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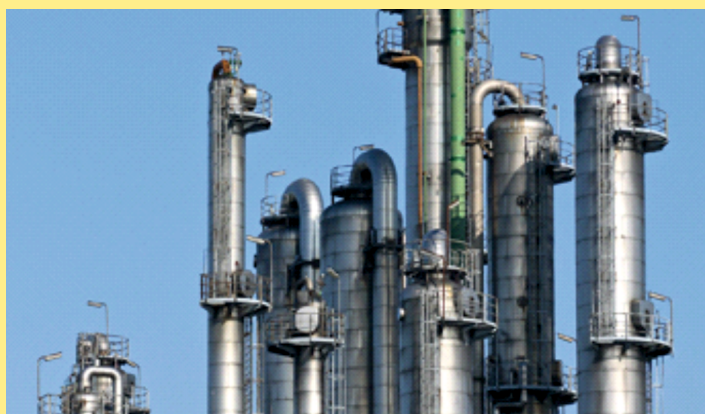
Name \_\_\_\_\_

Roll No. \_\_\_\_\_ Year 20\_\_\_\_ 20\_\_\_\_

Exam Seat No. \_\_\_\_\_

**CHEMICAL GROUP | SEMESTER - VI | DIPLOMA IN ENGINEERING AND TECHNOLOGY**

## **A LABORATORY MANUAL FOR MASS TRANSFER OPERATION (22609)**



**MAHARASHTRA STATE BOARD OF TECHNICAL EDUCATION, MUMBAI**  
(Autonomous) (ISO 9001 : 2015) (ISO / IEC 27001 : 2013)

## **VISION**

To ensure that the Diploma level Technical Education constantly matches the latest requirements of technology and industry and includes the all-round personal development of students including social concerns and to become globally competitive, technology led organization.

## **MISSION**

To provide high quality technical and managerial manpower, information and consultancy services to the industry and community to enable the industry and community to face the changing technological and environmental challenges.

## **QUALITY POLICY**

We, at MSBTE are committed to offer the best in class academic services to the students and institutes to enhance the delight of industry and society. This will be achieved through continual improvement in management practices adopted in the process of curriculum design, development, implementation, evaluation and monitoring system along with adequate faculty development programmes.

## **CORE VALUES**

MSBTE believes in the followings:

- Education industry produces live products.
- Market requirements do not wait for curriculum changes.
- Question paper is the reflector of academic standards of educational organization.
- Well designed curriculum needs effective implementation too.
- Competency based curriculum is the backbone of need based program.
- Technical skills do need support of life skills.
- Best teachers are the national assets.
- Effective teaching learning process is impossible without learning resources.

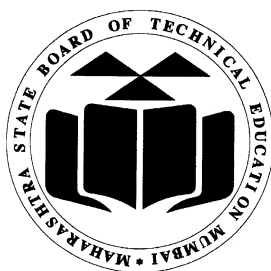
**A Laboratory Manual for**

# **Mass Transfer Operation**

**(22609)**

**Semester – VI**

**(CH)**

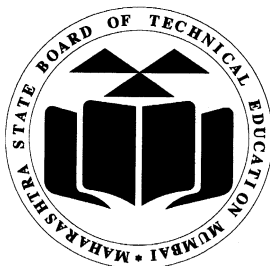


**Maharashtra State**  
**Board of Technical Education, Mumbai**  
**(Autonomous) (ISO:9001:2015) (ISO/IEC 27001:2013)**



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**4<sup>th</sup> Floor, Government Polytechnic Building, 49, Kherwadi,**  
**Bandra (East), Mumbai -400051.**  
**(Printed on November 2019)**





# Maharashtra State Board of Technical Education

## Certificate

This is to certify that Mr. / Ms .....  
Roll No.....of Sixth Semester of Diploma in  
Chemical Engineering of Institute .....  
..... (Code.....)  
has completed the term work satisfactorily in course **Mass  
Transfer Operation(22609)** for the academic year 20.....to  
20..... as prescribed in the curriculum.

Place .....

Enrollment No.....

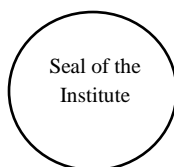
Date:.....

Exam Seat No. ....

**Course Teacher**

**Head of the Department**

**Principal**





## Preface

The primary focus of any engineering laboratory/ field work in the technical education system is to develop the much needed industry relevant competencies and skills. With this in view, MSBTE embarked on this innovative ‘I’ Scheme curricula for engineering diploma programs with outcome-based education as the focus and accordingly, relatively large amount of time is allotted for the practical work. This displays the great importance of laboratory work making each teacher; instructor and student to realize that every minute of the laboratory time need to be effectively utilized to develop these outcomes, rather than doing other mundane activities. Therefore, for the successful implementation of this outcome-based curriculum, every practical has been designed to serve as a **‘vehicle’** to develop this industry identified competency in every student. The practical skills are difficult to develop through ‘chalk and duster’ activity in the classroom situation. Accordingly, the ‘I’ scheme laboratory manual development team designed the practicals to **focus** on the **outcomes**, rather than the traditional age old practice of conducting practicals to ‘verify the theory’ (which may become a byproduct along the way).

This laboratory manual is designed to help all stakeholders, especially the students, teachers and instructors to develop in the student the pre-determined outcomes. It is expected from each student that at least a day in advance, they have to thoroughly read through the concerned practical procedure that they will do the next day and understand the minimum theoretical background associated with the practical. Every practical in this manual begins by identifying the competency, industry relevant skills, course outcomes and practical outcomes which serve as a key focal point for doing the practical. The students will then become aware about the skills they will achieve through procedure shown there and necessary precautions to be taken, which will help them to apply in solving real-world problems in their professional life.

This manual also provides guidelines to teachers and instructors to effectively facilitate student-centered lab activities through each practical exercise by arranging and managing necessary resources in order that the students follow the procedures and precautions systematically ensuring the achievement of outcomes in the students.

Chemical Engineers work as plant operators/ process engineer in various process industries. The processes and operations involve the exchange of mass and need to calculate the amount of material transferred. To operate a plant efficiently and economically, knowledge of mass transfer is essential. Chemical engineers have to deal with the equipment related to Mass Transfer Operations like Distillation, Extraction, Absorption, Drying, and Crystallisation. They have to handle various Mass Transfer Equipment like Distillation column, Dryer, Extractor, Crystalliser and Absorber and Extractor in safe and efficient manner.

Moreover the handling and operation of mass transfer equipment also play an important role in energy saving. Proper selection of equipment improves efficiency of the plant.

Although all care has been taken to check for mistakes in this laboratory manual, yet it is impossible to claim perfection especially as this is the first edition. Any such errors and suggestions for improvement can be brought to our notice and are highly welcome.

## Programme Outcomes (POs) to be achieved through Practical of this Course

Following POs and PSO are expected to be achieved through the practical of the chemical engineering.

**PO1. Basic knowledge:** *Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems*

**PO2. Discipline knowledge:** *Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.*

**PO3. Experiments and practice:** *Plan to perform experiments and practices to use the results to solve Technical problems related to Chemical Engineering.*

**PO4.Engineering tools:** *Apply relevant technologies and Chemical engineering tools with an understanding of the limitations*

**PO6. Environment and sustainability:** *Apply Chemical engineering solutions also for sustainable development practices in societal and environmental context.*

**PO7. Ethics:** *Apply ethical principles for commitment to professional ethics, responsibilities and norms of the practice also in the field of Chemical engineering.*

**PO8. Individual and team work:** *Function effectively as a leader and team member in diverse/ multidisciplinary teams.*

**PO9. Communication:** *Communicate effectively in oral and written form.*

**PSO1. Chemical Engineering Equipment:** *Operate equipment and materials effectively and efficiently used in chemical reactions.*

**PSO2. Material management and quality control:** *Manage chemicals and equipment to produce quality chemical products*

\*\*\*\*\*

## List of Industry Relevant Skills

The following industry relevant skills of the competency '*Use chemical process plant equipment for mass-transfer operations safely*'.

are expected to be developed in you by undertaking the practical of this practical manual.

- Operate distillation columns.
- Operate various dryers.
- Practice energy saving.
- Do maintenance of various mass transfer equipments.

### Practical- Course Outcome matrix

Course Outcomes (COs):						
a. Use various distillation methods in chemical industry. b. Use gas absorption operation and relevant equipment in chemical industries. c. Select relevant solvent for extraction process. d. Determine the time required for drying process. e. Determine the yield of crystals obtained.						
S. No.	Practical Outcome	CO a.	CO b.	CO c.	CO d.	CO e.
1.	Determine the diffusivity of given volatile liquids in air.	√	-	-	-	-
2.	Perform the simple distillation of methanol- water system.	√	-	-	-	-
3.	Measure purity of distillate in fractional distillation.	√	-	-	-	-
4.	Determine Diffusivity of liquid in liquid mixture.	√	-	-	-	-
5.	Measure the purity of distillate by carrying out Steam Distillation	√	-	-	-	-
6.	Carry out distillation to compare the purity of distillate in a packed column at total reflux and 0.5 reflux ratio	√	-	-	-	-
7.	Construct equilibrium diagram from total pressure- vapour pressure data and relative volatility values.	√	-	-	-	-
8.	Determine % Absorption of CO <sub>2</sub> in NaOH solution.	-	√	-	-	-
9.	Calculate the pressure drop of a given packed column for wet and dry packing.	-	√	-	-	-
10.	Determine distribution coefficient for toluene- acetic acid and chloroform- acetic acid mixture.	-	-	√	-	-
11.	Construct Ternary Diagram for system of three liquid, one pair partially soluble i.e. Acetic acid-Benzene-Water system.	-	-	√	-	-
12.	Carry out drying of wet saw dust or sand in a batch dryer to obtain the drying rate curve.	-	-	-	√	-
13.	Use the drum dryer to find the final moisture removal.	-	-	-	√	-
14.	Use a batch crystallizer to determine percent recovery and yield of crystallization.	-	-	-	-	√
15.	By heating or cooling method determine the solubility of a salt and obtain the solubility curve.	-	-	-	-	√

<b>16.</b>	Use the process simulator to analyze the parameters of distillation column .	√	-	-	-	√
<b>17.</b>	Use the process simulator to analyze the parameters of Dryer.	√	-	-	√	-
<b>18.</b>	Use the process simulator to analyze the parameters of Absorber.	√	√	-	-	-

## Guidelines to Teachers

1. **Teacher need to ensure that a dated log book** for the whole semester, apart from the laboratory manual is maintained by every student which s/he has to **submit for assessment to the teacher** in the next practical session.
2. There will be two sheets of blank pages after every practical for the student to report other matters(if any), which is not mentioned in the printed practicals.
3. For difficult practical if required, teacher could provide the demonstration of the practical emphasizing of the skills which the student should achieve.
4. Teachers should give opportunity to students for hands-on after the demonstration.
5. Assess the skill achievement of the students and COs of each unit.
6. One or two questions ought to be added in each practical for different batches. For this teachers can maintain various practical related question banks for each course.
7. If some repetitive information like data sheet, use of software tools etc. has to be provided for effective attainment of practical outcomes, they can be incorporated in Appendix.
8. For effective implementation and attainment of practical outcomes, teacher ought to ensure that in the beginning itself of each practical, students must read through the complete write-up of that practical sheet.
9. During practical, ensure that each student gets chance and takes active part in taking observations/ readings and performing practical.
10. Teacher ought to assess the performance of students continuously according to the MSBTE guidelines

## Instructions for Students

1. For incidental writing on the day of each practical session every student should maintain a **dated log book** for the whole semester, apart from this laboratory manual which s/he has to **submit for assessment to the teacher** in the next practical session.
2. For effective implementation and attainment of practical outcomes, in the beginning itself of each practical, students need to read through the complete write-up including the practical related questions and assessment scheme of that practical sheet.
3. Student ought to refer the data books, IS codes, Safety norms, Electricity act/rules, technical manuals, etc.
4. Student should not hesitate to ask any difficulties they face during the conduct of practical.

## Content Page

### List of Practicals and Progressive Assessment Sheet

S. No	Practical Outcome	Page No.	Date of performance	Date of submission	Assessment marks(25)	Dated sign. of teacher	Remarks (if any)
1.	Determine the diffusivity of given volatile liquids in air.	1					
2.	Perform the simple distillation of methanol- water system.	10					
3.	Measure purity of distillate in fractional distillation.	23					
4.	Determine Diffusivity of liquid in liquid mixture.	32					
5.	Measure the purity of distillate by carrying out Steam Distillation	41					
6.	Carry out distillation to compare the purity of distillate in a packed column at total reflux and 0.5 reflux ratio	47					
7.	Construct equilibrium diagram from total pressure- vapour pressure data and relative volatility values.	57					
8.	Determine % Absorption of CO <sub>2</sub> in NaOH solution.	67					
9.	Calculate the pressure drop of a given packed column for wet and dry packing.	74					
10.	Determine distribution coefficient for toluene- acetic acid and chloroform- acetic acid mixture.	83					
11.	Construct Ternary Diagram for system of three liquid, one pair partially soluble i.e. Acetic acid- Benzene-Water system.	92					
12.	Carry out drying of wet saw dust or sand in a batch dryer to obtain the drying rate curve.	103					
13.	Use the drum dryer to find the final moisture removal.	112					
14.	Use a batch crystallizer to determine percent recovery and yield of crystallization.	118					
15.	By heating or cooling method determine the solubility of a salt and obtain the solubility curve.	125					



S. No	Practical Outcome	Page No.	Date of performance	Date of submission	Assessment marks(25)	Dated sign. of teacher	Remarks (if any)
16.	Use the process simulator to analyze the parameters of distillation column..	134					
17.	Use the process simulator to analyze the parameters of Dryer.	145					
18.	Use the process simulator to analyze the parameters of Absorber.	155					
<b>Total</b>							

**Note:** To be transferred to Proforma of CIAAN-2017.



## **Practical No. 1: Measure the diffusivity of volatile liquid in air**

### **I. Practical Significance :**

Diffusion is the movement of an individual component through a mixture from a region of higher concentration to a region of lower concentration at fixed temperature and pressure with or without the help of an external force. Diffusion may occur in one phase or in both phases in all mass transfer operations.

### **II. Relevant Program Outcomes (POs)**

**PO 1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical engineering problems.

**PO2. Discipline knowledge:** Apply **Chemical Engineering** knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to **Chemical Engineering**.

### **III. Competency and Practical Skills**

**‘Use chemical process plant equipment for mass-transfer operations safely’.**

1. Use traveling microscope to measure level of liquid in tube.

### **IV. Relevant Course Outcomes**

Use various distillation methods in chemical industry.

### **V. Practical Outcome**

Determine the diffusivity of given volatile liquid in air.

### **VI. Relevant Affective Domain Related Outcomes.**

1. Follow safe practices
2. Practice energy conservation.

### **VII. Minimum Theoretical Background**

The process of transfer of mass as a result of the concentration difference of a component in a mixture or two phases in contact is called mass transfer. Diffusion is the movement of an individual component through a mixture from a region of higher concentration to a region of lower concentration at fixed temperature and pressure with or without the help of an external force. Diffusion may occur in one phase or in both phases in all mass transfer operations. A mixture which is non uniform initially will be ultimately brought to uniformity by diffusion since the concentration gradient which acts as a driving force for diffusion tends to move the component in such a direction as to equalize concentrations and destroy the gradient. If the concentration gradient is maintained by constantly supplying the diffusing component to the high concentration end and removing it at the low concentration end, then the flow of diffusing component is continuous. This movement is utilized in mass transfer operations.

When diffusion results from the random movement of the molecules, it is called molecular diffusion. When the movement of the molecules occurs with the help of an external force, then it is called eddy or turbulent diffusion. Molecular diffusion is a slow process, whereas eddy diffusion is a fast process. Molecular diffusion is the mechanism of stationary fluid, i.e., a fluid at rest and fluids in laminar flow. In case of fluids in turbulent flow, the mechanism of mass transfer is by eddy diffusion.

Diffusivity is defined as the ratio of the flux to the corresponding concentration gradient. Diffusivity of vapors is most conveniently determined by Winkelmann's method in which liquid is allowed to evaporate in a graduated vertical glass tube over the top of which a stream of air is passed, at a rate such that the vapor pressure is maintained almost at zero. If apparatus is maintained at a steady temp, there will be no eddy currents in vertical tube and mass transfer will take place from surface by molecular diffusion alone. The rate of evaporation can be followed by the rate of fall of the liquid surface, and since the concentration is known, diffusivity can be calculated.

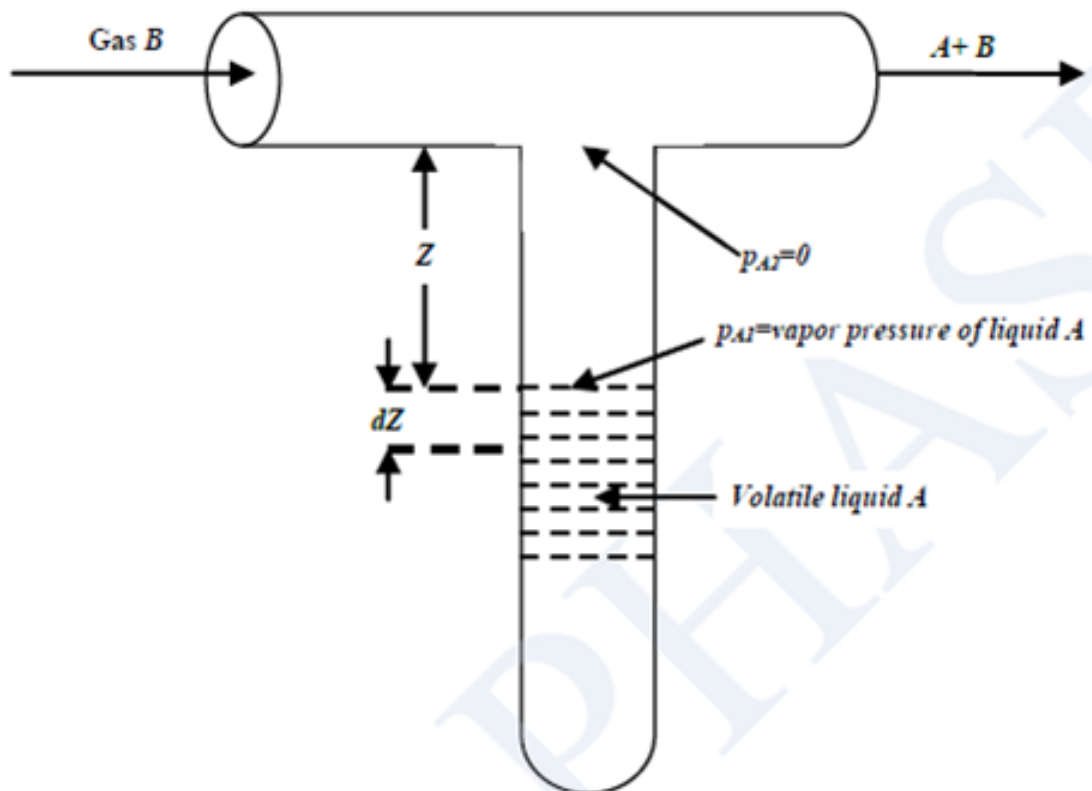


Figure 1 Stefan tube

**VIII. Experimental set up:****Figure 2****IX. Resources required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1.	Diffusivity measuring apparatus	Standard	1
2.	Level reading mechanism	Travelling microscope, least count=0.01	1

**X. Precautions to be followed.**

1. Evaporation tubes should be placed in well ventilated room.
2. Operating temperature should not exceed the boiling point of liquid

**XI. Procedure**

1. Fill up the beaker with clean water and place it on the heater plate. Connect electric supply to the set up and switch on the supply.
2. Adjust the temperature controller setting at desired temperature and ensure that the bath temperature is maintained at the set point.
3. Fill the liquid under test in the diffusion tube and dip the tube in the beaker. Connect blower outlet pipe to the diffusion tube.(for e.g. for  $\text{CCl}_4$ , adjust 45 to 50  $^{\circ}\text{C}$ )
4. Note the level of the liquid in the diffusion tube with regular interval.  
(Use magnifying glass for better measurement of the level).

**XII. Resources used**

Sr. No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					

**XIII. Actual procedure followed**

.....

.....

.....

.....

.....

**XIV. Precautions followed**

.....

.....

.....

.....

.....

**XV. Observations and Calculations:**

1. Substance under test: .....
2. Bath temperature = .....

Sr. No	Elapsed time (t) min	Liquid level (L) mm	Liquid level difference (L-L <sub>0</sub> ) mm	t/(L-L <sub>0</sub> )
1	0	L <sub>0</sub>		
2	5			
3	10			

4	15			
5	20			
6	15			
7	30			
8	35			
9	40			
10	45			

**Sample calculation for set no.**

1.  $\rho_L$  = Density of liquid is.....(for e.g.  $\text{CCl}_4 = 1540 \text{ kg/m}^3$ )

2. Molecular Weight of liquid is,  $M = \dots\dots\dots$ (for e.g.mol.wt of  $\text{CCl}_4 = 154$ )

3. From a plot of  $t/(L-L_0)$  against  $(L-L_0)$  as in above graph the slope of the graph

$$s = \dots\dots\dots \text{ min/mm}^2 = \dots\dots\dots \text{ s/m}^2$$

4. For 'x'(in Kelvin) temperature, Total Concentration is,

$$C_T = 273/('x' \times 22.4) = \dots\dots\dots \text{ kmol/m}^3$$

(Where  $22.4 \text{ m}^3$  is kilogram molecular volume at  $0^\circ\text{C}$  (273K) i.e.NTP)

Note: Refer Perry's Handbook or Internet for Vapour Pressure data,

5. The vapor pressure ( $P_A^0$ ) of Liquid ..... at 'x' K = .....  $\text{kN/m}^2$

(for e.g. The vapor pressure of  $\text{CCl}_4$  at 318K =  $37.5 \text{ kN/m}^2$ )

6. As  $P_A^0 / P = C_A / C_T$ ,

$$C_A = (P_A^0/101.325) \times C_T = \dots\dots\dots \text{ kmol/m}^3$$

7. As  $C_T = C_{A1} + C_{B1}$  &  $C_{A1}=0$  initially,  $C_T = C_{B1}$ , So,

$$C_{B1} = \dots\dots\dots \text{ kmol/m}^3$$

8. As  $P_B^0 / P = C_B / C_T$ ,

$$C_{B2} = (101.325 - P_A^0)/101.325 \times C_T$$

$$C_{B2} = \dots\dots\dots \text{kmol/m}^3$$

9.  $C_{BM}$  is Log Mean Concentration Difference, i.e.  $C_{BM} = (C_{B1} - C_{B2}) / \ln (C_{B1} / C_{B2})$

Put the values & calculate  $C_{BM} = \dots\dots\dots \text{kmol/m}^3$

10. Slope =  $s = \frac{\rho_L}{2MD} \frac{C_{BM}}{C_A C_T}$  (from graph), Rearrange the equation,

$$D = \frac{\rho_L}{2 M s} \frac{C_{BM}}{C_A C_T}$$

11. Put the values in the above equation & calculate the Diffusivity

$$D = \dots\dots\dots \text{m}^2/\text{s. at temperature} \dots\dots \text{K.}$$

## XVI. Results

The diffusivity of volatile liquid (.....) is.....

## XVII. Interpretation of results

.....  
 .....  
 .....  
 .....  
 .....  
 .....

## XVIII. Conclusions

.....  
 .....  
 .....  
 .....  
 .....  
 .....

## XIX. Practical related Questions

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- Explain diffusion.
- Explain Fick's law of diffusion.
- Differentiate between molecular diffusion and eddy diffusion.
- What is the driving force for mass transfer?



**[Space for Answers]**

[illegible]

.....

.....

.....

.....

.....

**XX. References / Suggestions for further Reading**

- <https://slideplayer.com/slide/4449134/>
- <https://www.khanacademy.org/science/chemistry/states-of-matter-and-intermolecular-forces/states-of-matter/v/vapor-pressure>
- <https://nptel.ac.in/courses/103103034/2>
- <https://nptel.ac.in/courses/103103034/6>

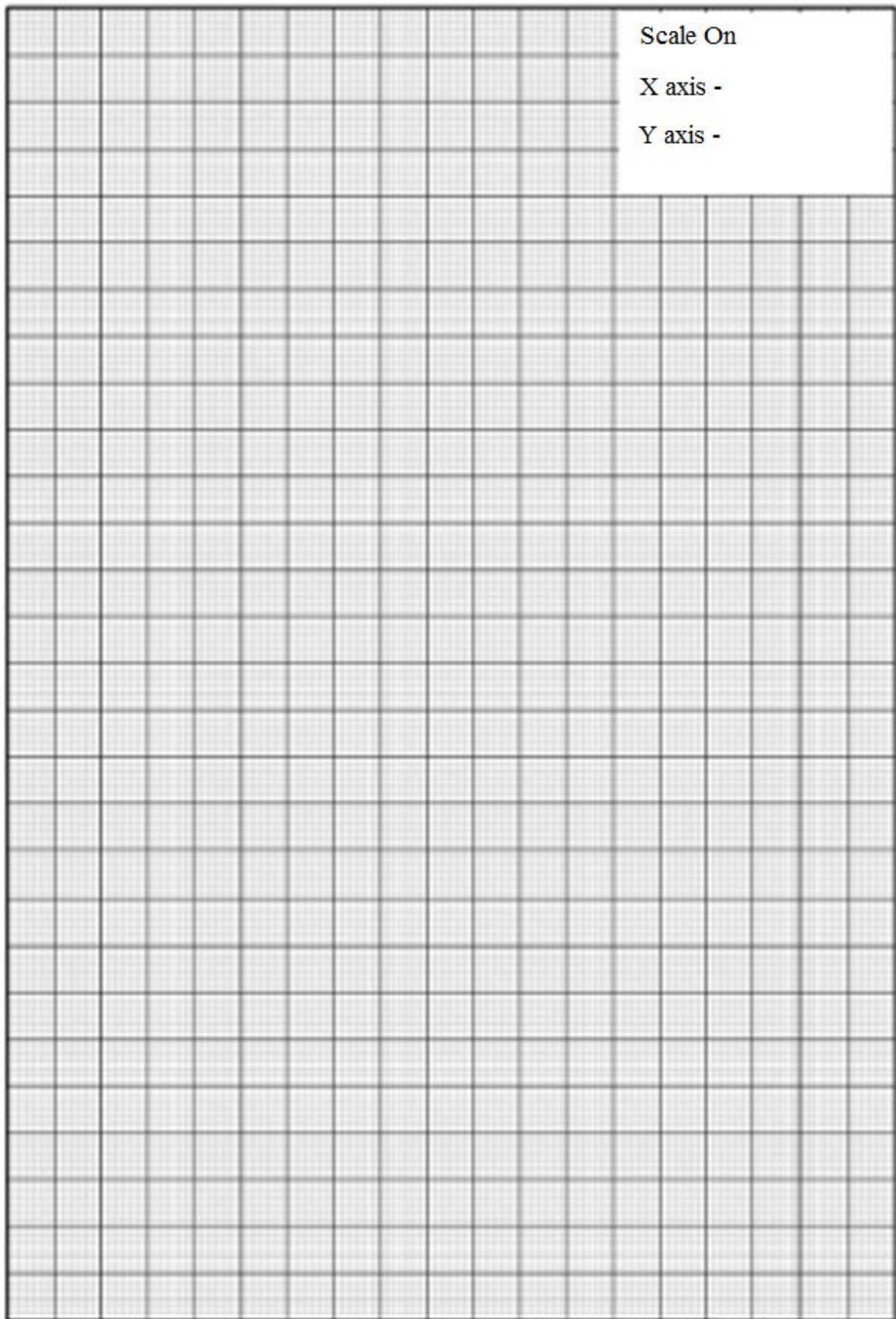
**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of the experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

*Name of student Team Members.*

1. ....
2. ....
3. ....
4. ....

Marks Obtained			Dated signature of Teacher
Process Related (15)	Product Related(10)	Total (25)	



## **Practical No. 2: Verify Rayleigh's equation by performing simple distillation of methanol- water mixture.**

### **I. Practical Significance**

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. Basically the difference in vapour pressure of different constituents at the same temperature is responsible for the separation. Distillation is used in chemical and petroleum industries as a means of separating the liquid mixture into its component parts.

### **II. Relevant Program Outcomes (POs)**

***PO 1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical engineering problems.*

***PO2. Discipline knowledge:** Apply **Chemical Engineering** knowledge to solve industry based Chemical Engineering problems*

***PO 3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical engineering.*

### **III. Competency and Practical Skills**

**'Use chemical process plant equipment for mass-transfer operations safely'.**

1. Use specific gravity bottle to measure density of liquid mixture.
2. Use volume measuring device to measure volume of liquid mixture.

### **IV. Relevant Course Outcomes**

Use various distillation methods in chemical industry.

### **V. Practical Outcome**

Perform the simple distillation of methanol- water system.

