Batch Distillation

BATCH DISTILLATION

Objectives

- We wish to explore the pressure drop across the column for various boil-up rates.
- We want to determine the overall column efficiency for various boil-up rates.
- We will run the column at a constant reflux ratio but vary the top and bottom compositions

Introduction

Before getting into the theory behind the distillation column we must first introduce a method for determining the composition of our binary liquid. Our ethanol/water mixture has an index of refraction that varies with composition. Next to the distillation column is a refractometer that will be used to determine the critical angle.

There is a pressure drop associated with vapor rising through the sieve trays. As the vapor velocity increases the pressure drop increases. To increase the vapor velocity one would increase boil-up rate. The boil-up rate is controlled by the power source. The pressure drop behaves like an orifice in that there is a square relationship between the pressure drop and the velocity:

$$\Delta P \propto v^2 \tag{1}$$

However, this relationship only exists if there is no liquid head and foaming on all of the trays.

We will be using Fenske's method to determine the theoretical number of trays. Fenske developed the following formula:

$$n+1 = \frac{\log\left[\left(\frac{x_A}{x_B}\right)_d - \left(\frac{x_B}{x_A}\right)_b\right]}{\log(\alpha_{AB})_{avg}} \tag{2}$$

where n is the number of theoretical trays, x_A is the mole fraction of the more volatile component, x_B is the mole fraction of the less volatile component, and α_{avg} is the average relative volatility.

$$\alpha_{avg} = \sqrt{\alpha_d \cdot \alpha_b} \tag{3}$$

where α_d and α_b are the relative volatilities in the distillate and bottoms, respectively. The efficiency can be calculated:

$$E = \frac{n}{\#\_of\_actual\_plates} \cdot 100(\%) \tag{4}$$

When the system is run at total reflux the liquid flow rate is equal to the vapor flow rate (at steady state). A mass balance around a tray using the most volatile component gives:

$$V \cdot y_n = L \cdot x_{n+1} \tag{5}$$

where V is the vapor flow rate, L is the liquid flow rate, y is the vapor concentration, and x is the liquid concentration. Since V=L

$$y_n = x_{n+1} \tag{6}$$

DESCRIPTION

Distillation Column

43 44 45

All numerical references relate to the diagrams on pages 9, 10 and 11.

The equipment is mounted on a floor standing, welded tubular steel framework (1) fitted with four adjustable feet (2). The frame is designed to allow the use of a fork lift or pallet truck to manoeuvre the unit into position initially.

The equipment comprises a 50mm diameter sieve plate column made up of two glass sections (3) and (4) each containing four sieve plates. The columns are separated by a central feed section and arranged vertically for counter-current vapour/liquid flow. Also installed within the framework are:- reboiler (13), condenser (8), top (10) product tank, decanter (11), reflux valve (12), vacuum pump (20) and all appropriate instrumentation.

The reboiler (13) situated at the base of the column is manufactured from 316 stainless steel and incorporates a flameproof immersion type heating element.

For batch operation, valve (V1) remains closed so that the reboiler can be filled with the initial charge (10 to 12 litres) of binary mixture.

Valve (V1) must remain closed at all times as it is only used on the continuous feed version of this equipment.

The column and reboiler are both insulated to minimise heat loss.

A level sensor (17) inside the reboiler protects the heating element from overheating due to low operating level and a sight glass (18) allows the level in the reboiler to be observed.

The glass column incorporates a total of eight sieve plates in two sections (3) and (4) each containing four plates. Each plate (D) is located by a central support rod (E) and incorporates a weir (F) and downcomer (G) to create a liquid seal between successive stages. The liquid seal on the final plate in each section is achieved by U-tube (H).

Vapour from the top of the column passes to a water-cooled, coil-in-shell condenser (8) which can be insulated to allow heat balances to be carried out. The shell of the condenser incorporates a pressure relief valve (PRV1) to protect the system in the event of a blocked vent and cooling water failure. Cooling water enters the condenser at a regulated rate through a rotameter (FI1) and the flowrate is controlled by diaphragm valve (V5). A cooling water supply is connected to the inlet nozzle (19) and serves also to operate the vacuum pump (20) when operation at reduced pressure is required. Water supply to the vacuum pump is controlled by valve (V14).

Condensate is collected in a glass decanter (11) (phase separator) which is by-passed for normal distillation experiments by opening valve (V10). When the decanter is in use (separation of two immiscible liquids as condensate), valve (V10) is closed so that the overflow (25) and underflow (26) pipes inside the vessel, can take effect.

