Momentum Transfer and Mechanical Operations Lab

Measurement of Diffusivity using Stefan's Tube

14th October, 2024

Team: MTMO 2

Team Members: Aayush Bhakna (CH22B008), Rapolu Pranay Reddy (CH22B018), Deepanjhan Das (CH22B020), Atharva Sunilkumar Ghodke (CH22B035), Anmol Upadhyay (CH22B053), Lakkireddy Vishnu Vardhan Reddy (CH22B076)

Smail IDs: ch22b008@smail.iitm.ac.in, ch22b018@smail.iitm.ac.in, ch22b020@smail.iitm.ac.in, ch22b035@smail.iitm.ac.in, ch22b053@smail.iitm.ac.in, ch22b076@smail.iitm.ac.in

Instructors: Dr. Abhijit Deshpande, Dr. Nitin Muralidharan, Dr. Sankha Karmakar, Dr. Khushboo Suman

Department of Chemical Engineering, IIT Madras

1 Abstract with Graphics

In this experiment, the primary objective was to determine the diffusivity of acetone in air at varying temperatures using Stefan's Tube (shown in figures 1 & 2). The motivation behind this study lies in the fundamental importance of molecular diffusion in mass transfer operations, which is crucial in various scientific and engineering applications. The experiment was conducted under controlled conditions to measure the rate of diffusion of acetone vapour through a stagnant air film.

Stefan's Tube provides a straightforward method for studying diffusion by establishing a one-dimensional diffusion model. The procedure involved setting up Stefan's Tube in a water bath at specific temperatures and introducing acetone into the tube. The rate of acetone evaporation was determined through the difference in the acetone meniscus position, which was noted using a microscope equipped with a Vernier scale. Using Fick's law and the pseudo-steady-state assumption, the diffusivity was calculated from the slope of a graph plotting the square of the meniscus height difference against time.

The error analysis revealed that the primary sources of uncertainty were in the measurements of temperature, time, and the acetone level. In conclusion, the experiment suc-

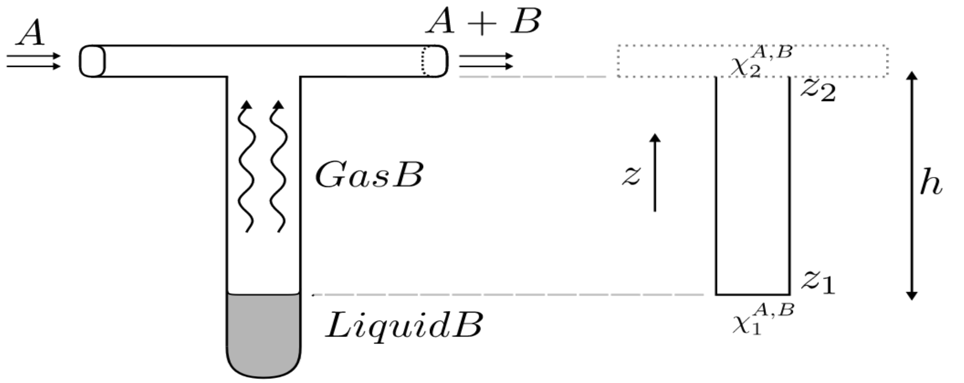


Figure 1

cessfully demonstrated the measurement of diffusivity using Stefan's Tube and provided valuable insights into the temperature dependence of molecular diffusion.The experiment highlights the critical role temperature plays in molecular diffusion The results contribute to a better understanding of mass transfer phenomena, which are essential in designing and optimising industrial processes.

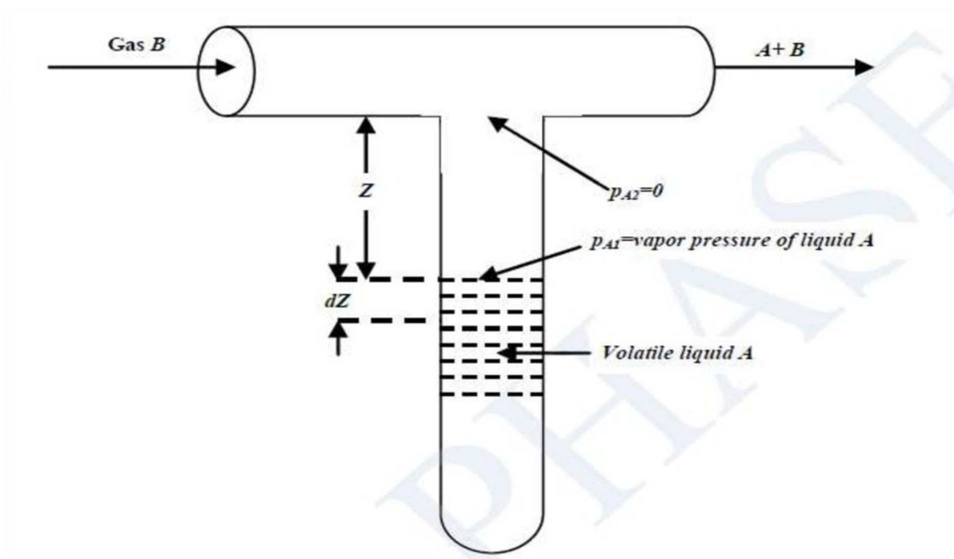


Figure 2

2 Aim & Objectives

- Using Fick's law to derive a pseudo-steady state equation for uni-molecular Diffusion
- Calculating the Vapour Diffusion Coefficient (D_{AB}) of Acetone-Air mixture using Experimental Data