### **VI. Relevant Affective domain related Outcome(s)**

1. Follow safe practices.
2. Maintain tools and equipment.
3. Practice good housekeeping

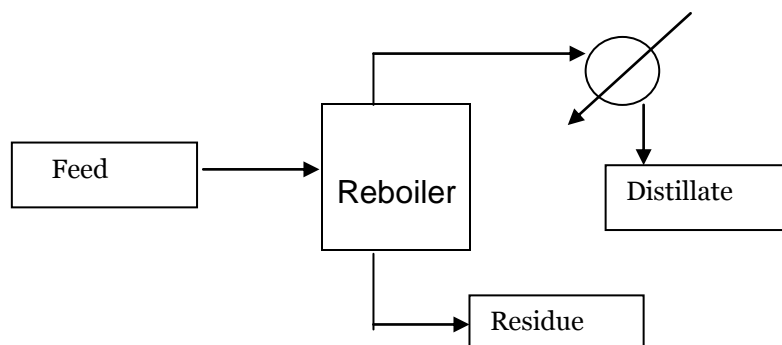
### **VII. Minimum Theoretical Background**

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. With this technique it is possible to separate the liquid mixture into its components in almost pure form and due to this, distillation is the most important of all the mass transfer operations. In distillation, the phases involved are liquid and vapour and mass is transferred from both the phases to one another by vaporization from the liquid phase and by condensation from the vapour phase. The

net effect is an increase in composition of the more volatile component in the vapour and that of the less volatile component in the liquid. The basic requirement for the separation of components by distillation is that the composition of the vapour be different from the composition of the liquid with which it is in equilibrium. The vapour is always richer in more volatile component than the liquid from which it is formed. If the vapour composition is the same as the liquid composition, distillation technique will not affect a separation.

Common methods used in distillation are: i) Differential or simple distillation ii) Flash or equilibrium distillation and iii) Rectification or fractionation. In simple distillation technique, a known quantity of a liquid mixture is charged into a jacketed kettle or still. The jacket is provided for heating the liquid mass in the still with the help of a heating medium such as steam. The charge is boiled slowly, the vapours formed is withdrawn and fed to a condenser where it is liquefied and collected in a receiver as distillate. In the early stage of distillation, the vapour leaving the still is rich in more volatile component and as the distillation proceeds the liquid in the still becomes lean with respect to more volatile component. The material balance equation for simple distillation is called Rayleigh's equation.

$$\ln(F/W) = \int_{x_w}^{x_F} \frac{dx}{(y-x)}$$



**Fig. 1 Block diagram of simple distillation**

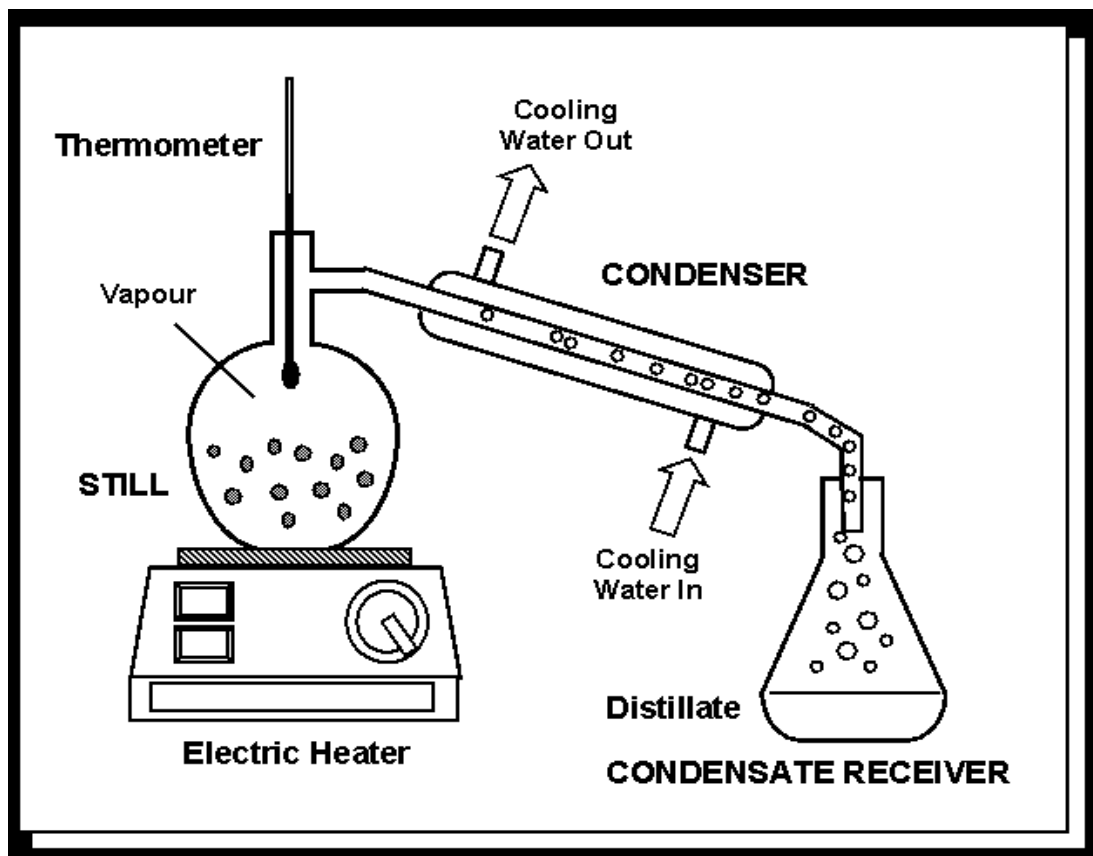


Fig. 2 simple distillation assembly

VIII. Experimental set up :



Fig 2

**IX. Resources required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Reboiler	Capacity: 3 lit	1
2	Receiver	Capacity: 1 lit	1

**X. Precautions to be followed**

1. Do not start heater before starting cooling water supply.
2. Operating temperature should not exceed 100°C.
3. Be careful while handling flammable chemicals.

**XI. Procedure**

1. Plot the graph between density of mixture and volume % of methanol
2. Take a known volume of methanol-water mixture in reboiler (1000 ml for a reboiler capacity of 3000 ml), find out its density using specific gravity bottle and note down the value.
3. Allow cooling water to pass through the condenser. Do not start the heater before starting the cooling water circulation.
4. Start heating and carry out distillation till sufficient distillate (about 100 ml) is collected in the receiver.
5. Stop heating, wait till all the distillate get collected in the receiver
6. Now stop cooling water supply.
7. Collect distillate, measure its volume and density
8. Plot the graph between  $1/(y-x)$  vs  $x$

**XII. Resources used**

Sr. No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					
3					

**XIII. Actual procedure followed**

.....

.....

.....

.....

.....

**XIV. Precautions followed**

.....

.....

.....

.....

.....

**XV. Observations and Calculations:**

Volume of feed = ..... ml

Density of feed = ..... g/ml

Volume of distillate = ..... ml

Density of distillate = .....g/ml

Table 1: (Weight of mixture of methanol- water for various compositions)

Volume of water(ml)	Volume of methanol (ml) 0	Weight of mixture (gms)	Density of mixture(g/ml)	Volume % of methanol
50	0	50		
40	10	47		
30	20	45.33		
20	30	43.188		
10	40	41.27		
0	50	39.08		

Table 2: (x,y data for methanol- water mixture at various temperatures)

Temperature	Composition of mvc in liquid phase(x)	Composition of mvc in gaseous phase(y)	1/(y-x)
100	0	0	
96.4	0.02	0.134	
93.5	0.04	0.23	
91	0.06	0.304	



89.3	0.08	0.365	
87.7	0.1	0.418	
84.4	0.15	0.517	
81.7	0.2	0.579	
78	0.3	0.665	
75.3	0.4	0.729	
73.1	0.5	0.779	
71.2	0.6	0.825	
69.3	0.7	0.87	
67.5	0.8	0.915	
66	0.9	0.958	
65	0.95	0.979	
64.5	1	1	

**Sample calculation for set no.**

Plot the graph between density of mixture and volume % of methanol

Feed

1. Volume of feed (V) = ..... ml
2. Density of feed ( $\rho$ ) = ..... g/ml
3. Vol % of methanol in feed (from density vs vol% of methanol

graph )= .....

4. Volume of methanol = volume % of methanol \* volume of feed

= ..... \* ..... = ..... ml

5. Weight of methanol = vol of methanol \* density of methanol

= ..... \* ..... = ..... gms

$$6. \text{ Moles of methanol} = \frac{\text{Weight of methanol}}{\text{molecular wt. of methanol}} =$$

$$7. \text{ Volume of water} = \text{volume of feed} - \text{volume of methanol}$$

$$= \dots\dots\dots - \dots\dots\dots = \dots\dots\dots \text{ ml}$$

$$8. \text{ Weight of water} = \text{volume of water} * \text{density of water}$$

$$= \dots\dots\dots * \dots\dots\dots = \dots\dots\dots \text{ gms}$$

$$9. \text{ Moles of water} = \frac{\text{Weight of water}}{\text{molecular wt. of water}} =$$

$$=$$

$$10. \text{ Total moles of feed (F)} = \text{moles of methanol} + \text{moles of water}$$

$$= \dots\dots\dots + \dots\dots\dots = \dots\dots\dots$$

$$11. \text{ Mol fraction of methanol in feed (x}_F\text{)} = \text{moles of methanol} / \text{total moles}$$

$$=$$

Distillate:

$$1. \text{ Volume of distillate} =$$

$$2. \text{ Density of distillate} =$$

$$3. \text{ Vol \% of methanol in distillate (from density vs vol\% of methanol graph)}$$

$$= \dots\dots\dots$$

$$4. \text{ Volume of methanol in distillate} = \text{volume \% of methanol} * \text{volume of distillate}$$

$$= \dots\dots\dots * \dots\dots\dots = \dots\dots\dots \text{ ml}$$

$$5. \text{ Weight of methanol} = \text{volume of methanol} * \text{density of methanol}$$

$$= \dots * \dots = \dots \text{gms}$$

6. Moles of methanol =  $\frac{\text{Weight of methanol}}{\text{molecular wt. of methanol}} =$

7. Volume of water = volume of distillate – volume of methanol

$$= \dots - \dots = \dots \text{ ml}$$

8. Weight of water = volume of water \* density of water

$$= \dots * \dots = \dots \text{gms}$$

9. Moles of water =  $\frac{\text{Weight of water}}{\text{molecular wt. of water}} =$

10. Total moles of distillate (D) = moles of methanol + moles of water

$$= \dots + \dots = \dots$$

11. Mol fraction of methanol in distillate ( $x_D$ ) = moles of methanol / total moles

$$=$$

Residue:

1.  $F = D + W$  or  $W = F - D$

$$= \dots - \dots = \dots$$

2.  $F * x_F = D * x_D + W * x_w$

OR

$$x_w = F \cdot x_F - D \cdot x_D / W$$

$$= \dots * \dots - \dots * \dots / \dots = \dots$$

Rayleigh's equation is

$$\ln(F/W) = \int_{x_w}^{x_F} \frac{dx}{y-x}$$

Plot the graph between  $1/(y-x)$  vs  $x$

Find the value of  $\ln(F/W) =$

Find out the area of one square = (x- axis scale \* y- axis scale)

$\ln(F/W) / \text{Area of one square} = \text{no. of squares}$

(Start counting no. of squares from  $x_F$  till you reach  $x_w$  value.)

$$x_w \text{ from graph} = \dots$$

#### XVI. Results

1.  $x_w$  (from graph) = .....
2.  $x_w$  (from calculation) = .....

#### XVII. Interpretation of results

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#### XVIII. Conclusions

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#### XIX. Practical related Questions

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- Write Rayleigh's equation and explain the terms involved in it.
- Why simple distillation is also called differential distillation?
- Write any two industrial application of simple distillation.
- Give any three advantages of simple distillation.

**[Space for Answers]**

[illegible]

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**XX. References / Suggestions for further Reading**

- [https://www.youtube.com/watch?v=0IWY\\_hdgKJM](https://www.youtube.com/watch?v=0IWY_hdgKJM)
- <https://nptel.ac.in/courses/103103034/29>
- <https://www.youtube.com/watch?v=vD8qMPrdMPU>
- <https://www.youtube.com/watch?v=Jgnw9HhX1CQ>
- [https://www.youtube.com/watch?v=v1Lx\\_ZoV5w4](https://www.youtube.com/watch?v=v1Lx_ZoV5w4)

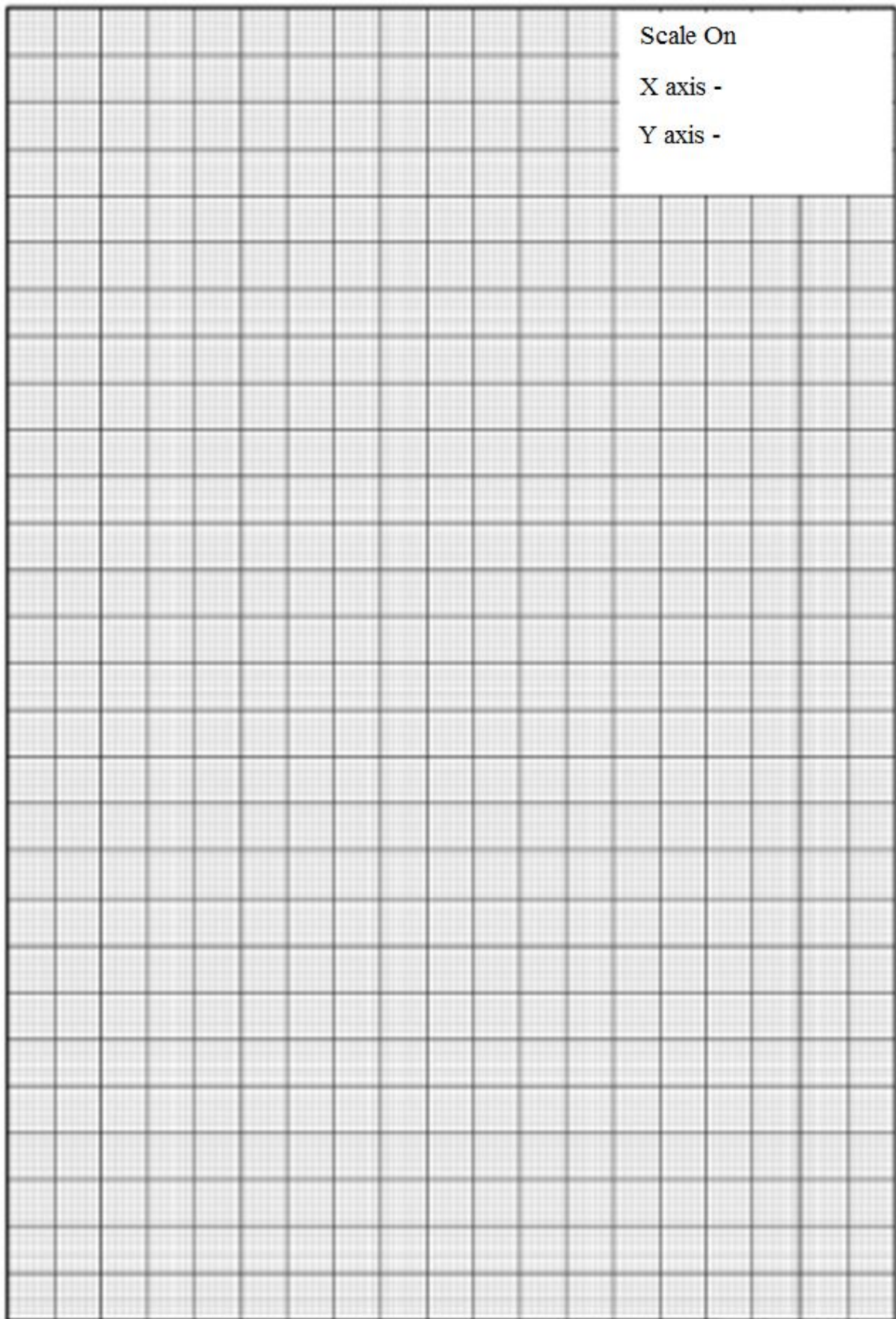
**XXI. Assessment Scheme**

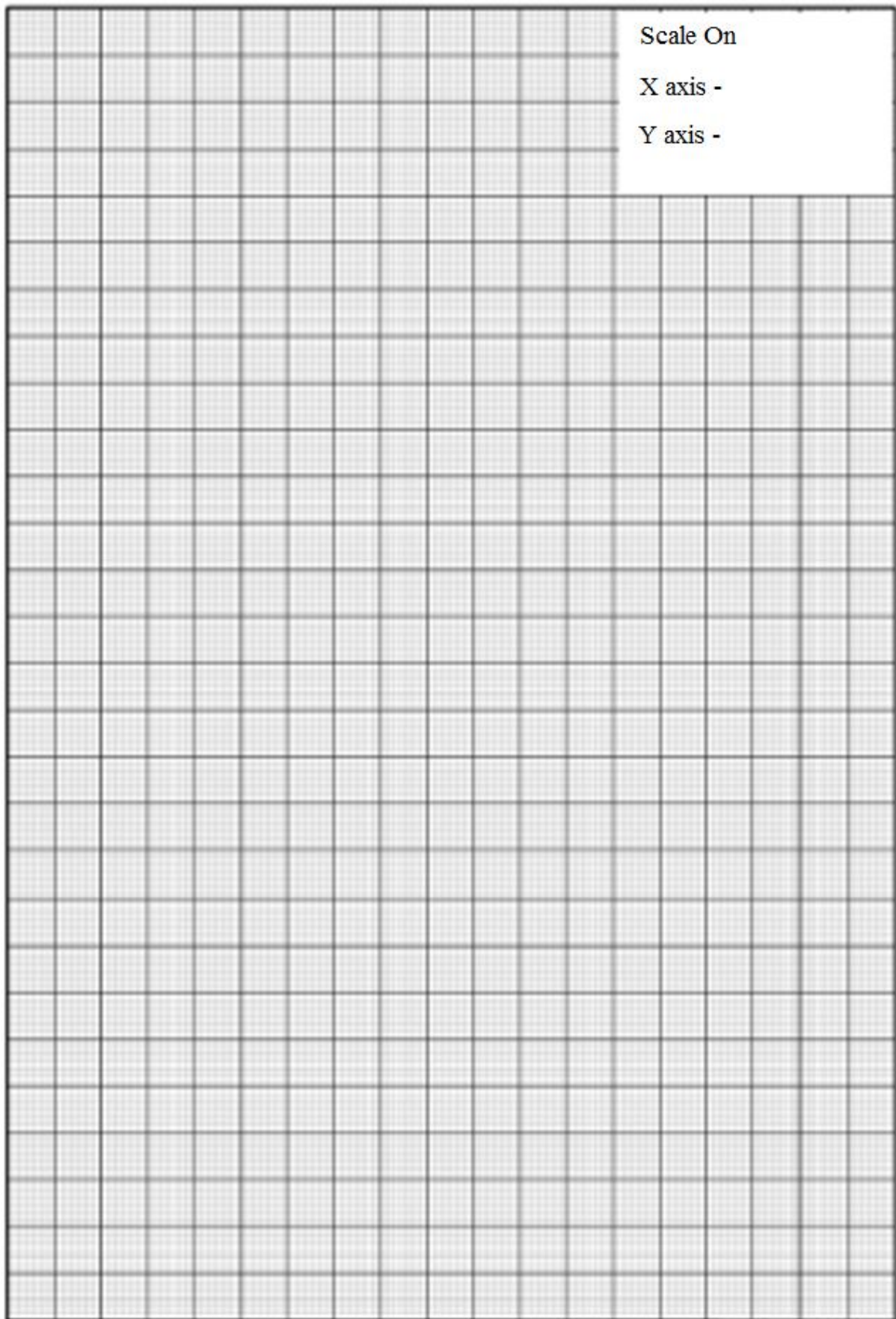
Performance Indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of the experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

- 1.....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	







### **Practical No. 3: Calculate purity of distillate obtained in fractional distillation.**

#### **I. Practical Significance**

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. Basically the difference in vapour pressure of different constituents at the same temperature is responsible for the separation. Distillation is used in chemical and petroleum industries as a means of separating the liquid mixture into its component parts.

#### **II. Relevant Program Outcomes (POs)**

***PO 1. Basic knowledge :** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical engineering problems.*

***PO 3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical engineering.*

***PO 4. Engineering tools:** Apply relevant technologies and Chemical engineering tools with an understanding of the limitations.*

#### **III. Competency and Practical Skills**

***‘Use chemical process plant equipment for mass-transfer operations safely’.***

1. Use specific gravity bottle to measure density of liquid.
2. Use volume measuring device to measure volume of liquid.

#### **IV. Relevant Course Outcomes**

Use various distillation methods in chemical industry.

#### **V. Practical Outcome**

Measure purity of distillate in fractional distillation.

#### **VI. Relevant Affective domain related Outcome(s)**

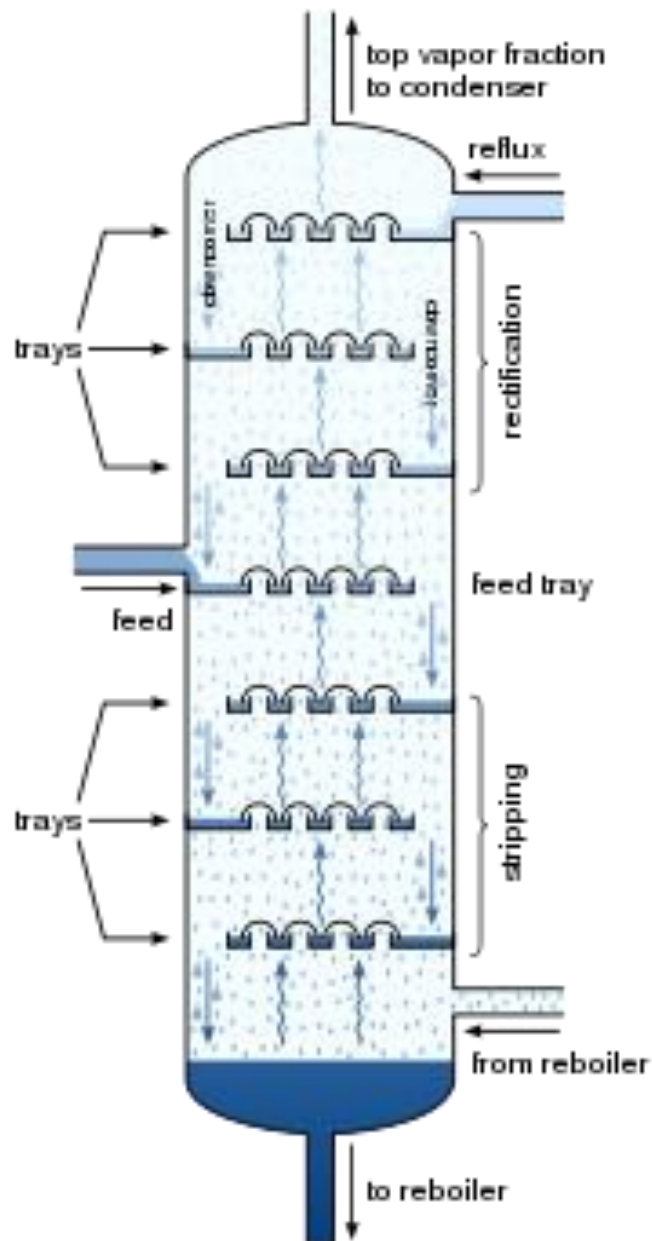
1. Follow safe practices
2. Maintain tools and equipment.

#### **VII. Minimum Theoretical Background**

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. With this technique it is possible to separate the liquid mixture into its components in almost pure form and due to this distillation is the most important of all the mass transfer operations. In distillation, the phases involved are liquid and vapour and mass is transferred from both the phases to one another by vaporization from the liquid phase and by condensation from the vapour phase. The net effect is an increase in composition of the more volatile component in the vapour and that of the less volatile component in the liquid. The basic requirement for the separation of components by distillation is that the composition of the vapour be different from the composition of the liquid with which it is in equilibrium. The vapour is always richer in more volatile component than the liquid from which it is

formed. If the vapour composition is the same as the liquid composition, distillation technique will not affect a separation.

Common methods used in distillation are: i) Differential or simple distillation ii) Flash or equilibrium distillation and iii) Rectification or fractionation. In fractional distillation, a part of the condensed liquid is returned back as reflux and a maximum enrichment of the more volatile component in the vapour is obtained by successive partial vaporization and condensation by a multistage contact of the vapour and the liquid. This is achieved in a single unit called a fractionating column.



**Figure 1 Fractionating column**

**VIII. Experimental set up:****Figure 2****IX. Resources required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Reboiler	Capacity: 5 lit	1
2	Distillate receiver	Capacity: 1 lit	1
3	Packed tower	Height : 43" Type of packing: Random, Raschig ring	1

**X. Precautions to be followed**

1. Do not start heater before starting cooling water supply.
2. Operating temperature should not exceed 100°C.
3. Be careful while handling flammable chemicals.

**XI. Procedure**

1. Take a known volume of methanol-water mixture in reboiler. Find out its density using specific gravity bottle and note down the value.
2. Charge the reboiler with the mixture. Allow cooling water to pass through the condenser. Do not start the heater before starting the cooling water circulation.
3. Start heating and carry out distillation with any value of reflux ratio (eg.0.5).
4. When about 500ml of distillate is collected, stop heating.
5. Allow all the distillate to get collected in the receiver.
6. Now stop cooling water supply.
7. Collect distillate, measure its volume and density

**XII. Resources used**

Sr. No.	Name of Resource	Suggested Broad Specification		Quantity
		Make	Details	
1				
2				
3				

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**

Volume of feed = ..... ml

Density of feed = ..... g/ml

Volume of distillate = ..... ml

Density of distillate = .....g/ml

Table 1: (weight of mixture of methanol- water for various compositions)

Volume of water(ml)	Volume of methanol(ml)	Weight of mixture (gms)	Density of mixture(g/ml)	Volume % of methanol
50	0	50		
40	10	47		
30	20	45.33		
20	30	43.188		
10	40	41.27		
0	50	39.08		

Distillate:

1. Volume of distillate =
2. Density of distillate =
3. Vol % of methanol in distillate (from density vs vol% of methanol graph)  
= .....

4. Volume of methanol = volume % of methanol \* volume of distillate  
= ..... \* ..... = ..... ml

5. Weight methanol = vol of methanol \* density of methanol  
= ..... \* ..... = .....gms

6. Moles of methanol =  $\frac{\text{Weight of methanol}}{\text{molecular wt. of methanol}}$  =

7. Volume of water = volume of distillate– volume of methanol  
= ..... - ..... = ..... ml

8. Weight of water = volume of water \* density of water  
= ..... \* ..... = .....gms

9. Moles of water =  $\frac{\text{Weight of water}}{\text{molecular wt. of water}}$  =

$$10. \text{ Total moles of distillate (D) } = \text{ moles of methanol } + \text{ moles of water } \\ = \dots\dots\dots + \dots\dots\dots = \dots\dots\dots$$

$$11. \text{ Mol fraction of methanol in distillate (x}_D\text{)} = \frac{\text{moles of methanol}}{\text{total moles}} =$$

**XVI. Results**

Purity of distillate is -----

**XVII. Interpretation of results**

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**XVIII. Conclusions**

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**XIX. Practical related Questions**

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- Give the main difference between simple distillation and fractional distillation.
- Define reflux ratio.
- Why purity of distillate is more in fractional distillation?
- What is the driving force for distillation?

**[Space for Answers]**

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**XX. References / Suggestions for further Reading**

- <https://nptel.ac.in/courses/103103034/37>
- <https://nptel.ac.in/courses/103103034/27>
- <https://nptel.ac.in/courses/103103034/31>
- <https://nptel.ac.in/courses/103103034/38>
- <https://www.youtube.com/watch?v=mvaQGt4TAOY>

**XXI. Assessment Scheme**

Performance Indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of the experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

1.....

