CH 3522: Unit Operations Lab

Packed Column Distillation

Batch - R, Group - 05
Date of Experiment: 19th March 2025
Date of Submission: 1st April 2025

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1. Objective

- Operate Vapor-Liquid Separation Experiment using a Packed Column Distillation Process Unit.
- To estimate the number of transfer-units (NTU) from experimental data.
- To estimate the height of transfer-unit (HTU) from total height (Z).

2. Introduction

Distillation is a fundamental separation technique used for liquid mixtures, exploiting the varying tendencies of components to transition between liquid and vapor phases. This method operates on the principle that each substance in the mixture has a distinct boiling point. As the mixture is heated, the more volatile components vaporize first, enabling their isolation. In continuous distillation, a high-purity product is achieved by integrating rectifying and stripping sections, with the feed introduced near the middle of the column. The liquid feed descends through the stripping section by gravity until it reaches the reboiler, a steam-heated unit responsible for vaporization. The generated vapor travels upward through the column, where it undergoes condensation, either being collected or partially recycled as reflux to sustain continuous operation.

Packed column distillation exploits differences in boiling points to achieve separation. When an ethanol-water mixture is heated at the base, ethanol, being more volatile, evaporates more readily. As the vapor rises through the packing material, it interacts with the downward-flowing liquid from the condenser, establishing a countercurrent exchange that enhances mass transfer. This interaction promotes the continuous transition of ethanol into the vapor phase while facilitating water’s

condensation back into the liquid phase. The effectiveness of this separation depends on multiple factors, including the choice of packing material, fluid flow rates, and column configuration.

This experiment aims to analyze these factors by collecting steady-state concentration data and computing key performance metrics, namely the Height of a Transfer Unit (HTU) and the Number of Transfer Units (NTU). Packed column distillation, which employs a structured or random packing medium to aid separation, finds widespread application in industries such as petrochemicals, oil refining, and food processing. In this study, Berl Saddles serve as the packing material. To evaluate column performance, temperature distribution and composition were measured at various points. These measurements were then analyzed to determine HTU and NTU, essential indicators for assessing and optimizing packed column efficiency.

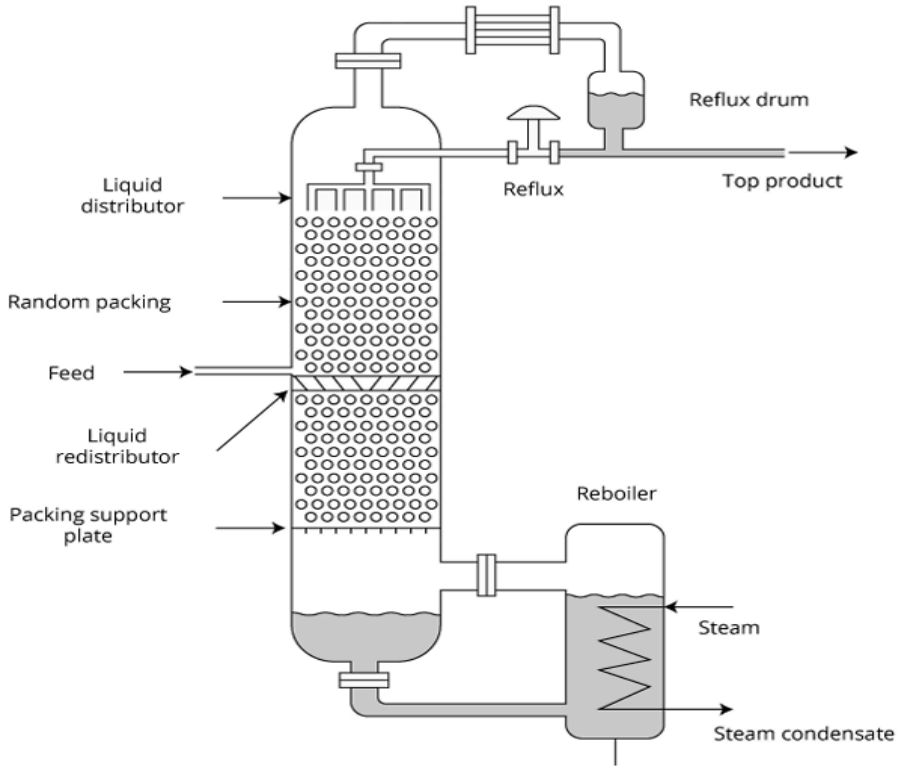


Figure 1: A schematic diagram of a packed distillation column with random packings