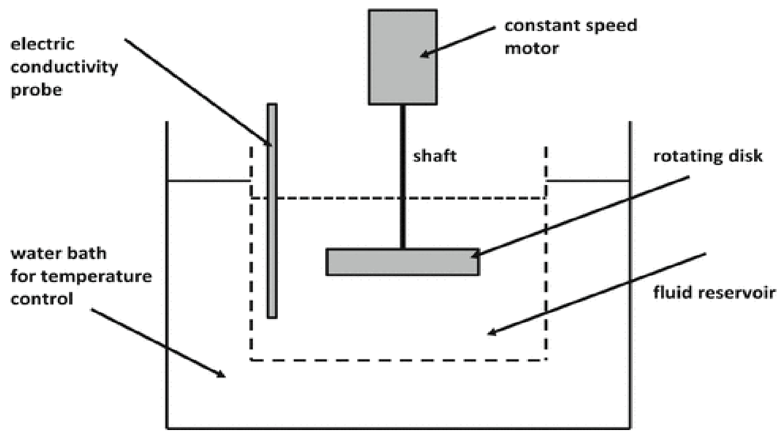


Figure 3: Schematic Setup of the Overall Experiment

3 Background and Motivation

The study of diffusion plays a crucial role in understanding various natural and industrial processes, from gas exchange in biological systems to mass transfer in chemical reactors. Stefan's Tube, named after physicist Josef Stefan, is a fundamental apparatus designed to measure diffusivity, particularly the diffusion of gases through liquids or air. Diffusivity, which quantifies how particles spread out over time due to concentration gradients, is a key parameter in applications such as environmental monitoring, chemical engineering, and material science.

In this experiment, the Stefan tube provides a straightforward yet effective means to examine the diffusion of gases into still air. By analyzing the rate at which a volatile liquid, such as acetone or ether, evaporates from a liquid surface within a vertical tube, we can measure the diffusivity of the vapor into the surrounding gas. This controlled setup allows for the direct observation of diffusion without the interference of external variables like convection.

The motivation behind this experiment is to gain hands-on experience with measuring diffusivity in a laboratory setting, which is vital for designing and optimizing processes such as drying, absorption, and even gas separation technologies. Additionally, the experiment enhances the understanding of fundamental transport phenomena, which are critical in various scientific and engineering disciplines. By using Stefan's tube, students and researchers can apply theoretical diffusion models to real-world scenarios, thus bridging the gap between theory and practice.

4 Materials and Methods

4.1 Apparatus & Materials Required

- **Materials**: Acetone, Distilled Water, Aluminum Foil.
- **Apparatus**: Stefan's Tube, Syringe Airflow Input, Inverting Microscope with Vernier Scale, Stirrer and Heater in a Water Bath.

4.2 Experimental Setup Description

The experimental setup is shown in the following figure 4.

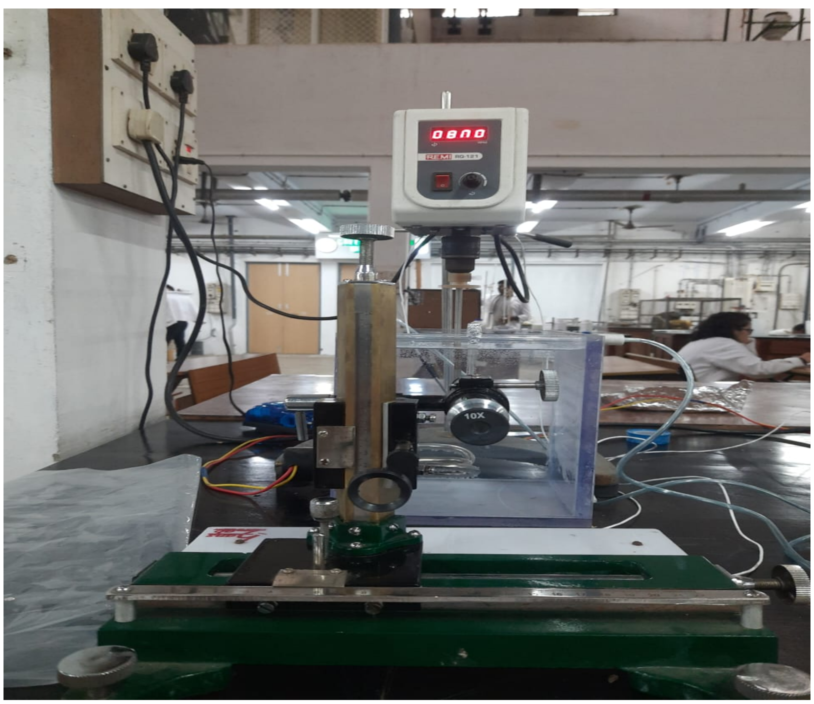


Figure 4: Complete Experimental Set Up

- **Water Bath with Heater and Stirrer**: A water bath equipped with a heater and stirrer is used to maintain and evenly distribute heat at controlled temperatures (40^{o}C, 45^{o}C, and 50^{o}C).