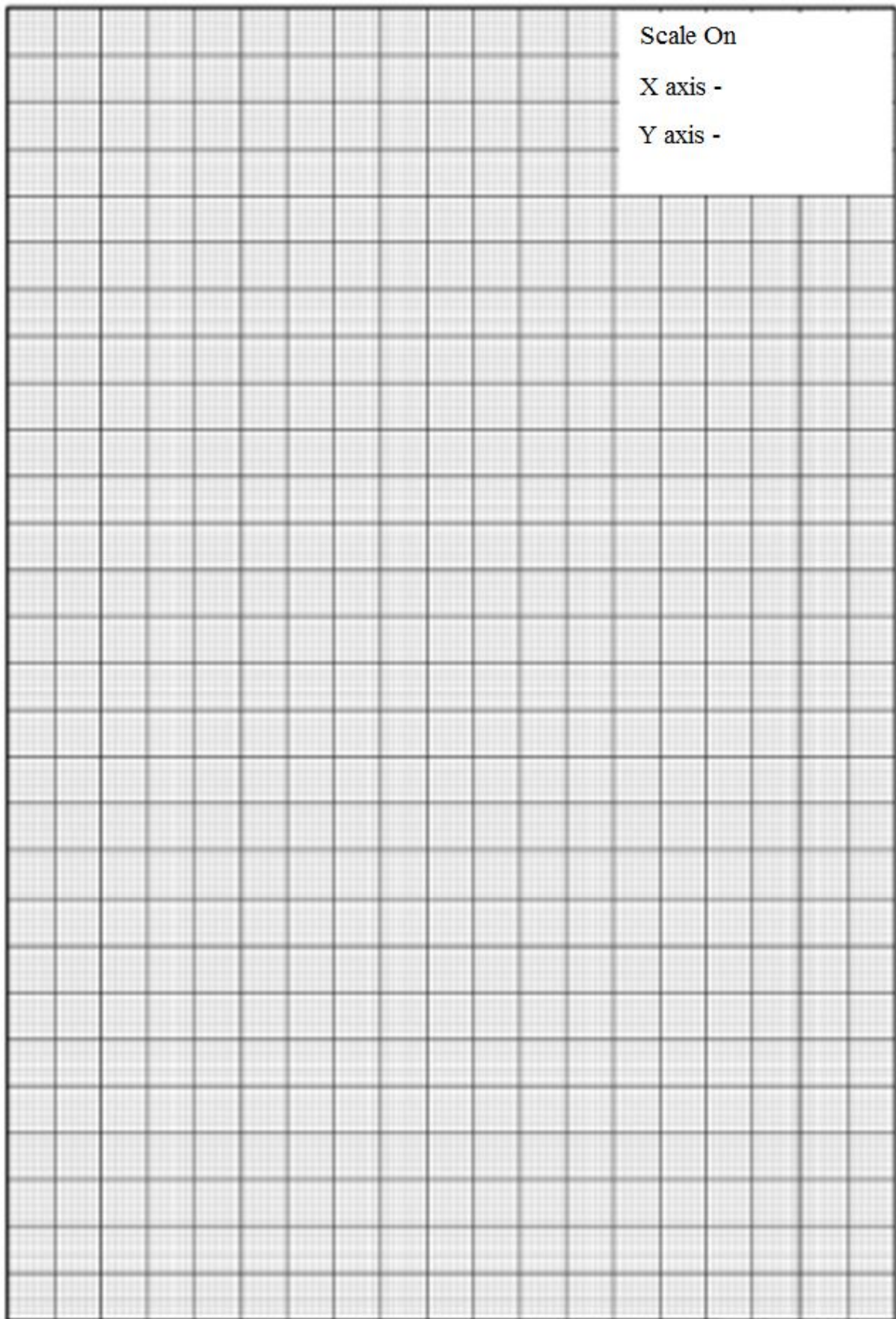
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Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	





**Practical No. 4: Calculate diffusivity of liquid in liquid mixture.****I. Practical Significance**

Diffusion is the movement of an individual component through a mixture from a region of higher concentration to a region of lower concentration at fixed temperature and pressure with or without the help of an external force. Diffusion may occur in one phase or in both phases in all mass transfer operations.

**II. Relevant Program Outcomes (POs)**

*PO 1. Basic knowledge: Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical engineering problems.*

*PO 3. Experiments and practice: Plan to perform experiments and practices to use the results to solve technical problems related to Chemical engineering.*

*PO 4. Engineering tools: Apply relevant technologies and Chemical engineering tools with an understanding of the limitations.*

*PO 7. Ethics: Apply ethical principles for commitment to professional ethics, responsibilities and norms of the practice also in the field of Chemical engineering.*

*PO 9. Communication: Communicate effectively in oral and written form.*

**III. Competency and Practical Skills**

*Use chemical process plant equipment for mass-transfer operations safely.*

1. Use traveling microscope to measure level of liquid in tube.

**IV. Relevant Course Outcomes**

Use various distillation methods in chemical industry.

**V. Practical Outcome**

Determine Diffusivity of liquid in liquid mixture.

**VI. Relevant Affective domain related Outcome(s)**

1. Follow safe practices
2. Practice good housekeeping.
3. Maintain tools and equipment.

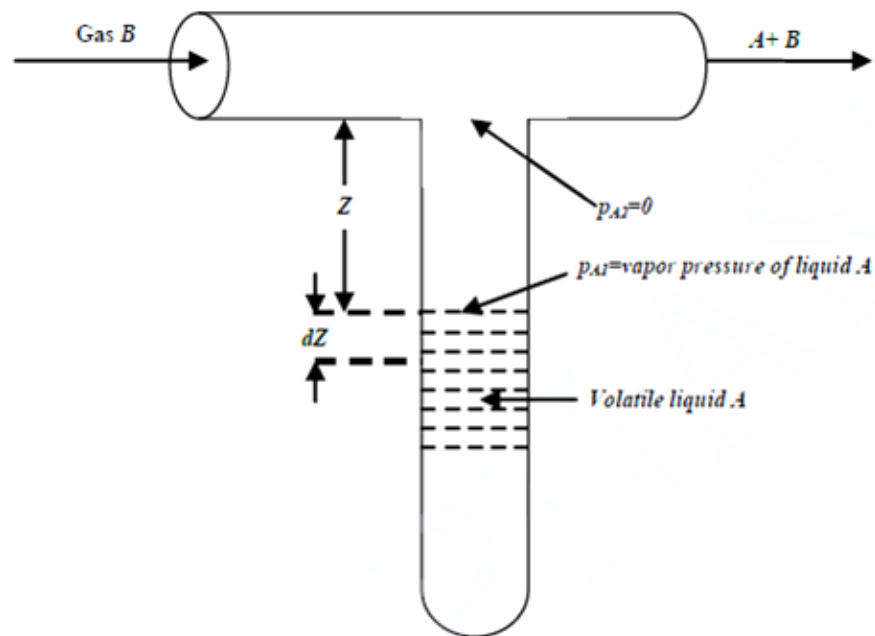
**VII. Minimum Theoretical Background**

The process of transfer of mass as a result of the concentration difference of a component in a mixture or two phases in contact is called mass transfer. Diffusion is the movement of an individual component through a mixture from a region of higher concentration to a region of lower concentration at fixed temperature and pressure with or without the help of an external force. Diffusion may occur in one phase or in both phases in all mass transfer operations.

A mixture which is non uniform initially will be ultimately brought to uniformity by diffusion since the concentration gradient which acts as a driving force for diffusion tends to move the component in such a direction as to equalize concentrations and

destroy the gradient. If the concentration gradient is maintained by constantly supplying the diffusing component to the high concentration end and removing it at the low concentration end, then the flow of diffusing component is continuous. This movement is utilized in mass transfer operations.

When diffusion results from the random movement of the molecules, it is called molecular diffusion. When the movement of the molecules occurs with the help of an external force, then it is called eddy or turbulent diffusion. Molecular diffusion is a slow process, whereas eddy diffusion is a fast process. Molecular diffusion is the mechanism of stationary fluid, i.e, a fluid at rest and fluids in laminar flow. In case of fluids in turbulent flow, the mechanism of mass transfer is by eddy diffusion.



**Fig 1. Stefan tube**

**VIII. Experimental set up:****Figure 2****IX. Resources required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Diffusivity apparatus	standard	1
2	Level reading mechanism	Travelling microscope mechanism, least count: 0.01	1

**X. Precautions to be followed**

1. Evaporation tube should be placed in well ventilated room.
2. Operating temperature should not exceed the boiling point of liquid.

**XI. Procedure**

1. Fill water bath with water.
2. Fill capillary tube with acetone-water mixture.
3. The vertical height of the microscope is adjusted until the capillary tube was visible.

4. When the capillary tube was viewed, the image of meniscus will be upside down so that the bottom of the meniscus of acetone would be at the top of image.
5. When the meniscus of the acetone has been determined, the sliding vernier scale should be aligned with a suitable graduation on the fixed scale.
6. Switch on air pump and water bath.
7. The temperature is set to be at  $40^{\circ}\text{C}$ .
8. The level of acetone inside the capillary tube is recorded for every 10 minutes.

**XII. Resources used**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**

1. Substance under test: .....
2. Bath temperature = .....

Sr. No	Elapsed time (t) min	Liquid level (L) mm	Liquid level difference (L-L <sub>0</sub> ) mm	t/(L-L <sub>0</sub> )
1	0	L <sub>0</sub>		
2	5			
3	10			
4	15			
5	20			
6	25			
7	30			

8	35			
9	40			
10	45			

**Sample calculation**

1.  $\rho_L$  = Density of liquid is.....(for e.g.  $\text{CCl}_4 = 1540 \text{ kg/m}^3$ )

2. Molecular Weight of liquid is,  $M = \dots\dots\dots$ (for e.g. 154 for  $\text{CCl}_4$ )

3. From a plot of  $t/(L-L_0)$  against  $(L-L_0)$  as in above graph the slope of the graph

$$s = \dots\dots\dots \text{ min/mm}^2 = \dots\dots\dots \text{ s/m}^2$$

4. For 'x'(in Kelvin) temperature, Total Concentration is,

$$C_T = 273/('x' \times 22.4) = \dots\dots\dots \text{ kmol/m}^3$$

(Where  $22.4 \text{ m}^3$  is kilogram molecular volume at  $0^\circ\text{C}$  (273K) i.e.NTP)

Refer Perry's Handbook or Internet for Vapour Pressure data,

5. The vapor pressure ( $P_A^0$ ) of Liquid ..... at 'x' K = .....  $\text{kN/m}^2$

(for e.g. The vapor pressure of  $\text{CCl}_4$  at 318K =  $37.5 \text{ kN/m}^2$  )

6. As  $P_A^0 / P = C_A / C_T$ ,

$$C_A = (P_A^0/101.325) \times C_T = \dots\dots\dots \text{ kmol/m}^3$$

7. As  $C_T = C_{A1} + C_{B1}$  &  $C_{A1}=0$  initially,  $C_T = C_{B1}$ , So,

$$C_{B1} = \dots\dots\dots \text{ kmol/m}^3$$

8. As  $P_B^0 / P = C_B / C_T$ ,

$$C_{B2} = (101.325 - P_A^0)/101.325 \times C_T$$

$$C_{B2} = \dots\dots\dots \text{ kmol/m}^3$$

9.  $C_{BM}$  is Log Mean Concentration Difference, i.e.  $C_{BM} = (C_{B1} - C_{B2}) / \ln (C_{B1} / C_{B2})$

Put the values & calculate  $C_{BM} = \dots\dots\dots \text{ kmol/m}^3$

10. Slope=  $s = \frac{\rho_L}{2MD} \frac{C_{BM}}{C_A C_T}$  (from graph), Rearrange the equation,

$$D = \frac{\rho_L}{2 M s} \frac{C_{BM}}{C_A C_T}$$

11. Put the values in the above equation & calculate the Diffusivity

$$D = \dots\dots\dots \text{m}^2/\text{s. at temperature} \dots\dots \text{K.}$$

#### XVI. Results

Diffusivity of acetone in water =.....

#### XVII. Interpretation of results

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 .....  
 .....  
 .....  
 .....

#### XVIII. Conclusions

.....  
 .....  
 .....  
 .....  
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#### XIX. Practical related Questions

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- What is the difference between mass concentration and molar concentration?
- Give the expression to find out rate of diffusion for equimolar counter diffusion
- Give the boiling point of the given volatile liquid.
- What is the effect of temperature on rate of diffusion?

**[Space for Answers]**

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**XX. References / Suggestions for further Reading**

- <https://nptel.ac.in/courses/103103035/module2/lec4.pdf>
- <https://nptel.ac.in/courses/103103034/2>
- <https://nptel.ac.in/courses/103103034/9>
- <https://nptel.ac.in/courses/103103145/7>

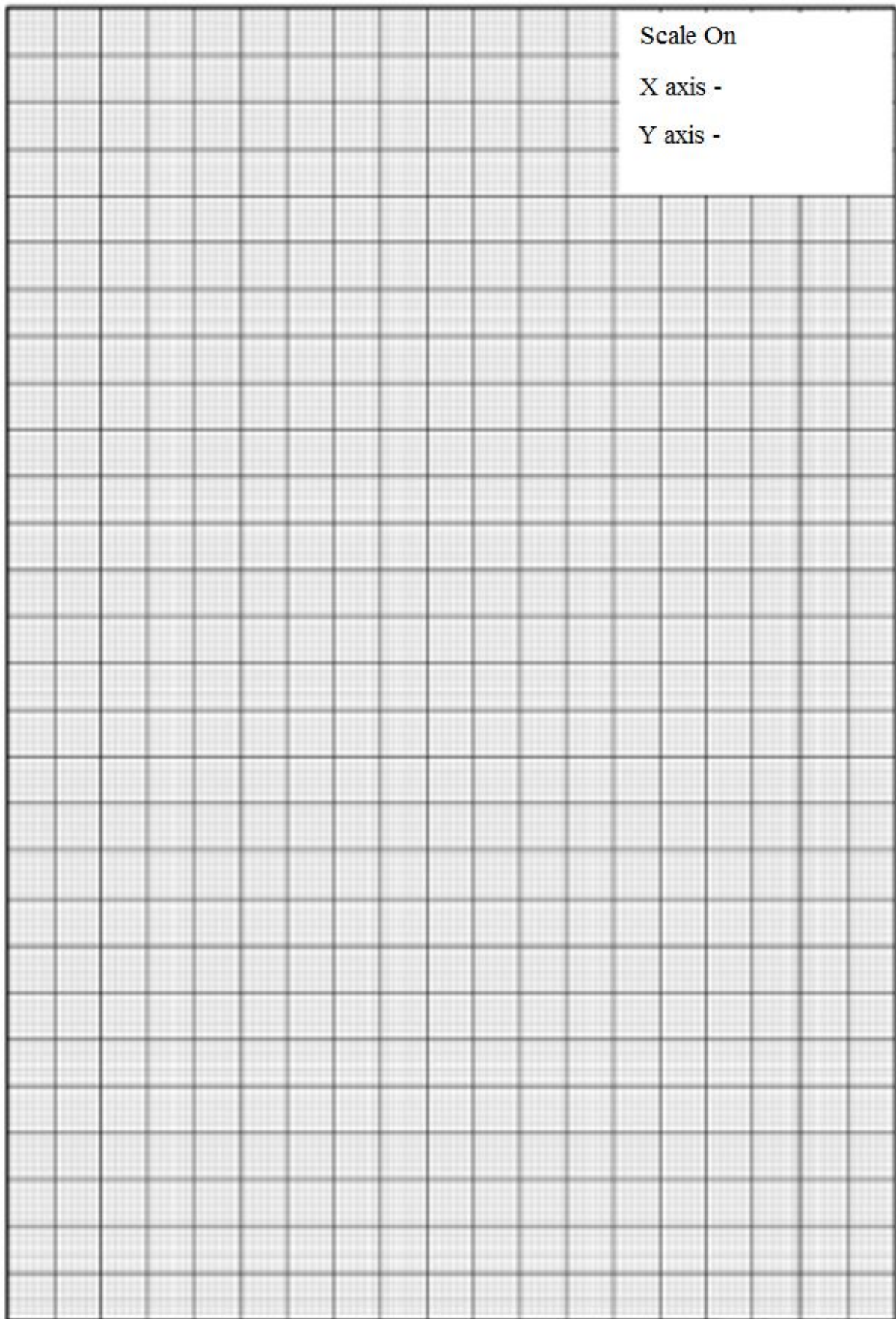
**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of the experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

- 1.....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related (15)	Product Related (10)	Total (25)	



**Practical No. 5: Calculate purity of distillate in steam distillation.****I. Practical Significance**

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. Basically the difference in vapour pressure of different constituents at the same temperature is responsible for the separation. Distillation is used in chemical and petroleum industries as a means of separating the liquid mixture into its component parts.

**II. Relevant Program Outcomes (POs)**

*PO 1. Basic knowledge: Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical engineering problems.*

*PO 3. Experiments and practice: Plan to perform experiments and practices to use the results to solve technical problems related to Chemical engineering.*

*PO 4. Engineering tools: Apply relevant technologies and Chemical engineering tools with an understanding of the limitations.*

**III. Competency and Practical Skills**

***'Use chemical process plant equipment for mass-transfer operations safely'.***

1. Use thermocouple to measure temperature.
2. Use U tube manometer to measure differential pressure

**IV. Relevant Course Outcomes**

Use various distillation methods in chemical industry.

**V. Practical Outcome**

Measure the purity of distillate by carrying out Steam Distillation.

**VI. Relevant Affective domain related Outcome(s)**

1. Follow safe practices
2. Maintain tools and equipment.

**VII. Minimum Theoretical Background**

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. With this technique it is possible to separate the liquid mixture into its components in almost pure form and due to this distillation is the most important of all the mass transfer operations. In distillation, the phases involved are liquid and vapour and mass is transferred from both the phases to one another by vaporization from the liquid phase and by condensation from the vapour phase. The net effect is an increase in composition of the more volatile component in the vapour and that of the less volatile component in the liquid. The basic requirement for the separation of components by distillation is that the composition of the vapour be different from the composition of the liquid with which it is in equilibrium. The vapour is always richer in more volatile component than the liquid from which it is formed. If the vapour composition is the same as the liquid composition, distillation technique will not affect a separation.

Steam distillation is used for (i) Separating a high boiling component from the non-volatile impurities, (ii) Separating a high boiling mixture into different fractions wherein the decomposition of material might occur if direct distillation were employed, (iii) In case where vaporization temperature cannot be reached by steam heat. Steam distillation is especially adopted in cases where the substance involved cannot withstand temperature of distillation and decompose. Substances of this kind can be separated by reducing the partial pressure of the volatile component. This can be done by making use of an inert vapour that decreases the temperature of distillation. The inert vapour used should be practically immiscible with the components to be distilled. Steam is usually used for this purpose.

In steam distillation, steam is directly admitted to the liquid in the still. The mixed vapour containing a desired component is taken as an overhead, condensed and the desired component is separated from the water phase by gravity, while the non volatile material remains behind in the still.

### VIII. Experimental set up:



Figure 1

**IX. Resources required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Reboiler flask	2000 ml capacity	2
2	Receiver	1000 ml capacity	1
3	Separating funnel	500 ml capacity	1

**X. Precautions to be followed.**

1. Pressure in the steam generating flask should not exceed the permissible value.
2. The temperature of reboiler should not exceed the boiling point of feed.

**XI. Procedure**

1. Take water in the steam generating flask.
2. Take 500 ml of the feed in the reboiler.
3. Pass steam to reboiler.
4. Heat the mixture to boil.
5. After 50 ml distillate is collected, stop heating.
6. Transfer the distillate into a separating funnel and allow the mixture to stand for 5-10 minutes.
7. Separate the layers; drain off aqueous layer.
8. Put the organic liquid in a few granules of anhydrous calcium chloride.
9. Measure the volume of organic liquid.
10. Test the purity of organic liquid.

**XII. Resources used (with major specifications)**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					
3					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**

1. Feed =
2. Volume of feed =
3. Volume of distillate collected =
4. Purity of distillate =

**XVI. Results**

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**XVII. Interpretation of results**

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**XVIII. Conclusions**

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**XIX. Practical related Questions**

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- a. Give an industrial example where steam distillation is used.
- b. What are the advantages of steam distillation?
- c. Mention the temperature at which the first drop of distillate collected.

**[Space for Answers]**

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[illegible]

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**XX. References / Suggestions for further Reading**

- <https://nptel.ac.in/courses/103103034/30>
- <https://www.youtube.com/watch?v=3HAW3rhXKic>
- <https://www.youtube.com/watch?v=7g4e3dhtgI>
- <https://www.youtube.com/watch?v=bmK9m2MLI4w>
- <https://www.youtube.com/watch?v=OVQC-6qIq-Y>

**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of the experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

- 1.....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related (15)	Product Related (10)	Total (25)	



## **Practical No. 6: Compare the purities of distillate at total reflux and 0.5 reflux ratio.**

### **I. Practical significance:**

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. Basically the difference in vapour pressure of different constituents at the same temperature is responsible for the separation. Distillation is used in chemical and petroleum industries as a means of separating the liquid mixture into its component parts.

### **II. Relevant Program Outcomes (POs)**

*PO 3.Experiments and practice: Plan to perform experiments and practices to use the results to solve technical problems related to Chemical engineering.*

*PO 4. Engineering tools: Apply relevant technologies and Chemical engineering tools with an understanding of the limitations.*

### **III. Competency and Skills**

*‘Use chemical process plant equipment for mass-transfer operations safely.’*

1. Adjust reflux ratio to required value.
2. Measure density of liquid mixture using specific gravity bottle.

### **IV. Relevant Course Outcome(s)**

Use various distillation methods in chemical industry.

### **V. Practical Outcome**

Carry out distillation to compare the purity of distillate in a packed column at total reflux and 0.5 reflux ratio

### **VI. Relevant Affective Domain Related Outcomes.**

- a. Follow safe practices.
- b. Practice good house keeping.

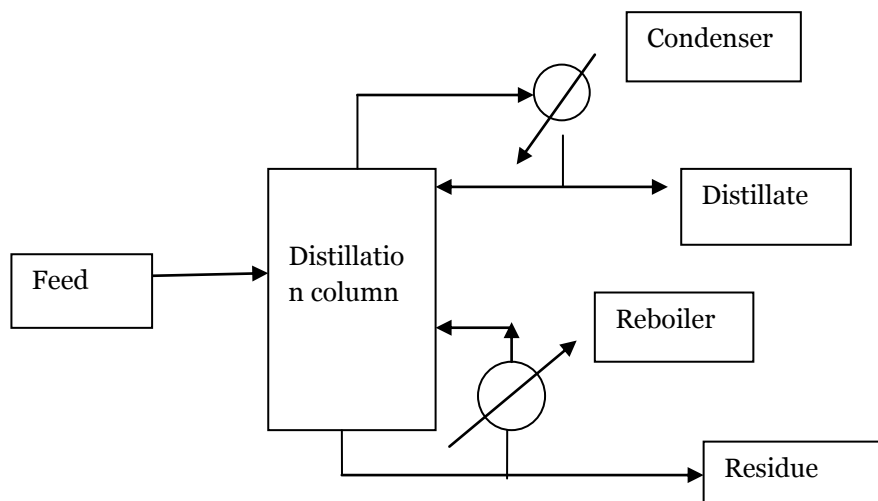
### **VII. Minimum Theoretical Background**

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. With this technique it is possible to separate the liquid mixture into its components in almost pure form and due to this distillation is the most important of all the mass transfer operations. In distillation, the phases involved are liquid and vapour and mass is transferred from both the phases to one another by vaporization from the liquid phase and by condensation from the vapour phase. The net effect is an increase in composition of the more volatile component in the vapour and that of the less volatile component in the liquid. The basic requirement for the separation of components by distillation is that the composition of the vapour be different from the composition of the liquid with which it is in equilibrium. The

vapour is always richer in more volatile component than the liquid from which it is formed. If the vapour composition is the same as the liquid composition, distillation technique will not affect a separation.

Common methods used in distillation are: i) Differential or simple distillation ii) Flash or equilibrium distillation and iii) Rectification or fractionation.

Rectification is commonly encountered in industrial practice as it is possible to get almost pure product by this method. The enrichment of the vapour stream as it passes through the column in contact with reflux is termed as rectification. In this separation method, a part of the condensed liquid is returned back as reflux and a maximum enrichment of the more volatile component in the vapour is obtained by successive partial vaporization and condensation by a multistage contact of the vapour and the liquid. This is achieved in a single unit called fractionating column.



**Fig 1 Block diagram for fractional distillation**

### VIII. Experimental setup



Figure 2

### IX. Resources Required.

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Reboiler	Capacity: 5 lit	1
2	Distillate receiver	Capacity: 1 lit	1
3	Packed column	Height : 43" Packing: random, raschig ring	1

**X. Precautions to be followed.**

1. Do not start heater before starting cooling water supply.
2. Operating temperature should not exceed  $100^{\circ}\text{C}$ .
3. Be careful while handling flammable chemicals.

**XI. Procedure****Part A : Total Reflux**

1. Take a mixture of methanol water, measure its volume, density and note down the values.
2. Plot the graph between density of mixture and volume % of methanol
3. Charge the reboiler with the feed
4. Start cooling water supply.
5. Start heating, keeping the reflux valve closed.
6. Allow distillation to carry out with total reflux for 1 hour.
7. After 1 hour open the valve and collect distillate.
8. When sufficient distillate is collected, stop heating.
9. Allow all distillate to get collected in the receiver, measure the volume and density of distillate.

**Part B : Reflux Ratio = 0.5**

1. Take a mixture of menthol water measure its volume and density
2. Charge the reboiler with the feed.
3. Start cooling water supply.
4. Adjust the valve for 0.5 reflux ratio.
5. Start heating, allow distillation to carry out.
6. Collect distillate, measure its volume and density.

**XII. Resources used.**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					

**XIII. Actual Procedure Followed.**

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**XIV. Precaution followed.**

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**XV. Observation and Calculation****Part A: Total Reflux**

Volume of distillate =

Density of distillate =

**Part B: Reflux ratio=0.5**

Volume of distillate =

Density of distillate =

Table 1: (weight of mixture of methanol- water for various compositions)

Volume of water(ml)	Volume of methanol(ml)	Weight of mixture (gms)	Density of mixture(g/ml)	Volume % of methanol
50	0	50		
40	10	47		
30	20	45.33		
20	30	43.188		
10	40	41.27		
0	50	39.08		

**Distillate: Total reflux**

1. Volume of distillate =
2. Density of distillate =
3. Vol % of methanol in distillate (from density vs vol% of methanol graph)  
= .....
4. Volume of methanol = volume % of methanol \* volume of distillate  
= ..... \* ..... = ..... ml
5. Weight methanol = vol of methanol \* density of methanol  
= ..... \* ..... = .....gms
6. Moles of methanol =  $\frac{\text{Weight of methanol}}{\text{molecular wt.of methanol}} =$
7. Volume of water = volume of distillate– volume of methanol  
= ..... - ..... = ..... ml
8. Weight of water = volume of water \* density of water  
= ..... \* ..... = .....gms
9. Moles of water =  $\frac{\text{Weight of water}}{\text{molecular wt.of water}} =$
10. Total moles of distillate (D) = moles of methanol + moles of water  
= ..... + ..... = .....
11. Mol fraction of methanol in distillate ( $x_D$ )=  $\frac{\text{moles of methanol}}{\text{total moles}} =$

**Distillate: 0.5 reflux**

1. Volume of distillate =
2. Density of distillate =
3. Vol % of methanol in distillate (from density vs vol% of methanol graph)  
= .....
4. Volume of methanol = volume % of methanol \* volume of distillate  
= ..... \* ..... = ..... ml
5. Weight methanol = vol of methanol \* density of methanol  
= ..... \* ..... = .....gms

$$6. \text{ Moles of methanol} = \frac{\text{Weight of methanol}}{\text{molecular wt. of methanol}} =$$

$$7. \text{ Volume of water} = \text{volume of distillate} - \text{volume of methanol}$$

$$= \dots\dots\dots - \dots\dots\dots = \dots\dots\dots \text{ ml}$$

$$8. \text{ Weight of water} = \text{volume of water} * \text{density of water}$$

$$= \dots\dots\dots * \dots\dots\dots = \dots\dots\dots \text{ gms}$$

$$9. \text{ Moles of water} = \frac{\text{Weight of water}}{\text{molecular wt. of water}} =$$

$$10. \text{ Total moles of distillate (D)} = \text{moles of methanol} + \text{moles of water}$$

$$= \dots\dots\dots + \dots\dots\dots = \dots\dots\dots$$

$$11. \text{ Mol fraction of methanol in distillate (x}_D\text{)} = \frac{\text{moles of methanol}}{\text{total moles}} =$$

## XVI. Results

Purity of distillate at total reflux = .....

Purity of distillate at 0.5 reflux = .....

## XVII. Interpretation of Results

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 .....  
 .....  
 .....  
 .....

## XVIII. Conclusions

.....  
 .....  
 .....  
 .....

## XIX. Practical Related Questions

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- What is the use of reflux in fraction distillation?
- How purity of product is increased in fractional distillation?
- Define external and internal reflux ratio?
- Give the capacity of reboiler and condenser used in the experiment.
- Identify the type of packing used in the column.

