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Balaji Institute of Technology & Science
Laknepally, NARSAMPET, Warangal – 506331
Accredited By NAAC & ISO 9001:2015 Certified Institution
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ENGINEERING CHEMISTRY
LAB MANUAL

Course Code	**:**	**CH106BS/CH206BS**
Regulations	**:**	**R18**
Class	**:**	**B.Tech**
Branch	**:**	**CE/EEE/ME/ECE/CSE**

Prepared by

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Vision of Department
To establish a centre of excellence in basic sciences such as Mathematics, Physics , Chemistry & Environmental Sciences that provides foundation for engineering studies and also in English Language Communication Skills that helps students to express themselves effectively and to create engineers with proficiency in engineering fundamentals – experimental, analytical, computational and designing abilities.

Mission of Department
M1: To create academic excellence in fundamental sciences and communication skills for the Students.
M2: To encourage advanced teaching learning process, quality based knowledge and Quality research at individual, department and institutional level.
M3: To impart personality development skills to students that will help them to succeed and lead.

Course Objectives
➢ Estimation of hardness and chloride content in water to check its suitability for drinking purpose.
➢ To determine the rate constant of reactions from concentrations as on function of time.
➢ The measurement of physical properties like adsorption and viscosity.
➢ To synthesize the drug molecules.
➢ To check the purity of organic molecules by thin layer chromatographic (TLC) technique.

Program Outcomes	
PO1:	**Engineering knowledge**: Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.
PO2:	**Problem analysis**: Identify, formulate, review research literature, and analyze complex engineering problems reaching substantiated conclusions using first principles of mathematics, natural sciences, and engineering sciences.
PO3:	**Design/development of solutions**: Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for the public health and safety, and the cultural, societal, and environmental considerations.
PO4:	**Conduct investigations of complex problems**: Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data, and synthesis of the information to provide valid conclusions.
PO5:	**Modern tool usage**: Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modeling to complex engineering activities with an understanding of the limitations.
PO6:	**The engineer and society**: Apply reasoning informed by the contextual knowledge to assess societal, health, safety, legal and cultural issues and the consequent responsibilities relevant to the professional engineering practice.
PO7:	**Environment and sustainability**: Understand the impact of the professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
PO8:	**Ethics**: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.
PO9:	**Individual and team work**: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.
PO10:	**Communication**: Communicate effectively on complex engineering activities with the engineering community and with society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.
PO11:	**Project management and finance**: Demonstrate knowledge and understanding of the engineering and management principles and apply these to one's own work, as a member and leader in a team, to manage projects and in multidisciplinary environments.
PO12:	**Life-long learning**: Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change.

Course Outcomes	
CO1:	Determine parameters like hardness and chloride content in water.
CO2:	Explain various titrations by different types of analysis using instrumental methods.
CO3:	Prove rate constant of a reaction from concentration – time relationships.
CO4:	Find physical and chemical properties like acid value ,adsorption, surface tension, partition coefficient, acid value and viscosity.
CO5:	Identify Rf values of some organic molecules by TLC technique and Analyze the synthesis of Drug molecules.

ATTAINMENT OF COURSE OUTCOMES & PROGRAM OUTCOMES												
COs	**PO1**	**PO2**	**PO3**	**PO4**	**PO5**	**PO6**	**PO7**	**PO8**	**PO9**	**PO10**	**PO11**	**PO12**
CO1	**3**	**2**	**-**	**-**	**-**	**-**	**2**	**-**	**-**	**-**	**-**	**-**
CO2	**2**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**
CO3	**2**	**-**	**-**	**-**	**-**	**-**	**2**	**-**	**-**	**-**	**-**	**-**
CO4	**3**	**3**	**-**	**2**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**
CO5	**3**	**-**	**2**	**2**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**
Average	**2.6**	**2.5**	**2**	**2**	**-**	**-**	**2**	**-**	**-**	**-**	**-**	**-**

1 = Slite (Low) **2 = Moderate (Medium)** **3 = Substantial (High)**

BALAJI GROUP OF INSTITUTION

LAKNEPALLY, NARSAMPET, WARANGAL

CERTIFICATE

Name of the Laboratory.....

This is to certify that, this is a bonafied record of laboratory experimental work done by .....bearing Hall Ticket Number ..... B.Tech/ MBA/ MCA ..... Semester student in the department of..... ofthis college during the year.....

(Signature of the concerned teacher)

APPROVED/ NOT APPROVED

External Examiner *Internal Examiner*

Department of……..

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4	Estimation of Acetic acid by Conductometric titrations				
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GENERAL PRECAUTIONS FOR AVOIDING ACCIDENTS IN A CHEMISTRY LABORATORY

- Use full length protective coat/ an apron.
- Wear safety goggles for preventing eye injuries by splashing of chemicals.
- A pair of gloves must be used when handling poisonous or toxic chemicals.
- A rubber bulb should be used for pipetting solutions.
- Before leaving the laboratory, wash and clean every apparatus, wipe the table and keep all the apparatus in its original position.
- Laboratory must be kept clean and tidy at all times.
- Each laboratory must be equipped with first aid box.
- Strong acids used for conducting experiments should be diluted before being poured into the sink.
- When the burner is not being used, lower the flame. As soon as the days work is over close the gas tap.
- Never handle chemicals with your finger. Always use fresh, clean spatula.
- Never try to lift the glass bottle holding the neck alone.
- Don't eat, drink in the laboratory.
- Do not throw solid wastes or filter paper or other wastes into the sink.

LAB SAFETY RULES

- **ALWAYS** wear your apron or protective clothing when working with chemicals.
- **ALWAYS** tie back loose hair.
- **ALWAYS** wear goggles or safety glasses to prevent getting materials in your eyes.
- **ALWAYS** read the labels an heed all warnings.
- **NEVER** eat, drink of smell the chemicals. Rather carefully "fan" the fumes to your nose.
- **NEVER** look directly into a test tube or flask. Look at the contents from the side.
- **NEVER** play around during experiments.
- **ALWAYS** wash your hands after handling lab materials.

VOLUMETRIC ANALYSIS

BASIC CONCEPTS OF VOLUMETRIC ANALYSIS

Chemical analysis of the compounds is carried out in two ways

1. **Qualitative analysis.**
2. **Quantitative analysis.**

Qualitative analysis shows what element a given contains.

Quantities analysis determines the quantity of a particular component present in substance.

It is carried out in two ways

1. **Gravimetric analysis.**
2. **Volumetric analysis.**

Gravimetric analysis involves the estimation of the amount of a given compound from the results of weighing.

Volumetric analysis is based on the measuring the volume of the solution of a substance.

Terms involved in volumetric analysis

1. **Titration:** The process of finding out the volume of one of the solution required to react completely with a definite volume of one the other solution of known concentration is called titration.
2. **Titrant:** The solution of known strength is called titrant.
3. **Titrate:** The solution whose concentration to be estimated.
4. **Indicator:** The reagent which indicates the endpoint or equivalent point of the titration.

The strength of concentration of a solution is expressed in the following ways.

NORMALITY: Number of gram equivalents of the substance dissolved per liter of the solution is called Normality. It is denoted by N

$$\textbf{Normality} = \frac{W_{solute}}{E_{solute}} \times \frac{1}{V_{solvent\ (in\ lit)}}$$

Where E is Gram equivalent weight

MOLARITY: Number of grams moles of a solute dissolved per liter of solution is called Molarity. It is denoted by M

$$\textbf{Morality} = \frac{W_{solute}}{M_{solute}} \times \frac{1}{V_{solvent\ (in\ lit)}}$$

Where M is Gram molecular weight

MOLALITY: It is the number of mole of the substance dissolved in 1kg of the solvent it is denoted by (m).

$$\textbf{Molality} = \frac{W_{solute}}{M_{solute}} \times \frac{1}{W_{solvent\ (in\ kg)}}$$

Standard Solution:

A solution whose concentration is known is called a standard solution. Again there are two types of standard solutions depending on the nature of the substance.

1. Primary Standard Substances:

Any substance stable, pure, readily soluble in water, with high equivalent weight and the composition of its solution should not change on standing or during storage is called primary standard substance.

For example crystalline oxalic acid, potassium dichromate and anhydrous sodium carbonate etc. are the primary standard substances.

2. Secondary Standard Substances:

The substance which do not fulfill the above mentioned requirements of primary standard substances, their solutions are not directly prepared by weighing and the exact strength of the solution is found by titrating it against some primary standard is called secondary standard substance and the process is called standardization.

Common secondary standard substances are NaOH, KOH which are hygroscopic, $KMnO_4$ which undergoes auto decomposition in permanganate solution on standing and inorganic acids like HCI, H_2SO_4 whose concentrations are known approximately.

Types of Titrations

Depending on the nature of chemical reaction involved, the volumetric titrations are classified into the following types.

1. **Acid Base Titrations**
2. **Oxidation-Reduction Titrations (Redox Titrations)**
3. **Complexometric Titrations**
4. **Precipitation titrations.**

1. **Acid Base Titrations:** Acid-base titrations, in which an acidic or basic titrant reacts with an analyte that is a base or an acid.
2. **Oxidation-Reduction Titrations (Redox Titrations):** The titrations in which reducting agent is titrated against an oxidizing agent and vice versa are called redox titrations
3. **Complexometric Titrations:** The titrations involving the formation of a stable soluble complex between the metal and the complexing reagent. The metal ion is called central atom and the complexing reagent is called ligand.
4. **Precipitation titrations:** Precipitation titrations, in which the analyte and titrant react to form a precipitate.

