# PHYSICAL AND ANALYTICAL CHEMISTRY

**NAME :**

**REG. NO. :**

**DEGREE : B. Tech.**

**BRANCH : CHEMICAL ENGINEERING**

**YEAR : I**

**SEMESTER : II**

**SUBJECT CODE : 21CHC101J**

**SUBJECT TITLE : PHYSICAL AND ANALYTICAL CHEMISTRY**

**ACADEMIC YEAR : 2023-2024 (EVEN)**


**DEPARTMENT OF CHEMICAL ENGINEERING**

**COLLEGE OF ENGINEERING AND TECHNOLOGY**

**SRM INSTITUTE OF SCIENCE AND TECHNOLOGY**

**KATTANKULATHUR – 603 203**

**CHENGALPATTU DISTRICT**

**TAMILNADU, INDIA**

**SRM INSTITUTE OF SCIENCE AND TECHNOLOGY**
(Under Section 3 of UGC Act, 1956)
**KATTANKULATHUR - 603 203**
CHENGALPATTU DISTRICT

# BONAFIDE CERTIFICATE

**Register Number: _____**

*Certified to be the bonafide record of work done by _____*

*of **I Year B. Tech. Chemical Engineering** Degree course in the Practical **21CHC101J - PHYSICAL AND ANALYTICAL CHEMISTRY** in SRM Institute of Science and Technology, Kattankulathur during the Even semester of the academic year 2023-2024 .*

Lab In charge

**Date:** **Head of the Department**

**INDEX**

**Name :** **Register No.:**

| Expt No. | Date | Title of the Experiment | Marks | Signature |
|---|---|---|---|---|
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
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| 12 | | | | |

**Department of Chemical Engineering**
**College of Engineering and Technology**
**SRM Institute of Science and Technology**

## B. Tech. in Chemical Engineering

### Vision of the Department

*To utilize Chemical Engineering and Technology and ensure overall socio-economic growth, welfare, and progress of Indian society and the World-at-large by supporting Academia, Industries through Research and Development, Consultancy and graduating high-quality Chemical Engineers*

### Mission of the Department

| | |
|---|---|
| Mission Stmt - 1 | *To facilitate high quality education, well grounded in the fundamental and applied areas of engineering necessary for learners to contribute effectively to chemical and allied industries* |
| Mission Stmt - 2 | *To educate, prepare, inspire and mentor learners with the technical and professional skill-set necessary to excel as professionals, grow in their careers and contribute to chemical engineering science and technology* |
| Mission Stmt - 3 | *To inculcate social-responsibility in learners and train them to contribute effectively to science and society* |

### Program Educational Objectives (PEO)

Within a few years of graduation, the students of the program will be able to attain the following:

| | |
|---|---|
| PEO - 1 | *Utilizing their strong fundamental knowledge from the program be able to solve technical problems and contribute to chemical and allied industries* |
| PEO - 2 | *Pursuing higher studies and/or continuously upgrading their skill-sets with technological advances leading to personal and professional growth and successful careers* |
| PEO - 3 | *Establishing themselves with successful careers in industry, academia and/or as entrepreneurs that will enable them to address social, economic and environmental challenges and contribute to science and society* |

### Mission of the Department to Program Educational Objectives (PEO) Mapping

| | Mission Stmt. - 1 | Mission Stmt. - 2 | Mission Stmt. - 3 |
|---|---|---|---|
| PEO - 1 | 3 | 2 | 1 |
| PEO - 2 | 2 | 3 | 1 |
| PEO - 3 | 2 | 1 | 3 |

3 – High Correlation, 2 – Medium Correlation, 1 – Low Correlation

**Mapping Program Educational Objectives (PEO) to Program Learning Outcomes (PLO)**

| | Program Outcomes (PO) | | | | | | | | | | | | Program Specific Outcomes (PSO) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Engineering Knowledge | Problem Analysis | Design/development of solutions | Conduct investigations of com- | Modern Tool Usage | The engineer and society | Environment & Sustainability | Ethics | Individual & Team Work | Communication | Project Mgt. & Finance | Life Long Learning | PSO - 1 | PSO - 2 | PSO - 3 |
| PEO - 1 | *3* | *3* | *3* | *3* | *2* | | | | *2* | *2* | *2* | | *3* | *3* | *2* |
| PEO - 2 | *3* | *3* | *3* | *3* | *2* | | *2* | *2* | | | | *2* | *3* | *2* | *2* |
| PEO - 3 | *2* | *2* | *2* | *2* | | *3* | *2* | *2* | *3* | *3* | *3* | *2* | *3* | *2* | *3* |

3 – High Correlation, 2 – Medium Correlation, 1 – Low Correlation , PSO – Program Specific Outcomes (PSO)

**PSO – Program Specific Outcomes (PSO)**

| | |
|---|---|
| PSO - 1 | *Ability to understand and differentiate processes* |
| PSO - 2 | *Apply the fundamentals to perform computations related to synthesis, design and analysis of chemical processes.* |
| PSO - 3 | *Analyze the process plants from Energy, Environment and Safety related aspects* |

# COURSE ARTICULATION MATRIX

## CO – PO MAPPING

**SYLLABUS**

| Course Code | 21CHC101J | Course Name | PHYSICAL AND ANALYTICAL CHEMISTRY | Course Category | C | Professional Core | L | T | P | C |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | 2 | 0 | 2 | 3 |

| Pre-requisite Courses | *Nil* | Co-requisite Courses | *Nil* | Progressive Courses | *Nil* |
|---|---|---|---|---|---|
| Course Offering Department | *Chemical Engineering* | Data Book / Codes/Standards | *Nil* | | |

| Course Learning Rationale (CLR): | *The purpose of learning this course is to:* |
|---|---|
| CLR-1 : | Describe the ideal and non-ideal behavior of liquids; learn colligative properties and their applications |
| CLR-2 : | Explain the concepts of chemical equilibrium and the effect of various factors on equilibrium constant |
| CLR-3 : | Compare the difference in behavior of different states of matter essential for separation operations |
| CLR-4 : | Describe the properties and applications of colloids; Understand the kinetics of photochemical reactions |
| CLR-5 : | Explain the principles of analytical instruments along with their limitations |

| Course Outcomes (CO): | *At the end of this course, learners will be able to:* | Program Outcomes (PO) | | | | | | | | | | | | PSO | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 1 | 2 | 3 |
| | | Engineering Knowledge | Problem Analysis | Design/development of solutions | Conduct investigations of complex problems | Modern Tool Usage | The engineer and society | Environment & Sustainability | Ethics | Individual & Team Work | Communication | Project Mgt. & Finance | Life Long Learning | PSO-1 | PSO-2 | PSO-3 |
| CO-1 : | Analyze ideal and non-ideal behavior of fluids and define the colligative properties | 3 | 2 | - | - | - | - | - | - | - | - | - | - | 1 | - | - |
| CO-2 : | Evaluate the significance of Gibbs' free energy and equilibrium constants | 3 | 2 | - | - | - | - | - | - | - | - | - | - | 2 | - | - |
| CO-3 : | Apply Gibbs' phase rule and draw the phase diagram of one- and three-component systems | 3 | - | 1 | - | - | - | - | - | - | - | - | - | 2 | - | - |
| CO-4 : | Analyze the properties of colloids and photochemical reactions | 2 | - | - | - | 3 | - | - | - | - | - | - | - | - | 1 | - |
| CO-5 : | Implement the appropriate analytical technique for various types of chemical compounds | 2 | - | - | 3 | - | - | - | - | - | - | - | - | - | 2 | - |

**Unit-1: PROPERTIES OF SOLUTIONS** **6 Hours**

Introduction to solutions, Raoult's law -Vapour pressures of ideal and non-ideal solutions - Deviations from ideality of Type I, Type II and Type III solutions - Completely miscible binary solutions: Vapor pressure-Composition and Boiling point-Composition curves of Type I, Type II, and Type III solutions - Fractional distillation of binary liquid systems, The Lever rule - Distillation of immiscible liquids, Steam ditillation - Partially miscible liquids, Critical solution temperature, Phenol-water system, Solutions of gases in liquids: Factors influencing solubility of a gas, Henry's law - Colligative Properties - Relative lowering of vapour pressure, Osmosis and osmotic pressure, Elevation in boiling point, Depression in freezing point, Determination of molecular weight from colligative properties, Effect of association/dissociation on colligative properties

**Unit-2: CHEMICAL EQUILIBRIUM** **6 Hours**

Introduction to Chemical equilibria - Gibbs' free energy and Chemical potential - Free energy of a spontaneous reaction - Law of mass action - Law of chemical equilibrium - Thermodynamic derivation of the law of chemical equilibrium - Significance of equilibrium constant - Equilibrium constants :$K_p$, $K_c$,and, $K_x$ - Relationship between $K_p$, $K_c$,and, $K_x$ - Temperature dependence of Equilibrium constant - Van't Hoff Equation - Pressure dependence of equilibrium constants - Le Chatelier's Principle - Effect of change in concentration, temperature, and pressure - Le Chatelier's principle and physical equilibria

**Unit-3: PHASE EQUILIBRIUM** **6 Hours**

Introduction to Phase equilibria - Component, phase and degrees of freedom - Conditions for equilibrium between phases - Derivation of Gibbs' phase rule - Representation of one component systems using phase diagrams - One component systems - water system, $CO_2$ system, sulphur system - Three component systems - Triangular phase diagram - Three component system: acetic

| | |
|---|---|
| | acid-chloroform-water system, Two salts and water system, The Nernst distribution law and distribution co-efficient, Conditions for the validity of the distribution law - Association of the solute in one of the solvents - Dissociation of the solute in one of the solvents - Applications of Nernst distribution law |
| **Unit-4: COLLOIDS AND PHOTOCHEMISTRY** | **6 Hours** |
| | Introduction to Colloids - General properties of colloids: Tyndall effect and Brownian movement - Electrical properties of colloids: electrical double layer, Zeta potential - Electrokinetic properties of colloids: electrophoresis and electro-osmosis - Gels and emulsions - Applications of colloids - Introduction to Photochemistry - Laws of photochemistry - Quantum yield - Photochemical reactions - Photochemical rate law - Determination of quantum yields - Kinetics of hydrogen-chlorine reaction: Mechanism and Derivation - Kinetics of hydrogen-bromine reaction: Mechanism and Derivation |
| **Unit-5: INSTRUMENTAL METHODS OF ANALYSIS** | **6 Hours** |
| | Instrumental Methods of Analysis - Accuracy, precision, common errors (system/manual) - Calibration curves - Classification of instrumental methods - spectroscopy, electrochemical and chromatography - Electro-magnetic (EM) spectrum, Interaction of EM radiation with matter - Generalities of optical methods (light source/ monochromator / sample introduction / detector / signal generator) - Principle, Instrumentation, Working, Applications, and Limitations of analytical techniques - UV –Vis spectroscopy - Infra-red spectroscopy - Atomic absorption spectroscopy - Chromatographic techniques: General principle - Column chromatography - Paper chromatography - Thin layer chromatography - Gas chromatography - High Performance Liquid Chromatography |

| | |
|---|---|
| **Practice** | **30 Hours** |
| | Practice 1: Determine the critical solution temperature (CST) of phenol-water system.<br>Practice 2: Determine the molecular weight of an unknown compound by Rast method.<br>Practice 3: Determine the strength of the given acid mixture by conductometric titration.<br>Practice 4: Determine the rate constant of acid catalyzed hydrolysis of an ester.<br>Practice 5: Phase diagram of three component system.<br>Practice 6: Determine the partition co-efficient of benzoic acid between benzene and water.<br>Practice 7: Estimation of sulphate by nephelometry.<br>Practice 8: Determine the amount of reducing sugar by DNS method.<br>Practice 9: Estimate amount of iron present in a sample using UV-Vis spectrophotometer.<br>Practice 10: Determine the amount of fatty acid methyl ester using gas chromatography.<br>Practice 11: Estimate the amount of Aspirin in the given tablet using pH meter.<br>Practice 12: Determine the amount of copper present in the given sample using AAS.<br>Practice 13: Determination of water quality parameters using Water analysis kit.<br>Practice 14: Estimate the percentage of manganese present in the given sample of ore.<br>Practice 15: Estimate the Saponification equivalent of the given oil. |

| | | |
|---|---|---|
| Learning Resources | *1.B. R. Puri, L. R. Sharma and Madan S. Pathania, Principles of Physical Chemistry, Vishal Publishing Co., 47th Ed, 2015*<br>*2. Arun Bahl, B. S. Bahl, and G. D. Tuli, Essentials of Physical Chemistry, S. Chand & Company Ltd., 2009.* | *3. Douglas A. Skoog , F. James Holler, and Timothy A. Nieman. "Principles of Instrumental Analysis, Thomson Learning Inc., Toronto, 1998* |

| | Bloom's Level of Thinking | Continuous Learning Assessment (CLA) - By the Course Faculty | | | | By The CoE | |
|---|---|---|---|---|---|---|---|
| | | **Formative** CLA-I Average of unit test (40%) | | **Life Long Learning** CLA-II- Practice (20%) | | **Summative** Final Examination (40% weightage) | |
| | | Theory | Practice | Theory | Practice | Theory | Practice |
| Level 1 | Remember | 20 | - | - | 20 | 20 | - |
| Level 2 | Understand | 20 | - | - | 20 | 20 | - |
| Level 3 | Apply | 30 | - | - | 20 | 30 | - |
| Level 4 | Analyze | 30 | - | - | 20 | 30 | - |
| Level 5 | Evaluate | - | - | - | 10 | - | - |
| Level 6 | Create | - | - | - | 10 | - | - |
| | Total | 100 % | | 100 % | | 100 % | |

| Course Designers | | |
|---|---|---|
| Experts from Industry | Experts from Higher Technical Institutions | Internal Experts |
| 1. Mr. A. Subramaniam, PESCO Beam Environmental Solutions Pvt. Ltd | 1. Dr. Lima Rose Miranda, Anna University | 1. Dr. K. Deepa, SRMIST |
| 2. Mr. S. Stalin, Course Director, Chem Skill Development Centre | 2. Dr. N. Anantharaman, Former Professor, NIT Trichy | 2. Dr. M. P. Rajesh, SRMIST |

## LIST OF EXPERIMENTS

| Exp. No. | Title of the Experiment |
|---|---|
| 1 | Determination of critical solution temperature for phenol - water system |
| 2 | Determination of molecular weight by Rast method |
| 3 | Conductometric titration |
| 4 | Determination of the rate constant of acid catalysed hydrolysis of an ester |
| 5 | Phase diagram of three component system |
| 6 | Partition coefficient of benzoic acid between benzene and water |
| 7 | Estimation of sulphate by nephelometry |
| 8 | Estimation of reducing sugar by dinitro salicylic acid (DNS) method |
| 9 | Estimation of iron in the given sample by using UV- visible spectrophotom-eter |
| 10 | Determination of fatty acid methyl ester using gas chromatography |
| 11 | Estimation of aspirin drug in tablets using pH meter |
| 12 | Estimation of manganese in the given sample of ore |

## MAPPING OF COURSE OUTCOMES WITH PROGRAM OUTCOMES

| S. No. | Course Outcomes (COs) | Program Outcomes (POs) | Experiment Details |
|---|---|---|---|
| 1 | Analyze ideal and non-ideal behavior of fluids and define the colligative properties | PO - 1: Engineering Knowledge<br>PO - 2: Problem Analysis | Exp. 1 - Determination of critical solution temperature for phenol - water system<br><br>Exp. 2 - Determination of molecular weight by Rast method |
| 2 | Evaluate the significance of Gibbs' free energy and equilibrium constants | PO - 1: Engineering Knowledge<br>PO - 2: Problem Analysis | Exp. 3 - Conductometric titration<br><br>Exp. 4 - Determination of the rate constant of acid catalysed hydrolysis of an ester |
| 3 | Apply Gibbs' phase rule and draw the phase diagram of one- and three-component systems | PO - 1: Engineering Knowledge<br>PO - 3: Design & Development | Exp. 5 - Phase diagram of three component system<br><br>Exp. 6 - Partition coefficient of benzoic acid between benzene and water |
| 4 | Analyze the properties of colloids and photochemical reactions | PO - 1 : Engineering Knowledge<br>PO - 5: Modern Tool Usage | Exp. 7 - Estimation of sulphate by nephelometry<br><br>Exp. 11 - Estimation of aspirin drug in tablets using pH meter |
| 5 | Implement the appropriate analytical technique for various types of chemical compounds | PO - 1 : Engineering Knowledge<br>PO - 4: Analysis, Design, Research | Exp. 8 - Estimation of reducing sugar by dinitro salicylic acid (DNS) method<br><br>Exp. 9 - Estimation of iron in the given sample by using UV-visible spectrophotometer<br><br>Exp. 10 - Determination of fatty acid methyl ester using gas chromatography<br><br>Exp. 12 - Estimation of manganese in the given sample of ore |

## Scheme of Evaluation

## 21CHC101J - Physical and Analytical Chemistry

| S. No. | Assessment Tool | Weight-age | Component | Marks |
|---|---|---|---|---|
| 1 | Total In Semester Assessment | 15 % | CLA 2A | 7.5 |
| | | | CLA 2B | 7.5 |
| 2 | End Semester Examination | Nil | Nil | Nil |
| 3 | **Total** | **15 %** | **Total** | **15** |

**RUBRICS**

| Course Outcomes | Allocated Marks | High (4 – 5) | Medium (2 – 3) | Low (0 – 1) |
|---|---|---|---|---|
| Pre-Lab | 5 | Thorough study and all the questions have been answered correctly | Adequate study and more than half of the questions have been answered correctly | No understanding of any experimental concepts |
| Post-Lab | 5 | Objectives of experiment are fully grasped; Developed theoretical understanding of concept | Objectives of experiment are fully grasped | No idea is built from experiment |
| Experiment Performance (Observation and Result Analysis) | 5 | Proper data is collected from experiment | Inconsistency in data | Wrong Observation made |
| | 5 | Accurate and reproducible results and analysis of data | Accurate and reproducible results and absence of analysis of data | No results calculated; No idea has been built from data and results |

## SAFETY PRECAUTIONS

### Lab Safety Do's and Don'ts for Students

**Use this handy checklist to acquaint students with safety dos and don'ts in the laboratory.**

### Conduct

- Never run in the laboratory.
- The use of personal audio or video equipment is prohibited in the laboratory.
- Do not engage in practical jokes or boisterous conduct in the laboratory.
- Do not sit on laboratory benches.