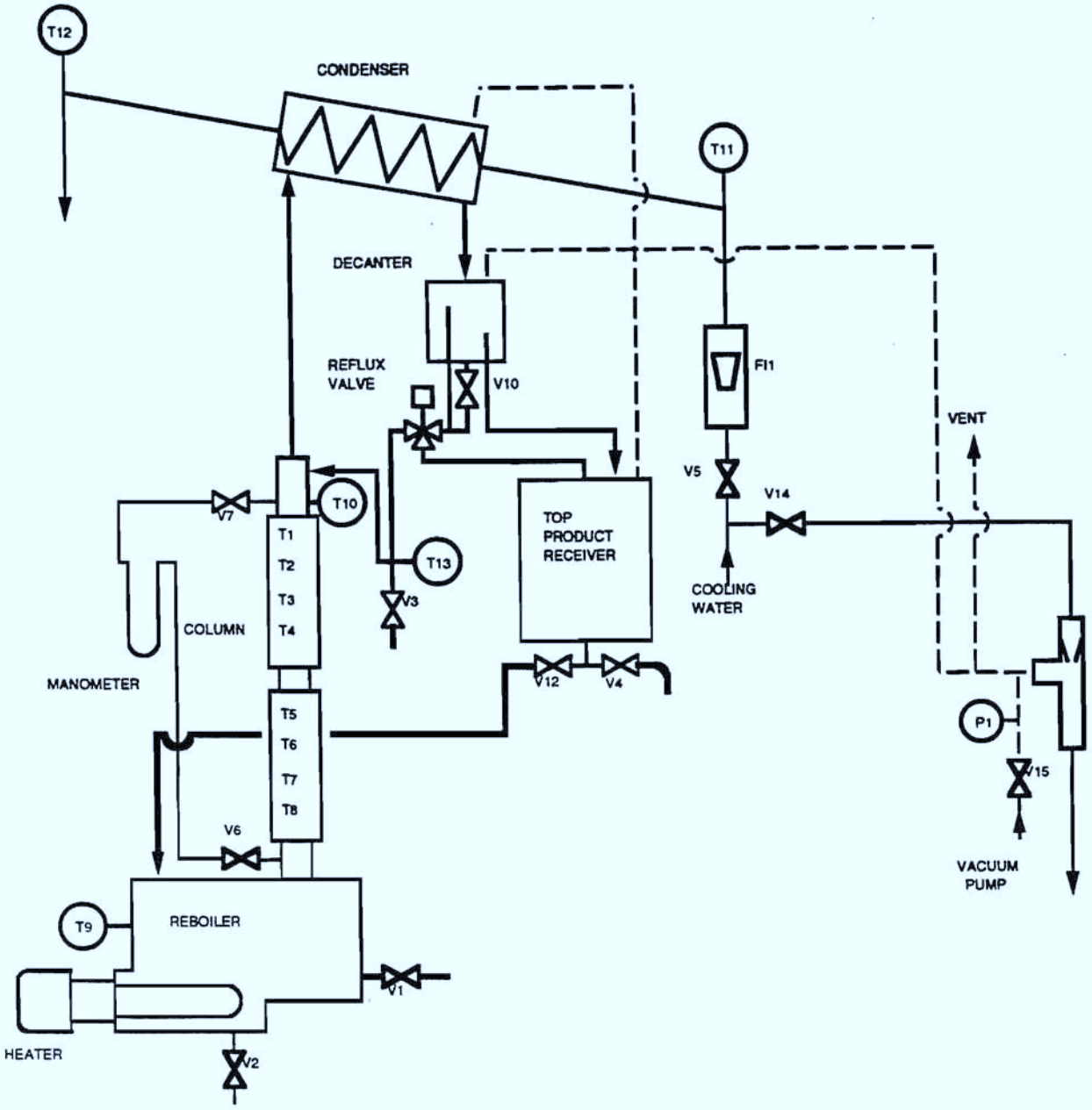
With valve (V10) open, condensate from the condenser outlet passes directly through the decanter to the inlet of the reflux ratio control valve (12) which is a 3-way solenoid operated valve. Depending on the setting of the reflux timers, condensate is directed by the reflux valve either back to the top of the column or to the top product collecting vessel (10). When directed to the column, the reflux passes through a U-seal where a valve (V3) can be used for measuring boil-up rate or for draining the U-seal. The contents of the top product tank (10) can be drained into the reboiler (13) for re-use via valve (V12) .

