



Determination of the diffusion coefficient of organic compound Carbon Tetrachloride (CCl₄) vapor

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Objective :

- Determination of the diffusion coefficient of organic compound Carbon Tetrachloride (CCl₄) vapor.
- Study the effect of temperature on the diffusion coefficient of the compound.

Theory:

Molecular diffusion is the phenomenon of individual molecules moving through a medium due to their inherent thermal energy. When a gas mixture comes into contact with a liquid in a two-phase system, spontaneous alteration via molecular diffusion occurs, eventually bringing the entire system to a condition of equilibrium. Finally, the concentration of any constituent remains constant throughout a phase, although it varies from phase to phase.

The rate expression for the steady-state diffusion of A through non-diffusing B is given as

$$N_A = \frac{D_{AB} P_T}{RT(z_2 - z_1)} \ln \left(\frac{P - P_{A2}}{P - P_{A1}} \right) = \frac{D_{AB} P_T}{RTx} \ln \left(\frac{P_{B2}}{P_{B1}} \right)$$

The rate of evaporation of the organic vapor is given by

$$N_A = \frac{\rho_A}{M} \frac{dx}{dt}$$

Hence, equating the diffusion flux for both the equations, we get

$$\frac{D_{AB} P_t}{RT x} \ln \left(\frac{P_{B2}}{P_{B1}} \right) = \frac{\rho_A}{M} \frac{dx}{dt}$$

The distance of the liquid surface below the open end of the tube is denoted by x in the expression. It is measured both before and after evaporation during a given duration of time. If the level variation is minor, the arithmetic mean of these two readings is used to calculate x. If there is a significant change in level, the value of x is obtained by integrating the initial and final level readings.

Integration of this expression with boundary conditions ($t = 0$, $x = x_0$; $t = t$, $x = x$) and rearranging the equation yields

$$(x^2 - x_0^2) = (2\theta M_A D_{AB} C_A C_T) / (\rho_A C_{BM}) \quad C_{BM} = \frac{C_{B_1} - C_{B_2}}{\ln(C_{B_1}/C_{B_2})}$$

$$\frac{\theta}{x - x_0} = \frac{\rho_{C_{BM}}}{2M_A D_{AB} C_A C_T} (x - x_0) + \frac{\rho_{C_{BM}}}{M_A D_{AB} C_A C_T} (x_0)$$

$$\frac{t}{x - x_0} = \frac{RT\rho_A}{2D_{AB}MP_t} \cdot \frac{1}{\ln(\frac{P_{B_2}}{P_{B_1}})} (x - x_0) + \frac{RT\rho_A}{D_{AB}MP_t} \cdot \frac{1}{\ln(\frac{P_{B_2}}{P_{B_1}})} x_0$$

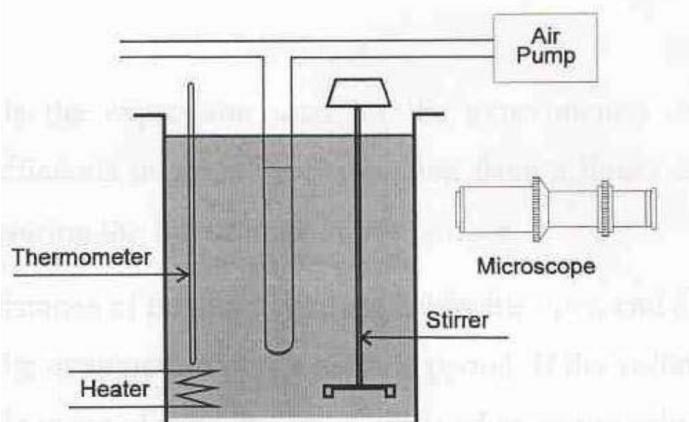
Assumptions made to obtain the earlier equation:

- Gas A and B behave as ideal gases under the experimental conditions.
- D is independent of concentration AB
- Quasi Steady-state conditions prevail throughout the diffusion time.
- Vapor B is insoluble in Liquid A.

Experimental Details:

EXPERIMENTAL SETUP

The apparatus consists of a glass T tube immersed in a constant temperature water bath. The DTC regulates the temperature of the bath. To supply the air that is passed through the T tube, an air pump is used. The T tube is filled with a volatile component, and air is passed across it by the pump, allowing the sliding microscope to detect changes in the level.



Make sure the device is clean and dust-free. Fill the water bath 3/4 full with water. Set the temperature of the water bath to the desired level (328K). Take note of the bath's consistent temperature. Fill the T-tube with CCl₄ until it reaches two centimetres above the capillary leg's top. Take note of the liquid's initial height in the capillary. Connect the air pump to the capillary and allow a gentle current of air to pass across it. At certain time stamps, measure the height of liquid (x) in the capillary. Compile the data and hold a discussion.



SPECIFICATIONS

- Capillary Material: Borosilicate Glass
- Water Bath Specifications:
 - Material: Stainless Steel 304 grade
 - Capacity: 8 L
 - Fitted with heater (Nichrome Wire Heater) and stirrer (Stainless Steel 304 grade)
 - Impeller and shaft coupled with FHP motor)
 - Air-Circulation by FHP pump
- Traveling Microscope: (0-150) × 0.1 mm resolution
- Temp. Sensor: RTD PT-100 type
- Control panel comprising of Digital temperature controller Cum-Indicator
 - (For Water Bath) 0-199.90 C
 - RTD PT-100 type
 - Standard make ON/OFF switch
 - Mains Indicator

The whole set-up is mounted on a powder coated base plate

Observations

- Density of CCl_4 = 1.59 g/cm³
- Total Pressure (p) = 1 atm;
- CCl_4 Molecular Weight = 0.15 Kg/mol

