing tolerance of tubes (±12.5%) and support arrangement at the bottom of the tubes will call for sufficient excess volume so that required volume of the catalyst can be packed.

Pressure drop of the catalyst packed tubes is usually much higher than that of clean tubes. Manufactures of catalysts provide data relating to pressure drop in the catalyst bed and suggest right size of the tube (i.e., diameter).

Over a period of operation some catalyst may settle or crumble and pressure drop may increase. All these aspects will have to be borne in mind while designing a reactor with the use of a catalyst.

EXERCISES

10.1 Sulphuryl chloride (SO_2Cl_2) formation is carried out in a batch reactor at 0°C and 330 kPa a. Reaction time is 0.6 h.

Reaction:

$$\underset{\text{Sulphur dioxide}}{SO_2(l)} + \underset{\text{Chlorine}}{Cl_2(g)} \rightarrow \underset{\text{Sulphuryl chloride}}{SO_2Cl_2(l)}$$

At the reaction conditions, sulphur dioxide is in liquid state and it is a limiting reactant. Conversion of SO_2 at the end of reaction (at equilibrium) is 60%. Chlorine supply rate to reactor is 10% higher than that consumed. Reaction is carried out in a glass lined reactor. Heat of reaction at 0°C is 75 kJ/mol (exothermic). For the production of 1000 kg of SO_2Cl_2 per batch,

(a) decide the diameter and height of batch reactor.
(b) select the suitable agitator for this gas-liquid reaction and calculate the rpm and power required by the agitator.
(c) calculate the heat duty of overhead condenser. Overhead condenser will recycle almost all excess SO_2 back to the reactor, and
(d) calculate the heat duty of jacket.

10.2 In Example 10.5, a jet reactor is used for hydrogenation of edible oil. Instead of the jet reactor, a reactor with gas induction type stirrer[6] and spiral jacket is to be designed as shown in Fig. 10.17. About 200 kg catalyst (1 to 2 μm particle size) is used with 10 t soybean oil. Use spiral baffled jacket (Fig. 10.9(c)) for heat transfer. Following equation[5] can be used to calculate jacket side heat transfer coefficient.

Fig. 10.17 *Gas Induction-Type Tube Strirrer for Hydrogenation*

For $Re > 10\ 000$

$$\frac{h_j D_e}{k} = 0.027\ Re^{0.8}\ Pr^{0.33}\left[\left(\frac{\mu}{\mu_w}\right)^{0.14}\left(1 + 3.5\left(\frac{D_e}{D_j}\right)\right)\right] \tag{10.30}$$

where, D_e = Equivalent diameter for cross section, m

$D_e = 4\ W$

W = Width of jacket, m

$$Re = \frac{D_e u \rho}{\mu}$$

$$u = \frac{\dot{m}'/\rho}{PW}$$

p = pitch of baffle spiral, m

$\dot{m}'$ = Effective mass flow rate through spiral, kg/s

$\dot{m}' = 0.6\,\dot{m}$

$\dot{m}$ = Actual mass flow rate through spiral jacket, kg/s.

$$D_j = \frac{D_{jo} + D_{ji}}{2}, \text{ mean diameter of jacket, m}$$

Inside heat transfer coefficient can be safely calculated by following equation[6]:

$$\frac{h_i D_i}{k} = 0.36\, Re^{2/3}\, Pr^{1/3} \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{10.31}$$

where, D_i = Inside diameter of shell of reactor, m

If heat transfer area provided hy spiral jacket is not sufficient for the given heat duty, then use internal helical coil. Coil side heat transfer coefficient can be calculated by Eq. (6.19).

For coil, $D_e = d_{ci}$, where, d_{ci} = Inside diameter of coil. Also velocity of fluid through coil, $\upsilon_c = \dfrac{\dot{m}/\rho}{\dfrac{\pi}{4} d_{ci}^2}$.

10.3 Acetic acid is manufactured via carbonylation of methanol. Reaction is carried out at 180°C and 50 atm a in presence of liquid phase rhodium promoted methyl iodide as catalyst. Reaction is carried out in a bubble column reactor. Acetic acid, itself is used as solvent.

Determine the following:

(a) Working volume of reactor.
(b) Diameter of reactor.
(c) Height of liquid inside the reactor.
(d) Heat duty of overhead condenser in which exit gas-vapour mixture is cooled down to 40°C.
(e) Design a spiral baffled jacket around bubble column reactor.

Data

(i) Reaction:

$$CH_3OH(l) + CO(g) \xrightarrow[180°\,C]{} CH_3COOH(l)$$

$$CH_3OH(l) + CH_3COOH(l) \rightarrow CH_3COOCH_3(l) + H_2O(l)$$

(ii) Bubble column reactor can be approximated as an ideal mixed flow reactor. Its uniform composition is 80% acetic acid, 10% methanol, 5% methyl iodide, 2.5% methyl acetate, and 2.5% water (by mass).
(iii) Feed rate of carbon monoxide is 10% higher than that consumed in the reaction.
(iv) Cooling water is available in the plant at 32°C.
(v) Overall rate of reaction does not depend on $K_L a$, if $S_g > 2$ cm/s.
(vi) Rate of chemical reaction

$$-r_A = \frac{1}{V_l}\frac{dN_A}{d\theta} = k\, C_A, \quad k = 89.26 \times 10^{-5}\ s^{-1}$$

C_A = Concentration of methanol, kmol/m^3

(vii) Production rate of acetic acid = 50 tpd
(viii) Heat of reaction at reaction temperature: $\Delta H_r = -137$ kJ/mol
(ix) Conversion per pass = 70%

10.4 Design the fixed bed catalystic reactor for 50 kg/h ethyl chloride plant based on the following data obtained on a pilot plant reactor[16]:

Data of pilot plant reactor:

(i) Feed flow rate = 0.025 53 kmol/h.

(ii) Feed composition

Component	mole %
Methane	86.55
Ethylene	7.40
Hydrogen chloride	6.05

(iii) Feed temperature = 171.1°C

(iv) Reaction is taking place at 171.1°C and 2859.22 kPa g.

$$\underset{\text{Ethylene}}{C_2H_4(g)} + \underset{\text{Hydrogen Chloride}}{HCl(g)} \rightarrow \underset{\text{Ethyl chloride}}{C_2H_5Cl\ (g)}$$

(v) Product composition

Component	mole %
CH_4	90.77
C_2H_4	2.88
HCl	1.47
C_2H_5Cl	4.88

(vi) Catalyst: Zirconium oxychloride supported on silica gel
Bulk density = 650 kg/m^3
Porosity of catalyst bed = 0.32

(vii) $$\frac{m_W}{\dot{m}_F} = 177.62\ \frac{\text{kg catalyst}}{\text{kmol of feed/h}}$$

(viii) Superficial mass velocity of feed gas, G_g = 58.93 kg/(m^2·h)

(ix) Rate of reaction

$$-r_A = 2.62 \times 10^{-4}\ \frac{\text{kg mol of ethylene converted}}{(\text{h} \cdot \text{kg catalyst})}$$

(x) Reaction is exothermic and heat of reaction at reaction temperature is 74 kJ/mol C_2H_4. It can be carried out in a shell and tube heat exchanger type reactor.

Feed composition, feed temperature, reaction temperature, operating pressure, catalyst, etc., are assumed same in pilot plant and commercial scale reactors.

10.5 Design a jet loop reactor for chlorination of benzene.

Stoichiometry

$$C_6H_6(l) + Cl_2(g) = C_6H_5Cl(l) + HCl(g) \quad \Delta H^o_{r1} = -131.5 \text{ kJ/mol}$$

$$C_6H_5Cl(l) + Cl_2(g) = C_6H_5Cl_2(l) + HCl(g) \quad \Delta H^o_{r2} = -124.4 \text{ kJ/mol}$$

Reaction is carried out in jet loop reactor (similar to one shown in Fig. 10.1 (i) at 2.4 bar a and 55°C. Rates of reactions are first order and are given by

$-r_B = k_1C_B$, $r_M = k_1C_B - k_2C_M$ and $r_D = k_2C_M$

where, $k_1 = 1.0 \times 10^{-4}$ s^{-1} and $k_2 = 0.15 \times 10^{-4}$ s^{-1}

Subscripts B, M and D denote benzene, monochloro-benzene and dichlorobenzene, respectively. C_B and C_M are concentrations in mol/L. Ferric chlorile ($FeCl_3$) is used as a catalyst (0.1 g $FeCl_3$/100 g benzene).

Overall conversion of 87% of benzene is to be considered. Assume negligible presence of chlorine in HCl gas stream, escaping the reactor and negligible leftover in the liquid phase.

It is desired to produe 1000 kg monochlorobenzene per batch together with byproduct dichlorobenzene and unreacted benzene. Calculate the following:

(a) Product composition.
(b) Working volume of the reactor.
(c) Considering 20% space for gas over liquid, find diameter and height of reactor.
(d) Heat duty of external heat exchanger.
(e) Capacity of circulation pump.

10.6 Hydrodenitrogenation of straight run heavy gas oil is to be carried out in a refinery in a trickle bed reactor[18,19]. Gas oil contains 900 mg/L nitrogen (as N_2). It is to be reduced to 15 mg/L by passing through a trickle bed reactor. Hydrogen (much in excess) and gas oil are passed cocurrently over a fixed bed of catalyst. Catalyst pellets are made of alumina, coated with nickel and tungsten. Reaction is carried out at 415 bar a and 370°C. Flow rate of gas oil is 5000 kg/h. Liquid Hourly Space Velocity (LHSV) is to be kept at 1000 h^{-1}. LHSV is defined as volume of liquid fed per hour per unit volume of catalyst. Porosity of the catalyst bed (ε) is 0.3. Assume that hydrodenitrogenation reaction is isothermal, irreversible and of first order with respect to reactant concentration.

Rate constant $k_L = 1.624\ 74 \times 10^{-3}$ cm^3 of liquid/(cm^3 of catalyst volume)

Density of gas oil is 800 kg/m^3 at the operating conditions.

Calculate the volume of catalyst bed to be packed in the reactor.

Following assumptions are made:

(i) Plug flow of liquid, i.e., no despersion in the axial direction in the catalyst bed.
(ii) There is no mass transfer or heat transfer limitations between gas and liquid, between liquid and solid catalyst or inside catalyst pellets.
(iii) Catalyst pellets are completely wet (i.e., bathed) with the liquid.
(iv) No vaporisation or condensation takes place.

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Process Design of Utility Systems

11

11.1 PREAMBLE

Every manufacturing industry is dependent on one or more utilities. Role of process utilities in chemical industry is of great importance. While it is observed that most large chemical complexes have well-designed process utility systems, small and medium scale chemical industry rather undermine the importance of utilities and they suffer; at times heavily, in operation in terms of production loss and product cost. Steam, heating by thermic fluid, cooling water, refrigeration, compressed air, inert gas, etc., are needed at various stages of production and careful design of these systems can pay rich dividends. This chapter is devoted to process engineering aspects of the utility systems. This will include the major parameters that are required for preparation of specifications of a utility system and its operation thereof. Equipments, required to provide these utilities, are described at suitable locations without going into the mechanical design aspects of the same.

11.2 PROCESS HEATING SYSTEMS

Unit operations as well as unit processes require heating. It is carried out by direct firing, supplying vapour condensation systems (steam and Dowtherm® type), thermic fluid (hot oil), hot water and hot air. While hot air and hot water systems are less common, steam and thermic fluid heating are most commonly employed in chemical industry. Spray dryers and furnaces for metal processing use hot air or hot flue gas for heating. Many heat sensitive product processing call for hot water heating. Except for such specialised applications, steam or thermic fluid are common heating mediums. Fig. 4.9(a), (b) and (c) show such heating applications in a continuous stirred tank reactor (CSTR).

11.2.1 Steam Heating

Use of steam is by far the most common in industry. Reboilers of distillation columns, jackets/limpet coils of CSTR or other reactors, desorption columns, evaporators, dryers, indirect heating applications, activation of carbon and numerous other applications can be listed where steam is used as a heating medium. Some use steam indirectly while a

few use live steam. Steam is non-toxic and inflammable and its leak in saturated state can be seen visually so that steps can be taken to stop the leakages.

Study of properties of steam will reveal some interesting facts. Two important facts are worth noting. Saturation temperature increases and latent heat of vapourisation is reducing with increase in steam pressure. These are rather contradictory parameters for heating applications. Hence, selection of steam temperature for heating application is critical. Unnecessary high temperature of steam may not be preferred in design as it will call for higher design pressure of the equipment. Also in heating applications, mostly latent heat of vapourisation (λ) is utilised while sensible heat is lost in condensate. Thus at higher pressure, more energy is wasted in the form of hot condensate. In most steam heating applications, condensing steam temperature is selected 20 to 25°C higher than the temperature requirement of the process fluid.

Consider manufacture of amino and alkyd resins. In the manufacture of amino resins temperature to be maintained in the reactor is in the range of 95 to 120°C while in the manufacture of alkyd resins, temperature requirement varies in the range of 220 to 240°C. Thus for amino resin manufacture one would try to use steam at 145 to 150°C (i.e., steam at saturation pressure in the range of 3.7 to 4.2 bar a). This selection is considered appropriate and one would design steam distribution system for about 5 bar a. If the same considerations are followed for alkyd resin manufacturing, steam temperature in the range 240 to 260°C would be required which will mean saturated steam in the pressure range 34 to 50 bar a will be required, Clearly design of steam equipment will be therefore at much higher pressure and its cost would be high. Steam at 4 bar a has $\lambda = 2133$ kJ/kg while at 50 bar a, $\lambda = 1639.7$ kJ/kg. This means energy usage will be lower by at least 23% at higher pressure. Thus for the manufacture of alkyd resin, use of thermic fluid or other condensable vapour should be considered rather than steam.

Consider a distillation system in which methanol-water is to be separated. It is clear that bottom temperature in the distillation column could be around 105°C, depending on pressure drop in the column. Therefore steam temperature in the reboiler should be kept at about 125 to 130°C (i.e., in the pressure range of 2.3 to 2.7 bar a). This is a common experience in industry. In vacuum creating systems, steam at 3.5 to 4 bar a with a superheat of about 10°C is commonly used as a primary fluid stream. These examples would suffice to justify the importance of steam pressure. Process engineer should therefore study the heating temperature requirement and select the right steam pressure. Saturated steam up to about 20 bar a (saturation temperature: 212.4°C) is recommended for process heating beyond which equipment cost becomes high due to high design pressure.

Steam in superheated condition is not preferred for indirect heating system. This is because superheated steam acts as a gas and heat transfer (film) coefficient of the superheated phase would be much lower than that for condensing. Much of the heat transfer surface would therefore be required for first desuperheating steam. This will increase the cost of heating equipment. However, live superheated steam at near atmospheric pressure is used for heating and activation of carbon as condensation of steam on carbon is not desired in the equipment. Similarly, regeneration of desiccant

beds can also be carried out with live superheated steam. Thus an application decides the steam conditions for use.

Use of wet steam for heating is not desired. This is because sensible heat (h) of condensate is much lower than latent heat of vapourisation (λ). For example, at 3 bar a, h = 561.4 kJ/kg while λ = 2163.2 kJ/kg. When steam is conveyed over a long distance, there could be condensate formation in the pipe and steam may have significant wetness. Under such circumstance, first moisture of steam should be removed before introducing to the heater. When steam pressure reduction is desired, a separator is provided on upstream pipe to ensure that saturated steam enters the pressure reducing valve. In this case in addition to the possibility of carry-over of moisture to the heater, erosion of internals of the pressure reducing valve can be experienced. A typical pressure reducing station is shown in Fig. 11.1.

Fig. 11.1 *Typical Steam Pressure Reduction System*

Steam is generated in a boiler. In small and medium scale chemical industry, steam generation is carried out up to a pressure of 12 to 15 bar g. Treated water is used in the boiler to avoid build up of solids in the steam drum which can cause fouling and/or corrosion. Requirements of boiler water quality are specified in IS:10 392 for such boilers. Blowdown from the boiler is maintained to meet these requirements. Scale build-up in the boiler will reduce heat transfer, leading to increase in flue gas temperature thereby waste of energy. Even explosions of boilers are reported due to excessive fouling. Combustion of fuels and steam generation are dealt in many books and reader is advised to refer them. Stoichiometric aspects of combustion are dealt at a length in Ref. 1.