DETERMINATION OF TOTAL HARDNESS OF WATER BY COMPLEXOMETRIC METHOD USING EDTA

Aim: To estimate the hardness of the given water sample by complexometric method using EDTA.

Instruments and Apparatus: Burette, pipette, conical flask, standard flask and burette stand.

Chemicals required: Magnesium Sulphate Heptahydrate ($MgSO_4.7H_2O$), Ethylene Diamine Tetra Acetic Acid (EDTA), Eriochrome Black T (EBT) indicator, Ammonia buffer solution, hard water and distilled water.

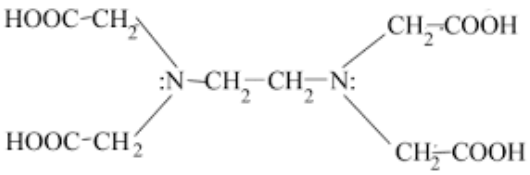
Principle: Hard water which contains Calcium and magnesium ions forms a wine red coloured complex with the Eriochrome Black-T indicator. EDTA forms a colourless stable complex with free metal ions like Ca^{2+} and Mg^{2+}.

Metal + Indicator ------→ Metal Indicator complex
(Blue) (Wine red colour)

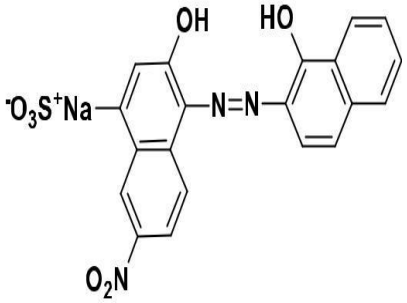
When EDTA is added from the burette, it extracts the metal ions from the metal ion-indicator complex thereby releasing the free indicator (The stability of metal ion-indicator complex is less than that of the metal ion-EDTA complex, and hence EDTA extracts metal ion form the ion-indicator complex).

EDTA + Metal indicator complex ------→ Metal ion – EDTA + Indicator
(Wine red colour) (Blue)

The reaction take place at a pH=10 and the buffer is made by Ammonium chloride and Ammonia solution.



structure of EDTA



Structure of EBT

Procedure

Preparation of Standard MgSO4 .7H_2O solution (0.05N):

Weight accurately 0.6162 g of Magnesium Sulphate and transfer it in to a clean 100 ml volumetric flask with the help of funnel, dissolved this crystal in minimum amount of distilled water and then made up to the mark with distilled water. It is shaken well to get uniform concentration and calculate the normality of the prepared Standard MgSO4 solution.

Normality of Standard $MgSO_4.7H_2O$ solution = 0.05 N

W= weight of $MgSO_4.7H_2O$ = ?

246.48= Molecular weight of $MgSO_4.7H_2O$

123.24=Equivalent weight of $MgSO_4.7H_2O$

$$\textbf{Normality} = \frac{Weight}{\text{Gr.Eq.wt}} \text{ x } \frac{1}{V}$$

$$\textbf{Weight= Normality x Gr. Eq.wt x } \frac{\text{V}}{1}$$

Titration I: Standardisation of EDTA Solution

Pipette out 20 ml magnesium sulphate solution in to a clean conical flask. To this add 2ml of buffer solution and 3 to 4 drops of EBT indicator. Then there develops a wine red colour. This solution is titrated against EDTA solution taken in the burette. The end point in this reaction is conversion of wine red colour into blue. Repeat the titrations till concurrent values are obtained. The normality of EDTA is calculated.

S. No.	Volume of Standard $MgSO_4$ (V_1) (ml)	Burette reading(ml)		Volume of EDTA Solution (V_2) (ml)
		Initial	Final	

Volume of standard $MgSO_4$ solution (V_1) = ml

Strength of standard $MgSO_4$ solution (N_1) = N

Volume of EDTA Solution (V_2) = ml

Strength of EDTA Solution (N_2) = N

$$N_1 V_1 = N_2 V_2$$

$$N_2 = N_1 V_1 / V_2$$

Titration II: Estimation of Total hardness of Water

Pipette out 20 ml of hard water sample in to a clean conical flask. To this add 2ml of buffer solution followed 3 to 4 drops of EBT indicator and titrate the solution till a clear blue colour persists. This marks the end point of the titration. Repeat the titrations for constant values and calculate the amount of total hardness present in the given sample.

S. No.	Volume of hard water (V_3) (ml)	Burette reading(ml)		Volume of EDTA Solution (V_2) (ml)
		Initial	Final	

Volume of hard water solution (V_3) = ml

Strength of hard water solution (N_3) = N

Volume of EDTA Solution (V_2) = ml

Strength of EDTA Solution (N_2) = N

$N_2 V_2 = N_3 V_3$

$N_3 = N_2 V_2 / V_3$

Amount of total hardness present in the given sample= N_3 x100 x 1000 = ppm

Titration III: Estimation of Permanent Hardness

Transfer 100 ml of the given sample water into a beaker and boil it gently for 20 minutes. Cool and filter it directly into a 100ml standard flask. Make up the solution with distilled water and mix well. Pipette out 20 ml of this solution into a clean conical flask. To this add 2ml of buffer solution followed 3 to 4 drops of EBT indicator and titrate the solution till a clear blue colour persists. This marks the end point of the titration. Repeat the titrations for constant values and calculate the amount of permanent hardness present in the given sample.

S. No.	Volume of sample water (V_4) (ml)	Burette reading(ml)		Volume of EDTA Solution (V_2) (ml)
		Initial	Final	

Volume of water solution (V_4) = ml

Strength of water solution (N_4) = N

Volume of EDTA Solution (V_2) = ml

Strength of EDTA Solution (N_2) = N

$$N_2 V_2 = N_4 V_4$$

$$N_4 = N_2 V_2 / V_4$$

Calculation:

Amount of permanent hardness present in the given sample= N_3 x100 x 1000 = ppm

Temporary hardness= Total hardness – Permanent hardness = ppm

Result: Total hardness of water sample = ppm

Permanent hardness of water sample = ppm

Temporary hardness of water sample = ppm

DETERMINATION OF CHLORIDE CONTENT OF WATER BY ARGENTOMETRY

Aim:

To determine the amount of chloride (in the form of Cl^-) present in the given water sample by Mohr's method.

Principle:

If water containing chlorides is titrated with silver nitrate solution, chlorides are precipitated as white silver chloride. Potassium chromate is used as indicator, which supplies chromate ions. As the concentration of chloride ions approaches extinction, silver ion concentration increases to a level at which reddish brown precipitate of silver chromate is formed indicating the end point.

$$Ag^+ + Cl^- \rightarrow AgCl(s)$$

$$2Ag^+ + CrO_4^{-2} \rightarrow Ag_2CrO_4(s)$$

Apparatus:

Burette, Pipettes, Erlenmeyer flasks, Measuring cylinder

Chemicals:

Chloride free distilled water, Standard silver nitrate solution (0.0141N), Potassium chromate indicator, Acid or alkali for adjusting pH.

Procedure:

Take 50mL of sample (V) and dilute to 100mL. If the sample is coloured add 3mL of aluminium hydroxide, shake well; allow to settle, filter, wash and collect filtrate. Sample is brought to pH 7-8 by adding acid or alkali as required. Add 1mL of indicator (Potassium chromate). Titrate the solution against standard silver nitrate solution until a reddish brown precipitate is obtained. Note down the volume (V_1). Repeat the procedure for blank and note down the volume (V_2).

Observation

S. No.	Volume of sample water (ml)	Burette reading(ml)		Volume of Silver nitrate (ml)
		Initial	Final	

$V =$

$V_1 =$

$V_2 =$

$N =$

$$\text{Chloride in mg/L} = \frac{(V_1 - V_2) \times N \times 35.46 \times 1000}{V}$$

$$= \frac{(V_1 - V_2) \times 500}{V} = \ldots\ldots\ldots \text{mg/L}$$

Results: Amount of Chloride ion present in water sample is:_____ mg/L

ESTIMATION OF HCL BY CONDUCTOMETRIC TITRATIONS

Aim: To determination the strength of given HCl solution by titrating against standard NaOH solution conductometrically.

Instruments and Apparatus: Burette, pipette, glass rod, beaker, standard flask, conductivity meter and conductivity cell with known cell constant.

Chemicals required: Hydrochloric acid (HCl), Oxalic acid and sodium hydroxide.

Principle:

According to Kohlarausch's law electrical conductivity of a solution depends on number of the ions present in it. In the titration of strong acid HCl with Strong base NaOH solution, before addition of NaOH solution there will be high conductance due to presence of large number H^+ of ions in it. Gradual addition of NaOH solution decreases the conductance due to the combination of H^+ ions with OH^- ions of the base to form undissociated water molecule. The conductance of solution decreases till neutralization point is reached and increases quickly due to free Na^+ and OH^- ions are added excess of NaOH solution. A plot of conductivity Vs volume of NaOH added will consist of two straight lines intersecting at the equivalent point. The shape of plot is V.

$$\mathbf{HCl + NaOH \rightarrow NaCl + } \boldsymbol{H_2O}$$

Procedure:

Preparation of standard oxalic acid solution (0.1 N):

Weigh 0.63 gram of oxalic acid crystals into a clean 100 ml standard flask, dissolve in small amount of water and make up the solution up to the mark with distilled water. Shake the flask well for uniform concentration.