The data (CCl_4 height in glass tube) is recorded accordingly using an optical microscope

Temp(°C)	Time(sec)	Length(cm)	x-x _o (cm)	θ/(x-x _o) (cm/s)
30	0	6.6850	0	-
	600	6.6925	0.0075	80000
	1200	6.7002	0.0152	78947.37
40	0	6.7120	0	-
	600	6.7238	0.0118	50847.46
	1200	6.7345	0.0225	53333.33
50	0	6.7414	0	-
	600	6.7625	0.0211	28436.02
	1200	6.7811	0.0397	30226.70

Calculated Data:

T(°C)	Vapour Pressure (atm)	C _T (Kmol/m ³)	C _A (Kmol/m ³)	C _{B1} (Kmol/m ³)	C _{B2} (Kmol/m ³)	C _{BM} (Kmol/m ³)	Slope, S (s/cm ³)	D _{AB} (m ² /s)
30	0.2	0.0402	0.0080	0.0402	0.0321	0.0360	548245.61	1.05239E-07
40	0.28	0.0389	0.010	0.0389	0.0280	0.0332	533333.33	7.59097E-08
50	0.437	0.0377	0.0164	0.0377	0.0212	0.0287	206978.70	1.15424E-07

Sample Calculations:

For $T = 30^\circ C$,

$$C_T = \frac{273.15}{22.4(T+273)} = 0.040245$$

$$C_A = \frac{0.2}{1} \times C_T = 0.008049 \text{ kmol/m}^3$$

$$C_{B_1} = \frac{273.15}{22.4(T+273)} = 0.040245 \text{ kmol/m}^3$$

$$C_{B_2} = \frac{1 - 0.2}{1} \times C_{B_1} = 0.032196 \text{ kmol/m}^3$$

$$S = \frac{\Delta \left(\frac{\theta}{\pi - \pi_0} \right)}{\Delta (\pi - \pi_0)} \quad (\because \text{only two points})$$

$$= \frac{30226.70025 - 34883.72093}{0.0397 - 0.0172} = 548245.61 \text{ s/cm}^2$$

$$D_{AB} = \frac{\rho C_{BH}}{2 M_A S C_A C_T} = \frac{1.59 \times 1000 \times 0.036041}{2 \times 0.15 \times 548245.61 \times 0.008049} \times \frac{1}{0.040245}$$

$$\therefore D_{AB} = 1.052 \times 10^{-3} \text{ m}^2/\text{s}$$

Similarly, For $T = 40^\circ C$, $D_{AB} = 7.591 \times 10^{-8} \text{ m}^2/\text{s}$

For $T = 50^\circ C$, $D_{AB} = 1.154 \times 10^{-7} \text{ m}^2/\text{s}$

Discussion:

- Volatile component is filled in the T-tube and air passed over it by the pump and change in the level is seen by the sliding microscope.
- We have made the following assumptions to obtain the diffusivity flux equation:
 - Gas A and B behave as ideal gases under the experimental conditions.
 - D_{AB} is independent of concentration
 - Quasi steady-state conditions throughout the diffusion time.
 - Vapor B = air is insoluble in Liquid A = CCl_4 .
- Diffusivities at different temperatures decreases as the temperatures increase gradually,
- which is evident from the equations. Effect of temperature and pressure on co-efficient of diffusion, D is expressed as: $D = \text{const. } T^{1.5}/P$.

Result and Conclusion:

- Relevant points:
 1. The difference in the values, at different temperatures may be accounted to the possible errors during the experiment and varying surrounding conditions though the diffusivity obtained from the experiment lies in the close vicinity of the theoretical values.
 2. The main components of the experimental setup are: glass T-Tube, constant temperature water bath, air pump, and the travelling microscope.
 3. Diffusivity of a binary compound system is a function of temperature, pressure and concentration of the substance.
 4. At the beginning of the experiment, one should check the water bath to see if it is turned on, then set at the right temperature, and filled with water. Water baths should be filled with distilled water. If someone using the water bath for an experiment then the temperature is to be checked frequently to make sure that the water bath is maintaining the proper temperature.
 5. Diffusivities at all three different temperatures are found. Diffusivities decreased as the temperatures increase gradually, however, the length readings for $T=50^{\circ}\text{C}$ doesn't seem to be consistent with other temperatures, due to which there is a little higher value in diffusivity than the expected value.
 6. Assumptions made:
 - Vapour B i.e (air) is insoluble in liquid A(CCl_4)
 - Ideal behaviour of A, B (air and CCl_4) even in an experimental setup
 - Quasi-steady-state prevails in the whole process of diffusion
- Precautions:
 1. We have to ensure that the experimental apparatus is clean and dustproof
 2. Make sure that the air pump is working fine and the water bath is maintaining a constant temperature through stirring for avoiding possible error
 3. Preheating the heater should be avoided and inspecting the heater device for avoiding any sort of electrocution through water in water bath
 4. Precaution should be taken to while setting up the U tube and the air pump to avoid possible errors
- Sources of errors:
 1. Human error can be caused through Parallax error which calibrates the CCl_4 liquid height through the microscope
 2. While slope finding, if done manually might be a great chance of error in drawing a regression line for the scatter plot
 3. Temperature fluctuations can give a possible error in the readings



Studies on Batch Distillation

Objective :

- To perform Batch Distillation for a Binary liquid mixture
- To verify Rayleigh equation for Batch Distillation

Theory:

Batch distillation is the process of distillation in which the feed is distilled in batches. There is only one stage of vapour-liquid contact in its most basic form. A specified amount of liquid mixture is charged into the vessel to be separated by boiling following condensation. In certain cases, an external heater is used to circulate the still charge. The vapor that exits the still is directed to a condenser, where it condenses and collects in the accumulator vessel. Because the distillate becomes heavier with time, it can be collected at varied time intervals as batches of product with the required composition.