Generation of steam at high pressures call for high quality boiler feed water. Quality requirements for boilers generating steam for turbines (i.e., for power generation) are extremely stringent as carry-over of solids (in particular silica) mean heavy erosion of steam turbine rotors.

Insulation of steam system is highly recommended. Apart from safety of the personnel, condensation of steam without utilisation in the heater can be avoided. Variety of insulating materials are in use; such as calcium silicate, mineral wool, etc.

Cascading of steam system is found very useful in its efficient utilisation. Superheated steam at high pressure and temperature is first utilised for power generation. Back pressure steam from the turbines can be used for heating applications. Different flow sheets can be developed for cascading which can result in optimum steam generation. Reference 1 describes several case studies of steam cascading systems. Such systems are common in heavy chemical plants, refinery, sugar mills, paper industry, etc. A captive power plant based on steam turbines, followed by use of saturated steam at lower pressures for heating can be ideal in many chemical plants to improve overall economy. Process engineer should explore the possibility of a cascading steam balance to utilise steam efficiently. Figure 4.4 is an example of a typical cascade steam system.

Use of a thermocompressor can be advantageous in reducing steam consumption. Ejectors are commonly used as thermocompressors. However, mechanical vapour compressors are also employed in industry. Twin lobe blowers are popular for use as mechanical vapour compressors. Since mechanical compressor discharges steam at higher temperature, compression ratio is usually low; say 1.2 to 1.3. However, even this compression of steam can be useful in a creating sufficient temperature difference across the heat transfer surface for the desired heat duty. In Sec. 8.9.1(*f*), applications of an ejector for reboiler steam are presented.

In multiple effect evaporators, thermocompressors are effectively used to improve steam economy. Concentration of effluents, concentration of sodium sulphate solution and many other application can be listed for which multiple effect evaporators are used with low pressure steam and thermocompressor. Following example will demonstrate the application.

Example 11.1 Sodium sulphate solution is produced in sizable quantity in a plant manufacturing sebacic acid. Strength of the feed solution is 8% by mass. In one plant, triple effect evaporator system is to be installed for concentrating solution to its saturation level. Slurry of Na_2SO_4 solution is obtained which is passed through a Oslo crystalliser where crystals of Na_2SO_4 having 10% moisture (by mass) is obtained. Saturated mother liquor from the crystalliser at the rate of 315 kg/h, containing 30% (by mass) Na_2SO_4, from the last effect is recycled and mixed with the fresh feed. Proposed system is shown in Fig. 11.2 below.

Two designs are being considered. In one design, saturated steam at 1.3 bar a is used in the first effect for heating. In second design, part of the vapour from first effect is compressed to 1.3 bar a in a thermocompressor (an ejector) with the help of motive saturated steam at 7 bar a. Last effect operates at 0.125 bar a (under vacuum) which is maintained by steam ejectors and a surface condenser. The condenser uses cooling water at 32°C which limits the pressure in the last effect. Anticipated operating conditions for both designs are listed in the following table:

Fig. 11.2 *Triple Effect Evaporator System for concerntrating Aqueous Sodium Sulphate Solution*

Table 11.1 Anticipated Operating Conditions of Evaporator Effects

Parameter	Average heat capacity of solution, C_p kJ/(kg·°C)	System without ejector			System with ejector		
		Temperature of Solution, °C		Pressure kPa a	Temperature of Solution, °C		Pressure kPa
		Liquid*	Cond. Vapour		Liquid*	Cond. Vapour	
1st Effect	0.95	103	100	101.325	97	95	84
2nd Effect	0.9	81	78	44	77	74	37
3rd Effect	0.7	56	50	12.5	56	50	12.5

*with elevation of boiling point

Thermocompressor uses motive saturated steam at 7 bar a and compresses 0.725 kg water vapour (at 84 kPa a) from the first effect per kg motive steam to 1.3 bar a. Assume heat of crystallisation in the last effect to be 20 kJ/kg Na_2SO_4 (exothermic). Make stoichiometric calculations for a feed rate of 5400 kg/h of aqueous solution containing 8% Na_2SO_4 at 35°C and find steam consumptions for both the designs.

Solution

Basis: Aqueous solution feed rate of 5400 kg/h containing 8% Na_2SO_4

Let $\dot{m}_s$ = Fresh steam supply at 1.3 bar a, kg/h

$\dot{m}_1$ = Solution flow rate leaving 1st effect, kg/h

$\dot{m}_2$ = Solution flow rate leaving 2nd effect, kg/h

Na_2SO_4 content of feed solution = 5400 × 0.08 = 432 kg/h

Crystals of Na_2SO_4 are produced with 10% moisture.

Moist crystal flow rate from crystalliser = 432/0.9 = 480 kg/h

Mother liquor rate = 315 kg/h with 30% or 94.5 kg/h Na_2SO_4

Mother liquor is recycled to the fresh feed and combined feed enters first effect.

Combined feed rate = 5400 + 315 = 5715 kg/h

Total solid in combined feed = 432 + 94.5 = 526.5 kg/h

Average concentration of Na_2SO_4 in the combined feed = 526.5 × 100/5715 = 9.21%

Total evaporation in all three effects = 5715 – 480 – 315 = 4920 kg/h

Table 11.2 Properties of Steam[1, 2]

Temperature, °C	Satd. steam pressure p, kPa a	Enthalpy*, kJ/kg	
		Sensible (h)	Latent (λ)
165	700	697.1	2064.9
107.1	130	449.2	2237.8
100	101.325	419.0	2256.9
95	84	397.3	2270.7
78	44	327.3	2313.4
74	37	309.8	2329.9
50	12.5	210.3	2382.3

*Reference state: Enthalpy of saturated liquid water at 273.15 K (0.01°C) = 0 kJ/kg

A. Case without Thermocompressor

Enthalpy balance equations for three effects can be written as under.

$$\dot{m}_s \times 2237.8 = 5400 \times 0.95(103 - 35) + 315 \times 0.7(103 - 56) + (5715 - \dot{m}_1)2256.9 \quad \text{(I)}$$

$$(5715 - \dot{m}_1)2256.9 = \dot{m}_1 \times 0.9(81 - 103) + (\dot{m}_1 - \dot{m}_2)2313.4 \quad \text{(II)}$$

$$(\dot{m}_1 - \dot{m}_2)2313.4 = \dot{m}_2 \times 0.7(56 - 81) + (\dot{m}_2 - 480 - 315\ )2382.3 - 480 \times 0.9 \times 20 \quad \text{(III)}$$

Solving Eq. (I), (II) and (III),

$$\dot{m}_s = 1827.10, \quad \dot{m}_1 = 4062.52 \text{ and } \dot{m}_2 = 2415.63 \quad \text{all in kg/h}$$

Average concentration of Na_2SO_4 in the solution, leaving first effect will be 12.96% and leaving second effect will be 21.8%.

Specific steam consumption = 1827.10/4920 = 0.3714 kg/kg evaporation

Steam economy = 4920/1827.10 = 2.6928 kg evaporation/kg steam

B. Case with Thermocompressor

In this case, vapour amounting 0.725 $\dot{m}_s$ is recycled through thermocompressor.

$$\dot{m}_s \times (2064.9 + 697.1 - 449.2) + 0.725\dot{m}_s \times 2270.7 = 5400 \times 0.95(97 - 35) + 315 \times 0.7(97 - 56) + (5715 - \dot{m}_1)2270.7 \quad \text{(IV)}$$

$$(5715 - \dot{m}_1 - 0.725\dot{m}_s)2270.7 = \dot{m}_1 \times 0.9(77 - 97) + (\dot{m}_1 - \dot{m}_2)2329.9 \quad \text{(V)}$$

$$(\dot{m}_1 - \dot{m}_2)2329.9 = \dot{m}_2 \times 0.7(56 - 77) + (\dot{m}_2 - 480 - 315)2382.3 - 480 \times 0.9 \times 20 \quad \text{(VI)}$$

Solving Eq. (IV), (V) and (VI),

$$m_s = 1305.37, \quad m_1 = 3583.08 \text{ and } m_2 = 2400.00 \quad \text{all in kg/h}$$

Vapour compressed = 0.725 m_s = 0.725 × 1305.37 = 946.4 kg/h from first effect

Average concentration of Na_2SO_4 in the solution, leaving first effect will be 14.69% and leaving second effect will be 21.94%.

Specific steam consumption = 1305.37/4920 = 0.2653 kg/kg evaporation

Steam economy = 4920/1305.37 = 3.769 kg evaporation/kg steam

Reduction in steam consumption = 1827.10 – 1305.37 = 521.73 kg/h or 28.56%

Note that incorporation of the thermocompressor results in significant reduction of fresh steam input. If low pressure (at 1.3 bar a) steam is used as fresh steam, a mechanical vapour compressor can be provided to compress vapour amounting 946.7 kg/h from the first effect (from 84 kPa a to 130 kPa a) and similar results can be achieved. Compression of vapour can be achieved in a twin lobe compressor which may consume about 40 kW power.

Saturated steam condensate when discharged from a steam trap results in flash steam due to pressure reduction. Quantum of flash steam depends on pressure reduction across the trap. Therefore a two phase flow is realised after the trap. It is a practice to convey both the phases simultaneously to a deaerator or to the boiler feed tank in the pipe size correctly sized and correct size trap is installed. Use of Fig. 5.17 can be made to size the pipe. Steam trap manufacturers provide trap selection chart for

a desired pressure drop across the trap and flow rate of condensate. Amount of flash steam for a given pressure drop for saturated condensate can be easily calculated by a simple energy balance. Figure 11.3 is the graph to be used as a guideline for flash steam estimation.

Fig. 11.3 *Flash Steam Production from Condensate*

11.2.2 Combined Heat and Power Systems

Competitive pressures to reduce cost of production has led to think of Combined Power and Heat (CHP) systems. This is also known as *Cogeneration*. When effectively applied, a CHP system can make a facility less dependent on the electrical grid or, more commonly, provide increased reliability for a strategic portion of the electric load at the site. Such a system produces less emissions per delivered unit of output which is a part of green engineering.

CHP or cogeneration is the sequential production of two forms of useful energy from a single fuel source. In most CHP applications, chemical energy in fuel is converted to both mechanical and thermal energy. The mechanical energy is generally used to generate electricity, while the thermal energy or heat is used to produce steam, hot water, or hot air. The principal technical advantage of a CHP system is its ability to extract more useful energy from fuel compared to traditional energy systems such as conventional power plants that only generate electricity and industrial boiler systems that only produce steam or hot water for process applications. Several CHPs can be thought of depending on thermodynamic possibilities and the need of the user. One typical generalized system is shown in Fig. 11.4.

Carnot engine[3] for power generation is the ideal machine and assumes two adiabatic steps. In the isothermal step at high temperature T_H, heat Q_H is absorbed by the working fluid in the engine and in the isothermal step at lower temperature T_C, heat amounting Q_C is discarded by the fluid. Work produced by the Carnot engine is

$$W = Q_H - Q_C \tag{11.1}$$

Gas Turbine Fuel Options	Options of Using Mechanical Energy	Examples of Heat-Recovery Option-I	Examples of Heat-Recovery Option-II	Potential Air Emissions
Liquid Fuel * Distillate Oil * Residual Oil * Naphtha Gaseous Fuel * Natural Gas * Refinery Gas * Biogas * LPG	* Electrical Generator * Fan * Pump Compressors * Air * Refrigerant	* Boiler Fired HRSG Unfired HRSG * Recuperator Gas-to-Liquid * Heat Exchanger (Feed Water Heating) * Absorption Chilling * Desiccant Dehumidification	* Boiler Feed Water Heating * Combustion Air Presheating * Power Generation using Kalina Cycle * Hot Water Generation	NO_x SO_2 PM CO CO_2 HAP VOC

Fig. 11.4 *Typical CHP System Options*

Thermal efficiency (η) of the Carnot engine is

$$\eta = W/Q_C = 1 - (T_C/T_H) \tag{11.2}$$

Since Carnot engine assumes both steps reversible, its efficiency is highest among all thermodynamic cycles of power generation.

For a Carnot engine, operating at $T_H = 773.15$ K and $T_C = 318.98$ K, engine efficiency (η) can be calculated at

$$\eta = 1 - (318.98/773.15) = 0.594 \text{ or } 59.4\%$$

No actual thermodynamic cycle of power generation is fully reversible. For a typical Rankine cycle[3], operating between these temperatures and other specific steam parameters and turbine efficiency of 100%, cycle efficiency is just 39.6%. For a steam turbine of 75% efficiency, the cycle efficiency drops to only 29.6%. Rest 70.4% energy is lost to atmosphere. It can be noticed that actual efficiency of the power cycle based on Rankine cycle is just 49.8% of that of Carnot cycle. Similarly, in boilers generating steam, overall efficiencies in the range of 60 to 70% (on GCV basic) are achieved. This is the basis of CHP system where energy is efficiently utilised by extracting energy in two forms. One can get as high as 80+% overall efficiency of CHP with proper design which means overall energy loss will be just 20% or less (mostly in flue gas and radiation).

Most CHP applications use gas turbines or reciprocating engines coupled to an electrical generator. Heat recovered from these prime movers is then used to produce steam or hot water to meet space heating or cooling requirements or to control humidity. These systems may include heat exchangers, absorption chillers, desiccant dehumidifiers, or other types of heat-recovery equipment. In another common CHP application, boiler steam passes through a steam turbine to generate electricity. Heat rejected from the steam turbine is then used for process applications. This suggests that one of the simplest strategies for implementing cogeneration technology is to replace steam pressure-reducing valves with backpressure steam turbines and electrical generators. Earlier mention of Fig. 4.4 was made which itself is an example of cogeneration. Newer technologies are being developed to improve overall efficiency of cogeneration. Use of low grade heat (i.e., primarily at low temperature) for power generation by Kalina cycle[4], extracting more heat from flue gas of a boiler by providing recuperators of suitable material of construction, use of hot water in process (instead of steam), etc., can be named under this category. From a practical viewpoint, CHP systems can be of any size and there is no limit as to the ratio in which the two types of energy are proportioned. In addition, power generated by a CHP system can be used on-site or exported, and the thermal energy can be used for heating, cooling, or both. Technical feasibility involves more than applying appropriate engineering principles in the design of a CHP system to meet performance criteria. Safety and reliability of the system are considered most important while selecting a CHP system. Next parameter is the availability of a fuel which should suit the requirements of the selected CHP system. Nevertheless, CHP system is very important for chemical process industry and its use must be critically evaluated by the process engineer in design.

11.2.3 Thermic Fluid Heating Systems

Hot oil heating system is next popular system in small- and medium-scale chemical industry in India. In earlier section, it was noted that steam is rarely used for heating beyond 200°C due to (i) high pressure equipment design requirements, and (ii) boilers generating steam above 24 bar a are expensive. Commonly, a hot oil system is used in plants where there are many relatively small high-temperature heating requirements. Instead of building several small fired heaters or providing small oil heating systems, it can be more economical to supply heat to the process from circulating hot oil streams and build a single fired heater that heats the hot oil. Use of hot oil reduces the risk of process streams being exposed to high tube-wall temperatures that might be experienced in a fired heater. Such heating systems are preferred even for relatively low temperature heating in small and medium-scale chemical industry where applications involve heating at low as well as at high temperatures. These systems do not come under the purview of Indian Boiler Regulations. Desired properties of thermic fluid are as given below.

(a) Fluid should be non-toxic, inflammable and non-corrosive.
(b) Fluid should be operable at high temperature without degradation in the operating temperature range.
(c) Fluid should offer good film coefficient so that heat transfer area is limited.
(d) Fluid should have low vapour pressure.
(e) Fluid should have low pour point and high auto-ignition temperature.

