ENGINEERING CHEMISTRY LABORATORY

MANUAL & RECORD

GOKARAJU RANGARAJU
INSTITUTE OF ENGINEERING & TECHNOLOGY

(Autonomous)

GOKARAJU RANGARAJU INSTITUTE OF ENGINEERING & TECHNOLOGY

(Autonomous Institute under J.N.T.University, Hyderabad)

Bachupally, Kukatpally, Hyderabad — 500 090

CERTIFICATE

This is to certify that this is the bonafide record of practical work done in the Engineering Chemistry Lab in B.Tech_____year, during the academic year_____

Name : _____

Regd. No. : _____

Branch : _____

Section : _____

External Examiner

Signature of faculty in-charge

(Name:)

Date:

PREFACE

The main objective of the laboratory manual entitled **"Engineering Chemistry Laboratory"** is to make the first year B.Tech students familiar with the Chemistry lab and to enhance practical skills. This manual is written according to the GR-20 regulation of GRIET (Autonomous) syllabus. This book is prepared to meet the objectives prescribed by AICTE and JNTUH for Practical Engineering Chemistry.

This manual is prepared by the faculty of the Department of Chemistry

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4. Bh. Saroja Rani, Assistant Professor
5. Dr. K. Kalpana, Assistant Professor
6. M. Haritha Kiranmai, Assistant Professor
7. M. Yesu, Assistant Professor
8. K. Lavanya, Assistant Professor
9. B. Ravi Kiran, Assistant Professor

INSTRUCTIONS

1) Students should wear their identity cards, formal dress and apron.

2) Students should come with their observation book and manual.

3) Students should maintain silence in the lab.

4) Observation note book should be signed by your concerned faculty before leaving the lab.

5) Record corrections in observation note book should be completed for the previous experiment before coming to the next lab class.

6) If any student is absent for a session, no repetition of experiment will be provided.

7) If any breakage is done by the students, fine should be paid.

8) Students should not drop any solid materials in the sinks.

9) Students should follow the rules and regulations according to the instructor.

10) Wash hands thoroughly with soap after completing the experiment.

11) First aid box is available in the lab.

SAFETY MEASURES

1. Students must wear apron in the lab.

2. Read label carefully before transferring a chemical from a container.

3. While pipetting, if any solution is swallowed, wash the mouth repeatedly with water.

4. Handle with care the chemicals, glassware and electrical instruments.

5. The electronic balance should be handled carefully under the directions of a technical assistant.

6. Do not perform unauthorized experiment in the laboratory.

7. Despite the best precautions sometimes an accident may occur. Seek immediate help and rescue from the laboratory staff.

8. Keep the work bench neat and tidy.

GOKARAJU RANGARAJU INSTITUTE OF ENGINEERING AND TECHNOLOGY

(AUTONOMOUS)

DEPARTMENT OF CHEMISTRY

ENGINEERING CHEMISTRY LAB

(Common to all Branches)

B. Tech I Year (I and II Semesters) 2018-19

L -0, T-0, P-3, C- 1.5

Course Objectives:

1. Introduce practical applications of chemistry concepts to engineering problems.
2. To determine the rate constant of reaction from change in concentrations as a function of time.
3. Measure the molecular or ionic properties such as conductance, redox potentials
4. Synthesize a drug molecule to learn how organic compounds are prepared in industry.
5. Know the laboratory practices implemented in a research and industrial chemistry laboratory setting.

Course Outcomes:

1. Ability to perform experiments illustrating the principles of chemistry relevant to the study of science and engineering.
2. Determination of parameters like hardness and chloride content in water.
3. Understand the kinetics of a reaction from a change in concentration of reactants or products as a function of time.
4. Synthesize a drug molecule as an example of organic synthesis methods widely used in industry.
5. Determination of physical properties like adsorption and viscosity.

ENGINEERING CHEMISTRY LABORATORY

List of Experiments

EVALUATION SHEET

S. No.	Exp. No.	Name of the Experiment	Date	Marks 5M	Signature of the Faculty
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					

BASIC CONCEPTS IN VOLUMETRIC ANALYSIS

Chemical analysis of the substances is carried out in two ways

1. **Qualitative analysis:** Identification of the constituents, e.g. elements or functional groups, present in a substance.
2. **Quantitative analysis:** Determines the quantity of a component present in substance. It is carried out in two ways
   a. Gravimetric analysis
   b. Volumetric analysis

   Gravimetric analysis involves the estimation of the amount of a given compound from the results of weighing. Volumetric analysis is based on measuring volume of the solution of a substance.
3. **Types of Titrations:**
   a. **Acid – Base titrations**: A titration in which an acid of known concentration is added to a solution of base of unknown concentration, or the converse.
   b. **Complexometric titrations:** Titrations in which complex is formed between the analyte and the titrant.
   c. **Redox titrations**: Redox titrations are based on an oxidation-reduction reaction between an oxidizing agent and a reducing agent.
   c. **Precipitation Titrations:** Titrations based on the formation of a precipitate between the titrant and the analyte are termed as precipitation titrations.

4. **Terms involved in volumetric analysis**
   a. **Titration:** The process of finding out the volume of one of the solution required to react completely with the definite volume of other solution
   b. **Titrant:** The solution of known strength.
   c. **Titrand or Analyte:** The solution which contains a substance whose strength is to be estimated.
   d. **Indicator:** The substance which indicates the end point of titration is called indicator. The indicator indicates the completion of reaction by change in colour at the end point. Eg: Phenolphthalein for acid base titration.

e. **Strength:** The amount of substance dissolved in one litre of a solution is expressed in terms of strength of a solution. Strength of solution can be expressed in any of the following ways.

i. **Normality**: It is the number of gram equivalents of solute present in one liter of solution. It is denoted by N.

ii. **Molarity:** The number of moles of a given substance per litre of solution. It is denoted by M.

iii. **Molality**: The number of moles of solute per kilogram of solvent. It is denoted by m.

f. **Standard solution:** It is a chemical term which describes a solution of known concentration. The concentration of the solution is normally expressed in units of moles per liter. Standard solutions are normally used in titrations to determine the unknown concentration of a substance in sample solution.

g. **Primary standard**: Any substance stable, pure, readily soluble in water, with high equivalent weight and the composition of its solution should not change on long standing or storage.
Examples: Oxalic acid, Magnesium sulphate, Potassium dichromate.

h. **Secondary Standard**: The composition of these solutions changes on long standing or storage with duration of time. These are standardized by Primary standards.
Examples: Ferrous sulphate, EDTA, Potassium permanganate

i. **Equivalent Weight:** Equivalent Weight of a substance may be defined as the number of parts by weight of it which can combine or displace 1.008 parts of hydrogen or 8 parts of oxygen or equivalent weight of any substance.

Equivalent weight of acid = Mol. Wt of acid / no. of replaceable H^+ ions

a. Molecular weight of sulfuric acid(H_2SO_4) = 98.07 g/mol
Number of replaceable H^+ ions or basicity = 2
Equivalent weight = 98.07 / 2 = 49.035 g/ equivalent of H^+

b. Molecular weight of Hydrochloric acid (HCl) = 36.5 g/mol

Number of replaceable H^+ ions = 1

Equivalent weight = 36.5 /1 = 36.5 g/ equivalent of H^+

Equivalent weight of base = Mol. Wt of base / no. of replaceable OH^- ions

a. Molecular weight of ammonium hydroxide (NH_4OH) = 35.00 g/mol

Number of replaceable OH^- ions or acidity = 1

Equivalent weight = 35.00 /1 = 35.00 g/equivalents of OH^-

b. Molecular weight of sodium hydroxide (NaOH) = 40.00 g/mol

Number of replaceable OH^- ions or acidity = 1

Equivalent weight = 40.00 /1 = 40.00 g/equivalents of OH^-

Equivalent weight of an oxidizing agent

Oxidation process involves loss of electrons. An oxidizing agent is one which accepts electrons; it oxidizes the other substance by stripping off electrons from it.

Equivalent weight of oxidizing agent = Mol. Wt / number of electrons gained

a. Potassium permanganate ($KMnO_4$):

i). Acidic **medium:**

Equivalent weight of $KMnO_4$ = Mol. Wt / 5

$MnO_4^- + 8\ H^+ + 5e^- \rightarrow Mn^{2+} + 4H_20$ **(**gained 5 electrons from reductant**)**

Equivalent weight of $KMnO_4$ = 158.04 / 5 = 31.608

ii). **Alkaline medium**

Equivalent weight of $KMnO_4$ = Mol. Wt / 1 = 158.04/1

MnO_4^- + e^- → MnO_4^{2-}

Permanganate ion Manganate ion

iii). **Neutral Medium**

Equivalent weight of $KMnO_4$ = Mol.Wt / 3 = 158.04/3 = 52.68

$MnO_4^- + 2\ H_2O + 6e^- \rightarrow MnO_2 \downarrow + 4\ OH^-$

Manganese dioxide ppt

b. Potassium dichromate($K_2Cr_2O_7$):

$Cr_2O_7^{2-} + 14\ H^+ + 6e^- \rightarrow 2\ Cr^{3+} + 7\ H_2O$

Equivalent weight of $K_2Cr_2O_7$ = Mol.Wt / 6 = 294.185/6 = 49.03

Equivalent Weight of reducing agent:

Reduction process involves gain of electrons. A reducing agent is one which loses the electrons; it reduces the other substance by donating electrons to it.