3. Theory

In this experiment, the feed is introduced only once into the system and then tightly sealed, allowing continuous distillation to take place within the column. Unlike a plate-column distillation setup, this process employs a continuous packed column with Berl Saddles as the packing material. Due to the continuous nature of the packing, the final liquid sample collected from the column is comparatively lower than what would be obtained from a plate-column system. Over time, the system reaches vapor-liquid equilibrium, making equilibrium data essential for determining the equilibrium mole fractions. These values are then used to calculate key performance parameters, such as the Number of Transfer Units (NTU) and the Height of a Transfer Unit (HTU).

Comparison Between Plate-Column and Packed-Column Distillation:

The performance of a packed column is evaluated using NTU and HTU, both of which provide insight into the efficiency of mass transfer and separation. In binary distillation systems where composition data is available along the column height, NTU can be determined through an integral method. This approach is particularly useful for non-ideal mixtures as it accounts for real vapor-liquid equilibrium behavior rather than assuming ideal stage-wise separation.

1. Number of Transfer Units (NTU)

The NTU quantifies the separation difficulty and indicates the theoretical number of mass transfer stages required to achieve the desired purity. It is calculated using the equation:

$$NTU = \int_{y_B}^{y_D} \frac{dy}{y - y^*}$$

where,

- y = Actual vapor-phase concentration of ethanol
- y* = Equilibrium vapor-phase concentration of ethanol
- y_B = Vapor concentration at the column bottom
- y_D = Vapor concentration at the column top
- x = Liquid-phase mole fraction of ethanol
- x_B = Ethanol mole fraction in the bottom product
- x_D = Ethanol mole fraction in the distillate

2. Height of Transfer Unit (HTU)

HTU represents the packing height required for one theoretical mass transfer unit. It is an empirical parameter known as the 'Height Equivalent to Theoretical Stage' (HETS) and plays a crucial role in translating theoretical stage calculations into practical design parameters. By using the McCabe-Thiele method, the number of theoretical stages required for separation can be determined and then converted into a physical column height using the following relation:

$$Z = HTU \times NTU$$

where:

- Z = Total packing height in the column
- NTU= Number of Transfer Units
- HTU = Height of Transfer Unit

A lower HTU value indicates better packing efficiency, leading to improved mass transfer and enhanced separation performance. High-efficiency packing materials reduce HTU, thereby optimizing the distillation process.

4. Apparatus Required

The apparatus and the chemicals used in this experiment are as follows,

- The chemicals required: Ethanol and water
- Triple-neck round bottom flask
- Plate column with Berl-Saddle packing
- Steady heat source
- Thermocouples and temperature display
- Glassware like beakers, measuring cylinders etc.
- Refractometer (to measure refractive index)

5. Schematic of Experimental Setup

Experimental setup includes the following components and we have also provided the setup used during the experiment in figure 4.

The experimental setup consists of a continuous packed distillation column with Berl-Saddle packing material. The process begins by preparing a feed mixture of ethanol and water, which is

placed in a triple-neck round-bottom flask. This flask is connected to the distillation column, which is equipped with various stages of packing material to facilitate mass transfer. The column is heated by a steady heat source, and the vaporized ethanol moves upward through the packed column while the liquid phase, composed mostly of water, descends. The column is connected to a condenser at the top, which cools and condenses the vapor back into a liquid phase. The condensed liquid can either be collected or returned as reflux into the column. Temperature probes are installed at multiple points along the column to monitor temperature variations during the experiment. The setup includes valves for collecting liquid samples at different heights in the column. A refractometer is used to measure the refractive index of the collected samples, which helps determine the ethanol concentration. Throughout the process, the condenser water supply is maintained to ensure proper condensation, and steady state is reached before collecting the samples for analysis.