Normality of Oxalic Acid =0.1 N, W= weight of Oxalic Acid =?

126.07= Molecular weight of Oxalic Acid 63=Equivalent weight of Oxalic Acid

$$\textbf{Normality} = \frac{\boldsymbol{Weight}}{\textbf{Gr.Eq.wt}} \textbf{ x } \frac{\mathbf{1}}{\boldsymbol{V}}$$

$$\textbf{Weight= Normality x Gr. Eq.wt x } \frac{\mathbf{V}}{\mathbf{1}}$$

Titration I: Standardisation of NaOH solution

Fill the burette with given NaOH solution. Pipette out 20 ml of oxalic acid into a conical flask and add 2 to 3 drops of phenolphthalein indicator and titrate the colourless solution against NaOH till pale pink colour is obtained as end point. Repeat the titration to get concurrent values.

S. No.	Volume of Oxalic acid solution (ml) (V_1)	Burette reading(ml)		Volume of NaOH (ml) (V_2)
		Initial	Final	

Volume of standard Oxalic acid solution (V_1) = ml

Strength of standard Oxalic acid solution (N_1) = N

Volume of Sodium hydroxide (V_2) = ml

Strength of Sodium hydroxide (N_2) = N

$$N_1 V_1 = N_2 V_2$$

$$N_2 = N_1 V_1 / V_2$$

Titration II: Determination of strength of HCl by conductivity meter

Transfer the given unknown HCl solution into a clean 100 ml standard flask and make up the solution up to mark with distilled water and mix well to obtain uniform concentration. Pipette out 20ml of the given acid into a clean 100ml beaker, dip the conductivity cell in it and measure the conductance initially. Fill the burette with standard NaOH solution and rundown into the beaker (at a time 1 ml) with gentle stirring of the contents of the beaker and note the conductance after each addition. The measured conductance are recorded and tabulated. Take about 20 readings, by plotting the graph (conductivity Vs volume of NaOH) we can get the amount of NaOH required for the neutralization of HCl taken in the beaker (i.e. end point) and calculate the concentration of HCl solution from the known concentration of NaOH solution.

S.NO	Volume of NaOH added(ml)	Conductance (Ohm^{-1} or Siemen's)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
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12		
13		
14		
15		
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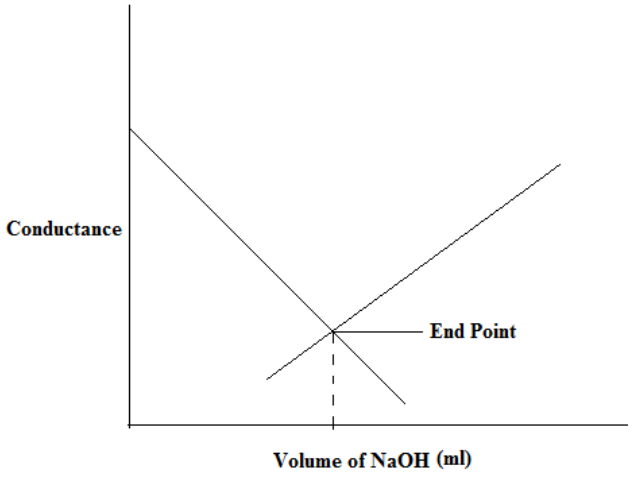
20		

Model Graph:



Calculation:

Normality of NaOH (N_2) = N

Volume of NaOH (from graph) (V_2) = ml

Normality of HCl (N_3) = N

Volume of HCl (V_3) = ml

$$N_2 V_2 = N_3 V_3$$

$$N_3 = N_2 V_2 / V_3$$

Strength of given HCl solution = Normality of HCl (N_3) x Eq. Wt. of HCl (36.54)

Result: The strength of HCl = g / lit

Advantage of Conductometric Titrations:

1. Coloured solutions which cannot be titrated by ordinary volumetric titration can be carried out without the help of indicator.

2. This method can also be employed in case of very dilute solution and also for weak acid and weak base.

3. No special care is necessary near the end point as it is determined graphically.

ESTIMATION OF ACETIC ACID BY CONDUCTOMETRIC TITRATIONS

Aim: To determination the strength of given HCl and Acetic acid solution by titrating against standard NaOH solution conductometrically.

Instruments and Apparatus: Burette, pipette, glass rod, beaker, standard flask, conductivity meter and conductivity cell with known cell constant.

Chemicals required: Hydrochloric acid (HCl), Acetic acid, Oxalic acid and sodium hydroxide.

Principle:

The electrical conductance of an electrolytic solution is proportional to

1. Number of ions present in solution i.e. ionic concentration.
2. The ionic mobilities.
3. Temperature of the solution.

The ionic mobilities of various ions in aqueous solution at 25 °C are generally in the range of 4-8 x 10^{-1} cm^2 $sec^{-1}volt^{-1}$. But the ionic mobilities of H^+ and OH- are abnormally high (H^+=36.8 x 10^{-4}, OH^- = 20.5 x 10^{-4} cm^2 $sec^{-1}volt^{-1}$). Thus the conductance of an electrolyte is quite sensitive to the concentration of H^+ and OH^-ions.

$$\mathbf{HCl + NaOH \rightarrow NaCl + H_2O}$$

$$\mathbf{CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O}$$

In the mixture of acids, the H^+ ion concentration is purely from HCl. So when the base is added HCl will react first, indicated by gradual decrease in the conductance because of fast moving H^+ ions are replaced by slow moving Na^+ ions. When all the HCl is used up, acetic acid starts reacting with NaOH. It is observed by gradual increase in the conductance. Beyond the equivalence point excess addition of NaOH there will be rapid increase in the conductance due to presence of fast moving OH^- ions.

The plot of conductance *Vs* the volume of alkali (NaOH) added will show two end points in the graph, the first one is the equivalence point for the titration of HCl *Vs* NaOH and the second for the equivalence point of acetic acid *Vs* NaOH.

Procedure:

Preparation of standard oxalic acid solution (0.1 N):
Weigh 0.63 gram of oxalic acid crystals into a clean 100 ml standard flask, dissolve in small amount of water and make up the solution up to the mark with distilled water. Shake the flask well for uniform concentration.

Normality of Oxalic Acid =0.1 N, W= weight of Oxalic Acid =? 126.07=

Molecular weight of Oxalic Acid 63=Equivalent weight of Oxalic Acid

$$\textbf{Normality} = \frac{Weight}{\text{Gr.Eq.wt}} \textbf{ x } \frac{1}{V}$$

$$\textbf{Weight= Normality x Gr. Eq.wt x } \frac{\text{V}}{1}$$

Titration I: Standardisation of NaOH solution

Fill the burette with given NaOH solution. Pipette out 20 ml of oxalic acid into a conical flask and add 2 to 3 drops of phenolphthalein indicator and titrate the colourless solution against NaOH till pale pink colour is obtained as end point. Repeat the titration to get concurrent values.

S. No.	Volume of Oxalic acid solution (ml) (V_1)	Burette reading(ml)		Volume of NaOH (ml) (V_2)
		Initial	Final	

Volume of standard Oxalic acid solution (V_1) = ml

Strength of standard Oxalic acid solution (N_1) = N

Volume of Sodium hydroxide (V_2) = ml

Strength of Sodium hydroxide (N_2) = N

$$N_1 V_1 = N_2 V_2$$

$$N_2 = N_1 V_1 / V_2$$

Titration II: Determination of strength of HCl and Acetic acid by conductivity meter

Transfer the given unknown acid mixture (HCl and acetic acid) solution into a clean 100 ml standard flask and make up the solution up to mark with distilled water and mix well to obtain uniform concentration. Pipette out 20ml of the given acid into a clean 100ml beaker, dip the conductivity cell in it and measure the conductance initially. Fill the burette with standard NaOH solution and rundown into the beaker (at a time 1 ml) with gentle stirring of the contents of the beaker and note the conductance after each addition. The measured conductance are recorded and tabulated. Take about 40 readings, by plotting the graph (conductivity Vs volume of NaOH) we can get the amount of NaOH required for the neutralization of HCl taken in the beaker (i.e. end point) and calculate the concentration of HCl and acetic acid solution from the known concentration of NaOH solution.