Advantages	Limitations
<ul style="list-style-type: none">● Simpler operation● Cheaper for small scale● Flexible in operation● Several products can be distilled from the same equipment	<ul style="list-style-type: none">● Low Productivity● Inherently less thermally efficient● Bigger sized equipment● Higher capital investment for a moderate to higher capacity distillation plant for average daily processing capacity● Higher risk of thermal degradation/decomposition

In a Batch Distillation, a batch of liquid is charged to a kettle fitted with a heating device. The charge is slowly boiled, and the vapours are condensed and collected as distillate. If this distillation proceeds infinitely slowly then the vapour issuing from the liquid would be in equilibrium with the liquid, if the heating is rapid the latter condition would never be achieved.

Applying a differential analysis on concentration balance to the batch process for obtaining Rayleigh equation,

Initial Amount of Feed-in still = Amount of Feed left in still + Amount evaporated

$$xL = (x - dx)(L - dL) + ydL$$

$$\Rightarrow Ldx = ydL - xdL$$

$$\Rightarrow \ln\left(\frac{L_1}{L_2}\right) = \int_{x_2}^{x_1} \frac{1}{y-x} dx$$

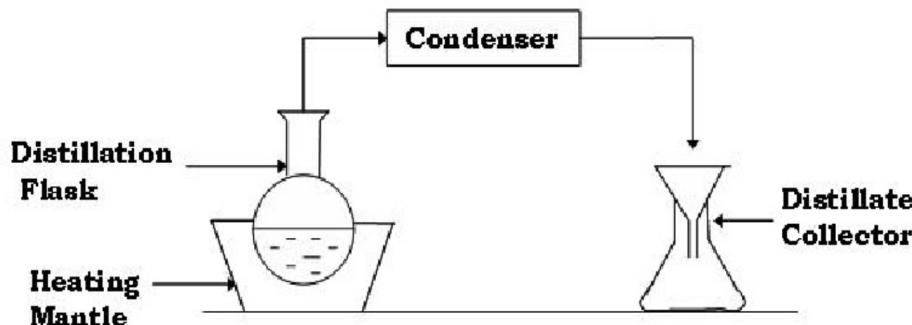
$$(or) \quad \ln\left(\frac{F}{W}\right) = \int_{x_w}^{x_F} \frac{1}{y^*-x} dx$$

The more volatile component is richer in the vapour than in the liquid remaining in the still. Over time, the liquid remaining in the still begins to experience a decline in the concentration of the more volatile component, while the distillate collected in the condenser becomes progressively more enriched in the more volatile component. The exact relationship for a particular mixture may be obtained from a thermodynamic analysis depending on temperature and pressure. For a system following the ideal behaviour given by Raoult's law, the equilibrium relationship between the vapour composition y (or x_D) and liquid composition X (or x_B) of the more volatile component in a binary mixture can be approximated using the concept of constant relative volatility (α), which is given by,

$$y = \frac{\alpha x}{(\alpha-1)x+1}$$

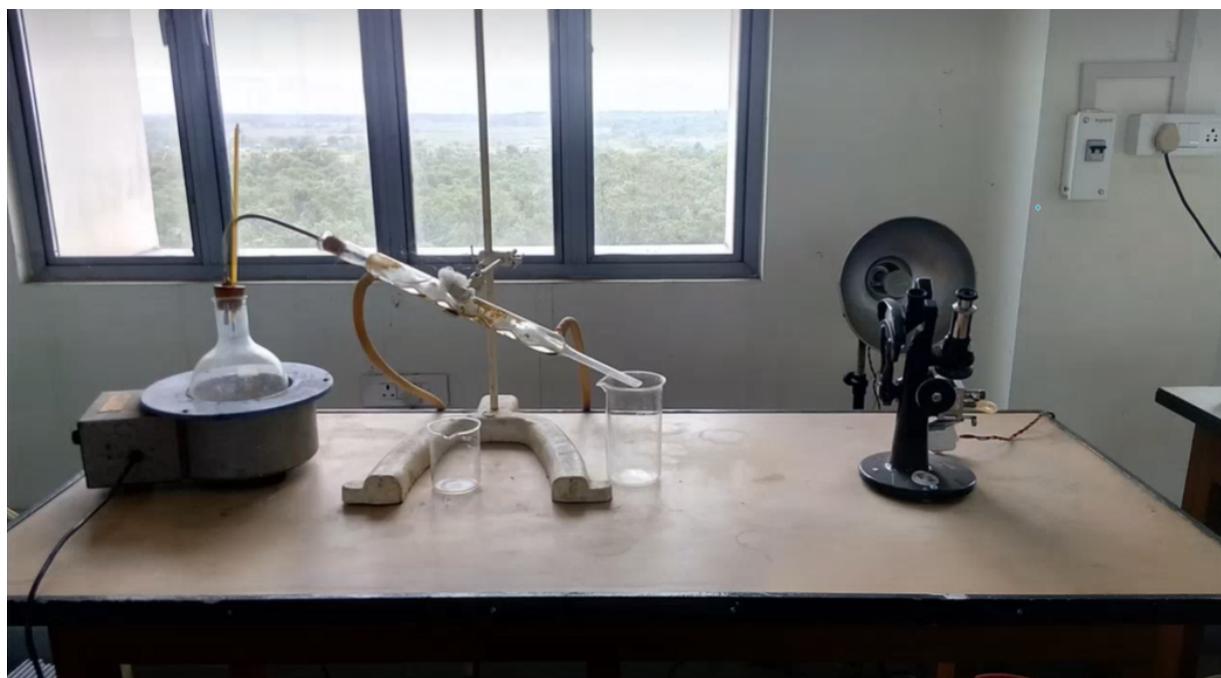
Experimental Details:

EXPERIMENTAL SETUP



The set-up consists of a round-bottomed flask, a condenser, distillate receiver and other accessories like thermometer, heating mantle, etc. In the round-bottomed flask, a binary liquid

combination in specific quantities is placed and slowly heated. The distillation is stopped when one-third of the liquid in the flask has been distilled over. The components of the distillate and residue are determined. For the material balance equation, the volumes of the distillate and residue are also measured. W and xW are derived using the experimental value of $x_{D,\text{avg}}$ for the finished distillate.