Temperatures within the system are monitored by thirteen thermocouple sensors (T1 to T13) located at strategic positions in the system. T1 to T8 are located in the column and measure the temperature of the liquid on each sieve plate.

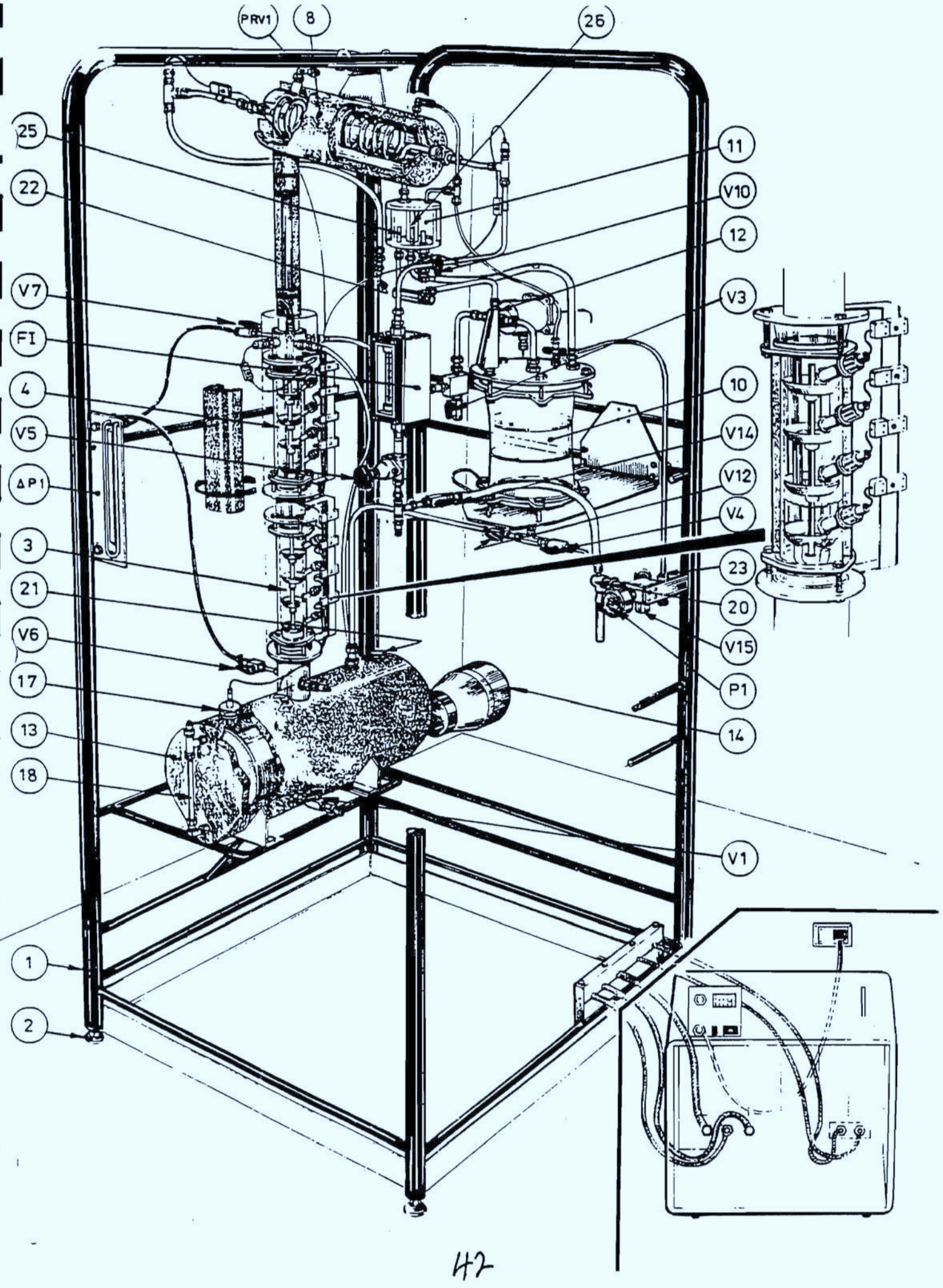
The total pressure drop across the column is indicated on a U-tube manometer (ΔP1) via appropriate tappings in the column fitted with isolating valves (V6) and (V7).