### General Work Procedure

- Know emergency procedures.
- Never work in the laboratory without the supervision of an instructor.
- Immediately report any spills, accidents, or injuries to your instructor.
- Never leave experiments while in progress.
- Do not remove any equipment or chemicals from the laboratory.
- Store coats, bags, and other personal items in designated areas.
- Keep the floor clear of all objects (e.g., ice, small objects, spilled liquids).

### Housekeeping

- Keep work area neat and free of any unnecessary objects.
- Thoroughly clean your laboratory work space at the end of the laboratory session.
- Do not block the sink drains with debris.
- Never block access to exits or emergency equipment.
- Properly dispose of broken glassware and other sharp objects (e.g., syringe needles)immediately in designated containers.
- Properly dispose of weigh boats, gloves, filter paper, and paper towels in thelaboratory.

### Apparel in the Laboratory

- Always wear personal protective equipment like goggles, gloves when handling hazardous materials.
- Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
- Wear shoes that adequately cover the whole foot.
- Secure long hair and loose clothing (especially loose long sleeves, neck ties, orscarves).

### Emergency Procedure

- Know the location of all the exits in the laboratory and building.
- Know the location of the emergency phone.
- Know the location of and know how to operate the following:Fire extinguishers, Eye washes, First aid kits

### Chemical Handling

- Check the label to verify it is the correct substance before using it.
- Always use a spatula to remove a solid reagent from a container.

- Do not directly touch any chemical with your hands.
- Hold containers away from the body when transferring a chemical or solution fromone container to another.
- Use a hot water bath to heat flammable liquids. Never heat directly with a flame.
- Clean up all spills properly and promptly.
- Dispose off chemicals as instructed.

**EXPERIMENT NO.** **DATE:**

# DETERMINATION OF CRITICAL SOLUTION TEMPERATURE FOR PHENOL - WATER SYSTEM

## AIM

To determine the critical solution temperature for phenol-water system and to find out the percentage of phenol in the given sample

## APPARATUS

Burette, boiling tube, thermometer, water bath, etc.

## PRINCIPLE

Phenol and water are partially miscible at ordinary temperatures. Therefore, on shaking these two liquids with each other, two saturated solutions of different compositions, one of phenol in water and the other of water in phenol, are obtained. Such solutions of different compositions co-existing with one another are termed 'conjugate solutions'.

The mutual solubility of phenol and water increases with rise in temperature and, therefore, the concentration of phenol in water as well as that of water in phenol goes on increasing with rise of temperature and ultimately at a certain temperature the two conjugate solutions change into one homogeneous solution. This temperature is known as consolute temperature or critical solution temperature. Above the consolute temperature, the two liquids become miscible with each other in all proportions.

## PROCEDURE

Take a clean dry boiling tube, fit it with a thermometer and a stirrer, and clamp it vertically to a clamp stand. Add 5 ml of phenol in the boiling tube using a burette. Add 3.0 ml of distilled water to it and mix well. Dip the boiling tube into a water bath and slowly heat the mixture with constant stirring. Note the temperature at which the turbidity of the mixture just disappears to become a clear solution. Remove the boiling tube from the heat and cool the mixture slowly with constant stirring, and note the temperature at which the turbidity reappears. The mean temperature is taken as the miscibility temperature. Add another 2.0 ml aliquot of water to

**OBSERVATION**

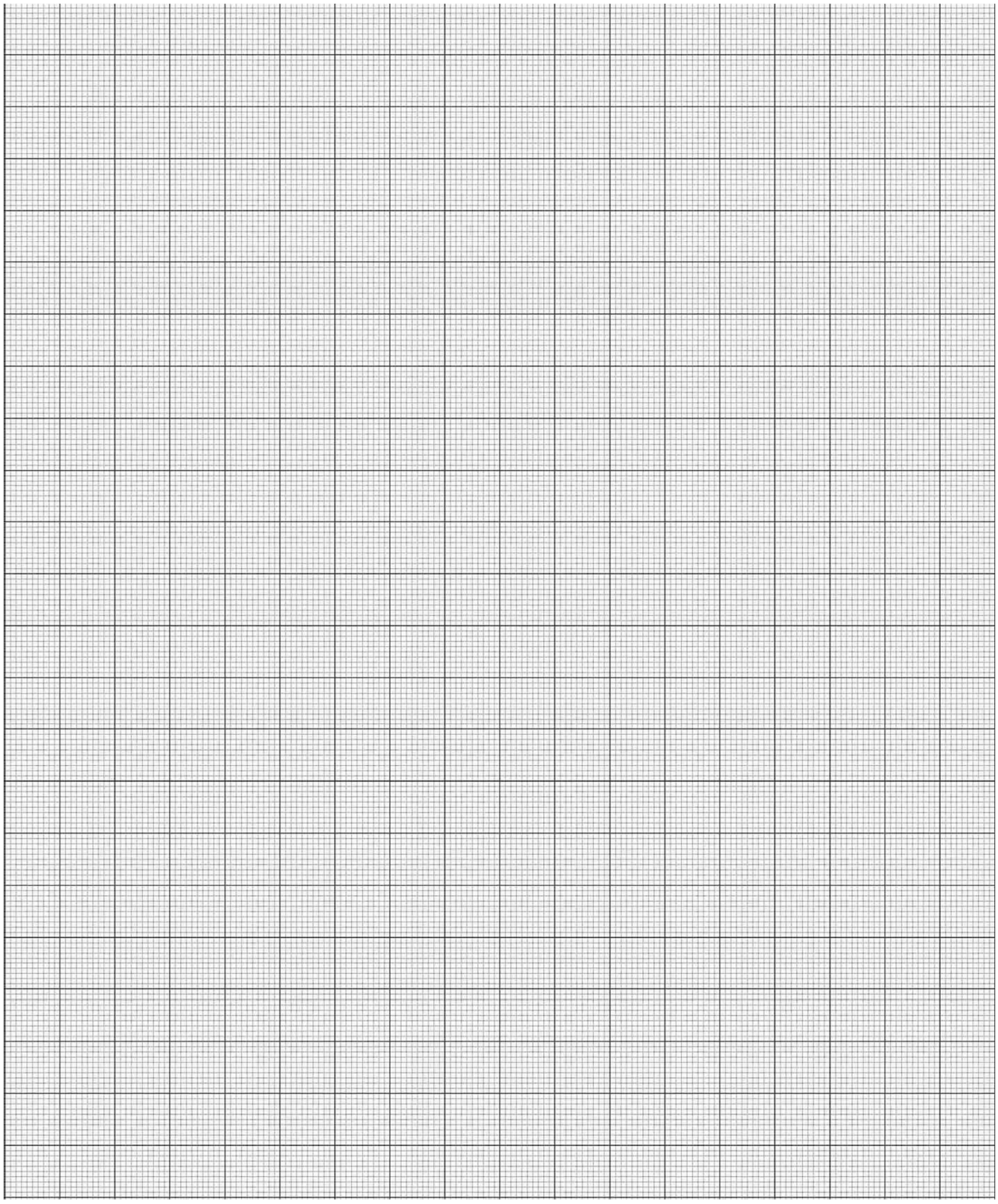
| S. No. | Vol. of phenol taken (ml) | Vol.($V_1$) of water added (ml) | Vol. percentage of phenol $= \left(\frac{5}{5+V_1}\right) x100$ | Temp. of disappear--ance of turbidity ( °C) | Temp. of appearance of turbidity (°C) | Mean-Temp. (°C) |
|---|---|---|---|---|---|---|
| 1 | 5 | 3 | | | | |
| 2 | 5 | 5 | | | | |
| 3 | 5 | 7 | | | | |
| 4 | 5 | 9 | | | | |
| 5 | 5 | 11 | | | | |
| 6 | 5 | 13 | | | | |
| 7 | 5 | 15 | | | | |
| 8 | 5 | 17 | | | | |
| 9 | 5 | 19 | | | | |
| 10 | 5 | 21 | | | | |
| 11 | 5 | 23 | | | | |
| 12 | 5 | 25 | | | | |
| 13 | 5 | 27 | | | | |
| 14 | 5 | 29 | | | | |
| 15 | 5 | 31 | | | | |
| 16 | unknown sample | | | | | |

the mixture and repeat the above procedure after every addition of 2 ml increments of water up to a final volume of about 36 ml. Obtain the miscibility temperatures for all these mixtures made up of different proportions of phenol and water.

**Scale**

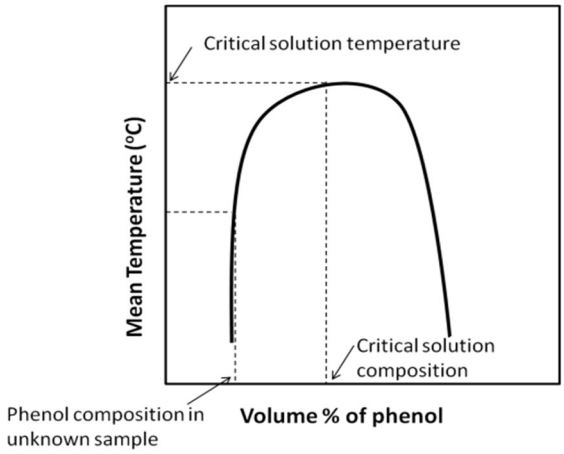
**In x-axis, 1 cm =**

**In y-axis, 1 cm =**



## MODEL GRAPH

Plot a graph between mean temperature and volume percentage of phenol. The y-axis co-ordinate of the maximum point on the graph corresponds to the critical solution temperature.




| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Define partially miscible systems.<br>2. Define critical solution temperature.<br>3. How does temperature affect the solubility of binary liquid systems? | 1. List the different types of partially miscible systems.<br>2. Give the significance of critical solution temperature.<br>3. How does temperature affect the solubility of phenol-water system? |

## RESULT

- The CST of phenol-water system was found to be _____ ºC.
- The critical solution composition was found to be _____% by volume of phenol.
- The percentage of phenol in the given sample was found to be _____% by volume.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT NO.** **DATE:**

# DETERMINATION OF MOLECULAR WEIGHT BY RAST METHOD

## AIM

a) To determine the molal depression constant of naphthalene using diphenyl-amine as solute

b) To find out the molecular weight of the given compound using the value of molal depression constant

## APPARATUS

Hot water bath, glass tube, thermometer, weighing bottles, etc.

## PRINCIPLE

The depression in freezing point of a solvent is a colligative property of dilute solution of a non-volatile solute in a volatile solvent. A colligative property is defined as that property which depends upon the composition of the solute (number of particles of solute) and not on the nature of solute.

When a non-volatile solute is dissolved in a volatile solvent, there occurs a depression in the freezing point of the solvent. This depression in freezing point of the solvent is given by the expression

$$\Delta T_f = k_f \text{ x } m$$

where $\Delta T_f$ = depression in freezing point

$k_f$ = molal depression constant
m = molality

Molality is given by

$$m = \frac{w_2}{M_2 \times w_1} \text{ x } 1000$$

where $w_2$ = weight of solute in grams
$w_1$ = weight of solvent in grams
$M_2$ = molal mass of solute

**OBSERVATION**

## DETERMINATION OF MOLECULAR WEIGHT BY RAST METHOD

### (A) MOLAL DEPRESSION CONSTANT

| Sl. No | Weight of Naphthalene $w_1$ (g) | Weight of Di-phenylamine $w_2$ (g) | Melting point , $T_m$ (°C ) | Freezing point, $T_f$ (°C ) | Mean Freezing point , T = $(T_m+T_f)/2$ (°C ) | $k_f = \frac{169.22 \times w_1 \times \Delta T_f}{w_2 \times 1000}$ |
|---|---|---|---|---|---|---|
| 1 | 2.0000 | 0.0000 | | | | --- |
| 2 | 2.0000 | 0.2000 | | | | |
| 3 | 2.0000 | 0.4000 | | | | |

Mean $k_f$ = _____ g °C $mol^{-1}$

### (B) MOLECULAR WEIGHT OF UNKNOWN COMPOUND

| Sl. No | Weight of Naphthalene $w_1$ (g) | Weight of un-known com-pound $w_2$ (g) | Melting point , $T_m$ (°C ) | Freezing point, $T_f$ (°C ) | Mean Freez-ing point , T = $(T_m+T_f)/2$ (°C ) | $M_2 = \frac{k_f \times w_2 \times 1000}{w_1 \times \Delta T_f}$ |
|---|---|---|---|---|---|---|
| 1 | 2.0000 | 0.0000 | | | | --- |
| 2 | 2.0000 | 0.2000 | | | | |
| 3 | 2.0000 | 0.4000 | | | | |

Mean molecular weight = _____ g $mol^{-1}$

$$\text{Thus } \Delta T_f = k_f \left( \frac{w_2}{M_2 \times w_1} \times 1000 \right)$$

The molal depression constant ($k_f$) is thus defined as that value of depression in freezing point which may be theoretically obtained for 1 molal solution.

$k_f$ can be experimentally determined, by determining $\Delta T_f$ using a solute of known molal mass ($M_2$) using the expression

$$k_f = \frac{M_2 \times w_1 \times \Delta T_f}{w_2 \times 1000}$$

Using the above value of $k_f$, the molal mass of unknown sample can be obtained using the expression

$$M_2 = \frac{k_f \times w_2 \times 1000}{w_1 \times \Delta T_f}$$

**PROCEDURE**

Take accurately 2.0000 g of pure naphthalene in a clean dry glass tube. Heat the tube in a constant heating water bath and determine the freezing point. Add about 0.2 g of diphenylamine into the tube and once again determine the freezing point. Repeat the above procedure after adding another 0.2 g of diphenylamine. Calculate the molal depression constant for naphthalene. Repeat the above procedure with 2.0000 g of naphthalene and 0.2 and 0.4 g of unknown compound to determine its molecular weight.

## CALCULATION

### (A) $k_f$ OF THE SOLVENT:

Weight of the solvent taken = $w_1$ g
Weight of the solute = $w_2$ g
Molecular weight of the solute, diphenylamine ($M_2$) = 169.22 g/mol
Freezing point of solvent = $T_A$ °C
Freezing point of solvent after addition of solute = $T_B$ °C
Depression in freezing point, $\Delta T_f$ = ($T_A$ - $T_B$) °C
Molal depression constant

$$k_f = \frac{169.22 \times w_1 \times \Delta T_f}{w_2 \times 1000}$$

### (B) MOLECULAR WEIGHT:

Weight of the solvent taken = $w_1$ g
Weight of the solute = $w_2$ g
Freezing point of the solvent = $T_A$ °C
Freezing point of the solvent after addition of solute = $T_B$ °C
Depression in freezing point $\Delta T_f$ = ($T_A$ - $T_B$ ) °C
Molecular weight of the solute

$$M_2 = \frac{k_f \times w_2 \times 1000}{w_1 \times \Delta T_f}$$

| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Define colligative property.<br>2. List the four colligative properties.<br>3. Define molality. | 1. Define 'depression in freezing point'.<br>2. Give the unit of molal depression constant.<br>3. Give the applications of 'depression in freezing point' in everyday life. |

## RESULT

The molal depression constant of naphthalene and the molecular weight of the unknown compound were determined and the results are as follows:

| Set. No. | $k_f$ (g °C $mol^{-1}$) | Molecular weight (g $mol^{-1}$) |
|---|---|---|
| 1 | | |
| 2 | | |
| Mean | | |

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT NO.** **DATE:**

# CONDUCTOMETRIC TITRATION

## AIM

To find out the strength of hydrochloric acid and acetic acid in a given mixture by titrating it against sodium hydroxide solution, conductometrically

## APPARATUS

Conductivity meter, burette, pipette, beakers, volumetric flask, etc.

## PRINCIPLE

Current is conducted through an electrolytic solution by the movement of positive and negative ions in opposite directions. The conductance of a solution is proportional to the number of ions, the charge of each ion and ionic speed. Conductance of any solution is generally expressed as equivalent conductance (mS). It is defined as the conductivity of all the ions produced by 1 g equivalent of the electrolyte in a given volume of solution.

When a strong acid such as HCl is titrated against strong base such as NaOH, the reaction taking place is

$$HCl + NaOH \longrightarrow Na^+Cl^- + H_2O$$

During the course of the titration, the fastest moving $H^+$ ions are replaced by the less mobile $Na^+$ ions. Hence, the conductivity decreases rapidly. At the equivalence point, all the $H^+$ ions are neutralized. When excess of NaOH is added it introduces the fast moving $OH^-$ ions and then the conductivity again increases. So two straight lines meeting at the minimum point is obtained in the plot of conductance against volume of NaOH solution. The volume of NaOH corresponding to the minimum point is the titre value.