Procedure

- In the round-bottomed flask, a binary liquid mixture in specific proportions is slowly heated.
- Distillation is stopped after one-third of the liquid in the flask has been distilled over. The components of the distillate and residue are determined.
- Samples from the distillate and residue are examined in a Refractometer to get the refractive index of both the mixtures to obtain the mixture volume composition.
- A linear calibration curve is plotted and the mixture composition is calculated from the equation for the curve which is further used to calculate the moles present in the distillate mixture.
- For the material balance equation, the volumes of the distillate and residue are also measured. W and xW are derived using the experimental value of $x_{D,\text{avg}}$ for the final distillate.

- The integral in equation (1) can be solved analytically (provided the relationship between y^* and x) or graphically (by calculating the area under the curve) for the plot of $1/(y^*-x)$ vs x .

Experimental data and Observation Table:

- $F = 100 \text{ ml of Toluene} + 100\text{ml of Acetone}$
- Total Volume Balance: $F = D + W$
- Toluene component Balance: $F (X_f) = D (X_d) + W (X_w)$
- Given, when one-third of the liquid in the flask is distilled over, the distillation is discontinued. So, $D = F / 3$; $W = 2 F / 3$
- Refractive Index,
 - i. Distillate = 1.375
 - ii. Residue = 1.464
 - iii. Feed = 1.4134
- Density,
 - i. Toluene (more volatile component) = 0.867 g/cc
 - ii. Acetone = 0.791 g/cc
- Calibration Data:

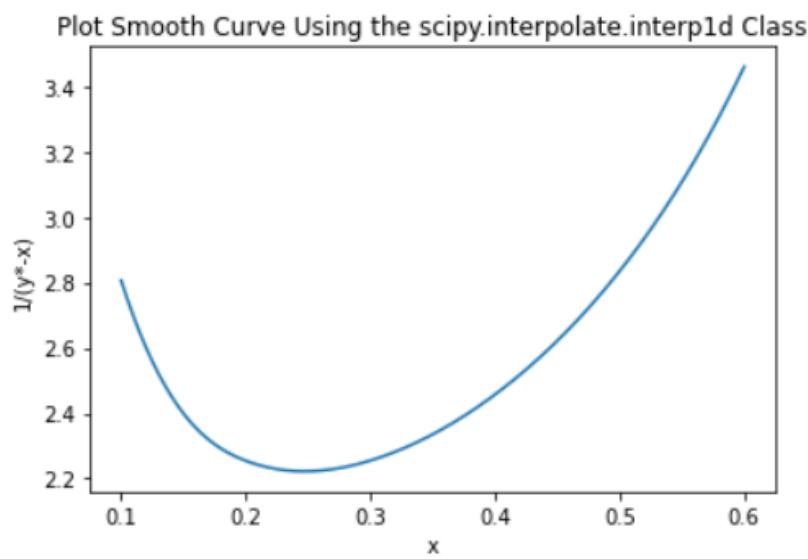
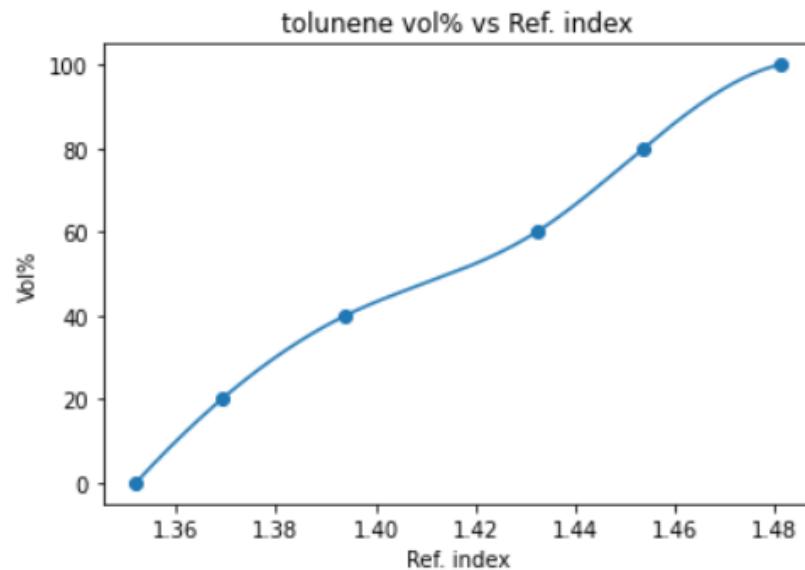
Toluene concentration (% volume) v/s Refractive index

Toluene concentration (% volume)	Refractive Index
0	1.3586
20	1.3692
40	1.3945
60	1.4323
80	1.4537
100	1.4965

- Equilibrium Data:

x	0	0.066	0.176	0.232	0.294	0.357	0.404	0.473	0.521	0.663	0.720	0.774	0.848	1.0
y	0	0.472	0.690	0.743	0.785	0.818	0.838	0.864	0.879	0.918	0.933	0.945	0.963	1.0

- Graph for computing vol% of toluene and for RHS of Reyleighs equation:



Sample Calculation

Given that the distillation is done $\frac{1}{3}$ rd of feed is distilled.
As, $F = 100 \text{ ml Acetone} + 100 \text{ ml Toulene}$

$$\therefore (F)_{\text{vol}} = 200 \text{ ml}$$

$$(D)_{\text{vol}} = \frac{1}{3} \times 200 = 66.67 \text{ ml}$$

$$(w)_{\text{vol}} = \frac{2}{3} \times 200 = 133.33 \text{ ml}$$

Now as we saw after interpolation of the refractive index v/s volume of toulene graph.