Equivalent weight of a reducing agent = Mol. Wt / No. of electrons lost

a. Equivalent Wt. of Mohr's Salt ($Fe\ SO_4(NH_4)_2\ SO_4.6\ H_2O$) = Mol.Wt / 1 = 392.13/1

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

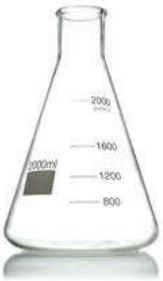
b. Equivalent Wt. of Oxalic acid ($H_2C_2O_4.2\ H_2O$) = Mol.Wt / 2 = 126/2

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e^-$$

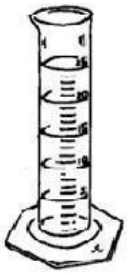
c. Equivalent Wt. of Sodiumthiosulphate($Na_2\ S_2O_3.5\ H_2O$)= Mol.Wt / 1=248.18/1

$$S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2\ e^-$$

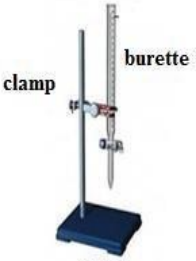
APPARATUS USED IN CHEMISTRY LABORATORY



Conical flask



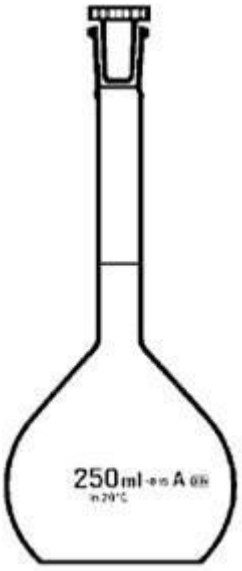
Measuring Cylinder



stand

Beakers

Pipette



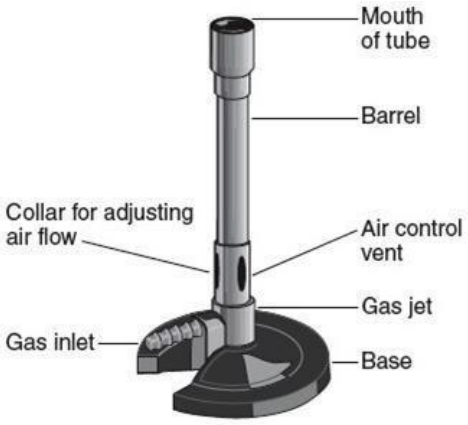
Volumetric flask

Test tubes

Watch Glass

Round Bottomed Flask

Wash bottle



Bunsen Burner

Seperating Funnel

Spatula

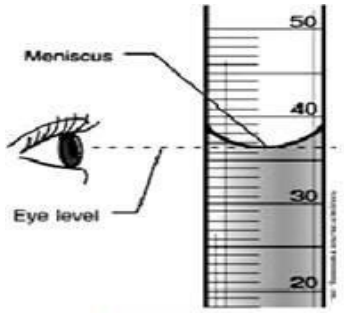
Reagent Bottle

Buchner Funnel

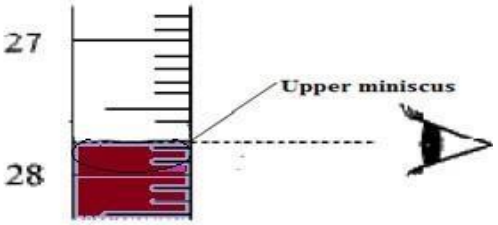
Funnel

How to note burette reading?

For colourless solutions lower meniscus is noted and for coloured solutions upper meniscus is noted.

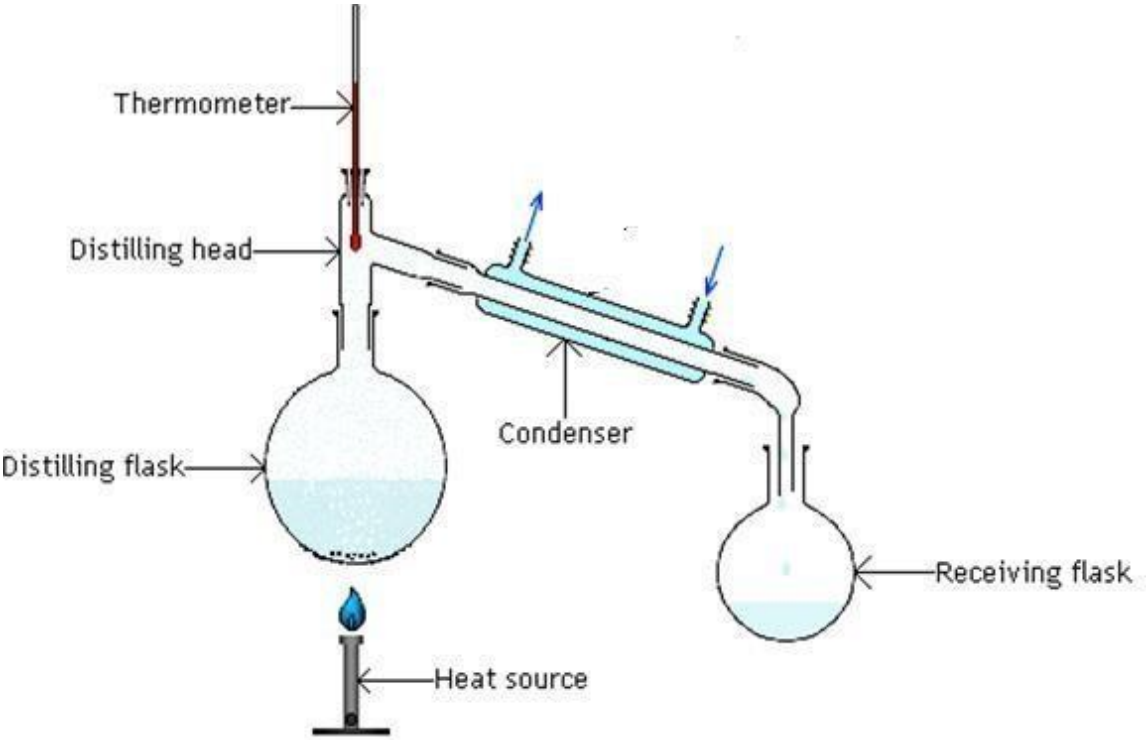


Colourless solution



Coloured solution

DISTILLATION APPARATUS



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COMPLEXOMETRY

1. ESTIMATION OF TOTAL HARDNESS OF WATER BY USING STANDARD EDTA SOLUTION

Aim: To estimate the total hardness of water using standard Ethylene Diamine Tetra-acetic Acid (EDTA) solution.

Apparatus: Pipette, burette, standard volumetric flask, conical flask, beaker, dropper

Chemicals Required: Disodium salt of EDTA, Eriochrome Black-T (EBT) indicator**,** Calcium carbonate, dil. HCl.

Indicator: Eriochrome Black-T (EBT)

End point: Change of color from wine red to blue

Principle: This method is based on the following facts; Eriochrome Black-T (a blue coloured dye) when added to a sample of hard water buffered to pH value of about 9-10, combines with Ca^{+2} and Mg^{+2} ions of hard water to form an unstable wine red colored complex.

$$Ca^{+2} + EBT \rightarrow [Ca^{+2}-EBT]$$

(From hard water) *(Unstable wine red complex)*

$$Mg^{+2} + EBT \rightarrow [Mg^{+2}-EBT]$$

(From hard water) *(Unstable wine red complex)*

EDTA solution when added to hard water abstracts calcium and magnesium ions from the above unstable complex and forms a colorless stable complex with regeneration of blue colored dye (EBT).

$$[Ca^{+2}-EBT] + EDTA \rightarrow [Ca^{+2}-EDTA] + EBT$$

(Colorless stable complex) *(Blue color)*

$$[Mg^{+2}-EBT] + EDTA \rightarrow [Mg^{+2}-EDTA] + EBT$$

(Colorless stable complex) *(Blue color)*

Thus, the amount of EDTA used corresponds to hardness of water. The end point of titration will be change of color from wine red to blue.

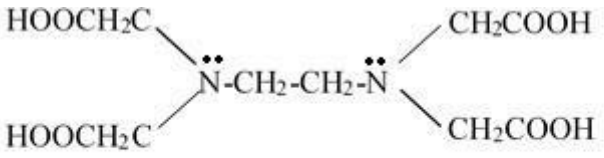

Fig. Structure of Ethylene Diamine Tetra-acetic Acid (EDTA)

Procedure: The experimental procedure involves the following steps

Step 1. Preparation of Standard Hard Water:

0.1 g of pure, dry $CaCO_3$ is weighed and dissolved in minimum quantity of dil. HCl to get clear solution and the volume is made up to the mark with distilled water in a 100ml volumetric flask.

Step 2: Standardization of disodium salt of EDTA

Pipette out 10 ml of standard hard water (V_1ml) into a clean conical flask, add 3 ml of buffer (NH_4Cl+NH_4OH) solution and 2 drops of Eriochrome Black-T indicator, upon which the solution would acquire a wine-red colour. Titrate with the solution of disodium salt of EDTA taken in the burette, till the wine red colour changes to blue which is the end point. Let the burette reading of EDTA be V_2 ml.

Step 3: Determination of Total Hardness

Repeat the above titration method for the given water sample instead of standard hard water. Let the burette reading of EDTA solution be V_4 ml.

Observations & Calculations:

Step 1: Molarity of standard hard water

Weight of $CaCO_3$ = _____

Molarity of $CaCO_3$ (M_1) = ____

Molecular weight of $CaCO_3$ =100

M_1 =

Step 2. Standardization of di sodium salt of EDTA:

S.No	Volume of Standard Hard water taken, V_1(ml)	Burette reading (ml)		Volume of EDTA solution consumed, V_2 (ml)
		Initial	Final	

By the law of equivalence, $\frac{M_1V_1}{n_1} = \frac{M_2V_2}{n_2}$ $(n_1 = n_2 = n)$

Where, M_1 = Molarity of standard hard water =

V_1 = Volume of standard hard water in conical flask=

M_2 = Molarity of EDTA =

V_2 = Volume of EDTA consumed =

$$M_2 = \frac{M_1V_1}{V_2}$$

Molarity of di sodium salt of EDTA solution =

Step 3. Determination of Total Hardness:

S.No	Volume of water sample taken, V_3 (ml)	Burette readings (ml)		Volume of EDTA solution consumed, V_4 (ml)
		Initial	Final	
1				
2				
3				

By the law of equivalence $\frac{M_3V_3}{n_3} = \frac{M_4V_4}{n_4}$ $(n_3 = n_4 = n)$

Where, $M_4 = M_2$ = Molarity of EDTA =

V_4 = Volume of EDTA consumed =

M_3 = Molarity of water sample

V_3 = Volume of water sample taken =

$$M_3 = \frac{M_4V_4}{V_3} = \frac{M_2V_4}{V_3} =$$

Molarity of given water sample =

Total Hardness = M_3 × Molecular Weight of $CaCO_3$ (100) × 1000

=

Result:

VIVA QUESTIONS:

1. Causes of Hardness.

   Presence of HCO_3^-, Cl^- , SO_4^{-2} ions of calcium and magnesium in water causes hardness.