When a weak acid like acetic acid is titrated against a strong base such as NaOH, the following reaction takes place

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$

## OBSERVATION

Weight of empty weighing bottle, $W_1$ = _____ g
Weight of bottle + Oxalic acid, $W_2$ = _____ g
Weight of bottle after transfer, $W_3$ = _____ g
Weight of oxalic acid taken, $(W_2 - W_3)$ = _____ g

$$\text{Conc. of oxalic acid } (x) = \frac{(W_2 - W_3) \times 1000}{250 \times 63} = \_\_\_\_\_ \text{ N}$$

## TABLE - 1 STANDARDIZATION OF NaOH SOLUTION

Volume of oxalic acid = 25 ml

| S. No. | Burette Reading (ml) | | Vol. of NaOH used ($V_1$) ml |
|---|---|---|---|
| | Initial | Final | |
| 1 | | | |
| 2 | | | |
| 3 | | | |

Volume ($V_1$) = ml

Acetic acid is partially ionized and the few $H^+$ ions present are replaced by $Na^+$ ions which are less mobile. But the product $CH_3COONa^+$ being a salt, is a strong electrolyte and it ionizes almost completely giving a large number of ions. Hence, from the beginning, the conductivity gradually increases instead of decreasing, as in the earlier case. After the equivalence point, excess of NaOH added gives fast moving $OH^-$ ions and conductivity sharply increases.

In the plot of conductance against volume of NaOH, two ascending lines, one steeper than the other is obtained and the volume of NaOH corresponding to the point at which these two curves meet gives the titre value. It must be noted that the first end point will be that of hydrochloric acid (strong), while the second that of acetic acid (weak). The curve will be a combination of the two curves as given below.

## PROCEDURE

Prepare a standard solution of oxalic acid by dissolving 1.5750 g of AR quality oxalic acid in 250 ml of distilled water. Using this standardize the given NaOH solution. Dilute the given sample of acid mixture into 100 ml with distilled water. Pipette out 10ml of the sample into a beaker and add 10 ml of water Mix well. Insert the conductivity cell into the solution and measure the conductance. Add 0.2 ml of 0.1 N solution of NaOH, from the burette, into the mixture, stir well and measure the conductance of the solution. Similarly measure the conductance of the solution after every addition of 0.2 ml of 0.1 N NaOH, until a final volume of about 8.0 ml of 0.1 N NaOH is added.

## MODEL GRAPH

Plot a graph between conductance and the volume of NaOH added and calculate the strength of HCl and $CH_3COOH$ in the given mixture.

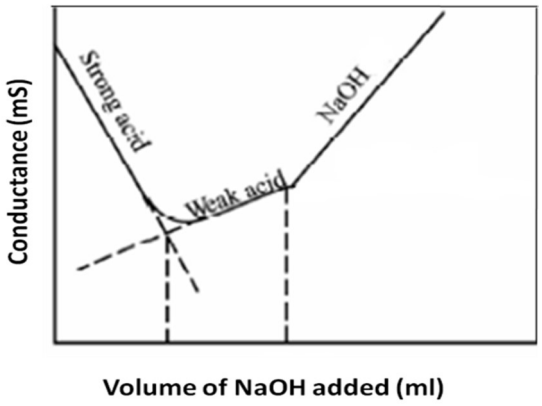


**TABLE-II CONDUCTOMETRIC TITRATION OF SAMPLE WITH NaOH**

Volume of sample taken = 10 ml

| S.No. | Volume of NaOH added (ml) | Conductance (mS) | S.No | Volume of NaOH added (ml) | Conductance (mS) |
|---|---|---|---|---|---|
| 1 | 0.0 | | 22 | 4.2 | |
| 2 | 0.2 | | 23 | 4.4 | |
| 3 | 0.4 | | 24 | 4.6 | |
| 4 | 0.6 | | 25 | 4.8 | |
| 5 | 0.8 | | 26 | 5 | |
| 6 | 1.0 | | 27 | 5.2 | |
| 7 | 1.2 | | 28 | 5.4 | |
| 8 | 1.4 | | 29 | 5.6 | |
| 9 | 1.6 | | 30 | 5.8 | |
| 10 | 1.8 | | 31 | 6.0 | |
| 11 | 2.0 | | 32 | 6.2 | |
| 12 | 2.2 | | 33 | 6.4 | |
| 13 | 2.4 | | 34 | 6.6 | |
| 14 | 2.6 | | 35 | 6.8 | |
| 15 | 2.8 | | 36 | 7.0 | |
| 16 | 3.0 | | 37 | 7.2 | |
| 17 | 3.2 | | 38 | 7.4 | |
| 18 | 3.4 | | 39 | 7.6 | |
| 19 | 3.6 | | 40 | 7.8 | |
| 20 | 3.8 | | 41 | 8.0 | |
| 21 | 4.0 | | | | |

## CALCULATION

### (a) CONCENTRATION OF NaOH

Volume of oxalic acid taken = 25 ml
Volume of NaOH used, $V_1$ = _____ ml
Concentration of oxalic acid, $x$ = _____ N
Concentration of NaOH, $N_1$ = ?
$25 \times x = V_1 \times N_1$

$\therefore N_1 = \frac{25x}{V_1}$ = _____ N

$\therefore$ Concentration of NaOH, $N_1$ = _____ N

### (b) STRENGTHS OF HCl AND $CH_3COOH$

From the graph,

Volume of NaOH consumed for HCl, $V_a$ = _____ ml
Volume of NaOH consumed for Acetic acid and HCl, $V_b$ = _____ ml

### (i) AMOUNT OF HCl IN THE MIXTURE:

Volume of sample taken = 10 ml
Normality of NaOH, $N_1$ = _____ N
Normality of HCl, $N_2$ = ?
$10 \times N_2 = V_a \times N_1$

$N_2 = \frac{N_1 \times V_a}{10}$ = _____ N

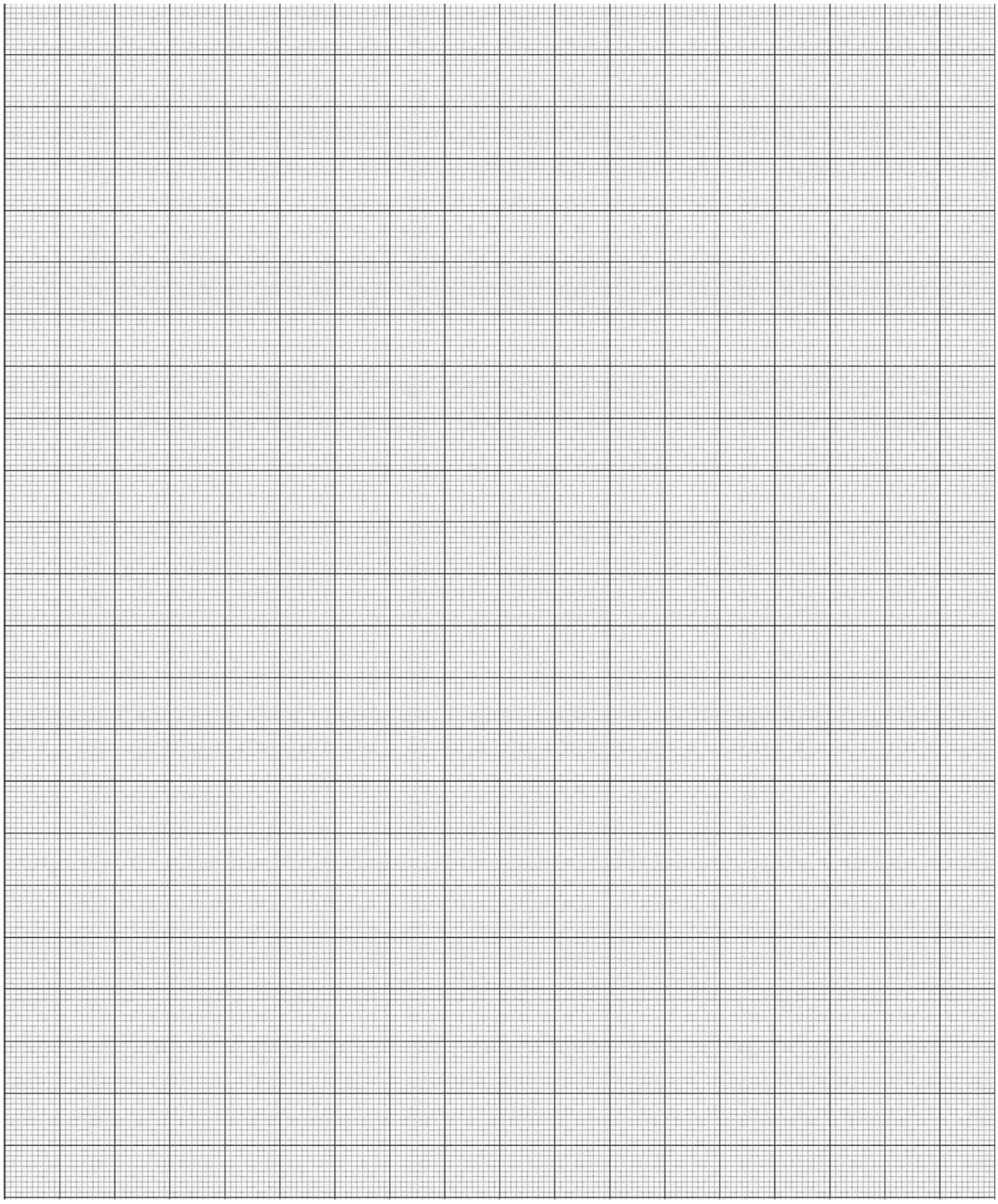
Concentration of HCl, $N_2$ = _____ N

Amount of HCl in the whole of solution, $W_{HCl} = \frac{N_2 \times 36.5 \times 100}{1000}$ = _____ g

**Scale**

**In x-axis, 1 cm =**

**In y-axis, 1 cm =**



## (ii) AMOUNT OF ACETIC ACID IN THE MIXTURE

Volume of sample taken = 10 ml
Volume of NaOH used for Acetic acid, $(V_b - V_a)$ =_____ ml
Normality of NaOH, $N_1$ = _____ N
Normality of acetic acid, $N_3$ = ?

$10 \times N_3 = (V_b - V_a) \times N_1$

$N_3 = \frac{(V_b - V_a) \times N_1}{10}$ = _____ N

Concentration of acetic acid, $N_3$ = _____N

Amount of $CH_3COOH$ in whole solution = $W_{CH_3COOH} = \frac{N_3 \times 60 \times 100}{1000}$ = _____ g

| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Define conductivity.<br>2. Give the unit of conductance.<br>3. Define an electrolyte. | 1. Differentiate between volumetric titration and conductometric titration.<br>2. How is the end point determined in the titration of a strong acid against a strong base?<br>3. How is the end point determined in the titration of a weak acid against a strong base? |

## RESULT

The amount of HCl in the given mixture was found to be = _____ g

The amount of $CH_3COOH$ in the given mixture was found to be = _____ g

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT NO.** **DATE:**

# DETERMINATION OF THE RATE CONSTANT OF ACID CATALYSED HYDROLYSIS OF AN ESTER

## AIM

To determine the rate constant of acid catalyzed hydrolysis of methyl acetate.

## APPARATUS

Thermostat, beakers, conical flasks, burettes, pipettes, etc.

## PRINCIPLE

A first order reaction is one in which the rate depends on concentration of only one of the reactants. Methyl acetate hydrolyses, in the presence of an acid, which acts as a catalyst, to give acetic acid and methyl alcohol.

$$CH_3COOCH_3 + H_2O \xrightarrow{\text{acid}} CH_3COOH + CH_3OH$$

The reaction rate is given by

Rate = k' [ $CH_3COOCH_3$] [$H_2O$], where k' is the specific reaction rate constant-(1)

Since water is present in large excess, its concentration remains practically constant throughout the reaction. As a result of this assumption, the above equation reduces to

Rate = k' [ $CH_3COOCH_3$] where k' = k [$H_2O$] = constant----------(2)

Hence, the rate of reaction is determined by the first power of the concentration of the ester and so the reaction is of the first order. It is however, a pseudo first order reaction which is not first order but is forced to obey the first order rate expression. Such reactions involve more than one molecule in the chemical reaction. As acetic acid is produced during the hydrolysis of methyl acetate, the reaction can be followed by titrating the reaction mixture with standard solution of an alkali.

The value of k can be calculated according to the first order rate expression which is given by

**OBSERVATION**

Room temperature = 30 ºC
Volume of 0.5 N HCl = 100 ml
Volume of ester = 10 ml

**TABLE I**

**TITRATION OF REACTION MIXTURE WITH 0.1 N NaOH**:

Volume of reaction mixture taken = 5 ml

| S.No | Time (min) | Burette Reading (ml) | | Volume of NaOH used, $V_t$ (ml) |
|---|---|---|---|---|
| | | Initial | Final | |
| 1 | 0 | | | |
| 2 | 5 | | | |
| 3 | 10 | | | |
| 4 | 20 | | | |
| 5 | 30 | | | |
| 6 | 40 | | | |
| 7 | 50 | | | |
| 8 | 60 | | | |
| 9 | $t_\infty$ at 90 min | | | |

$$k = \frac{2.303}{t} \times \log \frac{a}{a-x}$$

$$k = \frac{2.303}{t} \times \log \left( \frac{(V_\infty - V_O)}{(V_\infty - V_t)} \right)$$

where,
$V_o$ = Volume of alkali used at t = 0 min,
$V_t$ = Volume of alkali used at any time of reaction,
$V_\infty$ = Volume of alkali used at the end of the reaction.

The values of rate constant can be calculated at different intervals of time. It has the dimensions of time$^{-1}$.

**PROCEDURE**

Take 100 ml of 0.5 N HCl in a dry 250 ml conical flask and 10 ml of methyl acetate in a beaker. Place them separately in the thermostat, so as to reach an equilibrium temperature, for about 15 minutes. Fill the burette with 0.1 N NaOH solution and take 3 conical flasks containing crushed ice. Transfer 10ml of methyl acetate into the conical flask containing 100 ml of 0.5 N HCl and start the stop clock. Shake the flask well and immediately withdraw 5 ml of the reaction mixture, with the help of a pipette, into the conical flask with ice to arrest the reaction. Titrate this solution as rapidly as possible with 0.1 N NaOH solution using phenolphthalein (5 drops) as an indicator. This titre value gives the amount of HCl in the reaction mixture at the start of the reaction. This is taken as $V_o$.

Repeat the above procedure of withdrawing 5 ml of the reaction mixture adding into a conical flask containing crushed ice and titrating with 0.1 N NaOH at successive intervals of 5,10, 20, 30, 40, 50, 60, 70, 80 and 90 minutes. These titre values are taken as respective $V_t$ readings.

To find out the completion of hydrolysis of ester, transfer 25 ml of the reaction mixture into a separate conical flask, cover it with a beaker inverted in a water bath and heat it for about 30 min at 70-80°C in a hot air oven.

Pipette 5 ml of this mixture in to a conical flask and titrate against 0.1 N NaOH using phenolphthalein (5 drops) as an indicator. The titre value represents the total amount of HCl and the amount of acetic acid formed at the end of reaction and is denoted by $V_\infty$.

**TABLE - II**

**CALCULATION OF RATE CONSTANT ( k )**

| **Time, t (min)** | **Volume of NaOH, $V_t$ (m1)** | $\log\left(\frac{(V_\infty - V_O)}{(V_\infty - V_t)}\right)$ | $k = \frac{2.303}{t} \log \frac{(V_\infty - V_O)}{(V_\infty - V_t)}$ ($min^{-1}$) |
|---|---|---|---|
| 0 | | | |
| 5 | | | |
| 10 | | | |
| 20 | | | |
| 30 | | | |
| 40 | | | |
| 50 | | | |
| 60 | | | |
| $t_\infty$ at 90 min | | | |

Mean k = ---------- $min^{-1}$

**CALCULATION**

The rate constant $k = \frac{2.303}{t} \times \log\left(\frac{(V_\infty - V_O)}{(V_\infty - V_t)}\right)$

where,

t = time of reaction in minutes,

$V_0$ = Volume of NaOH required at start of reaction,

$V_\infty$=Volume of NaOH required for the completion of reaction,

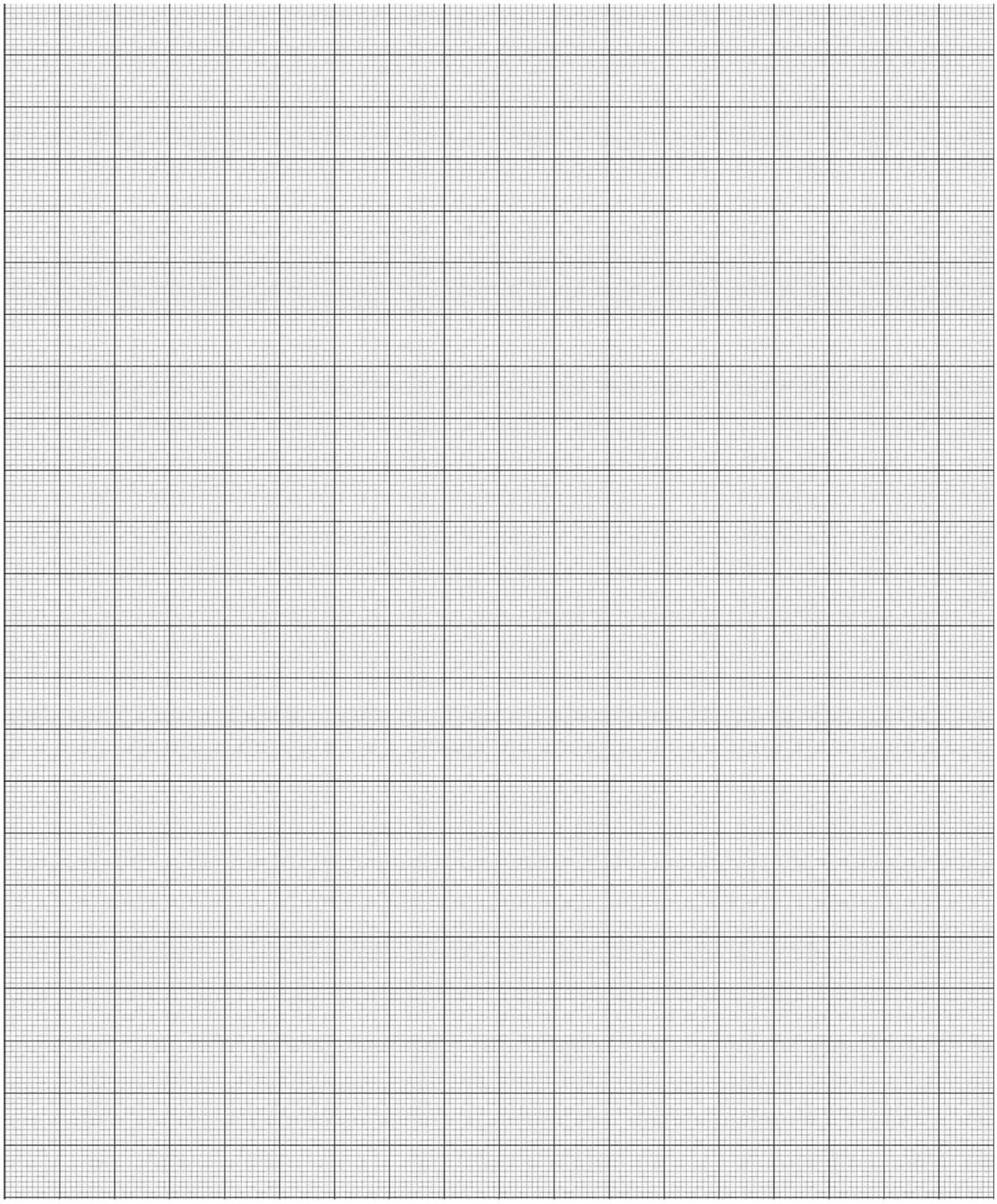
$V_t$ = Volume of NaOH required at regular interval of time, t.