Earlier it was seen that steam heating is unsuitable for alkyd resin manufacturing. For such a system, hot oil system can suit the requirements. As an alternate, an eutectic mixture of diphenyl-diphenyl oxide, abbreviated as DP/DPO (and popularly known as Dowtherm A®), condensing at 257.1°C at atmospheric pressure, can be considered without any pressure problem. DP/DPO is thermally stable and is usually operated in a vaporisation-condensation cycle, similar to the steam system. Condensed liquid DP/DPO is sometimes used for intermediate temperature heating requirements. However, λ of the condensing organic DP/DPO vapour is 296.2 kJ/kg at atmospheric pressure. This will mean condensing rate of the vapour would be about 5.6 times that of steam at 50 bar a. Cost of organic mixture is substantially high and any leakage of the mixture from the system would be a costly affair. DP/DPO mixture can be safely considered up to 400°C at which saturation pressure is 10.63 bar a and latent heat of vapourisation (λ) is 206.3 kJ/kg. It can be realised that steam is the preferred heating medium because of its high latent heat of vapourisation (λ) and low cost.

Properties of some of the thermic fluid in use in India are given in Table 11.3. Both mineral based and synthetic oils are in use.

Table 11.3 Properties of Thermic Fluids

	General purpose	High temperature applications	Food grade*	Heating/ cooling (combined) applications	DP/DPO mixture
Operating temperature, °C	0 to 305	0 to 345	–39 to 345	–80 to 260	13 to 400
Density (ρ), kg/m^3					
at 20°C	0.87	1.01	0.97	0.76	1.06
at 300°C	0.67	0.81	0.76	0.56 at 250°C	0.85
Kinematic viscosity (γ), mm^2/s					
at 20°C	108	122	7	1.7	1.02
at 300°C	0.6	0.5	0.33	0.26 at 250°C	0.27
Heat capacity, (C_p) kJ/(kg·°C)					
at 20°C	1.92	1.55	1.68	2.10	1.55
at 300°C	2.96	2.57	2.62	3.05 at 250°C	2.32
Thermal Conductivity, (k) [W/(m·K)]					
at 20°C	0.137	0.111	0.121	0.110	0.14
at 300°C	0.111	0.095	0.091	0.065 at 250°C	0.1
Fire point, °C	238	216	154	71	127
Auto-ignition temperature, °C	368	400	405	277	621
Pour point, °C	–48	–32	–48	< –85	12
Normal boiling point, °C	356	369	289	192	257.1
Average molar mass (M)	318	252	207	180	156

* US FDA approved

Properties listed in Table 11.3 are for synthetic thermic fluids. However, mineral oils are also available for use as thermic fluids. In a petroleum refinery, mineral oil from the operating stream is drawn and used for heating.

A typical P&I diagram of the oil heating system in use is given as Fig. 11.5.

Thermic fluid is pumped and is heated in fuel fired heater. Hot oil is sent to various heaters and its flow through each heater is controlled individually by throttling a valve on return line. Return oil is sent to a degassing tank and returns to the circulation pump. Location of the degassing tank is at an elevation above all heating equipments. This tank allows removal of any vapour formation along with non-condensables. All pipes are well insulated. Quality of thermic fluid must be checked periodically for degraded products and replacement is planned, if required.

Fig. 11.5 *P&I Diagram for Thermic Fluid Heating System*

In Fig. 4.9(a) thermic fluid is proposed for heating and cooling in CSTR. For this purpose, oil, proposed in the fourth column of Table 11.3 can be recommended.

Solid, liquid or gaseous fuel is used in the fired heater. Firing is controlled by fuel flow to the heater. Generally, the system is known as 'Thermo Pack' and is rated in terms of heat release rate in kW. Usually thermo packs are operated up to 80% duty of the design heat rating. Over a period, thermal efficiency of the heater get reduced due to fouling in the heater which raises flue gas temperature. It is recommended that flue gas temperature be controlled below 300°C.

11.2.4 Hot Air Systems

Hot air is produced by heating air at positive pressure in a fired heater. Hot air, so produced is used in spray dryers, tray dryers, in buildings for heating, etc. Air has low heat capacity and therefore large circulation for heating application is required which calls for large ducts to convey so that pressure drops are minimal. It is not recommended for indirect heating applications as its heat transfer coefficient is low and large heat transfer area will be required for the heater. A typical P&I diagram of air heating system is shown in Fig. 11.6.

Spray dryers are in use for producing dye powder, powders of pharmaceutical products, food products (such as tomato powder, catechu powder, etc.). Filtered hot air is extensively used in this equipment.

11.2.5 Hot Water Systems

As noticed earlier, hot water at high temperature is less preferred over steam. However, in some special cases, hot water system is employed. To exemplify, in extraction of herbs with an organic solvent, hot water is recommended. It is considered 'soft heating'. Hot water is also used in heating and ventilation equipment for air conditioning. Heating system is similar to the oil heating system. However, water pressure is normally kept about 10 bar higher than saturation pressure at the maximum operating temperature. Multistage centrifugal pumps, similar to boiler feed water pumps, but usually of stainless steel construction, are used for circulation. This system is prone to corrosion and fouling and so quality of water to be used for circulation must be very good. Use of demineralised water is recommended and dissolved oxygen (air) is removed by degassing or by chemical means.

Crude mentha oil is fractionated below 100°C under vacuum. Reboiler for the fractionating equipment is a falling film heater in which either hot thermic fluid or hot water can be used as heating medium. It is claimed that reboiler, heated with hot water, gives better recovery of products with less reject at the bottom.

11.2.6 Thermal Insulation for Hot Surfaces

Energy cost is one of the major concerns to industry. Various measures are adopted by the industry to reduce energy consumption. One of the important measures is provision of adequate thermal insulation to all hot surfaces. Consider a case of 50 mm (2 in) NB bare pipe carrying steam at 10 bar a (saturation temperature = 180°C). Assume ambient temperature as 30°C which means a temperature difference of 150°C. For this case, heat loss from bare pipe is estimated at 465 to 475 W/m. If this pipe

Fig. 11.6 *P&I Diagram for Solid Fuel Fired Hot Air System*

is provided with good insulation, the loss can be reduced to say 50 W/m. In simple terms, energy equivalent to 420 W/m can be saved by providing insulation. This loss could be as high as 2000 W/m for a 300 NB (12 in) pipe.

Several insulating materials are used in industry. For hot surfaces, calcium silicate, mineral wool, glass wool, saw dust, wood fibre, etc., are in use. Among these, mineral wool is the most commonly used insulating material in industry. Rock materials are blended, melted at about 1600°C and fibres are spun of the blend. Fibres are resin bonded and a felt is produced of desired density and resilience. Metallic wire mesh is used to stitch to produce mats of desired thicknesses. Typical thermal conductivities of mineral wool having 150 kg/m^3 density at 50°C and 200°C are 0.046 and 0.075 W/(m · °C), respectively. At first the surface is cleaned to make it free from oil, dust, etc. If the surface is of stainless steel, aluminium foil of about 0.1 mm thickness is applied. Over the pipe resin bonded mineral wool mattresses are wrapped. Finally, aluminium sheet (22 to 24 SWG, i.e., 0.61 to 0.71 mm thickness) with self-screws is wrapped over the mats.

As explained above increasing thickness of insulation can reduce heat loss. However, cost considerations restrict insulation thickness. Table 11.4 gives recommended economic insulation thickness for mineral wool mats.

Table 11.4 Economic Insulation Thickness for Rockwool Insulation*

Nominal pipe size		Recommended thickness, mm Temperature range, °C				
mm	in	50–100	101–200	201–250	251–300	301–400
15/20	½/¾	25	40	40	50	80
25	1	23	40	50	50	80
40	1½	25	40	50	65	80
50	2	25	40	50	65	80
80	3	25	40	50	80	100
100	4	40	50	65	80	100
150	6	40	50	65	80	100
200	8	40	50	75	100	125
300	12	40	50	75	125	150
400	16	50	75	75	125	150
500	20	50	75	100	125	150
600	24	50	75	100	125	175

* Ambient temperature is assumed to be 30–35°C.

For curved surface above 600 mm nominal diameter and flat surfaces, mattresses of thickness, mentioned for 600 mm NB, are recommended but metallic clits are recommended on the surface to hold them in place. Process engineer should provide for economic insulation requirements in design for all heating applications.

11.3 PROCESS COOLING SYSTEMS

Cooling of process fluids is carried out with the help of different fluids. When a process stream is to be cooled, its heat can be utilised by exchanging heat with other colder streams which may form a heat exchanger network. Such utilisation results in energy conservation, also known as *energy integration*. However, such an exchange is possible up to a level to recover heat economically. These aspects were discussed in Section 6.6.2 in which it was indicated that an external coolant will be needed beyond a pinch point to cool a stream. Where heat exchanger network is non-existent, stream cooling below less than 100°C to near ambient temperature, cooling medium is used in most cases. Cooling water or ambient air can be considered for the purpose; the latter can be an expensive proposition.

11.3.1 Air Cooling

Where water scarcity is experienced, air cooling is considered. Design of heat exchangers with air and water was dealt in detail in Chapter 6 where it was observed that heat transfer area requirement of an air cooler is higher than that with cooling water. Air coolers are usually finned tube heat exchangers with induced or forced draft air fans.

There are certain advantages of cooling with air:

1. Use of air cooler enables plant location independent of a water source.
2. Cost of treating cooling water is a factor that is usually high and it requires intricate knowledge of water chemistry. Air cooling does not need such hassle.
3. Air cooler can be placed near the process equipment which avoids cooling piping network.
4. Maintenance requirements are also relatively less for the air coolers.
5. During a power failure, although fan will stop air supply to the cooler but still some cooling will continue because of ambient air. However, in case of cooler with cooling water, supply of cooling water stops immediately and cooling effectiveness will totally be lost.
6. Fouling of a heat exchanger using cooling water is a perennial problem which calls for frequent cleaning of tubes. For this reason, excess heat transfer area of the cooler is provided. In comparison, air cooler fins are less prone to fouling and calls for less cleaning requirement.
7. Air is passed over the fins and process fluid (could be at higher pressure) is passed through the tubes which call for lower thickness of tubes as well as of tube sheets.
8. Size of an air cooler is usually is much bigger than a water cooler, calling for more ground area. However, air cooler can be placed at an elevation (say above the building) to save the space. For placing the air cooler at an elevation, however, civil structure should carefully be designed.

Despite above advantages, cooling with air is less preferred than water cooling in a tropical country mainly because the process fluid can be cooled to a higher temperature with air than with cooling water. Consider a location in India where air

attains 42 to 44°C dry bulb temperature and say 28°C wet bulb temperature (max.) in summer. A cooling tower at this location can deliver cooling water at 32°C. Process fluid at such a location can be cooled up to 55°C with air and up to 40°C with cooling water, a difference of 15°C in cooling. This is a significant advantage in most cooling systems. Besides, air cooler need be located in an open area so that required quantum of ambient air can be made available.

Several designs of air coolers are available (Fig. 6.36). Amongst these, cooler with forced draft air fan are more common. These fans take relatively cold ambient air in suction and therefore require somewhat less power than that is required for induced draft fans. Refrigerant condenser (Exercise 6.10), surface condenser for a steam turbine, compressed air/gas cooling for small compressors, etc., are some industrial applications of air coolers.

11.3.2 Water Cooling

Once through cooling systems are less prevalent in industry than a closed cooling water circuit. In most laboratories, cooling in glassware is carried by raw water flowing in once through manner. Sea water is used as a cooling medium in once through manner in large industrial plants (petroleum refinery, power plant, etc.) located near sea. Due to high salinity of sea water, it is highly corrosive and hence heat exchangers with tubes of special material of construction (say Admiralty brass or Cu-Ni alloys) making such exchangers expensive. Special care is also required to ensure clean sea water (free from fish and other external materials) pumping to such exchangers. Slime formation in tubes of a heat exchanger is a common problem when sea water is used. Despite all these problems, sea as large heat sink attracts industry to use sea water for cooling.

Closed cooling water circuits are more common in industry. Natural and forced-draft cooling towers are generally used to provide the cooling water required on a site. Cooling water is used both; in direct contact equipment and in heat exchangers in indirect manner. In an isolated case, cooling water can also be used for heating; e.g., evaporation of liquid ammonia at –33°C can be carried out with cooling water (refer to Example 2.10). Except for such isolated cases, cooling water is mainly used for cooling a process stream. A typical cooling circuit is represented in Fig. 11.7.

The minimum temperature that can be reached of cooling water depends on the local climate. Cooling towers function by evaporating a part of the circulating water to ambient air, causing the remaining water to cool. If the ambient temperature and humidity are high, then a cooling water system will be less effective. Most cooling towers have induced draft fans but cooling tower with forced draft fans or no fans are not uncommon. Spray ponds are also in use for circulating water in some air conditioning systems.

Difference in inlet cooling water temperature to and outlet cooling water temperature from cooling tower is called a *range* of cooling tower. Difference between outlet cooling water from cooling tower and ambient wet bulb temperature is called *approach*. Usually, cooling towers are designed for 5 to 8°C range. Design approach of a cooling tower is important and it should not be selected low. Usually, 4 to 5°C approach is specified for the design. Cooling tower supplier can furnish a graph in which predicted cooling water temperature can be read for different wet bulb temperature (than design) for the supplied model.

Fig. 11.7 *Typical Cooling Water Circuit*

Considering importance of this utility, usually two cooling water pumps are installed, each of full capacity of which one is standby. Suction pipe size of the pump is kept at least one size higher than that of suction nozzle size of the pump. Similarly, discharge size pipe be kept at least same size that of discharge nozzle size of the pump or higher. Non-return valves are provided in the discharge of each pump.

It is a good practice to provide a side stream filter for filtering cooling water. Its capacity could be 3 to 5% of design recirculation flow rate. Different types of filters are provided. Amongst these, a stratified sand bed filter and a bag filter (20 to 40 micron) are common. Clarified water from outlet of the filter is recycled to cooling tower sump. Turbidity in the circulating water should be checked once a day. It should preferably be below 5 mg/L on silica (as SiO_2) scale.

Cooling water is usually made up of clarified water with dissolved solids (DS) less than 500 mg/L. Because of evaporation of some water from the cooling tower, concentration of DS in cooling water increases. Ratio of DS in cooling water to that in make up water is known as *cycles of concentration.* For calculation of this parameter, normally chloride (as Cl) or silica (as SiO_2) ions are selected. It is desirable to keep cycle of concentration less than 3.5 so that fouling can be controlled. While some water droplets leave with evaporated vapour (known as drift loss), intentional blow down is maintained to keep cycles of concentration under control. Apart from cycles of concentration, other parameters; such as *p*H, hardness (as $CaCO_3$), chlorides, silica and sulphates (as SO_4) need be controlled in the cooling water. Need for control for these parameters is based on corrosion control and scale formation prevention. Cooling water chemistry is quite complex and it is suggested that standard reference books[7,8] on the subject may be referred. Chemicals such as phosphonates are being added to cooling water for control of fouling. Biocides are added to control bacterial growth in tubes Dichromates and/or phosphates are added to control corrosion, However, addition of these chemicals cause problems while handling blow down. For example, destruction of Cr^{+3} or Cr^{+6} must be carried out in the blow down water before disposing it as effluents. Biocides should preferably be biodegradable.

It is a common practice to select cooling water on tube side while designing a shell and tube heat exchanger. Desired velocity of cooling water be kept 1 m/s (min.) so as to get good film coefficient and also reduce possibility of fouling. Number of passes on tube side is based on this parameter. Cleaning of tubes is carried out periodically so that fouling resistance of cooling water is under control. Cleaning of tubes with a wire brush, attached to a rod, is used when mechanical cleaning is resorted to. Chemical cleaning is another method in which sulphamic acid is used with an inhibitor. Proprietary chemicals are also available from certain manufacturers for chemical cleaning which may not contain acidic chemicals. In another method, hydro-jetting technique is used for cleaning the tubes. High pressure (in excess of 150 bar) water is used in lances, attached to the tube, which loosen almost all types of scales and remove from the tubes without their damage. This technique is found quite effective and ensures clean tubes for the cooling service for a longer period.

A good practice is to provide a temperature indicator on cooling water line on exit of each cooler so that flow of cooling water flow can be effectively controlled. Also cooling water circuit should be designed such as no heat exchanger starves of it.

This is particularly important for heat exchangers located at elevations and at farthest locations where enough cooling water flow should be ensured.