Figure 2: Experimental setup of Plate Distillation Column

6. Procedure

1. The process begins by preparing a solution of ethanol in water. Add 200 mL of ethanol to 400 mL of water to form the feed solution. This feed is then transferred into a triple-neck round-bottom flask, which will be used in the distillation setup. Since this is a packed, batch distillation process, the feed is introduced only once.
2. Start by turning on the condenser water supply and the pump to ensure a continuous coolant flow, which is necessary to condense the vapor that rises through the column.

3. Next, switch on the power supply, setting it to a heating mode below 80% of its maximum capacity. Also, activate the temperature display and allow sufficient time for the system to reach steady-state conditions.

4. While the system stabilizes at steady-state conditions, prepare a calibration plot that relates the refractive index to the ethanol concentration in water. Begin the calibration by mixing 1 mL of ethanol with 9 mL of water, and measure the refractive index of the solution using a refractometer. Repeat this procedure for subsequent mixtures with increasing amounts of ethanol, e.g., 2 mL, 3 mL, and so on, up to 10 mL of ethanol, while keeping the total solution volume at 10 mL.

5. After preparing 11 different solutions, you will have corresponding refractive index readings for each ethanol-water concentration. This data can be used to create a calibration plot.

6. During the calibration procedure, monitor the temperatures from all five probes at regular intervals to track the onset of steady-state conditions.

7. Once the temperatures from the probes stabilize and become consistent, indicating that steady-state conditions have been reached, check the liquid levels at all four stages in the column. Then, collect samples from the valves at the top and bottom of the column.

8. After collecting all five samples, measure the refractive index of each sample and record the values. If possible, also measure the refractive index of the remaining liquid mixture left in the flask after the distillation process is complete.

7. Experimental Observations

The following table (refer to table 1, 2) contains the data collected while performing the experiment, corresponding to which the datasheet is also provided at the end of this report.

The tabulated values are as follows,

Table 1: Calibration data of Composition and Refractive Index

Volume of Water (ml)	Volume of Ethanol (ml)	Mole fraction of Water	Mole fraction of Ethanol	Refractive index (μ)
10	0	1.0000	0.0000	1.3306
9	1	0.9665	0.0335	1.3352
8	2	0.9277	0.0723	1.3412
7	3	0.8821	0.1179	1.3461
6	4	0.8279	0.1721	1.3505
5	5	0.7623	0.2377	1.3567
4	6	0.6813	0.3187	1.3591
3	7	0.5788	0.4212	1.3602
2	8	0.4449	0.5551	1.3608
1	9	0.2627	0.7373	1.3610
0	10	0.0000	1.0000	1.3571

