Engineering Chemistry Laboratory Manual

Department of Chemistry
GITAM Institute of Technology

GITAM

(Deemed to be University)

Accredited by NAAC with 'A^{+}' Grade

Visakhapatnam – Hyderabad – Bengaluru

INDEX

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1. GENERAL INSTRUCTIONS

1. Study theory behind the experiment before attending the Laboratory.

2. Keep the work bench and sink (wash basin) neat and clean. Do not allow used filter papers, broken pieces of glass, used match sticks, etc., to lie on the work bench – throw them into the available dust bin nearby.

3. Keep the apparatus clean and arrange them properly.

4. Handle the chemicals and reagent bottles carefully.

5. Take the prescribed quantities of chemicals and reagents only.

6. Do not pour any excess reagent, taken by chance, back into the reagent bottle, as it is likely to contaminate the entire solution in the reagent bottle.

7. Close the reagent bottles with their lids and keep them in their proper places, after use.

8. Water is a precious commodity; do not waste it; close the water tap immediately after use.

9. It is said, 'Prevention is better than cure' - take care to prevent fire accidents in the Lab.

10. If any piece of apparatus is broken, promptly bring it to the notice of either Staff Members or Lab Assistant.

11. Make it a habit to record all observations in your Observation Note Book, as and when you carry out an experiment; writing observations on loose bits of paper is a bad habit.

12. Do not forget to bring your Laboratory Record while attending the lab.

13. Always wear shoes and laboratory apron while you are in the lab.

14. Wash chemical spills on your body, if any, immediately with plenty of tap water.

15. Before leaving the Laboratory, wash the apparatus clean, keep them in proper place and make your work bench tidy.

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2. INTRODUCTION

The objective of a chemical analysis is to detect and identify different constituents present and finally determine their relative amounts in a substance. The process of detection and identification is commonly known as **Qualitative Chemical Analysis** (*Qualitative Analysis*) while the determination of the amounts of the constituents as **Quantitative Chemical Analysis** (*Quantitative Analysis*). Depending upon the technique used for the determination of the amount, the quantitative chemical analysis can be broadly divided into **Gravimetric Analysis** and **Titrimetric** (***Volumetric***) **Analysis**; the name **Titrimetric Analysis** is preferred over *Volumetric Analysis* as the later, now a day, means determination of volume of a gas evolved in a chemical reaction.

Gravimetric analysis is the process of isolating and weighing an element or a definite compound of the element in a pure form. The element or compound is precipitated from the solution of a weighed portion of the substance being examined. On the other hand, titrimetric analysis essentially consists in determining the volume of a solution of accurately known concentration which is required to react quantitatively with a known volume of the solution of the substance being determined. A solution of accurately known **strength** (***concentration***) is called the **Standard solution**; it contains a definite number of gram equivalents or gram moles per liter of the solvent. The weight of the constituent aimed to be determined is then calculated from the volume of the standard solution consumed and known laws of chemical equivalence. Generally, a standard solution is prepared by dissolving a **primary standard substance** in water (solvent). In order to serve as a primary standard substance, it has to satisfy the following requirements:

a) It must be easy to obtain, to purify, to dry (preferably at 110 - 120^0 C), and to preserve in a pure state.

b) The substance should be unaltered in air during weighing; it should not be hygroscopic, efflorescent, oxidized by air or affected otherwise.

c) It must be a high purity substance; impurities should not exceed 0.01 – 0.02 per cent.

d) It should have reasonably high equivalent weight so that the weighing errors are negligible.

e) It should be readily soluble under conditions it is employed.

f) The reaction of the standard solution with the analyte solution should be stoichiometric and practically instantaneous.

In practice, an ideal primary standard substance which satisfies all the above criteria is difficult to obtain and a compromise between the above ideal requirements is usually necessary. Some of the commonly employed primary standard substances are: Na_2CO_3, KCl, $K_2Cr_2O_7$, $Na_2C_2O_4$, $KBrO_3$, KIO_3, $(NH_4)_2$ $Ce(NO_3)_6$, and As_2O_3. Hydrated salts, as a rule, do not make good standards, because of the difficulty of efficient drying. However, salts which do not effloresce, such as oxalic acid $H_2C_2O_4$ $2H_2O$ and copper sulphate $CuSO_4$ $5H_2O$ are found

to be satisfactory secondary standards. The solutions prepared from less stable compounds need to be standardized using a primary standard solution before use.

The **Standard Solution** (***Titrant***) is usually added from a burette. The process of adding the standard solution to the ***analyte*** (***titrand)*** solution taken in the conical flask (titration/reaction vessel) until the reaction is just complete is termed as **Titration.** The point at which the reaction is just complete is called **Equivalence Point** or **Theoretical** (or **Stoichiometric**) **End Point.** The completion of the reaction should be detectable by some change, unmistakable to the eye, produced by the standard solution itself (e.g., $KMnO_4$) or more usually by the addition of an auxiliary reagent, known as **Indicator**. After the reaction is practically complete, the indicator would give a clear visual change (colour change or formation of turbidity) in the solution being titrated (***titrand/analyte***). The point at which this occurs is called the **End Point** of the titration.

For the purpose of discussion, the titrimetric analysis can be divided into four main types:

1. Neutralisation reactions or **Acidimetry** and **Alkalimetry**. These include the titration of free bases or those formed by hydrolysis of salts of weak acids, with a standard acid (**Acidimetry**) as well as the titration of free acids, or those formed by the hydrolysis of salts of weak bases with a standard base (**Alkalimetry**). In these reactions, hydrogen and hydroxide ions combine to form water.

$$H^+ + OH^- \longrightarrow H_2O$$

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

2. Oxidation – reduction or **Redox reactions**. These are the reactions involving change in oxidation number or transfer of electrons among the reacting substances. In these reactions, the substance which loses electrons is called a **Reducing agent** or **Reductant** while that which gains electrons an **Oxidising agent** or **Oxidant**; the overall reaction between the reductant and the oxidant is called **Redox reaction**.

$$\underset{\text{Oxidant}}{2\,Fe^{III}Cl_3} + \underset{\text{Reductant}}{Sn^{II}Cl_2} \longrightarrow 2Fe^{II}Cl_2 + Sn^{IV}Cl_4$$

3. Complex formation reactions. These depend upon the combination of ions or molecules, other than hydrogen and hydroxide ions, to form a soluble, slightly dissociated ion or compound.

$$CuSO_4 + 4KCN \longrightarrow K_2[Cu(CN)_4]\ (\text{Complex}) + K_2SO_4$$

$$Ag^+ + 2CN^- \longrightarrow [Ag(CN)_2]^-\ (\text{Complex})$$

4. Precipitation reactions. These reactions involve precipitation of sparingly soluble compounds from solutions of the two reactants.

$$\underset{\text{Solution}}{AgNO_3} + \underset{\text{Solution}}{NaCl} \longrightarrow \underset{\text{Precipitate}}{AgCl\downarrow} + \underset{\text{Solution}}{NaNO_3}$$

Usually, the strength of a solution is expressed either in **Molarity** or in **Normality**. A **molar solution** (**1M**) is one which contains one **gram molecular weight** (***gram mole***) of the reagent per liter of the solution. Similarly, a **normal solution** (**1N**) of a substance contains one gram equivalent weight of it per liter of the solution. Even though the gram molecular weight of a reagent does not vary, the gram equivalent weight varies with the type of the reaction; the same compound possesses different gram equivalent weights in different contexts.