	Ref. Index	VOL% Toulene
Distillate	1.375	24.769%
Residue	1.464	79.38462%

$$\therefore \text{For Feed, } (Toluene)_{\text{vol}} = 200 \text{ ml}$$

$$(\text{Acetone})_{\text{vol}} = 100 \text{ ml}$$

$$\rho_{\text{acetone}} = 0.79 \text{ g/ml}, \quad \rho_{\text{acetone}} = 79 \text{ g/mol}$$

$$\text{No. of moles of acetone} = \frac{W}{M_w} = \frac{79}{58.08}$$

$$[n_{\text{acetone}}]_F = 1.3601 \text{ moles}$$

$$\text{similarly, } \rho_{\text{toulene}} = 0.866 \text{ g/mol}$$

$$n_{\text{toulene}} = \frac{86.6}{92.14}$$

$$\text{Now, } x_F = \frac{n_{\text{toul}}}{n_{\text{acetone}} + n_{\text{toul}}} = 0.591371$$

$$F = 2.8006 \text{ moles.}$$

Similarly, for distillate

$$(D)_{\text{vol}} = 66.67 \text{ ml}; \quad (D_{\text{acetone}})_{\text{vol}} = 50.15385 \text{ ml}$$

$$(D_{\text{toulene}})_{\text{vol}} = 16.51282 \text{ ml}; \quad n_{\text{acetone}} = 0.68218 \text{ moles}$$

$$n_{\text{toulene}} = 0.1552 \text{ moles}$$

$$\therefore y_D = \frac{n_{\text{Tolu}}}{n_{\text{Tolu}} + n_{\text{acet}}} = 0.814662$$

Similarly, for residue

$$w_{\text{vol}} = 133.33 \text{ mL}$$

$$(w_{\text{Toulene}})_{\text{vol}} = 105.84 \text{ mL}$$

$$(w_{\text{acetone}})_{\text{vol}} = 27.487 \text{ mL}$$

$$n_{\text{acet}} = 0.79002 \text{ moles}$$

$$n_{\text{Toulene}} = 0.67298 \text{ moles}$$

$$\therefore x_w = 0.46$$

Now finding RHS of Rayleigh eqn., $\int_{x_w}^{x_f} \frac{1}{y^* - x} dx$
 finding using eqn. data, we get, $(\text{RHS}) = 0.46563$ using
 trapezoidal rule.

$$\text{Now, WKT, } (\text{LHS}) = \ln(F/w)$$

$$F = n_{\text{Tolu}} + n_{\text{acet}} = 2.3 \text{ moles}$$

$$w = n_{\text{Tolu}} + n_{\text{acet}} = 1.463 \text{ moles}$$

$$\therefore \ln \left(\frac{2.3}{1.463} \right) = 0.47526$$

$$\text{As } (\text{LHS}) = 0.47526, \quad (\text{RHS}) = 0.46563$$

$$\therefore \text{Error} = 2.08\%$$

As LHS \approx RHS, thus Rayleigh's eqn. is verified

Discussions:

- The experimental (LHS) and calculated (RHS) values in the Rayleigh equation were found to be approximately equal, thus, confirming the correctness of the equation.

-
- Calibration chart prepared for Acetone - Toluene binary mixture shows that the refractive index increases with increase in volume fraction of Toluene in the mixture, implying a higher value of refractive index of pure Toluene as compared to pure Acetone, which is in accordance with the scientific values.
 - From the x-y* equilibrium data, we can conclude that the rate of separation is comparatively lesser at higher mole fractions, and that complete separation of the two mixture is not feasible. This is not the case with continuous distillation, where there is no saturation and complete separation is possible. It was uncharacteristically low compared to the adjacent values so we didn't consider it in order to maintain the uniformity of values recorded.

Result and Conclusion:

Relevant points:

- Batch Distillation has been performed for the given Toluene-Acetone Mixture and respective refractive indices have been recorded.
- Rayleigh Equation is verified using the Equilibrium and Calibration data, by solving the integrand with a best-fit curve and then finding the area subtended by it.
- Error Analysis of Rayleigh Equation:
 - Experimental Value (LHS – obtained from Calibration data) = 0.47526
 - Theoretical Value (RHS - obtained from the Equilibrium data) = 0.4656
 - Error incorporated = 0.0208

Precautions and sources of errors:

- The distillation flask should be handled with care and the thermocouple should be placed in a vertical direction to avoid error in reading.
- Weight of residue and distillate should be measured when the subsystem cools down little to prevent the wrong calculation of volume.
- Constant heat flux should be provided for the distillation of the binary mixture of Acetone and Toluene.

Sources of errors:

- Take adequate amounts of mixture feed so that temperature sensor remains dipped in the solution throughout the experiment. This can result in system error.
- While taking sample solution for measuring the refractive index, excess amount could be taken than that is optimally required, as it is done manually using a pipette. If a micropipette was provided, the accuracy of the experiment could have increased.