In a special case a tube bundle is installed in a dedicated cooling tower in which condensation takes place. Such an equipment is called an *evaporative condenser*. Since evaporation of water takes place simultaneously with heat transfer, constant cooling water temperature is assumed for heat exchanger design calculations (refer to Exercise 6.10). Such an equipment can be used for condensation of a refrigerant, steam (such as turbine exhaust), etc.

In some cases, cooling water is used in two or more heat exchangers in series as each of them is designed for low temperature rise (say 2 to 3°C). While designing such a circuit, care should be exercised that pressure drop in cooler is low so that adequate flows are available in the coolers. Cooling in compressor jackets, cooling in a battery of glass condensers above a glass lined reactor, etc., are some examples which fall in this category.

Both carbon steel and stainless steel (SS) tubes are in common use in the coolers. When ERW SS tubes are used, chlorides in circulating water must be controlled, preferably below 300 mg/L (as Cl), as stress corrosion is a possibility in such coolers with chloride ions.

Special care is required while designing a closed circuit cooling water when cooling water is to be used in direct contact equipment such as a barometric condenser, venturi scrubber, etc. Cooling water could be contaminated with carry-over material in the barometric condenser which may call for special treatment of cooling water. Alternately excessive blow down may have to be resorted to control the concentration of the contaminant(s).

In general, water cooling is cheaper than air cooling.

11.3.3 Refrigeration and Associated Cooling Systems

When cooling of a process stream is required below ambient temperature, cooling medium must be at a lower temperature. Secondary coolant; chilled water or brine is used for this purpose. Term *refrigeration* is used for achieving temperature up to –50°C below which use of liquefied gases (such as liquid nitrogen) is required and such cooling is known as *cryogenics*. It is possible to achieve temperatures up to –80°C with cascade refrigeration but such systems are rather uncommon. Secondary coolant is cooled by a refrigerant and used for process heat transfer applications.

Chilled water systems In industry, chilled water is produced up to 7°C. Rise in chilled water is usually restricted to say 7°C in the chiller. Both absorption and mechanical refrigeration systems are in use in industry. A typical absorption refrigeration system, commonly used in industry, is shown in Fig. 11.8. It uses water (R-718) as a refrigerant and aqueous lithium bromide solution as an absorbent. Low pressure steam (say at 2 bar g) is used for evaporating water from strong aqueous LiBr solution.

Fig. 11.8 *Absorption Refrigeration Unit with LiBr Solution*

Extraction of heat in the evaporator is carried out at 5°C (min.) and 6 Torr a. Circulating chilled water is cooled by evaporating water. Steam condensate or water from a reverse osmosis plant (having low DS) is used as circulating water to avoid fouling problem. Secondary condensers for recovery of solvents such as methanol, ethyl acetate, *i*-propyl alcohol (IPA), etc., use chilled water as a coolant.

Mechanical refrigeration use ammonia (R-717) or a fluoro-chloro carbon refrigerant. Considering ozone depletion potential of various refrigerants, currently tetrafluoroethane (R-134a) is preferably used for chilled water production although use of chlorodifluoromethane (R-22) is also made in certain plants. In a conventional refrigeration cycle, refrigerant gas is compressed and condensed in condenser with help of cooling water. Liquid refrigerant is expanded to a lower pressure in an evaporator where latent heat of evaporation (λ) of the refrigerant takes heat from circulating chilled water. Ammonia is a low cost refrigerant but has toxic effects in comparison to fluoro-chloro carbon refrigerants. Leak of ammonia is a concern and special care is required to control the leaks.

Reciprocating, screw and scroll compressors are in use in industry. For chilled water applications, single stage reciprocating compressor is used. Screw compressor with jacket cooling provides compressed refrigerant at relatively lower temperature at the discharge than reciprocating compressor. In general, screw compressors are more costly than reciprocating machines but consume less power and hence screw compressors are preferred in current designs. Scroll compressors are used for relatively small chilling plants.

A process engineer is expected to specify the refrigeration duty (in kW), desired chilled water circulation rate and its temperature. If low cost steam (or waste steam is available), vapour absorption (VA) refrigeration may be selected. Cooling water temperature and design steam pressure are important parameters while designing VA system. System of mechanical refrigeration is selected on the basis of coefficient of performance and cost of the refrigerant. Currently, many evaporative condensers are in use in refrigerants systems and are claimed to consume considerably low power consumption than a shell- and tube-type condenser with cooling water as a coolant. However, selection of evaporative condenser should carefully be made such that cleaning of external surface of the tube bundle can be made with ease.

Brine systems Brines are secondary heat transfer fluids that are used for process heat transfer applications below 5°C. Most commonly used brine in industry was aqueous solution of calcium chloride. Corrosion was a serious problem faced with $CaCl_2$ brine in addition to other handling problems such as its slimy nature. Over a period, use of aqueous ethylene glycol (MEG) solution, aqueous methanol solution and other aqueous solutions have found applications as brines. Properties needed for calculation of a film coefficient; such as specific gravity, specific heat, freezing point, viscosity, etc., of brines are available in Refs. 2 and 9. Study of the properties help process engineer in selecting most suited brine for a given application. For cooling of brines both; ammonia absorption refrigeration and mechanical refrigeration are in use.

Ammonia absorption refrigeration system is used for achieving brine temperature up to –30°C. Both reciprocating and screw compressors are used for mechanical refrigeration; the latter is now preferred due to reliability and less power consumption. Usually reciprocating compressor has two or more stages. While aqueous MEG brines are used up to –35°C, aqueous methanol brines are used up to –50°C. Ammonia (R-717), tetrafluoroethane (R-134a) and chlorodifluoromethane (R-22) are used, depending on the desired brine temperature, coefficient of performance and cost per unit of refrigeration.

Design of refrigeration system involves brine handling system. Process engineer can design a single tank (3 to 6 m^3 capacity) for small refrigeration system; say for 15 kW capacity. Return brine from plant is returned to an insulated tank. A recirculation pump takes suction from the tank and passes the chilled water or brine through a chiller in which it exchanges heat with the refrigerant. Chilled water or brine is sent to various heat exchangers in the plant. In larger refrigeration plants, two tank (each of 3 to 7 m^3 capacity) system is provided. Also when storage of refrigerated brine is required, two tank system is preferred. Usually a bigger tank is provided with a partition and thus two tanks are made. In the event of internal circulation, overflow from cold well to hot well is permitted. One part is known as *hot well* in which return brine is stored. A primary pump takes suction from hot well and pass the brine through a chiller and colder brine is stored in *cold well*. Another pump takes suction from the cold well and circulates brine in the plant. Main advantage of the system is that the secondary pump is not required to operate continuously. Primary pump pumps brine through the chiller and thus provides sufficient storage of brine at desired temperature. Figure 11.9(a) and (b) depict P&I diagrams for brine handling systems.

Fig. 11.9(a) *Brine Handling System Incorporating Single Tank*

Fig. 11.9(b) *Brine Handling System Incorporating Two Tanks*

Capacity of tanks are decided on the basis of supply of brine (i.e., flow rate) required to number of equipments at a time. Thus these tanks act as buffer. When large refrigeration duty is required in a small period, particularly at a low temperature, required storage (or buffer) capacity is high, e.g., manufacture of antibiotics. A side stream filter (3 to 5% of main circulation flow) is recommended in the discharge of secondary pump and cold brine is recycled to the cold well. Many a times, tank size becomes very large and heat gain from atmosphere is excessive, adding to the basic load of refrigeration. A novel innovative system is developed to have balls in which a known aqueous solution is sealed. The solution freezes at somewhat higher temperature (say 3 to 5°C) than actual temperature at which brine is required. In practice, the balls freeze by transferring latent heat of fusion of the solution during chilling operation. When actual cold brine circulation is started, heat transfer takes place in plant's heat exchangers. To provide sufficient refrigeration, fluid in the balls starts melting and thus the brine temperature is maintained. Such a system requires relatively smaller tanks and heat gain from atmosphere can be kept under control.

In a small chemical plant, a common brine plant may suffice refrigeration requirements at process temperatures above 10°C and for process temperatures below say 5°C. However, in larger plants, more than one refrigeration plants are provided to cater refrigeration demands at different temperatures. When distances are long for brine travel in the plant, it may be economical to provide more than one refrigeration plants, located nearer to process requirements. For a specific requirement of refrigeration temperature (say below –35°C), a dedicated brine system is recommended.

Comparison of performance of various commonly used brines (secondary coolants) should prove interesting. In Table 11.5, some parameters are compared which may help in selecting a brine for the desired application.

Table 11.5 Brine Performance Comparison[9]

Brine (Secondary coolant)	Concentration % by mass	Recommended circulation rate $m^3/(h \cdot kW)$	Freeze point °C	Pressure drop[a] kPa	Heat transfer coefficient[b], $W/(m^2 \cdot °C)$	Energy factor[c]
Aq. calcium chloride	22	0.1800	–22.1	16.685	3214	1.447
Aq. sodium chloride	23	0.1652	–20.6	15.858	3169	1.295
Aq. ethylene glycol	38	0.1782	–21.6	16.410	2305	1.250
Aq. propylene glycol	39	0.1652	–20.6	20.064	1164	1.142
Aq. methanol	26	0.1685	–20.7	14.134	2686	1.078
Aqua ammonia	14	0.1602	–21.7	16.823	3072	1.000
Methylene chloride	100	0.4126	–96.7	12.824	3322	3.735

[a] Pressure drop in one length of 4.9 m tube having 26.8 mm ID and 2.134 m/s brine velocity

[b] Heat transfer coefficient based on Sieder & Tate equation [Eq. (6.18)] in tubes having 4.9 m length, 26.8 mm ID and brine velocity of 2.134 m/s

[c] Energy factor represents relative energy requirement for same suction/discharge pump conditions, –6.7°C average temperature of pumping, 6°C range in HE and freezing point 12°C below the lowest brine temperature.

Direct expansion of refrigerants When refrigeration is required at low temperature and heat transfer area (HTA) availability is restricted, direct expansion of refrigerant is found useful. In fact, use of direct expansion of refrigerant in various applications is in practice since long time. Expansion of ammonia (R-717) in cooling coils (of a serpentine cooler) of a cold storage room for preservation of vegetables, fruits, etc., is a common practice since decades. Similarly in a fishery, ammonia expansion in coils at different temperatures is carried out to preserve fish for long period. In some fisheries, different compressors (single or multistage) are provided and compressed ammonia is condensed in a common condenser. Liquid ammonia is stored in a common storage tank. A liquid ammonia pump then supplies liquid ammonia for direct expansion in different coils. Direct expansion of refrigerant is also commonly carried out in dairy and icecream plants.

Despite this experience, use of direct expansion of refrigerant is not a common practice in chemical plants. Use of chilled water/brine is more common in chemical industry. However, in many applications, use of brine does not provide sufficient refrigeration (in terms of kW at a definite temperature) due to restricted HTA. Consider a CSTR in which a chemical (say a dye intermediate or a bulk drug) reaction is to be carried out at low temperature but required refrigeration duty is high (say in excess of 10 kW). Jacket or limpet coil attached to CSTR may not provide sufficient HTA for heat exchange with a brine. For such cases. direct expansion of refrigerant is carried out at a specified pressure and liquid refrigerant is evaporated. Film coefficient of vaporising refrigerant is quite high in comparison to convective coefficient of brine. Also since evaporation takes place nearly at a constant temperature, temperature difference across hot and cold streams is higher than with brine circulation system. Both the factors result in high heat transfer rate in relatively less HTA. Such a system also eliminates a need for brine handling system. However, the heat transfer equipment is required to be designed for high pressure. For equipment in which chilled water/brine is used for chilling, operating pressure is usually 3 to 3.5 bar g while in equipment where direct expansion is carried out design pressure range from 15 to 20 bar g although actual expansion may take place to low pressure in normal run. This calls for thicker shell as well as jackets which means costly equipment. Also additional safety measures such as provision of safety relief valves/rupture discs on jackets are necessary. Despite this cost factor, direct expansion is found economical, particularly where a dedicated refrigeration system is provided for a single reactor.

Properties of refrigerants are extensively covered in Refs. 2 and 9. Process engineer can design an efficient refrigeration system with the help of these properties. Refrigeration cycles are well described in Refs. 3 and 9 and reader is advised to refer to them.

11.3.4 Thermal Insulation for Cold Surfaces

Heat absorption from atmosphere of cold surfaces is a concern. This could be a very expensive particularly for temperatures below 0°C. Insulation is provided on all cold surfaces for which different materials are used. Amongst these, rigid cellular polyurethane is the one which is commonly used for insulation of cold surfaces. This material finds application for temperatures up to well below –100°C. Its thermal conductivity is 0.05 W/(m · K) in the temperature range –20 to 10°C. Table 11.6 may be used as a guide for cold insulation

Table 11.6 Economic Insulation Thickness for Polyurathane Insulation*

Nominal pipe size		Recommended thickness, mm Temperature range, °C				
mm	in	Amb. to –4	–5 to –20	–21 to –30	–30 to –40	–40 to –50
15/20	½/¾	25	25	40	40	50
25	1	23	40	40	50	50
40	1½	25	40	40	50	65
50	2	25	40	50	50	65
80	3	40	50	50	50	65
100	4	40	50	50	65	65
150	6	40	50	50	65	75
200	8	40	50	50	65	75
300	12	40	50	50	75	100
400	16	50	50	65	75	100
500	20	50	65	75	100	125
600	24	50	65	75	100	125

* Ambient temperature is assumed to be 30–35°C.

11.4 COMPRESSED AIR SYSTEMS

In chemical plants, compressed air is used mainly for two purposes; for instrument air for control system and plant air for general use. For both the services, air at 6 to 7 bar g is stored and distributed in the plant. For small capacity, single stage reciprocating compressor is used. For larger capacity two-stage reciprocating compressor or a screw compressor is used. While oil-free reciprocating compressors provide oil-free air, efficient oil removal system is necessary for a screw compressor. At the suction of compressor, an air filter is provided to get rid of dust and other coarse foreign particles. In the discharge also a micron (fine) filter is provided to filter air. Compressed air is cooled in an aftercooler and stored in a tank. Piping network for compressed air is designed in accordance with the principles, described in Chapter 5. When compressed air requirement is cyclic or erratic, more than one compressor is provided with a 'start' signal based on pressure (on the tank) to start the standby compressor. Such an arrangement also results in energy saving. For more than two compressors in parallel a complex instrument control system is used for optimum energy use.

11.4.1 Plant Air System

Compressed air is used in pneumatic tools, breathing apparatus, in acid egg pumps, diaphragm pumps, during maintenance, etc. Literature provides data on air consumption of pneumatic tools, breathing apparatus, etc. Based on these data, requirement of plant air is estimated. Plant air is not dried. In fact, it is a necessity to have some moisture in air used in breathing apparatus.

11.4.2 Instrument Air System

A drying system is needed for the instrument air. Pneumatic transmitters and control valves need instrument air for operation. With the adoption of electronic instruments, air requirement for transmitters is not required. However, final control element is a control valve with a diaphragm and instrument air is inevitable for them. For normal control valves, dry air in the range of 0.2 to 1 bar g is needed. For on-off control valves, air at higher pressure is used. Selection of operating pressure for on-off valves is important and it is recommended that dry air pressure of 5 bar g (max.) may be specified when air receiver is maintained at 6 to 7 bar g. Instrument suppliers specify dry air requirement for the entire system. However, as a thumb rule, dry air consumption rate of 3 Nm^3/h per control loop may be assumed.

Drying systems for instrument air Drying of compressed air is carried out in different ways:

1. A system incorporating two desiccant dryers is a common system in the plants. A flow diagram for such a system is shown in Fig. 11.10.

   Compressed air enters one dryer from top in which desiccant is filled. Activated alumina, silica gel and molecular sieves can be used as desiccants. Dry air leaves from bottom and taken to a receiver after passing through a micron filter. It is recommended that final air should have foreign particles less than 0.1 gm/Nm^3 with particle sizes less than 3 μm. Recent trend is to use stainless steel piping from receiver outlet to usage point. For use of dry instrument air for control valves, 6 mm dia stainless steel (316 grade) tubes with flared fittings are used.