In neutralization reactions, the gram equivalent weight of an acid is obtained by dividing the gram molecular weight of it by the number of replaceable hydrogen ions that it contains. For example, the gram equivalent weight of HCl is its gram molecular weight divided by 1 (36.45/1), while that for H_2SO_4 is its gram molecular weight divided by **2** (98/2 = 49). Similarly, the gram equivalent weight of a base is obtained by dividing its molecular weight by the number of replaceable hydroxide ions that it contains. For example, the gram equivalent of $Ba(OH)_2$ is its gram molecular weight divided by **2**, as it contains two replaceable hydroxide ions.

With reference to redox reactions, the gram equivalent weight of an oxidant is obtained by dividing its gram molecular weight by the number of electrons that it gains while that of a reductant by dividing its gram molecular weight by the number of electrons it loses in the reaction. For example, thallous chloride (TlCl) is a reductant and thallium (I) loses two electrons in the process of oxidation and hence its gram equivalent weight is obtained by dividing its gram molecular weight by 2 (239.83/2 = 119.92). The case of $KMnO_4$ is very interesting: as an oxidant, it may gain one, three, four or five electrons depending upon the condition of the reaction medium and the reductant; accordingly its gram equivalent weight varies as 158/1, 158/3, 158/4 and 158/5 respectively.

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3. DESCRIPTION OF APPARATUS USED IN TITRIMETRY (VOLUMETRY)

While carrying out experiments in titrimetric analysis in the Chemistry Lab, you are going to use the following glass apparatus.

Burette: The commonly used laboratory burette (Fig. 1) is a long glass tube of uniform bore throughout its length. It has a holding capacity of 50 mL and is graduated up to $1/10^{th}$ of a milliliter, from top to bottom. At its lower end, a stopcock with nozzle is fixed to facilitate control of flow of a solution at the desired rate. You have to note the following points whenever you use a burette.

a) The burette should always be kept absolutely clean. For this purpose, it should be cleaned with chromic acid, thoroughly washed with tap water and then rinsed with distilled water.

b) Before starting the titration, it should be rinsed with the standard solution, filled with the same solution up to the zero mark, taking care to avoid air gaps in the nozzle and air bubbles in the stem and then fixed to burette stand, vertically (Fig. 2).

c) While noting down the burette readings, the lower meniscus of the solution in the burette has to be kept at the eye level to avoid parallax error; with dark coloured solutions like potassium permanganate, the lower meniscus may not be clearly visible and in such instances the upper meniscus is the choice.

Volumetric or **transfer** or **single mark Pipette**: It is a long narrow tube having cylindrical bulb in the middle, tapering into a fine nozzle at its lower end and an etched circular mark a little above the bulb on the upper truncated end. The volume of liquid delivered on starting from the etched mark to the tip of the nozzle is printed on the bulb of the pipette, in milliliters. As it is useful in transferring the same volume of a liquid always, it is called single mark pipette
(Fig.3). *Caution*: Do not blow off the little amount of liquid that remains at the tip of the nozzle, after transfer.

Volumetric Flask: A volumetric flask is a flat-bottomed, pear shaped vessel with a long narrow neck fitted with leak proof lid at the top (Fig. 5B). A thin line etched around the neck indicates the volume that it holds at a certain definite temperature (both the capacity and temperature are clearly marked on the flask). It is used in preparation of standard solutions and dilution of a sample to a definite volume. Flasks with capacities ranging from 5mL to 2 Liters are in use.

Graduated pipette: It is similar to a burette with a nozzle, but lacks a stopcock (Fig. 4). The regulation of flow of a liquid can be manipulated with forefinger kept at its top end. Just like a burette, it is useful in transferring variable volumes. There are pipettes with different total capacities starting from 1 mL and above.

Measuring Jar or Measuring Cylinder: This is a cylindrical jar provided with a sturdy base. It is graduated from bottom to top (Fig. 6). Usually, the cross-sectional area of the cylinder is relatively large and hence is useful for transfer of approximate volumes of reagents only. Jars with holding capacity ranging from 10mL to 2 Liters are available.

Beaker: Beaker is a flat bottomed wide cylinder with or without a spout at the top (rim) (Fig. 7). Beakers with capacities ranging from 5mL to 2 Liter are in use. However, 250mL and 400 mL beakers are more common in student laboratories. They are useful in preparation and handling of reagents, titrimetric and gravimetric analysis.

Conical flask or Erlenmeyer's Flask: It is a cone shaped flask mostly used as a reaction vessel in carrying out a titration (Fig. 5A). Even though flasks with capacities ranging from 10 mL to 1 Liter are available, 250mL and 500mL flasks are more commonly used.

Wash Bottle: A wash bottle is a flat bottomed flask designed to deliver a fine jet of distilled water or other liquid. This is used as a small size reservoir for storing and transfer of distilled water. It finds wide application in cleaning laboratory ware also. Both glass and squeeze type polythene bottles (Fig. 8) are known but, the latter type finds wider application. They are available from 100mL to 1 Liter capacities.

Glazed Tile: It is a ceramic plate glazed white on one side. It helps to perceive the colour change at the end point of a titration by providing a glossy white back ground.

Pictures of Some Apparatus and Equipment Used in the Lab

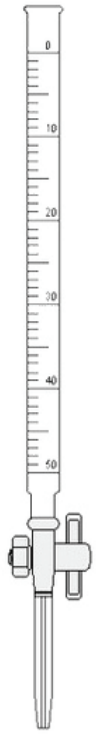

Fig. 1: Burette

Fig. 2: Burette and Clamp fixed to Stand

Fig. 3: Volumetric Pipettes

Fig. 4: Graduated Pipette

A B C

Fig. 5: Different types of Flasks

A - Conical Flask; B - Volumetric Flask; C – Round bottomed flask.