The value of **k** is obtained graphically by plotting graph of log $\frac{(V_\infty - V_O)}{(V_\infty - V_t)}$ against time, t. The plot gives a straight line passing through origin with slope equal to k.

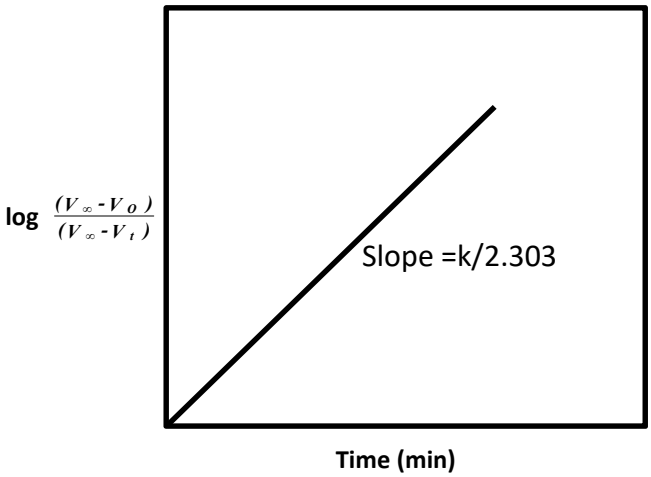
**Scale**

**In x-axis, 1 cm =**

**In y-axis, 1 cm =**



## MODEL GRAPH




From the graph k = slope = $\frac{y_2 - y_1}{x_2 - x_1}$ = _____

k = 2.303 x slope = 2.303 x _____ = _____ $min^{-1}$

| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Define rate of a reaction.<br>2. Define order of a reaction<br>3. State the equation governing the effect of temperature on rate constant. | 1. What is a pseudo first order reaction?<br>2. Give the unit of first order rate constant.<br>3. What are the different ways of finding the rate constant of a chemical reaction? |

## RESULT

The rate constant of the acid catalyzed hydrolysis of methyl acetate at 30 °C was found to be

a) From the graph, k = _____ $min^{-1}$

b) By calculation (analytically), k = _____ $min^{-1}$

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT NO.** **DATE:**

# PHASE DIAGRAM OF THREE COMPONENT SYSTEM

## AIM

To draw the phase diagram of three component (water-chloroform-acetic acid) system

## APPARATUS

Test tube, Burette, Reagent bottles

## THEORY

The phase rule states that, for a heterogeneous system at equilibrium, the sum of number of phases (P) and the degrees of freedom (F) is greater than the number of components (C) by two.

$$P + F = C + 2$$

or $\mathbf{F = C - P + 2}$

For a three component system, C = 3

Therefore, $F = 3 - P + 2 \Rightarrow F = 5 - P$

For a system containing one phase (P = 1)

$$F = 5 - 1 = 4$$

Hence the phase diagram should illustrate four variables which is difficult. Thus fixing pressure and temperature constant for a three component system, the phase rule reduces to F = 3 - P. A triangular graph is used to describe the ternary system in terms of the remaining variables –two of the three concentrations.

The simplest three components are those in which a liquid system breaks down into two phases. The system $CH_3COOH$-$CHCl_3$-$H_2O$ is such a system over a certain temperature range. A two phase region occurs in systems with relatively low amounts of acetic acid. The tie lines passing through the two phase region join the compositions of the two phases that are in equilibrium.

## PROCEDURE

Take three burettes. Fill each of them respectively with water, chloroform and acetic acid. Into a test tube add 5 ml of chloroform from the burette. Now add 0.5 ml of water and mix until two layers are formed. Now add acetic acid until the two layers merge to become a single homogeneous layer. Note down the value of acetic acid. Add another 0.5 ml of water in the test tube and repeat the addition of

## OBSERVATION

## PHASE DIAGRAM OF THREE COMPONENT SYSTEM

| Sl. No | Volume of | | | Volume % | | |
|---|---|---|---|---|---|---|
| | $CHCl_3$ $V_1$(ml) | $H_2O$ $V_2$(ml) | $CH_3COOH$ $V_3$(ml) | $CHCl_3$ $\left(\frac{V_1}{V_1+V_2+V_3}\right)x100$ | $H_2O$ $\left(\frac{V_2}{V_1+V_2+V_3}\right)x100$ | $CH_3COOH$ $\left(\frac{V_3}{V_1+V_2+V_3}\right)x100$ |
| 1. | 5 | 0.5 | | | | |
| 2. | 5 | 1 | | | | |
| 3. | 5 | 1.5 | | | | |
| 4. | 5 | 2 | | | | |
| 5. | 5 | 2.5 | | | | |
| 6. | 5 | 3 | | | | |
| 7. | 5 | 3.5 | | | | |
| 8. | 5 | 4 | | | | |
| 9. | 5 | 4.5 | | | | |
| 10. | 5 | 5 | | | | |
| 11. | 5 | 5.5 | | | | |
| 12. | 5 | 6 | | | | |
| 13. | 5 | 6.5 | | | | |
| 14. | 5 | 7 | | | | |
| 15. | 5 | 7.5 | | | | |
| 16. | 5 | 8 | | | | |
| 17. | 5 | 8.5 | | | | |
| 18. | 5 | 9 | | | | |
| 19. | 5 | 9.5 | | | | |
| 20. | 5 | 10 | | | | |
| 21. | 10 | 5 | | | | |

acetic acid dropwise. Repeat this process after each 0.5 ml addition of water until the volume of water reaches 10 ml. Take another test tube and add 5 ml of water and 0.5 ml of chloroform; add acetic acid dropwise from the burette until the two layers merge into a homogeneous layer. Repeat this process after each addition of 0.5 ml of chloroform until its volume reaches 9.0 ml. From these values, calculate the respective volume percentage of the three components and plot the phase diagram in the graph.

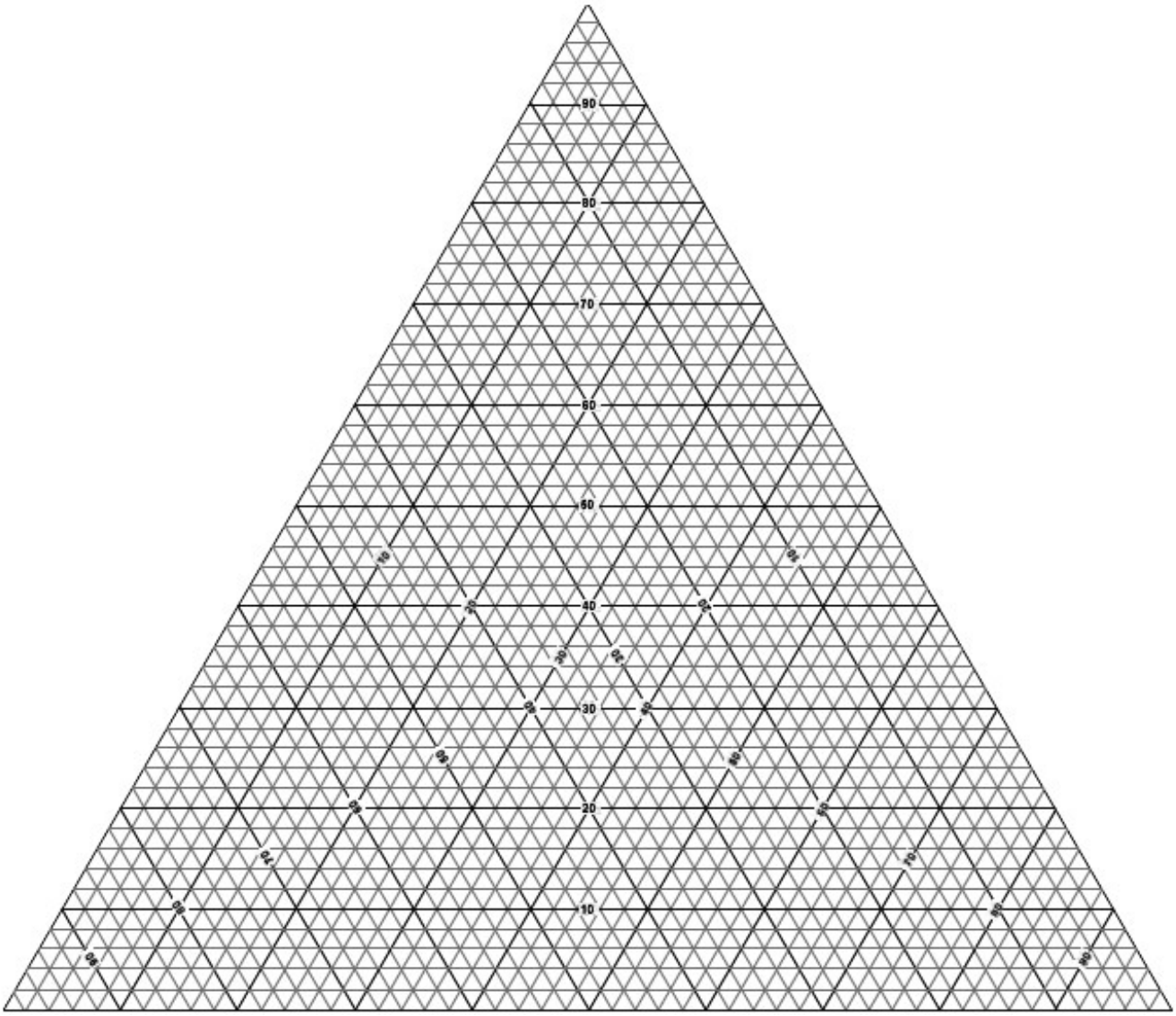
| | | | | | | |
|---|---|---|---|---|---|---|
| 22. | 9.5 | 5 | | | | |
| 23. | 9 | 5 | | | | |
| 24. | 8.5 | 5 | | | | |
| 25. | 8 | 5 | | | | |
| 26. | 7.5 | 5 | | | | |
| 27. | 7 | 5 | | | | |
| 28. | 6.5 | 5 | | | | |
| 29. | 6 | 5 | | | | |
| 30. | 5.5 | 5 | | | | |
| 31. | 5 | 5 | | | | |
| 32. | 4.5 | 5 | | | | |
| 33. | 4 | 5 | | | | |
| 34. | 3.5 | 5 | | | | |
| 35. | 3 | 5 | | | | |
| 36. | 2.5 | 5 | | | | |
| 37. | 2 | 5 | | | | |
| 38. | 1.5 | 5 | | | | |
| 39. | 1 | 5 | | | | |
| 40. | 0.5 | 5 | | | | |
| 41. | 0 | 5 | | | | |

**Scale**

**In x-axis, 1 cm =**

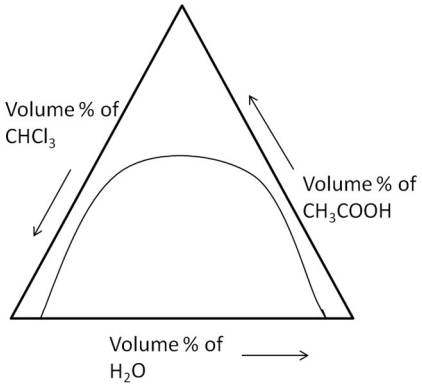
**In y-axis, 1 cm =**

**In z-axis, 1 cm =**



## MODEL GRAPH:

Plot the phase diagram.




| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Define a phase.<br>2. Define degrees of freedom.<br>3. Define a component. | 1. Give an example of a three component system.<br>2. What are tie lines?<br>3. Give the significance of a ternary plot. |

## RESULT

The phase diagram for $CHCl_3$ – $H_2O$ – $CH_3COOH$ system was plotted.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT NO.** **DATE:**

# PARTITION COEFFICIENT OF BENZOIC ACID BETWEEN BENZENE AND WATER

## AIM

To find out the partition coefficient of benzoic acid between benzene and water

## APPARATUS

Reagent bottles, burette, pipette, conical flask, separating funnel, shaking machine etc.

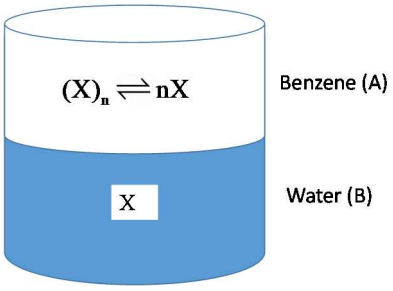
## THEORY

When a solute is added to a system of two immiscible liquids, it distributes itself between the two liquid layers in a definite proportion, irrespective of the total amount of the solute. Such systems follow the 'Nernst distribution law' according to which the ratio of concentrations of the solute in solvent A and solvent B is a constant.

$$K = \frac{C_A}{C_B}$$

where K is called the distribution coefficient or partition coefficient. This holds true when the solute has the same molecular weight in both the immiscible solvents, i.e., it is in the same molecular state.

When benzoic acid (X) is distributed between benzene (A) and water (B), it exists as $C_6H_5COOH$ in water and as $(C_6H_5COOH)_2$ in benzene due to association of the molecules forming dimers.



Suppose the solute exists as simple molecules of X in solvent B while n molecules of X associate to form $(X)_n$ molecules in solvent A. In such a case, the ratio $C_A/C_B$ will not be constant. The value of partition coefficient can now be calculated as follows.

Let $C_1$ be the concentration of the solute X in solvent B,
$C_2$ be the concentration of $X_n$ in solvent A, and
$C_3$ be the concentration of X in solvent A.

There exists an equilibrium between X in solvent A and X in solvent B. Therefore, by Nernst distribution law, we have

$$K_D = \frac{C_1}{C_3} \text{ ----------(i)}$$

In solvent A, the solute is associated and it exists in equilibrium with unassociated molecule. Applying the law of mass action to the chemical equilibrium, $(X)_n \rightleftharpoons nX$

Equilibrium constant, $K_C = \frac{[X]^n}{[(X)_n]} = \frac{(C_3)^n}{C_2}$ -------(ii)

Taking $n^{th}$ root on both sides of equation (ii),

$$\sqrt[n]{K_C} = \frac{C_3}{\sqrt[n]{C_2}} \text{ --------(iii)}$$

Dividing equation (i) by equation (iii), $\frac{K_D}{\sqrt[n]{K_C}} = \frac{C_1}{\sqrt[n]{C_2}} = K$ (a constant) -------(iv)

Thus when association occurs in one solvent, the distribution equation is modified as

$$K = \frac{C_1}{\sqrt[n]{C_2}} \text{ ------(v)}$$

In case of benzoic acid which generally exists as dimer in aprotic solvents such as benzene, the value of n = 2.

$$K = \frac{C_1}{\sqrt{C_2}}$$

**PROCEDURE**

Take three reagent bottles and mark them as 1, 2, and 3. Add 50ml of benzene and 50 ml of water into each of the 3 reagent bottles. Add 0.5, 1.0, 1.5 g of benzoic acid into bottle numbers 1, 2, and 3 respectively. Stopper the bottles properly and shake all the bottles for about 30-45 minutes in a shaking machine. Allow the mixture to separate into two layers, the lower layer will be the aqueous layer, while the upper layer will be the benzene layer.

Take 5 ml of the benzene layer, from each bottle by means of a pipette in a conical flask. Add 4-5 drops of phenolphthalein indicator. Titrate it against 0.1 N NaOH solution. Repeat the process till you get two concordant readings. Similarly titrate the benzene layer of each of the other two bottles by pipetting out 5 ml of the solution and titrating it against 0.1 N NaOH solution using 4-5 drops of phenolphthalein. Pipette out 5 ml of the aqueous layer of the first bottle and titrate it against 0.01 N NaOH solution, using phenolphthalein (4-5 drops) as the indicator. Repeat the process for the other bottles also.