S.NO	Volume of NaOH added(ml)	Conductance (Ohm^{-1} or Siemen's)
1		
2		
3		
4		
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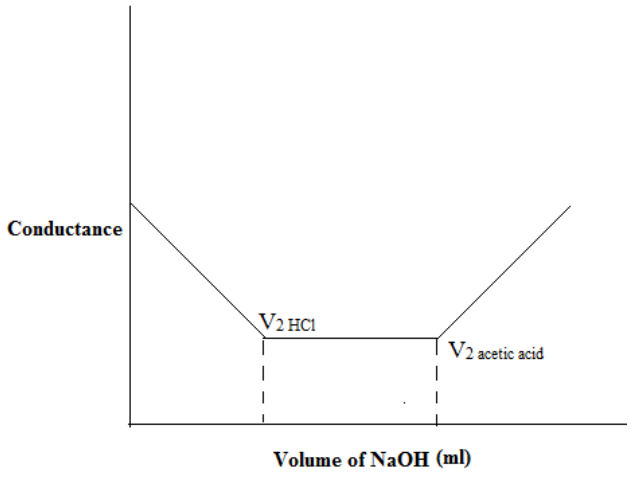
20		



Calculation:

Amount of HCl:

Normality of NaOH (N_2) = N

Volume of NaOH (from graph) ($V_{2\ HCl}$) = ml

Normality of HCl (N_3) = N

Volume of HCl (V_3) = ml

$$N_2\ V_2 = N_3\ V_3$$

$$N_3 = N_2\ V_2 / V_3$$

Strength of given HCl solution = Normality of HCl (N_3) x Eq. Wt. of HCl (36.54)

Amount of Acetic acid (CH3COOH):

Normality of NaOH (N_2) = N

Volume of NaOH (from graph) ($V_{2\ \text{acetic acid}}$) = $V_{2\ \text{acetic acid}}$ - $V_{2\ \text{HCl}}$ = ml

Normality of CH_3COOH (N_3) = N

Volume of CH_3COOH (V_3) = ml

$$N_2\ V_2 = N_3\ V_3$$

$$N_3 = N_2\ V_2 / V_3$$

Strength of given Acetic acid solution = Normality of Acetic acid (N_3) x Eq. Wt. of Acetic acid (60)

Result: The strength of HCl = g / lit

The strength of Acetic acid = g / lit

ESTIMATION OF HCL BY POTENTIOMETRIC TITRATIONS

Aim: To determination the strength of given HCl solution by titrating against standard NaOH solution potentiometrically.

Instruments and Apparatus: Burette, pipette, glass rod, beaker, standard flask, potentiometer, Platinum electrode and saturated calomel electrode (SCE).

Chemicals required: Hydrochloric acid (HCl), Oxalic acid and sodium hydroxide.

Principle:

When a solution of an acid is titrated with the solution of an alkaline, the change in the pH will be reflected in the change of E (potential). When a small amount of standard alkali is added to the acid a little change in the EMF is produced in the beginning. The change in the electrode potential depends upon the fraction of H^+ ions removed. As equivalence point reaches the fraction of the H^+ ions removed by constant volume of standard alkali increase rapidly. Thereby causing a rapid change in the EMF. Above the equivalence point there is again small change in the EMF by the addition of excess of alkali. Thus if the EMF of the cell is plotted against the volume of standard alkali added a curve is obtained.

$$\mathbf{HCl + NaOH \rightarrow NaCl + H_2O}$$

The experiment cell to be used is:

H2 (Pt) / acid solution ‖ KCl (aq.) / Calomel electrode

The EMF of the cell is given by

$$\mathbf{E = Ecell- EH = E^1+0.0591pH}$$

Procedure:

Preparation of standard oxalic acid solution (0.1 N):

Weigh 0.63 gram of oxalic acid crystals into a clean 100 ml standard flask, dissolve in small amount of water and make up the solution up to the mark with distilled water. Shake the flask well for uniform concentration.

Normality of Oxalic Acid =0.1 N, W= weight of Oxalic Acid =?

123.07= Molecular weight of Oxalic Acid
63=Equivalent weight of Oxalic Acid

$$\textbf{Normality} = \frac{Weight}{\text{Gr.Eq.wt}} \text{ x } \frac{1}{V}$$

$$\textbf{Weight= Normality x Gr. Eq.wt x } \frac{\text{V}}{1}$$

Titration I: Standardisation of NaOH solution

Fill the burette with given NaOH solution. Pipette out 20 ml of oxalic acid into a conical flask and add 2 to 3 drops of phenolphthalein indicator and titrate the colourless solution against NaOH till pale pink colour is obtained as end point. Repeat the titration to get concurrent values.

S. No.	Volume of Oxalic acid solution (ml) (V_1)	Burette reading(ml)		Volume of NaOH (ml) (V_2)
		Initial	Final	

Volume of standard Oxalic acid solution (V_1) = ml

Strength of standard Oxalic acid solution (N_1) = N

Volume of Sodium hydroxide (V_2) = ml

Strength of Sodium hydroxide (N_2) = N

$$N_1 V_1 = N_2 V_2$$

$$N_2 = N_1 V_1 / V_2$$

Calibration of Potentiometer:

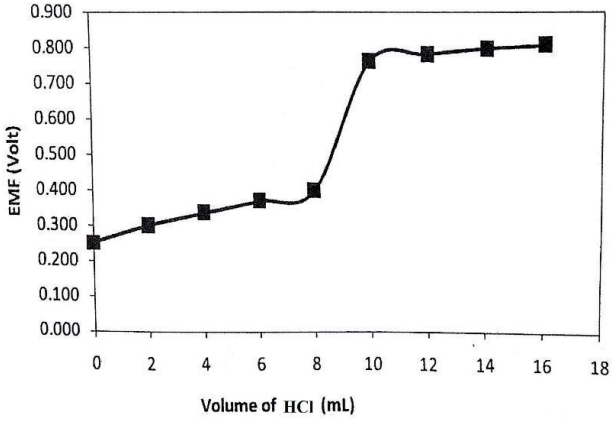
Switch on the potentiometer and connect the standard cell terminals to either channel A (move channel switch to position A) or channel B (move the channel switch to position B). The meter should read 1.018 V. In case it is not 1.018 V, adjust the Std. knob to obtain reference value.

Titration II: Determination of strength of HCl by potentiometry

Transfer the given unknown HCl solution into a clean 100 ml standard flask and make up the solution up to mark with distilled water and mix well to obtain uniform concentration. Pipette out 20 ml of above solution into a clean 100 ml beaker, place the electrode assembly (platinum electrode as indicator electrode and a SCE as reference electrode) in the beaker and connect the same to the potentiometer.

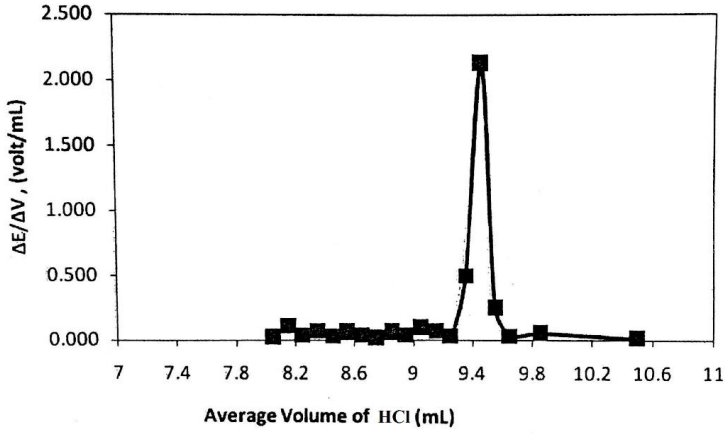
Add NaOH from burette in 1 ml portions to the acid solution, stir it and note down the EMF values. Continue the titration till a sudden inflexion in EMF occurs, then take about 6 to 8 readings after in 1 ml intervals. From the titration approximate volume of NaOH required is found out. Draw a graph of E cell Vs volume of NaOH added, the intersecting point gives an approximate equivalence point.

S.No	Volume of NaOH (ml)	EMF (volt)



The titration is repeated with addition of NaOH in 0.2 ml lots in the vicinity of end point (in 2 ml range). Plot a graph $\Delta E / \Delta V$ *Vs* volume of NaOH added. Calculate the normality of HCl solution and determine the amount of HCl in the given solution.

S.No	**Volume of NaOH (ml)**	**EMF(volt)**	**ΔE (volt)**	**ΔV (ml)**	**$\Delta E / \Delta V$ (volt /ml)**	**Average Volume (ml)**



Calculation:

Normality of NaOH (N_2) = N

Volume of NaOH (from graph) (V_2) = ml

Normality of HCl (N_3) = N

Volume of HCl (V_3) = ml

$$N_2 V_2 = N_3 V_3$$

$$N_3 = N_2 V_2 / V_3$$

Strength of given HCl solution = Normality of HCl (N_3) x Eq. Wt. of HCl (36.54)

Result: The strength of HCl = g / lit

ESTIMATION OF Fe^{+2} BY POTENTIOMETRY USING $KMnO_4$

Aim: To determination the amount of ferrous ion present in given whole solution by titrating against standard $KMnO_4$ solution potentiometrically.

Instruments and Apparatus: Burette, pipette, glass rod, beaker, standard flask, potentiometer, Platinum electrode and saturated calomel electrode (SCE).

Chemicals required: Ferrous Ammonium Sulphate (FAS), Potassium permanganate, Dil. Sulphuric acid.

Principle:
Redox titrations can be carried out potentiometrically using platinum-calomel electrode combination. For the reaction

$$\textbf{Reduction form} \leftrightarrows \textbf{Oxidised form + n electrons}$$

The potential is given by Nernst equation,

$$E = E^\circ + \frac{0.0591}{n} \log \frac{[\text{oxidized form}]}{[\text{reduced form}]}$$

Where, E° is the standard potential of the system. The potential of the system is thus controlled by the ratio of the concentration of the oxidized to that of the reduced species present. As the reaction proceeds, the ratio and hence the potential changes more rapidly in the vicinity of the end point of the titration. This may be followed potentiometrically and a plot of change in potential against volume (Titration curve) is characterized by sudden change in the potential at the equivalence point.