Fig. 6: Measuring Cylinders

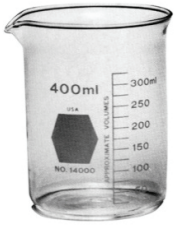

Fig. 7: Beaker

Fig. 8: Polythene Wash Bottles

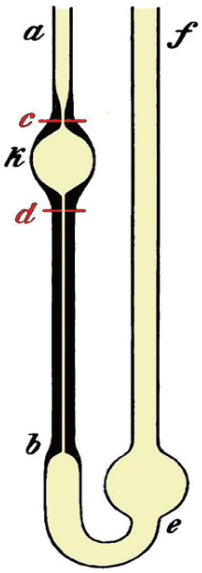

Fig. 9: Ostwald's Viscometer

Fig. 10: Stalagmometer

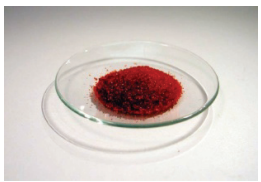

Fig. 11: Watch Glass

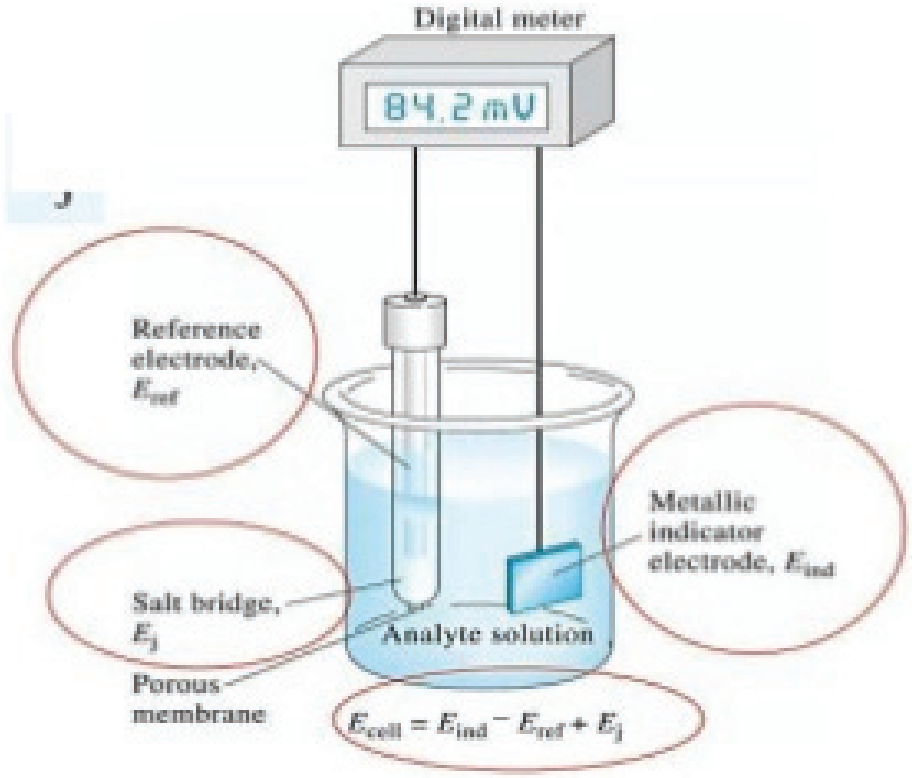

Fig. 12: Digital Potentiometer connected to Indicator and Reference Electrodes placed in Analyte solution

Fig. 13: Titration vessel & double junction electrode as connected to Digital pH meter

4. DETERMINATION OF SODIUM CARBONATE IN SODA ASH

AIM: To determine the amount of Na_2CO_3 present in the given 100mL sample solution using standard HCl solution.

APPARATUS: 50mL burette, burette stand and clamp, 10mL pipette, 250mL conical flask, 250 mL beaker, glazed tile, measuring jar, etc.

REAGENTS: Standard Na_2CO_3 solution, HCl solution, soda ash sample solution and Methyl orange indicator solution.

THEORY: Anhydrous Na_2CO_3, known as soda ash, is a salt formed from H_2CO_3, a very weak acid and NaOH, a strong base. Determination of Na_2CO_3 with a strong acid is a neutralization type of chemical reaction. In this reaction, two moles of HCl are consumed by one mole of Na_2CO_3 ; in fact, the base liberated by the hydrolysis of Na_2CO_3 is neutralised with HCl.

$$2HCl + Na_2CO_3 \longrightarrow 2NaCl + H_2O + CO_2 \uparrow$$

Methyl orange is used as internal indicator in this titration and its colour transition from yellow to orange red takes place in the pH-range of 3.1 to 4.4; the indicator exhibits yellow colour having pH greater than 4.4 and pink colour having pH less than 3.1. As the pH-transition of the said titration at the equivalence point also comes in this range, methyl orange suites as an internal indicator in the present context.

PART-A

STANDARDIZATION OF HCl SOLUTION USING STANDARD SOLUTION OF Na_2CO_3

All the given glass apparatus are washed thoroughly with tap water, rinsed with distilled water and the washings and rinsings are thrown out into the sink. The burette is first rinsed with the given HCl solution and then filled with the same solution up to the zero mark without any air bubbles. The burette is then clamped to burette stand. The pipette is rinsed with the given standard Na_2CO_3 solution and 10 mL of the Na_2CO_3 solution is pipetted out into a clean 250 mL conical flask. 30 mL of distilled water is added to the solution in the conical flask using a 50 mL measuring jar and two drops of methyl orange indicator solution is added to the conical flask. The solution attains yellow colour at this stage. The contents of the conical flask are titrated against HCl solution taken in the burette. The titration is continued until the colour of the solution in the conical flask changes from yellow to orange red, which marks the end point of the titration. The burette readings are noted down to the nearest 0.05 mL in Table-A. The titrations are repeated with fresh 10 mL portions of standard sodium carbonate solution, following the above procedure, until two successive titers are concordant. The data are entered in Table-A and the concentration of HCl solution is calculated as shown under the Table – A.

Table-A: Standardisation of HCl solution using standard Na_2CO_3 solution

S.No.	Volume of standard Na_2CO_3 solution pipetted, mL (V_1)	Burette readings, mL		Volume of HCl rundown from burette, mL (V_2)
		Initial	Final	
1	10.00			
2	10.00			
3	10.00			

CALCULATIONS: The concentration of HCl solution is calculated making use of the formula,

$N_1 V_1 = N_2 V_2$ where

V_1= Volume of Na_2CO_3 solution pipetted = 10.00 mL
V_2 = Volume of HCl solution rundown from burette= mL
N_1 = Normality of standard Na_2CO_3 solution =
N_2 = Normality of HCl solution = (given)
As V_1 , V_2 and N_1 are known, Normality of HCl solution is calculated as $N_2 = \frac{N_1 V_1}{V_2}$

Therefore, the concentration of HCl solution, N_2 = N

PART-B

DETERMINATION OF Na_2CO_3 IN THE GIVEN SODA ASH SAMPLE SOLUTION USING STANDARD HCl SOLUTION

The soda ash sample solution, given in 100 mL volumetric flask, is made up to the mark with distilled water. The solution is homogenized thoroughly by shaking, after closing the volumetric flask with its lid. The burette is again filled with the standardized HCl solution up to the zero mark and clamped to the burette stand. The pipette is washed with distilled water and then rinsed with the given soda ash sample solution. Now 10 mL of the sample solution is pipetted out into a clean 250 mL conical flask and 30 mL of distilled water is added using a measuring jar, followed by 2 drops of methyl orange indicator. The solution is yellow in colour at this stage. The contents of the conical flask are titrated against HCl solution taken in the burette. The titration is continued until the colour of the solution in the conical flask changes from yellow to orange red, which marks the end point. The readings of the burette are noted down to the nearest 0.05 mL in table-B. The titrations are repeated with fresh 10 mL portions till two successive titers are concordant. The results are entered in Table–B and the concentration and amount of Na_2CO_3 in the given sample of soda ash solution is calculated as shown under the Table – B.