Separation of two miscible 8-11 liquids in a Sieve plate Distillation column

Objective :

- To study the different parts of a distillation column.
- To determine the performance of the column.
- To compare the experimental results with those calculated by the McCabe-Thiele method and Fenske method.

Theory:

- Distillation is a process by which a liquid mixture is separated into fractions with higher concentrations of certain components by exploiting differences in relative volatility. The lighter, lower boiling point components evaporate and travel up the column to form the top product, and the heavier, higher boiling point components condense and travel down the column to form the bottom product.
- Nomenclature and brief write-up on the parts of the Column:
 1. **Feed** - The liquid and/or gas feed into the distillation column. The tray below the inlet nozzle is called the feed tray.
 2. **Stripping section** - The trays between the bottom of the column and the feed tray. In the stripping section, the aim is to concentrate the heavier component in the liquid phase.
 3. **Rectifying section** - The trays between the feed tray and the top of the column. In the rectifying section, the aim is to concentrate the lighter component in the vapour phase.
 4. **Reflux** - A portion of vapour from the top of the column has been condensed to a liquid and returned to the column as a liquid above the top tray.
 5. **Total Condenser** - All the vapour leaving the top of the column is condensed. Consequently, the composition of the vapour leaving the top tray is the same as that of the liquid distillate product and reflux.
 6. **Partial Condenser** - The vapour is only partially liquefied. The liquid produced is returned to the column as liquid, and a vapour product stream is removed. A partial condenser functions as an equilibrium separation stage, so columns with a partial condenser effectively have an extra ideal stage.
 7. **Reboiler** - A heat exchanger at the bottom of the column which boils some of the liquid leaving the column. The vapour generated returns to the column at the bottom of the stripping section.
 8. **Vapour-Liquid Equilibrium (VLE) Curve** - A plot of the actual composition of the lighter component in the vapour phase for a given composition in the liquid phase. Usually derived from thermodynamic data.

- For calculation purposes, It is assumed that the plates of the column behave as an ideal plate or theoretical plate where the time of contact between phases is sufficient so that the exit streams are in equilibrium. However, in reality, all the plates do not function to yield equilibrium composition for exit streams.
- We can find theoretical minimum trays required in the distillation column using **the Fenske equation** which goes as:

$$N = \frac{\log \left[\left(\frac{X_d}{1-X_d} \right) \left(\frac{1-X_b}{X_b} \right) \right]}{\log \alpha_{avg}}$$

Experimental Details:

EXPERIMENTAL SETUP

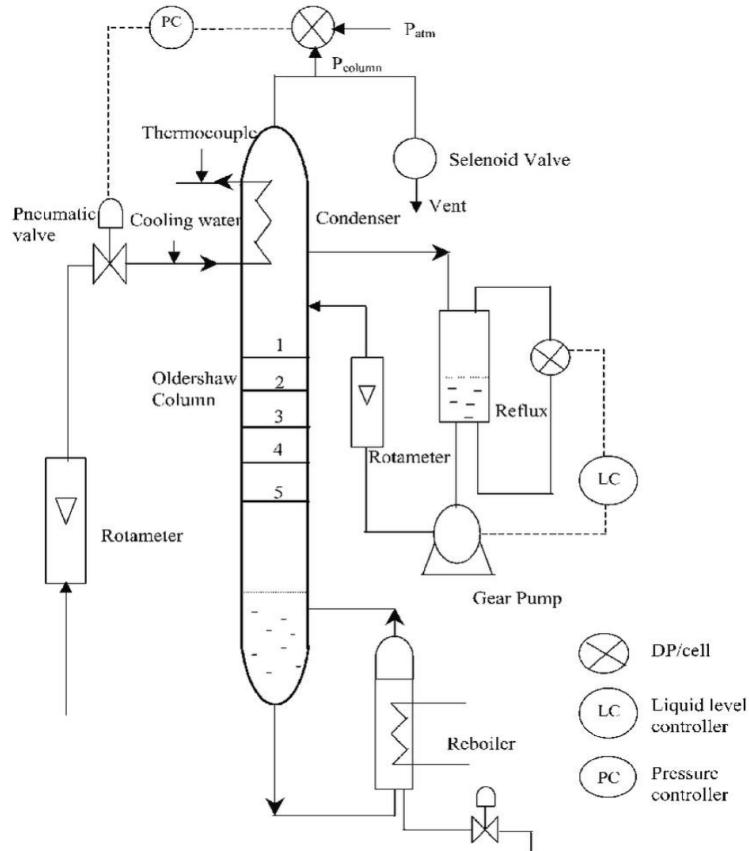


Fig. 01. Experimental setup

The experimental set-up consists of a column containing 8 sieve plates, reboiler, condenser, cooler, feed tank, distillate tank, etc. and accessories like rotameter, pump, heaters, digital temperature indicators, etc.

In the reboiler, a mixture of ethyl alcohol and water of unknown composition is placed (nearly half of the reboiler volume is filled up with this mixture). The refractive index method is used to determine its makeup. After that, the reboiler heaters are turned on. The distillation column gradually warms up. The cooler condenser allows the cooling water to pass through. When there is a significant amount of condensate in the distillation tank, the reflux pump is turned on to allow the condensate to enter the top plate.