The reaction that takes place in the determination of Fe (II) is

$$5Fe^{+2} \rightarrow 5Fe^{+3} + 5e$$

$$MnO_4^- + H^+ + 5e \rightarrow Mn^{+2} + 4H_2O$$

$$5Fe^{+3} + MnO_4^- + 8H^+ \rightarrow 5Fe^{+3} + Mn^{+2} + 4H_2O$$

The experiment cell to be used is:

$$\textbf{Hg / Hg2Cl2 (s), Saturated KCl} \parallel \textbf{Fe}^{3+}\textbf{, Fe}^{2+}\textbf{ / Pt}$$

Procedure:
Preparation of a Standard FAS solution (0.01N):

Weight accurately 0.392g of FAS and transfer it in to a clean 100 ml volumetric flask with the help of funnel, dissolve this crystal in minimum amount of distilled water and 2 ml of dil. Sulphuric acid and make up the solution up to the mark, shake well for uniform concentration and calculate the normality of the prepared FAS (Mohr's salt) solution.

Normality of FAS =0.01, W= weight of FAS = ?

392.13= Molecular weight of FAS

392.13=Equivalent weight of FAS

$$\textbf{Normality} = \frac{Weight}{\text{Gr.Eq.wt}} \text{ x } \frac{1}{V}$$

$$\textbf{Weight= Normality x Gr. Eq.wt x } \frac{\text{V}}{\text{1}}$$

Titration I: Standardisation of KMnO4

Pipette out 20 ml of standard FAS solution into a clean conical flask. Add one test tube full (about 20 ml) of dilute sulphuric acid solution. Titrate this mixture against potassium permanganate taken in the burette. The end point is the appearance of light pink violet colour. Repeat the titration for concordant titre values. From the strength of FAS calculate the strength of $KMnO_4$ solution.

S. No.	Volume of FAS (V_1) (ml)	Burette reading(ml)		Volume of $KMnO_4$ (V_2) (ml)
		Initial	Final	

Volume of standard FAS solution (V_1) = ml

Strength of standard FAS solution (N_1) = N

Volume of $KMnO_4$ (V_2) = ml

Strength of $KMnO_4$ (N_2) = N

$$N_1 V_1 = N_2 V_2$$

$$N_2 = N_1 V_1 / V_2$$

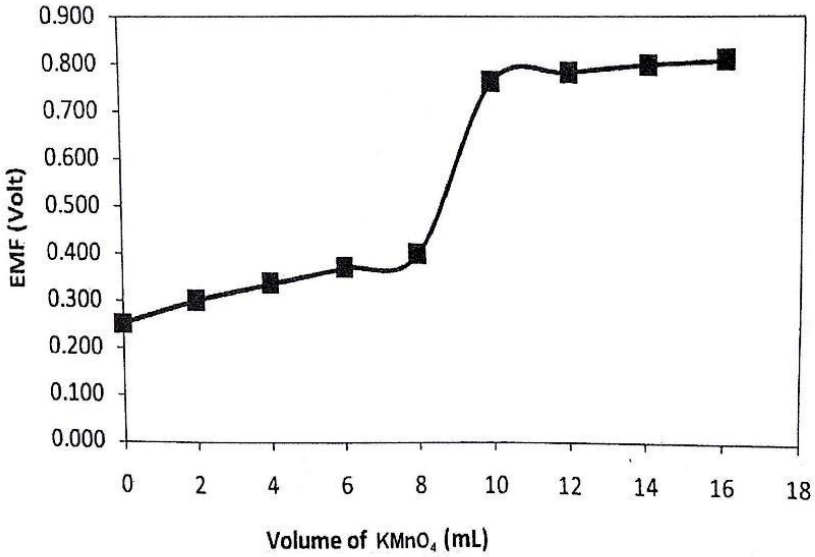
Calibration of Potentiometer:

Switch on the potentiometer and connect the standard cell terminals to either channel A (move channel switch to position A) or channel B (move the channel switch to position B). The meter should read 1.018 V. In case it is not 1.018 V, adjust the Std. knob to obtain reference value.

Titration II: Estimation Ferrous ion

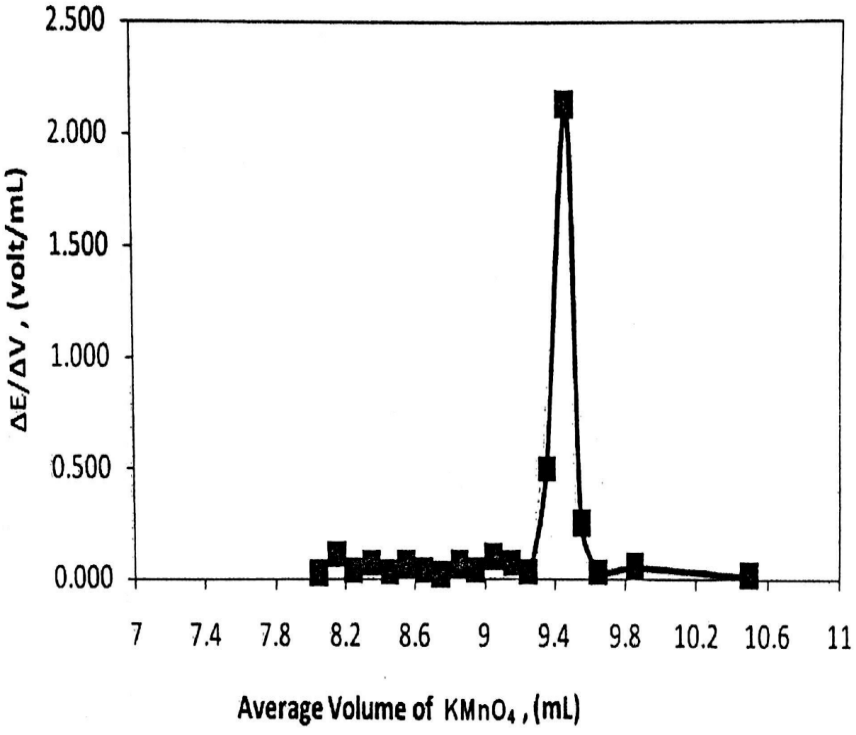
Transfer the given unknown ferrous ion solution into a clean 100 ml standard flask and make up the solution up to mark with distilled water and mix well to obtain uniform concentration. Pipette out 20 ml of above solution into a clean 100 ml beaker and add one test tube of Dil. Sulphuric acid. Place the electrode assembly (platinum electrode as indicator electrode and a SCE as reference electrode) in the beaker and connect the same to the potentiometer. Add permanganate from burette in 1 ml portions to the ferrous solution, stir it and note down the EMF values. Continue the titration till a sudden inflexion in EMF occurs, than take about 6 to 8 readings after in 1 ml intervals. From the titration approximate volume of permanganate required is found out. Draw a graph of E cell Vs volume of permanganate added, the intersecting point gives an approximate equivalence point.

S.No	Volume of $KMnO_4$ (ml)	EMF (volt)



The titration is repeated with addition of permanganate in 0.2 ml lots in the vicinity of end point (in 2 ml range). Plot a graph $\Delta E / \Delta V$ *Vs* volume of $KMnO_4$ added. Calculate the normality of ferrous solution and determine the amount of iron in the given solution.

S.No	Volume of $KMnO_4$ (ml)	EMF(volt)	ΔE (volt)	ΔV (ml)	ΔE / ΔV (volt /ml)	Average Volume (ml)



Calculation:

Normality of $KMnO_4$ (N_2) = N

Volume of $KMnO_4$ (from graph) (V_2) = ml

Normality of FAS (N_3) = N

Volume of FAS (V_3) = ml

$$N_2 V_2 = N_3 V_3$$

$$N_3 = N_2 V_2 / V_3$$

Weight of FAS per litre = Normality of FAS (N_3) x Eq. Wt. of FAS (55.85)

Weight of FAS per 100 ml = Weight of FAS per litre / 10

Result: The weight of Fe (II) present in the given solution by potentiometry is found to be_____gr/lit

DETERMINATION OF RATE CONSTANT OF ACID CATALYSED HYDROLYSIS OF METHYL ACETATE

AIM: To determine the rate constant of the hydrolysis of Ethyl acetate using an acid as a catalyst.

PRINCIPLE: The hydrolysis of an ester occurs according to the equation

$$CH_3COOC_2H_5 + H_2O \longrightarrow CH_3COOH + C_2H_5OH$$

This reaction follows pseudo first order kinetics.

PROCEDURE: 100 ml of 0.5 N HCl is taken in a clean dry conical flask. 5 ml of ester is pipetted out into the conical flask and the mixture is immediately withdrawn into another dry conical flask. A stop watch is started simultaneously. The reaction is then arrested by the addition of ice cubes and the mixture is titrated against 0.2 N NaOH using phenolphthalein as indicator. End point is the appearance of permanent pink colour. The volume of NaOH consumed in this titration is taken as V_0.

5 ml of acid – ester mixture is similarly withdrawn after 10, 20, 30, ..., 60 minutes respectively and titrated against NaOH using phenolphthalein as indicator. The volume of NaOH consumed for each of the above time intervals (t), is taken as V_t.

The contents are transferred into boiling tube with a cap and heated in a water bath for about 15 minutes. 5 ml of this mixture is withdrawn and titrated against NaOH to get V_∞.