Table-B: Determination of sodium carbonate in the given soda ash sample

S.No.	Volume of soda ash sample solution pipetted, mL (V_3)	Burette readings		Volume of HCl rundown from burette, mL (V_2)
		Initial	Final	
1 2 3	10.00 10.00 10.00			

CALCULATIONS: The strength of Na_2CO_3 in the sample is calculated by making use of the formula,

$N_2\ V_2 = N_3\ V_3$ where,

V_2 =Volume of HCl consumed for 10.00 mL of soda ash sample solution = mL

V_3 =Volume of soda ash sample solution pipetted = 10.00 mL

N_2 =Normality of HCl =

N_3 = Normality of Na_2CO_3 in the soda ash sample solution, which can be calculated as V_2 ! , N_2 and V_3 are known.

$$N_3 = \frac{N_2\ V_2}{V_3} =$$

Therefore, the strength of Na_2CO_3 in the sample, N_3 = N

Amount of Na_2CO_3 in the given sample (100 mL) = $\frac{N_3 \text{ x Eq.Wt. of } Na_2CO_3 \text{ x } 100}{1000} = \frac{N_3 \text{ x } 53}{10}$

$= N_3 \text{ x } 5.3$

Table: C Percentage Error Table

Roll/Regd. Number	Flask Number	Amount of Na_2CO_3 , g		% Error
		Given	Reported	

REPORT: Amount of Na_2CO_3 present in the given 100ml soda ash sample:_____gms.

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5. DETERMINATION OF IRON(II) USING POTASSIUM PERMANGANATE

AIM: To determine the amount of iron(II) present in the given sample solution using standard potassium permanganate solution.

APPARATUS: 50 mL burette, burette stand & clamp, 250 mL conical flask, glazed tile, 10 mL pipette, 100 mL volumetric flask, 50 mL measuring jar, beaker and wash bottle.

CHEMICALS REQUIRED: Standard oxalic acid solution, potassium permanganate solution, iron(II) test sample solution and 1:1 H_2SO_4 solution.

INDICATOR: $KMnO_4$ acts as a self indicator.

END POINTS: Colourless to permanent pale pink in Part-A and colourless/pale yellow to permanent pale pink in Part-B

THEORY : Reaction between $KMnO_4$ and Fe(II) is an example of a redox reaction where Mn(VII) in $KMnO_4$ oxidises Fe(II) to Fe(III) and itself gets reduced from Mn(VII) to Mn(II)state. The titration is carried out in H_2SO_4 medium.

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

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

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

According to the above equation 2 moles of $KMnO_4$ react with 10 moles of $FeSO_4$. The self colour of $KMnO_4$ solution is used to locate the end point. As potassium permanganate is not a primary standard, its solution has to be standardised using standard oxalic acid solution.

PART – A

STANDARDISATION OF $KMnO_4$ SOLUTION BY USING STANDARD SOLUTION OF MOHR'S SALT

All the glass apparatus are washed with tap water and rinsed with distilled water. The burette is rinsed with the given $KMnO_4$ solution and filled with the same solution up to the zero mark, avoiding air bubbles in it. The pipette is rinsed with standard Mohr's salt solution and 10 mL of the same solution is pipetted out into a clean 250 mL conical flask. To this 40 mL of distilled water and 5 mL of 1:1 dilute sulphuric acid solution are added. The contents of the conical flask are titrated against potassium permanganate solution taken in the burette until the colour of the solution in the conical flask changes from colourless/pale yellow to permanent pale pink. The above process is repeated, each time pipetting 10 mL

portions of standard Mohr's salt solution, until two successive concurrent titers are obtained. The results are entered in Table-A and the concentration of permanganate is calculated as shown under table-A.

Table-A :
Standardisation of $KMnO_4$ solution using standard Mohr's Salt solution

S.No	Volume of Mohr's Salt solution pipetted out , mL (V_1)	Burette Readings		Volume of $KMnO_4$ solution rundown from burette, mL (V_2)
		Initial	Final	
1.	10.00			
2.	10.00			
3.	10.00			

CALCULATIONS: The concentration of permanganate solution is calculated using the equation,

$$N_1V_1 = N_2V_2 \quad \text{where,}$$

N_1 = Normality of standard Mohr's Salt solution = (given)
V_1 = Volume of Mohr's Salt solution pipetted out = 10.00 mL
V_2 = Volume of $KMnO_4$ solution rundown from burette = mL and
N_2 = Normality of $KMnO_4$ solution
As N_1 , V_1 and V_2 are known the normality of permanganate, N_2 can be calculated as

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

Normality of $KMnO_4$ solution N_2 =

PART - B

DETERMINATION OF IRON(II) IN THE SAMPLE USING STANDARD $KMnO_4$ SOLUTION

The given iron(II) sample solution in 100 mL volumetric flask is made up to the mark with distilled water and it is homogenized properly. The burette is filled with $KMnO_4$ solution and clamped to the burette stand. The pipette is first washed with distilled water and then rinsed with the given iron(II) sample solution and 10 mL of the same solution is pipetted out into a clean 250 mL conical flask. To this 40 mL of distilled water and 5 mL of 1:1 H_2SO_4 solutions are added. The contents of the conical flask are then directly titrated against $KMnO_4$ solution until the colour of the solution in the conical flask changes from colourless to permanent pale pink. The burette readings are noted down to the nearest 0.05 mL in Table-B. The titrations are repeated with fresh 10 mL portions of iron(II) sample solution until two successive concurrent titers are obtained. The results are entered in Table–B and the concentration and amount of iron(II) in the sample solution are calculated as shown under the Table – B.

Table-B :

Determination of iron(II) using standard potassium permanganate solution

S.No	Volume of Fe(II) sample solution pipetted out, mL (V_3)	Burette Readings		Volume of $KMnO_4$ solution rundown from burette, mL (V_2)
		Initial	Final	
1.	10.00			
2.	10.00			
3.	10.00			

CALCULATIONS: The concentration of iron(II) in the sample solution is calculated from the equation $N_2V_2^1 = N_3V_3$ where

N_2 = Normality of $KMnO_4$ solution =

V_2 = Volume of $KMnO_4$ rundown from burette = mL

V_3 = Volume of iron(II) sample solution pipetted out = 10.00 mL and

N_3 = Normality of iron(II) in sample solution.

Therefore, $N_3 = \frac{N_2 V_2}{V_3}$ = N

The amount of iron(II) present in the given sample of 100 mL $= \frac{N_3 \times \text{Eq. Wt. of Fe(II)} \times 100}{1000}$

$= \frac{N_3 \times 55.85}{10}$ = g

Table: C Percentage Error Table

Roll/Regd. Number	Flask Number	Amount of iron(II), g		% Error
		Given	Reported	

REPORT: Amount of iron(II) present in the given 100 mL sample solution _____ g

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6. ESTIMATION OF CALCIUM IN PORTLAND CEMENT

AIM: To estimate the amount of calcium (as oxide) present in the given 100 mL Portland cement sample solution using standard solution of $KMnO_4$.

APPARATUS: 50 mL burette, burette stand & clamp, 10 mL pipette, 250 mL conical flask, beaker, 100 mL volumetric flask, measuring cylinder, glazed tile, wash bottle, quantitative filter papers, etc.

CHEMICALS REQUIRED: Portland cement sample, HCl, Ammonium hydroxide, 6% Ammonium oxalate, standard Oxalic acid, $KMnO_4$ and 1:1 H_2SO_4 solutions.

INDICATOR: $KMnO_4$ acts as a self indicator.

END POINT: Colourless to permanent pale pink.