   After a service period of the dryer, it is isolated for regeneration. Normally, a service cycle of 8 h is preferred for design. Simultaneously the standby dryer comes in line. Isolated dryer is depressurised. A portion of dry air from common outlet of the dryer is passed through a heater in which air is heated either by electric heater or steam to say about 110 to 120°C. Hot air is passed through the exhausted dryer (after depressurisation) from bottom and let out to atmosphere from top. After a definite period, heater is cut off and cool dry air passing is continued till the desiccant is cooled to say 50°C. Dryer is now ready for the next cycle. Regeneration period is usually 4 h for all the steps. Regeneration air requirement ranges from 8 to 12% of total air output of service cycle period.
2. As seen in (a), air requirement for regeneration could be up to 12% which is considered high. For reducing this requirement, a refrigerant condenser can be provided in an alternate arrangement on compressed air line. Chilled water or brine is used for chilling air and water is separated from air. Condensed water is drained out through an automatic trap. Such a system does not require twin dryer system but calls for refrigeration.
3. In another system, a dryer is provided with a special membrane. This membrane is designed to permeate moisture with some air to low pressure (say atmospheric) side. This system can provide air with relatively high moisture content than previous two designs and also lose up to 10% air in the permeate. This design is not common in India.

Fig. 11.10 *Desiccant-Type Air Drying System*

Dew point of instrument air Dew point (DP) of instrument air is an important parameter in designing a drying system. It is a common practice in industry to specify –40°C at atmospheric pressure for the design. Air with this dew point means moisture content of approx. 90 mg/Nm^3. However, drying up to this temperature calls for a desiccant dryer of much larger size. It is not necessary to dry air up to –40°C DP for all locations. For production of instrument air with –40°C DP at atmospheric pressure, compressed air 7 bar a needs to be dried up to –21.4°C under pressure. For dryer with refrigeration, brine at –30°C would be necessary for this purpose. Since high cost is involved in achieving this DP, it is important that right DP may be specified at the design stage. As a guideline, DP of instrument at atmospheric pressure may be specified 10°C less than minimum ambient temperature experienced at the desired location. Figure 11.11 gives DP of air (gas) at atmospheric pressure corresponding to DP at 7 bar a. A point on the line represents same moisture content (on molar basis) at both pressures.

Fig. 11.11 *Dew Points of Gas*

For example, at a particular location in India, minimum ambient temperature experienced in a year is 5°C. For this location, DP at atmospheric pressure may be specified –5°C which means moisture content of approx. 2850 mg/Nm^3. This corresponds to +23°C DP at 7 bar a for which chilled water at 7°C should be adequate in the refrigerated dryer. This represents substantial reduction of drying load on the dryer. Process engineer should therefore specify correct DP for design of the dryer. Online instruments are available for measurement of DP of air (for any gas) under operating pressure. In laboratory, DP is evaluated at atmospheric pressure. Figure 11.10 can be useful in correlating both values.

Figure 11.11 is universal and can be used for any gas but specific humidity (in mg/Nm^3) of the gas depends on its molar mass. Following equation represents relationship satisfactorily between DP at 7 bar a (T') and DP at atmospheric pressure (T) in the range of 5 to –60°C of DP at atmospheric pressure:

$$T' = 2.7355 \times 10^{-3}T^2 - 9.983 \times 10^{-2}T + 125.77 \tag{11.3}$$

where, T' = DP at 7 bar a, K
and T = DP at atmospheric pressure. K

Temperature of compressed air entering dryer Another important parameter is inlet temperature of compressed air entering the dryer. As noted earlier, compressed air is cooled in an aftercooler and stored in a receiver. If cooling water at 32°C is used for cooling in the aftercooler, it is possible to cool air up to say 40°C. Compressed air will be fully saturated at the operating pressure (say 7 bar a). This value may be specified for design of the air dryer. However, if lower temperature than 40°C is specified; say 35°C, it will result in substantially higher moisture content (at 40°C) in ingoing air in actual operation. In such a case, dryer will be under designed. Study of vapour pressure data of water indicates that vapour pressure of water increases 6+% for each degree rise in the temperature range 20°C up to 45°C. This means moisture removal load also increases in the same order. Hence, if the dryer is designed for inlet temperature of 35°C and fed with saturated compressed air at 40°C in actual situation, moisture load on the dryer will increase by say 31%. This will call for higher desiccant requirement in the dryer. Process engineer should therefore specify correct inlet air temperature to the dryer. Also this discussion leads to importance of cleaning compressed air aftercooler periodically. If an air cooler is used for cooling compressed air, temperature of air after the cooler could be still high (say up to 60°C).

Due to any reason, poor drying of air is achieved (i.e., high DP than the ambient), condensation of water is feared on the diaphragm of the control valve and it may malfunction.

11.5 INERT GAS SYSTEMS

Oxygen is required by all living animals for survival. Also oxygen is used for combustion of a fuel and in chemical industry for oxidation reactions. However, presence of oxygen is objectionable in many instances. For this reason, certain gases are used as *inert gases* (i.e., *non-reactive gases*). Nitrogen is used in majority of cases. However, use of carbon dioxide and argon are also being practised but they are relatively costly to nitrogen. Following are some of the reasons for use of inert gas:

1. Blanketing is required in many instances. For removal of a flammable solvent from a product in a dryer, presence of oxygen is objectionable as it can result in flammable (hazardous) mixture in the exhaust. As an example, in a bulk drug plant, a product with acetone (highly flammable solvent) is dried in a fluid bed dryer. If air is used for this drying operation, there is a probability of explosion due to a spark or static electricity at the outlet. For such a hazardous operation, inert gas (e.g., nitrogen) is used as a carrier gas in drying.

   Presence of oxygen over certain oxygen sensitive products can result in its degradation over a period. Consider transportation of mixed tocopherols, β-carotene concentrate, etc., over long distances in drums or in an isotank. If air is present in contact with such a product for a longer period, it will deteriorate. However, if oxygen concentration is kept below say 1% in the environment above such a product, its concentration does not get reduced for long periods. For this reason, blanketing of the product with an inert gas in a container is carried out at a slightly higher pressure than atmospheric.

   Many food products like potato chips, garlic powder, fruit juices, etc., use nitrogen during packing for blanketing so that its taste is maintained for longer duration.
2. In hazardous chemical plants, inert gas is used for different reasons. Consider a liquefied petroleum gas bullet which requires inspection of empty vessel. For man entry in such vessels, presence of oxygen is required. After depressurisation, for safety reasons direct purging of vessel with air is not carried out. Instead, the vessel is first purged with an inert gas and subsequently purged with air. Only after ensuring that all flammable gases are purged and desired oxygen concentration is achieved, man entry is permitted.

   Pressure purging with inert gas is commonly employed in industry. It is preferred over vacuum purging or sweep purging. Material balance calcutations[1] can be easily carried out to determine number of cycles of purging and quantum of inert gas required.

   In another case, consider replacement of catalyst in a methanator in ammonia plant. Presence of lethal gas; carbon monoxide, is anticipated in the vessel. Direct introduction of air is likely to result in explosion in the vessel. Here again, vessel is first purged with an inert gas and then with air.

   During a shutdown in large chemical plant like petroleum refinery, ammonia plant, methanol plant, etc., catalyst vessels are isolated and kept under inert gas blanket under pressure.
3. Dry inert gas is used for deriming (defrosting) of a cold box. In Chapter 6, use of plate-fin heat exchangers was mentioned for various applications, including in cryogenic gas separation. In this heat exchanger, gap between the plates is kept very less; say 2 mm. After a prolonged use of this equipment, a necessity is felt to flush out thin gaps of the heat exchanger due to increase in pressure drop. Flushing operation is carried out with an inert gas.
4. In gas chromatography, inert gas is used as a carrier gas.
5. Electronic industry and nuclear industry use inert gas for various purposes.

Apart from blanketing properties, nitrogen and carbon dioxide (as dry ice) are used for refrigeration applications below –50°C. Dry ice sublimes at –78.46°C by absorbing latent heat of sublimation (571.0 kJ/kg). Dry ice is useful in laboratory for achieving low temperatures as it can be conveniently handled in solid form. In plants, liquid nitrogen at –195.8°C is used for chilling applications by absorbing latent heat of vaporisation (198.7 kJ/kg).

Transportation of liquid nitrogen at temperature less than –195°C in the plant is carried out in special steel pipes (meant for cryogenic applications). Inner pipe is covered with another pipe and annulus is evacuated and sealed. Outer pipe is insulated by at least 150 mm thick polyurethane foam blocks. Despite this care, there can be a substantial vaporisation of liquid nitrogen (up to 25 to 30%) from storage to application point due to its low latent heat of vaporisation (λ). This refrigeration loss also must be taken into account while calculating liquid nitrogen requirements for any cryogenic application in the plant. Since liquid nitrogen is transported at very low temperature, expansion/compression study of the piping system must be carried out and suitable supports, expansion joints, etc., must be provided.

As mentioned earlier, nitrogen is used as an inert gas for most operations. Use of argon is restricted in limited applications because of its cost. Properties of common inert gases are listed in Table 11.7.

Table 11.7 Properties of Common Inert Gases[10]

Property	Nitrogen	Argon	Carbon dioxide
Symbol	N_2	Ar	CO_2
Molar mass	28.0134	39.948	44.0095
Normal boiling point/ Sublimation temperature, °C	–195.80	–185.85	–78.46
Specific volume at 1 atm and 35°C, m^3/kg	0.9025	0.6326	0.5701
Heat capacity at constant pressure (C_p) at 1 atm and 35°C, kJ/(kg · °C)	1.0414	0.5215	0.8595
Critical temperature (t_c), °C	–147.05	–122.45	30.96
Critical pressure, (p_c), bar	33.94	48.63	73.75

Quality of nitrogen varies from an application to application. For blanketing, drying or purging purpose, O_2 content up to 1% could be permitted. For production of nitrogen of this quality, Pressure Swing Adsorption (PSA) plant is adequate. Air is first compressed, dried and passed through carbon molecular sieve beds for separation of N_2.

For catalyst blanketing, O_2 content in nitrogen is desired below 100 ppm (v/v). In electronics industry, nitrogen with O_2 content less than 1 ppm (v/v) is demanded. For achieving high purity N_2, PSA plant with trace oxygen removal (by oxidising with H_2) facility is required. Alternately, cryogenic air separation technique is used which can produce either liquid or gaseous nitrogen of any desired quality. Nitrogen can also be generated onsite by cracking ammonia. Nitrogen so generated may have H_2 as an impurity which is acceptable for catalyst preservation. However, same nitrogen

when required to be used for blanketing, H_2 must be removed by oxidising H_2 with O_2 which is added (in the form of air) in calculated amount.

Purge gas from ammonia plant is processed in a cryogenic plant to recover hydrogen from it. Reject stream of this plant is normally 95 to 97% pure N_2 with some impurities of hydrogen, methane and argon. This reject stream can be used for blanketing of catalyst or purging of cold box. However, this stream cannot be used for purging vessels or for applications where impurities are not acceptable.

Thus selection of a nitrogen generation facility must be carefully made after ascertaining the quality requirements for the desired application. Liquid nitrogen is supplied in tankers at about 6 bar g and –195.8°C. For small requirements, liquid nitrogen is supplied in a stainless steel flask. Gaseous nitrogen is supplied at ambient temperature in cylinders (approx. 6 Nm^3) at about 150 bar g.

For deriming applications, nitrogen gas with –60°C DP at line pressure (approx. 1 mg/Nm^3 moisture by volume at 7 bar a) is required. For this purpose, nitrogen is dried on molecular sieves. For chromatographic applications also, impurity level must be very low and it is specified by the instrument supplier.

Carbon dioxide is available from ammonia plants which can be liquefied by compression and refrigeration at about 20 bar g (corresponding saturation temperature of –15°C). This corresponds to –44.2°C water dew point at atmospheric pressure and saturation humidity of approx. 60 mg/Nm^3 gas. Therefore when compressed (and liquefied) CO_2 is used as inert gas at low pressure, there is a possibility of ice (of water) formation unless gas DP is below –44.2°C at atmospheric pressure. This point is important while designing CO_2 liquefaction facility.

Carbon dioxide can also be produced from flue gas. At first flue gas is treated in an absorber with some solution (e.g., aqueous monoethanol amine solution) to remove CO_2 from the gas and then CO_2 is stripped off from the solution in a stripper by supplying heat. Purified gas is compressed and dried before liquefaction. Fermentation of molasses also produces CO_2 which can be washed, compressed and dried before liquefaction.

Liquid carbon dioxide is supplied in a tanker at about 20 bar g while gaseous CO_2 is supplied at ambient temperature in cylinders (approx. 6 Nm^3) at about 150 bar g. Dry ice blocks, each weighing 15 to 20 kg, are supplied. During transportation of dry ice, there could be substantial loss of CO_2 by sublimation unless transported in well-insulated container.

11.6 OTHER UTILITIES

There are a few other utilities that are of interest to chemical industry. Raw water usage, steam generation and utilisation, fire protection system, effluent treatment, electricity, heating, ventilation and air conditioning, etc., also need due attention at the design stage. Due to their prime dependence on the location, design aspects significantly vary from place and place. For example, raw water could be a surface water source or an underground water source. Qualities of theses sources vary significantly at different locations and hence their treatments are also specific for general purpose use (including sanitation), boiler feed, cooling tower make-up, etc. Similarly, extent of effluent treatment depends on stipulations by local effluent control authority, and

so on. For design of systems related to these utilities, standard books on the related subjects may be referred.

EXERCISES

11.1 Name two chemicals used for removal of final traces of oxygen from boiler feed water or (compressed) water used for heating application.

11.2 Consider process heating requirement at 110°C. Suggest steam pressure for indirect heating application.

11.3 An indirect heater uses hot water at 130°C for heating a heat sensitive organic solution. Suggest pressure of water to be circulated in the system.

11.4 A distillation column separates an aqueous solution of acetic acid (refer to Example 8.5). Assume that column operates essentially at atmospheric pressure. Suggest steam pressure in the reboiler.

11.5 A vacuum system consists of two steam ejectors in series. Both use steam at 4.5 bar a with 10°C superheat as motive steam. What will be the steam temperature of motive steam?

11.6 A high pressure boiler, generating steam for power generation, uses water having conductivity equal to 5 μS/cm and silica content of 0.02 mg/L. Suggest water treatment steps for an underground water source having 1200 mg/L dissolved solids.

11.7 A horizontal pipe of 200 mm (8 in) carries steam at 200°C (t_s). It is insulated with rockwool insulation (having 150 kg/m^3 density) of 50 mm thick mattress which is expected to have a surface temperature of 50°C (t_c) above the aluminium sheet with ambient temperature of 35°C.

Heat transfer coefficient (h) for horizontal bare pipe can be calculated using the following equation:

$$h = [0.85 + 0.005(t_s - t_a)] \times 10 \text{ W/(m}^2 \cdot \text{°C)}$$

where, t_s = Bare surface temperature, °C

t_a = Ambient temperature, °C

Heat transfer coefficient (h') for insulated pipe can be calculated by using the following equation from insulated surface:

$$h' = [0.31 + 0.005(t_m)] \times 10 \text{ W/(m}^2 \cdot \text{°C)}$$

where, $t_m = (t_s + t_c)/2$, °C

t_c = Cold surface (over insulation) temperature, °C

Calculate energy saving for 100 m steam pipe by providing insulation. If cost of energy of saturated steam at 10 bar a (from a solid fuel fired boiler) is Rs. 1.25/kg, calculate annual saving.

Assume that energy requirement is 2475 kJ/kg for 10 bar a steam generation and overall boiler efficiency (η) is 75%.

11.8 In a particular plant, CO_2 is liquefied at 35 bar a and (t_s =) 0°C. What should be the water DP (max.) of compressed CO_2 at atmospheric pressure before liquefaction?