- **Stefan's Tube**: A Stefan's tube filled with acetone is placed in the heated water bath for diffusion studies.
- **Sealing**: Aluminum foil is used to seal the openings of the tube to prevent acetone vapor from escaping, except for a small air inlet for airflow input.
- **Airflow Input**: Airflow is introduced into the system through a controlled inlet to facilitate the acetone evaporation and diffusion process.
- **Inverting Microscope**: An inverting microscope with a Vernier scale is used to track the movement of the acetone meniscus. The microscope is aligned with the meniscus and adjusted periodically to record position changes.

This setup allows precise monitoring of acetone evaporation and diffusion under controlled temperature conditions.

4.3 Procedure

- Using a syringe, pour acetone into the Stefan tube until it reaches an approximate depth of 7 cm.
- Insert the capillary tube through a rubber ring and into the metal nut, ensuring that the top of the tube rests securely on the nut.
- Carefully screw the capillary tube assembly onto the top plate, positioning the 'T' piece perpendicular to the microscope.
- Attach a flexible air tube to one end of the 'T' piece.
- Adjust the microscope, focusing the objective lens until the capillary tube is clearly visible.
- If the capillary tube remains unclear, modify the vertical height of the microscope or adjust the distance between the objective lens and the tank until clarity is achieved.
- Fine-tune the microscope's view to ensure the meniscus inside the capillary tube is sharp and well-defined. Note that the image will appear inverted, with the top corresponding to the bottom of the capillary tube.
- Align the sliding Vernier scale with an appropriate graduation on the fixed scale.
- Turn on the air pump to initiate the experiment.
- Activate the temperature-controlled water bath, setting it to 40°C, and wait until the temperature stabilizes.

- Measure and record the acetone level in the Stefan tube using the Vernier scale.
- Record the acetone level within the capillary tube at intervals of 10, 20, and 30 minutes.
- Repeat the procedure at temperatures of 45^{o}C and 50^{o}C, ensuring recordings at the same time intervals.
- Ensure that the water bath temperature does not exceed the boiling point of acetone (56^{o}C).

5 Observation Tables

The tabulations include the observed data from each of the sub-parts of the overall experiment and are tabulated in the following tables (tables 1, 2 & 3):

t (min)	MSR (mm)	VSR (mm)	TSR (mm)
0	23.0	3	23.03
5	22.5	28	22.78
10	22.5	11	22.61
15	22.0	25	22.25
20	22.0	0	22.00
25	21.5	33	21.83

Table 1: [Case 1] T = 40^oC

t (min)	MSR (mm)	VSR (mm)	TSR (mm)
0	21.5	23	21.73
5	21.5	32	21.82
10	21.5	5	21.55
15	21.0	27	21.27
20	21.0	0	21.00
25	20.5	12	20.62

Table 2: [Case 2] T = 45^oC

t (min)	MSR (mm)	VSR (mm)	TSR (mm)
0	20.5	10	20.60
5	20.0	0	20.00
10	19.5	15	19.65
15	19.0	10	19.10
20	18.5	30	18.80
25	18.5	5	18.55

Table 3: [Case 3] T = 50^oC

Notations used: t = Time in minutes, T = Temperature, MSR = Main Scale Readings (in mm), VSR = Vernier Scale Readings (in mm), TSR = Total Scale Readings (in mm).

6 Theory & Calculations

6.1 Fick's Law of Diffusion

The Fick's law for diffusion is given by :

$$J_A = -D_{AB}\frac{dC_A}{dz} \tag{1}$$

Rewriting this equation in mole fractions, we get :

$$J_A = -C_A D_{AB}\frac{dy_A}{dz} \tag{2}$$

Now, assuming unimolecular diffusion (Acetone is diffusing, Air is not), we can say that $N_B = 0$. As such, the equation for molar flux becomes :

$$N_A = -C_A D_{AB}\frac{dy_A}{dz} + y_A N_A \tag{3}$$

$$N_A = \frac{-C_A D_{AB}}{1 - y_A}\cdot\frac{dy_A}{dz} \tag{4}$$

Putting this into species material balance, we get :

$$\frac{dN_A}{dz} = 0 \tag{5}$$

$$\frac{d}{dz}\left(\frac{1}{1-y_A}\cdot\frac{dy_A}{dz}\right)=0 \tag{6}$$

We define the Boundary equations as:

$$At\ bottom\ (z=z_1)\ :\ y_A=y_{A1},\ y_B=y_{B1} \tag{7}$$

$$At\ top\ (z=z_2)\ :\ y_A=y_{A2},\ y_B=y_{B2} \tag{8}$$

Finally, after solving the 2nd order ODE equation, we get :

$$N_A=\frac{C_A D_{AB}}{z_2-z_1}\cdot\frac{C_T}{C_{Bm}} \tag{9}$$

This can be rewritten as :

$$N_A=\frac{C_T D_{AB}}{z}\cdot ln\left(\frac{1-y_{A2}}{1-y_{A1}}\right) \tag{10}$$

Here,

$$Total\ Conc\ :\ C_T=\frac{P}{RT} \tag{11}$$

$$Liquid\ Depth\ from\ Top\ :\ z=z_2-z_1 \tag{12}$$

$$y_{A1}=\frac{C_{A1}}{C_T}=\frac{P_{vap}}{P} \tag{13}$$

$$y_{A2}=0 \tag{14}$$

6.2 Deriving Pseudo Steady-State Equation

For the case of pseudo steady-state :

$$N_A=\frac{\rho_A}{M_A}\cdot\frac{dz}{dt} \tag{15}$$

Equating this with the N_A derived using Fick's Law, we get the following dynamic relation:

$$z_t^2=z_o^2+\tau D_{AB}\cdot t \tag{16}$$

This can be rewritten as :

$$D_{AB}=\frac{z_t^2-z_o^2}{\tau\cdot t} \tag{17}$$

Where,

$$\tau=\frac{2\ C_T M_A}{\rho_A}\cdot ln\left(\frac{1-y_{A2}}{1-y_{A1}}\right) \tag{18}$$