THEORY: Portland cement, on boiling with dilute hydrochloric acid decomposes yielding soluble chlorides of calcium, iron and aluminium and insoluble silica.

$$\underset{\text{Solid}}{\text{Portland cement}} + \underset{\text{Solution}}{\text{Dil. HCl}} \xrightarrow{\text{Boiling}} \underbrace{CaCl_2 + FeCl_3 + AlCl_3 + H_2O}_{\text{Solution}} + \underset{\text{Ppt.}}{SiO_2}\downarrow$$

The solution containing the metal ions is separated from the insoluble silica (SiO_2) by filtration through a quantitative filter paper. Calcium in the filtrate is then selectively and quantitatively precipitated as CaC_2O_4 at near neutral pH using ammonium oxalate, $(NH_4)_2C_2O_4$ as precipitating agent. The CaC_2O_4 precipitate is then filtered through a quantitative filter paper, freed from other metal ions by repeated washing with distilled water and dissolved in dilute sulphuric acid. This dissolution results in the liberation of oxalic acid from CaC_2O_4.

$$\underset{\textit{Precipitate}}{CaC_2O_4}\downarrow + \underset{\textit{Solution}}{H_2SO_4} \longrightarrow \underset{\text{------}\textit{Solution}\text{---------}}{CaSO_4 + H_2C_2O_4}$$

Further, it may be seen from the above stoichiometric reaction that the amount of oxalic acid so liberated is equivalent to the amount of calcium initially present. The solution is quantitatively transferred into a volumetric flask and made up to the mark with distilled water and homogenized. An aliquot of this solution is used for the estimation of calcium in Portland cement. The reaction between oxalic acid and potassium permanganate is an example of a redox reaction and Mn(VII) in $KMnO_4$ oxidizes the $H_2C_2O_4$ to CO_2 and itself gets reduced from Mn(VII) to Mn(II) state in H_2SO_4 medium.

$$2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2\uparrow$$

According to the above reaction 2 moles of $KMnO_4$ react with 5 moles of $H_2C_2O_4$. The titration is initiated at a temperature of about 70 – 80°C. Although the reaction mixture is heated to 70°C, the rate of reaction is initially slow but gets efficiently catalysed by the *in-*

situ generated Mn^{+2} ions in the solution; Mn^{+2} acts as an auto catalyst. The self colour of $KMnO_4$ is used to locate the end point, as all other reactants and products are colourless; $KMnO_4$ acts as a self indicator. The given $KMnO_4$ solution is first standardized using standard oxalic acid solution as the former is not a primary standard.

PART – A

STANDARDISATION OF $KMnO_4$ SOLUTION USING STANDARD OXALIC ACID SOLUTION

All the given glass apparatus are washed with tap water and then rinsed with distilled water. The burette is rinsed with the given $KMnO_4$ solution and is filled with the same solution. Pipette is rinsed with the given standard oxalic acid solution and 10 mL of standard oxalic acid solution is pipetted out into a clean 250 mL conical flask followed by the addition of 45 mL distilled water and 5mL of 1:1 diluted H_2SO_4. The colourless contents of the conical flask are heated to 70 – 80^oC. Now the hot contents of the flask are titrated with $KMnO_4$ solution, taking all the precautions until a permanent pale pink colour appears which indicates the end point of the titration. The burette readings are noted in Table–A and titrations are continued with 10 mL portions of oxalic acid until 2 successive titers are concurrent. The burette readings are tabulated in Table–A and the concentration of $KMnO_4$ solution calculated as shown under Table–A.

Table-A: Standardisation of $KMnO_4$ solution with standard oxalic acid solution

S.No	Volume of Oxalic acid solution pipetted out, mL (V_1)	Burette Readings		Volume of $KMnO_4$ solution rundown, mL (V_2)
		Initial	Final	

CALCULATIONS: The strength of $KMnO_4$ solution is calculated using the equation,

$N_1V_1 = N_2V_2$ where

N_1 = Normality of standard oxalic acid solution = (given)

V_1 = Volume of oxalic acid pipetted out = 10.00 mL

V_2 = Volume of $KMnO_4$ solution rundown from burette = mL and

N_2 = Normality of $KMnO_4$ and N_2 calculated as $N_2 = \frac{N_1V_1}{V_2}$

Therefore, normality of $KMnO_4$ solution N_2 =

PART- B

ESTIMATION OF CALCIUM IN PORTLAND CEMENT USING STANDARD $KMnO_4$SOLUTION

PROCEDURE: The burette is filled with $KMnO_4$ solution, the initial reading set to zero without any air bubbles and clamped to the stand. Pipette is rinsed with the sample solution prepared from calcium oxalate and 10 mL of the same solution is pipetted out into a clean 250 mL conical flask. To this 45 mL of distilled water and 5mL of 1:1 diluted H_2SO_4 are added and the solution is heated to about 70 – 80^oC. The hot contents of the conical flask are then titrated against $KMnO_4$ solution taken in the burette, until the colour of the solution

changes from colourless to permanent pale pink. The burette readings are noted down to the nearest 0.05 mL in Table–B. The above process is repeated with fresh 10 mL portions obtained from the cement sample until two successive titers are concurrent. The results are also entered in Table-B. The final calculations are carried out as shown under Table – B and the amount of calcium reported in terms of CaO.

Table-B: Estimation of calcium in Portland cement with standard solution of $KMnO_4$

S.No	Volume of Sample solution pipetted out, mL (V_3)	Burette Readings		Volume of $KMnO_4$ solution rundown from burette, mL (V_2)
		Initial	Final	
1. 2. 3.	10.00 10.00 10.00			

CALCULATIONS: The concentration of oxalic acid in the sample solution obtained from calcium fraction is calculated using the equation,

$N_2V_2^{I} = N_3V_3$ where,

N_2 = Normality $KMnO_4$ =

V_2 = Volume of $KMnO_4$ rundown = mL

V_3 = Volume of Sample solution Pipetted = mL and

N_3 = Normality of oxalic acid in Sample solution

Therefore, $N_3 = \frac{N_2V_2}{V_3}$

From the balanced chemical equations shown in the earlier part, it may be understood that Normality of Calcium in the sample solution = Normality of oxalic acid in the sample solution.

Equivalent weight of CaO = $\frac{\text{Mol.Wt.of CaO}}{2}$

Amount of Calcium present in the given 100ml Portland cement sample as expressed in terms of CaO = $\frac{\text{Normality of } H_2C_2O_4 \text{ in sample x Eq.Wt.of CaO x 100}}{1000} = N_3 \times 2.804$

Table: C Percentage Error Table

Roll/Regd. No.	Flask No.	Amount of Ca present as CaO, g		% Error
		Given	Reported	

REPORT: The amount of calcium, as CaO, present in the given sample of cement = g

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7. DETERMINATION OF VOLUME STRENGTH OF HYDROGEN PEROXIDE

AIM: To determine the amount and volume strength of H_2O_2 present in the given sample solution using standard $KMnO_4$ solution.

APPARATUS : 50 mL burette, burette stand and clamp, 250 mL conical flask, glazed tile, 10 mL pipette, 100 mL volumetric flask, wash bottle, beaker, measuring jar, etc.

CHEMICALS REQUIRED: Standard oxalic acid, $KMnO_4$, H_2O_2 and 5N sulphuric acid solutions.