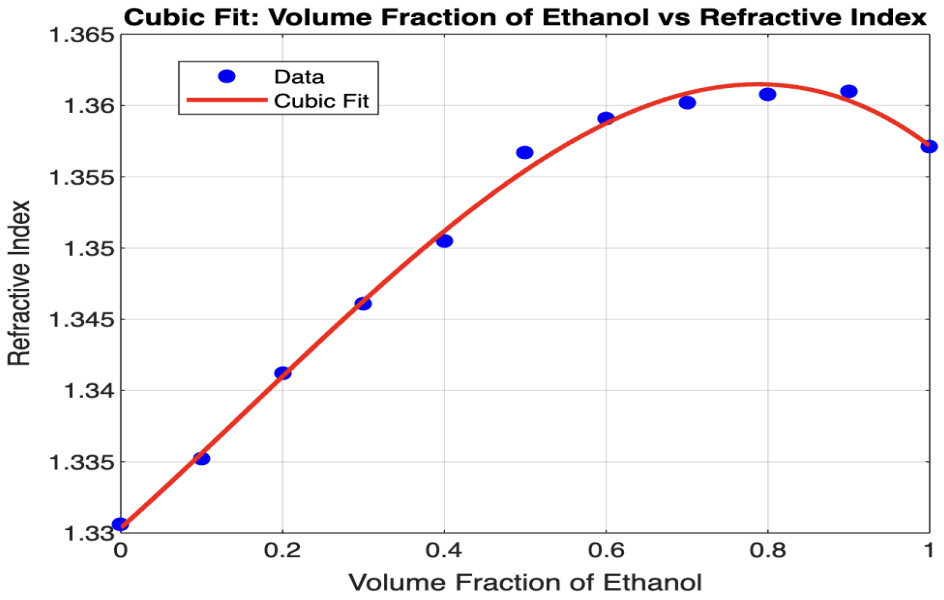


Figure 3: Equation : $y = \mu(x) = -0.0458x^3 + 0.0220x^2 + 0.0506x + 1.3304$

Table 2: Density of samples from different trays of the distillation column

Thermocouple No.	Temperature(with offset)	Refractive index (μ)
1	76	1.3599
2	75.5	1.3605
3	75.4	1.3604
4	74.8	1.3604
5	75.1	1.3606

This Equilibrium data was extrapolated through the equilibrium data found in literature as:

Table 3: Equilibrium data for x vs y

T(℃)	x	y
95.5	0.0190	0.1700
89.0	0.0721	0.3891
86.7	0.0966	0.4375
85.3	0.1238	0.4704
84.1	0.1661	0.5089
82.7	0.2337	0.5445
82.3	0.2608	0.5580
81.5	0.3273	0.5826
80.7	0.3965	0.6122
79.8	0.5079	0.6564
79.7	0.5198	0.6599
79.3	0.5732	0.6841

78.74	0.6763	0.7385
78.41	0.7472	0.7815
78.15	0.8943	0.8943

8. Sample Calculations & Results

Calculating the volume fractions and mole fractions of the 5 thermocouples:

We consider thermocouple 2, for it we have μ = 1.3605. Hence, we can calculate (x) by

$$y = \mu(x) = -0.0458\,x^3 + 0.0220x^2 + 0.0506\,x + 1.3304$$

$$1.3605 = -0.0458\,x^3 + 0.0220x^2 + 0.0506\,x + 1.3304$$

The value of x can be calculated out to be x = 0.6754 (67.54 %)

Converting this volume fraction to mole fraction by the formula:

$$Mole\ fraction\ of\ ethanol = \frac{\frac{x_e \rho_e}{M_e}}{\frac{x_e \rho_e}{M_e} + \left(\frac{(1-x_e)\rho_w}{M_W}\right)}$$

Where,

x_e = Volume Fraction of Ethanol

ρ_e = Density of Ethanol

ρ_w = Density of Water

M_e = Molecular mass of ethanol

M_w = Molecular mass of water

Now, for thermocouple 2:

T = 75.5

$\rho_w = 0.9725\ g/cm^3$

$\rho_e = 0.730\ g/cm^3$

$M_e = 46\ g/mol$

$M_w = 18\ g/mol$

Substituting these values we get, the mole fraction as 0.3793 (37.9 %)

Table 4: Mole fractions

Thermocouple	Temperature	Refractive index	Volume Fraction	Mole fraction
1	76	1.3599	0.6450	0.3482
2	75.5	1.3605	0.6754	0.3793
3	75.4	1.3604	0.6698	0.3735
4	74.8	1.3604	0.6698	0.3733
5	75.1	1.3606	0.6812	0.3852