Here, z_t is the liquid depth at time t and z_o is the initial depth at $t = 0$. M_A is the molecular weight of Acetone and ρ_A is the density of Acetone in liquid phase.

$$z(t) = H - TSR(t) \tag{19}$$

$$H = 55\ mm \tag{20}$$

6.3 Theoretical Diffusivity

To get the theoretical values of D_{AB} at different temperatures, we will be using the equation given in Perry's Chemical Engineers' Handbook (Eq. 2-152), which gives the diffusivity of Air-Hydrocarbon gas mixtures.
The equation is given below :

$$D_{AB} = \frac{0.01013 \cdot T^{1.75} \left(\frac{1}{M_A} + \frac{1}{M_B}\right)^{0.5}}{P\left[\left(\sum v_A\right)^{1/3} + \left(\sum v_B\right)^{1/3}\right]^2} \tag{21}$$

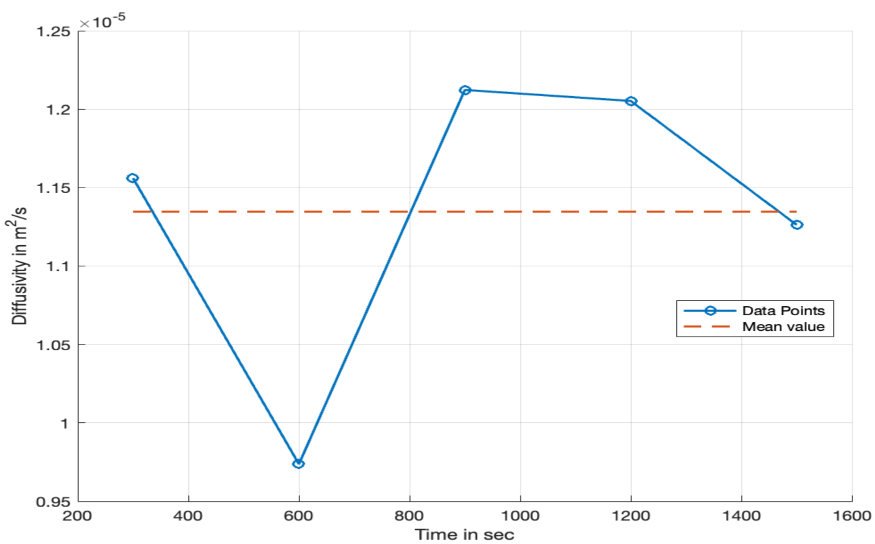


Figure 5: Case 1 : Diffusivity at 40^{o}C

The temperature is in Kelvin, pressure is in Pascals, D_{AB} is in m^2/s, M is the molecular weight in $kg/kmol$ and other values are give below :

$$Air\ :\ \sum v_B = 20.1 \tag{22}$$

$$Acetone\ :\ \sum v_A = 66.861 \tag{23}$$

6.4 Final Results

For n samples collected, the equation for experimental D_{AB} becomes :

$$D_{AB,\ exp} = \frac{1}{n-1} \sum_{i=2}^{n} \frac{z_i^2 - z_o^2}{\tau \cdot t_i} \tag{24}$$

It is to be noted that we are ignoring the $i = 1$ case as $t_1 = 0$ and $z_1 = z_o$.

The graphs of the $D_{AB,\ exp}$ values at different temperatures is given below (figures 5, 6, 7):

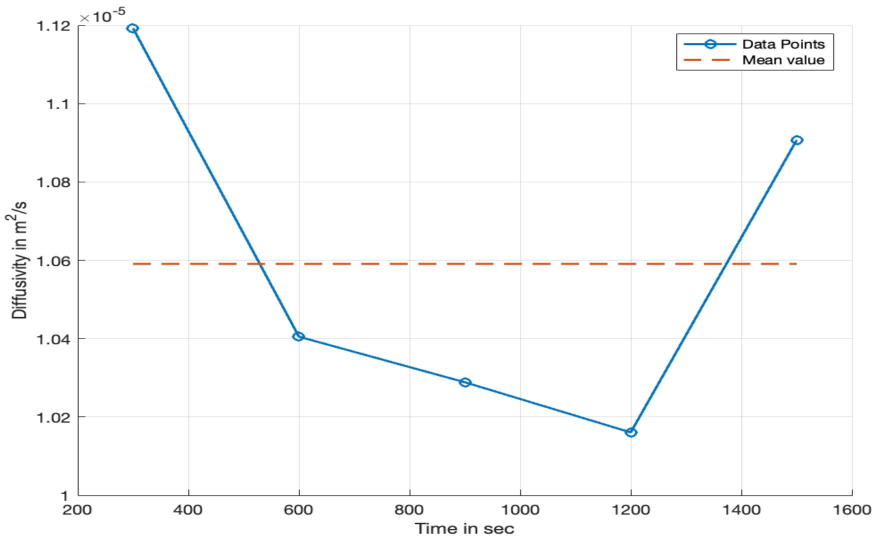


Figure 6: Case 2 : Diffusivity at 45^{o}C

The tabulations of all results is given below :