INDICATOR: $KMnO_4$ serves as a self indicator

END POINT: Colourless to permanent pale pink

THEORY: The reaction between H_2O_2 and $KMnO_4$ is also an example of a redox reaction wherein $KMnO_4$ oxidizes hydrogen peroxide to water and oxygen; Mn(VII) in permanganate gets reduced to Mn(II) state, in the process.

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

$$5H_2O_2 + 5(O) \longrightarrow 5H_2O + 5O_2$$

$$2KMnO_4 + 3H_2SO_4 + 5H_2O_2 \longrightarrow K_2SO_4 + 2MnS_4 + 5O_2 + 8H_2O$$

According to the above equation 5 moles of H_2O_2 react with 2 moles of $KMnO_4$. The self colour of $KMnO_4$ solution is used to locate the end point because all the other reactants and products are almost colourless. Moreover, initially the rate of the reaction is very slow and it gets catalyzed by the *in-situ* generated Mn^{+2} which acts as a catalyst and hence Mn^{+2} plays the role of an auto- catalyst in this reaction also.

DETERMINATION OF VOLUME STRENGTH OF HYDROGEN PEROXIDE USING STANDARD $KMnO_4$ SOLUTION

Procedure:

The given H_2O_2 sample solution is made up to the mark with distilled water in the 100 mL volumetric flask and homogenized properly. Burette is filled with $KMnO_4$ solution. Pipette is washed with tap water, rinsed with distilled water and then with H_2O_2 sample solution. Now 10 mL of the sample solution is pipetted out into a clean 250 mL conical flask. To this 40 mL of distilled water followed by 5 mL of 1:1 diluted H_2SO_4 are added. The contents of the conical flask are titrated, at room temperature, against $KMnO_4$ solution until the colour of the solution changes from colourless to permanent pale pink. The burette readings are noted down to the nearest 0.05 mL in the Table–B. The titrations are repeated with fresh 10 mL aliquots of H_2O_2 until two successive titers are concurrent and the readings are recorded in Table-B. The amount of H_2O_2 present in the given sample is calculated as shown under Table–B.

Table-A: Determination of volume strength of H_2O_2 using standard $KMnO_4$ solution

S.No	Volume of H_2O_2 solution pipetted, mL (V_3)	Burette Readings		Volume of $KMnO_4$ solution rundown from burette, mL (V_2)
		Initial	Final	
1. 2. 3.	10.00 10.00 10.00			

CALCULATIONS: The concentration of H_2O_2 in the sample, N_3 , is calculated by substituting the known values of N_2, V_2^{I}, and V_3 in the equation,

$N_3 = N_2V_2^{I} / V_3$ where

N_2 = Normality of $KMnO_4$ solution=
V_2 = Volume of $KMnO_4$ rundown from burette = mL
V_3 = Volume of H_2O_2 solution pipetted out = 10.00 mL and
N_3 = Normality of H_2O_2

Therefore, the calculated concentration of H_2O_2 in the sample solution = N

Amount of H_2O_2 in 1 litre of solution = N_3 x Eq.Wt. of H_2O_2 = N_3 x 17.01 g = **Y**

The amount of H_2O_2 present in the given 100ml of H_2O_2 sample solution,

$$\mathbf{Z} = \frac{\mathbf{Y} \times 100}{1000} =$$

Calculation of volume strength of H_2O_2:

The basic chemical reaction and calculations involved are:

$$2H_2O_2 \longrightarrow 2H_2O + O_2$$
(2 Moles) (2Moles) (1 Mole)

Therefore, 2 moles of H_2O_2 give rise to 1 mole of O_2 and it is also well known that 1 gm mole of oxygen gas at STP occupies 22.4 liters. Therefore,

2 x 34.02 (=68.04) gm of H_2O_2 is equivalent to 22.4 liters of O_2 or

1 gm of H_2O_2 is equivalent to $\frac{22.4 \times 1}{68.04}$ liters of O_2 = 0.3292 Lt = **X** L

Volume strength of H_2O_2 = (Weight of H_2O_2 per L) x **X** = **Y** x **X** = **Y** x 0.3292 = Lt.

Table: B Percentage Error Table

Roll/Regd. No.	Flask No.	Amount of H_2O_2 , g		% Error
		Given	Reported	

REPORT: The amount of H_2O_2 present in given 100ml sample solution, **Z** .

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8. DETERMINATION OF CHROMIUM(VI) USING FERROUS AMMONIUM SULPHATE

AIM: To determine the amount of chromium(VI) present in the given sample solution using ferrous ammonium sulphate solution of known concentration.

APPARATUS : 50 mL burette, stand & clamp, 10 mL pipette, 250 mL conical flask, 50 mL measuring jar, beaker, 100 mL volumetric flask, glazed tile, wash bottle, etc.

CHEMICALS REQUIRED: Ferrous ammonium sulphate, standard potassium dichromate, and 1:1 H_2SO_4 solutions and syrupy phosphoric acid (H_3PO_4).

INDICATOR: Diphenylamine (DPA)

END POINT: Bluish green to bluish violet

THEORY: The reaction between $K_2Cr_2O_7$ and Fe(II) is another example of a redox reaction. In the presence of H_2SO_4, $K_2Cr_2O_7$ oxidises Fe(II) to Fe(III) and Cr(VI) in dichromate gets reduced to Cr(III) state.

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3(O)$$

$$6FeSO_4 + 3H_2SO_4 + 3(O) \longrightarrow 3Fe_2(SO_4)_3 + 3H_2O$$

$$K_2Cr_2O_7 + 6FeSO_4 + 7H_2SO_4 \longrightarrow K_2SO_4 + 3Fe_2(SO_4)_3 + 7H_2O$$

According to the above reaction 1 mole of $K_2Cr_2O_7$ reacts with 6 moles of $FeSO_4$. Diphenylamine (DPA) is used as internal indicator to locate the end point. Premature end points result due to the closeness of redox potential of the indicator system to that of Fe(III)/Fe(II) system. However stiochiometric end point can be achieved by adding syrupy phosphoric acid to the reaction mixture; phosphoric acid removes ferric iron as phosphato complex and thereby lowers the redox potential of Fe(III)/Fe(II) system, providing sufficient difference in the redox potentials of the two systems concerned such that premature end points are avoided. Therefore, in the presence of H_3PO_4 , once all the Fe(II) ions react completely with dichromate, the next drop of dichromate oxidizes the indicator and the oxidized form of the indicator is violet blue in colour. Hence the colour transition at the end point is from bluish green to bluish violet.

As ferrous ammonium sulphate is not a primary standard, its solution has to be standardized using a standard solution like that of potassium dichromate.

PART – A

STANDARDISATION OF IRON(II) SOLUTION USING STANDARD $K_2Cr_2O_7$ SOLUTION

All the glass apparatus are washed with tap water first and then rinsed with distilled water. The burette is rinsed with the given standard potassium dichromate solution and filled with the same solution up to the zero mark without any air bubbles. It is clamped to the burette stand. Pipette is rinsed with iron(II)) solution and 10 mL of the same solution is

pipetted out into a clean 250 mL conical flask. To this 40 mL of distilled water, 5 mL of 1:1 diluted H_2SO_4 and 3 mL of syrupy phosphoric acid are added followed by 2 drops DPA indicator. The colourless solution in the conical flask is titrated against standard $K_2Cr_2O_7$ solution until the colour changes from bluish green to bluish violet. The burette readings are noted down to the nearest 0.05 mL in the Table–A. The titrations with 10 mL parts of iron(II) solution are repeated until two successive concurrent titers are obtained and the readings are entered in the same table. The concentration of iron(II) is calculated as shown under Table-A.