CALCULATION:

The rate constant K is determined using the equation,

$$K = \frac{2.303}{t} \log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$$

Rate constant is also determined graphically by plotting $\log (V_\infty - V_t)$ Vs **time**.

TABULATION:

S.No.	Time Min	Volume of NaOH ml	$(V_\infty - V_t)$ ml	$log\ (V_\infty - V_t)$ ml	$K = \frac{2.303}{t} \log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$ min^{-1}
1	0				
2	10				
3	20				
4	30				
5	40				
6	50				
7	60				
8	∞				

RESULT: The Rate Constant for the hydrolysis of an ester from

1. Calculated value =
2. Graphical value =

SYNTHESIS OF ASPIRIN AND PARACETAMOL

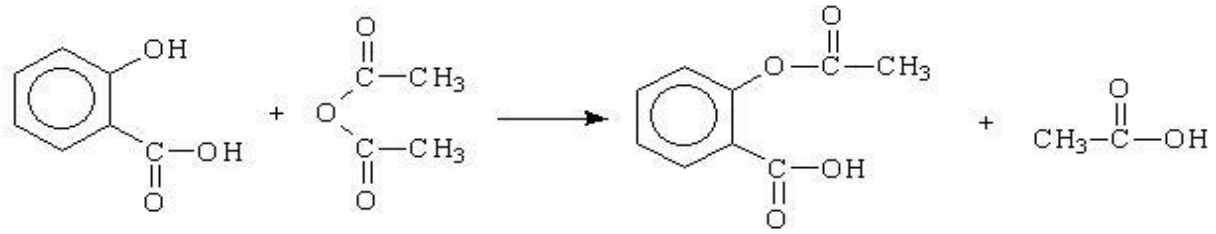
a) Synthesis of Aspirin

Aim: To synthesize aspirin

Apparatus: Beaker, Conical flask, watch glass, stirring rod, Buchner funnel, filter paper.

Chemicals: Salicylic acid, acetic anhydride, glacial aceticacid, sulphuric acid, ice and distilled water.

Principle: Aspirin is acetylsalicylic acid. It is a synthetic organic derivative derived from salicylic acid by acetylation in acidic medium. Salicylic acid is a natural product found in the bark of the willow tree and was used by the ancient Greeks and Native Americans, among others, to counter fever and pain. It was first synthesized in 1987 by a German chemist named Felix Hoffman.



Salicylic acid	Acetic anhydride	Acetylsalicylic acid	Acetic Acid
($C_7H_6O_3$)	($C_4H_6O_3$)	($C_9H_8O_4$)	($C_2H_4O_2$)

Procedure:

1. Take 2.0 g (0.015 mole) of salicylic acid in a 125-mL Erlenmeyer flask.
2. Add 5 mL (0.05 mole) of acetic anhydride, followed by 5 drops of conc. H_2SO_4 (*use a dropper, H_2SO_4 is highly corrosive*) and swirl the flask gently until the salicylic acid dissolves.
3. Heat the flask gently on the steam bath for at least 10 minutes.
4. Allow the flask to cool to room temperature. If acetylsalicylic acid does not begin to crystallize out, scratch the walls of the flask with a glass rod. Cool the mixture slightly in an ice bath until crystallization is completed. The product will appear as a solid mass when crystallization is completed.
5. Add 50 mL of water and cool the mixture in an ice bath. Do not add the water until crystal formation is complete.
6. Filter the solution through a Buchner funnel to remove any insoluble impurities or polymers that may have been formed. Wash the beaker and the funnel with 5 to 10 mL of water.
7. Take the crude product and recrystalise with 1:1 ratio of acetic acid and alcohol.
8. When the product is completely dry, weigh the product, determine its melting point (lit mp 135-136 °C) and calculate the percentage yield.

Result:

The percentage yield of Aspirin is_____.

Melting point_____.

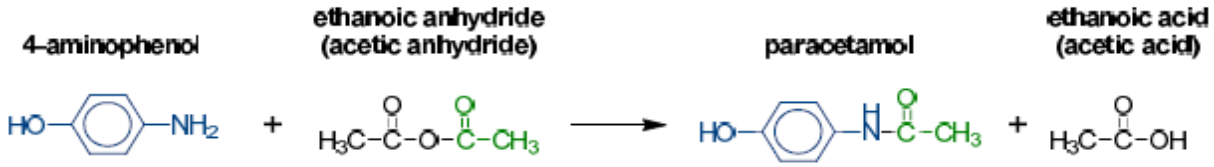
b) Synthesis of Paracetamol

Aim: To synthesize analgesic and antipyretic drug paracetamol.

Apparatus: Round bottom flask, measuring jar, glass rod, spatula funnel, filter paper, water bath, watch glass.

Chemicals: 4-amino phenol, acetic anhydride, distilled water.

Principle: Paracetamol is also known as acetaminophen or APAP, is a medicine used to treat pain and fever. Paracetamol is made by reacting 4-aminophenol with acetic anhydride. This reaction forms an amide bond and acetic acid as a byproduct. When the reaction is complete the Paracetamol is then isolated and purified.



Procedure: Weigh about 1.1 g of p- amino phenol in a round bottomed flask and add 30ml of distilled water with a measuring jar and add 12 ml of acetic anhydride. Stirr the reaction mixture vigorously with a glass rod and warm on water bath till the solid disappear. After 10 min cool the reaction mixture and filter the solid material to separate acetyl derivative. Dry and recrystallise the crude product from hot water. Dry the pure product and measure melting point (literature 169 ºC) report the yield.

Result:

The yield of pure Paracetamol is_____.

Melting point_____.

THIN LAYER CHROMATOGRAPHY CALCULATION OF Rf VALUES. Eg ORTHO AND PARA NITRO PHENOLS

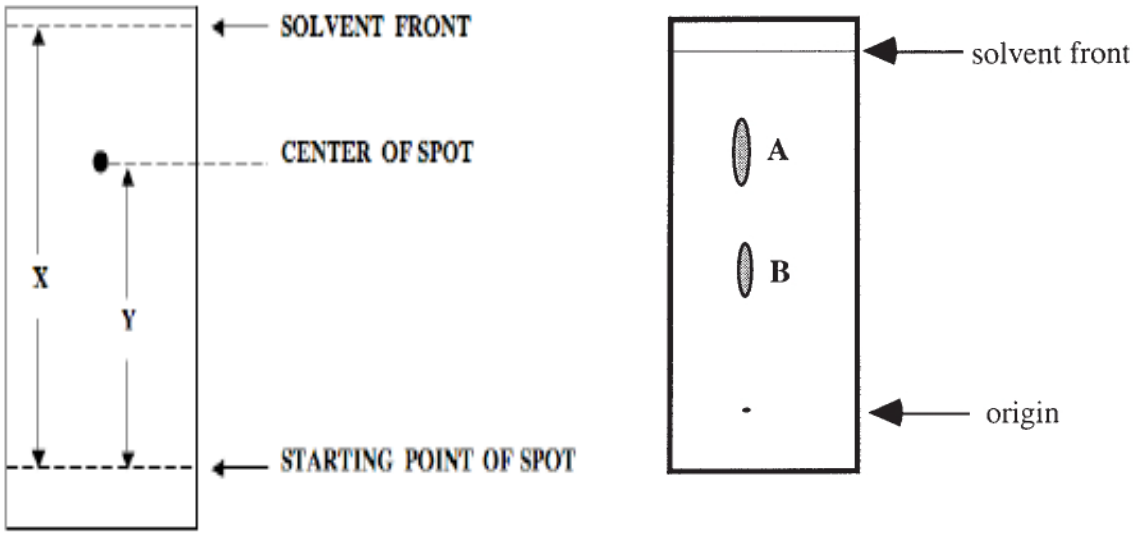
Aim: To determine the R_f values of given compounds using Thin-Layer Chromatography.

Apparatus: TLC, TLC-Chamber

Chemicals: ortho and para nitro phenols.

Principle: Thin layer chromatography (TLC) is a useful technique for the separation and identification of compounds in mixtures. Thin layer chromatography (TLC) uses the same principles as extraction to accomplish the separation and purification of compounds: that is, the separation of different compounds between two phases based on differences in solubility of compounds in the two phases. In the case of TLC, one phase is a mobile liquid solvent phase and the other phase is a stationary solid phase with a high surface area. The **stationary phase** normally consists of a finely divided **adsorbent**, silica (SiO_2) or alumina (Al_2O_3) powder, used in the form of a thin layer (about 0.25 mm thick) on a supporting material. The support is usually a sheet of glass or metal foil. The **mobile phase** consists of a volatile organic solvent or mixture of solvents.

TLC is used routinely to follow the progress of reactions by monitoring the consumption of starting materials and the appearance of products. Commercial applications of TLC include the analysis of urine for evidence of "doping", the analysis of drugs to establish purity or identity of the components, and analysis of foods to determine the presence of contaminants such as pesticides.



Retention factors are numbers between zero and one, representing the position of the spot on the TLC plate. The R_f (rate of flow) value is usually constant under fixed conditions such as TLC plate, developing solvent, and temperature. Therefore, each compound can be identified based on the R_f value along with the shape and color of its spot. The R_f value is usually recorded to the second decimal place.