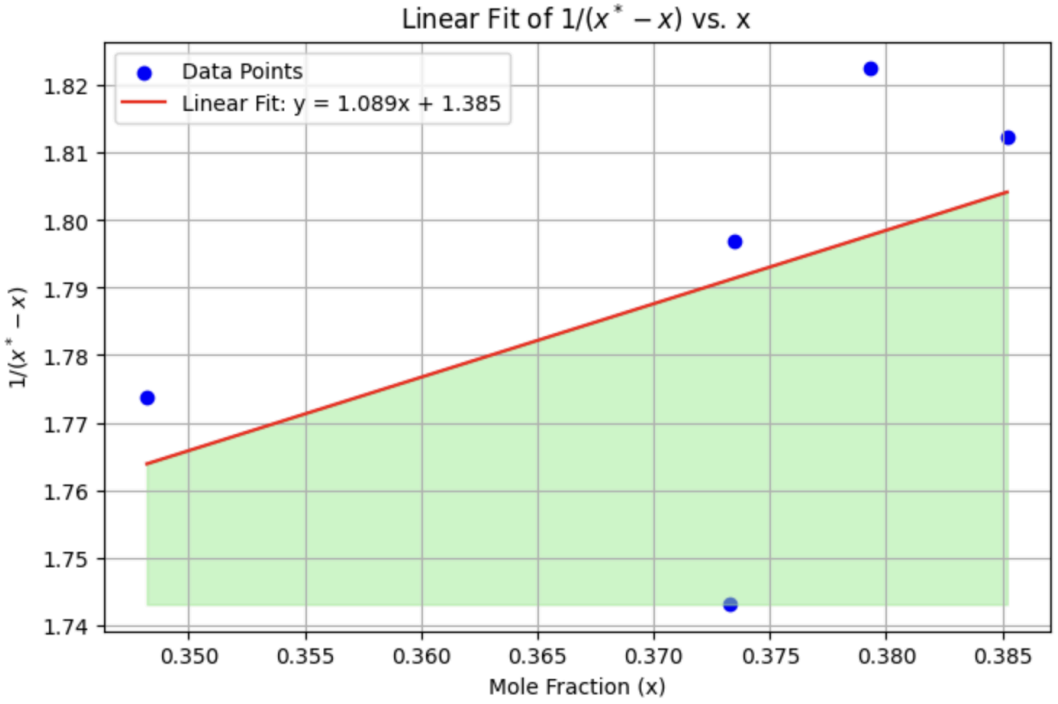


Figure 4: NTU calculations

Table 5 : Equilibrium mole fractions

Thermocouple	Temperature	Mole fraction	Equilibrium Mole Fraction
1	76	0.3482	0.912
2	75.5	0.3793	0.928

3	75.4	0.3735	0.930
4	74.8	0.3733	0.947
5	75.1	0.3852	0.937

Calculation of NTU:

$$NTU = \int_{x_b}^{x_d} \frac{dx}{x^* - x}$$

Hence, NTU = Area under curve = $NTU = \int_{x_b}^{x_d} \frac{dx}{x^* - x}$ = 0.066 (By Simpson's rule)