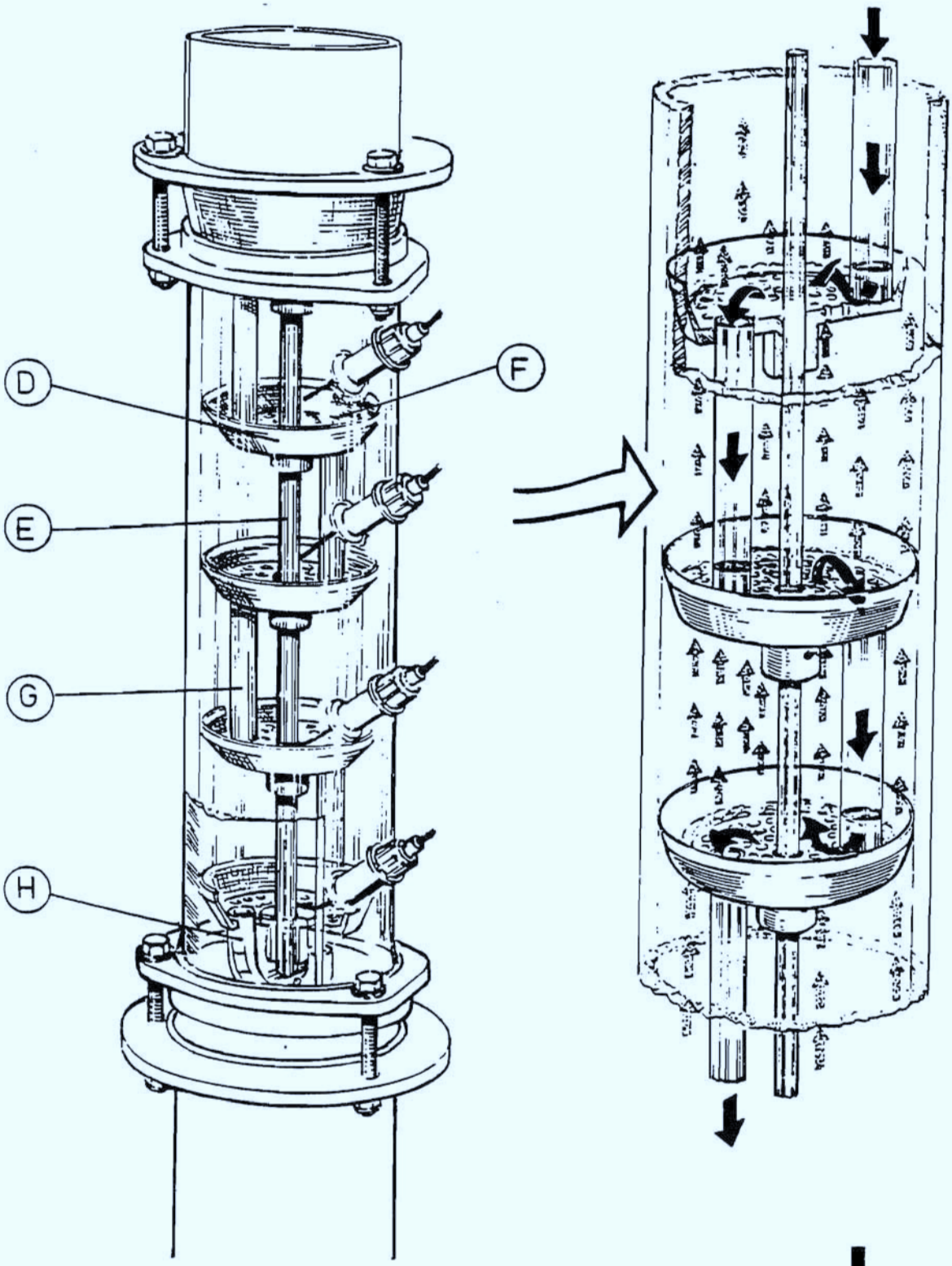
All of the vessels in the system are connected to a common vent (23). This vent is normally connected through a 4.0m length of tubing to a fume cupboard or safe atmospheric vent outlet.

Operation at reduced system pressures is achieved using the water powered vacuum pump (20). When in use, the flexible vent pipe is attached to the inlet of this vacuum pump at (23), and motive water admitted via valve (V14). The level of vacuum is adjusted using needle valve (V15) and is indicated on pressure gauge (P1).