**[Space for Answers]**

[illegible]



**XX. References/ Suggestions for Further Reading**

- <https://www.youtube.com/watch?v=u6De71IGcsE>
- <https://www.youtube.com/watch?v=AzK7K601cAE>
- <https://www.youtube.com/watch?v=p5S6Ri5aemA>
- [https://www.youtube.com/watch?v=\\_Cc1a8s0zQA](https://www.youtube.com/watch?v=_Cc1a8s0zQA)
- <https://www.youtube.com/watch?v=aZQ6KS2MKhc>
- <https://nptel.ac.in/courses/103103034/33>

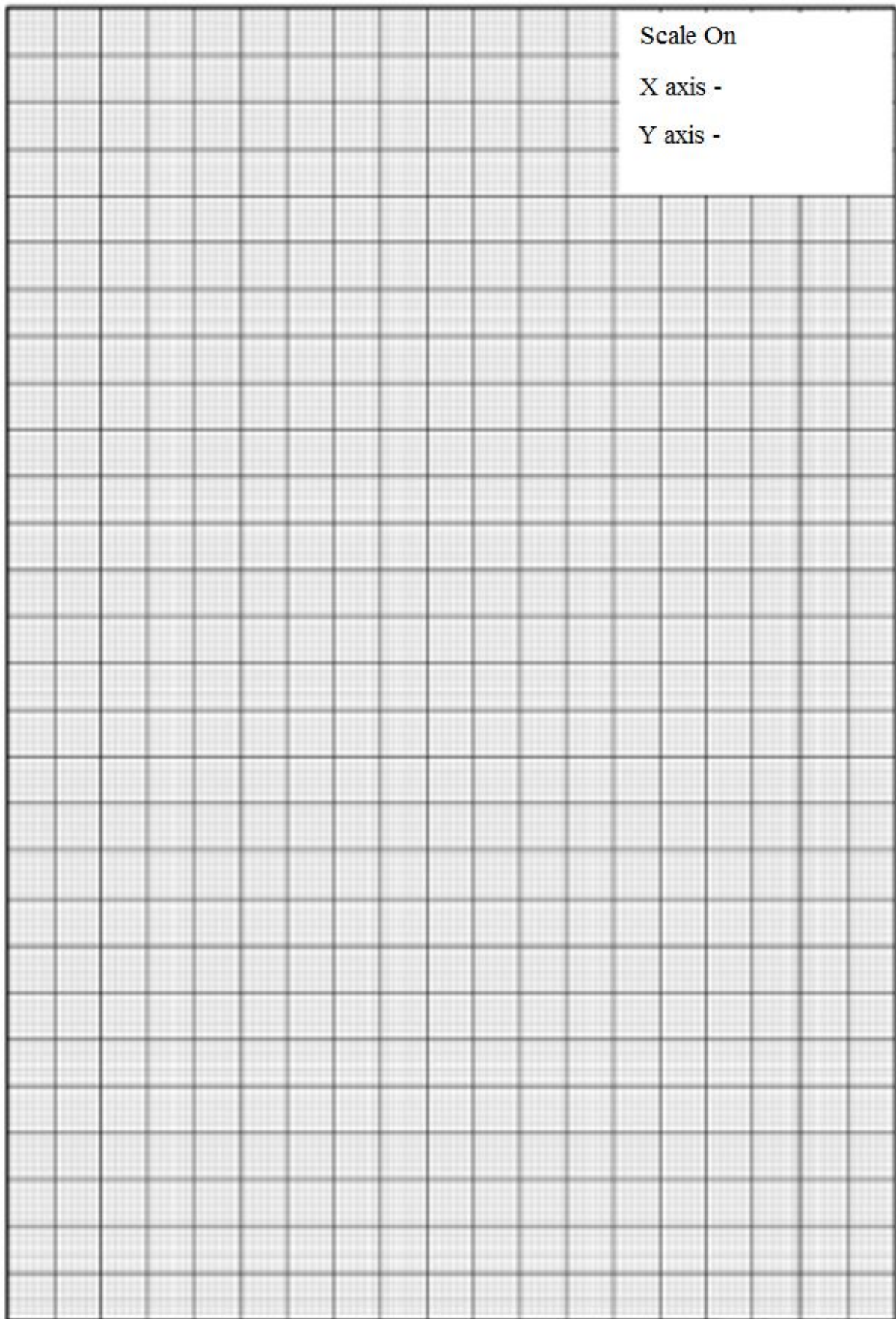
**XXI. Assessment Scheme.**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of the experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

- 1.....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related (15)	Product Related(10)	Total (25)	



## Practical No. 7: Construct equilibrium diagram.

### I. Practical Significance

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. Basically the difference in vapour pressure of different constituents at the same temperature is responsible for the separation. Distillation is used in chemical and petroleum industries as a means of separating the liquid mixture into its component parts. Plot of equilibrium vapour-liquid composition is used for distillation calculations.

### II. Relevant Program Outcomes (POs)

**PO 1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical engineering problems.

**PO 2. Discipline knowledge:** Apply Chemical engineering knowledge to solve industry based Chemical Engineering problems.

**PO 8. Individual and team work:** Function effectively as a leader and team member in diverse/ multidisciplinary teams.

**PO 9. Communication:** Communicate effectively in oral and written form.

### III. Competency and Practical Skills

**'Use chemical process plant equipment for mass-transfer operations safely.'**

1. Calculate x-y data from the given values.
2. Plot equilibrium diagram.

### IV. Relevant Course Outcomes

Use various distillation methods in chemical industry.

### V. Practical Outcome

Construct equilibrium diagram from total pressure- vapour pressure data and relative volatility values.

### VI. Relevant Affective domain related Outcome(s)

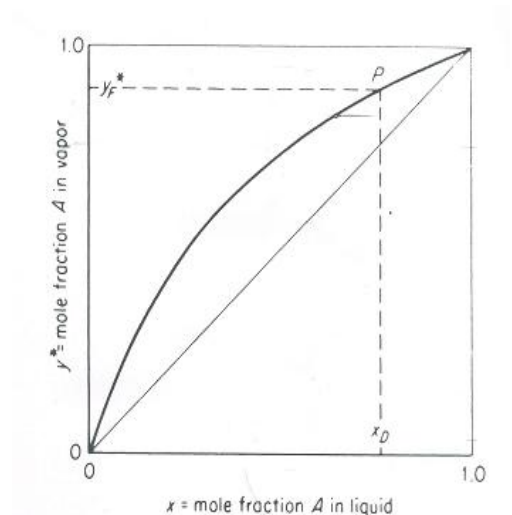
Follow ethical practices

### VII. Minimum Theoretical Background

Distillation is a unit operation in which the constituents of a liquid mixture are separated using thermal energy. With this technique it is possible to separate the liquid mixture into its components in almost pure form and due to this distillation is the most important of all the mass transfer operations. In distillation, the phases involved are liquid and vapour and mass is transferred from both the phases to one another by vaporization from the liquid phase and by condensation from the vapour phase. The net effect is an increase in composition of the more volatile component in the vapour and that of the less volatile component in the liquid. The basic requirement for the separation of components by distillation is that the composition of the vapour be different from the composition of the liquid with which it is in equilibrium. The vapour is always richer in more volatile component than the liquid from which it is

formed. If the vapour composition is the same as the liquid composition, distillation technique will not affect a separation.

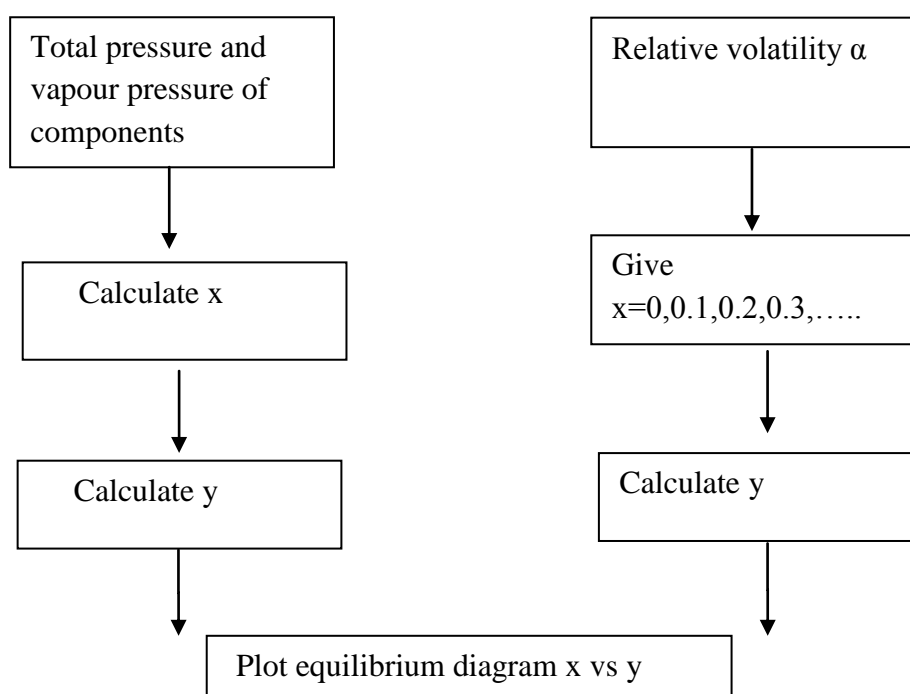
Majority of distillation operation are carried out at a constant total pressure. For distillation calculations, the equilibrium vapour- liquid composition data can also be plotted where vapour phase composition ( $y$ ) is plotted as ordinate and the liquid phase composition ( $x$ ) is plotted as abscissa. Such a diagram is called as equilibrium diagram or equilibrium curve. As the vapour is richer in more volatile component than the liquid, the equilibrium curve lies above  $45^\circ$  diagonal line which is drawn for comparison.



**Fig 1 Equilibrium diagram**

### VIII. Experimental set up:

### Figure 2 Concept structure



**IX. Resources required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Values of partial pressures and total pressure	standard	sufficient
2	Value of relative volatility	standard	1

**X. Precautions to be followed**

1. Calculate the values correctly.
2. Plot the graph with pointed pencil.

**XI. Procedure****Part A: From total pressure- vapour pressure data**

1. Calculate mole fraction of more volatile component in liquid phase ( $x_A$ ) using the formula  $x_A = (P - P_B^0) / (P_A^0 - P_B^0)$  where  $P_A^0$  and  $P_B^0$  are vapour pressures of more volatile and less volatile components respectively.
2. Calculate mole fraction of more volatile component in vapour phase ( $y_A$ ) using the formula  $y_A = x_A * P_A^0 / P$
3. Construct equilibrium curve by plotting  $x_A$  values on x-axis and  $y_A$  values on y-axis.
4. Plot equilibrium diagram with values of x on x-axis and values of y on y-axis with same scale.

**Part B: From relative volatility ( $\alpha$ ) data**

1. Put  $x=0, 0.1, 0.2, 0.3, \dots$  and calculate y using the formula  $y = \frac{\alpha * x}{1 + x(\alpha - 1)}$  ( $\alpha = 2.1$ )
2. Construct equilibrium curve by plotting x values on x-axis and y values on y-axis with same scale.

**XII. Resources used**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:****Part A: From total pressure- vapour pressure data**

Sr No	Temperature (K)	Vapour pressure of benzene (KPa)	Vapour pressure of toluene (KPa)	$x_A$	$y_A$
1	353	101.325	---		
2	355.9	108.12	41.863		
3	358	117.6	45.996		
4	360	127.6	50.396		
5	363.5	138.25	55.195		
6	366.4	149.72	60.262		
7	369.1	161.85	65.861		
8	372	174.65	71.727		
9	374.6	188.251	77.993		
10	377.5	202.65	84.66		
11	380.2	216.65	91.86		
12	383	234.11	99.592.		
13	383.1	---	101.325		

**Part B: From relative volatility data**

$$\alpha = 2.1$$

x	y
0	
0.1	
0.2	
0.3	
0.4	
0.5	
0.6	
0.7	
0.8	
0.9	
1	

**Sample calculation for set no.**

Part A: From total pressure- vapour pressure data

$$x_A = (P - P_B^0) / (P_A^0 - P_B^0)$$

$$= (\dots\dots\dots - \dots\dots\dots) / (\dots\dots\dots - \dots\dots\dots) =$$

$$y_A = x_A * P_A^0 / P$$

$$= \dots\dots\dots * \dots\dots\dots / \dots\dots\dots = \dots\dots\dots$$

Part B: From relative volatility data

$$\alpha = 2.1$$

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

=

Plot equilibrium diagram with values of x on x-axis and values of y on y-axis.

**XVI. Results**

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**XVII. Interpretation of results**

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**XVIII. Conclusions**

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**XIX. Practical related Questions**

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- a. Define volatility and relative volatility.
- b. Define Raoult's law.
- c. Which is the more volatile component in Benzene- Toluene mixture?
- d. How equilibrium diagram is plotted? Give the significance of x and y.
- e. Define more volatile component.

**[Space for Answers]**

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**XX. References / Suggestions for further Reading**

<https://nptel.ac.in/courses/103103145/26>

<https://nptel.ac.in/courses/103103034/27>

<https://www.youtube.com/watch?v=-XcTEknC9Aw>

[https://www.youtube.com/watch?v=dBoF\\_9ParfA](https://www.youtube.com/watch?v=dBoF_9ParfA)

[https://www.youtube.com/watch?v=xt\\_5vY1U0ps](https://www.youtube.com/watch?v=xt_5vY1U0ps)

<http://demonstrations.wolfram.com/PXYAndTXYDiagramsForVaporLiquidEquilibriumVLE/>

**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of the experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

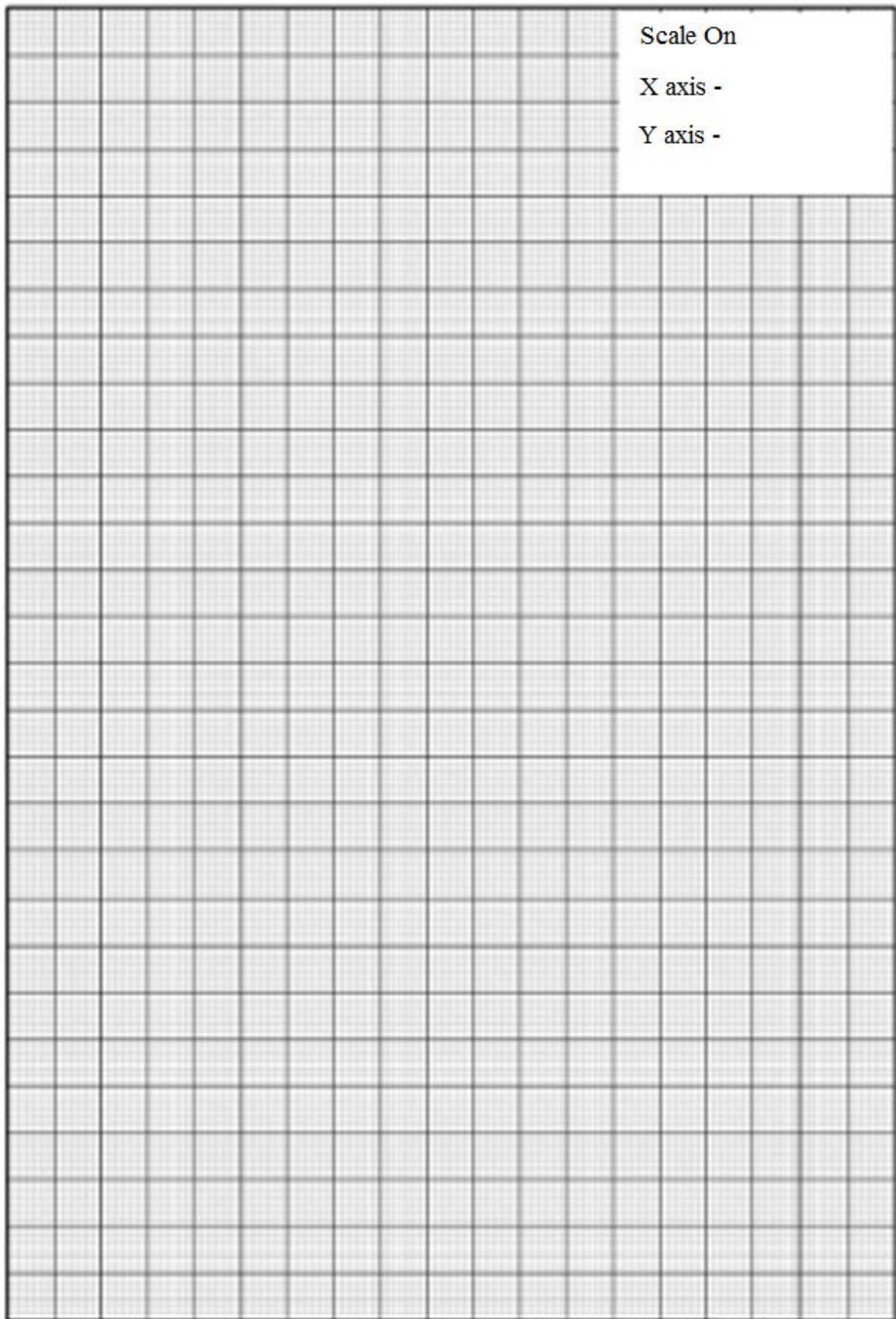
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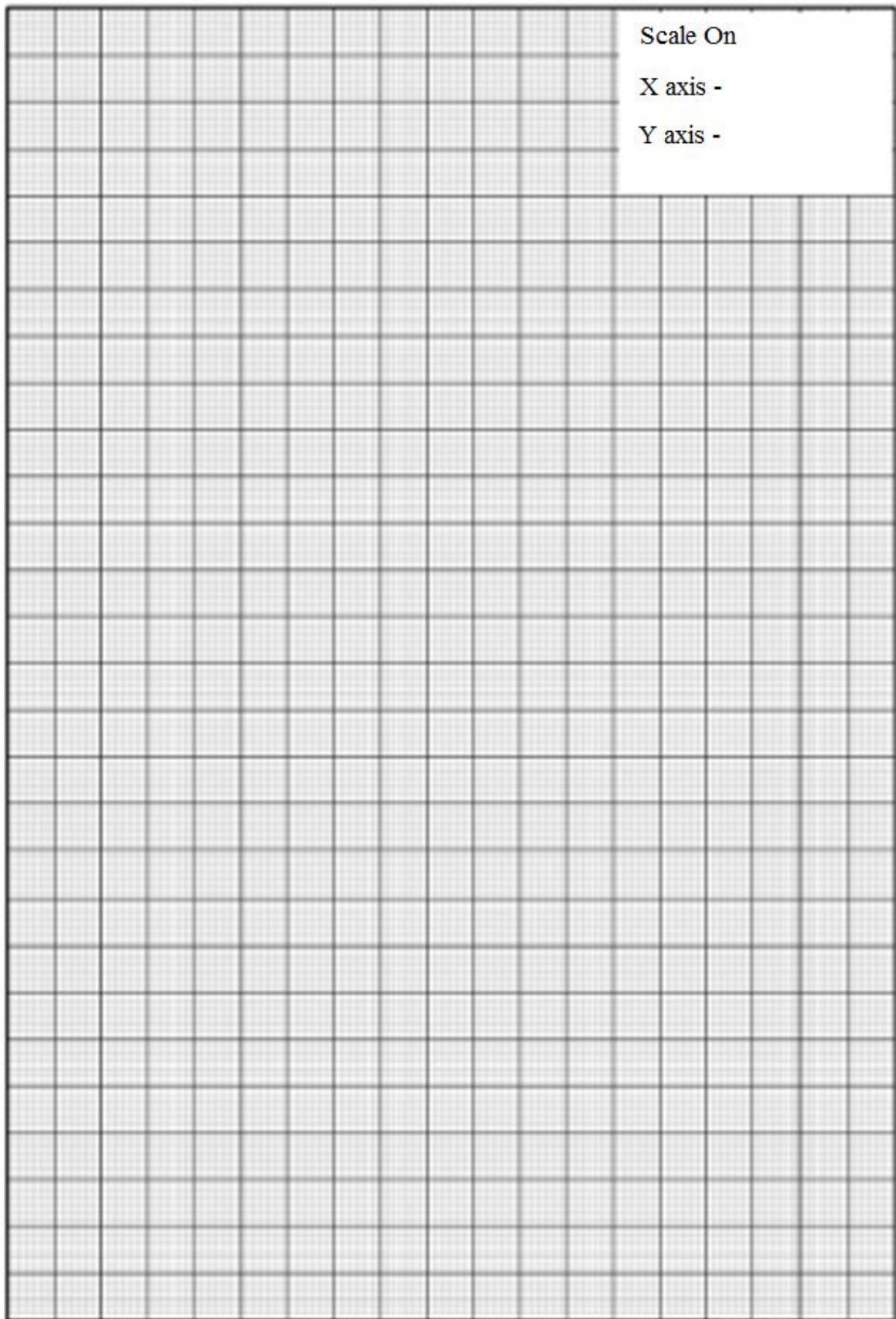
2.....

3.....

4.....

Marks Obtained			Dated signature of Teacher
Process Related (15)	Product Related (10)	Total (25)	





**Practical No. 8: Determine % absorption of CO<sub>2</sub> in NaOH solution.****I. Practical Significance**

Gas absorption refers to an operation in which a gas mixture is contacted with a liquid to preferentially dissolve one or more soluble components of the gas mixture in the liquid. The difference in solubility of gases in a given solvent are utilized to effect such a separation. Gas absorption is the second most important mass transfer operation encountered in the chemical industry and is used for the recovery or removal of the solute gas.

**II. Relevant Program Outcomes (POs)**

**PO 1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical engineering problems

**PO 2. Discipline knowledge:** Apply Chemical engineering knowledge to solve industry based Chemical Engineering problems.

**PO 3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical engineering.

**PSO 2. Material management and quality control:** Manage chemicals and equipment to produce quality chemical products.

**III. Competency and Practical Skills**

**'Use chemical process plant equipment for mass-transfer operations safely.'**

1. Use rotameter for accurate measurement of flow rate.

2. Do acid-base titration.

**IV. Relevant Course Outcomes**

Use gas absorption operation and relevant equipment in chemical industries.

**V. Practical Outcome**

Determine % absorption of CO<sub>2</sub> in NaOH solution.

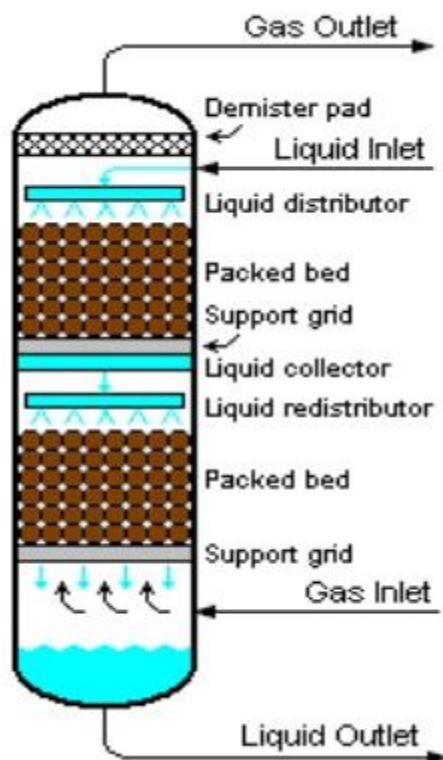
**VI. Relevant Affective domain related Outcome(s)**

1. Follow safe practices
2. Maintain tools and equipment.
3. Practice good housekeeping.

**VII. Minimum Theoretical Background**

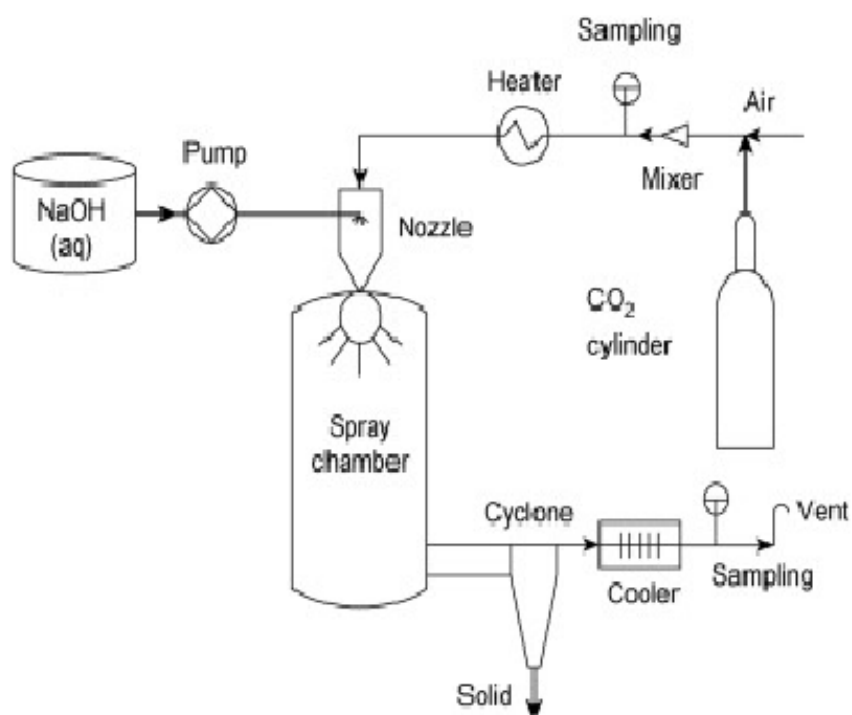
In gas absorption, a gas phase contacts a liquid phase and mass is transferred from the gas phase to the liquid phase. In absorption, the soluble component of a gas mixture is called as the solute gas, the insoluble component is called as inert gas or carrier gas and the liquid used for absorption is called as the solvent or absorber. The reverse of absorption is called desorption or stripping. Absorption may be of two types-physical and chemical.

Physical absorption is the type of absorption in which the process is purely physical. Eg: absorption of ammonia from ammonia-air mixture with water. Chemical absorption is the type of absorption in which chemical reaction also occurs with absorption. Eg: Absorption of NO<sub>2</sub> in water to produce nitric acid.



**Fig 1 Absorption column**

### VIII. Experimental set up:



**Figure 2**

**IX. Resources Required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Gas absorption column	Raschig ring packing	1
2	Compressor	1 HP, working pressure: 5 kgf/cm <sup>2</sup>	1
3	CO <sub>2</sub> cylinder	standard	1

**X. Precautions**

1. Stay away from moving parts of compressor.
2. Rotameter valves are adjusted for correct flow rate.
3. Titration should be carried out to get accurate reading.

**XI. Procedure**

1. Prepare 5 lit of approximately 1N NaOH solution.
2. Transfer this solution to the supply tank.
3. Maintain the flow rate of NaOH at 15 LPH.
4. Adjust CO<sub>2</sub> flow rate such that % of CO<sub>2</sub> in air-CO<sub>2</sub> mixture is about 8-10%.
5. Adjust CO<sub>2</sub> flow rate at 25 LPH and air flow rate at 250 LPH.
6. Allow the system to attain steady state.
7. After attaining steady state, outlet liquid sample is withdrawn .
8. To a known volume of sample add 25% (w/w) Barium chloride solution till precipitation occurs.
9. After completion of precipitation, add some excess amount.
10. The solution is titrated against standard HCl solution with methyl orange as indicator.

**XII. Resources used**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					
3					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**

NaOH flow rate = 15 LPH

CO<sub>2</sub> flow rate = 25 LPH

Air flow rate = 250 LPH

**Sample calculation**

Volume of CO<sub>2</sub> = 25 lit/h

$$= 25 / 1000 * 3600 \text{ m}^3/\text{sec}$$

$$= 6.94 * 10^{-6} \text{ m}^3/\text{sec}$$

**Before absorption**

1. Normality of HCl (N<sub>1</sub>) = 1N

2. Volume of HCl used (V<sub>1</sub>) = .....kg ml

3. Volume of NaOH (V<sub>2</sub>) = 10 ml

4. Normality of NaOH (N<sub>2</sub>) = V<sub>1</sub>.N<sub>1</sub> / V<sub>2</sub> = .....

= .....

5. gms / lit of NaOH (C<sub>1</sub>) = N<sub>2</sub> \* equivalent weight of NaOH

=

6. No. of gmoles of NaOH per lit (n<sub>1</sub>) = C<sub>1</sub>/molecular weight

= .....