Procedure: Thin-layer chromatography is carried out with a glass, aluminum, or plastic plate coated with silica gel. When one of the edges of the plate is dipped in a developing solvent, the sample moves along with the solvent. The mobility of samples depends on their properties. This method is utilized for checking the purity of a product and the progress of a reaction.

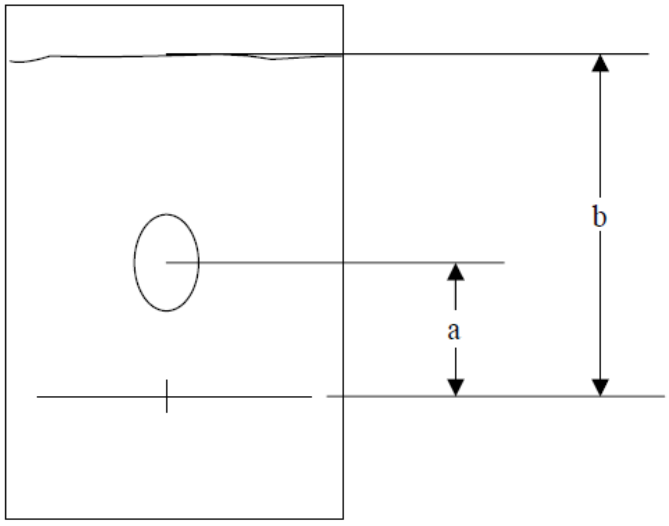
Preparation

1. Take a small amount of a sample with a micro spatula and dissolve in a small amount of an appropriate solvent.
2. Draw the starting line lightly with a pencil at about 10 mm above from a shorter edge of a silica gel plate.
3. Draw cross marks on the line as chromatography starting points.
4. Take the sample solution with a capillary, spot the solution lightly on one of the starting points, and dry the spot with a dryer if necessary. Repeat this operation a few times to concentrate the sample in the small spot, which should be less than 2 mm in diameter.
5. Use a new capillary when a different sample is taken.

Development

1. Pour the developing solvent into a wide-mouth bottle to about 5 mm in height.
2. Close the cap and wait a little while until the bottle is saturated with the solvent vapor.
3. Open the cap and hold the upper end of the TLC plate with tweezers. Place the TLC plate in the bottle so that the bottom of the plate is dipped in the solvent, and the top of the plate is leaned against the wall of the bottle. The solvent should be drawn straight upright.
4. Finish the development when the solvent front reaches at about 10 mm below the upper end of the TLC plate.

Calculation of the Rf value:



Measure the distance from the starting point to the center of the spot on the TLC plate (distance a).

Measure the distance from the starting point to the solvent front (distance b).

Calculate the Retention Factor as:

$$R_f = \frac{a}{b}$$

1. Take out the TLC plate and immediately mark the front line of the developing solvent with a pencil.
2. Wait for the solvent to evaporate or dry the TLC plate with a dryer.
3. Turn on a UV lamp. Trace the outlines of the spots detected with UV ray with a pencil.
4. Record the shape and color of the spots.
5. Calculate Rf value.

   $R_f = a/b$

   a = The distance from the starting point to the gravity center of the sample spot.
   b = The distance from the starting point to the front of the developing solvent.

Results:

1. R_f value of ortho nitro pnenol_____
2. R_f value of para nitro pnenol_____

DETERMINATION OF ACID VALUE OF COCONUT OIL

Aim: To determine the acid value present in given Coconut oil.

Chemicals: Oxalic acid, KOH Solution, Alcohol, Coconut oil, Phenolphthalein indicator and distilled water.

Apparatus: Burette, pipette, conical flask, Wash bottle, Dropper.

Principle: Acid value indicates the proportion of free fatty acid present in an oil or fat and may be defined as the number of milligrams of caustic potash required to neutralize the acid in 1 gm of the sample. The normal acid value for most samples lies within 0.5. If any titrable acid other than a fatty acid is present in the sample, it will be an error. A high acid value indicates a stale oil or fat stored under improper conditions.

Procedure

Standardization of KOH: Pipette out 20 ml of 0.1 N oxalic acid solution in a 250 ml conical flask. Add 1 or 2 drops of phenolphthalein indicator to this solution. Titrate this solution against KOH taken in a burette. The appearance of pink color indicates the end point. From the volume of the KOH solution in burette, find the normality of KOH.

Determination of Acid value in coconut oil: Weigh 5 gm of coconut oil and transfer it into 250 ml conical flask. Add 50 ml of neutralized alcohol solution to the oil solution. Heat this mixture for 10 minutes by using the heater. Take the solution after 10 minutes and add 1 or 2 drops of phenolphthalein indicator. Titrate this against the KOH solution from the burette. The appearance of pink color indicates the end point

Calculation

Titration I: Standardisation of Potassium hydroxide solution:

S.No	Volume of Oxalic acid (ml)	Burette Reading		Concordant Volume of KOH (ml)
		Initial	Final	

Burette: KOH
Pipette solution: Oxalic acid
Indicator: Phenolphthalein
End point: Appearance of pink colour

Volume of oxalic acid (V_1)=

Normality of oxalic acid (N_1)=

Volume of KOH consumed (V_2)=

Normality of KOH consumed (N_2)= V_1 N_1 / V_2

Normality of KOH (N_2) = _____.

Titration II: Estimation of acid value

S.No	Volume of Oxalic acid (ml)	Burette Reading		Concordant Volume of KOH (ml)
		Initial	Final	

Burette solution: KOH

Pipette solution: Oil + 50 ml of neutralized alcohol

Indicator: Phenolphthalein

End point: Appearance of pink colour

Acid value = (Volume of KOH x Normality of KOH x Eq. wt x 1000) / Weight of Oil sample

Acid Value = _____.

RESULT

The acid value of the given Coconut oil sample was found to be_____.

VERIFICATION OF FREUNDLICH ADSORPTION ISOTHERM- ADSORPTION OF ACETIC ACID ON CHARCOAL

Aim: To verify Freundlich Adsorption Isotherm for adsorption of acetic acid on a given sample of charcoal.

Apparatus: Stoppered reagent bottles, burette, pipette, conicalflask, beakers, funnels, glazed paper (filter paper).

Chemicals: Acetic Acid (0.5M) NaOH (0.1M), Distilled water, phenolphthalein indicator.

Principle: The property of accumulating other substances on the surface of porous solid without a sensible penetration into the bulk of solid phase is called **adsorption**.

At a given temperature, the variation in the amount of solute adsorbed with change in concentration of the solution is given by an empirical relation suggested by Freundlich known as **Freundlich adsorption isotherm**. This is

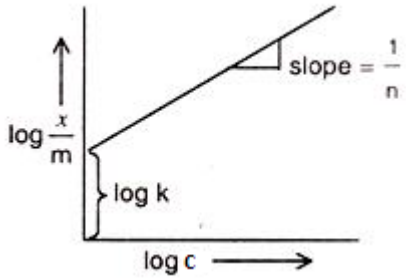
$$\frac{x}{m} = KC^{1/n}$$

Where *C* stands for the equilibrium concentration of the solute in solution.

Taking logarithms of the terms in the Freundlich isotherm we get,

$$\log \frac{x}{m} = \log K + \frac{1}{n} \log C$$

If we plot a graph between log (*x/m*) (along ordinate) versus log *C* (along abscissa), a straight line would get. The slope of the line will equal 1/*n* and log *K* can be obtained as the intercept which the straight line makes on the ordinate for a value of log *C* equal to zero.



Procedure: Weigh accurately about 2gms of finely powdered charcoal in each of the thoroughly cleaned and dried bottles numbered as 1 to 6. Prepare 0.5 M acetic acid solution for 250 ml by means of a burette add 10, 20, 30, 40, 50 ml of acetic acid solutions and 40, 30, 20, 10 and 0ml distilled water in the bottles 1, 2, 3, 4 and 5 respectively. Take the unknown solution in the bottle no. 6. Shake the bottles vigorously and leave them at a desired temperature for about half an hour. Prepare 0.1 M NaOH for 250 ml and standardise with standard oxalic acid solution using phenolphthaline indicator and determine the concentration of acid solution.

Filter the solution of each bottle by means of filter paper. Collect the filtrates properly in separate conical flasks numbered with 1, 2, 3, 4 and 5. While filtering reject about 5 ml of initial filtrate in each case. Take 10 ml from each filtrate and then titrate against standard NaOH solution. Calculate the equilibrium concentration of acid in each bottle.

Observations and Calculations

Tabulate the observation and plot a graph.

Flask No	NaOH solution required for 10 ml solution after adsorption.	Initial concentration of Acetic acid (C_i)	Equilibrium concentration of Acetic acid (C_e)	Amount adsorbed $x = \frac{C_i - C_e}{20}$	x/m	$\log \frac{x}{m}$	$\log C_e$

Result: Freundlich Adsorption Isotherm-Adsorption of Acetic Acid on Charcoal is:_____

DETERMINATION OF VISCOSITY OF CASTOR OIL AND GROUND NUT OIL BY USING OSTWALD'S VISCOMETER.