UOP3BM
BATCH DISTILLATION COLUMN

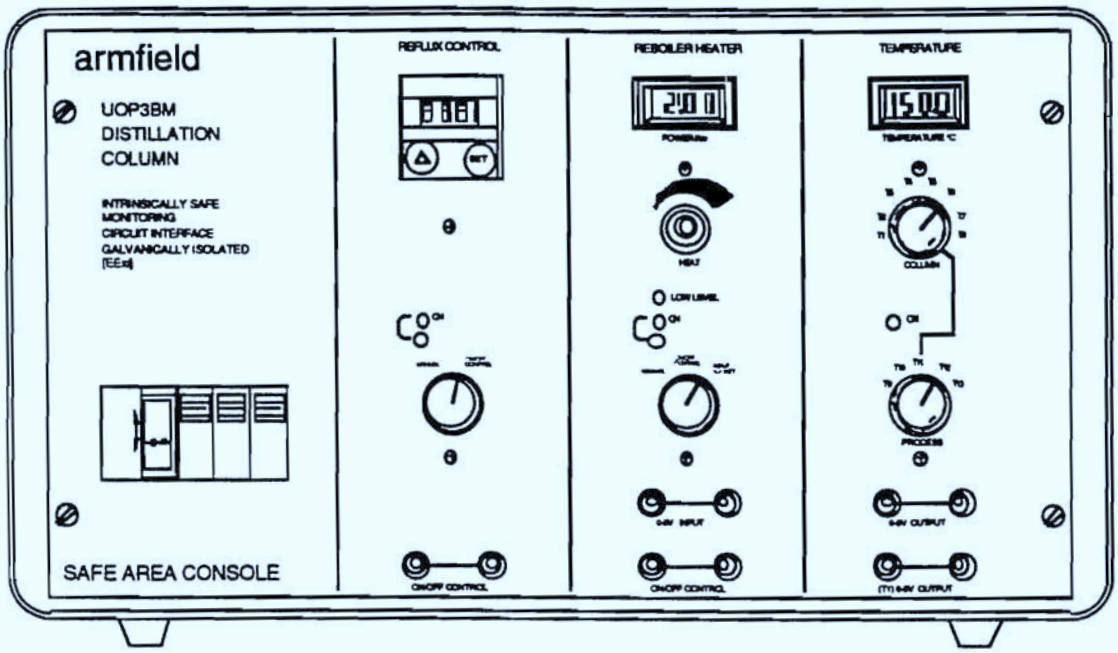

PRV1
8
26
25
22
V7
FI
4
V5
ΔP1
3
21
V6
17
13
18
1
2
11
V10
12
V3
10
V14
V12
V4
23
20
V15
P1
14
V1

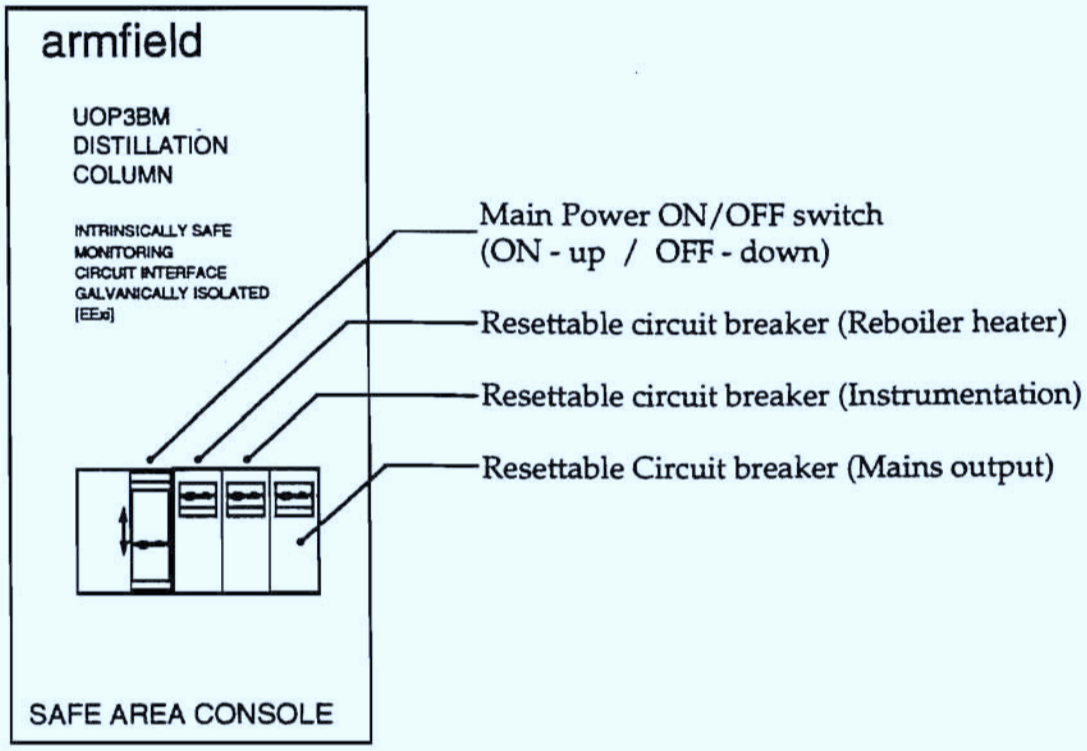

D
F
E
G
H
Liquid Movement
Vapour Movement

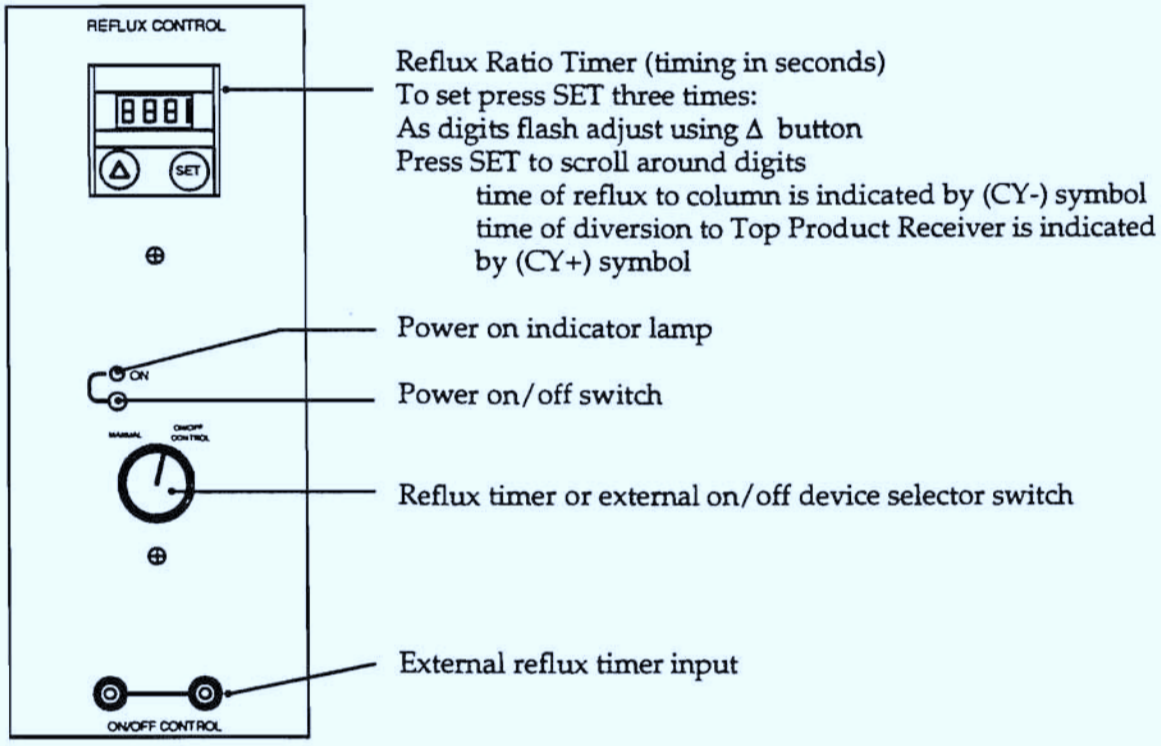
Control Console

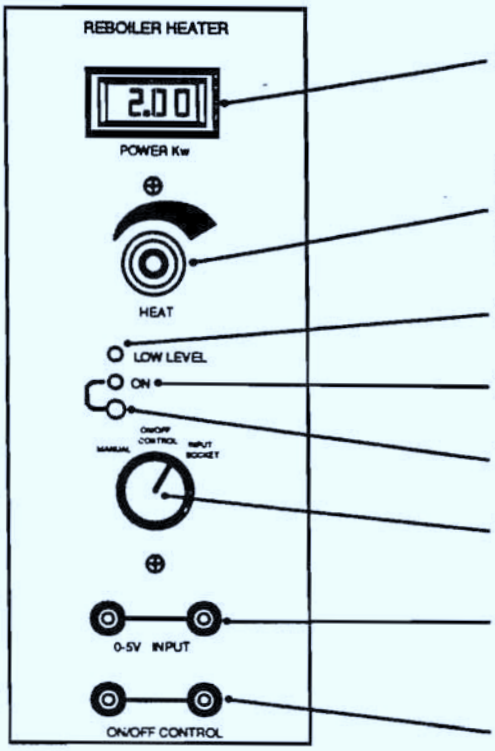
The individual sections of the console are described on pages ~~13~~ and ~~14~~.

The console is attached to the process unit by an umbilical cable which is of adequate length to allow the console to be positioned at least 2.0m away (outside the "Zone 2" area). See "Installation Requirements".




armfield
UOP3BM
DISTILLATION
COLUMN
INTRINSICALLY SAFE
MONITORING
CIRCUIT INTERFACE
GALVANICALLY ISOLATED
[EExi]
SAFE AREA CONSOLE
Main Power ON/OFF switch
(ON - up / OFF - down)
Resettable circuit breaker (Reboiler heater)
Resettable circuit breaker (Instrumentation)
Resettable Circuit breaker (Mains output)


REFLUX CONTROL
SET
ON
ON/OFF CONTROL