**After absorption**

1. Normality of HCl (N<sub>1</sub>) = 1N

2. Volume of HCl used (V<sub>1</sub>) = .....kg ml

3. Volume of NaOH (V<sub>2</sub>) = 10 ml



$$4. \text{ Normality of NaOH } (N_2) = V_1 \cdot N_1 / V_2 = \dots\dots\dots$$

$$= \dots\dots\dots$$

$$5. \text{ gms / lit of NaOH } (C_2) = N_2 * \text{ equivalent weight of NaOH}$$

$$=$$

$$6. \text{ No. of gmols of NaOH per lit } (n_2) = C_2 / \text{molecular weight}$$

$$= \dots\dots\dots$$



$$2 \text{ moles of NaOH} = 1 \text{ mol of CO}_2$$

$$(n_1 - n_2) \text{ moles of NaOH} = X$$

$$7. X = (n_1 - n_2) / 2 = \dots\dots\dots \text{ gmols of CO}_2 \text{ used per lit} =$$

$$8. \text{ gmols / sec CO}_2 \text{ absorbed} = (\text{gmols of CO}_2 / \text{lit}) * \text{ Volumetric flow rate of CO}_2 \text{ in lit/sec}$$

$$= X * 25/3600$$

$$= \dots\dots\dots = \dots\dots\dots \text{ kmols/sec}$$

$$9. PV = nRT \text{ or } V = nRT/P$$

$$\text{Volume of CO}_2 \text{ absorbed} = \dots\dots \text{ m}^3/\text{sec}$$

$$10. \% \text{ absorption of CO}_2 = (\text{CO}_2 \text{ absorbed} / \text{CO}_2 \text{ fed}) * 100 =$$

## XVI. Results

$$\% \text{ absorption of CO}_2 \text{ in NaOH solution} = \dots\dots\dots$$

## XVII. Interpretation of results

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## XVIII. Conclusions

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**XX References / Suggestions for further Reading**

- <https://nptel.ac.in/courses/103103034/21>
- <https://www.youtube.com/watch?v=u0titKpYl8w>
- <https://www.youtube.com/watch?v=2owJnTE3Xow>

**XXI. Assessment Scheme**

Performance Indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of the experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

1. ....
2. ....
3. ....
4. ....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	

## Practical No: 9: Calculate the pressure drop of packed column.

### I. Practical Significance:

Gas absorption refers to an operation in which a gas mixture is contacted with a liquid to preferentially dissolve one or more soluble components of the gas mixture in the liquid. The difference in solubility of gases in a given solvent are utilized to effect such a separation. Gas absorption is the second most important mass transfer operation encountered in the chemical industry and is used for the recovery or removal of the solute gas.

### II. Relevant Program Outcomes (POs)

**PO1. Basic knowledge :** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems

**PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.

**PO 8.Individual and team work:** Function effectively as a leader and team member in diverse/ multidisciplinary teams.

**PO 9.Communication:** Communicate effectively in oral and written form.

### III. Competency and Practical Skills

**'Use chemical process plant equipment for mass-transfer operations safely.'**

1. Use thermocouple to measure temperature.

### IV. Relevant Course Outcomes

Use gas absorption operation and relevant equipment in chemical industries.

### V. Practical Outcome–

Calculate the pressure drop of a given packed column for wet and dry packing.

### VI. Relevant Affective domain related Outcome(s)

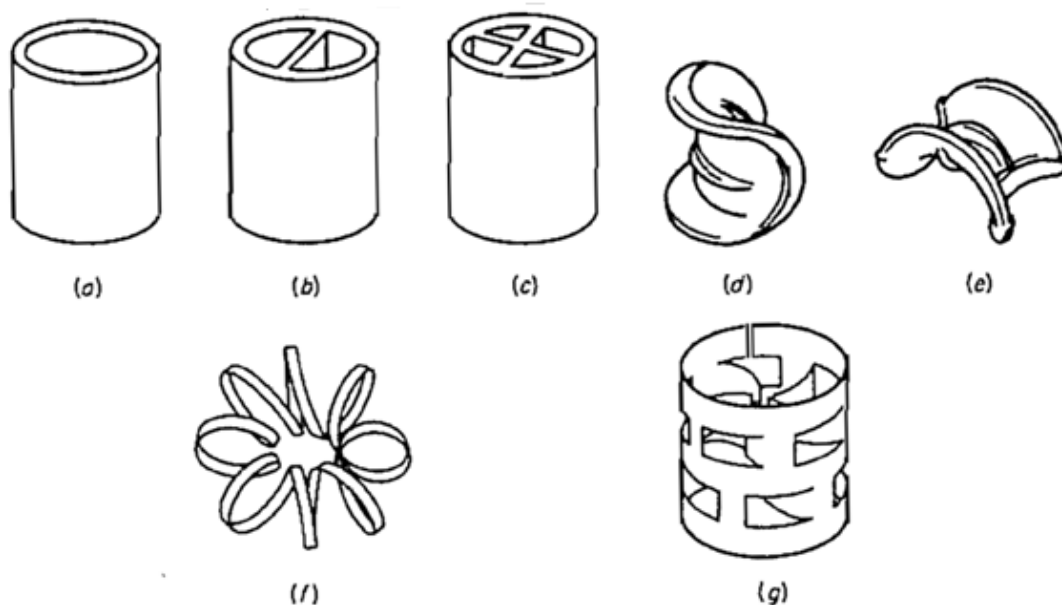
1. Follow safe practices
2. Maintain tools and equipment.

### VII. Minimum Theoretical Background

In gas absorption, a gas phase contacts a liquid phase and mass is transferred from the gas phase to the liquid phase. In absorption, the soluble component of a gas mixture is called as the solute gas, the insoluble component is called as inert gas or carrier gas and the liquid used for absorption is called as the solvent or absorber. The reverse of absorption is called desorption or stripping. Absorption may be of two types-physical and chemical. Physical absorption is the type of absorption in which the process is purely physical. Eg: absorption of ammonia from ammonia-air mixture with water. Chemical absorption is the type of absorption in which chemical reaction also occurs with absorption. Eg: Absorption of  $\text{NO}_2$  in water to produce nitric acid.

Hydrodynamics is the study of pressure drop in a packed column with changing gas velocity for wet and dry packing. Loading point is that point at which the liquid hold up in the column starts to increase and the slope of the pressure drop line changes. From this point onward the pressure drop increases more rapidly with an increase in gas velocity. Flooding point is that velocity of gas at which the tower is flooded. At this velocity entrainment of liquid by the rising gas occurs. If the tower is to operate practically, the operating gas mass velocity must be lower than the flooding velocity. For safe operations, the packed towers are designed using gas velocities of about 50 to 70 percent of flooding velocity at the expected liquid rate.

Packings are used to fill the column and is used to increase the interfacial area for gas – liquid contact. Packing are generally classified as random packing and regular packing. If the packing are simply dumped into the tower during installation and the individual pieces are not arranged in any particular pattern, they are known as random packing. The packing arranged in a particular pattern are called regular (stacked) packing.



Some random tower packings: (a) Raschig rings, (b) Lessing ring, (c) partition ring, (d) Berl saddle (courtesy of Maurice A. Knight), (e) Intalox saddle (Chemical Processing Products Division, Norton Co.), (f) Tellerette (Ceilcote Company, Inc.), and (g) pall ring (Chemical Processing Products Division, Norton Co.).

**Fig 1 Different types of packings**

**VIII. Experimental set up :****Figure 2****IX. Resources required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Packed column	Diameter: 0.0556 m, height ;28''	1
2	Packing	Raschig ring	
3	Manometric fluid	Water	
4	Compressor	1 HP, working pressure: 5 kgf/cm <sup>2</sup>	1

**X. Precaution**

1. Stay away from moving parts of compressor.

2. Be sure rotameter valves are positioned properly.
3. By pass valves of water tank must be positioned properly.

## **XI. Procedure**

### **Part A: For dry packing**

1. Flush the packing with liquid, stop liquid flow.
2. Start the flow of air.
3. Adjust the flow rate of air using rotameter float.
4. Note down the manometer reading.
5. Increase the flow rate of air and note down the manometer reading.
6. Repeat the procedure for various flow rates of air
7. Plot the graph between  $\log V_G$  (gas velocity) on x- axis and  $\log \Delta P$  on y-axis

### **Part B: For wet packing**

1. Start the flow of water and adjust the flow rate of water at a constant value.
2. Start the flow of air.
3. Adjust the flow rate of air using rotameter float.
4. Note down the manometer reading.
5. Increase the flow rate of air and note down the manometer reading.
6. Repeat the procedure for various flow rates of air.
7. Observe the loading point. Still increase the flow rate, observe the flooding point where there is reversal in the flow of liquid.
8. Plot the graph between  $\log V_G$  (gas velocity) on x- axis and  $\log \Delta P$  on y-axis.

## **XII. Resources used (with major specifications)**

Sr. No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks(If any)
		Make	Details		
1					
2					
3					
4					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**

Diameter of the column = .....m

Area of column (A) = ..... m<sup>2</sup>

Height of column = .....m

**Part A: For dry packing**

Sr No	Volumetric flow rate of air		Pressure drop across the column ( $\Delta P$ )			Velocity of gas ( $V_G$ ) m/hr	Log $V_G$	Log $\Delta P$
	In LPM	In m <sup>3</sup> /hr	cm of water	cm of air	m of air			
1								
2								
3								
4								
5								
6								
7								



**Part B: For wet packing:**

Volumetric flow rate of water= .....LPM

Sr No	Volumetric flow rate of air		Pressure drop across the column ( $\Delta P$ )			Velocity of gas ( $V_G$ ) m/hr	Log $V_G$	Log $\Delta P$
	In LPM	In $m^3/hr$	cm of manometric fluid	cm of air	m of air			
1								
2								
3								
4								
5								
6								
7								

**Sample calculation for set no.**1. Volumetric flow rate of air = .....LPM = .....  $m^3/hr$ 2. Velocity of air ( $V_G$ ) =  $\frac{\text{volumetric flow rate of air}}{\text{Area of column}} =$ 3. Log  $V_G = \log$  ..... = .....4. Pressure drop ( $\Delta P$ ) = .....cm of watercm of air =  $\Delta P_{\text{air}} = \Delta P_{\text{water}} (\rho_{\text{water}} - \rho_{\text{air}}) / \rho_{\text{air}} =$  ..... cm of air = ..... m of air5. Log ( $\Delta P$ ) = log ..... = .....6. Plot the graph between log  $V_G$  on x- axis and log  $\Delta P$  on y-axis**XVI. Results**

For an air mass velocity of ..... the pressure drop is.....

**XVII. Interpretation of results**

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**XX. References / Suggestions for further Reading**

- <https://nptel.ac.in/courses/103103034/20>
- <https://www.youtube.com/watch?v=8DiWty6Uh2Y>

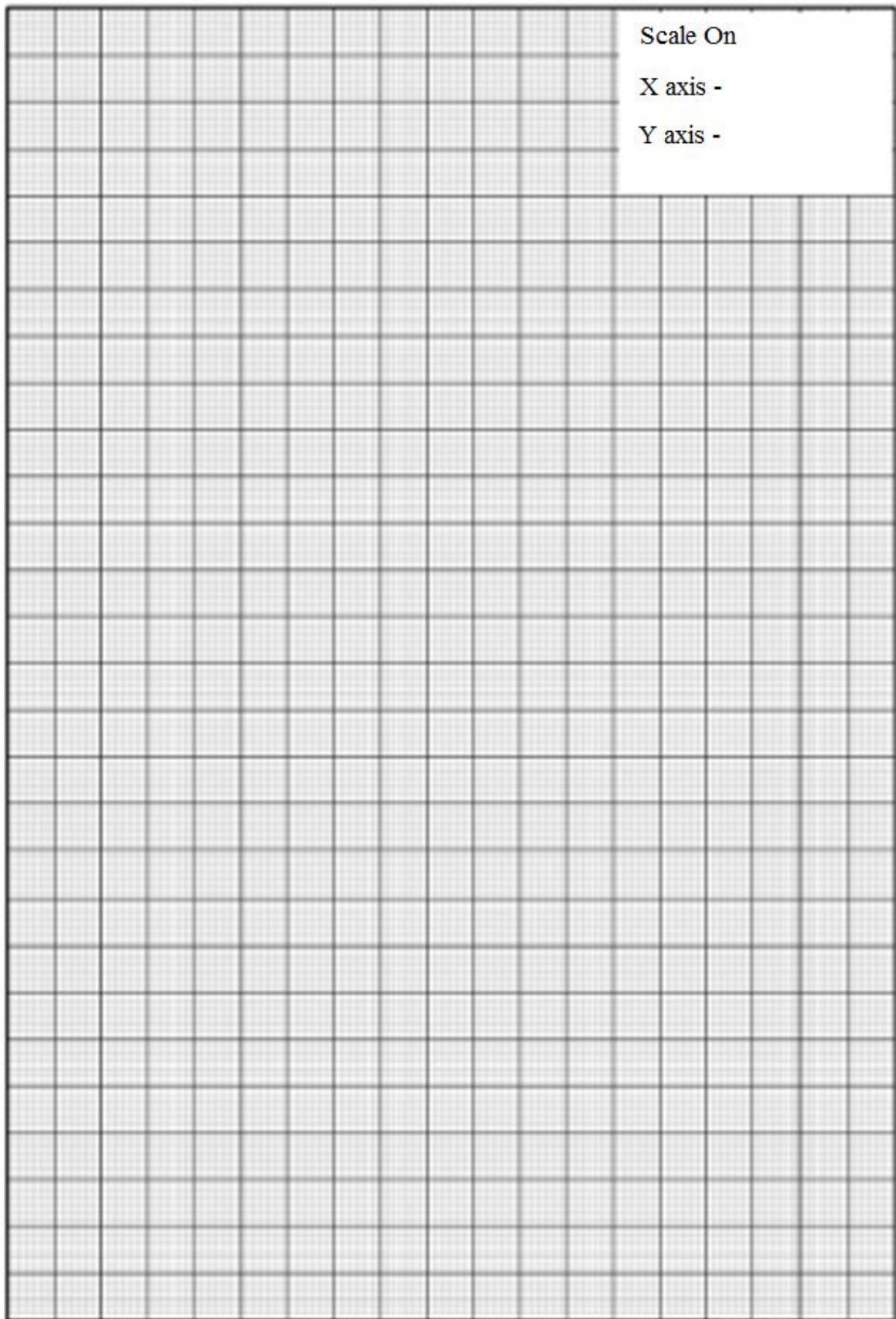
**XXI Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
1	Calculation and Interpretation of result	20%
2	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

1. ....
2. ....
3. ....
4. ....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	



## Practical No.10: Calculate distribution coefficient

### I. Practical Significance:

The technique of removing one component from a liquid mixture by means of a liquid solvent is termed as liquid extraction. When a mixture of liquids is not easily separable by distillation, liquid extraction is used. Close boiling mixture or substances that cannot withstand the temperature of distillation, even under vacuum may often be separated by extraction.

### II. Relevant Program Outcomes (POs)

**PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical **Engineering** problems

**PO2. Discipline knowledge:** Apply **Chemical Engineering** knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical **Engineering**.

**PO 9.Communication:** Communicate effectively in oral and written form.

**PSO 2.Material management and quality control:** Manage chemicals and equipment to produce quality chemical products.

### III. Competency and Practical Skills

**'Use chemical process plant equipment for mass-transfer operations safely.'**

1. Use volume measuring device to measure volume.
2. Use burette to perform acid-base titration.

### IV. Relevant Course Outcomes

Select relevant solvent for extraction process.

### V. Practical Outcome

Determine distribution coefficient for toluene-acetic acid and chloroform – acetic acid mixture.

### VI. Relevant Affective domain related Outcome(s)

1. Follow safe practices.
2. Maintain tools and equipment.

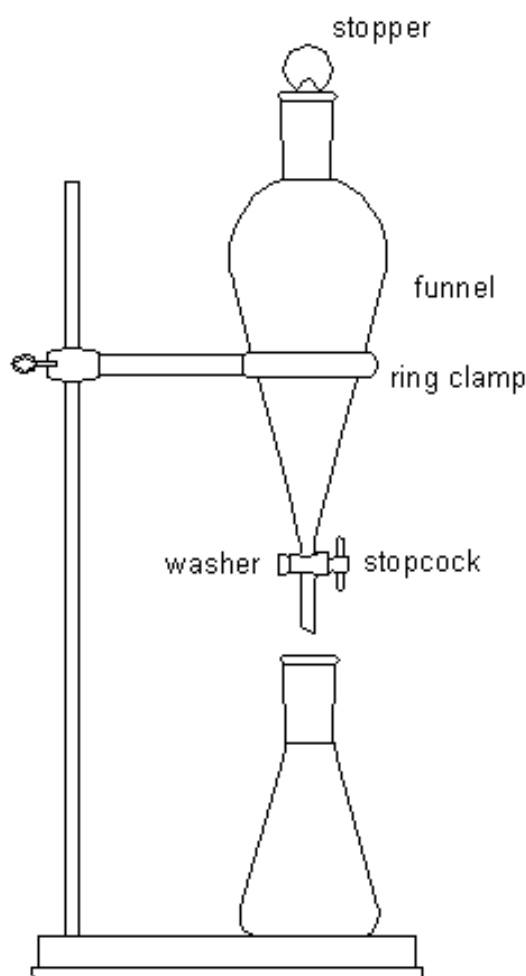
### VII. Minimum Theoretical Background

Liquid extraction is an operation in which the constituents of liquid mixture are separated by contacting it with a suitable insoluble liquid solvent which preferentially dissolves one of them. When solvent is added to the liquid mixture, two immiscible layers are formed, both containing varying amounts of different components. The isolated layers are then separated using density difference as extract phase and raffinate phase. For the recovery of solvent for reuse, extraction is followed by distillation or evaporation. Extraction utilizes the difference in solubility of constituents/ components to effect a separation. In this operation, a solute in a liquid

solution is removed by contacting the solution with another liquid solvent. The solvent is relatively immiscible with the solution. In liquid extraction, the feed solution to be handled represents one phase and the solvent to be used to effect separation represents the second phase. The mass transfer of the solute takes place from the feed solution to the solvent phase.

In extraction, the solution which is to be extracted is called the feed and the liquid with which the feed is contacted for the extraction of solute is called solvent. The solvent lean, residual liquid solution from which solute is removed is called as raffinate and the solvent rich product of the operation, containing the extract solute, is called as extract. The extract phase contains the desired product in a larger proportion.

Distribution coefficient ( $K$ ) is the ratio of concentration of solute in extract phase to the in raffinate phase.  $K = C_E / C_R$ . Higher values of distribution coefficient are generally desirable as less solvent will then be required for a given extraction duty.



**Figure 1 : Separating Funnel**

**VIII. Experimental set up:****Figure 2: Separating Funnel Photo****IX. Resources required**

Sl No.	Name of Resource	Suggested Broad Specification	Quantity
1	Burette	50 ml	2
2	Measuring cylinder	100 ml, 10 ml	2
3	Beaker	Borosil, 250 ml, 10 ml	2
4	Conical flask	Borosil, 250 ml	2

**X. Precautions**

1. Add the solution from burette slowly to get exact end point.
2. Accurate measurement of volume should be done.

**XI. Procedure****For Toluene- acetic acid**

1. Wash all the apparatus thoroughly.
2. Take 20 ml acetic acid and 30 ml toluene in a beaker.

3. Add 50 ml water to this mixture and shake well for about 20 minutes.
4. After 20 minutes, pour the mixture into a separating funnel and allow it to settle and separate into two layers.
5. Take 10 ml of bottom layer and titrate it against standard 1 N NaOH solution using phenolphthalein indicator. End point is colourless to pink.
6. Repeat titration to get constant burette reading, note down (CBR).
7. Similarly titrate 10 ml of the top layer against standard 1 N NaOH solution using phenolphthalein indicator. End point is colourless to pink.
8. Note down CBR.

#### For Chloroform- acetic acid

1. Wash all the apparatus thoroughly.
2. Take 20 ml acetic acid and 30 ml chloroform in a beaker.
3. Add 50 ml water to this mixture and shake well for about 20 minutes.
4. After 20 minutes, pour the mixture into a separating funnel and allow it to settle and separate into two layers.
5. Take 10 ml of bottom layer and titrate it against standard 1 N NaOH solution using phenolphthalein indicator. End point is colourless to pink.
6. Repeat titration to get constant burette reading, note down CBR.
7. Similarly titrate 10 ml of the top layer against standard 1 N NaOH solution using phenolphthalein indicator. End point is colourless to pink.
8. Note down CBR.

#### XII. Resources used (with major specifications)

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks(If any)
		Make	Details		
1					
2					
3					
4					

#### XIII. Actual procedure followed

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**XIV. Precautions followed**

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**XV. Observations and Calculations:****Toluene- acetic acid mixture:**

CBR for extract phase = ..... ml

CBR for raffinate phase = ..... ml

**Chloroform - acetic acid mixture:**

CBR for extract phase = ..... ml

CBR for raffinate phase = ..... ml

**Sample calculation****Toluene – acetic acid**

Extract phase

Volume of acetic acid  $V_1 = 10$  ml

Normality of acetic acid  $N_1 = \dots\dots\dots$  N

Volume of NaOH  $V_2 = \dots\dots\dots$  ml

Normality of NaOH  $N_2 = 1$  N

$$V_1 N_1 = V_2 N_2$$

$$\text{Normality of acetic acid } N_1 = V_2 N_2 / V_1$$

$$= \dots\dots\dots * \dots\dots\dots / \dots\dots\dots = \dots\dots\dots \text{N}$$

$$\text{Concentration of acetic acid } (C_E) = N_1 * \text{equivalent weight of acetic acid}$$

$$= \dots\dots\dots * 60 = \dots\dots\dots \text{g/lit}$$

Raffinate phase

Volume of acetic acid  $V_1 = 10$  ml

Normality of acetic acid  $N_1 = \dots\dots\dots$  N

Volume of NaOH  $V_2 = \dots\dots\dots$  ml

Normality of NaOH  $N_2 = 1$  N

$$V_1 N_1 = V_2 N_2$$

Normality of acetic acid  $N_1 = V_2 N_2 / V_1$

$$= \dots\dots\dots * \dots\dots\dots / \dots\dots\dots = \dots\dots\dots \text{N}$$

Concentration of acetic acid ( $C_R$ ) =  $N_1$  \* equivalent weight of acetic acid

$$= \dots\dots\dots * 60 = \dots\dots\dots \text{g/lit}$$

Distribution coefficient ( $K$ ) =  $C_E / C_R = \dots\dots\dots / \dots\dots\dots$

=

### **Chloroform – acetic acid**

Extract phase

Volume of acetic acid  $V_1 = 10$  ml

Normality of acetic acid  $N_1 = \dots\dots\dots$  N

Volume of NaOH  $V_2 = \dots\dots\dots$  ml

Normality of NaOH  $N_2 = 1$  N

$$V_1 N_1 = V_2 N_2$$

Normality of acetic acid  $N_1 = V_2 N_2 / V_1$

$$= \dots\dots\dots * \dots\dots\dots / \dots\dots\dots = \dots\dots\dots \text{N}$$

Concentration of acetic acid ( $C_E$ ) =  $N_1$  \* equivalent weight of acetic acid

$$= \dots\dots\dots * 60 = \dots\dots\dots \text{g/lit}$$

Raffinate phase

Volume of acetic acid  $V_1 = 10$  ml

Normality of acetic acid  $N_1 = \dots\dots\dots$  N

Volume of NaOH  $V_2 = \dots\dots\dots$  ml

Normality of NaOH  $N_2 = 1$  N

$$V_1 N_1 = V_2 N_2$$

Normality of acetic acid  $N_1 = V_2 N_2 / V_1$

$$= \dots * \dots / \dots = \dots N$$

Concentration of acetic acid ( $C_R$ ) =  $N_1$  \* equivalent weight of acetic acid

$$= \dots * 60 = \dots \text{ g/lit}$$

Distribution coefficient ( $K$ ) =  $C_E / C_R = \dots / \dots =$

## XVI. Results

1. Distribution coefficient for toluene- acetic acid =
2. Distribution coefficient for chloroform- acetic acid =

## XVII. Interpretation of results

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## XVIII. Conclusions

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## XIX. Practical related Questions

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- a. Identify the solute and added solvent in the liquid mixtures.
- b. Define distribution coefficient.
- c. What should be ideal value of distribution coefficient?
- d. Define leaching.
- e. Which liquid mixture (from the experiment) has more value of  $K$ ?

**[Space for Answers]**

.....  
 .....  
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[illegible]

**XX. References / Suggestions for further Reading**

- <https://nptel.ac.in/courses/102106022/16>
- <https://www.youtube.com/watch?v=TdU-Pw4tbyg>
- <https://www.youtube.com/watch?v=zvRuMxB0bZY>
- <https://www.khanacademy.org/test-prep/mcat/chemical-processes/separations-purifications/v/extractions>

**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
1	Calculation and Interpretation of result	20%
2	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

1. ....
2. ....
3. ....
4. ....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	

## **Practical No. 11: Construct Ternary diagram for Acetic acid- Water- Benzene system**

### **I. Practical Significance**

The technique of removing one component from a liquid mixture by means of a liquid solvent is termed as liquid extraction. When a mixture of liquids is not easily separable by distillation, liquid extraction is used. Close boiling mixture or substances that cannot withstand the temperature of distillation, even under vacuum may often be separated by extraction.

### **II. Relevant Program Outcomes (POs)**

**PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems

**PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.

**PO 8. Individual and team work:** Function effectively as a leader and team member in diverse/ multidisciplinary teams.

### **III. Competency and Practical Skills**

**'Use chemical process plant equipment for mass-transfer operations safely.'**

1. Use volume measuring device for accurate measurement of volume.
2. Do titration of acid- base to get end point.

### **IV. Relevant Course Outcomes**

Select relevant solvent for extraction process.

### **V. Practical Outcome**

Construct ternary diagram for system of three liquids, one pair partially soluble i.e. Acetic acid-Benzene-Water system

### **VI. Relevant Affective domain related Outcome(s)**

1. Follow safe practices
2. Maintain tools and equipment.

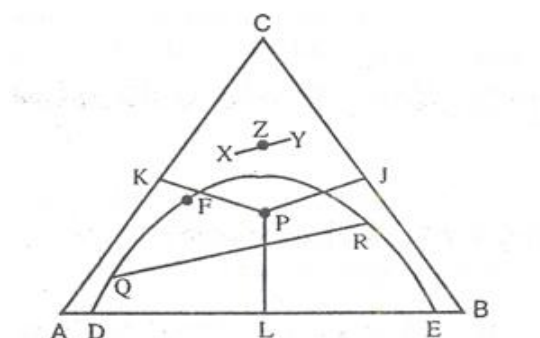
### **VII. Minimum Theoretical Background**

Liquid extraction is an operation in which the constituents of liquid mixture are separated by contacting it with a suitable insoluble liquid solvent which preferentially dissolves one of them. When solvent is added to the liquid mixture, two immiscible layers are formed, both containing varying amounts of different components. The isolated layers are then separated using density difference as extract phase and raffinate phase. For the recovery of solvent for reuse, extraction is followed by distillation or evaporation. Extraction utilizes the difference in solubility of constituents/ components to effect a separation. In this operation, a solute in a liquid solution is removed by contacting the solution with another liquid solvent. The solvent is relatively immiscible with the solution. In liquid extraction, the feed solution to be

handled represents one phase and the solvent to be used to effect separation represents the second phase. The mass transfer of the solute takes place from the feed solution to the solvent phase.

In extraction, the solution which is to be extracted is called the feed and the liquid with which the feed is contacted for the extraction of solute is called solvent. The solvent lean, residual liquid solution from which solute is removed is called as raffinate and the solvent rich product of the operation, containing the extract solute, is called as extract. The extract phase contains the desired product in a larger proportion.

In liquid -liquid extraction, when the solvent is partially miscible with the original solvent, the solubility and equilibrium relations are often shown on a triangular diagram. The composition of ternary system can be shown by a point lying inside an equilateral triangle. This is used to find equilibrium between extract phase and raffinate phase. It is used to give concept of tie line, plait point. It can be used for finding out number of stages for complete recovery of solute. Binodial curve represents two phase region that will split up into two layers in equilibrium with each other. Tie line is a line which connects two phases in equilibrium with each other. The plait point on binodial curve represent a single phase where the length of tie line is zero.