T (^oC)	P_{vap} (kPa)	y_{A1}	τ	$D_{AB,\ exp}$ (cm^2/s)	D_{AB} (cm^2/s)	Error (%)
40	56.187	0.5545	0.004627	0.11347	0.11541	01.68
45	67.834	0.6695	0.006235	0.10591	0.11865	10.74
50	81.366	0.8030	0.009009	0.12635	0.12194	03.62

Table 4: Diffusivity at different temperatures

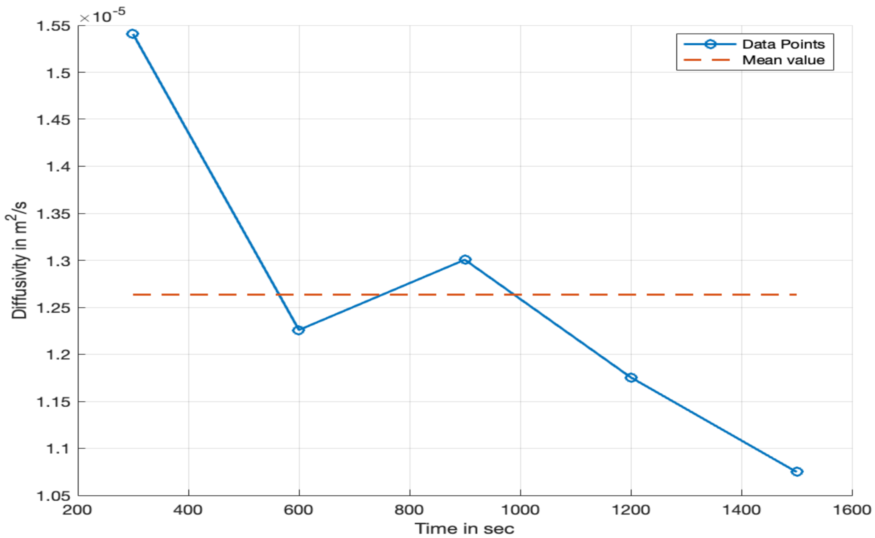


Figure 7: Case 3 : Diffusivity at 50^{o}C

7 Conclusions and Remarks

- It is seen that the Vapour Diffusion Coefficient (D_{AB}) tends to increase as the Temperature (T) rises.
- The value of Molar flux (N_A) also increases as temperature is increased.
- The Case-1 and Case-3 show a very low error. We can thereby conclude that the Pseudo Steady-State equation derived from Fick's law gives a reasonably accurate value of D_{AB}, which can be used in future calculations.
- The high error in Case-2 will be discussed in the Error Analysis section.

8 Error Analysis

Error in Timer $\equiv \Delta t = 5$ sec $= 0.0833$ min
Least Count of Vernier Calliper $\equiv \Delta z_t = 0.01\ mm$
Least Count of Main Scale $= 0.5$ mm
Least Count of Thermocouple $\equiv \Delta T = 1^oC$

Fractional Error in Vapour Pressure (P_{vap}) :

$$\frac{\Delta P_{vap}}{P_{vap}} = 2.303\left(A + \frac{B}{T+C}\right) \cdot \frac{B}{(T+C)^2} \cdot \Delta T \tag{25}$$

Fractional Error in Mole Fraction of Acetone (y_{A1}) :

$$\frac{\Delta y_{A1}}{y_{A1}} = \frac{\Delta P_{vap}}{P_{vap}} \tag{26}$$

Fractional Error in Tau (τ) :

$$\frac{\Delta \tau}{\tau} = \frac{\Delta T}{T} + \frac{\Delta y_{A1}}{(1 - y_{A1}) \cdot ln\left(\frac{1}{1-y_{A1}}\right)} \tag{27}$$

Fractional Error in Diffusivity ($D_{AB,exp}$) :

$$\frac{\Delta D_{AB,exp}}{D_{AB,exp}} = \frac{\Delta \tau}{\tau} + \frac{\Delta t}{t} + 2 \cdot \frac{\Delta z_t}{z_t} \tag{28}$$

8.1 Correcting Outlier in Case-2

In the Case-2 ($T = 45^o C$), it is seen that the first point is an Outlier. To correct this, we will use linear regression on the other points, and back-calculate (predict) the true value of the first point.

$$Outlier\ :\ t = 0\ sec,\ TSR = 21.73\ mm \tag{29}$$

After performing Linear Regression on the other points, we get :

$$Equation\ :\ TSR\ (mm) = -0.0009833\ t\ (sec)\ + 22.137 \tag{30}$$

$$Validation\ :\ R^2 = 1 - \frac{SS_{Res}}{SS_T} = 0.994935 \tag{31}$$

Seeing as the value of R^2 is fairly high, we can use this equation to predict the true value of TSR at t = 0 sec. The answer comes out to be :

$$At\ t = 0\ sec\ :\ TSR = 22.137\ mm \tag{32}$$

8.2 Sources of Error

- There is a significantly larger error in the Case-2 (T = $45^o C$). This is because the temperature of the water container overshoot to $46^o C$ for the first few minutes, before returning back to $45^o C$ after about 270 seconds. As such, the first two reading of Case-2 have very high error in them.