Table-A: Standardisation of iron(II) using standard potassium dichromate solution

S.No	Volume of Fe(II) solution pipetted, mL (V_2)	Burette Readings		Volume of $K_2Cr_2O_7$ solution rundown from burette, mL (V_1)
		Initial	Final	
1. 2. 3.				

CALCULATIONS: The concentration of iron(II) is calculated using the equation,

$N_1V_1 = N_2V_2$ where

N_1 = Normality of $K_2Cr_2O_7$ =

V_1 = Volume of $K_2Cr_2O_7$ rundown from burette= mL

V_2 = Volume of Fe(II)solution pipetted out = mL and

N_2 = Normality of Fe(II)solution, which can be calculated as, $N_2 = \frac{N_1V_1}{V_2}$ =

Therefore, Normality of Fe(II) solution N_2 =

PART – B

DETERMINATION OF CHROMIUM(VI) IN THE SAMPLE SOLUITON USING STANDARD IRON(II) SOLUTION

The given chromium(VI) sample solution in 100 mL volumetric flask is made up to the mark with distilled water and shaken thoroughly for complete homogenization. The burette is rinsed with the given chromium(VI) sample solution and filled with the same solution up to zero mark avoiding air bubbles and clamped to the burette stand. 10 mL of iron(II) solution is pipetted out into a clean 250 mL conical flask. To this 10 mL of distilled water, 5 mL of 1:1 diluted H_2SO_4 and 3 mL of syrupy phosphoric acid are added followed by 2 drops of DPA indicator. The solution in the conical flask is titrated against chromium(VI) sample solution taken in the burette until the colour of the solution changes from bluish green to bluish violet and the burette readings are noted down to the nearest 0.05 mL in Table B. Titrations with fresh 10.0 mL aliquots of iron(II) are repeated following the above procedure until two successive concurrent titers are obtained and the readings are also noted down in Table-B.

Table-B: Determination of chromium(VI) with standard iron(II) solution

S.No	Volume of iron(II) sample solution pipetted out, mL (V_2)	Burette Readings		Volume of Chromium(VI) solution rundown from burette, mL (V_3)
		Initial	Final	
1. 2. 3.				

CALCULATIONS : Finally the amount of chromium(VI) in the given sample solution is Calculated using the formula,

$$N_2V_2 = N_3V_3 \quad \text{where}$$

N_2 = Normality of iron(II) solution=

V_2 = Volume of iron(II) pipetted out= 10.00 mL

V_3 = Volume of chromium(VI) rundown from burette =

N_3 = Normality of chromium(VI) sample solution = $\frac{N_2V_2}{V_3}$

Normality of chromium(VI) in the sample solution N_3 =

Finally the amount of chromium(VI) present in the given sample solution is calculated using the formula

$$\frac{N_3 \times \text{Eq. Wt. of Cr(VI)} \times 100}{1000} = \frac{N_3 \times 17.33}{10} = 1.733 \times N_3$$

Table: C Percentage Error Table

Roll/Regd. No	Flask No.	Amount of Chromium(VI), g		% Error
		Given	Reported	

REPORT: The amount of Cr(VI) in the given sample solution is _____ gm.

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9. DETERMINATION OF COPPER(II) USING SODIUM THIOSULPHATE

AIM : To determine the amount of copper(II) present in the given sample solution using sodium thiosulphate (**hypo**) solution (IODOMETRIC METHOD).

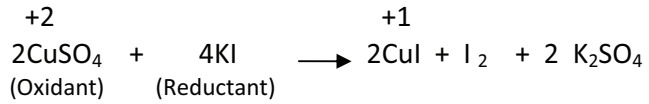
APPARATUS: 250 mL conical flask, 50 mL burette, burette stand and clamp, glazed tile, 10 mL pipette, 100 mL volumetric flask, 50 mL measuring jar, Wash bottle, etc.

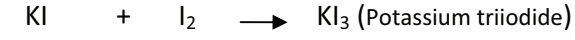
CHEMICALS REQUIRED: Standard copper sulphate solution, sodium thiosulphate (hypo), and 10% potassium iodide solutions.

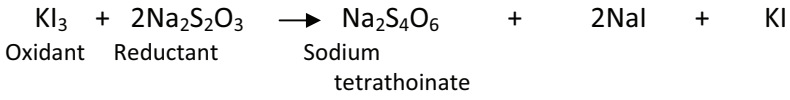
INDICATOR: 1 % Starch solution

END POINT: Blue to pale cream colour

THEORY: Copper(II) cannot be titrated with hypo as there is no direct reaction between Cu(II) and hypo. However Copper(II) directly oxidizes iodide to iodine and the liberated iodine can be titrated with sodium thiosulphate solution quantitatively. Therefore, Cu(II) is indirectly estimated iodometrically by adding potassium iodide solution to Cu(II) solution and titrating the liberated iodine with hypo provided, the Cu(II) solution is free from mineral acids. The basic reactions involved are:







According to the above reaction, 2 equivalents of $CuSO_4$ liberate 2 equivalents of molecular iodine, which in turn oxidize 2 equivalents of $Na_2S_2O_3$. If free mineral acids are present as impurities in the Cu(II) solution, they cause interference in the above reaction; in the presence of mineral acids, iodide gets oxidised to iodine by the dissolved oxygen present in the medium. However the above interference can be overcome by adding a small amount of Na_2CO_3 and dissolving the precipitated copper carbonate in dilute acetic acid; sodium carbonate neutralizes the mineral acid and the slight excess of carbonate causes precipitation of a small amount of $CuCO_3$ which dissolves in dilute CH_3COOH. As sodium thiosulphate (Hypo) is not a primary standard its solution has to be standardized with a primary standard solution like potassium iodate. But as copper(II) in the sample is to be determined, and to avoid methodical errors, a standard solution of copper(II) is prepared from Analytical Grade $CuSO_4.5H_2O$ (a secondary standard substance) to standardize hypo solution.

PART - A

STANDARDISATION OF HYPO USING STANDARD COPPER SULPHATE SOLUTION

All the given glass apparatus are washed with tap water and then rinsed with distilled water. The burette is rinsed with hypo and filled with the same solution up to the zero mark avoiding air bubbles and it is clamped to burette stand. The given standard copper(II) solution is made up to the mark with distilled water and homogenized. The pipette is rinsed with standard copper(II) solution and 10 mL of the same solution is pipetted out into a clean 250 mL conical flask. To this sodium carbonate solution is added drop wise until the solution becomes turbid. And then dilute Acetic acid (1:1) is added drop wise to re-dissolve the precipitate obtained and the solution becomes the clear. To this 10 mL of 10 % KI solution is added. The blue Cu(II) solution becomes dark brown in color due to the liberation of iodine. To this 30 ml of distilled water is added now and allowed to stand for about two minutes to ensure complete liberation of iodine. Now the contents of the conical flask are then titrated against hypo taken in the burette until it becomes straw yellow in colour. Then 1 mL of starch indicator is added. The conical flask contents now attains blue or violet colour. The titration with hypo is continued until the blue colour disappears sharply with one drop addition of hypo. The burette readings are noted down to the nearest 0.05 mL in Table-A. The above procedure is continued with fresh 10 mL portions of copper(II) solution until two successive concurrent titers are obtained and the readings are entered in Table-A.