**Figure 1 Triangular diagram**

**C, A, B- Acetic Acid, Benzene, Water**

**F-Plait Point (Length of Tie Line is Zero)**

**P- Triple Point**

**Curve DQFRE – Binodal Curve**

**QR- Tie Line**

**VIII. Experimental set up:****Figure 2****IX. Resources required**

Sl No.	Name of Resource	Suggested Broad Specification	Quantity
1	Beaker	500 ml	2
2	Burette	50 ml	2
3	Measuring cylinder	100 ml, 10 ml	2
4	Conical flask	500 ml	2

**X. Precautions to be followed**

1. Add the solution from burette slowly to get exact end point.
2. Accurate measurement of volume should be done.



**XI. Procedure****A) By keeping volume of water constant**

1. Fill every conical flask with 10 ml water.
2. To the first conical flask add 10 ml chloroform.
3. Titrate this heterogeneous mixture against glacial acetic acid from burette.
4. End point is clear homogeneous phase. Note down the volume of acetic acid consumed.
5. To the second flask add 9 ml chloroform and titrate this mixture against acetic acid.
6. Note the volume of acetic acid used.
7. Repeat the procedure by adding chloroform to each flask in the descending order(8,7,6...) and then titrating against acetic acid

**B) By keeping volume of chloroform constant**

1. Fill every conical flask with 10 ml chloroform.
2. To the first conical flask add 10 ml water.
3. Titrate this heterogeneous mixture against glacial acetic acid from burette.
4. End point is clear homogeneous phase. Note down the volume of acetic acid consumed.
5. To the second flask add 9 ml water and titrate this mixture against acetic acid.
6. Note the volume of acetic acid used.
7. Repeat the procedure by adding water to each flask in the descending order(8,7,6...) and then titrating against acetic acid

**XII. Resources used**

Sr. No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks(If any)
		Make	Details		
1					
2					
3					
4					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**

1. Density of water = 1 gm/cc

1. Density of chloroform = 1.49 gm/cc

2. Density of acetic acid = 1.038 gm/cc

**A) By keeping volume of water constant**

Sr.No	Volume of water in ml	Volume of chloroform in ml	Volume of acetic acid in ml
1	10	10	
2	10	9	
3	10	8	
4	10	7	
5	10	6	
6	10	5	
7	10	4	
8	10	3	
9	10	2	
10	10	1	

**B) By keeping volume of Chloroform constant**

Sr.No	Volume of chloroform in ml	Volume of water in ml	Volume of acetic acid in ml
1	10	10	
2	10	9	
3	10	8	
4	10	7	

5	10	6	
6	10	5	
7	10	4	
8	10	3	
9	10	2	
10	10	1	

**Calculations****Part A) Sample calculation for set no:**

Weight of water (A) = volume of water \* Density of water

=

Weight of chloroform (B) = volume of chloroform \* Density of chloroform

=

Weight of acetic acid (C) = volume of acetic acid \* Density of acetic acid

=

Total weight = A+B+C

=

Weight % of water =  $A * 100 / (A+B+C)$

=

Weight % of Chloroform =  $B * 100 / (A+B+C)$

=

Weight % of acetic acid =  $C * 100 / (A+B+C)$

=

Sr.No	Wt of H <sub>2</sub> O(gm)	Wt of CHCl <sub>3</sub> (gm)	Wt of CH <sub>3</sub> COOH (gm)	Total weight	Wt % of H <sub>2</sub> O	Wt % of CHCl <sub>3</sub>	Wt % of CH <sub>3</sub> COOH
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							

**Part B) Sample calculation for set no:**

Weight of water (A) = volume of water \* Density of water

=

Weight of chloroform (B) = volume of chloroform \* Density of chloroform

=

Weight of acetic acid (C) = volume of acetic acid \* Density of acetic acid

=

Total weight = A+B+C

=

Weight % of water =  $A * 100 / (A+B+C)$

=

Weight % of Chloroform =  $B * 100 / (A+B+C)$

=

Weight % of acetic acid =  $C * 100 / (A+B+C)$

=

Sr.No	Weight of H <sub>2</sub> O(gm)	Weight of CHCl <sub>3</sub> (gm)	Weight of CH <sub>3</sub> COOH (gm)	Total weight	Wt % of H <sub>2</sub> O	Wt % of CHCl <sub>3</sub>	Wt % of CH <sub>3</sub> COOH
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							

**XVI. Results**

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**XVII. Interpretation of results**

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**XVIII. Conclusions**

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**XIX. Practical related Questions**

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- a. Identify the solute in water-chloroform-acetic acid mixture.
- b. Define tie line in triangular diagram.
- c. What is the use of triangular diagram?
- d. Define plait point.

**[Space for Answers]**

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[illegible]

**XX. References / Suggestions for further Reading**

- <https://freevideolectures.com/course/3132/mass-transfer/25>
- <https://nptel.ac.in/courses/126105011/55>
- <https://www.youtube.com/watch?v=gGYHXhcKM5s>
- <https://www.youtube.com/watch?v=J3QFoXRoKEg>

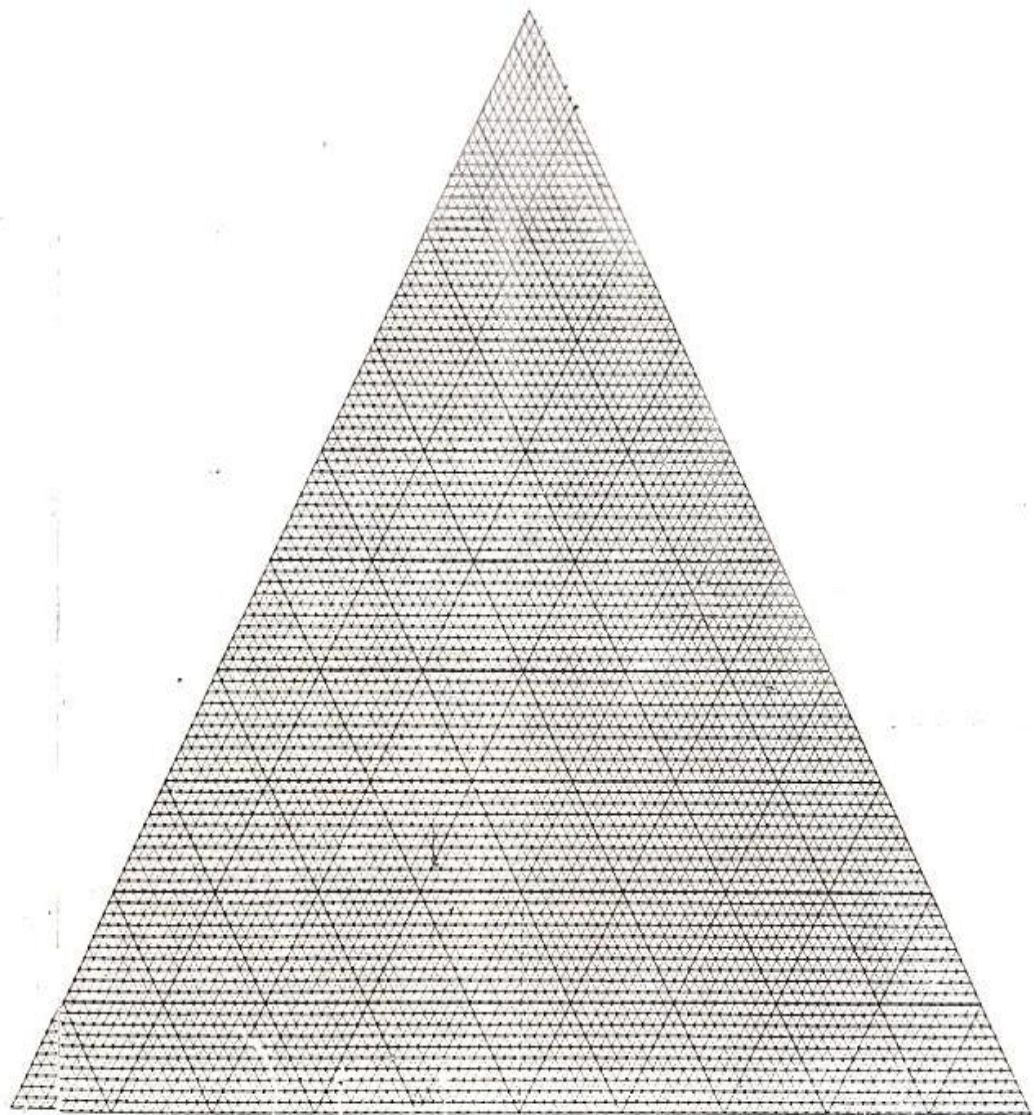
**XXI. Assessment Scheme**

Performance Indicator		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
1	Calculation and Interpretation of result	20%
2	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

1. ....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	





**Practical No. 12: Operate batch dryer to carry out drying of wet saw dust****I. Practical Significance**

Drying refers to the removal of relatively small amount of water from a solid or nearly solid material. It involves the transfer of liquid from a wet solid into an unsaturated gas phase. It is the last operation in manufacturing process and is usually carried after evaporation, filtration or crystallisation. This operation is carried out in food, agricultural, pharmaceutical and textile industries.

**II. Relevant Program Outcomes (POs)**

**PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems

**PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.

**PO4 Engineering tools:** Apply relevant technologies and Chemical engineering tools with an understanding of the limitations

**PO 8.Individual and team work:** Function effectively as a leader and team member in diverse/ multidisciplinary teams.

**III. Competency and Practical Skills**

**'Use chemical process plant equipment for mass-transfer operations safely.'**

1. Use thermocouple to measure temperature.

2. Use heater switch to maintain temperature.

**IV. Relevant Course Outcomes**

Determine the time required for drying process.

**V. Practical Outcome**

Carry out drying of wet saw dust or sand in a batch dryer to obtain the drying rate curve.

**VI. Relevant Affective domain related Outcome(s)**

1. Follow safe practices.

2. Maintain tools and equipment.

3. Practice energy conservation .

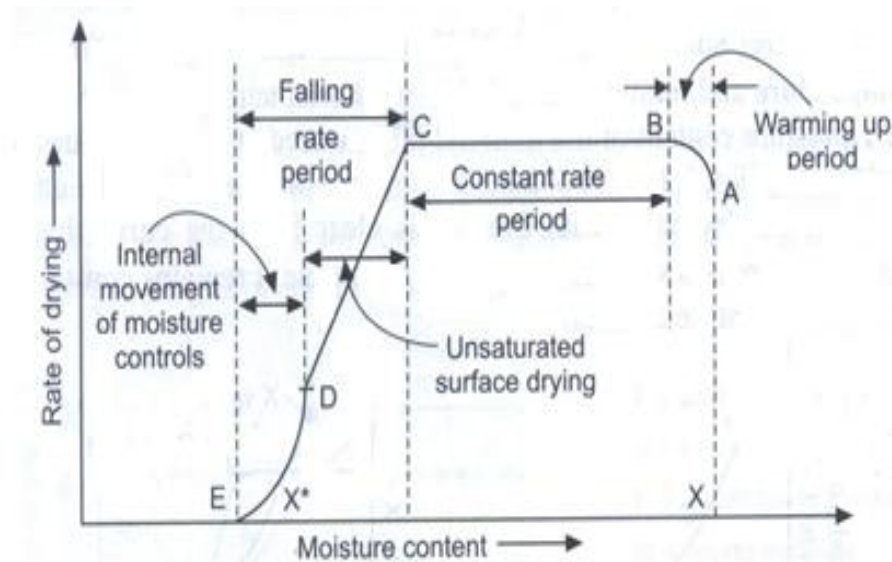
4. Demonstrate working as a leader/ a team member.

**VII. Minimum Theoretical Background**

Drying refers to an operation in which the moisture of a substance is removed by thermal means. During drying operation, mass and heat transfer occur simultaneously. Heat is transferred from the bulk of the gas phase to the solid phase and mass is transferred from the solid phase to the gas phase in the form of liquid and vapour through various resistances. The material that is transferred is the solute and

transfer takes place as the gas phase is always unsaturated with respect to the solute material.

The moisture content of a wet feed material, on wet basis, is defined as the ratio of the weight of the moisture to the weight of the wet feed material. Rate of drying depends on gas velocity, humidity of gas, area of drying surface and temperature of the gas. Rate of drying curve is plotted with rate of drying on y-axis against moisture content on x-axis.



**Figure 1 Rate of drying curve under constant drying conditions**

In a batch dryer, a definite size of batch of the wet feed is charged to the dryer and drying is carried out over a given period of time. Drying in batches is relatively expensive operation and consequently batch dryers are preferred for small scale production, pilot plant and for drying valuable materials. Tray dryer is a batch dryer.

**VIII. Experimental set up:****Figure 2****IX. Resources required**

Sl No.	Name of Resource	Suggested Broad Specification	Quantity
1	Tray dryer	Area of tray: 307.04 cm <sup>2</sup>	1
2	Blower	1 HP	1

**X. Precautions**

1. Do not start the heater before starting the blower.
2. Temperature should be maintained at the required value.

**XI. Procedure**

1. Weigh about 100 gms of sand or saw dust, add it to a pan.
  2. Add water (about 80 ml) to it so that it will just wet the sand/ saw dust.
  3. Switch on the air supply by starting the blower.
  4. Switch on the heater and maintain a constant temperature of 55-60 °C.
  5. When the required temperature is attained, place the pan inside the dryer compartment and note down the initial weight at t=0.
  6. For a definite time interval (5 minutes), note down the weight of material.
  7. Repeat the procedure till complete moisture is removed from the system.
- (This can be decided when two consecutive weight of material remains same)

**XII. Resources used**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks(If any)
		Make	Details		
1					
2					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV Observations and Calculations:**

Weight of drying sand / saw dust = .....gm

Weight of moisture = .....gm

Area of pan / tray (A) = ..... m<sup>2</sup>**Observation Table:**

Sr No	Total weight	Weight of moisture inside the system	Moisture removed from the system	Time in seconds	dw	dt	dw/dt	Rate of drying	Moisture content	Average moisture content
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

1.  $dw = w_1 - w_2 = \dots - \dots = \dots \text{gm}$

2.  $dt = t_2 - t_1 = \dots - \dots = \dots \text{Seconds}$

3.  $dw/dt = \dots / \dots = \dots$

4. Rate of drying =  $1/A(dw/dt) = \dots * \dots = \dots$

5. Moisture content ( $X_1$ ) =  $\frac{\text{Weight of water remaining}}{\text{weight of dry material}} =$

6. Average moisture content =  $\frac{X_1 + X_2}{2} =$

7. Plot drying rate curve by plotting average moisture content on x-axis and rate of drying on y axis

$$\text{Total time} = t_c + t_f = \left[ \frac{W' (X_1 - X_2)}{A R_c} \right] + \left[ \frac{W' (X_c - X_e)}{A R_c} \right] \ln \left[ \frac{(X_c - X_e)}{(X_2 - X_e)} \right]$$

=

## XVI Results

Total time required for drying (by calculation) =

(from experiment) =

## XVII. Interpretation of results

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## XVIII. Conclusions

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## XIX. Practical related Questions

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- What is the effect of velocity of gas on rate of drying?
- How drying is different from evaporation?
- How temperature of gas affect rate of drying?

**[Space for Answers]**

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[illegible]

**XX. References / Suggestions for further Reading**

- <https://nptel.ac.in/courses/102106022/36>
- <https://www.youtube.com/watch?v=PGAWyD4xiGE>
- <https://www.youtube.com/watch?v=9TsW77o3BWQ>

**XXI. Assessment Scheme**

Performance Indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

1. ....

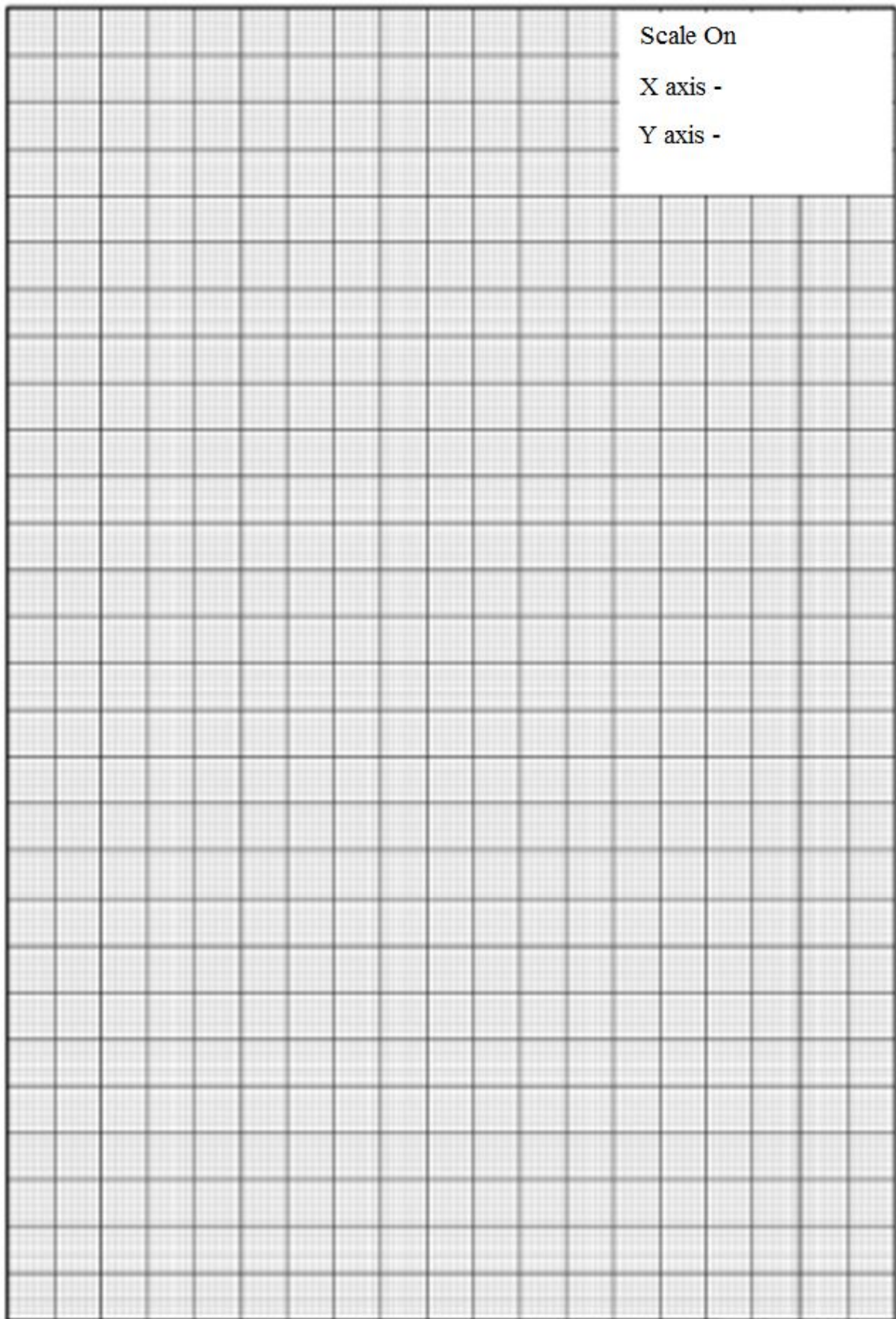
2.....

3.....

4.....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	





## Practical No. 13: Operate drum dryer to find moisture removal

### I. Practical Significance

Drying refers to the removal of relatively small amount of water from a solid or nearly solid material. It involves the transfer of liquid from a wet solid into an unsaturated gas phase. It is the last operation in manufacturing process and is usually carried after evaporation, filtration or crystallisation. This operation is carried out in food, agricultural, pharmaceutical and textile industries.

### II. Relevant Program Outcomes (POs)

**PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems

**PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.

**PO4.Engineering tools:** Apply relevant technologies and Chemical engineering tools with an understanding of the limitations

**PO 8.Individual and team work:** Function effectively as a leader and team member in diverse/ multidisciplinary teams.

**PO 9.Communication:** Communicate effectively in oral and written form.

**PSO 2.Material management and quality control:** Manage chemicals and equipment to produce quality chemical products

### III. Competency and Practical Skills

**'Use chemical process plant equipment for mass-transfer operations safely.'**

- 1 Use weighing balance to measure weight.

### IV. Relevant Course Outcomes

Determine the time required for drying process.

### V. Practical Outcome

Use the drum dryer to find the final moisture removal.

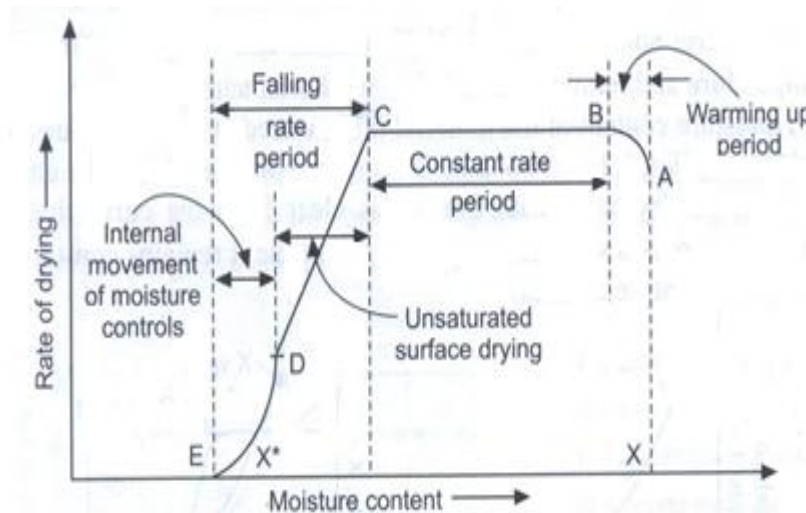
### VI. Relevant Affective domain unrelated Outcome(s)

1. Follow safe practices
2. Maintain tools and equipment.
3. Practice energy conservation.

### VII. Minimum Theoretical Background

Drying refers to an operation in which the moisture of a substance is removed by thermal means. During drying operation, mass and heat transfer occur simultaneously. Heat is transferred from the bulk of the gas phase to the solid phase and mass is transferred from the solid phase to the gas phase in the form of liquid and vapour through various resistances. The material that is transferred is the solute and transfer takes place as the gas phase is always unsaturated with respect to the solute material.

The moisture content of a wet feed material, on wet basis, is defined as the ratio of the weight of the moisture to the weight of the wet feed material. Rate of drying depends on gas velocity, humidity of gas, area of drying surface and temperature of the gas. Rate of drying curve is plotted with rate of drying on y-axis against moisture content on x-axis.

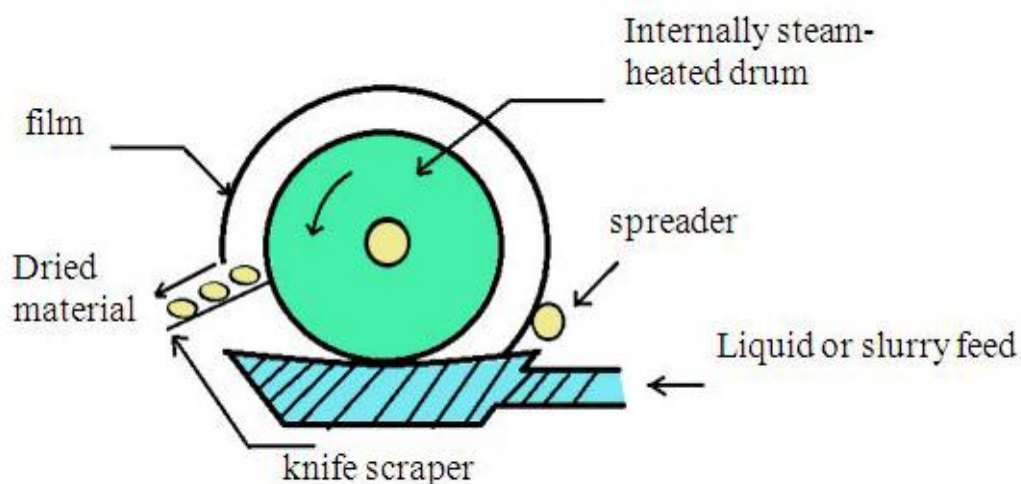


**Figure 1 Rate of drying curve under constant drying conditions**

In a batch dryer, a definite size of batch of the wet feed is charged to the dryer and drying is carried out over a given period of time. Drying in batches is relatively expensive operation and consequently batch dryers are preferred for small scale production, pilot plant and for drying valuable materials. Tray dryer is a batch dryer.

Drum dryer consists of one or more metal rolls that are heated internally by steam and rotates at about 1-10 revolutions per minute. The drum is submerged into a pool of solution or slurry contained in a trough. A spreader regulates the thickness of the film of substance on the outside of the drum and a knife on the other side to scrap the dried material from the slowly revolving drum. Vapour hood collects the vaporized moisture.

Drum dryers are suitable for handling fluid and semi fluid materials such as slurries, solutions etc.

**VIII. Experimental set up:****Figure 2****IX. Resources required**

Sr. No.	Name of Resource	Suggested Broad Specification	Quantity
1	Drum dryer assembly	Internally steam heated drum, knife scrapper and spreader	1

**X. Precautions**

1. The speed of rotating drum should not exceed the limit.

**XI. Procedure**

1. Take slurry in the trough and agitator is worked to prevent settling of solids.
2. Rotate the internally heated drum at about 1-10 rpm so that a part of the drum is submerged in the slurry.
3. Scrap the dried solid with help of scrapping knife.
4. Weigh the dried material.

**XII. Resources used**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks(If any)
		Make	Details		
1					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**

1. Weight of dried material =

**XVI. Results**

Weight of dried material = .....

**XVII. Interpretation of results**

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**XVIII. Conclusions & Recommendation**

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**XIX. Practical related Questions**

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- a. Give the classification of dryer.
- b. Give applications of drum dryer.
- c. How heating is done in drum dryer?

**[Space for Answers]**

This image shows a full page of primary-ruled paper. It features approximately 28 horizontal dotted lines spaced evenly down the page, providing a guide for handwriting practice. The paper is otherwise blank, with no margins, text, or other markings.

**XX. References / Suggestions for further Reading**

- <http://textofvideo.nptel.ac.in/103104046/lec36.pdf>
- <https://www.youtube.com/watch?v=wQcBtS2KsmQ>
- <https://www.youtube.com/watch?v=0XpSnRXkmCE>
- [https://www.youtube.com/watch?v=62\\_WIhwcfQo](https://www.youtube.com/watch?v=62_WIhwcfQo)

**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

1. ....
2. ....
3. ....
4. ....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	

## **Practical No. 14: Determine percent recovery and yield of crystallization in a batch crystallizer**

### **I. Practical Significance**

Crystallisation is an important operation in chemical industry as the number of salable products has to be in the form of crystals. This operation gives almost pure product in the form of crystals of a desired size. Crystallisation requires much less energy for separation as compared to other purification methods.

### **II. Relevant Program Outcomes (POs)**

**PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems

**PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.

**PO4. Engineering tools:** Apply relevant technologies and Chemical engineering tools with an understanding of the limitations

**PO6. Environment and sustainability:** Apply Chemical engineering solutions also for sustainable development practices in societal and environmental contexts.

**PSO 2. Material management and quality control:** Manage chemicals and equipment to produce quality chemical products

### **III Competency and Practical Skills**

**'Use chemical process plant equipment for mass-transfer operations safely.'**

1. Use thermocouple to measure temperature.
2. Use rotameter to measure volumetric flow rate.

### **IV. Relevant Course Outcomes**

Determine the yield of crystals obtained.

### **V. Practical Outcome**

Use a batch crystallizer to determine percent recovery and yield of crystallization

### **VI. Relevant Affective domain unrelated Outcome(s)**

1. Follow safe practices
2. Maintain tools and equipment.

### **VII. Minimum Theoretical Background**

Crystallisation is an operation in which solid particles are formed from a liquid solution. It is a solid- liquid operation used to separate a solute from its solution in the form of crystals. In this operation, mass is transferred from the liquid phase to a pure solid crystalline phase. Crystallisation requires much less energy and may be carried out at relatively low temperature and on a scale ranging from a few grams to thousands of tons per day.



Supersaturation is the quantity of solute present in a solution compared with the quantity of solute that is in equilibrium with the solution. Crystallisation cannot take place unless a solution is supersaturated. The amount of crystals formed depends upon the difference in saturation concentration since once the crystallization begins, the extra solute held in the solution due to supersaturation also comes out of the solution in the form of crystals.

Batch crystallization is characterized by the fact that the system is always in the unsteady state. The initial super saturation at which crystallization starts will drop quickly from relatively high value to the saturation value. If crystal growth is to continue, the solution must be maintained in the meta-stable region. As a consequence, cooling must continue and the batch temperature must continue to drop during the growth period. In batch crystallization it is comparatively easy to penetrate the labile zone producing a fine mass of fine crystals. By using controlled seeding the solution will not become labile thereby aiding crystal growth. The percentage yield of a crystallization process is the amount of the solute crystallised expressed as a percentage of the amount of the solute present in the feed solution.

### VIII. Experimental set up:



**Figure 1: Batch Crystallizer**

**IX. Resources required**

Sl No.	Name of Resource	Suggested Broad Specification	Quantity
1	Agitated tank crystalliser	S.S. 304, size: 1 lit, jacketed for cooling	1
2	Heating bath cum receiving tank	S.S. 304, 50 lit, insulated	1
3	Temperature sensor	RTD.PT-100	4

**X. Precautions**

1. Before switching on the heater, ensure that water level in the tank is at least 6" above the heating element.
- 2 Crystallization will not occur if the solution is not supersaturated.
- 2 After experimentation wash the tanks with water and drain.

**XI. Procedure**

1. Prepare a saturated solution of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in water at 80 °C by dissolving 66.2 gm  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in 100gms of solution (1.9586 gm  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  per gm of free water). The crystallizer should be filled to  $3/4^{\text{th}}$  the capacity. Prepare about 1.5 L of solution of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  and water. During mixing, the agitator should be used for effective mixing.
2. After uniform mixing has been achieved, stop the electric supply.
3. Allow the flow of cold water to pass through the jacket at a pre-fixed flow rate with the help of water by-pass valve & a Rotameter. Record the flow rate (flow rate may be fixed such that the rise in cooling water temperature is maximum around 5-7°C maintained by using ice).
4. Record the temperature of inlet water, outlet water and solution temperature with the help of respective Sensors.
5. Put 10 gm  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in the crystallizer when the temp reaches to 50°C for seeding.
6. Carry out the crystallization process for about 2 hrs.
7. After two hours, stop the cooling water supply, open the valve fixed at the cone of the crystallizer and collect the slurry in the bottom receiving tank that is fixed with a mesh at the top. The crystals shall be collected on the mesh and liquid in the tank.
8. Collect all the crystals from the mesh on a filter paper and weigh. Let the weight of the product crystals be P (kg). For collecting all the crystals you may flush the crystallizer tank with 100-200ml of saturated  $\text{MgSO}_4$  solution.