2. Give the types of hardness.

   Hardness is of two types

   a) **Temporary Hardness:** It occurs due to the presence of HCO_3^- ions of calcium and magnesium in the water.

   b) **Permanent Hardness:** It occurs due to the presence of Cl^-, NO_3^- and SO_4^{-2} ions of calcium and magnesium in water.

3. What are the units of Hardness?

   The units of Hardness are mg/l, ppm, Degree Clark, Degree French

   (1ppm = 1mg/l)

4. Give the range of hardness (in ppm)

>75	Soft Water
75 – 150	Moderately Hard
150 -300	Hard
> 300	Very Hard

5. Define Buffer Solution.

   A solution which resist the change in pH on addition of small amount of strong acid or strong base.

6. Why do we use ammonia buffer?

   To maintain the pH in the range of 9 to 10.

   Ammonia Buffer: Ammonium Chloride and Ammonium Hydroxide. (pH of 9- 10).

7. Why the pH value is adjusted to 10?

   The efficiency of the complex formation of calcium and magnesium with EDTA is high at pH 9- 10, and the complexing efficiency decreases when the pH falls below 7.5 and with the increase in pH>10, the calcium and magnesium ions tend to form metal Hydroxides. The pH is adjusted to 10 by adding ammonia buffer.

8. Give the full form of EDTA.

   EDTA: Ethylene diamine tetra acetic acid. It is a complexing agent which forms chelating complexes with metal ions (Calcium and Magnesium ions).

9. Name the indicator and its color used in the titration.

   EBT: Eriochrome Black-T / Solochrome Black T. It is Blue in colour.

10. What are the different complexes formed in EDTA titration?

    Metal – Indicator Complex [Ca – EBT] or [Mg- EBT], Colour: Wine Red

    Metal – EDTA Complex [Ca – EDTA] or [Mg- EDTA], Colour: Colorless

    End Point: Wine red to Blue.

11. Why do we get blue colour as an end point?

    When EDTA is added to Metal – EBT complex, a stable Metal – EDTA complex is formed and EBT is released which is blue in colour.

12. Why do we use 100 in the final calculation?

    As molecular weight of $CaCO_3$ is 100.

    [Hardness of water sample = Molarity × Molecular Weight of $CaCO_3$ (100) × 1000]

13. Why is the hardness of water generally expressed as $CaCO_3$ equivalents?

    Hardness is expressed in terms of $CaCO_3$ equivalents because it is insoluble in water and its molecular weight is 100 which is easy for calculation.

14. Give the formula to calculate temporary hardness.

    Temporary Hardness of water sample = Total Hardness - Permanent Hardness

15. What is the other name for temporary and permanent hardness?

    Carbonate and Non – Carbonate hardness respectively.

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PERMANGANOMETRY

2. ESTIMATION OF IRON (II) USING POTASSIUM PERMANGANATE

Aim: To estimate the amount of Ferrous iron present in the given iron sample solution by Permanganometric method.

Apparatus: Burette, pipette, conical flask, beaker.

Chemicals: Potassium permanganate, Ferrous ammonium sulphate hexahydrate, H_2SO_4

Indicator: Potassium permanganate acts a self-indicator

End point: colorless to pale pink

Theory: Potassium permanganate is a powerful oxidizing agent in which manganese is present in its +7 oxidation state and Mohr's salt ($FeSO_4.(NH_4)_2SO_4.6H_2O$) contains iron in its +2 oxidation state which acts as a reducing agent. These two react in dilute sulphuric acid medium according to the partial equation:

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(O)$$

$$10FeSO_4 + 5H_2SO_4 + 5(O) \rightarrow 5Fe_2(SO_4)_3 + 5\,H_2O$$

Adding, the overall equation is

$$2\,KMnO_4 + 10\,FeSO_4 + 8\,H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5\,Fe_2(SO_4)_3 + 8\,H_2O$$

According to this overall balanced equation, one mole of permanganate reacts with five moles of Fe^{+2}. In this reaction, except permanganate, all other reactants and products are colorless. When all the Fe^{+2} ions are completely reacted, the excess drop of permanganate imparts pink color to the solution, which marks the end point of the titration. i.e., permanganate acts as self-indicator in this titration.

Procedure: - The experiment involves three steps

Step 1: Preparation of standard Mohr's salt solution:

About 0.8 g of Mohr's salt is carefully weighed and transferred in to a 100ml volumetric flask through the funnel, dissolve the salt in small amount of distilled water and add 2ml conc. sulphuric acid and the solution is made up to the mark with distilled water.

Step 2: Standardization of potassium permanganate solution with standard ferrous ammonium sulphate solution:

The burette is rinsed with the permanganate solution and filled with the same solution up to the mark. The initial reading of burette is noted.

10 ml of the Mohr's salt solution is pipetted out carefully into a clean 250 ml conical flask, followed by the addition 5 ml of 1:1 sulphuric acid by using a measuring jar and titrate with potassium permanganate solution until the first permanent pale pink colour appears which marks the end point. The titration is repeated until two successive titre values are concurrent. The observations are recorded in Table 1.

Step 3: Titration of Iron(II) sample with Standard $KMnO_4$:

10 ml of the Mohr's salt solution of unknown concentration is pipetted out carefully into a clean 250 ml conical flask, followed by the addition of 5 ml of 1:1 sulphuric acid by using a measuring jar. After gently swirling the contents of the conical flask to homogenize the solution, it is titrated with the permanganate solution drop-wise until a drop in excess imparts a permanent pale pink color, which marks the end point. The titration is repeated until two successive concurrent titre values. The observations are recorded in Table 2.

Observations and Calculation:

Step1: Normality of standard Ferrous ammonium sulphate

Weight of the salt =

$$\text{Normality } (N_1) = \frac{\text{weight of the salt}}{\text{Equivalent Weight of Mohr's salt}} \times \frac{1000}{100}$$

Equivalent Weight of Mohr's salt is 392.13

Table 1: Standardization of Potassium permanganate solution with standard Mohr's salt solution

S.No.	Volume of Standard Mohrs salt solution taken, V_1 (ml)	Burette reading (ml)		Volume of pot. permanganate solution run down, V_2 (ml)
		Initial	Final	
1				
2				
3				

Normality of standard Mohr's salt solution taken = N_1=

Volume of standard Mohr's salt solution taken = V_1=

Normality of Potassium permanganate solution consumed =N_2=?

Volume of Potassium permanganate solution consumed =V_2 =

By the law of equivalence: $N_1V_1 = N_2V_2$ or

$$N_2 = \frac{N_1V_1}{V_2}$$

Normality of Potassium permanganate solution (N_2) =

Table 2: Titration of unknown iron(II) sample solution with standard potassium permanganate solution

S. No.	Volume of Iron(II)solution taken, V_3 (ml)	Burette reading (ml)		Volume of pot. permanganate solution run down, V_4 (ml)
		Initial	Final	

Normality of Iron (II) solution taken = N_3=

Volume of Iron (II) solution taken =V_3=

Normality of Potassium permanganate solution consumed =N_4= N_2=

Volume of Potassium permanganate solution consumed =V_4 =

By the law of equivalence: $N_3V_3 = N_4V_4$

Or $N_3 = \frac{N_4V_4}{V_3}$ =

Normality of Iron (II) solution = N_3 =

Amount of ferrous iron present in the given iron sample: -

Equivalent weight of ferrous iron = Atomic weight /1 = 55.85/1 = 55.85

Amount of Fe^{+2} present in 1000 ml of iron solution (X)g = N_3 ×Eq. Wt of Fe^{+2} (55.85)

Amount of Fe^{+2} present in the given 100 ml of an Iron solution = X / 10 =

Result:

VIVA QUESTIONS

1. What type of titration is this?
   Redox titration (A titration which involves both oxidation and reduction)

2. Define oxidizing agent. Is $KMnO_4$ oxidizing or reducing agent?
   A substance that tends to bring about oxidation by being reduced and gaining electrons. $KMnO_4$ is an oxidizing agent.

3. Define reducing agent and name the reducing agent involved in the titration?
   A reducing agent is one that loses electrons and is oxidized to a higher valency condition. Ferrous ammonium sulphate, where Fe^{+2} is oxidized to Fe^{+3}

4. Write the balanced chemical equation of this experiment.
   $2\ KMnO_4 + 10\ FeSO_4 + 8\ H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$

5. What is the chemical name of the Mohr's salt?
   $FeSO_4.(NH_4)_2SO_4.6H_2O$

6. What were the oxidation states of Mn in $KMnO_4$ before and after the reaction?
   $K\ ^{+1}Mn^{+7}\ O_4^{-8} \rightarrow Mn^{+2}S^{+6}O_4^{-8}$

7. What is the purpose of adding sulphuric acid during the titration?
   Normal reduction of potassium permanganate to manganous salt is carried out only in the acidic medium.

8. Name the indicator used in the titration?
   Potassium permanganate itself acts as a self-indicator, and one excess drop of $KMnO_4$ gives pink colour at the endpoint indicating that the reaction is complete.

****

ARGENTOMETRY

3. DETERMINATION OF CHLORIDE CONTENT OF WATER

Aim: Determination of chloride content of water by Mohr's method or (Argentometric titration).

Apparatus: Conical flask, burette, pipette, volumetric flask, beakers.