Each plate's liquid samples are collected and reboiler (before the start of the operation and during the operation). Temperatures are recorded at many locations. The refractive index technique is used to analyze the liquid samples. We operate the same in total Reflux condition, which is $R=L/D=(\text{infinity})$

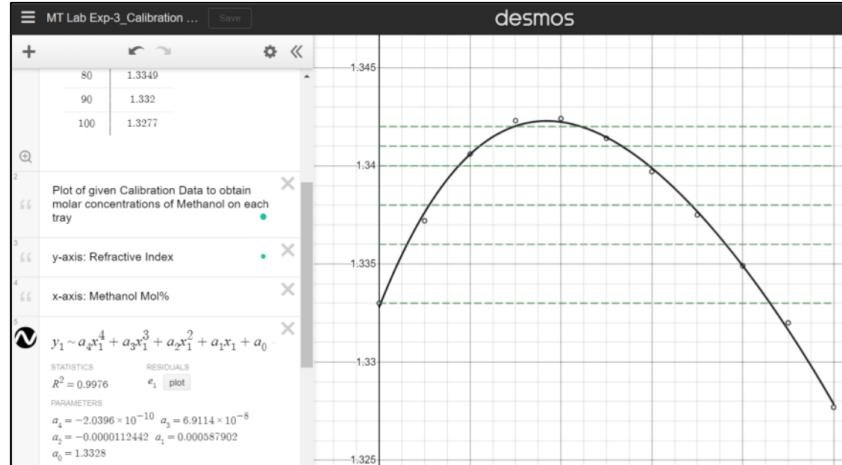
Procedure

- A mixture of ethyl alcohol and water of some unknown composition is put in the reboiler. Its composition is determined by the refractive index method.
- When considerable condensate accumulates in the distillate tank, the distillate is allowed to enter the top plate by switching on the reflux pump.
- The analysis of the liquid samples is made using the technique of refractive index.
- The column is operated under total reflux condition.
- A linear calibration curve is plotted and used to calculate the mole fraction in the distillate and reboiler mixture.
- Number of minimum ideal trays and ideal trays are calculated using Fenske's equation and McCabe Thiele's graphical method, respectively and the data is used to calculate the tray efficiency for the column.

Experimental Data and Observation Table:

Using the above data we found the trend line to find the ethanol vol% in each tray,

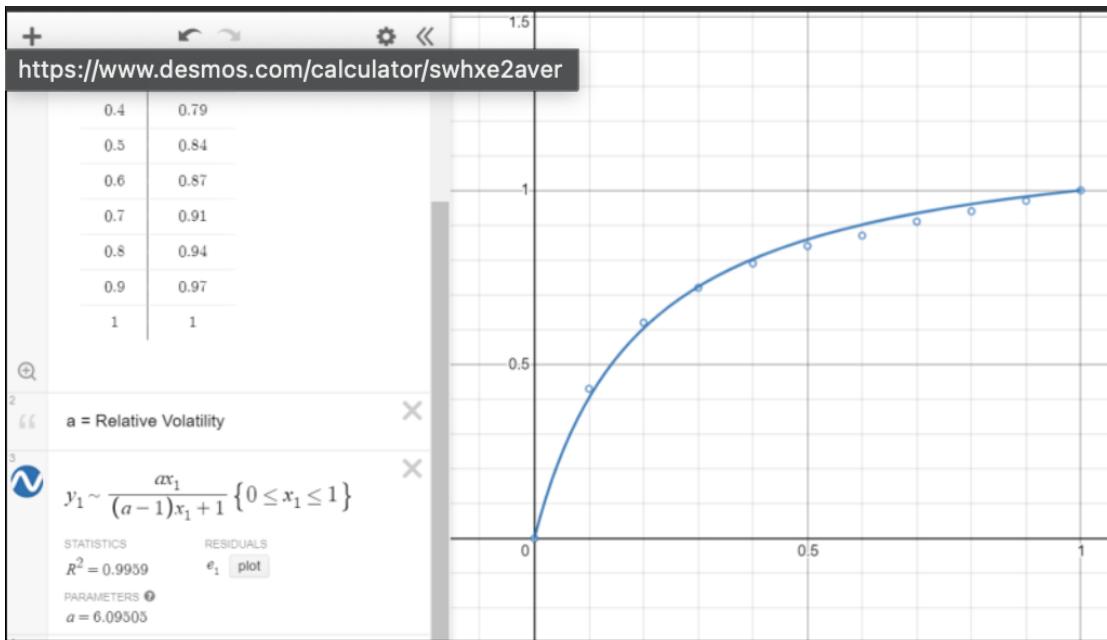
Mol% Methanol	Refractive index
0	1.333
10	1.3372
20	1.3406
30	1.3423
40	1.3424
50	1.3414
60	1.3397
70	1.3375
80	1.3349
90	1.332
100	1.3277



Location	Refractive index	Methanol %Molar Concentration
Distillate	1.333	86.019
Plate 1	1.336	76.272
Plate 2	1.338	68.664
Plate 3	1.341	53.408
Plate 4	1.342	44.376
Plate 5	1.342	29.71
Plate 6	1.34	17.496
Plate 7	1.34	17.496
Plate 8	1.338	10.999
Reboiler	1.336	6.126

- Equilibrium Data Analysis:

x	y*
0	0
0.1	0.43
0.2	0.62
0.3	0.72
0.4	0.79
0.5	0.84
0.6	0.87
0.7	0.91
0.8	0.94
0.9	0.97
1	1



- From the above two data tables & Plots:

$$xF = 0.44376$$

Feed Tray = Plate 4

$x_D = 0.86019$ (Distillate)