## OBSERVATION

Room temperature = 30 °C

### TABLE I: TITRATION OF AQUEOUS LAYER WITH 0.01 N NaOH SOLUTION

Volume of water layer = 5 ml

| Bottle No. | Burette Reading (ml) | | Volume of 0.01 N NaOH solution ($V_1$ ml) |
|---|---|---|---|
| | Initial | Final | |
| I | | | |
| | | | |
| II | | | |
| | | | |
| III | | | |
| | | | |

### TABLE II: TITRATION OF BENZENE LAYER WITH 0.1 N NaOH SOLUTION

Volume of benzene layer = 5 ml.

| Bottle No. | Burette Reading (ml) | | Volume of 0.1N NaOH solution ($V_1$ ml) |
|---|---|---|---|
| | Initial | Final | |
| I | | | |
| | | | |
| II | | | |
| | | | |
| III | | | |
| | | | |

**CALCULATION:**

**TABLE III: CONCENTRATION OF BENZOIC ACID IN AQUEOUS LAYER ($C_1$)**

Volume of water layer = 5 ml

| Bottle No. | Vol. of 0.01N NaOH used ($V_1$ / ml) | Normality of benzoic acid $N_1 = \frac{V_1 X 0.01}{5}$ | Concentration of benzoic acid $C_1 = N_1$ X 122 (g /l ) |
|---|---|---|---|
| I | | | |
| II | | | |
| III | | | |

**TABLE IV: CONCENTRATION OF BENZOIC ACID IN BENZENE LAYER ($C_2$)**

Volume of benzene layer = 5 ml

| Bottle No. | Vol. of 0.1N NaOH used ($V_2$ / ml) | Normality of benzoic acid $N_2 = \frac{V_2 X 0.1}{5}$ | Concentration of benzoic acid $C_2 = N_2$ X 122 (g /l ) |
|---|---|---|---|
| I | | | |
| II | | | |
| III | | | |

## TABLE V: CALCULATION OF PARTITION COEFFICIENT

| Bottle no. | Conentration of benzoic acid in water layer, $C_1$ (g/l) | Concentration of benzoic acid in benzene layer, $C_2$ (g/l) | $K = \frac{C_1}{(C_2)^{1/2}}$ |
|---|---|---|---|
| I | | | |
| II | | | |
| III | | | |

Mean K = ----------

| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Define partition coefficient.<br>2. Give the Nernst distribution law.<br>3. What are the reasons for deviations in Nernst distribution law? | 1. Discuss the molecular state of benzoic acid in benzene and in water.<br>2. Give the unit of partition coefficient.<br>3. What are the applications of Nernst distribution law? |

## RESULT

The partition coefficient of benzoic acid between water and benzene at 30 °C was found to be _____.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT NO.** **DATE:**

## ESTIMATION OF SULPHATE BY NEPHELOMETRY

### AIM

To determine the amount of sulphate in the given sample by using turbidity meter

### APPARATUS

Pipette, Burette, Volumetric flask, etc.

### REAGENTS

Standard sulphate solution, Sodium chloride-hydrochloric acid mixture, Barium chloride, Glycerol-ethanol solution.

### INTRODUCTION

Turbidity can be casually defined as the observable cloudiness or haziness of a liquid which is caused by suspended solids. It is defined by the Standard Methods for the Examination of Water and Wastewater as, an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in straight lines through the sample.

Turbidity is typically determined by measuring the attenuation (intensity loss) of light as it is passed through a sample. Light which is passed through pure water continues undisturbed, but light which is passed through water containing suspended particles reacts with the particles, which absorb some light and scatter the remainder.

It is important to note that turbidity is not the direct measurement of suspended particles but more so of the scattering effect the particles have on incident light. Considering this statement, particle shape and size does largely determine light scattering effects, as does the refractive index and color of the sample.

### INSTRUMENTATION

The most common turbidity instrument is the turbidimeter, or turbidity meter. These devices consist of three main component groups: the light source, sample container, and photodetectors.

- **LIGHT SOURCE**

A turbidimeter's light source provides the incident light to be passed through the sample. Most light source consist of tungsten filament lamps, which provide polychromatic light with a wide wavelength range. At times, however, this wide wavelength can cause interference in turbidity measurement as some natural colors and organic matter can absorb artificially high amounts of certain wavelengths. In addition, tungsten lamps are highly dependent on the stable voltage of the light source power supply and are subject to burnout over time.

For the reasons listed above, some turbidimeters use monochromatic, narrow-wavelength light-emitting diodes (LED), lasers, or mercury lamps as light sources. While these lamps eliminate the interference common in tungsten types, they are also less sensitive to small particles.

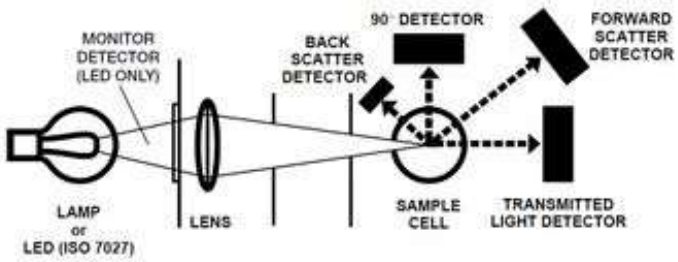
- **SAMPLES**

Samples are typically introduced in glass cuvettes in the case of benchtop and portable turbidimeters. On-line devices occasionally use glass sample cells but more often use sample liquids which flow through a chamber between the light source and photodetectors.

- **PHOTODETECTORS**

Photodetectors output an electrical signal proportional to the amount of detected light. Commonly-used detectors include photodiodes, photomultiplier tubes, and cadmium sulfide photoconductors. Because each of these detector types is especially sensitive to certain wavelengths, users must factor detector differences into turbidity measurements when using polychromatic light sources. For example, photodiodes are much more sensitive to short wavelengths when used with tungsten filament lamps, therefore making them more sensitive to the detection of small particles.

The detector configuration for a nephelometric ratio turbidimeter—one of the most common modern types—is shown below. All nephelometers primarily measure light scattered at a 90° position and often include additional detectors for sensing back- and front-scattered as well as transmitted radiation. The output of these sensors would be mathematically combined and formatted into a turbidity measurement.



## APPLICATIONS

Turbidity is a key component of any comprehensive water quality analysis. Suspended particles can be the result of phytoplankton within the water source, or sediment from nearby human factors such as construction, farming, and mining. Turbidity measurement is common in the following water quality applications.

- Drinking water: As the turbidity of drinking water increases, the incidence and risk of gastrointestinal disease generally increases.
- Aquatic life: High turbidity reduces the amount of sunlight which reaches deeper waters. This can affect aquatic plant (and subsequently aquatic fauna) growth.

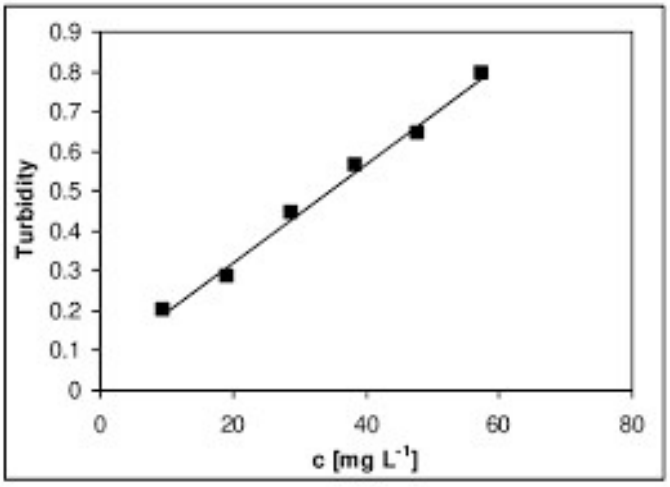
## PRINCIPLE

Barium Chloride is added to standard sulphate to precipitate Barium Sulphate.

$$BaCl_2\ (aq) + K_2SO_4\ (aq) \text{----->} BaSO_4\ (s) + 2KCl\ (aq)$$

The rate of dissolution of barium chloride controls the velocity of the reaction. Sodium chloride and hydrochloric acid are added before the precipitation in order to inhibit the growth of micro crystals of barium sulphate. The optimum pH is maintained to minimize the effect of variable amounts of other electrolytes present in the sample. A glycerol-ethanol solution helps to stabilize the turbidity. The turbidity of a dilute barium sulphate suspension is difficult to reproduce. The velocity of the precipitation, as well as the concentration of the reactants, must be controlled by adding pure solid barium chloride of definite grain size.

## MODEL GRAPH



## OBSERVATION AND CALCULATION

Concentration of the standard potassium sulphate stock solution (C) = --------- ppm

| S.No. | Volume of potassium sulphate solution taken ($V_i$ ml)) | Conc. of potassium sulphate (ppm) = (C x $V_i$ ) /100 | Turbidity meter reading (NTU) |
|---|---|---|---|
| 1. | 0 | | |
| 2. | 0.5 | | |
| 3. | 1 | | |
| 4. | 1.5 | | |
| 5. | 2 | | |
| 6. | 2.5 | | |
| 7. | 3.0 | | |
| 8. | Unknown Sample | | |

## PROCEDURE

Run 0.5, 1, 1.5, 2.0, 2.5 and 3.0 ml of the standard potassium sulphate solution from a calibrated burette into separate 100 ml graduated flasks. To each flask add 10 ml of the sodium chloride-hydrochloric acid mixture, 20 ml of glycerol-ethanol solution and dilute to 100 ml with distilled water. Add 0.3 g of the sieved barium chloride to flask. Stopper the flask and shake for 1 minute by inverting each flask once per second. All the barium chloride should dissolve. Allow each flask to stand for 2-3 minutes and measure the turbidity in the turbidity meter.

Take care to avoid small air bubbles adhering to the walls of the matched test tubes. Use the most concentrated solution as standard and by means of the sensitivity control adjusts the galvanometer reading to 100 divisions. Prepare a blank solution (without sulphate) and repeat the above sequence of operations. Place the blank solution in the turbidity meter and adjust to zero reading of the galvanometer scale by means of the zero control above the galvanometer suspension.

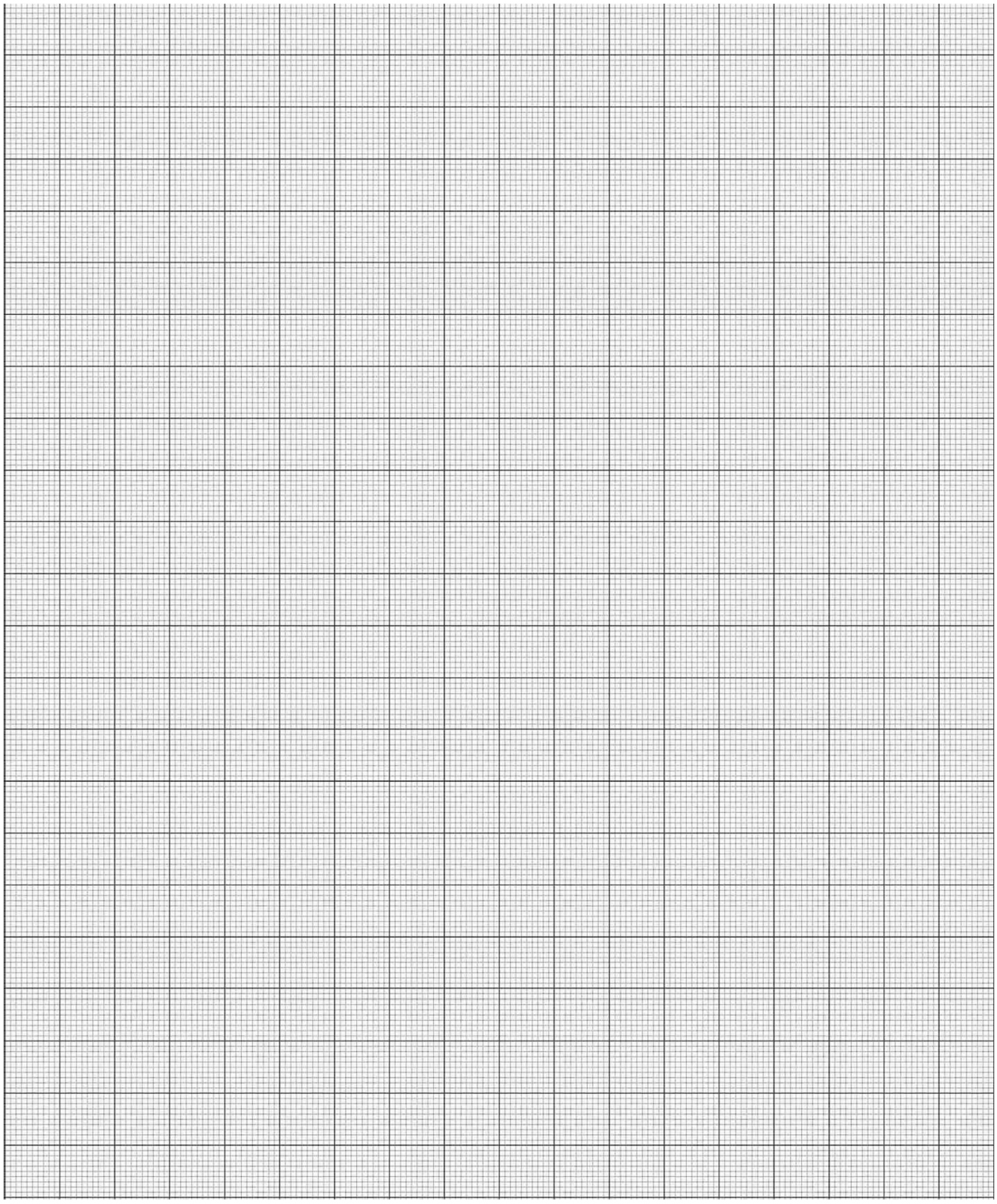
Check the reading of the most turbid solution, and adjust any deviation from 100 by means of the sensitivity control. Repeat the measurements with the five other standard sulphate solutions. Plot the galvanometer reading against the sulphate-ion content per ml. Determine the sulphate-ion content of an unknown solution.

The reaction vessel should be shaken at the same rate and the same number of times. The unknown must be treated exactly like the standard solution. The internal between the time of precipitation and measurement must be kept constant.

**Scale**

**In x-axis, 1 cm =**

**In y-axis, 1 cm =**



| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. What are the different water quality parameters?<br>2. Define turbidity.<br>3. What is a calibration plot? | 1. What are the components of a nephelometer?<br>2. Give the role of glycerol - ethanol mixture and NaCl - HCl micture in generating a turbid $BaSO_4$ mixture.<br>3. Explain the phenomenon of scattering of light. |

## RESULT

The amount of sulphate in the given sample was found to be _____ ppm.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT No.** **DATE:**

## ESTIMATION OF REDUCING SUGAR BY DINITRO SALICYLIC ACID (DNS) METHOD

### AIM

To estimate the amount of reducing sugar present in the given sample by using DNS method.

### APPARATUS

Pipette, Test tube, Volumetric flasks, Beaker, Heating mantle, UV-Visible spectrophotometer etc.,

### REAGENTS

Glucose stock solution, 3,5 dinitrosalicylic acid, sodium potassium tartrate solution.

### PRINCIPLE

The DNS method for estimating the concentration of reducing sugars in a sample was originally invented by G. Miller in 1959. Reducing sugars have the property to reduce many of the reagents. A reducing sugar is one that in a basic solution forms an aldehyde or ketone.

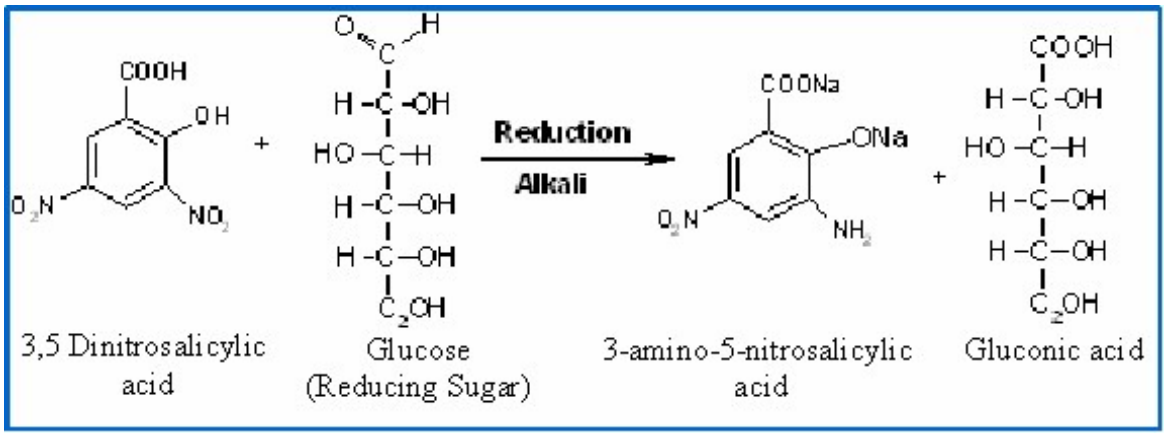
This method tests for the presence of free carboxyl group in reducing sugars. This involves the oxidation of the aldehyde functional group present in glucose. Simultaneously, 3,5-dinitrosalicylic acid (DNS) is reduced to 3-amino, 5-nitrosalicylic acid under alkaline conditions.

Aldehyde group ---Oxidation---> carboxyl group

3, 5-dinitrosalicylic acid ---Reduction---> 3-amino, 5-nitrosalicylic acid

**OBSERVATION**

| S.No | Vol. of standard glucose solution, $V_1$ (ml) | Concentration of standard solution $N_2 = \frac{V1}{7}$ x 100 (mg/ml) | Absorbance at 540 nm |
|---|---|---|---|
| 1 | 0 | | |
| 2 | 0.5 | | |
| 3 | 1.0 | | |
| 4 | 1.5 | | |
| 5 | 2 | | |
| 6 | 2.5 | | |
| 7 | Unknown | | |




The above reaction scheme shows that one mole of sugar will react with one mole of 3,5-dinitrosalicylic acid. However, it is suspected that there are many side reactions, and the actual reaction is more complicated than that previously described. The type of side reaction depends on the exact nature of the reducing sugars. Different reducing sugars generally yield different color intensities; thus, it is necessary to calibrate for each sugar. The absorbance measured using a spectrophotometer is directly proportional to the amount of reducing sugar.