Chemicals: Standard silver nitrate, potassium chromate

Indicator: Potassium chromate

End Point: Light yellow to brick red colour

Theory: The Sources of chloride in natural water is due to the presence of NaCl, KCl, $MgCl_2$ and $CaCl_2$. This method for determination of chloride in water is performed in the pH ranges 7-8. The Mohr method uses chromate ions as an indicator in the titration of chloride ions with a standard silver nitrate solution. After all the chloride has been precipitated as white silver chloride, the first excess of titrant results in the formation of a brick red silver chromate precipitate, which signals the end point. In the Mohr's method, the determination of end point is based on the formation of a second precipitate which is coloured. The requirement here is that silver chromate precipitate should have solubility slightly greater than the precipitate between the analyte and the titrant (Silver chloride precipitate).

$$Ag^+ + Cl^- \rightarrow AgCl \downarrow \quad \text{White precipitate}$$

$$2Ag^+ + CrO_4^{2-} \rightarrow Ag_2CrO_4 \downarrow \text{ Brick red precipitate}$$

Procedure:

Estimation of Chloride by using standard silver nitrate solution

1. Wash the burette with distilled water and rinse with standardised solution of silver nitrate ($AgNO_3$) and then fill the burette with the same.
2. Pipette out 25 ml of given water sample and transfer into a conical flask. Add 1ml of the potassium chromate indicator solution (advisable to use a pipette for this purpose).

3. Titrate the resultant solution with silver nitrate solution and ensure that the flask is constantly swirled.
4. As the titration proceeds slowly a red colour is formed and disappears slowly after addition of each drop of silver nitrate.
5. At this stage slow down the rate of addition of silver nitrate. The end point is indicated by sudden development of a faint but distinct change in colour to reddish-brown indicates the precipitation of silver chromate which does not disappear on swirling the conical flask.
6. Repeat titration for concurrent readings.

Observations and Calculations:

Estimation of Chloride by using standard silver nitrate solution

Table:

S.No.	Vol. of Water sample (ml)	Burette Reading		Vol. of silver nitrate run down (V_1 ml)
		Initial	Final	

Normality of Standard silver nitrate solution (N_1) =

Vol. of Silver nitrate (V_1) =

Volume of water sample = V_2 =

Amount of Chloride ions in mg/litre =

$$\frac{\text{Normality of } AgNO_3 \text{ x Volume of } AgNO_3 \times \text{equivalent weight of chlorine}}{\text{Volume of water sample}} \text{ x } 1000$$

Result: The Amount of Chloride in water sample is ppm or mg/L.

Viva Questions:

1. What are precipitation Titrations?

   Titrations based on the formation of a precipitate between the titrant and the analyte are termed as precipitation titrations.

2. How many precipitates are formed during the titration, Name them.

   Silver chloride white precipitate and silver chromate -brick red colour precipitate

3. Name any two methods for determination of chloride by using silver nitrate

   Mohr's method and Fajan's method

4. What is the role of chromate ions in chloride determination?

   In the presence of excess silver ions, solubility product of silver chromate (K_{sp}=1.1 $\times 10^{-12}$) exceeded and it forms a reddish-brown precipitate. (AgCl solubility product Ksp = 1.7 $\times 10^{-10}$).

5. Why pH range is important in chloride determination?

   If pH > 9.0, the silver ions react with hydroxide ions and precipitated as silver hydroxide. In contrast, at lower pH (acidic), potassium chromate may be converted into potassium dichromate ($K_2Cr_2O_7$) and mask the end point.

$$Ag^{+}_{(aq)} + OH^{-}_{(aq)} \rightarrow Ag(OH)_{(s)} \text{ (brown ppt)}$$

$$CrO_4^{2-}{}_{(aq)} \rightarrow Cr_2O_7^{2-}{}_{(aq)} \quad \text{(orange colour)}$$

ACIDIMETRY

4. DETERMINATION OF ACID VALUE OF COCONUT OIL

Aim: To determine the acid value of given coconut oil sample

Apparatus: Conical flask, pipette, burette

Chemicals: Sodium hydroxide/ potassium hydroxide, phenolphthalein indicator, ethanol, diethyl ether

Theory: Acid value of oil or fat indicates the amount of free fatty acids present in it. Acid value is defined as the number of mg of potassium hydroxide required to neutralize the free acid in 1g of fat in fatty oil. The value is a measure of the amount of fatty acids which have been liberated by hydrolysis from the glycerides due to the action of moisture, temperature and/or lipolytic enzyme lipase.

The extent of rancidity can be determined by acid value. Fats undergo rancidification and produce foul odour due to release of free fatty acids, low molecular weight aldehydes and ketones. Rancidity is of three types- oxidative, hydrolytic, and microbial rancidity.

Principle: A solution of known quantity of fat to be analysed is treated with a mixture of ethanol and diethyl ether, followed by titration of free fatty acid present in the ethanoic solution of KOH.

$$\text{Oil} + \text{alcohol} \rightarrow \text{free fatty acid} + \text{glycerol}$$

$$\underset{\text{(Free fatty acid)}}{RCOOH} + KOH \rightarrow RCOOK + H_2O$$

Procedure: Weigh accurately 1-5g of coconut oil in a 250ml conical flask. Dissolve oil in 25ml of solvent mixture (ethanol and ether) Shake the mixture to dissolve oil. Heat the mixture with shaking in a water bath until the substance is dissolved. Allow it to cool and add few drops of phenolphthalein and titrate the mixture with continuous swirling by using standard 0.1M KOH solution until permanent pink colour is obtained. (If solution became cloudy during titration add few ml of solvent mixture). Record the volume of KOH required for neutralization, repeat the experiment for at least two concurrent readings.

Observations and Calculations:

Estimation of acid value of an Oil:

S.No.	Weight of oil sample	Burette Reading		Vol. of KOH run down (V_{KOH} ml)
		Initial	Final	

Weight of empty bottle (w_1) = mg

Weight of bottle + oil (w_2) = mg

Weight of bottle after transferring the oil (w_3) = mg

Weight of the oil transferred = ($w_2 - w_3$) =

$$\text{Acid Value} = \frac{\text{Normality of KOH x Volume of KOH} \times \text{equivalent weight of chlorine x1000}}{\text{Weight of oil sample}}$$

Result: The acid value of coconut oil is

Viva Questions

1. Define Acid Value.
   Acid value is defined as the number of mg of potassium hydroxide required to neutralize the free acid in 1g of fat in the fatty oil.

2. What is an oil?
   Oil is an organic molecule of carbon, hydrogen, oxygen and sometimes nitrogen and sulphur. It is composed of long chain fatty acids and esters (glyceride esters) along with some other derivatives like sulphates, sulphonates etc.

3. What happens when alcohol is mixed with oil?
   Oil is non-polar but alcohol contains both polar and non-polar parts. Basing on the principle of 'like dissolves like' non-polar parts of oil evenly dissolves with the non-polar parts of alcohol.

4. Why determination of neutralization number or acid value number is important for engineers?
   i. Acid number represents the free acids that can corrode parts.
   ii. It is indicative of the age of the oil and determines when the oil should be changed.
   iii. Low Acid value indicates good cleansing by soap.
   iv. Aging of biodiesel can be determined by analysing its acid value (acid value of biodiesel should be < 0.5)

5. Difference between acid value and saponification number.
   Saponification Number: Alkaline hydrolysis of fat and oils is known as saponification number. This indicates average molecular weight of oil or fat and also forms the basis of soap making.
   Acid value indicates the proportion of free fatty acid present in oil or fat. Generally acid value for most of the samples lies within 0.5

6. What is Rancidity?

   The moisture and oxygen cause deterioration of fats. The combined action of both moisture and oxygen causes rancidity which is characterised by bad smell, development of red colour, increase in acidity, increase in viscosity. During frying, cooking and baking oils undergo hydrolytic and polymerisation reactions which are undesirable in edible oils. Prolonged exposure of oil to air develops resinous products causing rancidity.

7. What are the uses of oil?
   Oil is used for producing ghee, detergents, vanaspati, cosmetics, medicines, polymers, paints, varnishes and many other applications.

8. Acid value of Oils:
   Coconut oil – not more than6.0
   Edible Oils - <1%
   Pharmaceutical oils - acid value should be zero

CONDUCTOMETRY

5. CONDUCTOMETRIC TITRATION OF STRONG ACID WITH STRONG BASE

Aim: To determine the strength of HCl solution by titrating it against standard NaOH solution conductometrically.

Apparatus: Conductivity meter, burette, beaker

Chemicals: HCl, NaOH

Principle:

Conductometric titration is based upon the measurement of the conductance during course of titration. The number of free ions and mobility of the ions affect the conductance of an aqueous solution. When one electrolyte is added to another electrolyte, the change in number of free ions causes a change in the conductance. For example, when a strong acid (HCl) is titrated against a strong base (NaOH), the acid solution has high conductivity due to highly mobile H^+ ions just before NaOH solution is added from the burette. The conductivity of acid solution decreases while adding the NaOH, due to the neutralization of highly mobile H^+ ions of the acid with OH^- ions of the base.

$$H^+Cl^- + Na^+OH^- \longrightarrow NaCl + H_2O$$

Thus, the conductance of the solution continues to decrease until the equilibrium point is reached. Further addition of NaOH solution will increase the conductance by highly mobile hydroxyl (OH^-) ions. The reason for this is electrical conductance of solution depends upon the number of ions and their mobilities. Two straight lines are obtained on drawing a graph between conductance and volume of NaOH. On extrapolation, these lines intersect at a point that gives the end point and volume of titrant required for neutralization.

Procedure:

Measurement of the conductivity of the solution:

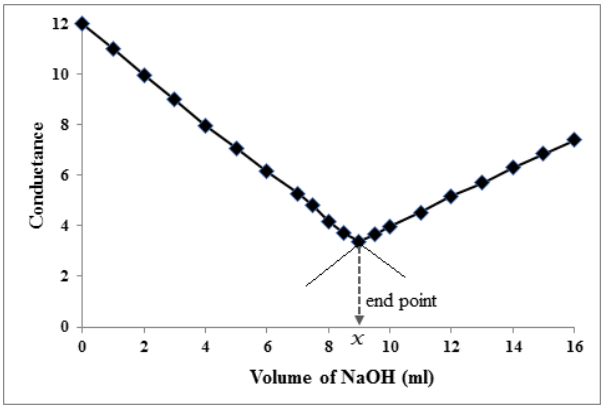
Pipette out 40 ml of the HCl solution into a 100ml beaker. Dip the conductivity cell in HCl solution after rinsing the conductivity cell with distilled water and HCl solution. Note the conductance value of HCl solution. Add 1 ml of NaOH solution taken in the burette to HCl solution and stir well. Note the conductivity of the solution after the addition of NaOH solution. Repeat the procedure by addition of 1 ml NaOH solution every time and the conductivity

readings of the remaining solution. Take 15 − 20 readings and note the readings in the given table.