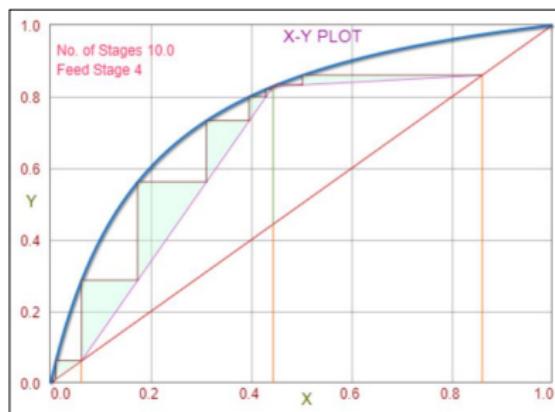
$x_W = 0.06126$ (Reboiler)

quality, $q = 1$ (Feed is pure liquid)

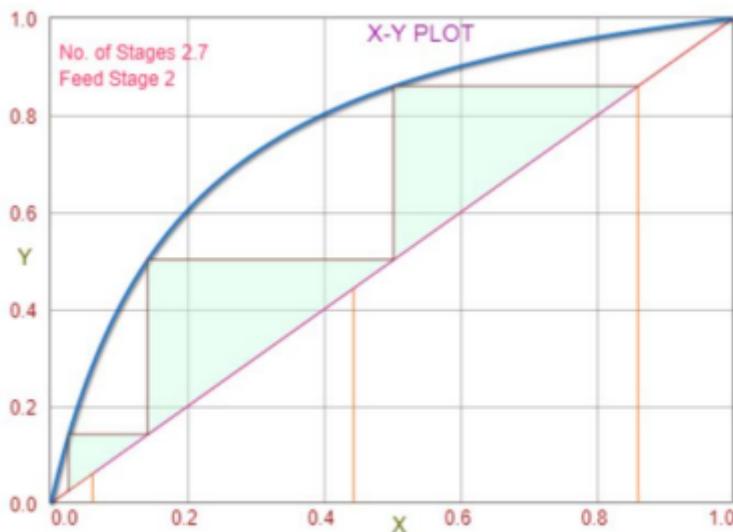
Therefore, feed line in McCabe-Thiele plot is vertical

- McCabe-Thiele Method:

- To obtain 10 theoretical stages for given Feed configuration, required Reflux Ratio (R) = 0.0812



- To obtain a minimum number of plates using the McCabe-Thiele method, we need to consider the Total Reflux condition i.e., $D = 0$ and $R = \infty$, hence, Rectifying line and Stripping line are $y=x$. This is because the entire area between the equilibrium curve and diagonal line is used for separation, with the largest possible driving force.



The minimum number of theoretical Stages = 3 (To obtain similar concentrations in Distillate and Condensate for the given Feed)

- Section Efficiency using theoretical minimum and actual stages:
 $N_{\text{section}} = n_{\text{theoretical minimum}}/n_{\text{actual}} = 3/9 \times 100 = 33.33\%$

Murphree Tray Efficiency considering the compositions on Plate-1:

Actual compositions: $y_n = 0.763$; $y_{n-1} = 0.687$;
Equilibrium composition leaving nth tray $y_n^* = 0.859$

$$n_{\text{tray}} = (0.763 - 0.687) / (0.860 - 0.687) = 44\%$$

Discussion

- For Total Reflux ratio, the area between $y = x$ and equilibrium curve increases implying the infinite reflux ratio; due to this difference between each stage concentrations are more pronounced and required distillation is achieved in minimum number of stages.
- But Total Reflux can be achieved only in instances when we reduce the feed to zero and return all the overhead product back to the column as reflux and re-boil all the bottoms product. Such instances can be interpreted as infinite reboiler heat duty and condenser cooling capacity for a given feed rate.
- For operation at total reflux, sufficient amount of feed is charged into the reboiler, and flow of steam and of cooling water are started. The entire condensate is fed back as reflux. The rate, concentration and thermal state of the feed are not relevant to the column operated at these conditions since, there is no feed input at all.
- Assumptions made for the experiment:
 - Perfect mixing of liquid on any tray with uniform composition over it o liquid and vapor leaving each tray are in thermal equilibrium
 - Negligible operating pressure influence along the column

Result and Conclusion:

- Relevant points:
 1. The low-efficiency values in both sectional and tray calculations are due to the configuration chosen. For the given concentrations of Methanol in Feed, Distillate, and Condensate, we can get better efficiency either by changing the Reflux Ratio or by changing the feed tray location.
 2. When chosen Total Reflux ratio, the area between $y=x$ and equilibrium curve increases implying the infinite reflux ratio; due to this difference between each stage concentrations are more pronounced and required distillation is achieved in a minimum number of stages (steps in McCabe-Thiele plot).

But practically, Total Reflux can be achieved only in instances when,

- Reducing the feed to zero.
- Returning all the overhead products back to the column as reflux and Reboiling all the bottoms products.

Such instances can be interpreted as infinite reboiler heat duty and condenser cooling capacity for a given feed rate.

3. Few assumptions we made while running the experiment are:
 - Perfect mixing of liquid on any tray with uniform composition over it.
 - Liq. and Vap. leaving each tray are in thermal equilibrium.
 - Negligible Operating Pressure influence along the column.
 - Vapour hold up is negligible compared to Liquid hold up.

- Negligible coolant and steam dynamics in condenser and reboiler, respectively.
 - Readings are noted down once the setup crosses the start-up phase and reaches the production phase i.e., a steady-state operation with maximum obtainable purity.
4. We see that the McCabe Theile method gave better efficiencies as it's a standard practise method and less approximation is done while comparing to the Fenkse method
- Precautions and sources of errors:
 - If we don't obtain any Distillate even while all the accessories are properly functioning, we can increase the steam flow rate in Reboiler, so that we can increase the vapours making it reach the condenser.
 - We need to minimize the errors in readings of rotameter, pump, heaters, digital temperature indicators, etc, if the data is turning out to be inconsistent such as rate of change in the refractive indices values, abnormal local maxima & minima, etc.