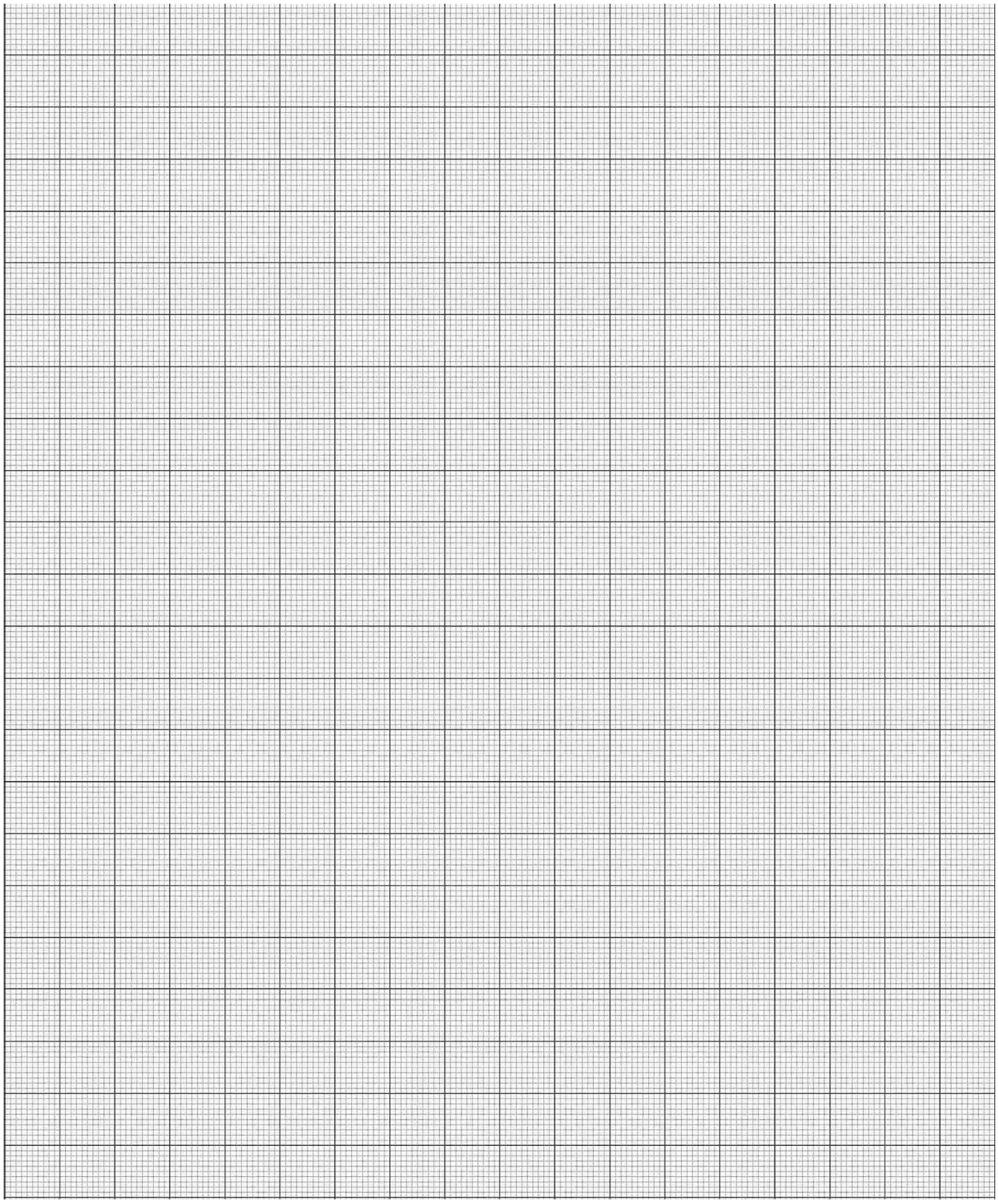
**PROCEDURE**

- Prepare 1 mg/ml of glucose stock solution by weighing 100 mg of glucose dissolving in 100 ml of distilled water.
- Pipette out 0, 0.5, 1.0, 1.5, 2.0 and 2.5 ml of the standard glucose solution into series of dry clean and labeled test tubes.
- Make it up to 3 ml with distilled water
- 3 ml of distilled water alone serves as a blank
- Add 3 ml of dinitro salicylic acid to all the test tubes then kept in water bath at 70º C for 10 -15 min until the red-brown color develops
- Add 1 ml of 40% sodium- potassium tartrate (Rochelle salt) solution to all the test tubes
- Cool the test tubes thoroughly and read the optical density (absorbance) of the colored solutions at 540 nm.
- Plot the standard curve of the absorbance (Y-axis) against the glucose concentration
  (X-axis) and determine the amount of reducing sugar in the unknown sample.

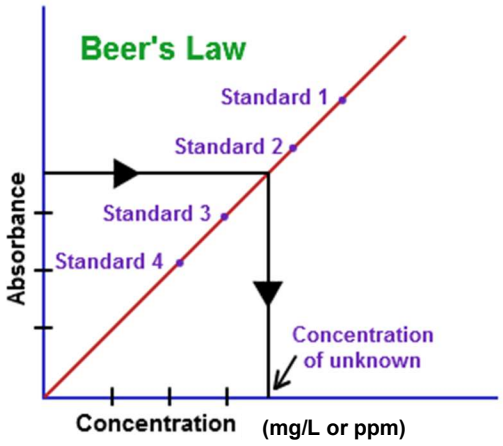
**Scale**

**In x-axis, 1 cm =**

**In y-axis, 1 cm =**



## MODEL GRAPH




| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. What are reducing and non-reducing sugars?<br>2. Define turbidity.<br>3. What is a calibration plot? | 1. Give the principle of DNS method.<br>2. Give the applications of DNS method of analysis in everyday life.<br>3. Convert ppm to $g/m^3$. |

## RESULT

The concentration of reducing sugars present in the given sample was found to be _____ ppm.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT No.** **DATE:**

## ESTIMATION OF IRON IN THE GIVEN SAMPLE BY USING UV- VISIBLE SPECTROPHOTOMETER

### AIM

To estimate the amount of iron present in the given sample by using UV-Visible spectrophotometer

### APPARATUS

Pipette, Burette, Volumetric flasks, Weighing bottle

### REAGENTS

Ammonium ferric sulphate, Ammonium thiocyanate solution (2M), Nitric acid (4N).

### PRINCIPLE

UV-visible spectroscopy is type of absorption spectroscopy in which light of ultra-violet radiation is absorbed by the molecule. Absorption of the ultra-violet radiations results in the excitation of the electrons from the ground state to higher energy state. The energy of the ultra-violet radiation that are absorbed is equal to the energy difference between the ground state and higher energy states. The Beer-Lambert law states that the absorbance of a solution is directly proportional to the concentration of the absorbing species in the solution and the path length. Thus, for a fixed path length, UV/Vis spectroscopy can be used to determine the concentration of the absorber in a solution

UV spectroscopy obeys the Beer-Lambert law, which states that when a beam of monochromatic light is passed through a solution of an absorbing substance, the rate of decrease of intensity of radiation with thickness of the absorbing solution is proportional to the incident radiation as well as the concentration of the solution.

The expression of Beer-Lambert law is

$A = \log (I_0/I) = ECL$

Where, A = absorbance

$I_0$ = intensity of light incident upon sample cell

I = intensity of light leaving sample cell
C = molar concentration of solute
L = length of sample cell
E = molar absorptivity

From the Beer-Lambert law it is clear that greater the number of molecules capable of absorbing light of a given wavelength, the greater the extent of light absorption. This is the basic principle of UV spectroscopy.

Iron present in the water in ferric state is reduced to ferrous state by reacting with thiocynate and nitric acid. It forms a soluble chelated complex having intense red color with the ferrous ion present. This can be used for the spectrophotometric determination of iron in the water sample

**Instrumentation and working of UV spectroscopy**

Instrumentation and working of the UV spectrometers can be studied simultaneously. Most of the modern UV spectrometers consist of the following parts-

Light Source- Tungsten filament lamps and Hydrogen-Deuterium lamps are most widely used and suitable light source as they cover the whole UV region. Tungsten filament lamps are rich in red radiations; more specifically they emit the radiations of 375 nm, while the intensity of Hydrogen-Deuterium lamps falls below 375 nm.

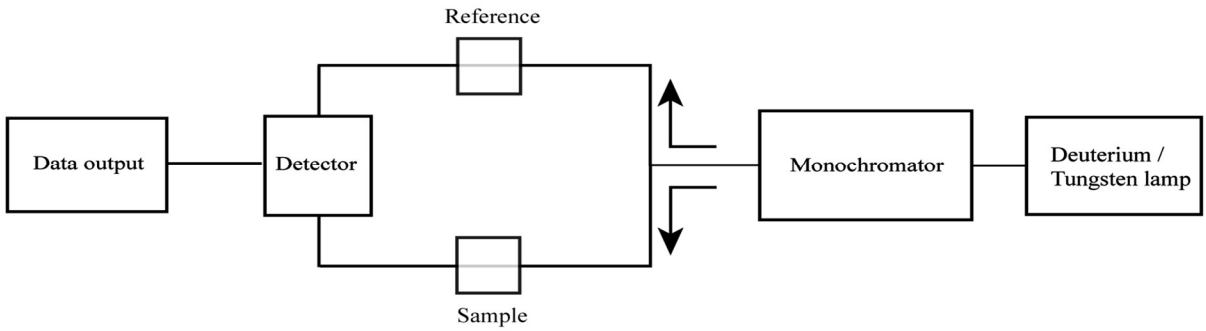
Monochromator- Monochromators generally composed of prisms and slits. The most of the spectrophotometers are double beam spectrophotometers. The radiation emitted from the primary source is dispersed with the help of rotating prisms. The various wavelengths of the light source which are separated by the prism are then selected by the slits such the rotation of the prism results in a series of continuously increasing wavelength to pass through the slits for recording purpose. The beam selected by the slit is monochromatic and further divided into two beams with the help of another prism.

Sample and reference cells- One of the two divided beams is passed through the sample solution and second beam is passed through the reference solution. Both sample and reference solution are contained in the cells. These cells are made of either silica or quartz. Glass can't be used for the cells as it also absorbs light in the UV region.

Detector- Generally two photocells serve the purpose of detector in UV spectroscopy. One of the photocell receives the beam from sample cell and second detector receives the beam from the reference. The intensity of the radiation from the reference cell is stronger than the beam of sample cell. This results in the generation of pulsating or alternating currents in the photocells.

Amplifier- The alternating current generated in the photocells is transferred to the amplifier. The amplifier is coupled to a small servometer. Generally current generated in the photocells is of very low intensity, the main purpose of amplifier is to amplify the signals many times so we can get clear and recordable signals.

Recording devices- Most of the time amplifier is coupled to a pen recorder which is connected to the computer. Computer stores all the data generated and produces the spectrum of the desired compound.




**Applications of UV-visible spectroscopy**

1. Detection of functional groups- UV spectroscopy is used to detect the presence or absence of chromophore in the compound. This is technique is not useful for the detection of chromophore in complex compounds. The absence of a band at a particular band can be seen as an evidence for the absence of a particular group. If the spectrum of a compound comes out to be transparent above 200 nm than it confirms the absence of –
a) Conjugation b) A carbonyl group c) Benzene or aromatic compound d) Bromo or iodo atoms.

2. Detection of extent of conjugation- The extent of conjugation in the polyenes can be detected with the help of UV spectroscopy. With the increase in double bonds the absorption shifts towards the longer wavelength. If the double bond is

increased by 8 in the polyenes then that polyene appears visible to the human eye as the absorption comes in the visible region.

3. Identification of an unknown compound- An unknown compound can be identified with the help of UV spectroscopy. The spectrum of unknown compound is compared with the spectrum of a reference compound and if both the spectrums coincide then it confirms the identification of the unknown substance.

4. Determination of configurations of geometrical isomers- It is observed that cis-alkenes absorb at different wavelength than the trans-alkenes. The two isomers can be distinguished with each other when one of the isomers has non-coplanar structure due to steric hindrances. The cis-isomer suffers distortion and absorbs at lower wavelength as compared to trans-isomer.

5. Determination of the purity of a substance- Purity of a substance can also be determined with the help of UV spectroscopy. The absorption of the sample solution is compared with the absorption of the reference solution. The intensity of the absorption can be used for the relative calculation of the purity of the sample substance.

**OBSERVATION**

| S.No. | Volume of iron (III) solution (Vi) (ml) | Conc. of ferric sulphate solution (ppm) $= \frac{V_i \, X \, C}{50}$ | Absorbance |
|---|---|---|---|
| 1. | 0.0 | | |
| 2. | 0.5 | | |
| 3. | 1.0 | | |
| 4. | 1.5 | | |
| 5. | 2.0 | | |
| 6. | 2.5 | | |
| 7. | Unknown sample | | |

## PROCEDURE

Prepare standard 100 ppm ferric sulphate solution by weighing 0.2160 g of Ammonium ferric sulphate in 2.5 ml conc. $H_2SO_4$ and dissolving with 250 ml of distilled water. Take 0.5, 1.0, 1.5, 2.0 and 2.5 ml of the ferric sulphate solution, through burette into separate 50 ml volumetric flask. Add 5 ml ammonium thio cyanate and 3 ml of 4 N nitric acid into each of volumetric flasks. Dilute the contents of each flask upto 50 ml with distilled water. Prepare a blank solution without the iron sample and measure the absorbance at 480 nm. Repeat the above procedure with suitable aliquot of water sample. Plot a calibration graph between absorbance and concentration of ferric sulphate, and compare this standard graph to determine the amount of iron in the unknown sample.

## CALCULATIONS

Weight of empty weighing bottle ($W_1$) = ---------- g

Weight of bottle + ammonium ferric sulphate ($W_2$) = ---------- g

Weight of bottle after transfer ($W_3$) = ---------- g

Weight of ammonium ferric sulphate ($W_2 - W_3$) = ---------- g

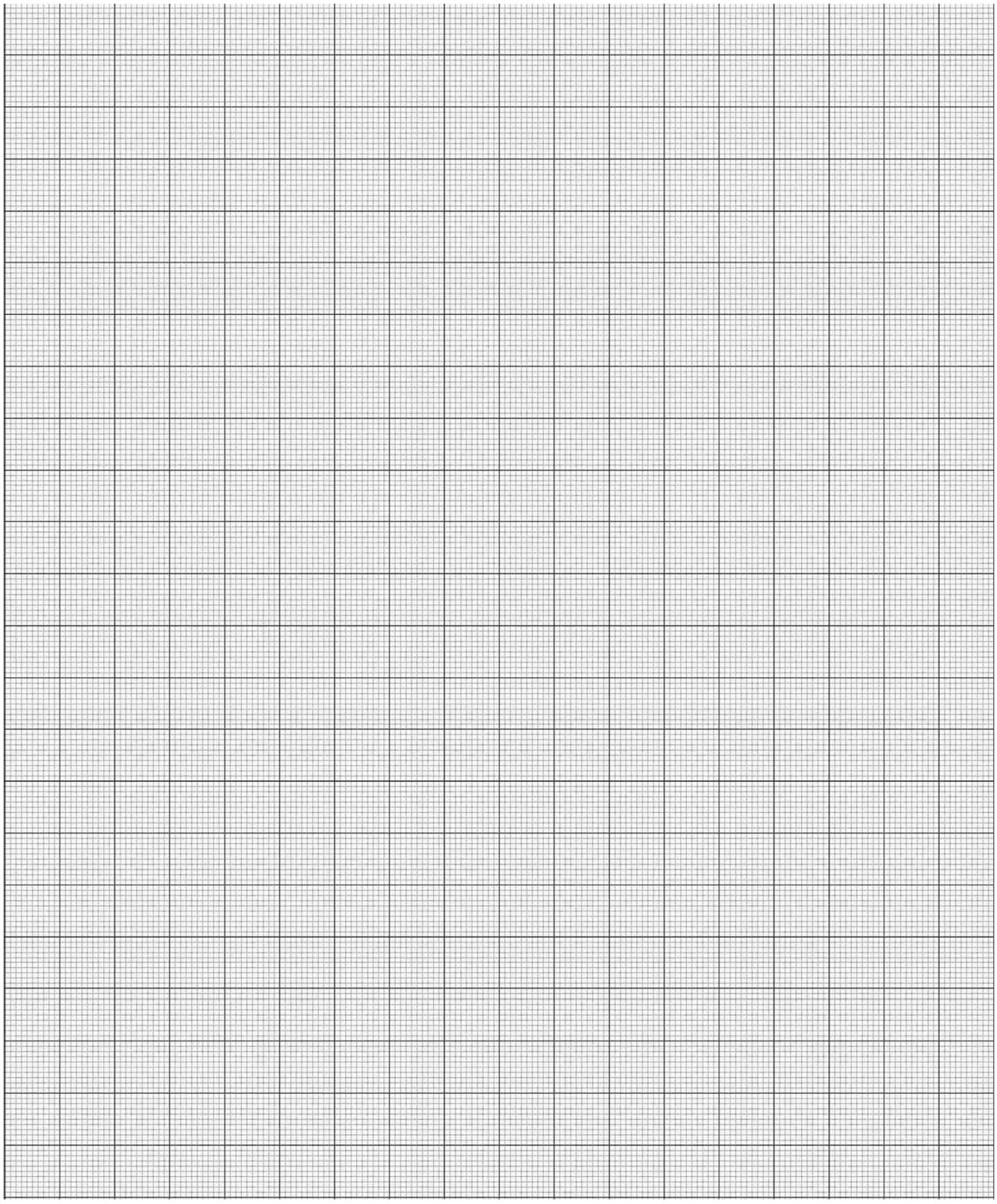
$$\text{Concentration of iron in solution (C)} = \frac{(W_2 - W_3)\ X55.85\ X\ 1000X1000}{250\ X\ 482.2}$$