**XII. Resources used**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					
3					

**XIII Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**

Concentration of feed solution of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  at  $80^\circ\text{C}$

= 66.2 gm /100 gm of solution

= 1.9586 gm  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  / gm of water

Wt. of Feed solution, F = ..... kg

Weight of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  = ..... kg

Amount of water = ..... kg

Solubility of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  at  $20^\circ\text{C}$  = 53.8 gm/100gm of solution

= 1.1645 gm  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  / gm of water

Wt. of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  = ..... kg

Amount of water = ..... kg

Conc. of feed solution at  $50^\circ\text{C}$  = 1.9586 gm  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  per gm of water

= 0.662 gm  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in 1 gm of solution

Time T, min.	Cooling water		Solution temp. T <sub>s</sub> °C
	Inlet, T <sub>wi</sub>	Outlet, T <sub>wo</sub>	

Amount of crystal of MgSO<sub>4</sub>.7H<sub>2</sub>O collected after 2 hrs.(P)=      kg

Wt. of mother liquor      (M)=      kg

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#### XVI. Results

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#### XVII. Interpretation of results

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#### XVIII. Conclusions

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#### XIX. Practical related Questions

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- Define yield of crystallisation.

- Which are the steps involved in crystallisation?
- What is the effect of impurities on crystal formation?
- What is mother liquor and magma?

**[Space for Answers]**

[illegible]

**XX. References / Suggestions for further Reading**

<http://textofvideo.nptel.ac.in/102106022/lec35.pdf>

<https://www.youtube.com/watch?v=2F9NEoXvkQE>

<https://www.youtube.com/watch?v=rgdrkbbkZ4Q>

**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

1. ....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	

**Practical No. 15: Determine solubility of a salt and plot solubility curve.****I. Practical Significance**

Crystallisation is an important operation in chemical industry as the number of products has to be in the form of crystals. This operation gives almost pure product in the form of crystals of a desired size. Crystallisation requires much less energy for separation as compared to other purification methods.

**II. Relevant Program Outcomes (POs)**

**PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems

**PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.

**PO 8.Individual and team work:** Function effectively as a leader and team member in diverse/ multidisciplinary teams.

**PO 9.Communication:** Communicate effectively in oral and written form.

**III. Competency and Practical Skills**

**'Use chemical process plant equipment for mass-transfer operations safely.'**

1. Use weighing balance for accurate weighing of salt.

2. Use thermometer to measure temperature.

**IV. Relevant Course Outcomes**

Determine the yield of crystals obtained.

**V. Practical Outcome**

By heating or cooling method determine the solubility of a salt and obtain the solubility curve.

**VI. Relevant Affective domain related Outcome(s)**

1. Follow safe practices

2. Maintain tools and equipment.

3. Demonstrate working as a leader / a team member.

**VII. Minimum Theoretical Background**

Crystallisation is an operation in which solid particles are formed from a liquid solution. It is a solid- liquid operation used to separate a solute from its solution in the form of crystals. In this operation, mass is transferred from the liquid phase to a pure solid crystalline phase. Crystallisation requires much less energy and may be carried out at relatively low temperature and on a scale ranging from a few grams to thousands of tons per day.

Supersaturation is the quantity of solute present in a solution compared with the quantity of solute that is in equilibrium with the solution. Crystallisation cannot take place unless a solution is supersaturated. The amount of crystals formed depends upon the difference in saturation concentration since once the crystallization begins, the extra solute held in the solution due to supersaturation also comes out of the solution in the form of crystals.

Solubility of a solute in a given solvent is the concentration of the solute in a saturated solution at a given temperature. A graphical relationship between the solubility of a solute and temperature is termed as solubility curve. The concentration necessary for crystal formation and chemical species that separate can be determined from solubility curves. It shows the effect of temperature on the solubility of the solute. The solubility of solute in a given solvent may increase, decrease or remain more or less constant with temperature. Solubility curves have no general shape or slope.

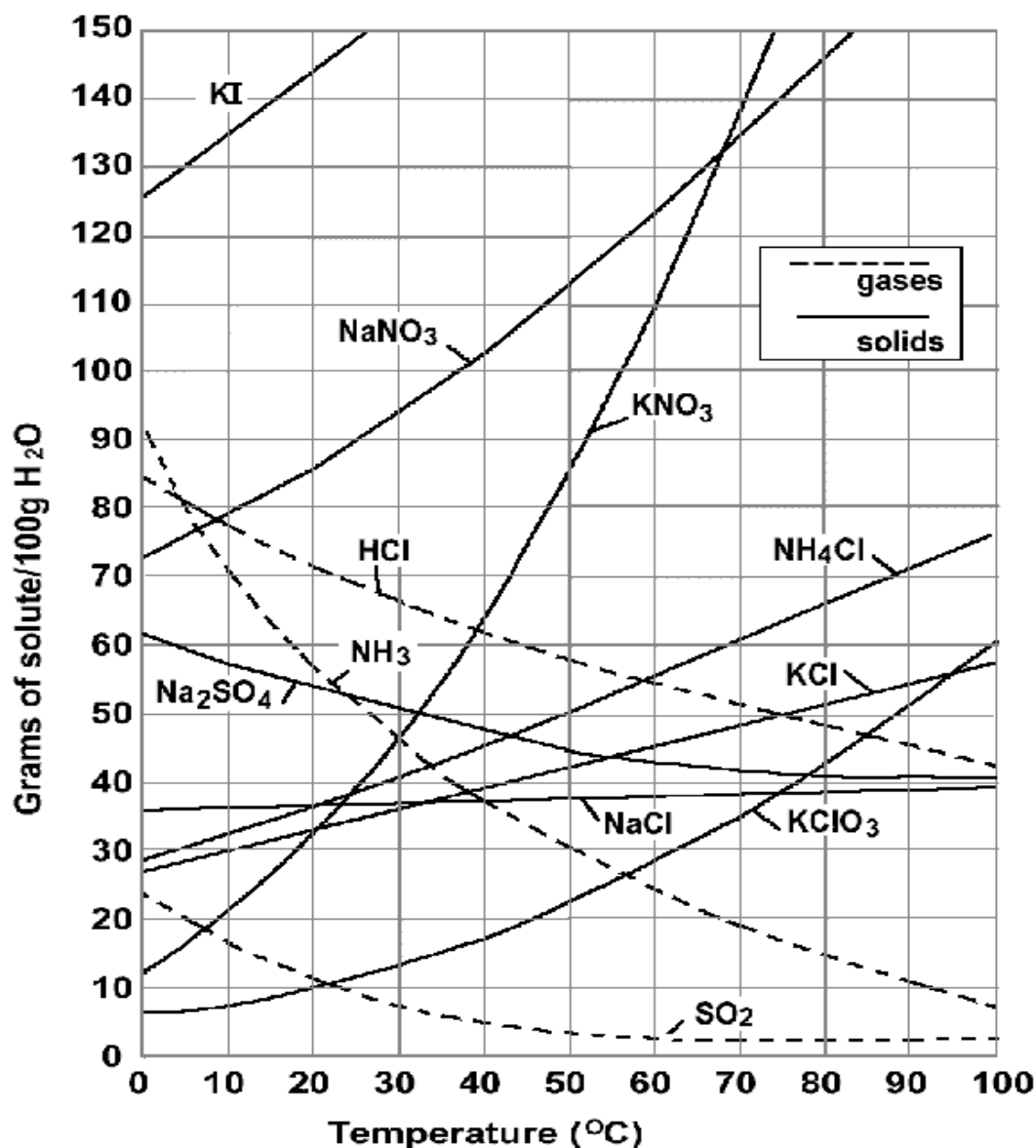


Figure 1 solubility curve for different salts



**VIII. Experimental set up: -****Figure 2****IX. Resources required**

Sl No.	Name of Resource	Suggested Broad Specification	Quantity
1	Beaker	500 ml capacity borosil	2
2	Heater	Electrical	1
3	Test tubes	standard	4

**X. Precautions**

1. Temperature should be noted carefully..
2. Accurate weight should be taken.

**XI Procedure :-****Part A: While heating**

1. Take 100 ml water in a beaker.
2. Weigh 100 gram potassium nitrate in a watch glass.
3. Add salt (potassium nitrate) into water in the beaker, dissolve by stirring.
4. Keep on adding the salt and dissolving it till the solution becomes saturated.
5. When saturation is attained, note down temperature of solution, height of solution in beaker and weight of salt dissolved.
6. Increase the temperature of solution by  $10^{\circ}\text{C}$  and repeat steps 4 & 5.
7. Repeat the experiment for 4 more temperature, increasing  $10^{\circ}\text{C}$  every time.
8. Measure the diameter of the beaker.
9. Calculate solubility and plot solubility curve by plotting solubility on y-axis and temperature on x-axis.

**Part B: While cooling**

1. Using a marking pencil, number four test tubes 1 through 4. Place the tubes in a test tube rack.
2. On the balance, measure out exactly two grams of potassium nitrate. Pour the salt into test tube 1.
3. Repeat step 2 for the following masses of  $\text{KNO}_3$ . Add each amount to the test tube indicated.  
4.0 g to test tube #2  
6.0 g to test tube #3  
8.0 g to test tube #4
4. Add exactly 5.0 mL distilled water to each test tube.
5. Fill a 400 mL beaker about three-fourths full of tap water. This will be used as a water bath. Heat the water to 90 degrees Celsius and adjust the flame to maintain the water at this temperature.
6. Stir the  $\text{KNO}_3$  water mixture with a glass stirring rod until the  $\text{KNO}_3$  is completely dissolved. Remove the stirrer and rinse it off. Loosen the clamp and, using a test tube holder, remove the tube.
7. While lab partner number one repeats step 6 for test tube #2, have the other lab partner place the warm thermometer dipped into the warm solution into test tube #1. Hold the test tube up to the light and watch for the first sign of crystallization in the solution. At the instant crystallization occurs observe and report the temperature. Should crystallization start so quickly, re-dissolve the liquid and restart this part.
8. Steps 6 and 7 should be followed for all four test tubes. One lab partner should stir the potassium nitrate until it dissolves, and the other should record the temperature.
9. If any inaccurate results are obtained, repeat the procedure by reheating in a hot water bath.
10. Calculate solubility and plot solubility curve by plotting solubility on y-axis and temperature on x-axis.

**XII. Resources used**

Sr.No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks(If any)
		Make	Details		
1					
2					
3					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XIV. Observations and Calculations:****Part A: Heating**

Diameter of the beaker = .....cm

Area of the beaker = .....cm<sup>2</sup>

Temperature	Amount of salt dissolved(gm)	Height of solution	Volume of solution(cm <sup>3</sup> )	Volume of solution(lit)	Solubility (gm/ lit)

**Part B: Cooling**

Test tube #	Grams of KNO <sub>3</sub> /5.0mL H <sub>2</sub> O	Solubility(grams of KNO <sub>3</sub> /1 lit H <sub>2</sub> O)	Crystallization temperature (Celsius)
1	2.0g/5.0mL		
2	4.0 g/5.0 mL		
3	6.0g/5.0mL		
4	8.0g/5.0mL		

**Part A : While heating**

1. Temperature of solution = .....<sup>0</sup>C

2. Volume of solution = Area of beaker \* height of solution

$$= ..... * ..... = ..... \text{ cm}^3 = ..... \text{ lit}$$

3. Amount of salt dissolved = ..... gms

4. Concentration of solution = Amount of salt / volume of solution in lit

$$= ..... / ..... = ..... \text{ gm/lit}$$

5. Plot solubility curve by plotting solubility on y-axis and temperature on x-axis.

**Part B: While cooling**

1. Using Proportions convert the experimental mass/volume ratios to equivalent mass/100mL ratios.

$$2.0\text{g}/5.0 \text{ mL} = 40\text{g}/100\text{mL}$$

$$4.0\text{g}/5.0\text{mL} = 80\text{g}/100\text{mL}$$

$$6.0\text{g}/5.0\text{mL} = 120\text{g}/100\text{mL}$$

$$8.0\text{g}/5.0\text{mL} = 160\text{g}/100\text{mL}$$

2. Plot mass of solute per 100mL of water on the Y axis and temperature on the X-axis.

3. Construct a solubility curve by connecting the plotted points on your graph.

**XVI. Results**

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**XVII. Interpretation of results**

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**XVIII. Conclusions**

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**XIX. Practical related Questions**

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- a. Define solubility.
- b. What is the effect of temperature on solubility?
- c. Define saturated and supersaturated solution.
- d. How super saturation is attained in this experiment?

**[Space for Answers]**

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**XX References / Suggestions for further Reading**

- <http://textofvideo.nptel.ac.in/102106022/lec35.pdf>
- <http://www.eolss.net/ebooks/sample%20chapters/c06/e6-34-03-02.pdf>
- <https://www.youtube.com/watch?v=rgdrkkbkZ4Q>

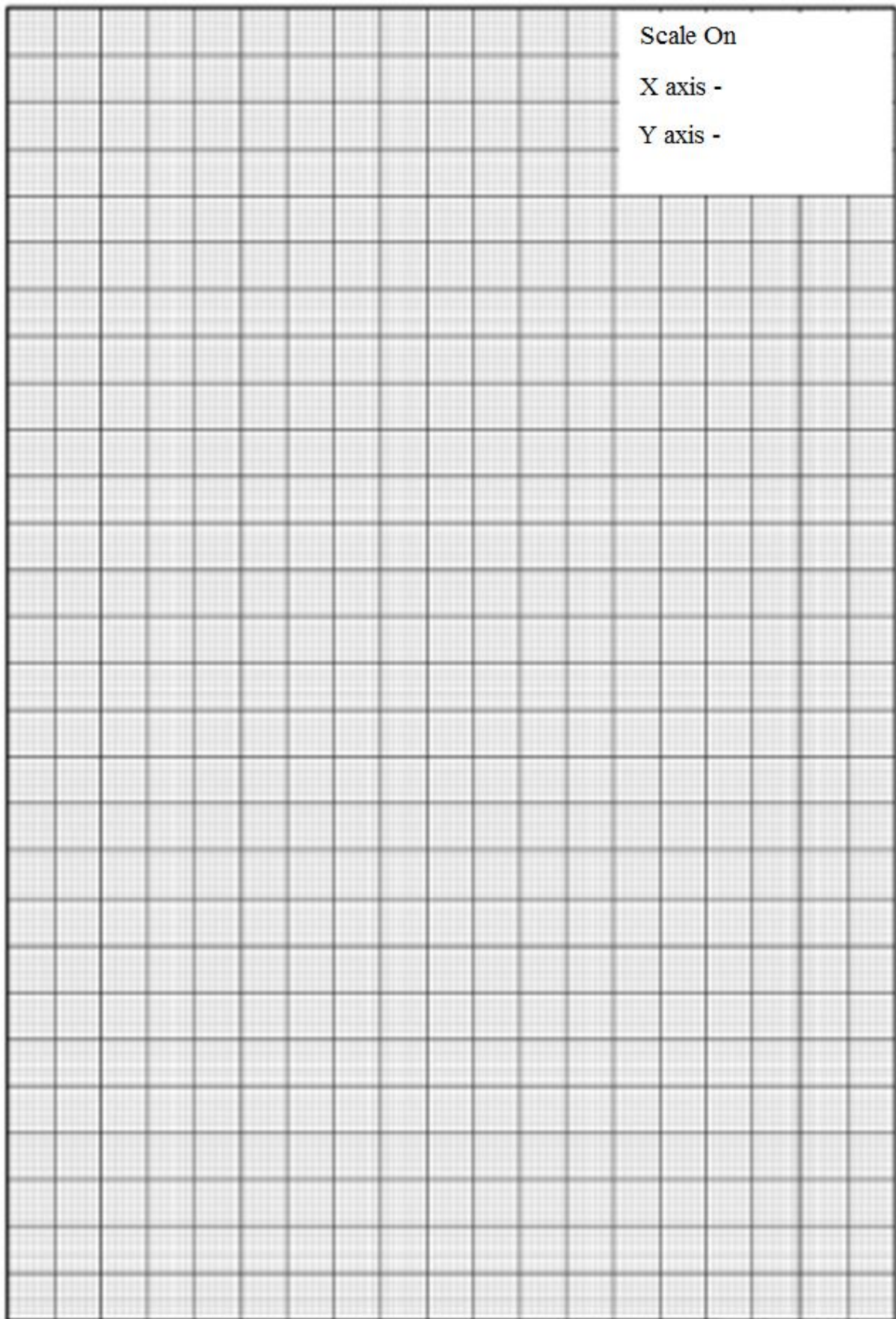
**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

- 1.....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	



## **Practical No. 16: Use the process simulator to analyze the parameters of Distillation Column**

### **I. Practical Significance**

Simulation software is used to study the effect on various process variables such as pressure, temperature, flow, level by introducing / activating malfunctions in the normal operation.

### **II. Relevant Program Outcomes (POs)**

***PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems*

***PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.*

***PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.*

### **III. Competency and Practical Skills**

***‘Use chemical process plant equipment for mass-transfer operations safely.’***

To control and study the effects of various parameters on Distillation process.

### **IV. Relevant Course Outcomes**

Use various distillation methods in chemical industry.

### **V. Practical Outcome**

Use the process simulator to analyze the parameters of distillation column.

### **VI. Relevant Affective domain related Outcome(s)-**

1. Load the exercise and run the simulation.
2. Record the change in values of process variables with introduction of malfunction.

### **VII. Minimum Theoretical Background**

#### **Process**

The distillation tower operations program uses conventional bubble cap plate distillation tower. These are so named because they have a number of horizontal plates or trays, which are usually equally spaced in the upright tower shell. These bubble plates provide time for mixing and interchange of vapor and liquid flows required for carrying out distillation separation. The relatively large number of trays used generally produce tall and upright structure, and is the reason for the term "column" or "tower".

The physical arrangement of the distillation tower used in this training and its auxiliary equipment, are shown in the graphics. The column contains 42 plates, which is required to achieve desired separation. Liquid feed enters from the feed pump to some point way down the column. The plate on which feed enters is called "feed plate". The part of the column above the feed plate is called rectifying Section and that



below including the feed plate is called stripping section. The vaporizer or reboiler is provided at the bottom with the steam coil for heating the liquid at the bottom of the tower to its boiling point. Vapor leaves from the top of the tower and passes through the overhead condenser, where large part of it is condensed. The condensed liquid flows into the reflux drum. The high boiling material leaves the bottom of the tower as bottom product.

The liquid in the reflux drum, which has high percentage of "fraction" of low boiling material, is pumped out by reflux pump. A large part of pump flow is returned directly to the column as "liquid reflux". The remaining liquid leaves the tower as "overhead liquid" product. Uncondensed overhead vapors leave the reflux drum through the off-gas line as overhead vapor product.

The vapor rise through each tray of the tower is series path. In passing through each plate, the vapor is deflected beneath the surface of the liquid and is distributed into the liquid by means of bubble cap. Intimate mixing of the two streams of liquid and the vapor, throughout their countercurrent flow paths, is maintained.

The hot vapors entering the plate give up the heat to the liquid. Some of high boiling material in the vapors is condensed and passes to the lower plate as liquid. The heat supplied condensing hot vapor causes some of the low boiling material to vaporize and passes upward to the tray above. The combined action of all the plates causes the low boiling material to work its way up the column as vapor, while the high boiling material drops to the bottom of the column as liquid.

The temperature of the boiling liquid is fixed by the column pressure and liquid composition and heat balance is always maintained. For the continuous balanced operation, the sensible heat of the liquid and vapor flows into the plate plus the latent heat of the condensing vapor must equal the sensible heat of the liquid and the vapor flow out, plus the heat loss and the heat of mixing. Since the temperature change from one plate to the another is usually small, the heat balance effectively reduces the balance between the heat given up by the condensing vapor and the heat required to vaporize the liquid leaving the plate. And as the heat of vaporization and condensation have similar values, the vapor rate out of the plate is nearly equal to the vapor rate into the plate.

Liquid enters the bottom part of the tower from the bottom plate down-flow pipe. Part of this liquid flows into the reboiler section, where heat from the steam supply is applied to raise the liquid temperature to its boiling point and produce a vapor flow. The vapor from the reboiler reenters the bottom plate and proceed upward in the distillation column. Liquid containing high percentage of boiling material is drawn off as the bottom's liquid product.

The heat required to raise the liquid at its bubble point temperature depends upon the tower bottom pressure and the nature of the liquid mixture. More heat tends to boil off the low boiling mixture, producing the vapor flow to the bottom of the tower. The more vapor produced, the richer the bottom liquid becomes, in high boiling material. Therefore, for reasonably constant column pressure, the bottom plate temperature can be used to adjust the fraction of high boiling material in the bottom

product flow. Increasing the bottom plate temperature will increase the bottom fraction of boilers.

Vapour containing a high percentage of low boiling material leaves the top plate and goes to the overhead condenser, where a large part of it is condensed. Part of the condenser vapours are drawn off as an overhead liquid product, and the remainder is returned as liquid reflux. This reflux becomes the liquid input to the top plate and flows from the top of the column down to the feed plate. Note that the liquid reflux contains about the same fraction of low boiling material as the vapour from the top plate.

The reflux rate effectively determines the time that the feed materials remains in the column . A large reflux rate means high portion of the overhead vapours are returned to the column giving the material a longer time exposure to the separation operation, making it richer ( higher percentage) in low boiling material. A smaller reflux flow will produce a less pure overhead product.

### **Instructor Function :**

#### **1 TAG READS :CYCLE TIME**

RANGE :0.25 to 2 secs.

NORMAL :1 sec.

FUNCTION : This setting selects the step time interval DT for calculating all the variables in one cycle. To have real simulation, DT must match the time the computer takes to complete one cycle. If DT is higher, simulation is fast and if DT is lower | simulation is slow.

#### **2 TAG READS : TIME LAG**

RANGE : 0 – 50 secs.

NORMAL : 50 secs.

FUNCTION : This can be used to change the plate liquid time constant i.e. hold up on each tray.

#### **3 TAG READS : FEED RATE**

RANGE : 0-15 kg/s

NORMAL : 6.06

FUNCTION : Sets the maximum feed rate (the feed flow rate when feed control valve is fully open). Can be used to vary the size of the feed control valve.

4 TAG READS : FEED COMPOSITION

RANGE : 0-100 %

NORMAL : 28%

FUNCTION: Specifies the weight fraction of the low boiling (high volatility) component or key component in the feed stream - a setting of 28 means 28% by weight of the liquid feed is the lower boiling component.

5 TAG READS : CW TEMP

RANGE : 0-100C

NORMAL : 21.11C

FUNCTION : This setting decides the temperature of cooling water entering the overhead condenser. Increasing the setting increases temperature of cooling water supply.

6 TAG READS : FOUL COND

RANGE : 0-100 %

NORMAL : 50%

FUNCTION: Increasing the setting increases the fouling in condenser and decreases the heat transfer rate from hot side to cold side of E-102.

7 TAG READS : VAPOR FLOOD

RANGE : 0-100%

NORMAL : 100%

FUNCTION : Simulates effect of column flooding when dial setting is made smaller vapor rate at top of tower reduces and is dumped back to tower. Liquid starts building in column.

### VIII. Experimental set up :

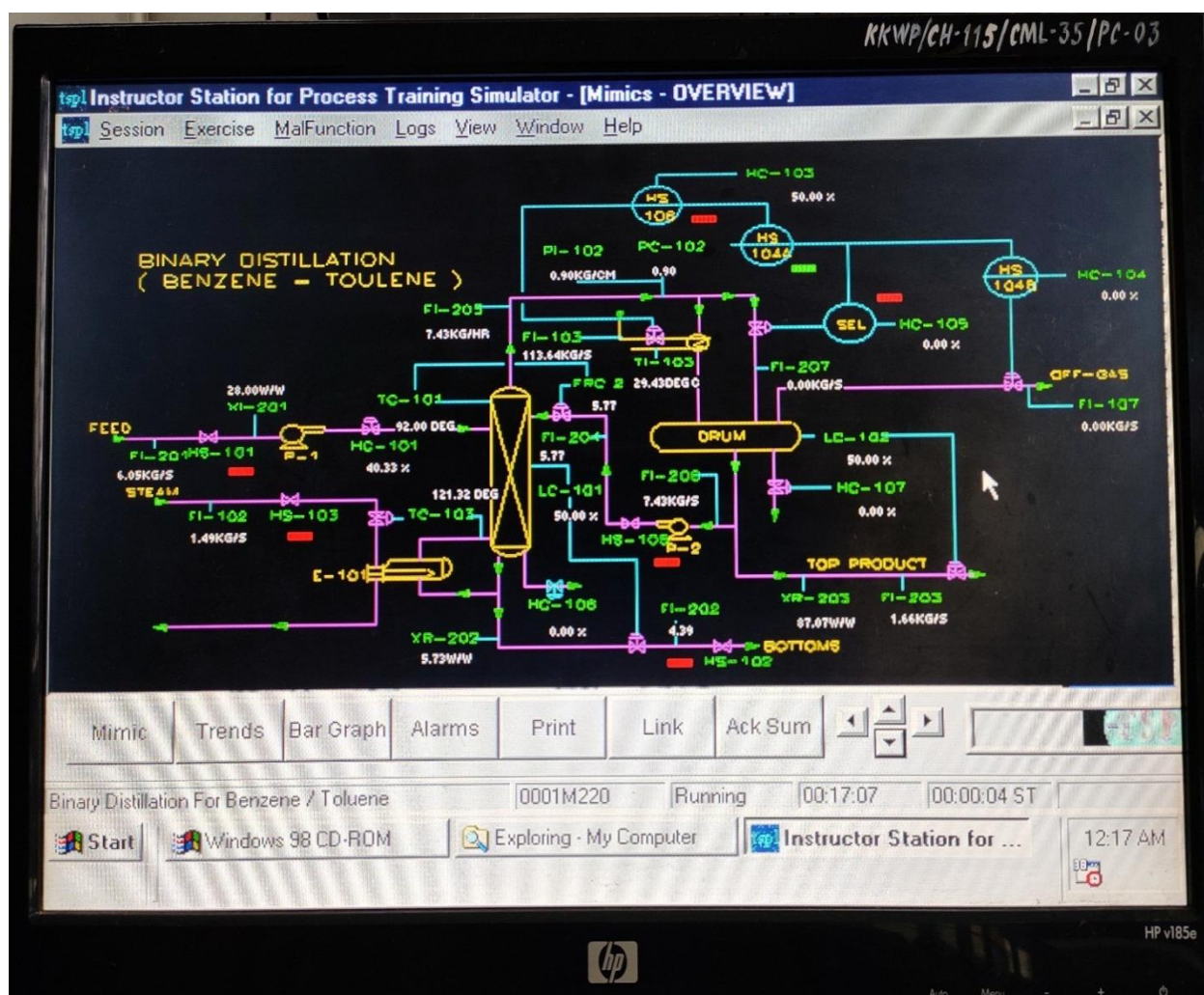


Fig. 1

### IX. Resources required

Sl No.	Name of Resource	Suggested Broad Specification	Quantity
1	Triangle Simulation Private Limited or MSBTE Sponsored TSPL	As per standard specification Version 2017	1
2	Personal Computer	P-IV or higher version PC	1

**X. Precautions to be followed**

After starting the software, ensure that the link of Binary / Multicomponent Distillation is showing in the dialogue box.

**XI. Procedure**  
**Design start**

1. Set Student Loading Function as per the settings given in setup manual for design start.
2. Set Student Switches as per settings given in setup manual.
3. Set the controllers as per settings given in setup manual.
4. Adjust controller outputs manually as per setup manual.
5. You will get display of operating variables on the screen.
6. Check value of all the instructor functions whether they are as per up data. If not you can change it from keyboard

**Cold start (optional)**

The tower should be cold that is :

- Cooling water valve closed.
  - Feed pump P-1 and reflux pump P-2 off.
  - All controllers on manual control.
  - All controller outputs at zero (control valves closed).
1. Set the proportional band, reset rate and mode for each controller used in controlling the tower as per the values recommended by the Instructor Manual.  
The Instructor will give you these settings.
  2. Start the feed pump P-1.
  3. Ask the Instructor to start loading the reboiler with some feed.
  4. During the time it takes to load the reboiler, open the C-2 Condenser Cooling water valve using the manual control on the operator's console. Set cooling water valve to 53.1 %.
  5. Using the manual control set the output of controller PC-102 to 50 % (both the hot vapor bypass control valve and the off-gas vent control valve will be closed).
  6. Check the Reboiler level, as indicated by LC-101. When the level reaches 25-30 %, stop the loading operation.
  7. Put all the controllers in AUTO mode and let the plant settle at design conditions.
  8. After the system reaches to steady state, you can do all the exercises.