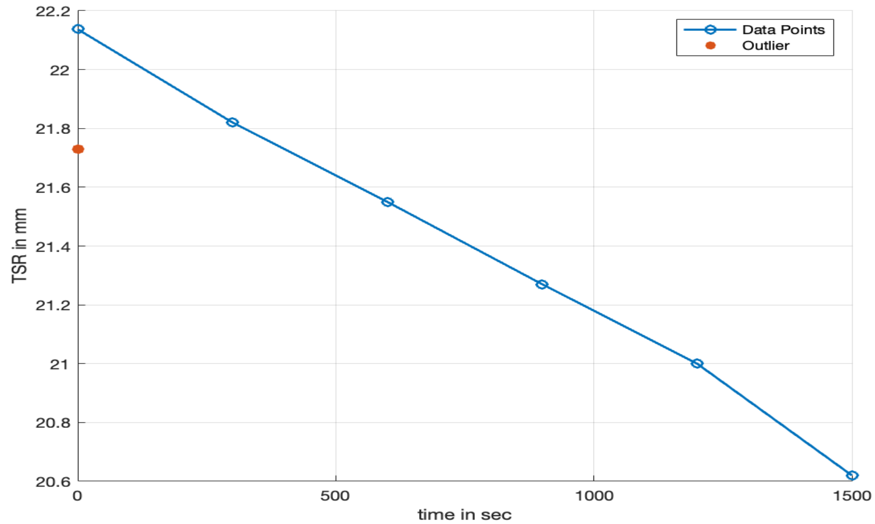


Figure 8: Outlier in Case-2

- After about an hour or so, the Stefan's tube gets surrounded by some air bubbles, making reading measurements difficult.
- The pressure may change slightly if there are variations in flow rate of the Air flowing in the upper channel. This can cause deviations in calculation of D_{AB}.
- In real life, the value of N_B is not actually equal to zero. Some air (a very small amount) does diffuse into the Acetone solution at the bottom. Our equations neglect this.
- Human error (taking reading in parallax, changing temperature by mistake, etc.)

9 Precautions

- Ensure the temperature is consistently maintained at the desired set points (e.g., 40^oC, 45^oC, 50^oC) to avoid fluctuations that may affect diffusion measurements. The water bath temperature must not exceed the boiling point of acetone (56^oC).
- Ensure all connections, including the capillary tube, Stefan tube, and air pump, are tightly sealed to prevent leaks that could affect the acetone diffusion rate.
- Use clean and dry equipment to prevent any contamination of acetone or air that could alter diffusion behavior.

- Ensure that the air pump provides a steady, controlled airflow throughout the experiment. Variations in airflow may introduce inconsistencies in the diffusion process.
- Properly calibrate and focus the microscope to accurately observe the meniscus inside the capillary tube. An unclear view can lead to inaccurate measurements.
- Minimize exposure to open air to avoid unintended evaporation of acetone, which could alter concentration levels and affect diffusion rates.

10 Thought Question / Open-Ended

Q. Consider yourself to be in a James Bond movie. The detective is tied to a chair in a closed room, and there is a lethal perfume leak in his pocket. Considering the diffusivity of the perfume to be 0.135 cm^2/s, estimate how much time you have to save the detective.

A. Using Fick's Law in spherical coordinates in case of uni-molecular Diffusion, we get the following relation :

$$N_A = \frac{-C_T D_{AB}}{1 - y_A} \cdot \frac{\partial y_A}{\partial r} \tag{33}$$

Putting this in species material balance, we get :

$$\frac{\partial y_A}{\partial t} = \frac{D_{AB}}{r^2} \cdot \frac{\partial}{\partial r}\left(r^2 \frac{1}{1 - y_A} \frac{\partial y_A}{\partial r}\right) \tag{34}$$

We define our Boundary conditions as :

$$At\ r = 0\ :\ y_A = y_{A1} \tag{35}$$

$$At\ r = R\ :\ y_A = 0 \tag{36}$$

We define our Initial condition as :

$$At\ t = 0\ :\ y_A = 0,\ \forall\ r \neq 0 \tag{37}$$

We can solve this PDE equation numerically using MATLAB. Then, we can find the time at which $y_A = y_{A,\ lethal}$ at $r = R_{person}$.

Values given : $D_{AB} = 0.135\ cm^2/s$
Values required : y_{A1}, R, $y_{A,\ lethal}$ and R_{person}.

11 Acknowledgements

We as a group contributed our respective parts into completing the above report on "Measurement of diffusivity using Stefan's Tube".

In terms of specifications, Rapolu Paranay Reddy helped with “Apparatus & Materials" and “Experimental Setup Description" part of the report. Atharva Sunilkumar Ghodke contributed in “Procedure" & “Precaution" parts. Anmol Upadhyay delivered the content for “Background & Motivation" along with Lakkireddy Vishnu Vardhan Reddy helping in “Abstract” part of the report and rest of all the parts are collaboratively done & organized by Deepanjhan Das (general editor) & Aayush Bhakna (proof reader).

Regarding AI transcript for the open-ended thought question asked, we didn't use ChatGpt for our thought question. It was more confusing and so we, after discussing the scenario and after reading some related papers, we wrote as per our understanding. Therefore no such transcript is provided in the **Appendix** section.

And at last but not the least, we specially thank the respective TA Jayesh bhaiya for this experiment for his kind help and to let us have a thorough understanding of the whole process and the concept. We thank all the course instructors for their effective control and high co-operation as per the need.