= ppm

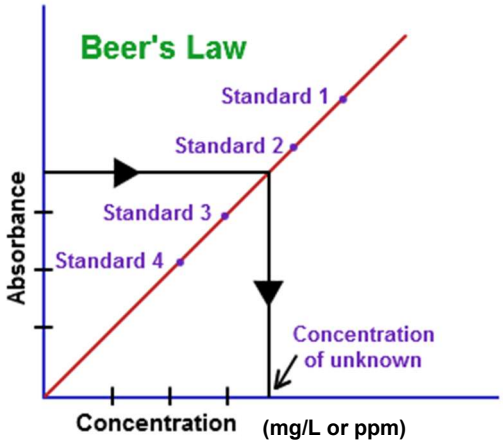
**Scale**

**In x-axis, 1 cm =**

**In y-axis, 1 cm =**



## MODEL GRAPH




| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Define absorbance.<br>2. What is a calibration plot? | 1. What are the components of a UV-visible spectrophotometer?<br>2. What is the role of a monochromator?<br>3. Give the Beer-Lambert's law.<br>4. Give the applications of a UV-visible spectrophotometer. |

## RESULT

The concentration of iron in the given sample was found to be _____ ppm.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT No.** **DATE:**

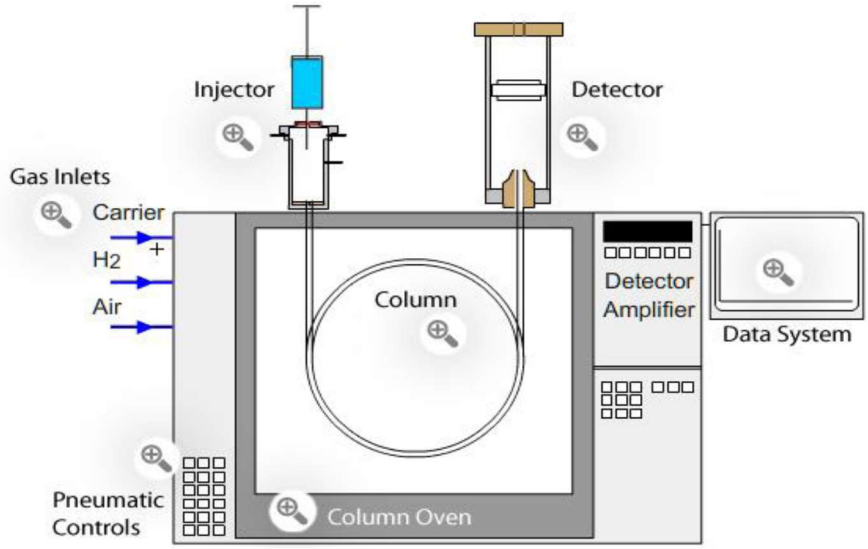
## DETERMINATION OF FATTYACID METHYL ESTER USING GAS CHROMATOGRAPHY

### AIM

To determine the amount of Fatty Acid Methyl Ester (FAME) in the given sample using Gas Chromatography.

### INSTRUMENTATION

Gas chromatography uses a gaseous mobile phase to transport sample components through either packed columns or hollow capillary columns containing a polymeric liquid stationary phase.




**GAS CHROMATOGRAPHY**

### GAS INLETS:

Gas is fed from cylinders through supply piping to the instrument. It is usual to filter gases to ensure high gas purity and the gas supply may be regulated at the bench to ensure an appropriate supply pressure. Required gases might include:
Carrier - ($H_2$, He, $N_2$)
Make-up gas - ($H_2$, He, $N_2$)
Detector Fuel Gas - ($H_2$ & Air, Ar or Ar & $CH_4$, $N_2$) depending on the detector type.

## PNEUMATIC CONTROLS:

The gas supply is regulated to the correct pressure (or flow) and then fed to the required part of the instrument. Control is usually required to regulate the gas coming into the instrument and then to supply the various parts of the instrument. A GC fitted with a Split/Splitless inlet, capillary GC column and Flame Ionization detector may have the following different gas specifications:
Carrier gas supply pressure, column inlet pressure (column carrier gas flow), inlet split flow, inlet septum purge flow, detector air flow, detector hydrogen flow, detector make-up gas flow.

## INJECTOR:

Here the sample is volatilized and the resulting gas entrained into the carrier stream entering the GC column.

Inlet types :

- Split / Splitless
- Programmed Thermal Vaporizing (PTV)
- Cool-on-column (COC) etc.
  The COC injector introduces the sample into the column as a liquid to avoid thermal decomposition or improve quantitative accuracy.

## COLUMN:

In GC, retention of analyte molecules occurs due to stronger interactions with the stationary phase than the mobile phase. This is unique in GC and, therefore, interactions between the stationary phase and analyte are of great importance. The interaction types can be divided into three broad categories:

- Dispersive
- Dipole
- Hydrogen bonding

The sample is separated into its constituent components in the column. Columns vary in length and internal diameter depending on the application type and can be either packed or capillary. Packed columns (typical dimension 1.5 m x 4 mm) are packed with a solid support coated with immobilized liquid stationary phase material

(GLC). Capillary columns (typical dimension 30 m x 0.32 mm x 0.1 mm film thickness) are long hollow silica tubes with the inside wall of the column coated with immobilized liquid stationary phase material of various film thickness.
Many different stationary phase chemistries are available to suit a host of applications. Columns may also contain solid stationary phase particles (GSC) for particular application types.

**COLUMN OVEN:**

Temperature in GC is controlled via a heated oven. The oven heats rapidly to give excellent thermal control. The oven is cooled using a fan and vent arrangement usually at the rear of the oven.

A hanger or cage is usually included to support the GC column and to prevent it touching the oven walls as this can damage the column.

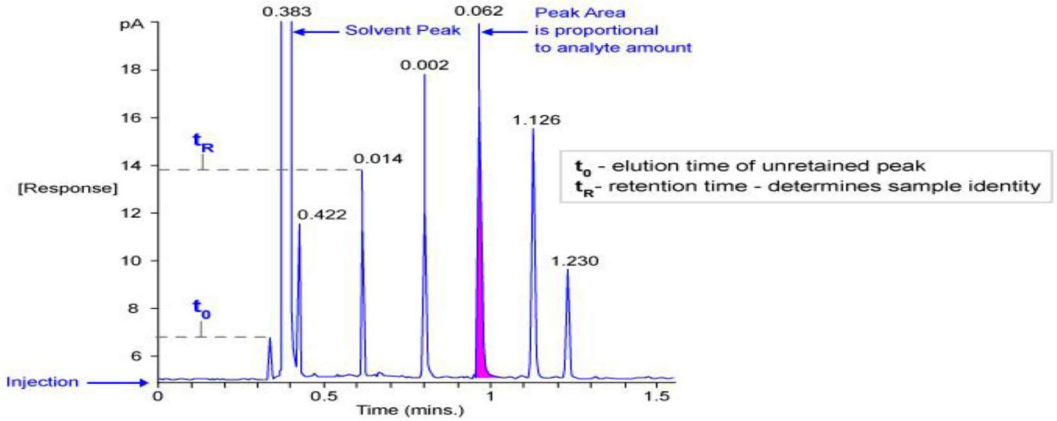
The injector and detector connections are also contained in the GC oven. For Isothermal operation, the GC is held at a steady temperature during the analysis. In temperature programmed GC (pTGC) the oven temperature is increased according to the temperature program during the analysis.

**DETECTOR:**

The detector responds to a physicochemical property of the analyte, amplifies this response and generates an electronic signal for the data system to produce a chromatogram.
Many different detector types exist and the choice is based mainly on application, analyte chemistry and required sensitivity – also on whether quantitative or qualitative data is required.
Detector choices include:
Flame Ionization (FID)
Electron Capture (ECD)
Flame Photometric (FPD)
Nitrogen Phosphorous (NPD)
Thermal Conductivity (TCD)
and Mass Spectrometer (MS)

## DATA SYSTEM:

The data system receives the analogue signal from the detector and digitizes it to form the record of the chromatographic separation known as the 'Chromatogram'. The data system can also be used to perform various quantitative and qualitative operations on the chromatogram – assisting with sample identification and quantitation.




**GAS CHROMATOGRAM**

The following information gives an indication of the type of sample (analyte) analyzed by GC

- Samples analyzed by GC must be volatile (have a significant **vapor pressure** below 250 °C).
- **Derivatization** to increase volatility is possible but can be cumbersome and introduces possible **quantitative** errors.
- Most GC analytes are under 500 Da Molecular Weight for volatility purposes.
- Highly polar analytes may be less volatile than suspected when dissolved in a polar solvent or in the presence of other polar species due to intermolecular forces such as hydrogen bonding.

## PRINCIPLE

When a sample is injected into the column, a carrier gas sweeps the sample through the column. An oven heats the system to vaporize the sample and speed its passage through the column. The different components of the sample will be separated by the column because each of the components "sticks" to the liquid coating

on the column packing differently. The greater the "stickiness," the longer it takes for a substance to pass through the column.

When a substance leaves the column, it is sensed by a detector. The detector generates a voltage that is proportional to the amount of the substance. The signal from the detector is then displayed by a chart recorder and fed into a computer.

Gas chromatographs are connected to a computer which displays the peaks of all the substances in the sample. This is called the chromatogram.

The kind of signal displayed by a chart recorder is more-or-less a triangular shaped peak. This is because the detector signal causes a vertical deflection of the recorder pen at the same time that the chart paper is moving under the pen.

The time that it takes a substance to pass through the instrument from injection to detection is called the retention time, $t_r$. The retention time, $t_r$, is measured from the injection point to the peak height. The peak height is the highest point of the peak and is the only reproducible point on the peak. Since the chart paper moves at a constant speed, the box divisions are proportional to $t_r$ and can measure the tr in box divisions for the experiment.

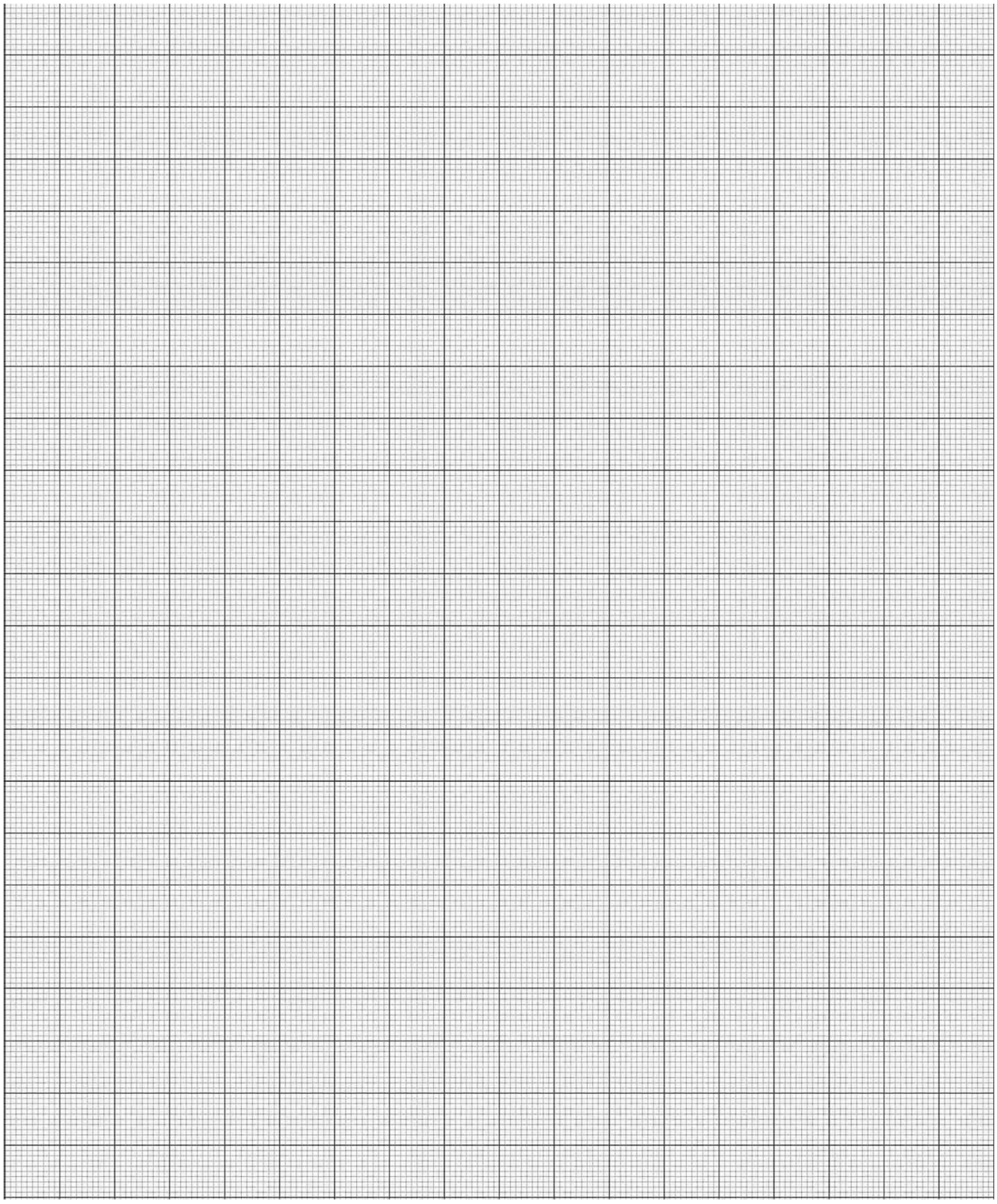
The amount of substance in a sample is proportional to the area under the peak of that substance. The proportionality constant is different for each substance and detector. Therefore, to do quantitative analysis by gas chromatography, we must first determine the proportionality constant for each substance in the sample.

Since most signals from the detector are too large for the recorder mechanism to handle, there is a switch on the chromatograph that attenuates or reduces the size of the signal. This is the attenuation (attn). For example, if the attenuation of a peak is 512, the signal has been reduced 512 times. So you must multiply the area under the peak (the area that you measure on the chart) by 512 to get the true area that is proportional to the original signal. Thus, it is area x attn that is proportional to the amount of substance injected.

**Scale**

**In x-axis, 1 cm =**

**In y-axis, 1 cm =**



There are two kinds of information available on the recordings in the packet.

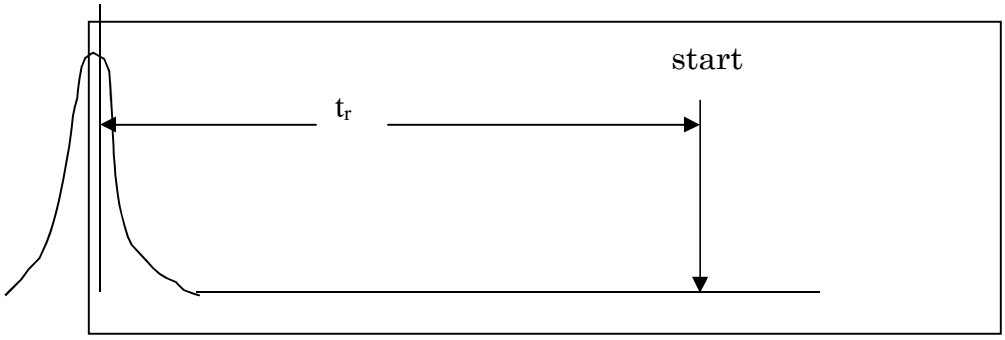
1. The retention times of each substance as determined from the distance in box units between the injection point and peak of each curve. As stated above, we will use box units for retention times because they are proportional to the time and serve us just as well.

2. The peak areas corresponding to injection of different amounts of each pure substance.

Plot a calibration line for each substance using Vernier's Graphical Analysis program. Then perform a linear regression on each line to determine the slope and y-intercept of the line. After the calibration lines are plotted and approved by the instructor, you will be given the chromatogram of an unknown mixture to identify. By determining the retention time of each peak of the unknown, you can identify the substance that the peak corresponds to. By measuring the area x attn of each *peak, you can use the formula of a straight line (y = mx + b) from the calibration line to determine how much of each substance was injected.

**DETERMINING RETENTION TIMES AND PREPARING CALIBRATION GRAPHS**

On the chromatogram of all the pure substances, measure the retention time of each peak in box units, to the nearest 0.1 box units, and record it directly on the graph near the peak. For each peak, the retention time is measured from injection




point to the top of the peak.

Using Graphical Analysis, you will plot the area x attn on the vertical axis (the y-axis) and the microliters ( μL) injected on the horizontal axis (the x-axis). A separate graph will be prepared for each substance.

**OBSERVATION**

Collect the data from the computer such as peak area, peak height, retention time, concentration.

| S.No. | Concentration (mg/L) | Peak area ($mm^2$) | Peak Height (mm) | Retention Time (min) |
|---|---|---|---|---|
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| Unknown | | | | |

## PROCEDURE

## PREPARATION OF FAME STANDARDS:

Weigh accurately 1mg of FAME (Fatty Acid Methyl Ester) and transfer into a vial. Then add 1 mL of Hexane to it. This is the stock solution (1mg/mL). From the stock solution prepare 0.1mg/ mL,0.3mg/ mL,0.5mg/ mL,0.7mg/ mL and 1mg/ mL of varying concentration.