Reflux Ratio Timer (timing in seconds)
To set press SET three times:
As digits flash adjust using Δ button
Press SET to scroll around digits
time of reflux to column is indicated by (CY-) symbol
time of diversion to Top Product Receiver is indicated by (CY+) symbol
Power on indicator lamp
Power on/off switch
Reflux timer or external on/off device selector switch
External reflux timer input



Digital display (kW)

Power adjustment
clockwise to increase (max 2.0kW)
(selector switch in manual position)

Low liquid level in reboiler

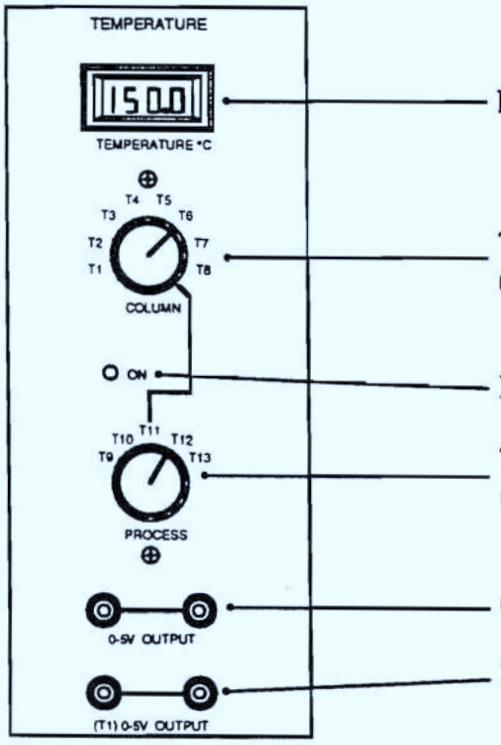
Power on indicator lamp

Power on/off switch

Type of power adjustment selector switch

Power adjustment by external 0-5V source
(selector switch in INPUT SOCKET position)

Power adjustment by external on/off source
(selector switch in on/off control position)



Digital display (°C)

Thermocouple selector switch
(sensors in column)

Power on indicator lamp

Thermocouple selector switch
(sensors in process)

0-5V output of any selected temperature sensor

Permanent 0-5V output of sensor T1

Procedure

Refractive Index Calibration Curve

1. Prepare various mixtures of ethanol and water using 200 proof grade ethanol and distilled water.
2. Turn on the switch to the water bath to start the flow of water through the system and to bring the water to 25°C.
3. Put a couple of drops of pure ethanol on the sample surface in the R.I. machine and clamp the sample in with the upper part of the apparatus.
4. Flip the switch on the left side of the machine down to the first setting. This will turn on the light.
5. To take a measurement, push the switch on the left down to the lowest setting and adjust the large knob on the right until the light/dark horizon is in the center of the cross-hairs.
6. Repeat this procedure for the ethanol/water standard mixtures you prepared.
7. Plot mol% ethanol vs. R.I. to obtain the calibration curve to be used later in the lab (To do this, you will need to know the molecular weight, and density for each component).