Vapour (sublimation) pressure of ice can be calculated from the following equation in the temperature range 273.16 K to 193.15 K.

$$\ln\left(\frac{p}{611.657}\right) = -13.928[1 - (T/273.16)^{-1.5}] + 34.7078[1 - (T/273.16)^{-1.25}]$$

where, p = vapour (sublimation) pressure of ice, Pa

T = temperature at which vapour pressure is desired, K

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Appendix

1 Conversion Tables

Table A1.1 Length Units

	Metres (m)	Centimetres (cm)	Inches (in)	Feet (ft)	Yards (yd)
m	1	100	39.370 08	3.280 84	1.093 613
cm	0.01	1	0.393 701	$3.280\ 84 \times 10^{-2}$	$1.093\ 613 \times 10^{-2}$
inch	0.0254	2.54	1	$8.333\ 333 \times 10^{-2}$	$2.777\ 78 \times 10^{-2}$
ft	0.3048	30.48	12	1	0.333 333
yd	0.9144	91.44	36	3	1

Table A1.2 Area Units

	Square metres (m^2)	Square centimetres (cm^2)	Square inches (in^2)	Square feet (ft^2)	Square yards (yd^2)
m^2	1	10^4	1550.003	10.763 91	1.195 99
cm^2	10^{-4}	1	0.155	$1.076\ 391 \times 10^{-3}$	$1.195\ 99 \times 10^{-4}$
$inch^2$	6.4516×10^{-4}	6.4516	1	$6.944\ 444 \times 10^{-3}$	$7.716\ 049 \times 10^{-4}$
ft^2	0.092 903	929.0304	144	1	0.111 111
yd^2	0.836 127	8361.273	1296	9	1

Table A1.3 Density and Concentration Units

	Kilograms per cubic meter (kg/m^3)	Grams per cubic centimetre (g/cm^3)	Pound per cubic foot (lb/ft^3)	Pound per UK gallon (lb/UK gal)	Pound per US gallon (lb/US gal)
kg/m^3	1	10^{-3}	$6.242\ 795 \times 10^{-2}$	$1.002\ 2 \times 10^{-2}$	$8.345\ 402 \times 10^{-3}$
g/cm^3	1000	1	62.427 95	10.0224	8.345 402
Ib/ft^3	16.018 462	$1.601\ 846 \times 10^{-2}$	1	0.160 54	0.133 681
Ib/UKgal	99.776 37	9.9776×10^{-2}	6.228 842	1	0.832 675
Ib/USgal	119.8264	0.119 826	7.480 519	1.200 95	1

*$1 g/cm^3 = 1 t/m^3 = 1.000\ 028$ g/mL = 1.000 028 kg/L

Table A1.4 Volume and Capacity Units

	Cubic Metres (m^3)	Cubic Centimetres (cm^3)	Cubic Inches (in^3)	Cubic Feet (ft^3)	UK (Imperial) gallons (UK gal)	US gallons (US gal)
m^3	1	10^6	$6.102\,374 \times 10^4$	35.314 67	219.9625	264.172
cm^3	10^{-6}	1	$6.102\,374 \times 10^{-2}$	$3.531\,467 \times 10^{-5}$	$2.199\,69 \times 10^{-4}$	$2.641\,722 \times 10^{-4}$
in^3	$1.638\,706 \times 10^{-5}$	16.387 06	1	$5.787\,037 \times 10^{-4}$	$3.604\,651 \times 10^{-3}$	$4.329\,007 \times 10^{-3}$
ft^3	$2.831\,685 \times 10^{-2}$	$28.316\,85 \times 10^3$	1728	1	6.2289	7.480 52
U K gal	$4.546\,095 \times 10^{-3}$	$4.546\,095 \times 10^3$	$2.774\,194 \times 10^2$	0.160 54	1	1.200 95
U S gal	$3.785\,412 \times 10^{-3}$	$37.854\,12 \times 10^2$	231	0.133 681	0.832 674	1

Table A1. 5 Mass Units

	Kilograms (kg)	Grams (g)	Tonnes (t)	Pounds (av.) (lb)	Tons (T)	Tons (short) (Ts)
kg	1	1000	10^{-3}	2.204 623	9.8421×10^{-4}	$1.102\,312 \times 10^{-3}$
g	10^{-3}	1	10^{-6}	$2.204\,623 \times 10^{-3}$	9.8421×10^{-7}	$1.102\,312 \times 10^{-6}$
t	1000	10^6	1	2204.623	0.984 21	1.102 312
lb	$453.592\,37 \times 10^{-3}$	453.592 37	$4.535\,924 \times 10^{-4}$	1	4.4643×10^{-4}	5×10^{-4}
T	1016.047	$1.016\,047 \times 10^6$	1.016 271	2240	1	1.12
Ts	907.185	$9.071\,847 \times 10^5$	0.907 185	2000	0.892 86	1

Table A1.6 Force Units

	Newtons (N)	Kilograms force (kgf)	Dynes (dyn)	Pounds force (lbf)	Poundals (pdl)
N	1	0.101 972	10^5	0.224 809	7.233
kgf	9.806 65	1	$9.806\,65 \times 10^5$	2.204 62	70.932
dyn	10^{-5}	$1.019\,72 \times 10^{-6}$	1	2.2046×10^{-6}	7.233×10^{-5}
Ibf	4.448 22	0.453 592	$4.448\,22 \times 10^5$	1	32.174
pdl	0.138 255	1.4098×10^{-2}	$1.382\,25 \times 10^4$	3.1081×10^{-2}	1

Table A 1.7.1 Pressure or Stress Units

	Newtons per square metre N/m^2 (Pa)	Bars (bar)	Standard atmospheres (atm)	Kilograms-force per square centimetre (kgf/cm^2 or at)	Dynes per square centimetre (dyn/cm^2)
N/m^2 (Pa)	1	10^{-5}	$9.869\,233 \times 10^{-6}$	$1.019\,716 \times 10^{-5}$	10
bar	10^5	1	0.986 923	1.019 716	10^6
atm	101.325×10^3	1.013 25	1	1.033 227	101.325×10^4
kgf/cm^2	908.665×10^2	0.980 65	0.967 841	1	$9.806\,65 \times 10^5$
dyn/cm^2	0.1	10^{-6}	$9.869\,233 \times 10^{-7}$	$1.019\,716 \times 10^{-6}$	1
Torr	133.3224	$1.333\,224 \times 10^{-3}$	$1.315\,79 \times 10^{-3}$	$1.359\,51 \times 10^{-3}$	$13.332\,24 \times 10^2$
in Hg	3386.388	$3.386\,395 \times 10^{-2}$	$3.342\,105 \times 10^{-2}$	$3.453\,155 \times 10^{-2}$	$33.863\,886 \times 10^3$
m H_2O	9806.65	9.8067×10^{-2}	9.6784×10^{-2}	0.1	$9.806\,65 \times 10^4$
ft H_2O	2989.063	$2.989\,068 \times 10^{-2}$	$2.949\,89 \times 10^{-2}$	3.048×10^{-2}	$2.989\,063 \times 10^4$
Ibf/in^2	6894.757	$6.894\,757 \times 10^{-2}$	$6.804\,596 \times 10^{-2}$	$7.030\,696 \times 10^{-2}$	$6.894\,753 \times 10^4$

Table A 1.7.2 Pressure or Stress Units (Contd.)

	Torr or Barometric millimetres of mercury (Torr or mm Hg*)	Barometric inches of mercury (in Hg*)	Head of water**		Pounds-force per square inch (lbf/in^2)
			m H_2O	ft H_2O	
N/m^2 (Pa)	$7.500\,616 \times 10^{-3}$	$2.952\,999 \times 10^{-4}$	$1.019\,72 \times 10^{-4}$	$3.345\,53 \times 10^{-4}$	$1.450\,377 \times 10^{-4}$
bar	750.061 6	29.529 99	10.1972	33.4553	14.503 77
atm	760	29.921 26	10.332 27	33.8985	14.695 95
kgf/cm^2	735.5592	28.959 03	10	32.8084	14.223 34
dyn/cm^2	$7.500\,617 \times 10^{-4}$	2.953×10^{-5}	$1.019\,72 \times 10^{-5}$	$3.345\,53 \times 10^{-5}$	$1.450\,377 \times 10^{-5}$
Torr	1	$3.937\,008 \times 10^{-2}$	$1.359\,51 \times 10^{-2}$	$4.460\,35 \times 10^{-2}$	$1.933\,678 \times 10^{-2}$
in Hg	25.4	1	0.345 316	1.132 92	0.491 154
m H_2O	73.5556	2.8959	1	3.280 84	1.422 334
ft H_2O	22.4198	0.882 67	0.3048	1	0.433 526
Ibf/in^2	51.714 92	2.036 021	0.703 072	2.306 67	1

*at 0°C **at 4°C

Table A 1.8.1 Energy and Heat Units

	Joules (J)	Kilowatt hours (kWh)	Kilocalories ($kcal_{IT}$)	Kilogram–force metres (kgf · m)
T	1	$2.777\,778 \times 10^{-7}$	$2.388\,459 \times 10^{-4}$	0.101 972
kWh	3.6×10^{6}	1	859.845 2	$3.670\,98 \times 10^{5}$
$kcal_{IT}$	4186.8	1.163×10^{-3}	1	426.935
kgf · m	9.806 65	$2.724\,07 \times 10^{-6}$	2.3423×10^{-3}	1
L · atm	101.325	$2.814\,583 \times 10^{-5}$	$2.420\,107 \times 10^{-2}$	10.332 31
Btu_{IT}	1055.056	$2.930\,711 \times 10^{-4}$	0.251 996	107.586
lbf · ft	1.355 818	$3.766\,161 \times 10^{-7}$	$3.238\,315 \times 10^{-4}$	0.138 255

Table A 1.8.2 Energy and Heat Units (Contd.)

	Litre atmospheres (L · atm)	British thermal units (Btu_{IT})	Pound-force feet (lbf · ft)
J	$9.869\,233 \times 10^{-3}$	$9.478\,17 \times 10^{-4}$	0.737 562
kWh	$3.552\,924 \times 10^{4}$	$3.412\,142 \times 10^{3}$	$2.655\,224 \times 10^{6}$
$kcal_{IT}$	41.320 5	3.968 32	3088.96
kgf · m	$9.678\,38 \times 10^{-2}$	$9.294\,88 \times 10^{-3}$	7.233
L · atm	1	$9.603\,759 \times 10^{-2}$	74.733 51
Btu_{IT}	10.412 59	1	778.1693
lbf · ft	$1.338\,088 \times 10^{-2}$	$1.285\,067 \times 10^{-3}$	1

Table A 1.9 Specific Energy Units

	Joules per kilogram (J/kg)	Kilocalories per kilogram ($kcal_{IT}$/kg)	British thermal unit per pound (Btu_{IT}/lb)
J/kg	1	$2.388\,459 \times 10^{-4}$	$4.299\,226 \times 10^{-4}$
kcl_{IT}/kg	4186.8	1	1.8
Btu_{IT}/lb	2326	0.555 556	1

Table A 1.10 Power Units

	Kilowatts (kW)	Kilogram force metre per second (kgf · m/s)	Metric horse-powers (mhp)	Pound-force feet per second (lbf · ft/s)	Horsepowers (hp)
kW	1	101.971 62	1.359 62	737.561 2	1.341 022
kgf · m/s	$9.806\,65 \times 10^{-3}$	1	1.3333×10^{-2}	7.233	1.3151×10^{-2}
mhp	0.7355	75	1	542.4761	0.986 32
lbf · ft/s	$1.355\,82 \times 10^{-3}$	0.138 25	1.8434×10^{-3}	1	1.8182×10^{-3}
hp	0.7457	76.040 2	1.013 87	550	1

Table A 1.11 Temperature Units

	Degrees Kelvin (K)	Degrees Celcius (°C)	Degrees Fahrenheit (°F)	Degrees Rankine (°R)
K	T	$t + 273.15$	$5/9\ (t' + 459.67)$	$5/9\ T'$
°C	$T - 273.15$	t	$5/9\ (t' - 32)$	$5/9\ (T' - 459.67)$
°F	$9/5\ T - 459.67$	$9/5\ t + 32$	t'	$T' - 459.67$
°R	$9/5\ T$	$9/5\ t + 491.67$	$t' + 459.67$	T'

Table A 1.12 Absolute Viscosity Units

	kg/(m · s) or N · s/m^2	Poise (P) or g/(cm · s) or dyn · s/cm^2	lb/(ft · s)
kg/(m · s)	1	10	0.671 97
Poise	0.1	1	0.067 197
Ib/(ft · s)	1.488 164	14.881 64	1

1 Poise = 100 certipoises (cP) = 100 mPa · s

Table A 1.13 Kinematic Viscosity Units

	m^2/s	Stoke (St)	ft^2/s
m^2/s	1	10^4	10.763 91
Stoke	10^{-4}	1	$1.076\ 391 \times 10^{-3}$
ft^2/s	0.092 903	929.03	1

1 St = 100 cSt

Table A 1.14 Thermal Conductivity Units

	W/(m · K)	kcal$_{IT}$/(h · m · °C)	Btu$_{IT}$/(h · ft · °F)
W/(m · K)	1	0.859 854	0.577 789
kcal$_{IT}$/(h · m · °C)	1.163	1	0.671 969
Btu$_{IT}$/(h · ft · °F)	1.730 735	1.488 164	1

Table A 1.15 Heat Transfer Coefficient Units

	W/(m^2 · °C)	kcal$_{IT}$/(h · m^2 · °C)	Btu$_{IT}$/(h · ft^2 · °F)
W/(m^2 · C)	1	0.859 845	0.176 11
kcal$_{IT}$/(h · m^2 · °C)	1.163	1	0.204 816
Btu$_{IT}$/(h · ft^2 · °F)	5.678 264	4.882 43	1

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(i) ***Thermodynamics Research Centre (TRC)***, USA from its Publication: *Reprint of the Introduction to the TRC Thermodynamic Tables: Non–Hydrocarbons*, December 31, 1991 and

(ii) ***International Union of Pure and Applied Chemistry (IUPAC)***, UK from its Publication: *Quantities, Units and Symbols in Physical Chemistry*, Edited by I. Mills, T. Cvitas, K. Homann, N. Kallay and K. Kuchitsu, 2nd Ed., Blackwell Science Ltd., UK, 1993.

(iii) ***Guide for the Use of the International System of Units (SI),*** NIST Special Publication 811, 2008, National Institute of Standards and Technology, USA.