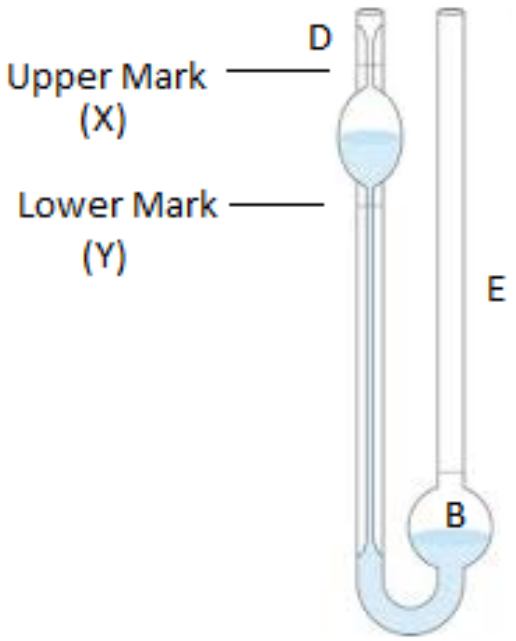
Aim: To determine the viscosity of a given liquid sample by Oswald's viscometer.

Instruments and Apparatus: Oswald's viscometer and stop watch.

Chemicals required: Liquid sample and water sample.

Principle: Viscosity is the most important property of any lubricating oil. It describes the suitability of liquid for lubrication purposes. Viscosity of any liquid is that characteristics of fluid by virtue of which it hinder its own flow. Absolute viscosity is a measure of the internal resistance. Absolute viscosity is the tangential force per unit area required to move one horizontal plane with respect to the other at unit velocity when maintained a unit distance apart by the fluid. Kinematic viscosity is the ratio of absolute or dynamic viscosity to density- a quantity in which no force is involved. Kinematic viscosity can be obtained by dividing the absolute viscosity of a fluid with its mass density.

Viscosity index (vi) is an arbitrary measure for the change of viscosity with temperature. It is used to characterize lubricating oil in the automotive industry.



Procedure:

Determination of density of given sample

Weight of specific gravity bottle + given sample W_1 = g

Weight of specific gravity bottle W_2 = g

Weight of given sample ($W_1 - W_2$) = g

Volume of given sample (V) = ml

Density of given sample (d_2) = $W_1 - W_2$ / V (gm/cc) =

Take a clean and dry Oswald viscometer and set it vertically on a stand. Introduce 10 ml of water through E (refer figure) into the large bulb (B). Suck the liquid up into the bulb 'A' through a rubber tubing attached to the end 'D' to a level above the mark 'X'. Allow the liquid to flow freely through the capillary and note the time t_1 (use the wrist watch or stop watch) for the liquid flow from X to Y. repeat steps two or more times and get an average value of t_1. Repeat the process to obtain the average time for the liquids to flow from X to Y. Tabulate the data as follows.

S. No.	Sample	Density	Time flow time average		Time average
			Exp 1	Exp 2	
1	Water	1			
2					
3					

Calculations:

$$\eta 1 / \eta 2 = (d1t1) / (d2t2)$$

Where η_1 = viscosity of water,
η_2 = viscosity of given liquid
d_1 = density of water,
d_2 = density of given liquid
t_1 = time flow of water from X toY
t_2 = time flow of given liquid from X to Y

Result: The viscosity of the given liquid = (units of viscosity are poise, Pascal - second or stokes)

DETERMINATION OF PARTITION COEFFICIENT OF ACETIC ACID BETWEEN n-BUTANOL AND WATER

Aim: To determine the partition co-efficient of Acetic acid between n-butanol and water.

Chemicals: n-butanol, acetic acid, distilled water, NaOH Solution.

Apparatus: Burette, pipette, 100 ml conical flask, 250 ml Stoppered bottle, Wash bottle, Dropper.

Principle: Nernst distribution law states that at constant temperature, when a solute are allowed to distribute between two immiscible solvents in contact with each other, then at equilibrium the ratio of the concentration of the solute in two solvent layers is constant.

When a solute is shaken in two immiscible liquids, then the solute is found to be distributed between the liquids in a definite manner, if the solute is soluble in each of the solvent.

According to distribution law, the distribution co-efficient at a particular temperature is given by

$K_d = C_1 / C_2$, where

C_1 and C_2 represent concentration of acetic acid in water and concentration of acetic acid in buty1 alcohol respectively. When the solute molecules in each solution phase are in same state of association.

Procedure: A 250 ml stoppered bottle is taken and washed properly with distilled water. This bottle is filled up with 40 ml of n-butanol, 50 ml distilled water and 10 ml of acetic acid. Then it is allowed to shake in a mechanical shaker for about an hour. After shaking, it is kept aside for about 5 minutes so that two layers separate out clearly. 10ml of aqueous layer is pipette out in a 250 ml conical flask. 2 -3 drops of phenolphthalein indicator is added and titrated against standard NaOH solution until colour changes from colourless to pink.

Similarly 10ml of organic layer is pipette out in a 250 ml conical flask. 2 -3 drops of phenolphthalein indicator is added and titrated against standard NaOH solution until colour changes from colourless to pink. The experiments are repeated to get concordant values.

Observations & Calculations

Layers Taken	**Pipette Reading (ml)**	**Burette Reading (ml)**		**Volume of NaOH consumed (ml)**	**$K_d = C1 / C_2$**
		Initial	**Final**		
Aqueous Layer					
Organic Layer					

Aqueous Layer (C_1):

Vol. of aqueous layer x C_1 = Vol. of NaOH required (V) x strength of NaOH(S)

10ml x C_1 = V x S

$C_1 = (V \times S) / 10$

Organic Layer (C_2):

Vol. of Organic layer x C_2 = Vol. of NaOH required (V) x strength of NaOH(S)

10 ml x C_2 = V x S

$C_2 = (V \times S) / 10$

Hence, $K_d = C_1 / C_2$

Result: Partition Coefficient of Acetic acid between water & n-butanol is ……..

Precautions:

1. Stoppered bottle should be thoroughly cleared & dried.
2. During withdrawing of liquid for titration, one layer must not be contaminated with other layer.

DETERMINATION OF SURFACE TENSION OF A GIVEN LIQUID USING STALAGMOMETER

Aim: To determine the surface tension of a given liquid.

Apparatus: Stalagmometer, stand, beakers.

Chemicals: Distilled water, test liquid.

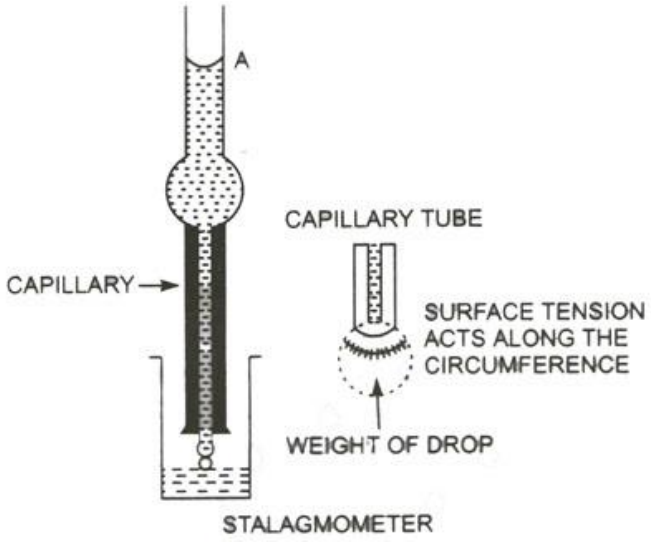
Principle: Surface tension is defined as the force in dynes acting on a surface at right angles to any line of unit length or per centimeter.

Surface tension of a liquid is measured using an apparatus called stalagmometer. The method is based on the principle that the weight of a liquid falling from a capillary tube held vertical is approximately proportional to the surface tension of the liquid. It is more convenient to count the number of drops of the liquid. The weight of equal volumes of two liquids are proportional to their densities. If n1 & n2 are the same volumes of two liquids then

$$\gamma_1/\gamma_2 = n_2d_1/n_1d_2$$

If the surface tension of the one liquid is known then that of the other can be easily calculated from the equation. Generally liquid 2 is a reference which is water, whose data can be obtained from literature.

Description of Apparatus: The apparatus "Stalagmometer" is used to determine surface tension is a pipette with a capillary at the lower end, a bulb in the middle. The upper end is provided with one screw pinch cock. It is provided two marks A and B, A is in the upper end and B is in the lower end. These marks A & B define the volume of a lubricant to be delivered for counting the drops. The rate of flow can be adjusted using screw pinch cock.



Procedure: Take a clean and dried stalagmometer. Fill the stalagmometer with distilled water by sucking it while immersing the lower end into the distilled water. Now bring the level of the water to mark A. Open the pinch cock, adjust it , so that the rate of flow of liquid is about 12-15 drops per minute and count the number of drops formed till the level of the liquid reaches the mark B. Note down the time interval in the given table. Repeat the experiment to get two concordant values. Repeat the procedure for unknown liquid to determine the surface tension.

Observation and calculations

S. No	Number of drops of water (n_1)	Number of drops of unknown liquid (n_2)

Surface tension of water at 25 ºC = 72 dynes/cm

γ_1 = surface tension of water (reference liquid)

γ_2 = surface tension of unknown liquid.

$$\gamma_1/\gamma_2 = n_2d_1/n_1d_2$$

Precautions:

1. The tip of the lower end should not come in contact with hand or anything else as it will be contaminated with traces of grease, which will alter the size of the drop.
2. The stalagmometer should be held vertical ant shaken, otherwise the drop fall out before attaining its maximum size.

Result:

The surface tension of the given liquid = ……. dynes/cm