Observations & Calculations:

Observed conductance readings are noted from the Table -1. A graph is plotted between observed conductance (Y-axis) and volume of NaOH added (X- axis). Equivalence point (neutralization point) is noted down from the graph.

Fig:- Graph of conductance Vs volume of NaOH solution



Operating Instructions:

Digisun Digital Conductivity meter:

1. Switch on the instrument and allow it to warm up for a minute or two.
2. Keep check / read switch at check position and selectors switch in 10K range and calibrate to 10.00
3. Dip the conductivity cell in HCl solution
4. Bring the switch to read position and note down the titration values.

EI -611 Conductivity Meter:

1. Switch on the instrument and allow it to warm up for a minute or two.
2. Set the temperature component to room temperature ($25/30^0$ C).
3. Select the range to 200ms
4. Keep the conductivity cell in the distilled water and the function knob in the check position and adjust the cell constant value to 1.000
5. Keep the conductivity cell in the test solution (HCl solution) and note the reading on the display.

Table:1 Determination of the strength of the given HCl:

S.No	volume of NaOH (ml)	Observe Conductance (Milli Mhos)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

The strength of the given acid (HCl) solution is determined in the following way

Volume of NaOH solution (V_1) =

Normality of NaOH solution (N_1) =

Volume of HCl solution (V_2) =

Normality of HCl solution (N_2) =

Normality of HCl (N_2) = $\frac{V_1 \times N_1}{V_2}$ =

Amount of HCl in the given solution = $N_2 \times$ Eq. wt of HCl

Result:

VIVA QUESTIONS

1. What are the advantages of conductometry titrations?
   Colored solutions for which no indicator is found can be successfully titrated by this method. Conductometric titrations are useful for very dilute solutions

2. Define conductance.
   Conductance: The reciprocal of resistance is called conductance or the flow of ions / flow of free electrons through a conducting medium. Units: Siemens, Mhos.

3. Define Conductometric titration.
   Titrations in which conductivity measurements are made use of in determining the end point of acid-alkali reactions are called Conductometric titration.

4. What is the shape of the graph obtained in the experiment, give reasons?
   V shaped graph is obtained; Conductivity decreases as the number of H^+ ions decreases gradually due to neutralization by the OH^- ions of NaOH and then increases due to the accumulation of the OH^- ions.

5. Give reason for decrease and increase of conductance.
   The conductance depends on the mobility of the ions, initially the conductance is high but with decrease in concentration of H^+ ions, conductance decreases and after reaching end point the conductance increases because of OH^- ions.

6. Name the Electrode used?
   Conductivity cell: It consists of two platinum electrodes separated by unit distance apart and the cell is filled with electrolytic solution.
   Its cell constant is 1. (units Cm^{-1})

7. How can we plot the neutralization point from the conductivity graph?
   The point of Intersection of the two linear curves plotted graphically gives the neutralization point.

****

POTENTIOMETRY

6. ESTIMATION OF Fe (II) USING POTENTIOMETER

Aim: To estimate the amount of Fe (II) present in the given solution potentiometrically.

Apparatus: Pipette, burette, beakers, calomel and platinum electrodes, glass rod

Chemicals: Standard potassium dichromate, ferrous ammonium sulphate and H_2SO_4

Principle: Potentiometric titration involves the measurement of potential between two electrodes, one is an indicator electrode whose potential changes with that of the pH of the solution and the other a reference electrode of constant potential. The equivalence point of the reaction will be revealed by sudden change in the potential.

Estimation of ferrous iron by using potassium dichromate is a redox titration which involves oxidation and reduction. During their titration, the solution potential changes due to the changein the concentration of oxidised/reduced form. From the Nernst equation, we know that the potential of a given reaction will depend on the relative concentration of oxidised/reduced form.

Potassium dichromate is an oxidising agent and in acid medium, it follows the half reaction to give Cr (III) as the reduction product.

$$Cr_2O_7^{-2} + 14H^+ + 6e \rightarrow 2\ Cr^{3+} + 7\ H_2O\ (Cr^{6+}/\ Cr^{3+} - 1.33V)$$

While Fe^{2+} gets oxidised to Fe^{3+} as per the reaction

$$Fe^{2+} \rightarrow Fe^{3+} + e^- \qquad (Fe^{2+}/\ Fe^{3+} - 0.77V)$$

The overall ionic equation of this titration can be obtained by adding the above two:

$$Cr_2O_7^{-2} + 6\ Fe^{2+} + 14H^+ \rightarrow 6\ Fe^{3+} + 2Cr^{3+} + 7\ H_2O$$

$$H_2O\ (or)\ Cr^{6+} + Fe^{2+} \rightarrow Fe^{3+} + 2Cr^{3+}$$

The EMF of cell is given by $E = E_{cathode} - E_{anode}$

Cell representation: SCE | Fe^{2+} | Fe^{3+} || Cr^{6+} | Cr^{3+} |Pt

Procedure:

1. **Pilot Titration:** Take 40 ml of ammonium ferrous sulphate in a 100ml beaker, add 10 ml of dilute sulphuric acid and 5ml of distilled water. SCE is used as a reference electrode and platinum foil which acts as indicator electrode are dipped in Fe^{2+} solution. Carry out

necessary connections. Add 1ml of 0.04N $K_2Cr_2O_7$ from the burette and stir the solution, wait for a minute and note the EMF. After every addition of 1ml titrant, stir the solution and note the potential reading. An abrupt change in the potential gives a rough estimate of the equivalence point.

Graphs:

1. Potentiometric titration curve (S-shaped curve) is obtained by plotting a graph between volume of $K_2Cr_2O_7$ (V) and potential (E)
2. The volume of $K_2Cr_2O_7$ required for titration of Fe^{2+} (endpoint) is located from the first derivative curve (**V** and **$\Delta E/\Delta V$**).

Model Graphs:

Fig-1: Potentiometric curve

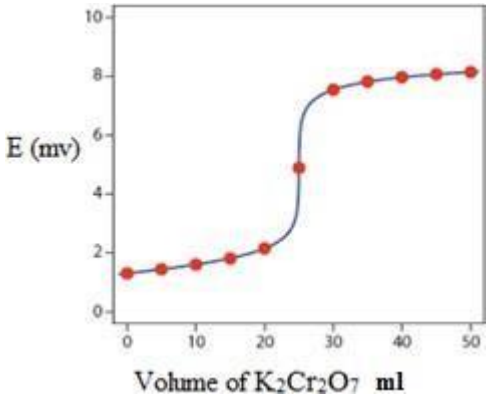
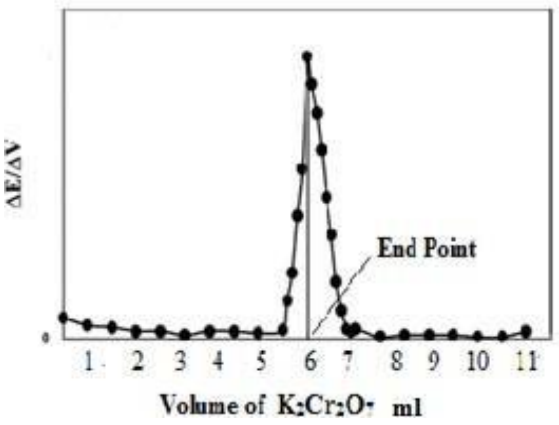


Fig-2: First derivative curve



Observations and Calculations:

1. Pilot Titration:

S.No.	Vol. of $K_2Cr_2O_7$ ml	Potential (E) (mv)	ΔV	ΔE	ΔE/ΔV
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					

From the plotted curve, the volume of $K_2Cr_2O_7$ used corresponding to the equivalence point (V_1) ml =

$$N_1 \times V_1 = N_2 \times V_2$$

Normality of $K_2Cr_2O_7$ (N_1) =

Volume of $K_2Cr_2O_7$ (V_1) =

Volume of Fe (II) solution (V_2) =

Normality of Fe (II) solution (N_2) =

Normality of Fe (II) solution (N_2) = $\dfrac{N_1 \times V_1}{V_2}$ =

Amount of ferrous iron present in the given iron sample: -

Equivalent weight of ferrous iron = Atomic weight /1 = 55.85/1 = 55.85

Amount of Fe^{+2} present in 1000 ml of iron ore solution = N_2 ×Eq.Wt of Fe^{+2} (55.85) =

Amount of Fe^{+2} present in the given 100 ml of an Iron ore solution = x / 10 =

Result: Amount of Fe^{+2} present in the given 100 ml of an Iron sample solution is

VIVA QUESTIONS

1. What are potentiometric titrations?
   Potentiometric titrations involve EMF measurement and applied for colored and very dilute solutions.
2. Name the electrodes used in Potentiometric redox titration?
   Saturated Calomel Electrode (SCE) as reference Electrode, Platinum as an indicator electrode at which potential change is observed.
3. Which electrode serves as a measurement of change in potential & give the end point?
   Indicator Electrode (Platinum)
4. What is indicator electrode?
   The electrode whose potential is dependent upon the concentration of the ion to be determined.
5. Give the principle involved in potentiometric titration?
   The principle involved in potentiometric titration is the measurement of the EMF betweentwo electrodes, an indicator electrode and reference electrode. In these titrations, measurement of EMF are made while the titration is in progress. The equivalence point of the reaction is revealed by a sudden change in the potential.
6. Why the beaker solution gradually changes to green during the course of titration?
   When Fe(II) reacts with $K_2Cr_2O_7$, $Cr_2(SO_4)_3$ is formed which is green salt solution.
7. What is the reaction between ferrous ion and $K_2Cr_2O_7$?
   Acidified potassium dichromate oxidises ferrous sulphate to ferric sulphate and itself gets reduced to chromic sulphate.
8. Why sulphuric acid is added during the titration?
   $K_2Cr_2O_7$ oxidises ferrous ion only in presence of acidic medium.
9. Give the EMF of total cell
   $E_{cell} = E_{cathode} - E_{anode}$

****

ADSORPTION

7. Determination of Adsorption Isotherm of Acetic acid by Charcoal

Aim: To determine the Freundlich adsorption isotherm of acetic acid by activated charcoal.