Appendix

2 Viscometer Conversion Chart

Index

Absorber 652
criteria of selection 653–656
process design of 660–677, 695–697, 734
Absorption 652
exothermic 654, 712
of HCl gas in 712–714
falling film type absorber 712–717
in a packed tower 656–677
in a spray tower 653, 703–705
of SO_2 in spray chamber 653
in venturi scrubber 709–710
physical 660
with chemical reaction 694–697
Active (bubbling) area of a sieve tray 506
Activity coefficient 215, 369, 439
its determination by
NRTL method 439, 640
UNIFAC method 439
UNIQUAC method 439
van Laar equation 599
Activity of a component 369
Adiabatic compressor 116
Adjacent keys 435
Adsorptive distillation 594
Advantages and disadvantages of
air cooler over water cooler 173
batch reactors over continuous reactors 764
brazed aluminium plate fin heat exchanger 329
bubble type reactor over stirred tank type reactor 805
centrifugal extractors 393
fixed tube heat exchangers 168
gas induction type agitator 786
jet reactors over agitated vessel type reactors 786
mixed flow reactors 774
mixer settler 385
PHE over shell and tube heat exchanger 272
plug flow reactor 774
pulse column 390
rotating disc contactors 393
supercritical extraction over liquid-liquid extraction 420
U-tube heat exchangers 168
vacuum distillation over atmospheric distillation 437
Agitator 396–397, 762–763, 784–788
anchor type 762, 785
crossbeam and blade type 762, 784
gas induction type 786, 840
helical ribbon type 762, 785
high shear mixer type 762
paddle type 762, 785
Pfaudler 762, 785
pitch blade type 762, 787
power correlations for 397
propeller type 762, 785
turbine type 762, 785
AIChE method
for efficiency in distillation 516–518
Air cooled heat exchanger 173, 283
induced draft type 283
forced draft type 283
Air cooling 173–175, 283, 861–862
Air heater 283
Anchor agitator (stirrer) 762, 785
Annular flow 181
ANSI standards for PFD and P&ID 67
°API 14
Atomic Mass 11
Avogadro's number 12
Antoine equation 14
Approach to wet bulb temperature 173, 862
Artificial vapour pressure 97
ASME Code 149
Auxiliary heat transfer medium 629
Azeotropes 589
its compositions 590–592
separation thereof 593
heterogeneous 592
homogeneous 590–591

maximum boiling 589
minimum boiling 589
Azeotropic distillation 594

Back-mix reactor 775
Back mixing 773
Bare tube area 285
Baffles 160–163
dam type 161
disc and doughnut type 161
for condensers 163
orifice type 161
nest type 161
segmental type 161
Baffle cut 161–162
Bale packing 582–583
Batch distillation 540–562
binary 540, 545–549
multicomponent 541, 561–562
settlers 398
simple 540–541
with rectification 545–549, 561–562
Batch operations 44
Batch reactors 759–764
performance equation 764
Batch system 16
Batch type supercritical extractor 421
^{o}Baume 14
Beam type packing support 676
Bell cap liquid redistributor 676
Bell's method 234
Binary distillation 540, 545–549
Binodal curve 367
Blower 116–117
centrifugal 116
Lobe type 116
Block diagram 61
Bogart equation 548
Boiling point 14
normal (at atmospheric pressure) 14
Boiling of liquid mixture 257
Boyko-Kruzhilin equation 181
Brazed aluminium plate-fin exchanger 329–330
Brines 171, 867
systems 867
Bubble cap tray 500
Bubble column reactor 802–804
factors affecting its performance 802
process design of 806–808
Bubble point 216
calculations of 216
Bypassing stream 20

Calibration of
orifice meter 127
rotameter 131
Cap type packing support 676
Capacity ratio curve 626
Carbon dioxide (CO_2)
critical properties of 419
p-t diagram 417
Catalysis 824–826
a key to waste minimization 827–828
Catalytic distillation 576–589
contacting devices for 582
bale packing for 582–583
structured packing for 582
Centrifugal extractors 393–394
Centrifugal pump 93–113
characteristics of 114
viscosity correction charts for 111–112
Ceramic packing 657, 658
Channel cover of shell & tube type heat exchanger 170
Channeling in packed tower 675
Checking for
liquid entrainment 257, 508
weeping 510–511
Characteristics of random packing 658–659
Chemical technology 44
Chilled water system 272, 865–867
Coefficient of heat transfer
boiling 256
condensation 181, 185
with no phase change 178, 235
Coefficient of performance 53, 628
Cogeneration 852
Combined heat and power systems 852–854
Common arrangements for jackets and coils 800–801
Compressed air systems 872
instrument air 873–875
plant air 872
Conversion in a chemical reaction 18
Conversion tables 882–888
Cooler
air type 283, 861
of solids 347–350
Cooling medium 171–175, 861–871
air 171, 861–871
brine 171–172, 867–870
cooling water 171–173, 862–865
chilled water 172, 865–867
oil (thermic fluid) 171
Compressor 80, 116–118
adiabatic 116
polytropic 116
Compressible fluid 80
Comparison of pervaporation, adsorption and distillation 637
Condensation coefficient 181, 182
shell side 185
tube side 181
Condensation with noncondensables 206
Condensation with subcooling 197
Continuous extractor 385
Continuous flow reactor 772
Continuous stirred tank reactor (CSTR) 775
Continuous phase 366
Conversion 18, 764
Conveyor cooler 350
Coriolis flow meter 124
Cornell's method 670–673
Cosolvents and surfactants 423
Cost cutting measures 53
Countercurrent type packing support
mesh type 676

welded ring type 676
Crawford-Wilke correlation 405
Criteria of selection of condensers 197
Criteria of selection of extractors 395
Critical heat flux 256
Critical pressure 15
Critical properties 15
pseudo 263
Critical surface tension of packing Material 669
Critical temperature 15
Critical temperature drop 254
Critical volume 15
Cross beam and blade stirrer 763, 785
Crystallization
freeze 638

Dam baffle 161
Data sheets of equipments 53
Decaffeination of coffee 423–424
Decantation 398
Decanter 398–400
design calculations of 398–400
horizontal type 398–400
vertical type 398, 399
Degree of completion of reaction 783–784
Degree of freedom 435, 498
Delaware method 234
Density 4, 14
Derived dimensions (units) 2–5
Design of a hydrogenation reactor 788–799
Desired properties of solvent
for absorption 661
for extraction 369–370
Determination of
activity coefficient 369, 439
LLE data 369
VLE data 439
Determination of transfer units
for absorption 664
for extraction 376
Determination of theoretical stages
for absorption 739
for distillation 452, 460, 466, 475
for extraction 376
Determination of tower diameter
for sieve tray tower 504–508, 734
for packed tower
for absorber/distillation 665–667, 576–578
for extractor 405
Dew point 216
calculations of 216
of instrument air 874
atmospheric 874
pressure 874
Different type of agitators 396–397, 762–763, 784–788
Different type of reactors 758
Differential continuous contact extractor 387–394
Differential pressure type volumetric flow meters 120
Differential thermal expansion 167
Diluent 365
Dimensions 1–5
fundamental 2
derived 2–5
DIN standards for PFD and P&ID 67
Dispersed phase 366
Distillation 432
adsorptive 594
azeotropic 594
batch 540–562
binary 540, 545–549
catalytic 576–589
extractive 594
multicomponent 465, 475, 561
pressure swing 597
reactive 576–589
short path 569
Distillation columns 432
process design steps 434
selection criteria for 433
Distributed components 435
Distribution coefficient 369
Dittus-Bolter equation 178
Divided flow shell 160
Divided wall column 625
Downcomer 513–515
checking of residence time 515
flooding in 505
pressure drop in 514
various types 513
Dummy tubes 233

Eduljee's equation 510
Efficiency of pumps 98
Efficiency of sieve tray 515–518
Ejectors for distillation system 625–630
Electrical units 7
quantity of 7
Electromagnetic flow meters 113
Energy 7, 20
Energy balances 20
Energy conservation in distillation 620
energy efficient distillation by design 620
efficient operation of column 621
by advanced process control 622
by heat integration 621
by use of high efficiency packing/tray 622
with the use of heat pump 625–630
Enriching section 445
Enthalpy 7
Entrainer 594
Entrainment 257, 509
Estimation of
density of liquid mixture 229
density of vapour/gas mixture 213
molar heat capacity of liquid and gas mixtures 21
surface tension of liquid 408
surface tension of liquid mixture 268, 409
thermal conductivity of gas mixture 249
thermal conductivity of binary liquid mixture 770
thermal conductivity of multicomponent liquid mixture 229
viscosity of liquid mixture 229, 413
viscosity of multicomponent gas mixture 248
viscosity of binary gas mixture 686
mean particle diameter of solids 355
Equation of state 15

ideal gas law 15
Equation tearing procedure for distillation 496–498
Equilibrium constant 215, 217
Equilibrium curve 375, 446
Equilibrium line 373
Equipment data sheet 53
Equivalent diameter 184
for square pitch 184
for triangular pitch 184
of fin 285
of flow channel of plate HE 321
Equivalent length
of fittings and valves 86–87
Equivalent number of velocity head
for fittings and valves 87
ERW pipes 85
ETBE manufacture 584–588
catalytic distillation process 584
effect of various parameters on 587
conventional process 584
Evaporative condenser 361
Exothermic absorption of gases 617–619
of HCl and NH_3 714–717
Expansion joint 167
Extract phase 365, 368
Extractive distillation 594
Extractor 384–396
agitated column type 391
batch type 385
centrifugal type 393
continuous 387
design of 405–407
mixer settler 386
packed tower type 387
rotating disc type 391

Fans 80, 115
axial flow 116
centrifugal 116
Fanning or Darcy equation 82
Fanning friction factor 82
chart 83
Falling film absorber 712–717
advantages an disadvantages of 713
of HCl and NH_3 (its design) 714–717
Fensky-Underwood-Gillalnad equation 465
Film wise condensation 181
Fin efficiency 287–290
Finned tube heat exchanger 284
Fixed bed catalytic reactor 829
design of 832
examples of 832
scale-up 834
Fixed tube sheet shell & tube heat
exchanger 152, 168
advantages and disadvantages of 168
Flash steam from steam condensate 851
Floating head shell & tube heat exchanger 168–169
advantages and disadvantages of 169
pull through type 169
split ring type 169
Flooding
in packed tower type absorber 665–667
in packed tower type extractor 405
in tray tower 504
downcomer flooding 505
jet flooding 505
Flooding velocity
in falling film absorber 716
in packed tower 665–667
in absorber/distillation column 505, 744
in extractor 405
in sieve tray tower 505
Flow arrangements in PHE 307
Flow meters 120–122
electromagnetic type 124
Coriolis 124
flow nozzles 125
orifice type 121–122, 125–127
rotameters 123, 130–131
ultrasonic 124
venturi type 114. 122, 125
volumetric 120
vortex 121
weirs 123
Flow sheet of batch supercritical extraction 421
Fluid 80
compressible 80
incompressible 80
Fluid allocation 174–175
Fluid bed cooler 349–350
Fluid moving devices 93
Force 6
Newton 6
Forced circulation reboiler 275, 282
Forced draft air cooled heat exchanger 283
Fouling coefficient
for shell and tube heat exchangers 189
for plate heat exchangers 309
Francis formula 510
Freeze crystallization 638
Friction factor
for air side 294
for plate heat exchangers 310
for pipes 82–83
shell side 188, 242,
tube side 183
Freezing point 21
Froude number 345
FUG method 465–466
Fugacity coefficient 215, 439
Fundamental quantities 1, 2

Gas absorption 652
Gas expansion and shrinkage 803
Gas film controlled absorption 664
Gas injection type packing support
beam type 676
cap type 676
Gas induced agitator 44, 785, 840
Gas phase transfer unit 664
Gas phase mass transfer coefficient 664
Gases 15
Gasket material for PHE 306
Generalized flooding and pressure drop correlation 667
Gilliland's correlation 466

Gilmore's equation 206
Grand composite curve 335
Green engineering 53
Gross calorific value 23

Heavy key component 435
Heat 7
Heat capacity 21
  molar (isobaric/isochoric) 21
  of ideal gas mixtures 21
  of liquid mixtures 21
Heat duty 169, 254
Heat of absorption 31
Heat of combustion 22
Heat of formation 22
Heat of mixing 31
Heat exchanger design software 152
Heat exchangers
  brazed aluminium film type 329
  finned tube type 283
  plate type 305
  shell & tube type 149
  spiral type 317
Heat exchanger networking 331
Heat of reaction 23
  endothermic 23
  exothermic 23
  standard 23
Heat of mixing 31
Heat of solution 31
Heat integration in distillation column 621
Heat pump in distillation 625–630
Heat transfer area 178
  calculations thereof 190
Heat transfer coefficient
  overall, estimation of 176–177
    calculations of 189
  for air cooler/heaters 287
  for agitated vessel 769
  for bubble column reactor 808
  for condensation 181
  for condensation with noncondensables 206
  for falling film absorber 717
  for no phase change 178
  for nucleate boiling 256
  for plain jacket 814
  for plate heat exchanger 308
  for spiral baffle jacket 840
  for spiral heat exchanger 322–324
  overall 178
    typical values 177
  shell side 182–187, 235–240
  tube side 178–182
Heat transfer factor
  shell side 186, 236
  tube side 180
  for air side 287
Heater
  air type 283
Heat transfer in batch reactors 795
Heat transfer in solids 347–350
Heating medium 171–172
  auxiliary medium 629
  condensing fluids 171, 845
  flue gas/hot air 171, 858
  molten salt 172
  oil (thermic fluid) 171– 172, 855–857
  saturated steam 171, 845
Hegstebeck-Geddes equation 440
HETP of packings 518
Height of equivalent overall transfer
  unit for absorber
  based on gas phase 663, 667
  based on liquid phase 663, 667
Height of equivalent overall transfer unit for extractor
  based on raffinate phase 396
  based on continuous phase 406
  determination by experiment 407
Helical ribbon impeller 763, 785
Hess's law 23
HETS values fo extractors 407
High efficiency packing/trays 540
High shear mixer 662
High speed tray for absorption 655
Heterogeneous azeotrope 592
Hold down plate 677
Homogeneous azeotrope 590–591
  maximum boiling 590
  minimum boiling 590
Horizontal condenser 197
Horizontal spray chamber 703, 705
Hot air systems 858
Hot oil (thermic fluid) cycle 171–172, 855–857
Hot water systems 858
Hydraulic flooding 505

Ideal batch reactor 759
  advantages and disadvantages of 764
  its performance equation 764
Ideal gas law 15
Ideal mixed flow reactor 775–776
Ideal plug flow reactor 772–775
Inclined downcomer 514
Incompressible fluid 80
Induced draft air cooled heat exchanger 283
Industrial applications of liquid-liquid extraction 366
Industrial examples
  of batch reactor 759
  of bubble column reactor 804
  of mixed flow reactor 776
  of plug flow reactor 774
Industrial extractors 384–394
Inert gas systems 876–879
Inline mixer 56, 773
Internal coil in a vessel 801–802
International System of Units 1

Jackets 800
  common arrangements of 801
  limpet coil type 801
  plain 800
  spiral baffle type 801
  heat transfer coefficients for
    calculations thereof 814, 840
  selection criteria of 800
Jet flooding 505

Jet reactor 761
Joint efficiency 85
Joule 5, 7

Kay's rule 263
KATMAX structured packing 582
Kettle type reboiler 151, 254–258
  process design steps 254–258
  selection criteria of 282
Key components 435
  light key 435
  heavy key 435
  split key 435
K_i values for hydrocarbons 217
Kirk Bride equation 472

Lacy's equation 741
Latent enthalpy 21
  of fusion 22
  of sublimation 22
  of vaporization 21
Le Chatelier's principle 576
Lewis-Matheson method 475–479
Li method 770
Light key component 435
Limitations of heat exchanger networking 347
Limiting reactant 18
Limpet coil 801
Linde trays 503
Liquid back-up in downcomer 514
Liquid crest over weir 510
Liquid distributors 674–675
  orifice type 674, 675
  perforated pipe type 674, 675
  trough type 674, 675
  weir type 674, 675
Liquid redistributors 675–676
Liquid entrainment 257, 509
Liquid flow patterns in sieve tray tower 507
Liquid phase transfer unit 663, 667
Liquid phase mass transfer coefficient 663, 668
Liquid redistributors 675–676
  bell cap 676
  wiper type 676
Locations of pressure taps for orifice meter 126
Logarithmic mean temperature difference (LMTD) 175
Loop reactor 660, 685
LMTD correction factors 175
  for shell and tube heat exchangers 175–176
  for air coolers/heaters 291– 292
  for plate heat exchangers 308–309

McAdams, Drew and Bay's equation 619
Malokanov equation 466
Mass 2
  atomic 11
  molar 2, 11
Mass fraction 12
Mass flow meter
  Coriolis type 124
Mass flow rate
  through orifice meter 126
  through rotameter 130
  through venturi meter 147
Mass transfer coefficients
  of gas phase 664
  of liquid phase 665
Mass velocity 179, 184
Material balances 16–18
  without chemical reactions 17
  involving chemical reactions 18
MATHCAD® 448
McCabe-Thiele method 445–446
McAdams, drew and Bay's equation 717
Mean temperature difference (MTD)
  logarithmic 175
  weighted average 215
MESH equations 497
Minimum amount of solvent
  for gas absorption 662
  for liquid-liquid extraction 372
Minimum reflux ratio, 443
  for batch distillation 546, 562
  for binary distillation 443–445
  for multicomponent distillation 445
Minimum wetting rate for different packing 663
Mist eliminator 673
Mixed flow reactor 775–776
Mixer settler 386
Mixing of reaction systems
  gas-liquid mixture 785–786
  homogeneous liquid mixture 785
  immiscible liquid mixture 787
  slurry 788
MKS system of units 1, 2
Molar heat capacity 4, 21
  isobaric 4, 21
  isochoric 4, 21
Molar mass 2, 11
Molar volume of gas at NTP 15
Mole 2, 12
Mole fraction 12
Molecular distillation 570
Molecular sieve drying 611, 637, 874
Monolithic reactor 761–762
Morris and Jackson equation 663
Mostinski equation 255
Multicomponent condensation 214–216
Multicomponent distillation 465, 561
Multiple quench bed reactor 830–831
  axial flow 831
  radial flow 831
Multistage countercurrent extraction 371