The measured height of packing is 58 cm = 0.58 m

HTU * NTU = 0.58

HTU= 8.7878 m

9. Discussions & Conclusions

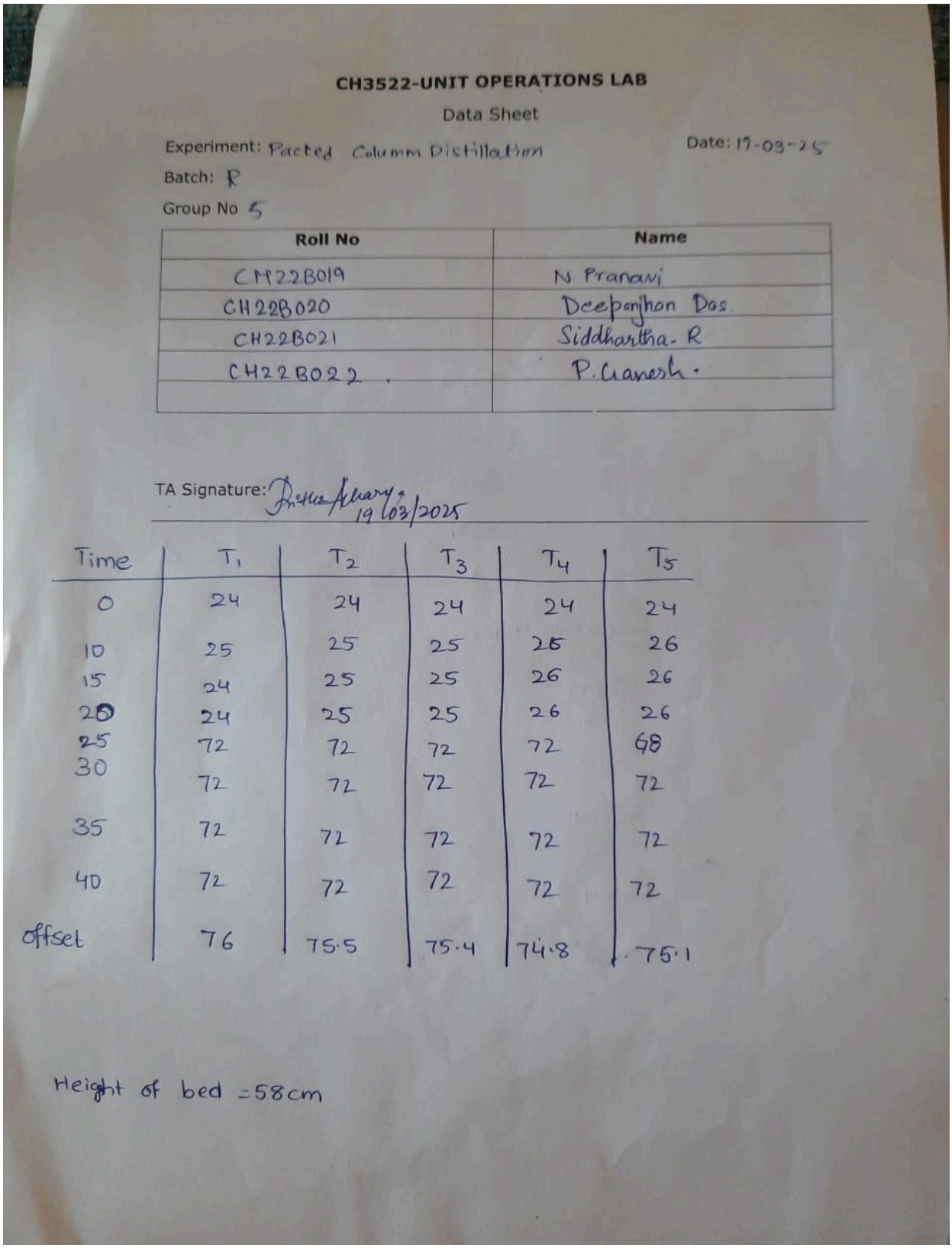
- The ethanol - water mixture attains steady state at around 75°C.
- The Number of Transfer Units (NTU) was calculated to be 0.066 using the simpson's rule of integration.
- The Height of a Transfer Unit (HTU) was determined to be 8.7878 m, based on the known packed height of m and the calculated NTU.