Experiment A – Pressure Drop With Respect to Various Bio-up Rates

1. Before starting, make sure all valves are closed.
2. Open valve V10 on the reflux pipe.
3. Turn on power to control panel and switch the temperature selector to T9 (reboiler temperature).
4. Open valve V5 and set a cooling water flow rate of 3L/min on FI1.
5. On the control panel, turn on the power to the heating element to “power on” and turn the knob to a power of 0.8kW. If foaming along the lower column occurs, lower power by 0.1kW until foaming stops.
6. Open valves V6 then V7. Confirm that there is no initial pressure drop in the column. Close the valves in the same order.
7. Wait for equilibrium to occur (no temperature change in each of the temperature probes in the column).
8. Measure boil-up rate by opening valve V3 and allowing the condensate to flow into a graduated cylinder. First, drain into a non-measuring beaker until a steady flow is observed. This will not disturb the system if as long as a liquid level is maintained in the condensate feeding pipe. Once a steady flow is observed, start collecting a sample and start tracking time at the same time. Take about 90 mL of a sample. The boil-up rate is the volume of the sample divided by the time.
9. Take manometer readings by opening valve V6 then V7 and recording the pressure difference. Close the valves in the same order as soon as reading is taken.
10. Ensure that steady state is reached by confirming with a similar manometer reading 5 to 10 min. later. Continue taking readings until two very similar manometer readings are taken.
11. Repeat steps 7-10 after decreasing the reboiler power by 0.1 kW each time until no condensate is observed from the distillate.
12. Comment on the degree of foaming for each power setting.

Experiment B – Determination of Column Efficiency

1. Start the column in a similar fashion to Experiment A but start the power at 0.6 kW. Let the apparatus reach equilibrium (wait about 30 min and check temperatures to make sure they don't change).
2. As described in Experiment A, step 8, determine the boil-up rate three different times and take an average.
3. When taking samples, it is good to take a "discardable sample" (about 10 ml) first so that you get a truly representative sample from the column.
4. Take samples from the distillate and bottoms and determine the refractive index, and the molar concentration for each. BE VERY CAREFUL AS THE SAMPLE FROM THE BOTTOM WILL BE HOT!!!! WEAR THERMAL GLOVES!!!!
5. Record the temperature from the top of the column T1, and the temperature from the bottom of the column T8. Calculate the average temperature of the column.
6. Repeat this process for various boil-up rates to cover the operating range of the column.

Experiment C – Batch Distillation with a Constant Reflux Ratio

1. Start the column in a similar fashion to Experiment A but start the power at 0.6 kW. Let the apparatus reach equilibrium (wait about 30 min and check temperatures to make sure they don't change).
2. On the reflux controller set the reflux settings so that a 5:1 ratio is obtained. To do this:
    a. Press the set button so that a C- shows on the display. This is the time, in seconds, that the distillate will return to the column. Set this to 50 by pressing the delta button until the display reads 50.
    b. Press the set button so that a C+ shows on the display. This is the time, in seconds, that the distillate will go to the top product receiver. Set this to 10 by pressing the delta button until the display reads 10.
    c. Switch the reflux valve on by pressing the power on/off button on the reflux control panel.
3. Measure the boil-up rate as described in step 8 of Experiment A. Do this three times and take an average.
4. Take samples from the top, through valve V3, and bottom, through valve V2, of the column. Remember to allow a small "discardable sample" out first and then take your sample. BE VERY CAREFUL AS THE SAMPLE FROM THE BOTTOM WILL BE HOT!!!! WEAR THERMAL GLOVES!!!
5. Record the refractive index for both samples.
6. Repeat the above steps for different reflux ratios.
7. Repeat the above steps for different boil-up rates.

Data Analysis

Experiment A

- Plot Boil-up rate vs. Pressure Drop on a log-log plot and verify the square relationship.
- Comment on the foaming on the trays.

Experiment B

- Use the Fenske Equation to calculate the theoretical number of plates.
- Calculate the efficiency of the column for various boil-up rates.
- Comment on any noticeable trends or interesting results.

Experiment C

- Use a McCabe-Thiele diagram to calculate the theoretical number of plates and the composition on each plate. Repeat the calculation using a familiar simulation software such as PROII and compare results.
- Calculate the efficiency of the different reflux ratios and boil-up rates.
- Comment on any noticeable trends or interesting results.