Net area for vapour-liquid disengagement on a tray 506
Net positive suction head (NPSH) 94–97
  available 94–85
  for saturated liquid with dissolved gases 96–97
  required 94
  for calculations thereof 94
  correction factors for high temperature 95
Non-distributed components 436
Normal boiling point 14
Normal temperature and pressure (NTP) 14

NRTL equation for determination of activity coefficient 439, 640
Number of overall gas phase transfer units 664
of packed tower 664
of spray tower 704–705
of venturi scrubber 709
Number of transfer units (NTU)
for gas absorption 664
for liquid-liquid extraction 376
Nucleate boiling 254
Nusselt equation
for shell side coefficient 185
for tube side coefficient 178
Nusselt number 178,

Onda, Takeuchi and Okumoto correlation 668–673
Open steam
use in distillation 458
Operating instructions manual (OIM) 51–52
Operating range of trays 503
Operating flooding 505
Optimum amount of solvent for gas absorption 662
Optimum pipe size 81
Optimum pressure drop
in shell and tube heat exchanger 187
Optimum reflux ratio 451
Optimum solvent to feed ratio for extraction 373
Optimum tray spacing 508
Orifice coefficient
for orifice meter 126
for sieve tray 512
Orifice meter 120–121, 125–127
advantages and disadvantages 125
process design steps 126
various tap locations 121, 126–127
Orifice type liquid distributor 675
Overall heat transfer coefficients
calculations thereof 176, 189
for shell & tube heat exchangers 189
for plate heat exchangers 313
for spiral heat exchangers 322
typical values for SHE 177

p-V-T relationship 15
p-t diagram for CO_2 417
p-t diagram for H_2O 418
Packed tower for extraction 387
internals thereof 389
Packing
different types 656–657
random 657
structured 657
selection criteria for 657
Packing factor 658–659
Packing supports 676–677
beam type 676
cap type 676
gas injection type 676
mesh type 676
welded ring type 676
Paddle type agitator 762, 785
Pall rings 657
Partial pressure 5, 16
Partition coefficient 368
Partition plate
shell side 159
tube side 163
Partitioned distillation column 625
Pascal 6
Pass partition plate
shell side 159
tube side 163
Pelen and Small equation 257
Peng-Robinson equation 439
Percent void space in packing 658–659
Perforated pipe type distributor 675
Performance of centrifugal pump
while handling viscous liquids 111–113
Performance test run 52
Pervaporation 637
Petyluk column 625
Pfaudler agitator 762, 785
Phases
continuous 366–367
dispersed 366–367
extract 365–368
raffinate 365–368
Phase diagram
of carbon dioxide (CO_2) 417
of water (H_2O) 418
Phase equilibrium
liquid-liquid 369
of a ternary system of liquids 367
vapour-liquid 439
Physical absorption 660
isothermal 660
non-isothermal 660
Pilot studies 45
Pinch point 341
Pinch technology 331
Pinch temperature difference 331
Piping & instrumentation diagram (P&ID) 60, 66–77
Plait point 368
Plate heat exchanger 305–310
Plug flow reactor 772–775
Point efficiency of sieve tray 515
Ponchon-Savarit Method 460
Polar solvents 423
Positive displacement pump 80
Power 7
Power correlations for agitators 397
Power number 397
Power requirement of
agitators 397
blower/adiabatic compressor 115
fan 116
pump 98
Prandtl number 179
Pressure 6
Pressure drop in a sieve tray 511
Pressure swing adsorption 433, 637
Pressure swing distillation 597–599
Process intensification 55
Projected perimeter of fin 285
Propeller 762, 785
Properties of common inert gases 878

Properties of solvents
for absorption 661
for extraction 369
Properties of supercritical fluids 419
Pressure 3, 6
absolute 6
critical 15
gauge 2
partial 4, 15
Pressure drop calculations for
fluid bed cooler 357
orifice meters 126
packed tower 666–667
piping system 82
in fittings and valves 86
plate heat exchanger 310
shell side 187, 241
sieve tray tower 511–513
spiral heat exchanger 322–323
tube side 182
two phase system 141
venturi scrubber 710
Process design 47
alternate designs 43, 47–49
Process flow diagram (PFD) 60, 62–66
Process engineering 43
alternate routes 43
Process cooling systems 172, 861–865
Process heating systems 171, 845–846, 855–858
Process intensification 55–56
Process research 45–46
Projected perimeter of finned tube 285
Pseudo critical properties 263
Pulsed column for extraction 390
Pump 80, 93–98
different types 93

Quench bed reactor
axial 830–831
radial 830–831
q-line 445

Raffinate phase 365–368
Rankine coefficient of performance 854
Random packing 657
Raoult's law 16, 439
Raschig rings 657
Reaction completion 18, 783–784
degree of completion 783–784
Reactive distillation 576–589
advantages and disadvantages of 580
applications 583
different ways of 581
Reactors
different types 758–759
batch type 759–764
batch loop type 759–760
baffle tank type 760
bubble column type 802–808
continuous flow reactors 772
continuous stirred type 775–776
fixed bed catalytic type 829–834
fluid bed catalytic 760
gas induction type 785, 840
heterogeneous continuous type 785–788
jet reactor 761
mixed flow reactor 775
monolith loop type 761–762
plug flow type 772–775
thin film absorber type 817
tubular type 772
quench bed type 830
Reactive and catalytic distillation 576–589
advantages and disadvantages 580–581
applications 583
contact devices for catalytic distillation 582
design of experiment for 647
Reboiler 254
forced circulation type 254
kettle type 254–258
thermosyphon type 271–276
Recirculation ratio in reboiler 272
Recommended fluid velocities 81, 82
Rectification 445
Recycle stream 20
Refrigeration 865
Reflux ratio 20
minimum 443–445, 546, 562
optimum 451
Regular packing 657
grids 657
stacked rings 657
structured packing 657
Relative volatility 440, 465
Research & development 45
Residence time in downcomer 515
Residual pressure drop 513
Reynolds number 82
Rigorous methods for multicomponent distillation 475
equation tearing procedure 496
Lewis-Matheson method 475
relaxation methods 475
Thiele-Geddes method 488
Riedel equation 22
Roller wiper system 571
Rotary cooler for solids 349
ROSE process 424–427
Rotameter 123, 130–131
calibration of 135
magnetic type 123
standard 123
Rotating disc columns for extraction 391–393
Rotating distributor plate 571

Saddles 657
berl type 657
Intalox type 657
Shapes of solid catalysts 826
Scale-up
of agitated vessels 400, 788
of fixed bed catalytic reactor 834
Schedule number 85
Scrubbing 652
Sealing strip 166
Seamless pipes 85

Segmental baffle 161
Selection criteria for condensers 197
Selection criteria for different types of absorbers 653
Selection criteria for different types of extractors 395
Selection criteria for different types of reboilers 254
Selection criteria for tray tower and packed tower 433
Selection criteria for solvents
  for absorption 661
  for extraction 369
  for extractive distillation 596
Selection criteria for various types of gas-liquid reactors 805
Selection criteria for various types of trays 503–504
Selection of cooling/heating medium 171–173
Selection of key component 435
Selection of operating pressure for distillation column 436
Selectivity in extraction 369, 418
Selectivity in reaction 18
Sensible heat changes 20
Settler 398–400
Shear mixer 763
Shell 152
  divided flow type 160
  single pass 152
  split flow 160
Shell side pass partition plate 159
Shell & tube heat exchangers 149
  as absorber 712
  as reactors 817
  different parts of 152–167
  different types 150, 151
Sherwood-Leva=Eckert correlation chart 667
Short path distillation 569–576
  applications 572–576
  design and working of 571–572
SI prefixes 8
SI system of units 1–5
  base units 2
  derived units 2–5
Sieder-Tate equation 178
Sieve tray 499
  efficiency 515–518
  in extraction 389–390
  process design of 504–518
Simple batch distillation 540–541
Slurry reactions 788
Solute 365
Solvent 365, 369–370
Smith-Brinkley method 465
Smoker's equation 452
Spacer 165
Sparged vessel type reactor 760
Specific gravity 3, 14
Split keys 435
Spiral plate heat exchanger 317–319
  advantages and disadvantages 317
  design of 320–323
  flow arrangement 318–319
  standard dimensions of 320–321
Spray chamber/tower 653, 703–705
  horizontal 705
  vertical 704
Spray chamber design 703–705
  for SO_2 scrubbing 705–709
Spray cooling in rotary cooler 349
Square pitch arrangement 159, 184, 255
Stagewise extractor 384–385
Standard heat of combustion 23
Standard heat of formation 23
Standard heat of reaction 23
Standard pipes 85
Standards for PFD 67
Standards for P&ID 67
Steam heating 845–848
Stratified flow 181
Static mixers 773
Stationary cooler for solids 348
Steam ejector 625–628
Steam stripping 608
Stripping section 445
Stirred tank as absorber 656
Stoichiomtric factor 18
Stoichiometry 11
Stoke's law 399
Structured packing 657
Subcooling 197
Sudgen equation 408
Supercritical extraction 416–420
  advantages and disadvantages 420
  applications 422
Supercritical fluids 419
  critical properties of 419
  properties 416
  reactions in 787
Superficial gas velocity 704, 835
Superficial liquid velocity 411
Supplementary units 2
Surface area of packing 658–659
Surface roughness 82
  of various materials 82
Surface tension 408, 458, 459, 580
  critical of packing material 669
Symbols
  of equipments 68–69
  of piping, instruments and controls 70–72
Synthesis of ETBE 584–589

Tail gas scrubber 715
TEMA 149, 169
  designation 169–170
Tellerettes 657
Temperature 1, 2, 15
  absolute 1
  critical 15
Theoretical stages in distillation 456, 465–466
Thermal conductivity 5, 770
Thermal energy 3, 853
Thermal insulation for cold surfaces 871–872
Thermal insulation for hot surfaces 858, 860
Thermally coupled
  distillation 623–624
  direct sequence 623

indirect sequence 623
side column rectifier 623
side stream stripper 623
Thermic fluid heating systems 855–857
Thermocompressor for distillation system 626
Thermosyphon reboiler 235–247
horizontal 235
vertical 235–247
Thickness of pipe 85
Thiele-Geddes method 488–493
Tie line 368, 377,
Tie rod 164
Tinker's flow model 232–233
Tray efficiency 515–518
Actual 515
AIChE method 516
Van Winkle's correlation 516
Tray spacing 508
Tray tower
different types 499
bubble cap 500
high capacity 503
sieve tray 499
valve tray 501
Tray tower as absorber 734
Triangular pitch arrangement 159
Tridiagonal matrix method 496–498
Triple point 22
of CO_2 417
of H_2O 418
Trouble shooting 52–53, 144
Trough type distributor 675
Tube 163
Tube bundle diameter 159
Tube hole count 153–158
Tube to tubesheet joint 165
expanded 165
welded 165
Tubeside pass partition plate 163,–164
Tube sheet (tube plate) 165
tube hole count 153–158
Turbine type agitator
curved blade 762, 785
flat blade 762, 785
pitched blade 761, 787
Turndown ratio 503
^{O}Twaddell 14
Two phase flow 138–139, 141
estimation of pressure drop in steam-condensate system 140

Ultrasonic flow meter 124
Unagitated static columns for extraction 387
Underwood's method 445
Units 1, 2, 5
conversion factors 882–888
different systems 1, 2
UNIQUAC method for determination of activity coefficient 439
Universal gas constant (*R*) 4, 15
US customary units 1
Use of open steam in distillation 458
U-tube shell & tube heat exchanger 150, 168
advantages and disadvantages 168

Vacuum distillation 433
advantages and disadvantages 437–438
Valves & fittings
equivalents lengths of pipe for 85–86
equivalent number of velocity heads for 86
Valve tray 501
van Larr equations 599
Vapour–liquid equilibrium data 437
Vapour–liquid equilibrium coefficients
for hydrocarbons 217
Vapour recompression system for distillation system 546, 547
Valve trays 500
Vapour pressure 14
Antoine equation 14
artificial 97
Vaporizer 254
van Laar equation 599
Variable area flow meter 123
Vena contracta 121
Vertical condenser 197
Vertical spray tower 705
Vertical thermosyphon reboiler 271–276
VLE data 438
Volume 7
Vortex meter 121
Venturi meter 120
Venturi scrubber 656, 709–710
for SO_2 scrubbing 710–712
Viscosity
conversion diagram 889
correction for centrifugal pump 111–112

Water (H_2O)
critical properties of 419
p-t diagram 418
Water cooled heat exchanger 173–174
Water cooling 862–865
Water hammer 142–143
Watson equation 22
Weeping 510
Froude number for checking weeping 511
Weep point correlation 511
Weir on a tray 508–510
Welded ring type packing support 676
Wetted surface of packing 669
Wetting rate 663
Weirs
for distillation tower 508
for flow measurement 123
Weir type distributor 675
Weighted temperature difference 215
Wiped film evaporator 493
Wiper type redistributors 676
Wiremesh type packing support 676
Work 7

Yield 18

Zeolites 637
Zuber's equation 256

Stream Number	1		2		3		4		5		6	
Stream	Methanol		Air		Mixed Feed		Reactor Exit		Absorber Outlet		HCHO Solution	
Component	kg/h	mass %	kg/h	mass %	kg/h	mass %	kg/h	mass %	kg/h	mass %	kg/h	mass %
CH_3OH	4000.0	100.00	---	---	4000.0	9.29	40.0	0.09	---	---	40.0	044
O_2			9000.6	23.05	9000.6	20.91	6806.8	15.81	6806.8	17.1		
N_2			29 626.8	75.86	29 626.8	68.81	29 626.8	68.81	29 626.8	74.90	5649.1	
H_2O			427.1	1.09	427.1	0.99	2766.0	6.42	2649.0	6.70	3341.3	62.56
HCHO							3341.3	7.76	---	---		37.00
CO_2							386.6	0.90	386.6	0.98		
CO							27.7	0.07	27.7	0.07		
H_2							4.0	0.01	4.0	0.01		
CH_4							9.9	0.02	9.9	0.02		
$(CH_3)_2O$							45.5	0.11	45.5	0.11		
Total	4000.0	100.00	39 054.5	100.00	43 054.6	100.00	43 054.6	100.00	39 556.3	100.00	9030.4	100.00

Fig. 4.2 *Process Flow Diagram for Manufacture of Formaldehyde*

Fig. 4.3 *Process Flow Diagram for Liquefying Oxygen at Low Pressure (Linde Process)[5] (Reproduced with the Permission of McGraw Chemical Engineering by Access Intelligence, USA)*

Summary of Major Streams

Component	Composition, mole %					
	1. Feed Gas	2. LP Drum Vapour	3. Demethanizer Feed	4. Net Overhead Vapour	5. Residue Gas	6. NGL Product
N_2	3.0	4.44	0.37	0.64	3.52	0
CO_2	0.5	0.19	1.07	1.10	0.43	0.91
C_1	80.90	94.16	56.76	96.09	94.63	1.93
C_2	12.00	1.19	31.68	2.08	1.41	72.93
C_3	3.20	0.02	9.00	---	0.01	21.53
i-C_4	0.15	---	0.42	---	---	1.01
n-C_4	0.15	---	0.42	---	---	1.01
i-C_5	0.04	---	0.11	---	---	0.27
n-C_5	0.04	---	0.11	---	---	0.27
C_6+	0.02	---	0.06	---	---	0.14
Total	100.00	100.00	100.00	100.00	100.00	100.00
Conditions :-						
Pressure, bar a	56.2	11.0	15.6	15.2	24.1	15.7
Temperature, °C	38	–111.1	–40	–101.1	48.9	–13.3
Gas Flow, 10^6 m^3/d	2.8316	1.8292	---	0.5861	2.4154	---
Liquid Flow, m^3/d	---	---	---	---	---	1500
Molar Flow Rate, kmol/h	5000	3220	1768	1030	4256	744

Fig. 4.4 *Process Flow Diagram for NGL Recovery from Natural Gas by Expander Process (Reproduced with the Permission of the McGraw-Hill, Inc., USA)*

Fig. 4.10 *Piping and Instrumentation Diagram for Manufacture of Formaldehyde*