Chemicals: 0.5M acetic acid, activated charcoal powder, 0.1M NaOH, phenolphthalein.

Apparatus: stoppered bottles (5 nos.), burette, pipette, funnel

Theory:

Adsorption is the phenomenon of accumulation of high concentration of molecules (called as adsorbate) on a solid surface (called as adsorbent). This occurs because of the unsatisfied bonds present on the surface of solid and do not penetrate deeper into the bulk. For a given weight of adsorbent, its adsorptive capacity is directly proportional to the surface area. There are mainly three types of isotherms namely Freundlich, Langmuir, and BET isotherms. In this experiment we will study and verify Freundlich adsorption isotherm of acetic acid (adsorbate) on charcoal (adsorbent) at a constant temperature.

In a Freundlich isotherm, the mass of a solute (or gas) adsorbed by a solid adsorbent at various concentrations (or pressures) is given by an empirical equation:

$$\frac{x}{m} = k \cdot c^{1/n}$$

Where 'x' is the mass of the solute (or gas) adsorbed by 'm' grams of solid adsorbent at various concentrations (or pressures), k and n are constants for a given solid adsorbent and solute.

The above equation can be written as,

$$\text{Log } x/m = \log k + 1/n \log C$$

A plot of log x/m versus log C gives a straight line at low concentration indicating the direct relationship of the amount of adsorption with concentration for low values with intercept log k and slope equal to 1/n.

Procedure:

1. Take 5 clean, dry, stoppered bottles of 100 ml capacity. Number them from 1 to 5.

2. Take 2 burettes and fill them with acetic acid and distilled water respectively.

3. Fill the bottles with different volumes of acetic acid and water as given in the table 1.

4. Add 2 g of charcoal in each bottle.

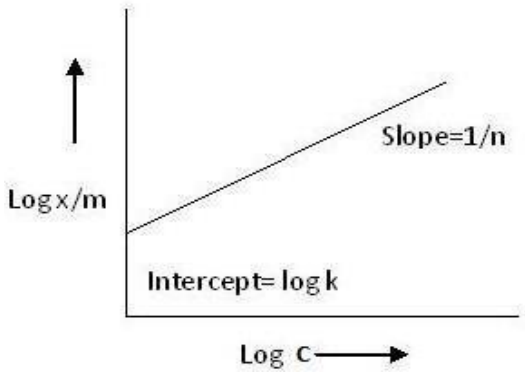
5. Shake the bottles for 20 minutes vigorously and allow to stand.

6. Filter the contents of each bottle through filter paper. Pipette out 10 ml of the filtrate into 100ml conical flask and titrate against standard NaOH using phenolphthalein indicator. Tabulate the results in table 2.

7. Calculate the change in concentration of acetic acid for all the 5 bottles in gms and tabulate the results in the table 3.

8. Plot the data by taking log C on X-axis and log x/m on Y-axis, Note the slope and intercept of the straight line obtained.

Model Graph:



Observations and Calculations:

Table 1: Different proportions of acetic acid and water to get different concentrations of acetic acid in the 5 bottles.

Bottle No.	Volume of acetic acid (ml) 0.5M	Volume of water (ml)	Total volume of mixture (ml)	Intial conc. Of acetic acid (M)
1	50	0	50	0.5
2	40	10	50	0.4
3	30	20	50	0.3
4	20	30	50	0.2
5	10	40	50	0.1

Table 2: Titrations of acetic acid in the filtrates of all the bottles with NaOH solution

Bottle No.	Volume of filtrate (ml)	Volume of NaOH consumed (ml)
1	50ml	Initial Burette Reading:
		Final Burette Reading:
		V_1=
2	40ml	Initial:
		Final:
		V_2=
3	30ml	Initial:
		Final:
		V_3=
4	20ml	Initial:
		Final:
		V_4=
5	10ml	Initial:
		Final:
		V_5=

Calculation for Bottle 1:

Molarity of NaOH (M_1) =

Volume of NaOH (V_1) =

Volume of acetic acid taken from the filtrate (V_2) = 10ml

Molarity of acetic acid (M_2) =

$$\frac{M_1V_1}{n_1} = \frac{M_2V_2}{n_2}$$

Since $n_1 = n_2 = 1$, $M_1 V_1 = M_2 V_2$

i). Concentration of acetic acid after adsorption (M_1) = $\frac{M1\ V1}{V2}$

ii). Change in equilibrium concentration of acetic acid after adsorption = $(0.5 - M_1)$

iii). Change in concentration expressed in terms of amount (gm) per 50 ml of the solution (Bottle1)

$X_1 = \frac{\text{Change in concentration in moles x Volume taken}}{1000}$ x Mol.Wt. of acetic acid

$= \frac{(0.5 - M1) X 50}{1000}$ x 60

iv). Similarly, calculate X_2, X_3, X_4, and X_5 for the remaining bottles numbered 2, 3, 4, 5

Tabulate the results in Table 3 below.

Table 3: Calculated data of log(x/m) and logC

Bottle No.	Initial conc. of Acetic acid (M)	Equilibrium conc. of acetic acid after adsorption	Amount of acetic acid adsorbed (x)	Weight of adsorbent (m)	x/m	Log x/m	Log C
1	0.5						
2	0.4						
3	0.3						
4	0.2						
5	0.1						

Viva Questions

1. What is the difference between adsorption and absorption?
   Adsorption is the accumulation of gas or liquid solute (adsorbate) on surface of a solid or a liquid (adsorbent) forming a molecular or atomic film. Absorption is the diffusion of a substance into a liquid or solid to form a solution.

2. Give some examples of Adsorption materials.

   Charcoal, silica gel, clay etc.

3. What are the applications of adsorption materials?
   Adsorbent materials are widely used at water treatment plants to remove especially organic impurities and chlorine as purification treatment.

4. What do you mean by adsorption isotherm?
   The relation between the amount adsorbed by an adsorbent and the equilibrium pressure of the absorbate at a constant temperature is called an adsorption isotherm.

5. What are the applications of adsorption?
   Various applications of adsorption are
   i. To remove poisonous gases from air
   ii. To remove colouring impurities in production of sugar, vinegar etc.
   iii. To create vaccum e.g., charcoal adsorbs all gases at low temperature
   iv. Graphite acts as a lubricant due to adsorption of gases

6. What factors influence the adsorption on solid surfaces?
   The extend of adsorption of a gas on a solid surface depends upon the following factors:
   i. Nature of gas and the solid
   ii. Surface area of the adsorbent
   iii. Pressure of the gas
   iv. Temperature
   v. Activated state of adsorption

CHEMICAL KINETICS

8. DETERMINATION OF RATE CONSTANT OF ESTER HYDROLYSIS

Aim: To determine the rate constant of the acid-catalysed hydrolysis of methyl acetate.

Chemicals: Methyl acetate, hydrochloric acid, sodium hydroxide, phenolphthalein indicator.

Apparatus: Burette, pipette, conical flasks, thermostat, ice.

Principle: Methyl acetate undergoes hydrolysis reaction in the presence of an acid to produce acetic acid and methyl alcohol.

$$CH_3COOCH_3 + H_2O \xrightarrow{H+} CH_3OH + CH_3COOH$$

The rate of the reaction is given by $dx\ dt = [CH3COOCH3]\ [H2O]$ Where k is called the reaction rate constant.

Since, water is present in large excess, the above equation reduces to pseudo first order by including the water term in the rate constant, $dx/$ dt$= k'$ $[CH_3COOCH_3]$ i.e., the rate of the reaction is determined by ester concentration term. Rate constant k' is given by $k' = 2.303\ t\ log\ a/\ a$–x Where the term 'a' is initial concentration of ester, (a-x) is concentration of ester after time 't'.

By substituting the volume terms of the reaction mixture at different stages of the experiment, the equation becomes

$k' = 2.303/\ t^*\ log\ (V\infty - V_0\ )/\log(V\infty - Vt)$

Procedure: The progress of the reaction is followed by removing a definite volume of reaction mixture at definite intervals of time, followed by quenching the reaction, and titrating the acetic acid formed against standard sodium hydroxide solution.

1. Take 100 ml of HCl solution in a reagent bottle
2. Add 5 ml of ester to HCl. Start the Stop clock immediately. Shake the mixture thoroughly.

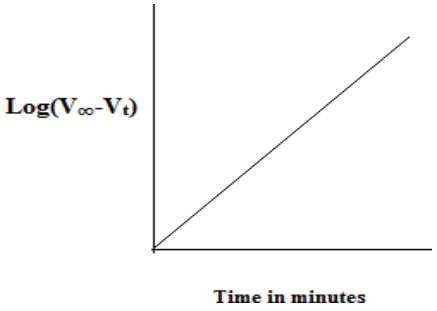
3. Pipette out 5 ml of reaction mixture and pour it onto 50 ml of ice cold distilled water in a 100 ml conical flask.
4. Titrate the reaction mixture against standard NaOH using phenolphthalein indicator. Titre value corresponds to V_0.
5. Steps 3 & 4 are repeated at intervals of 5, 10, 15, 20, 30, 45, 60 min. Titre values correspond to V_t.
6. The remaining solution is left overnight to repeat steps 3 & 4. Titre value gives V_∞.
7.
8. Draw a graph between $Log(V_\infty - V_t)$ and t, to determine the slope of the straight line obtained.
9. Calculate the theoretical and graphical k' values.

The data obtained is summarized in the table below.