**XII. Resources used**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks(If any)
		Make	Details		
1					
2					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**  
**Transmitter details:**

Instrument No.	UNITS	Description	Hi Range	Lo Range	PV
TC-101	DEG C	COL TOP TEMP	150.000	50.000	
TC-103	DEG C	COL BTM TEMP	200.000	100.00	
PC-102	Kg/cm <sup>2</sup>	COL TOP PRESS	2.000	0.00	
LC-102	m	REFLUX DRM_LV	00.000	0.000	
LC-101	m	COL_BTMT LVL	100.000	0.000	
FRC-26	Kg/hr	COL RX	10.000	0.000	
HC104	%	VENT	100.000	0.000	
HC105	%	VAPOR BYPASS	100.000	0.000	
HC107	%	RX_DRM DRAIN	100.000	0.000	
HC106	%	COL_BTMT DRAIN	100.000	0.000	
FI-102	Kg/s	STM TO REBOILER	2.000	0.000	
FI-201	Kg/s	FEED RATE	15.000	0.00	
FI-204	Kg/s	REFLUX FLOW	0.000	0.00	
FI-202	Kg/s	BTM PRODUCT	10.000	0.000	
FI-207	Kg/s	OFF GAS	15.000	0.000	
FI-206	Kg/s	LIQUID COND	15.000	0.000	

FI-107	Kg/s	OFF GAS	2.000	0.000	
FI-103	Kg/s	CW TO COND	225.000	0.000	
TI-103	DEG C	CW OUTLET	100.000	0.000	
FI-203	Kg/s	TOP PRODUCT	5.00	0.000	
XI-201	W/W	FEED COMP	100.000	0.000	
XR-202	W/W	BTM COMP	30.000	0.000	
XR-203	W/W	TOP COMP	100.000	0.000	
PI-102	KG/CM2	O/H PRES	2.000	0.000	
TI-001	KG/CM/2	O/H PRES	2.000	0.000	
TI-002	DEG C	TRAY-2 TEMP	200.000	0.000	
TI-003	DEG C	TRAY-3 TEMP	200.000	0.000	
TI-004	DEG C	TRAY-4 TEMP	200.000	0.000	
TI-005	DEG C	TRAY-5 TEMP	200.000	0.000	
FI-205	Kg/s	VAPOR FLOW	20.000	0.000	
HC-101	%	FEED FLOW	100.000	0.000	
CYC_TIM %	CYCLE TIME	115.000	0.000	115.000	
PRES_TAU		TOW FOR PRESS	115.000	0.000	
LIQ_TAU		TIME LAG	0.000	0.000	

### Sample Exercise.

#### Ex .1. TOWER TOP TEMPERATURE

**ACTION** : The student makes a small change in the output of TC-101 (96.150) in manual mode

**RESULT** : A display of the result appears on FI-204 (5.900).

**DISCUSSION** The changed controller output causes a change in the top temperature, by changing the reflux flow. do not bother about other changes.

## XVI. Results

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### **XVII. Interpretation of results**

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### **XVIII. Conclusions & Recommendation**

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### **XIX. Practical related Questions**

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- a) Define Binary Distillation process with example.
- b) How Temperature affect on distillation?
- c) List out the major components in Distillation Equipment with their function.
- d) What is Simulation?
- e) List out the companies where Simulation softwares used.

f) *Ex .1.* TOWER BOTTOM TEMPERATURE

ACTION : The student makes a very small change <approx .5%> to the student setpoint of TC-103.

RESULT :

DISCUSSION:

*Ex .2.* TOWER OVERHEAD PRESSURE

ACTION : The student a small change to the setpoint of PC-102.

RESULT :

DISCUSSION:

**[Space for Answers]**

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[illegible]

**XX. References / Suggestions for further Reading**

- <https://www.trianglesimulation.com/operator-training-simulator-for-engineering-institutes.html>
- <http://www.rroij.com/open-access/modeling-and-control-of-binary-distillationcolumn.php?aid=43804>
- <http://controlguru.com/distillation-introduction-to-control/>
- <https://www.controleng.com/articles/distillation-columns-internal-reflux-control/>

**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

- 1.....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	

## **Practical No. 17: Use the process simulator to analyze the parameters of Dryer**

### **I. Practical Significance**

Simulation software is used to study the effect on various process variables such as pressure, temperature, flow, level by introducing / activating malfunctions in the normal operation.

### **II. Relevant Program Outcomes (POs)**

***PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems*

***PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.*

***PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.*

### **III. Competency and Practical Skills**

***‘Use chemical process plant equipment for mass-transfer operations safely.’***

- Use chemical process plant equipment for mass-transfer operations safely.
- To control and study the effects of various parameters on Dryer process.

### **IV. Relevant Course Outcomes**

Use various Drying methods in chemical industry.

### **V. Practical Outcome**

Use the process simulator to analyze the parameters of Dryer

### **VI. Relevant Affective domain related Outcome(s)-**

1. Load the exercise and run the simulation.
2. Record the change in values of process variables with introduction of malfunction.

### **VII. Minimum Theoretical Background**

In unit operations of chemical engineering, drying is a mixture of “art” based on experience and “science” based on analytic approaches. The technique for design and operation in the industrial world depend largely on the art form; the scientific approach is to be found in the studies.

The rotary dryer is the workhorse of the solids process industry and is to be found in such diverse plants as fertilizer production, pharmaceutical chemicals manufacture, lead or zinc concentrate production for smelting, cement manufacture, and many more industries.

**Dryer Process**

The dryer chosen for the purpose of simulation is rotary dryer. A rotary dryer consists of a revolving cylindrical shell, inclined slightly toward outlet. Wet feed enters from one end of the cylinder and dry material leaves from the other end. As the material passes through the dryer, it is lifted up by internal flights and showered in a stream of hot gases. The movement of solid within the dryer, is by cascade action kiln or rolling action. In other words, particle is lifted stationary in a flight from the bottom half of the drum to the upper half from where it cascades down depending on its angle of repose, and then it is moved forward by sliding downhill of rolling/kiln action under pressure from the particles coming behind.

In summary, there are three separate components making up the total movement of the particles down the drum :

1. Lifting up of the particles by flights.
2. Forward or backward movement due to drag on the particles from the drying gases.
3. Action between flights in the lower half of the drum.

The process is simulated using 'Shrinking Core Model' concept. This concept assumes that each granule consists of a shell and a core, which has original moisture content. The core shrinks towards centre as drying continues. The drying rate is determined depending on the relative rates of external mass transfer coefficient and effective diffusivity of water vapor-air in a porous medium. From above discussion it is clear that separate processes performed in the rotary dryer are:

1. The movement or dynamics of the particles as they progress by cascade and kiln action through the drum length.
2. Heat transfer from hot gases to the particles providing the latent heat of vaporization to the moisture with the particles.
3. Mass transfer of the moisture from within the particles to their surface and then to the hot gases in the drum.

In case of rotary dryers, the mechanism is usually described as being heat and mass transfer, although strictly speaking, they are consecutive processes with heat transfer preceding mass transfer. The process of drying described above appears to me simple but the same becomes complicated in case of rotary dryers because of complex motion of the solids through the drum. The cascade cycle maybe divided into 'cascading' and 'resting in flights' periods; in the cascading period the solids are subjected to the heat and mass -transfer, in the resting periods; both the processes are slowed down, possibly stopped altogether.

For the purpose of simulation, instead of considering entire drum as only zone and assuming it to be well stirred (which is not valid at all) the dryer is divided into

number of zones. Although in practice, any number of zones can be considered, for optimization (depending on the accuracy and the time), it is divided into four zones. The student can see the profiles of moisture content of air and solid and the temperatures of air and solid in each zone and get the feel of working of dryer.

### Significant Operating Variables.

The following variables are controlled for efficient operation of DRYER.

#### 1. Furnace Temperature :

To have fine control over temperature of air entering dryer, this is controlled by flow of dilution air to furnace. This sends a control to dilution air flow controller. If temperature increases flow decreases.

#### 2. Dryer Temperature :

It is controlled by adjusting flow of combustion air to furnace. This sends a control to combustion air flow controller. Higher the temperature, lower is the flow.

#### 3. Dilution Air :

This receives remote control from furnace temperature controller.

#### 4. Combustion Air :

This also receives remote control from dryer temperature controller.

### VIII. Experimental set up:

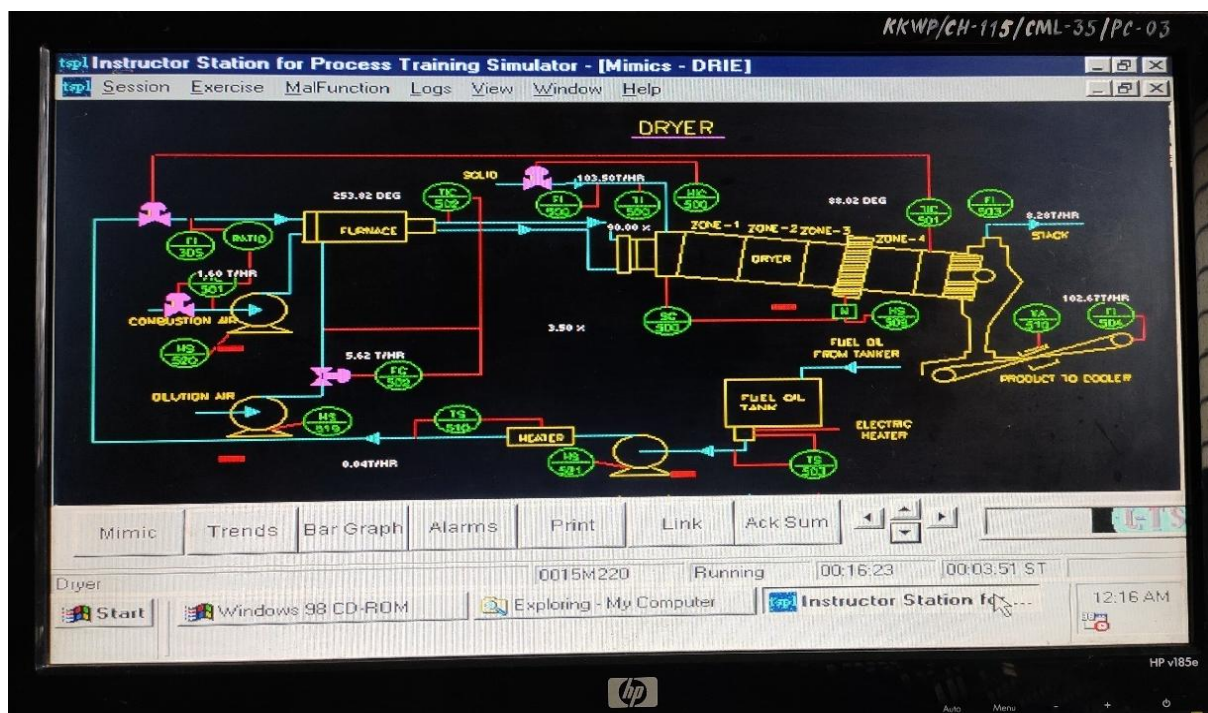


Fig. 1

**IX. Resources required**

Sl No.	Name of Resource	Suggested Broad Specification	Quantity
1	Triangle Simulation Private Limited or MSBTE Sponsored TSPL	As per standard specification Version 2017	1
2	Personal Computer	P-IV or higher version PC	1

**X. Precautions to be followed**

After starting the software, ensure that the link of Dryer is showing in the dialogue box.

**XI. Procedure****Design Start**

1. Set Student Loading Function as per the settings given in set up manual for design start.
2. Set Student Switches as per settings given in set up manual.
3. Set the controllers as per settings given in set up manual.
4. Adjust controller outputs manually as per set up manual.
5. You will get display of operating variables on the screen.
6. Check value of all the instructor functions whether they are as per up data. If not you can change it from keyboard.
7. Put all the controllers in AUTO mode and let the plant settle at design conditions.
8. After the system reaches to steady state, you can do all the exercises.

**Instructor Loading Function**

1. TAG READS : CALORIFIC VALUE OF FUEL  
RANGE : 8000 - 13000 KCAL/KG OF FUEL  
NORMAL : 10000 KCAL/KG OF FUEL  
FUNCTION : Sets the heating value of fuel.
2. TAG READS : FFAIR/FFUEL RATIO

- RANGE : 25 to 35
- NORMAL : 32
- FUNCTION : Sets heating efficiency of fuel.
3. TAG READS : HUMIDITY OF AIR IN
- RANGE : 0.02 - 0.04 kg moisture/kg dry air
- NORMAL : 0.034 kg moisture/kg dry air
- FUNCTION : Gives the moisture content of entering air.
4. TAG READS : SOLID MOISTURE CONTENT
- RANGE : 0.02 - 0.03 kg moisture/kg dry solid
- NORMAL : 0.0256
- FUNCTION : Specifies water content in entering solid.
5. TAG READS : SOLID TEMPERATURE IN
- RANGE : 80 - 100 C
- NORMAL : 92
- FUNCTION : Decides temp of solid going to dryer

## XII. Resources used

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					

## XIII. Actual procedure followed

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**XIV. Precautions followed**

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**XV. Observations and Calculations:****1 Transmitter Details**

Sr. No.	Tag Name	Units	Description	High Range	Low Range	PV
1	FC501	T/HR	FLOW OF COMB.AIR	5.00	0.00	
2	FC502	T/HR	FLOW OF DILUTION AIR	20.00	0.00	
3	TC502	DEG C	TEMP OF AIR INLET	300.00	0.538236	
4	TC501	DEG C	TEMP OF SOLID OUT	150.00	50.00	
5	FV500	T/HR	FLOW	150.00	0.00	
6	TI500	DEGC	TEMP OF SOLID IN	100.00	0.00	
7	SC500	RPM	DRYER	100.00	0.00	
8	FOIL	T/HR	FLOW OF OIL	0.5	0.00	
9	SIM_SPED		SIMULATION SPEED	100.00	0.00	
10	FI305	T/HR	FUEL OIL TO FURNACE	0.00	0.00	
11	HIC500	%	SOLIDS INLET	100.00	0.00	



**Instructor Function :-**

TAG NO	DESCRIPTION	COLD	DESIGN	RANGE
CV_FUEL	CAL. VALUE OF FUEL	0.00	10000	8000 - 13000
FA_FF_R	FFAIR/FFUEL RATIO	0.00	32	25 - 35
HUM_AIR	HUMIDITY OF AIR IN	0.00	0.034	0.025 - 0.04
SOL_MOIS	SOLID MOIST. CONT	0.00	0.0256	0.02 - 0.03
SOL_TEMP	SOLID TEMP. IN	0.00	92	80 – 100 C
LEN_DRY	LENGTH DRYER	0.00	24	20 – 30 C
DIA_DRY	DIAMETER DRYER	0.00	3.1	2.5 - 4
SPD_DRY	SPEED DRYER	0.00	3.5	2 – 4
INCL_DRY	INCLI. DRYER	0.00	2.5	2 – 3
RAD_GRAN	RADIUS GRANULE	0.00	0.002	0.001 - 0.003
PORO_PAR	POROS. PARTICLE	0.00	0.13	0.1 - 0.2
OVAL_HTC	OVERALL HTC	0.00	8.5	5 – 10
FLAG_UP	FLAG HOLD UP	ON	ON	ON / OFF

**Sample Exercise**

*Ex. 1 MOISTURE CONTENT OF SOLID IN.*

**ACTION** : Increase Moisture Content of Solid Feed.

**RESULT** : Flow of combustion air increases.

*DISCUSSION : More moisture content means more moisture will have to be removed. Others conditions remaining same, more moisture causes decrease in rate of drying. More moisture goes out with the solids. Temperature of air decreases so flow of combustion air increases.*

#### **XVI. Results**

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#### **XVII. Interpretation of results**

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#### **XVIII. Conclusions & Recommendation**

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#### **XIX. Practical related Questions**

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

*Define Dryer process with example.*

*b) How Temperature affects on Dryer?*

*c) List out the major components in Dryer Equipment with their function.*

*d) Search the Virtual Lab Simulation Practical for Dryer and write down the URL.*

*e) Exercise. 1 HUMIDITY OF AIR IN.*

*ACTION : Increase humidity of entering air.*

*RESULT :*

*DISCUSSION :*

*Exercise. 2 HEAT TRANSFER COEFFICIENT.*

*ACTION : Increase Heat Transfer Coefficient (HTC).*

*RESULT :*

*DISCUSSION :*

**[Space for Answers]**

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**XX. References / Suggestions for further Reading**

- <https://www.trianglesimulation.com/operator-training-simulator-for-engineering-institutes.html>
- [http://vmt-iitg.vlabs.ac.in/Rotary\\_dryer\(theory\).html](http://vmt-iitg.vlabs.ac.in/Rotary_dryer(theory).html)
- <https://www.sciencedirect.com/science/article/pii/S0032591013001459>
- <https://aiche.onlinelibrary.wiley.com/doi/abs/10.1002/aic.690320214>

**XXI. Assessment Scheme**

Performance indicators		Weightage
<b>Process related (15 Marks)</b>		<b>60%</b>
1	Preparation of experimental set up	20%
2	Setting and operation	20%
3	Safety measures, observation and recording	20%
<b>Product related (10 Marks)</b>		<b>40%</b>
4	Calculation and Interpretation of result	20%
5	Practical related question and submission of report	20%
<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

- 1.....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	

## **Practical No. 18: Use the process simulator to analyze the parameters of Absorber.**

### **I. Practical Significance**

Simulation software is used to study the effect on various process variables such as pressure, temperature, flow, level by introducing / activating malfunctions in the normal operation.

### **II. Relevant Program Outcomes (POs)**

**PO1. Basic knowledge:** Apply knowledge of basic mathematics, sciences and basic engineering to solve the Chemical Engineering problems

**PO2. Discipline knowledge:** Apply Chemical Engineering knowledge to solve industry based Chemical Engineering problems.

**PO3. Experiments and practice:** Plan to perform experiments and practices to use the results to solve technical problems related to Chemical Engineering.

### **III. Competency and Practical Skills**

**'Use chemical process plant equipment for mass-transfer operations safely.'**

- To control and study the effects of various parameters on Absorption process.

### **IV. Relevant Course Outcomes**

Use gas absorption operation and relevant equipment in chemical industries.

### **V. Practical Outcome**

Use the process simulator to analyze the parameters of Absorber

### **VI. Relevant Affective domain related Outcome(s)-**

1. Load the exercise and run the simulation.
2. Record the change in values of process variables with introduction of malfunction

### **VII. Minimum Theoretical Background**

#### **Process**

Gas absorption is an operation in which a gas mixture is contacted with a liquid for the purposes of preferentially dissolving one or more components of the gas and to provide a solution of them in the liquid. When mass transfer occurs in the opposite direction, i.e. from the liquid to the gas, the operation is called desorption, or stripping. The principles of both absorption and desorption are basically the same.

Exhaust gases from the reactor and granulator containing ammonia are scrubbed with phosphoric acid. Co-current void tower is used to dissolve the gas in the liquid (phosphoric acid). The scrubber liquid is re-circulated via an integral sump and pump.

A constant bleed of liquor is fed to the ammonia scrubber under level control.

The gases at the ammonia scrubber inlet are maintained below 90°C by a constant bleed of cold air or water injection into the hot gases. Specific gravity in ammonia scrubber is maintained below 1.6 by addition of water and the liquor bleed is fed to the reactor under level control.

### VIII. Experimental set up :

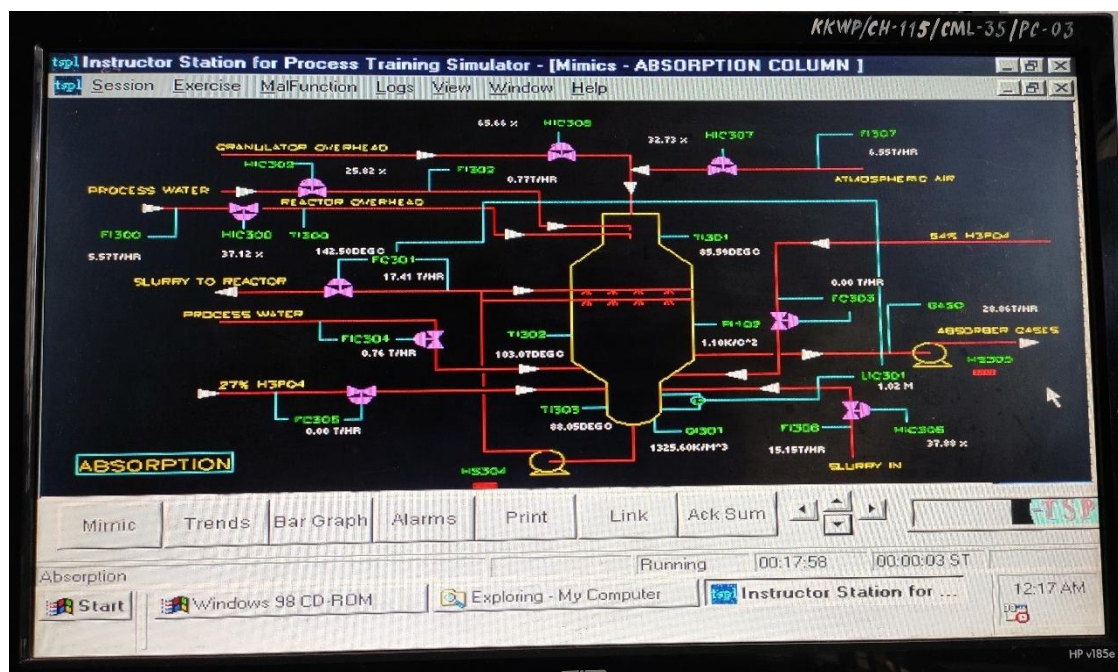


Fig. 1

### IX. Resources required

Sl No.	Name of Resource	Suggested Broad Specification	Quantity
1	Triangle Simulation Private Limited or MSBTE Sponsored TSPL	As per standard specification Version 2017	1
2	Personal Computer	P-IV or higher version PC	1

### X. Precautions to be followed

After starting the software, ensure that the link of Absorption is showing in the dialogue box.

### XI. Procedure DESIGN START

1. Set Student Loading Function as per the settings given in set up manual for design start.

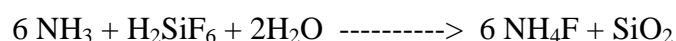
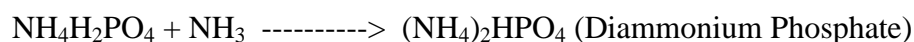
2. Set Student Switches as per settings given in set up manual.
3. Set the controllers as per settings given in set up manual.
4. Adjust controller outputs manually as per set up manual.
5. You will get display of operating variables on the screen.
6. Check value of all the instructor functions whether they are as per up data. If not you can change it from keyboard.
7. Put all the controllers in AUTO mode and let the plant settle at design conditions.
8. After the system reaches to steady state, you can do all the exercises.

### **COLD START**

1. Start the flow of slurry into the reactor by opening FIC-206. Simultaneously start the flow of ammonia into the reactor by opening HIC-203 and the flow of 54% P<sub>2</sub>O<sub>5</sub> by opening HIC-204. FC208 should be opened to allow flow of H<sub>2</sub>SO<sub>4</sub> into the reactor.
2. Level must be maintained at 5.3, which is indicated by a level indicator LI203.
3. Density also is to be maintained at design value (it is measured by DI-203).
4. Slurry in the reactor is mixed thoroughly with NH<sub>3</sub>, 54% P<sub>2</sub>O<sub>5</sub> and H<sub>2</sub>SO<sub>4</sub> due to constant stirring in the reactor. As the mixing approaches 100% reaction starts up.

("In Simulator, it is assumed that 100% mixing is approached without stirring"). As the reaction proceeds temperature in the reactor goes up (Exothermic reactions) when it crosses its upper limit i.e. 110 C, TIC-202 (Temp. controller) opens the valve in water inlet line allowing the water to flow into the reactor to cool down the reactants. While cooling the reactants, water itself gets heated up and gets vaporized and is ejected out.

5. Reactions that take place in the reactor are as follows:



6. Slurry containing MAP, DAP, DAS and other products, goes out through FC207 to the granulator.

**Significant Operating Variables.**

The following variables are controlled for efficient operation of Absorption Column:

Temperature: The gases at the ammonia scrubber inlet are maintained below 90°C by a constant bleed of cold air or water injection into the hot gases.

Specific gravity: Specific gravity in ammonia scrubber is maintained below 1.6 by addition of water and the liquor bleed is fed to the reactor under level control.

**XII. Resources used**

Sl No.	Name of Resource	Suggested Broad Specification		Quantity	Remarks (If any)
		Make	Details		
1					
2					

**XIII. Actual procedure followed**

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**XIV. Precautions followed**

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**XV. Observations and Calculations:**  
**Transmitter Details:**

Instrument No.	UNITS	Description	Hi Range	Lo Range	PV
TC-101	DEG C	COL TOP TEMP	150.000	50.000	
TC-103	DEG C	COL BTM TEMP	200.000	100.00	
PC-102	Kg/cm <sup>2</sup>	COL TOP PRESS	2.000	0.00	



LC-102	m	REFLUX DRM_LV	00.000	0.000	
LC-101	m	COL_BTM LVL	100.000	0.000	
FRC-26	Kg/hr	COL RX	10.000	0.000	
HC104	%	VENT	100.000	0.000	
HC105	%	VAPOR BYPASS	100.000	0.000	
HC107	%	RX_DRM DRAIN	100.000	0.000	
HC106	%	COL_BTM DRAIN	100.000	0.000	
FI-102	Kg/s	STM TO REBOILER	2.000	0.000	
FI-201	Kg/s	FEED RATE	15.000	0.00	
FI-204	Kg/s	REFLUX FLOW	0.000	0.00	
FI-202	Kg/s	BTM PRODUCT	10.000	0.000	
FI-207	Kg/s	OFF GAS	15.000	0.000	
FI-206	Kg/s	LIQUID COND	15.000	0.000	
FI-107	Kg/s	OFF GAS	2.000	0.000	
FI-103	Kg/s	CW TO COND	225.000	0.000	
TI-103	DEG C	CW OUTLET	100.000	0.000	
FI-203	Kg/s	TOP PRODUCT	5.00	0.000	
XI-201	W/W	FEED COMP	100.000	0.000	
XR-202	W/W	BTM COMP	30.000	0.000	
XR-203	W/W	TOP COMP	100.000	0.000	
PI-102	KG/CM2	O/H PRES	2.000	0.000	
TI-001	KG/CM/2	O/H PRES	2.000	0.000	
TI-002	DEG C	TRAY-2 TEMP	200.000	0.000	
TI-003	DEG C	TRAY-3 TEMP	200.000	0.000	
TI-004	DEG C	TRAY-4 TEMP	200.000	0.000	
TI-005	DEG C	TRAY-5 TEMP	200.000	0.000	
FI-205	Kg/s	VAPOR FLOW	20.000	0.000	
HC-101	%	FEED FLOW	100.000	0.000	
CYC_TIM	CYCLE	115.000	0.000	115.000	

%	TIME				
PRES_TAU		TOW FOR PRESS	115.000	0.000	
LIQ_TAU		TIME LAG	0.000	0.000	

### Sample Exercise

Ex .1: Flow of 54%  $P_2O_5$ .

ACTION : Close HIC 204.

RESULT : Reaction will not take place.

DISCUSSION : Since one of the reactants viz. 54%  $P_2O_5$  is absent in the reactor the reaction will not take place. So neither there will be temperature rise nor there will be any sudden pressure rise.

### XVI. Results

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### XVII. Interpretation of results

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### XVIII. Conclusions & Recommendation

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### XIX. Practical related Questions

*Note: Below given are few sample questions for reference. Teachers must design more such questions so as to ensure the achievement of identified CO.*

- Define Absorption process with example.*
- How Solubility affects on absorption?*

- c. *List out the major components in Absorption Equipment with their function.*

EX. 1 : Flow Of Ammonia

ACTION : Close HIC-203.

RESULT :

DISCUSSION :

EX.2: Flow of slurry in

ACTION : Close FIC-206.

RESULT :

DISCUSSION :

EX.3: Flow of water

ACTION : Close the valve in water input line.

RESULT :

DISCUSSION :

**[Space for Answers]**

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**XX. References / Suggestions for further Reading**

- <https://www.trianglesimulation.com/operator-training-simulator-for-engineering-institutes.html>
- <http://vlabs.iitb.ac.in/vlab/chemical/exp2/index.html>
- <https://www.sciencedirect.com/science/article/pii/S0009250907006768>
- <https://kx.lumerical.com/t/how-to-properly-set-up-an-absorption-simulation/3690/12>
- <https://www.ncbi.nlm.nih.gov/pubmed/23007435>

**XXI. Assessment Scheme**

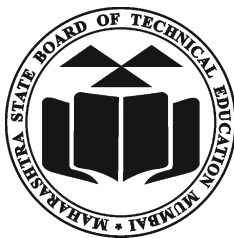
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<b>Total (25 Marks)</b>		<b>100 %</b>

***Names of Student Team Members***

- 1.....
- 2.....
- 3.....
- 4.....

Marks Obtained			Dated signature of Teacher
Process Related(15)	Product Related(10)	Total (25)	

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