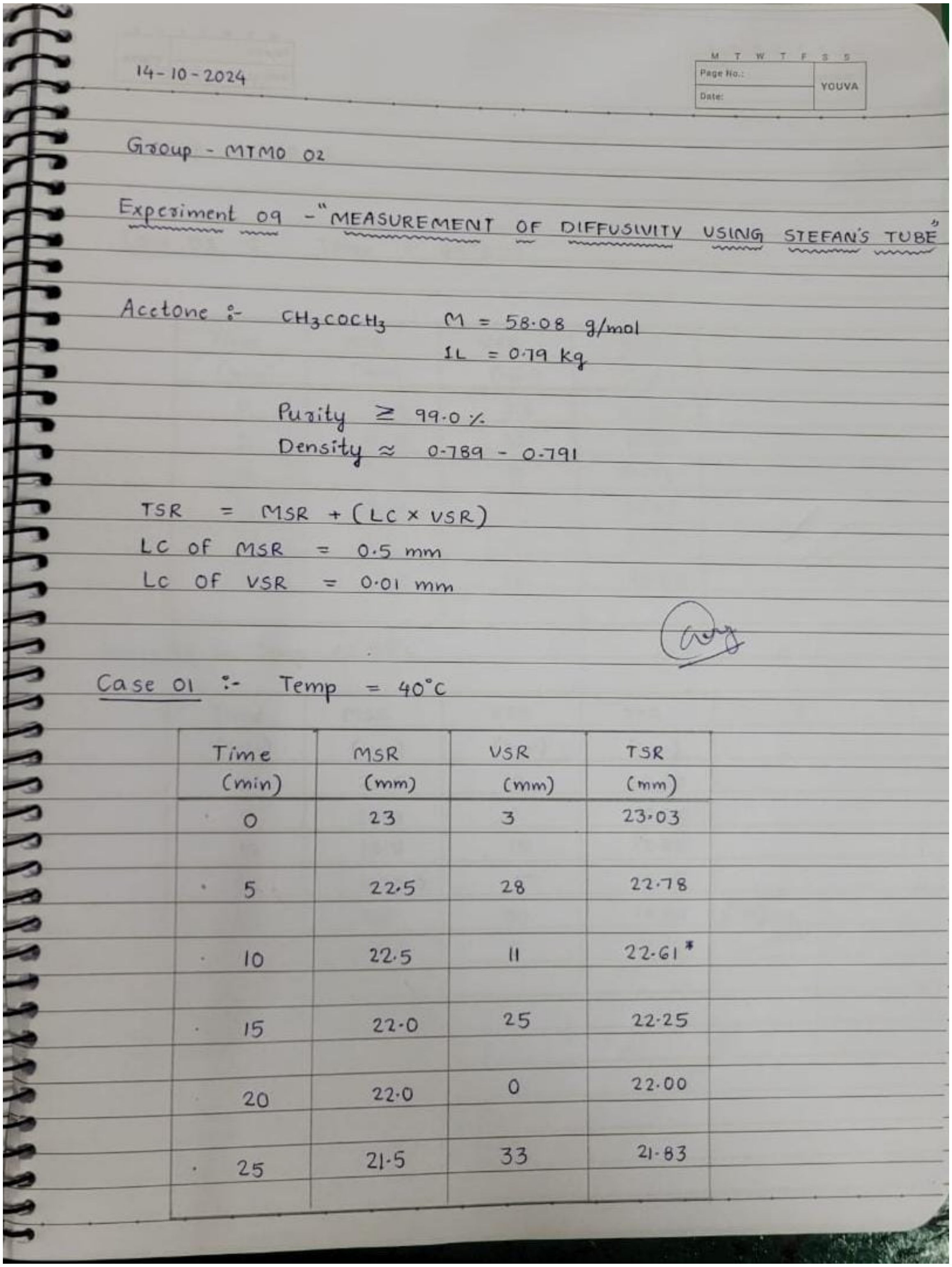
References

- Notes from the course CH2014 : Heat and Mass Transfer by Prof Renganathan.
- Equations used in calculating vapour diffusion coefficient where taken from the paper - "MOLECULAR DIFFUSION IN GASES" | K.S.R. Murthy, Srinivas Tadepalli, Adel Alfozan
  https://ymerdigital.com/uploads/YMER220136.pdf
- Antoine equation parameters of Acetone
  https://webbook.nist.gov/cgi/cbook.cgi?ID=C67641&Mask=4&Type=ANTOINE&Plot=on
- Fick's law of diffusion | Eq 2-149 | Page 2-370 | Perry's Chemical Engineers' Handbook
- Equation to calculate theoretical diffusivity of Air-Hydrocarbon gas mixture | Eq 2-152 | Page 2-370 | Perry's Chemical Engineers' Handbook 7th Edition

Appendix

Lab Data: All the experimental observations with each of the sub-parts of the main experiment that was performed and tabulated during the laboratory session are included in order in the following (in figures 9 & 10).

Reference to all the contents: The official GitHub repository which contains all the related data and coded scripts for calculations is also provided below: https://github.com/deep183Das/CH3510_MTMO_Lab_Group_2/tree/main/Experiment_9. One can easily refer to all the related lab resources from this GitHub repository from where screenshots of few instances are shown in the above figures, in in this report.