Table: Titration data of the volumes of NaOH consumed at different stages of the reaction

S.No.	Time (min)	Volume of reaction mixture Taken (ml)	Volume of NaOH Consumed (V_t ml)	$V_\infty - V_t$	$Log(V_\infty - V_t)$	$\frac{Log(V\infty - V0)}{Log(V\infty - Vt)}$	$K = \frac{2.303}{t(sec)} \frac{Log(V\infty V0)}{Log(V\infty - Vt)}$
1.	0		V_0				
2.	5						
3.	10						
4.	15						
5.	20						
6.	30						
7.	45						
8.	60						
9.			V_∞				

Model Graph:



Result:

Rate constant obtained, k' =

Viva Questions:

1. What are the factors on which rate of a reaction depend?
   Concentration of reactants, temperature, catalyst.

2. Why certain reactions are very fast?
   Very low activation energy

3. What will be the effect of temperature on reaction rate?
   For most of the reactions the rate becomes nearly double or even more for 10^0C rise of temperature.

4. Why is the reaction rate increases with increase in temperature?
   The number of collisions among the reactant molecules increase with increasing temperature.

5. What is the role played by kinetics and thermodynamics for a chemical reaction?
   Chemical reactivity is controlled by two broad factors: thermodynamics and kinetics. Thermodynamics considers the question: which state is more stable, reactants or products; Kinetics- what controls the rate of a reaction?

PARTITION COEFFICIENT

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

Aim: To determine the partition coefficient of acetic acid distributed between n-butanol and water.

Chemicals: Acetic acid, n-butanol, distilled water, phenolphthalein indicator, NaOH solution

Apparatus: Stoppered glass bottle, burette, pipette, conical flask

Theory: A solute in contact with two immiscible solvents (which are in contact with each other), will distribute itself between these two solvents at constant temperature and the ratio of the concentrations of solute in the two layers is a constant. According to Nernst distribution law, partition coefficient or distribution coefficient

$$K_d = \frac{c_1}{c_2}$$

where c_1 and c_2 represent the concentrations of solute in solvent 1 and solvent 2 respectively.

Procedure:

1. Take 25 ml of CO_2-free distilled water in a 100 ml glass stoppered bottle and add 20 ml of n-butanol followed by 5 ml of acetic acid.

2. Shake the bottle vigorously for about 10 min and keep it aside for 5 min for the two liquid layers to separate out.

3. Pipette out 5 ml of aqueous layer into a 100 ml conical flask. Add 2 to 3 drops of phenolphthalein indicator and titrate against standard NaOH solution until the colourless solution changes to pale pink.

4. Similarly, pipette out 5 ml of organic layer into 100 ml conical flask. Add 2 to 3 drops of indicator and titrate against standard NaOH solution until the colourless solution turns to pale pink.

5. Experiment is repeated till concurrent readings are obtained.

The following table summarises the data obtained during the experimentation

S.No.	Volume of organic layer taken (ml)	Volume of aqueous layer Taken (ml)	Amount of NaOH consumed	Partition coeff. $K_d = \frac{C1}{C2}$

Result:

Partition coefficient of acetic acid between n-butanol and water, K_d =

Viva Questions:

1. Why does n-butanol and water are immiscible?

   As a general rule in Chemistry, like dissolves like. Water is a polar compound, and thus readily will dissolve or solubilize other polar compounds, such as ethanol. Polarity of n-butanol is low.

2. What are the factors that effect the distribution coefficient?

   The value of distribution coefficient depends upon, nature of the solute, nature of the solvent, and temperature

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VISCOMETRY

10. DETERMINATION OF VISCOSITY OF GIVEN LIQUID BY OSTWALD VISCOMETER

Aim: To determine the viscosity of given liquid by using Ostwald viscometer.

Apparatus: Ostwald viscometer, stop watch, pipette

Chemicals: Distilled water, unknown liquids.

Theory: When a liquid flows, it has an internal resistance or an internal friction to flow. Viscosity is the property of the fluid which offers resistance to its own flow. Honey flows less readily than water because honey has higher viscosity than water.

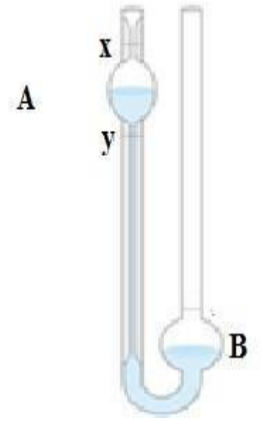
In the lab for comparing the viscosities of different liquids, the commonly used instrument is Ostwald's viscometer. In this operation, different liquids are taken exactly in the same volume. Ostwald's viscometer has two bulbs one at upper end (A) and other at lower end(B). The liquids are sucked up in the upper bulb A. Initially, enough liquid volume is taken in bulb B such that when the liquid stands above the mark x in bulb A, a little is still left in the bulb B. The time of flow (t) is measured for each liquid for its flow from x to y. The driving pressure (p) at all stages of flow in the liquid is given by 'hdg', where h is difference in heights of liquids in upper and lower ends and d is density of liquid and g is acceleration due to gravity.

Viscosity is measured by using the formula $\frac{\eta_l}{\eta_w} = \frac{t_l d_l}{t_w d_w}$

Procedure:

1. Take a clean and dry Ostwald viscometer and set it vertically on a stand.
2. Introduce 10ml of distilled water into the larger bulb B.
3. Suck the liquid up into the bulb A through a rubber tubing attached to the end to a level above the mark 'x'.
4. Allow the liquid to flow freely through the capillary and note the time t_w for the liquid to flow from mark x to y.
5. Repeat the procedure 2 to 3 times and get an average value of t_w.
6. Dry the viscometer in an oven and then repeat steps 2 and 5 to obtain average time for the unknown liquid to flow from mark x to y.

Ostwald's Viscometer



$$\frac{\eta_l}{\eta_w} = \frac{t_l d_l}{t_w d_w}$$

η = viscosity

t = time of flow

d = density

Observations & calculations:

S.No.	Liquid	Time of flow in seconds			Time (Average)	Viscosity (milli poise)
		t_1	t_2	t_3		
1.	Water					
2	Unknown liquid					

$$\frac{\eta_l}{\eta_w} = \frac{t_l d_l}{t_w d_w}$$

η_l =Viscosity of given liquid=?

η_w = Viscosity of distilled water = 0.7975millipoise

d_l = density of liquid=

d_w= density of distilled water=1g/cc

t_l= average time of flow of liquid=

t_w = average time of flow of distilled water=

$$\eta l = \frac{t_l d_l}{t_w d_w} \eta_w$$

Result:

VIVA QUESTIONS

1. Define Viscosity.
   Property of a fluid that determines its resistance to flow. Units: Poise

2. Give the formula to calculate viscosity?
   Viscosity is measured by using the formula $\frac{\eta_l}{\eta_w} = \frac{t_l d_l}{t_w d_w}$

3. Viscosity of distilled water=0.7975milli poise

4. Name some viscometers used to determine viscosity of lubricants?
   Red Wood Viscometer, Saybolt Viscometer, and Englers
   Viscometer

5. As the temperature increases viscosity decreases, because the time of flow of lubricant decreases with increasing of temperature.

6. In light machinery, thin oil with low viscosity is used, in clocks thin lubricant oil is used. In Heavy machinery, heavy oil with high viscosity is used, example Grease.

7. Give some practical application of Viscosity?
   i) Viscosity affects heat generation in bearings, cylinders and gear sets related to an oil's internal friction.
   ii) If toothpaste does not have the correct viscosity, it can either be too difficult to pump out from the tube, or pump out too much.
   ii) The viscosity of sea water makes the waves subside during the storm.
   iv) The motion of the objects in fluids depends upon the viscosity of the fluids
   v) If the arteries and veins of human body contract, their diameter decreases, and flow of blood is affected due to the viscosity of blood and blood pressure increases.
   vi) Viscosity determines the ease with which machine get started as operated under varying temperature conditions, particularly in cold climates. It also governs the sealing effect of oils and the rate of oil consumption.

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SURFACE TENSION

11. DETERMINATION OF SURFACE TENSION OF A LUBRICANT BY USING STALAGMOMETER

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

Apparatus: Stalagmometer, beaker etc.

Chemicals: Unknown liquid, distilled water

Theory: 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. There are two methods of determining surface tension

1. Drop weight method and 2. Drop number method

The liquids with large intermolecular forces would have higher surface tension. Thus, water has highest surface tension (72.8 dyne/cm or 0.0728 Newton/m). Surface tension of a liquid is measured by using an apparatus called Stalagmometer.

Principle: The size of the drop falling down at the end of the tube depends on surface tension of the liquid and size of capillary end. Thus, when a liquid is allowed to flow through a capillary tube, a drop will increase in size to a certain extent and then fall off.

- The total surface tension of supporting the drop is $2\pi r \gamma$, where r= radius of outer circumference of dropping end of capillary tube.
- Surface tension of liquid can therefore be determined from weight of single drop and external radius of dropping tube as W= $2\pi r$ where W= weight of the drop and $2\pi r$ is the circumference of the external wall of the capillary tube.
- For two liquids, $W_1 = 2\pi r\gamma_1$, $W_2 = 2\pi r\gamma_2$

$$\frac{w_1}{w_2} = \frac{\gamma_1}{\gamma_2}$$

- Thus, drop weight method can be employed for comparing surface tension of 2 different liquids. However, if it is easy to count the no. of drops by equal volumes of 2 liquids than finding the weight of single drop.

- For two different liquids, the weight of equal volumes are proportional to their density. Let N_1, N_2 be the no. of drops of 2liquids.