## CONDITIONS FOR GC METHOD

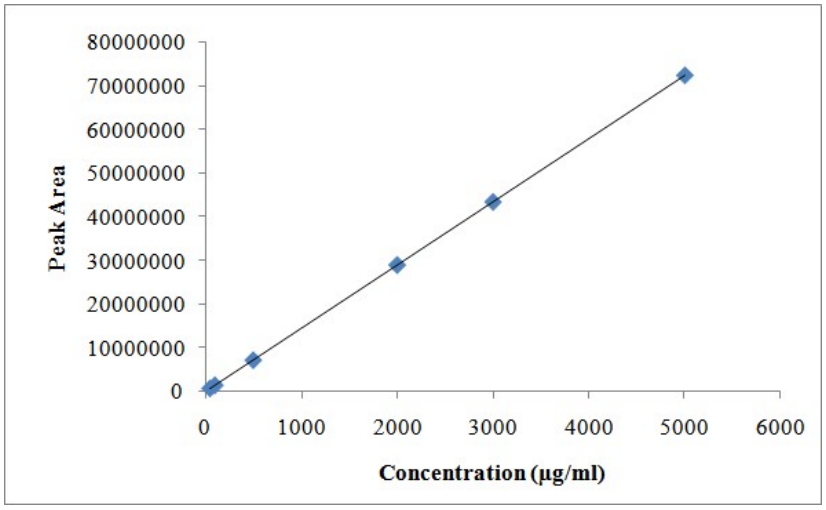
| | |
|---|---|
| Flow rate | : 1 mL/min |
| Inlet Temperature | : 250 ºC |
| Detector Temperature | : 300 ºC |
| Oven Temperature Programme | : 80ºC withhold time 2 min<br>Ramp 10 ºC/min at 200 ºC<br>Ramp 5 ºC/min at 240 ºC withhold time 8 min, Total run time 32 min. |

## OPERATION OF THE INSTRUMENT

For this experiment all the standards and samples are being run under the same conditions, so the method will be the same for all the runs.

1. Select FAME. M" in the "method" menu bar.
2. Go to "method" then "edit entire method" and make sure the parameters such as the solvent mixtures are correct.
3. Then select "FAME. S" in the "sequence" menu bar.
4. Go to "sequence" then "sequence template" to see the order of the samples and make sure the method is the correct for each run.
5. Then click run sequence.
6. Run the sequence two times.
7. Make sure that the peak heights of the repeated runs are within 5% of each other. If they are not, repeat the runs until they are reproducible.

## MODEL GRAPH




| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Give the working principle of a gas chromatograph.<br>2. What are carrier gases?<br>3. What is a calibration plot? | 1. What are the components of a gas chromatograph?<br>2. Define retention time.<br>3. Give the information obtained from a chromatogram. |

## RESULT

The concentration of the unknown sample of FAME was found to be _____ ppm.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT NO.** **DATE:**

## ESTIMATION OF ASPIRIN DRUG IN TABLETS USING pH METER

### AIM

To estimate the amount of Aspirin in the given tablet using pH meter

### APPARATUS

Pipette, Conical flask, Volumetric flask, Beaker etc.

### REAGENTS

Alcoholic KOH solution, oxalic acid, absolute alcohol

### PRINCIPLE

pH is the common way of expressing the hydrogen ion concentration $[H^+]$. pH is defined as: $pH = -\log [H^+]$

A pH meter is a scientific instrument that measures the hydrogen-ion concentration (or pH) in a solution, indicating its acidity or alkalinity. The pH meter measures the difference in electrical potential between a pH electrode and a reference electrode. It usually has a glass electrode plus a calomel reference electrode, or a combination electrode.

A pH meter measures essentially the electro-chemical potential between a known liquid inside the glass electrode (membrane) and an unknown liquid outside. Because the thin glass bulb allows mainly the agile and small hydrogen ions to interact with the glass, the glass electrode measures the electro-chemical potential of hydrogen ions or the potential of hydrogen.

**Aspirin** is also known as **acetyl salicylic acid** (**ASA**). Acetyl salicylic acid is a weak organic acid. It can be estimated by using a standard strong base either by titrimetry or by pH metry. Aspirin is a medication used to treat pain, fever and inflammation. Aspirin is also used to prevent heart attacks, strokes, and blood clot formation in people at high risk of developing blood clots.

## OBSERVATIONS AND CALCULATIONS

Weight of empty weighing bottle $(W_1)$ = ---------- g
Weight of bottle + Oxalic acid $(W_2)$ = ---------- g
Weight of bottle after transfer $(W_3)$ = ---------- g
Weight of Oxalic acid taken $(W_2 - W_3)$ = ---------- g

Concentration of Oxalic acid $(N_1) = \frac{(W_2 - W_3) \times 1000}{250 \times 63} =$

## TABLE 1: STANDARDIZATION OF KOH

| Sl.No. | Volume of standard Oxalic acid (ml) | Burette Reading | | Volume of KOH $(V_2)$ (ml). |
|---|---|---|---|---|
| | | Initial | final | |
| 1. | 25 | | | |
| 2. | 25 | | | |
| | | | | $V_2$ = --------- ml |

Concentration of Oxalic acid $(N_1)$ =

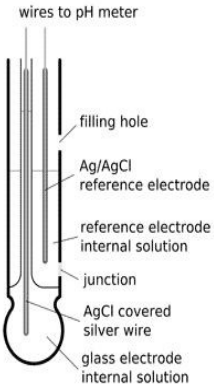
Volume of Oxalic acid $(V_1)$ =

Volume of KOH $(V_2)$ =

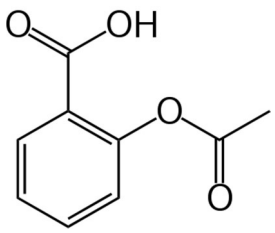
Concentration of KOH $(N_2)$ = $N_2 = \frac{N_1 \times V_1}{V_2}$

## PILOT TITRATION (A) SAMPLE vs ALCOHOLIC KOH

| Sl. No. | Volume of alc. KOH (ml) | pH |
|---|---|---|
| 1. | 0 | |
| 2 | 1 | |
| 3 | 2 | |
| 4 | 3 | |
| 5 | 4 | |
| 6 | 5 | |
| 7 | 6 | |
| 8 | 7 | |
| 9 | 8 | |
| 10 | 9 | |
| 11 | 10 | |




Aspirin




**PROCEDURE**

**STANDARDIZATION OF ALCOHOLIC KOH**

Prepare 0.05 N oxalic acid by dissolving 0.7875g of oxalic acid in 250 ml water. Pipette out 25 ml of this solution into a clean conical flask. Add 2drops of phenolphthalein indicator. It is titrated against the alcoholic KOH solution taken in the burette. The end point is appearance of pink color.

**ACETYL SALICYLIC ACID vs KOH**

Accurately weigh the given tablet and grind it in a porcelain mortar to powder. Dissolve the powder in 25 ml of ethanol and transfer the whole into a 100 ml volumetric flask and make it up to the mark using distilled water. Pipette out 25 ml of the sample solution into a beaker. Immerse the electrode in to the drug solution. Note the pH value after the addition of each ml of N/20 alcoholic KOH solution from the burette after well stirring. Continue the addition up to 10 ml. Note the jump in pH value. Repeat the experiment, by adding 0.1ml after the jump in pH value.

Draw a graph by plotting pH vs. the volume of alcoholic KOH solution. Calculate the strength of acetyl salicylic acid in the given drug sample using the volume of alcoholic KOH from the graph.

**(B) ACTUAL TITRATION**

| S. No. | Volume of alc. KOH (ml) | pH | ΔpH / ΔV |
|---|---|---|---|
| 1. | 0 | | |
| 2. | 1 | | |
| 3. | 2 | | |
| 4. | 3 | | |
| 5. | 4 | | |
| 6. | | | |
| 7. | | | |
| 8. | | | |
| 9. | | | |
| 10. | | | |
| 11. | | | |
| 12. | | | |
| 13. | | | |
| 14. | | | |
| 15. | | | |
| 16. | | | |
| 17. | | | |
| 18. | | | |
| 19. | | | |
| 20. | | | |
| 21. | | | |
| 22. | | | |
| 23. | | | |
| 24. | | | |
| 25. | | | |
| 26. | | | |
| 27. | | | |
| 28. | | | |
| 29. | | | |
| 30. | | | |

**CALCULATION**

Volume of sample drug taken = 25 ml

Concentration of alcoholic KOH = $N_2$ = ------- N

Volume of KOH consumed for drug = $V_2$ = --------- ml (from graph)

Concentration of aspirin in drug ($N_3$) = $\frac{N_2 \text{ X } V_2}{25}$ =

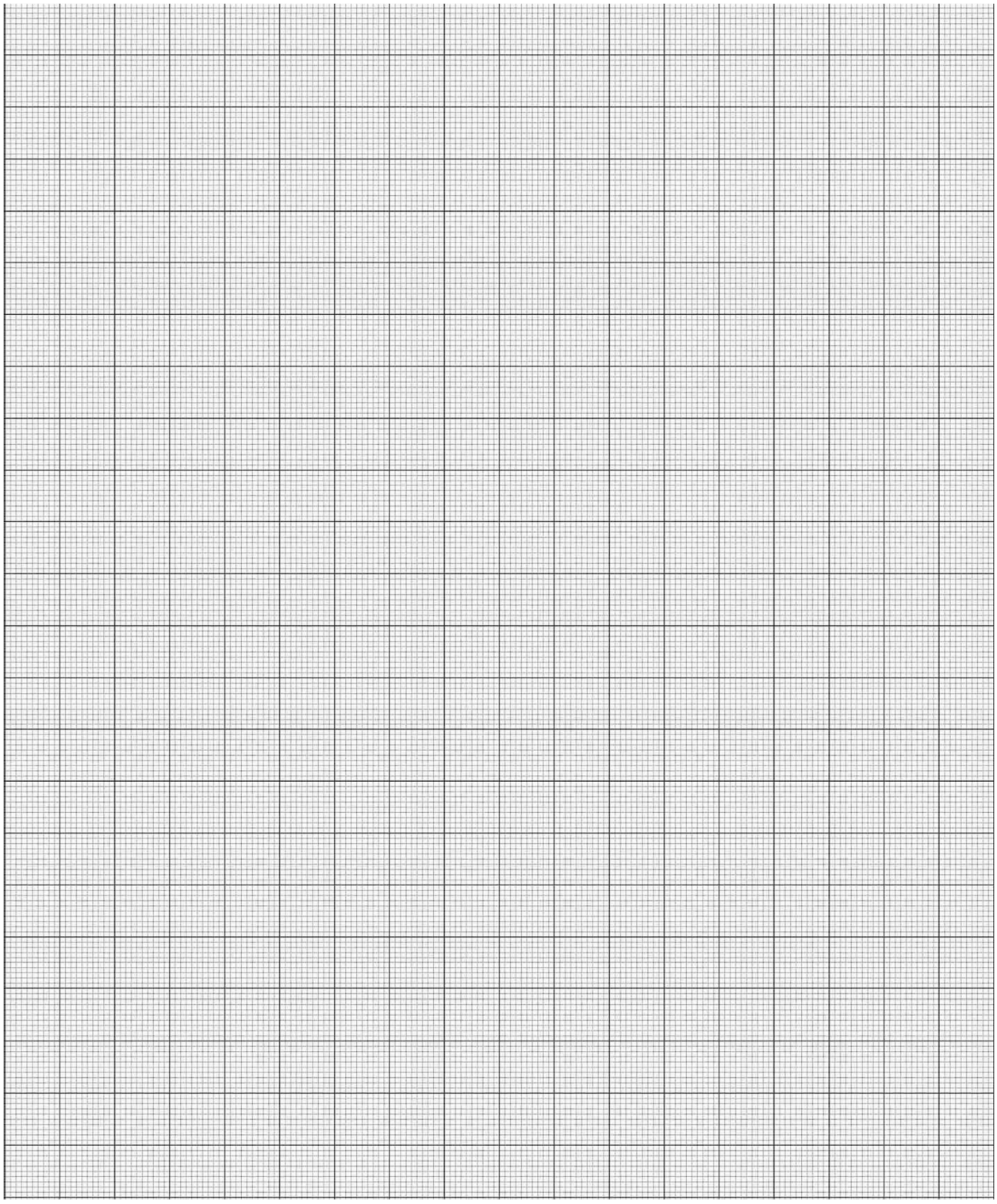
Amount of the aspirin in the drug = $\frac{N_3 \text{ X } 180 \text{ X } 100 \text{ X } 1000}{1000}$

= $N_3$ x 180 x 100 =

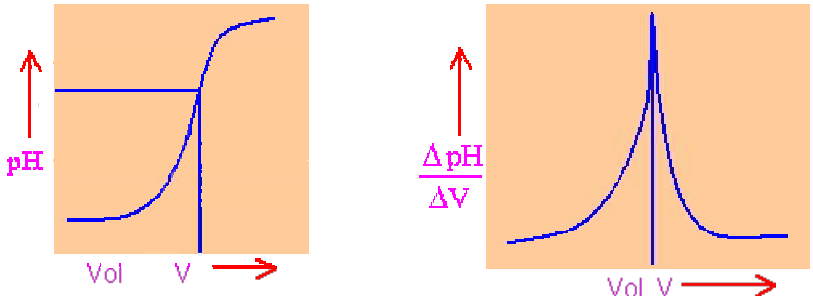
**Scale**

**In x-axis, 1 cm =**

**In y-axis, 1 cm =**



## MODEL GRAPH




| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. Give the chemical name of aspirin.<br>2. Define pH.<br>3. Give the uses of aspirin. | 1. Differentiate between pH-based titration and a volumetric titration.<br>2. Explain the working of a pH meter. |

## RESULT

The amount of aspirin in the given sample of drug was found to be ---------- mg.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |

**EXPERIMENT No.** **DATE:**

## ESTIMATION OF MANGANESE IN THE GIVEN SAMPLE OF ORE

### AIM

To estimate the percentage of manganese present in the given sample of ore.

### APPARATUS

Burette, Pipette, Volumetric flasks, Heating mantle, Conical Flask, Funnel etc.

### REAGENTS

Oxalic acid, Sulfuric acid, $KMnO_4$

### PRINCIPLE

Pyrolusite consists of $MnO_2$. Since $MnO_2$ has oxidizing property, its determination can be carried out by a suitable redox reaction, $MnO_2$ quantitatively oxidizes oxalic acid in sulfuric acid medium. The unreacted oxalic acid can be estimated by permanganometry. Knowing the amount of oxalic acid consumed. The quantity of $MnO_2$ in the given weight of sample is calculated.

$$MnO_2 + H_2C_2O_4 + 2H^+ \rightarrow Mn^{2+} + 2H_2O + 2CO_2$$

### PROCEDURE

Prepare 0.1N solution of oxalic acid by dissolving 1.575g of oxalic acid in 250 ml of water. Pipette out 50 ml of this solution into a clean conical flask. Then add 25 ml of 6N dilute sulfuric acid. Heat gently up to warm condition, and then titrate the warm solution against $KMnO_4$ solution until just appearance of permanent pale pink color.

### ESTIMATION OF MANGANESE IN THE ORE

Weigh out accurately 0.25 g of given sample of ore into a clean conical flask. Add 25 ml of dilute sulfuric acid and boil for 10 minutes. Cool the flask then add 50 ml of oxalic acid solution through pipette and heat gently until black residue disappears. Titrate the contents of the conical flask against already standardized $KMnO_4$ taken in the burette, until the appearance of permanent pale pink color.

## OBSERVATIONS AND CALCULATIONS

Weight of empty weighing bottle $(W_1)$ = ---------- g
Weight of bottle + Oxalic acid $(W_2)$ = ---------- g
Weight of bottle after transfer $(W_3)$ = ---------- g
Weight of Oxalic acid taken $(W_2 - W_3)$ = ---------- g

Concentration of Oxalic acid $(N_1) = \frac{(W_2 - W_3) \times 1000}{250 \times 63} =$

## TABLE 1: STANDARDIZATION OF $KMNO_4$

| S .No. | Volume of standard Oxalic acid (ml) | Burette Reading (ml) | | Volume of $KMnO_4$ $(V_2)$ (ml). |
|---|---|---|---|---|
| | | Initial | final | |
| 1. | 50 | | | |
| 2. | 50 | | | |
| | | | | $V_2$ = --------- ml |

Concentration of Oxalic acid $(N_1)$ =

Volume of Oxalic acid $(V_1)$ = 50 ml

Volume of $KMnO_4$ $(V_2)$ =

Concentration of $KMnO_4$ $(N_2)$ $= N_2 = \frac{N_1 \times V_1}{V_2}$ =

## TABLE 2: DETERMINATION OF MANGANESE IN ORE

Sample vs $KMnO_4$

| Sample No. | Volume of sample soln. | Burette Reading (ml) | | Volume of $KMnO_4$ ,$V_3$ (ml). |
|---|---|---|---|---|
| | | Initial | final | |
| 1. | 50 | | | |

## CALCULATION

Weight of ore sample ($w_1$) = ----------g

Volume of $KMnO_4$ used for blank ($V_2$) = --------- ml

Volume of $KMnO_4$ used for sample 1 ($V_3$) = ---------- ml

Volume of $KMnO_4$consumed by ($V_2 - V_3$) = ----------ml

(1 ml of 1 N $KMnO_4$ = 1 ml of 1 N $H_2C_2O_4$ = 27 mg of Mn)

Percentage of Mn in given sample of ore 1 = $\frac{27 \times N_2 \times (V_2 - V_3)}{(w_1 \times 1000)} \times 100$

| Pre-Lab Questions | Post Lab Questions |
|---|---|
| 1. What is pyrolusite?<br>2. What is permanganometry? | 1. Give the redox reaction used in estimation of Mn by permanganometry.<br>2. Give the applications of permanganometry. |

## RESULT

The percentage of manganese in the given sample of ore was found to be _____ %.

## REPORT

| Particulars | Max. Marks | Marks Obtained |
|---|---|---|
| Pre-Lab Q & A | 5 | |
| Post-Lab Q & A | 5 | |
| Experiment Performance | 10 | |