14-10-2024

Page No.: Date: YOUVA

Group - MTMO 02

Experiment 09 - "MEASUREMENT OF DIFFUSIVITY USING STEFAN'S TUBE"

Acetone :- CH_3COCH_3 M = 58.08 g/mol

1L = 0.79 kg

Purity ≥ 99.0 %

Density ≈ 0.789 - 0.791

TSR = MSR + (LC × VSR)

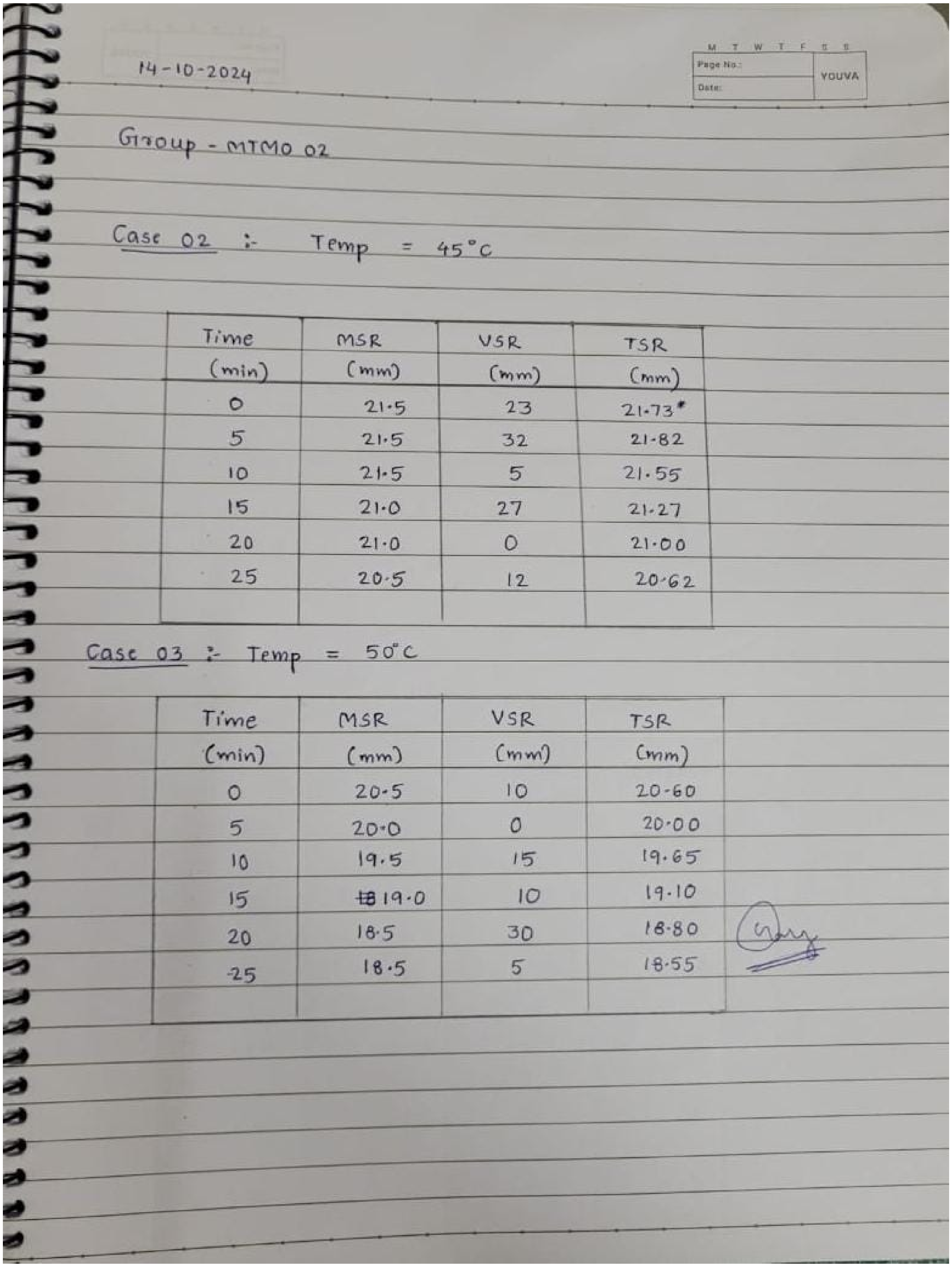
LC of MSR = 0.5 mm

LC of VSR = 0.01 mm

Case 01 :- Temp = 40°C

Time (min)	MSR (mm)	VSR (mm)	TSR (mm)
0	23	3	23.03
5	22.5	28	22.78
10	22.5	11	22.61*
15	22.0	25	22.25
20	22.0	0	22.00
25	21.5	33	21.83

Figure 9: Case of T = 40^{o}C



14-10-2024

Group - MTMO 02

Case 02 :- Temp = 45°C

Time (min)	MSR (mm)	VSR (mm)	TSR (mm)
0	21.5	23	21.73*
5	21.5	32	21.82
10	21.5	5	21.55
15	21.0	27	21.27
20	21.0	0	21.00
25	20.5	12	20.62

Case 03 :- Temp = 50°C

Time (min)	MSR (mm)	VSR (mm)	TSR (mm)
0	20.5	10	20.60
5	20.0	0	20.00
10	19.5	15	19.65
15	~~18~~ 19.0	10	19.10
20	18.5	30	18.80
25	18.5	5	18.55

Figure 10: Cases with T = 45^oC and 50^oC