Volume of single drop $= \frac{V}{N_1}$

Weight of single drop $= V\frac{d_1}{N_1}$

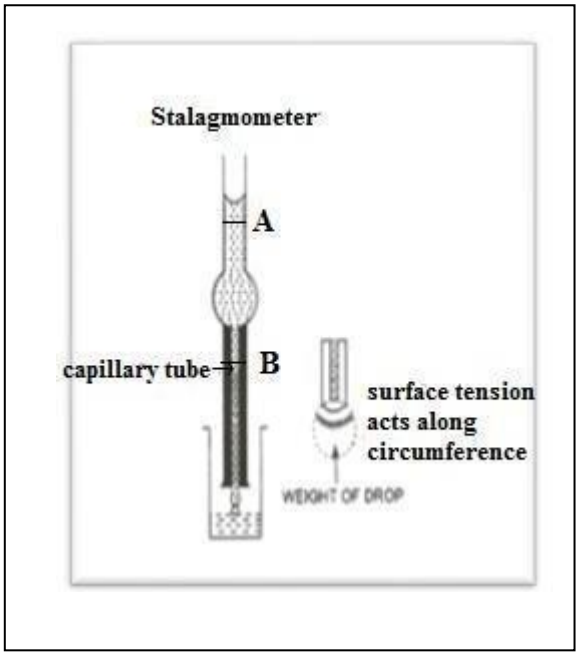
$$\therefore 2\pi r\gamma_1 = V\frac{d^1}{N_1} \longrightarrow (1)$$

Similarly, for 2nd liquid

$$\therefore 2\pi r\gamma_2 = V\frac{d^2}{N_2} \longrightarrow (2)$$

Dividing these two equations i.e., 1/2 , we get,

$$\frac{\gamma_1}{\gamma_2} = \frac{N_2 d_1}{N_1 d_2} \text{ or } \frac{\gamma_l}{\gamma_w} = \frac{N_w d_l}{N_l d_w}$$



Procedure: Take a clean and dry Stalagmometer.

- Suck the distilled water to the level above the mark in stalagmometer.
- Now allow the liquid to flow and count the no. of drops falling from upper mark to the lower mark.
- Repeat the above procedure with the given liquid.

Observations &Calculations:

S.No	Liquid	No. of drops			Surface tension (dynes/cm)
		N_l	N_2	Average	
1	Distilled water				72.8
2	Unknown organic liquid				

Calculate the surface tension by using the formula:

$$\frac{\gamma_l}{\gamma_w} = \frac{N_w d_l}{N_l d_w}$$

Surface tension of water (γ_w) = 72.8 dynes/cm

Density of water (d_w) = 1 g/cc.

Density of unknown liquid (d_l) = g/cc

No. of drops of water (N_w) =

No. of drops of liquid (N_l) =

Surface tension of the liquid (γ_l) =

$$\text{Now } \gamma_l = \frac{N_w d_l \gamma_w}{N_l d_w}$$

Result:

VIVA QUESTIONS

1. Define Surface tension.

   The tangential cohesive force acting along the unit length of the surface of a liquid or

   The tension of the surface film of a liquid caused by the attraction of the particles in the surface layer by the bulk of the liquid, which tends to minimize surface area.

   Formula to calculate surface tension

$$\frac{\gamma_l}{\gamma_w} = \frac{N_w d_l}{N_l d_w}$$

2. What are units of surface tension in C.G.S. and S.I. (M.K.S.) system?
   Dynes/cm(C.G.S.) and Newton / meter (M.K.S.)

3. What are cohesion and adhesion forces?
   Cohesive force is the attractive force between like molecules, whereas, the adhesion is the attractive force between unlike molecules, e.g. attraction between glass slide and the liquid.

4. What are the factors affecting the surface tension?
   (a) Nature of liquid (b) Nature of the surface in contact (c) Temperature

5. What is the effect of temperature on surface tension?
   Surface tension decreases with the rise of temperature.

6. Define critical temperature?
   The temperature at which the surface tension is zero.

7. What is the shape of free surface at critical temperature?
   At any critical temperature, the surface tension because zero hence the free surface is flat.

8. Give some practical applications of surface tension
   i) A drop of falling liquid is always in spherical shape.
   ii) We use oily substances to set out hairs.
   iii) Functioning of ink jet printer depends on surface tension.
   iv) Capillary action e.g. rising of oil in the wick of a lamp.
   v) Accurate method of measuring surface tension is capillary action, capillary action in part determines the behaviour of ground water in the soil which makes it important to civil and environmental engineers in understanding the stability of building and roads as well as environmental impact of human development.
   vi) Flying insects can walk on water surface without getting their feel wet.
   vii) The surface tension of paints and other surface coating is carefully designed to ensure that the coating spreads easily while maintaining a desired film thickness.
   viii) Environmental and chemical engineers study the interaction between surface tension and chemicals as they develop technologies to remove pollutants from water and air resources.

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DRUG SYNTHESIS -1

12. PREPARATION OF ASPIRIN

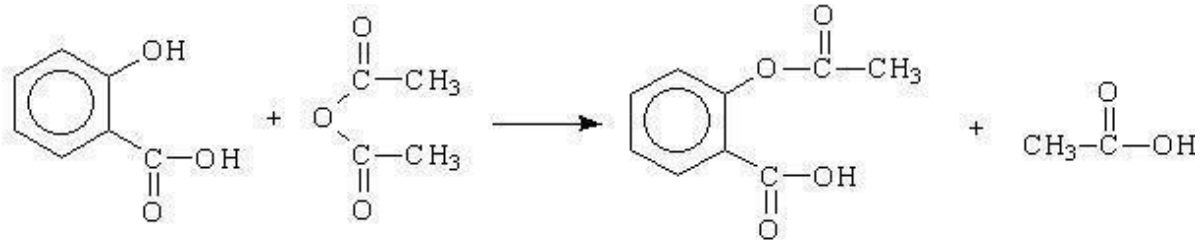
Aim: To prepare a sample of aspirin drug from salicylic acid.

Chemicals: Salicylic acid, conc. H_2SO_4, acetic anhydride.

Apparatus: Beakers, funnel, conical flask, glass rod, filter papers, filtration unit etc.

Theory: The chemical name of aspirin is acetyl salicylic acid. Aspirin is extensively used as an antipyretic, analgesic, anti-inflammatory drug, as a pain killer in treatment of arthritis and as a medicine which prevents heart attack by checking clotting in arteries.

Principle: Salicylic acid undergoes O-acetylation with acetic anhydride in the presence of conc.H_2SO_4 to give O-acetyl salicylic acid.



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

Procedure:

Take 2gm of salicylic acid, 4 ml of acetylating mixture (acetic anhydride and glacial acetic acid) and 1-2 drops of conc. sulphuric acid in a round bottomed flask/ conical flask. The mixture is warmed on water bath at 50-60^{0}C with occasional shaking for 30minutes. Allow the solution to cool to room temperature and pour the solution into 100ml of cold water taken in 250ml beaker with stirring (to destroy the excess of reagents). Scratch the sides of beaker with a glass rod to induce crystallization and stir the solution till complete precipitation. Filter the colourless crystalline solid and wash it with cold water. Spread the solid aspirin on a filter paper and dry the solid.

The crude aspirin so obtained is recrystallised by dissolving it in alcohol and pouring it into 40ml of warm water. The solution which then becomes turbid is warmed to give a clear solution and then allowed to cool slowly. The needles which separate out are filtered. The melting point of the sample of aspirin is 130^0-135^0 C.

Precautions:

- Take acetyl chloride in excess as it can act as acetylating reagent and solvent. Make sure that all the salicylic acid is dissolved in acetylating mixture.
- The presence of un-reacted salicylic acid is checked by adding a drop of 1% of FeCl3 solution to reaction mixture and observing the colour.

Calculations:

Result: The yield of prepared Aspirin is_____gm.

Melting Point of Aspirin is _____

Viva questions:

1. What is the chemical name of Aspirin?

   The chemical name of aspirin is acetyl salicylic acid.

2. What are the uses of aspirin?

   It is used as antipyretic, anti-inflammatory and as analgesic.

3. What is meant by acetylating mixture?

   Acetic anhydride and glacial acetic acid is acetylating mixture.

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DRUG SYNTHESIS -2

13. PREPARATION OF PARACETAMOL

Aim: To prepare paracetamol from 4- aminophenol.

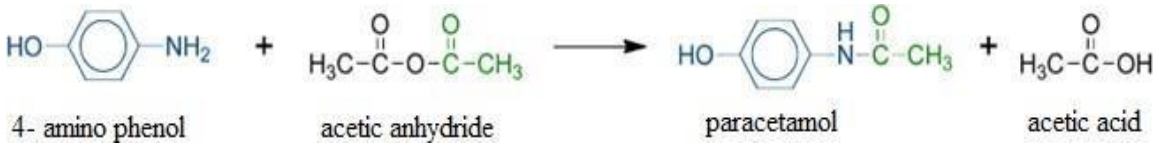
Chemicals: 4- aminophenol, ethanoic anhydride (acetic anhydride).

Apparatus: Beakers, buchner funnel, conical flask, magnetic stirrer.

Principle:

Synthesis of paracetamol by acetylation:

The objective of the present experiment is to synthesize an aromatic amide by addition-elimination reaction with acetic anhydride. In this case, p-aminophenol, the amine, is treated with acetic anhydride to form acetaminophen (p-acetamidophenol), the amide.



PROCEDURE:

2.0 grams of 4-aminophenol, 20ml of distilled water and 3ml of acetic anhydride are taken in a round bottom flask fitted with a reflux condenser. The mixture is refluxed for 15 mins until the reaction mixture becomes colorless. The hot liquid is poured on crushed ice with stirring in a beaker. Filter the colorless crystalline solid, wash it with cold water and then dried. For recrystallization (to attain purity) solid is dissolved in 150ml of hot distilled water and allowed to cool at room temperature. Repeat recrystallization process to achieve a pure product. Melting point of paracetamol is ~170^0C.

Precautions:

4-aminophenol and ethanoic anhydride are both irritant's- avoid contact with hands or breathing in vapors. If contact with skin or eyes wash with water: Paracetamol is dangerous in large quantities. Wear safety glasses and lab coat.

Calculations:

Result: The yield of prepared Paracetamol is_____gm.

Melting Point of Paracetamol is _____

VIVA QUESTIONS:

1. What are the uses of paracetamol?

   It acts as a painkiller (analgesic) and relieves from fever.

2. What are the side effects of paracetamol if taken in excess?

   It causes increased sweating, Diarrohea, nausea or vomiting etc:-

3. What is meant by recrystallisation?

   It is a technique used to purify chemicals (Either the desired compound or impurities can be removed from solution leaving the other behind).

4. What are the advantages of recrystallisation?

   Helps in purification of a compound.

5. What is the biproduct formed in paracetamol synthesis?

   Acetic acid

6. Why acetic anhydride readily attack the amino group of P-aminophenol but not hydroxyl group?

   Amines are more basic and nucleophilic than hydroxyls.

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