10. Error Analysis

- Some vapors may escape from the top instead of being fully captured and condensed by the upper condenser, which can alter the equilibrium composition of the collected samples.
- The refractometer contains an inherent instrumental error, and residual amounts of a previously measured solution with a different refractive index can affect subsequent readings.
- The temperature indicator recorded a continuous increase in temperature for the last two probes rather than stabilizing, suggesting that a steady state had not been reached; given more time, nearly all the liquid might have vaporized.
- Since the samples are collected at elevated temperatures and are volatile, ethanol; the more volatile component can evaporate, leading to expected changes in concentration; this error is unavoidable due to the time delay during refractive index measurements.

11. References

I. AMT Course Notes
II. Cengel, Y. A., & Ghajar, A. J. (2015). Heat and mass transfer: Fundamentals and applications, McGraw-Hill Education
III. Study of Packed Columns
IV. Article on Identification of best model and parameters for T-X-Y equilibrium data of ethanol-water mixture.
V. Packed Column Distillation



CH3522-UNIT OPERATIONS LAB

Data Sheet

Experiment: Packed Column Distillation Date: 17-03-25

Batch: R

Group No 5

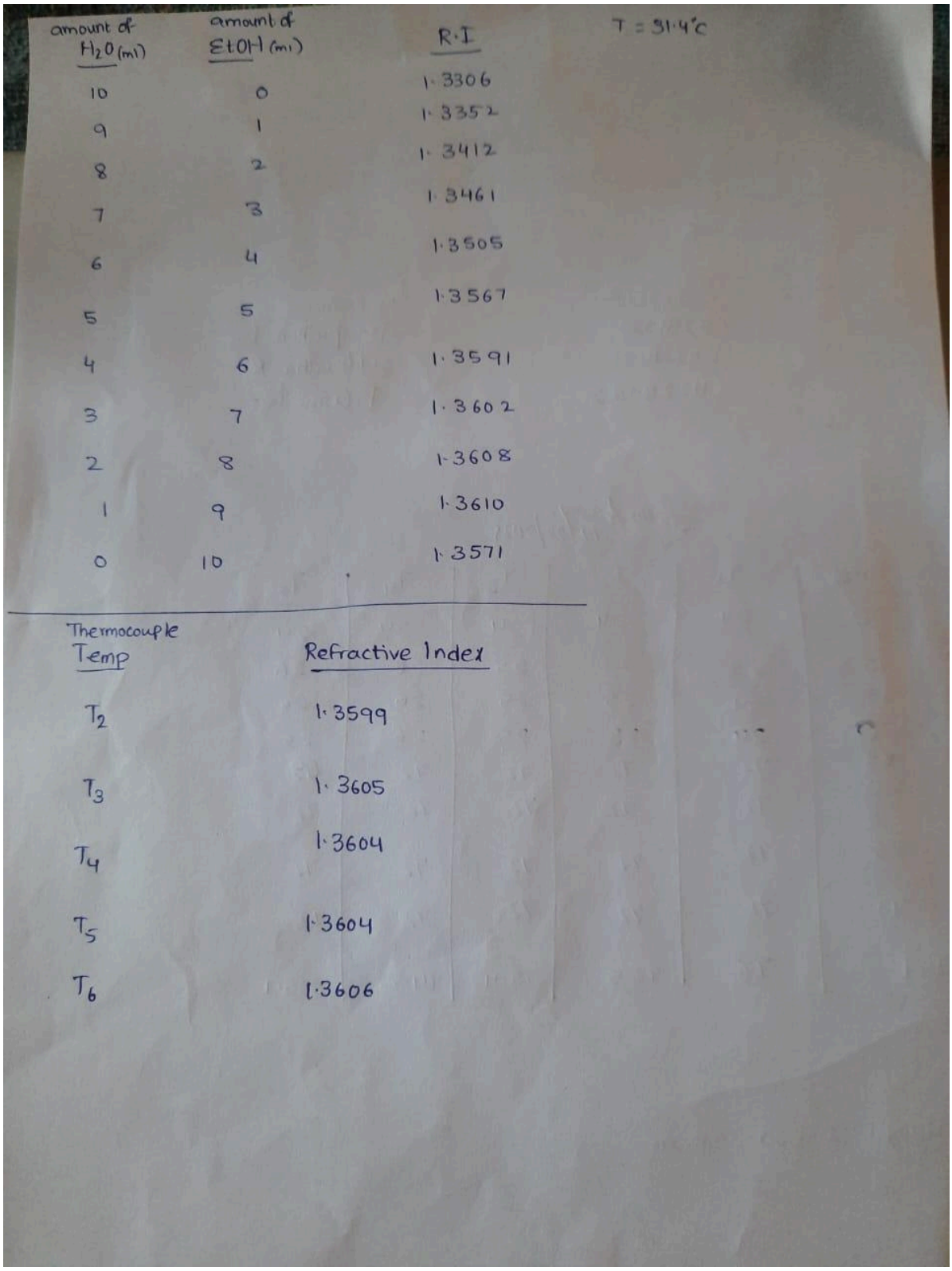
Roll No	Name
CH22B019	N Pranavi
CH22B020	Deepanjhon Das.
CH22B021	Siddhartha. R
CH22B022	P. Ganesh.

TA Signature: 19/03/2025

Time	T_1	T_2	T_3	T_4	T_5
0	24	24	24	24	24
10	25	25	25	25	26
15	24	25	25	26	26
20	24	25	25	26	26
25	72	72	72	72	68
30	72	72	72	72	72
35	72	72	72	72	72
40	72	72	72	72	72
offset	76	75.5	75.4	74.8	75.1

Height of bed = 58cm

Figure 5: Datasheet containing Lab-Data.



amount of H_2O (ml)	amount of EtOH (ml)	R.I
10	0	1.3306
9	1	1.3352
8	2	1.3412
7	3	1.3461
6	4	1.3505
5	5	1.3567
4	6	1.3591
3	7	1.3602
2	8	1.3608
1	9	1.3610
0	10	1.3571

T = 31.4°C

Thermocouple Temp	Refractive Index
T_2	1.3599
T_3	1.3605
T_4	1.3604
T_5	1.3604
T_6	1.3606

Figure 6: Datasheet containing Lab-Data.