Table-A: Standardisation of hypo with standard Cu(II) solution.

S.NO	Volume of copper(II) solution pipetted, mL (V_1)	Burette Readings		Volume of hypo Rundown, mL (V_2)
		Initial	Final	

CALCULATIONS: The concentration of hypo can be calculated using the equation,

$V_1N_1 = V_2N_2$ where

V_1 = Volume of standard Cu(II) solution pipetted = 10.00 mL
N_1 = Normality of standard Cu(II) solution =
V_2 = Volume of hypo solution rundown form burette =
N_2 = Normality of hypo solution
Therefore, normality of hypo solution, N_2 is calculated by substituting the known values of N_1, V_1 and V_2 in the equation, $N_2 = V_1 N_1 / V_2$

Concentration of Hypo solution, N_2 = N

PART - B

DETERMINATION OF COPPER (II) IN SAMPLE SOLUTION USING STANDARD HYPO SOLUTION

The given sample of copper (II) solution is made up to the mark of the volumetric flask by adding distilled water and homogenized. Burette is filled with the hypo solution up to the zero mark avoiding air bubbles and clamped to the burette stand. 10 mL of copper(II) sample solution is pipetted down into a clean 250 mL conical flask using a 10 mL pipette, which was initially washed with tap water, distilled water and then rinsed with the copper(II) sample solution, following the usual procedure. To this sodium carbonate solution is added drop wise until the solution becomes turbid. And then dilute Acetic acid (1:1) is added drop wise to re-dissolve the precipitate obtained and the solution becomes the clear. To this 10 ml of 10% KI solution is added, immediately followed by 30 ml of distilled water and the flask is closed the reaction is allowed to complete by giving **2** minutes time. Now the contents of the conical flask is then titrated against hypo solution until it attains straw yellow colour. At this stage 1 mL of starch indicator is added and the titration with hypo is continued until the blue colour gets discharged with just 1 drop of hypo. The burette readings are noted down to the nearest 0.05 mL in Table–B. The above procedure is continued with 10 mL portions of sample solution until two successive concurrent titers are obtained and the readings are also entered in Table-B.

Table-B: Determination of Copper(II) in the given sample solution

S.NO	Volume of copper(II) sample solution pipetted, mL (V_3)	Burette Readings		Volume of hypo rundown, mL (V_2)
		Initial	Final	

CALCULATIONS : The concentration of Cu(II) in the sample is calculated using the formula

$V_2^{\prime} N_2 = V_3 N_3$ where

V_2= Volume of hypo rundown from burette =

N_2 = Normality of hypo =

V_3 = Volume of Cu(II) sample solution pipetted out =10.00 mL

and N_3 = Normality of Cu(II) in sample solution

Therefore, [Cu(II)] in the sample, $N_3 = \frac{V_2^{\prime} N_2}{V_3}$ =

Finally, the amount of Cu(II) present in the given sample is calculated using the formula,

$$= \frac{N_3 \times \text{Eq.Wt. of Cu(II)} \times 100}{1000} = \frac{N_3 \times 63.55}{10} = 6.355 \times N_3 =$$

Table: C Percentage Error Table

Roll/Regd. No.	Flask No.	Amount of Copper(II), g		% Error
		Given	Reported	

REPORT: The amount of Copper(II) present in the sample solution is g.

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10. ESTIMATION OF ACTIVE CHLORINE CONTENT IN BLEACHING POWDER

AIM: To estimate the amount of active chlorine content present in the given bleaching powder sample using standard hypo solution (IODOMETRIC METHOD).

APPARATUS : 250 mL conical flask, 10 mL pipette, 50 mL burette, burette stand with clamp, Glazed tile, 100 mL volumetric flask, measuring jar, wash bottle, etc.

CHEMICALS REQUIRED: Standard copper sulphate solution, 10 % KI solution, hypo solution, glacial acetic acid and bleaching powder sample.

INDICATOR: 1% Starch solution

END POINT: blue to colourless

THEORY: The available chlorine content in a bleaching powder sample can be estimated with hypo by iodometric method based on the redox reaction which involves the conversion of elemental iodine to iodide ions as given by

$$I_2 + 2e^- \longrightarrow 2I^-$$

Bleaching powder contains a mixture of Ca(OCl)Cl and basic calcium chloride $CaCl_2$. $Ca(OH)_2$ H_2O. The active component of bleaching powder is hypochlorite ion which is responsible for the bleaching action of bleaching powder. Bleaching powder hydrolyses in water forming hypochlorous acid, which is a good disinfectant.

$$Ca(OCl)Cl + H_2O \longrightarrow Ca(OH)_2 + Cl_2$$

$$Cl_2 + H_2O \longrightarrow \underset{\text{Hypochlorous acid (Germicide)}}{HOCl} + HCl$$

Hypochlorous acid inactivates certain enzymes in the cells of microorganisms leading to their death. Available chlorine in bleaching powder can be determined by treating a suspension of bleaching powder in water with potassium iodide In presence of acetic acid, which liberates active chlorine, which in turn liberates iodine from iodide quantitatively.

$$Ca(OCl)Cl + 2H^+ \longrightarrow Ca^{+2} + Cl_2 + H_2O$$

$$Cl_2 + 2KI \longrightarrow 2KCl + I_2$$

Hence, available chlorine can be determined iodometrically by adding KI and acetic acid to a suspension of the bleaching powder in distilled water. The liberated chlorine, in the presence of acetic acid, releases iodine from potassium iodide solution quantitatively and the liberated iodine is titrated against hypo solution of known concentration.

$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$$

As the hypo solution is not a primary standard, it is standardized with standard copper(II) solution, as was mentioned earlier.

PART – A

STANDARDISATION OF HYPO SOLUTION USING STANDARD COPPER(II) SOLUTION

The given glass apparatus are washed with tap water and then rinsed with distilled water. Burette is rinsed with hypo solution and filled with the same solution up to the zero mark without any air bubbles and clamped to the burette stand. Pipette is rinsed with standard copper(II) solution and 10 mL of the same solution is pipetted out into a clean 250 mL conical flask. To this 10 mL of 10% KI solution is added immediately followed by 40 mL of distilled water. The flask is closed with its lid and the reaction is allowed to complete by giving 2 minutes time. Now the contents of the conical flask is titrated against hypo solution taken in the burette until the solution turns straw yellow in colour. At this stage about 2 mL of 1% starch indicator is added; the colour of the solution turns blue. The titration against hypo is continued until the blue colour disappears with 1 drop addition of hypo. The burette readings are noted down to the nearest 0.05 mL in Table A and the above process is continued taking 10 mL aliquots of copper(II) solution until 2 successive concurrent titers are obtained and the readings are entered in Table-A. From the known concentration of copper(II) solution the concentration of hypo is calculated as shown under the same table.

Table-A: Standrdisation of hypo solution using standard copper(II) solution

S.No	Volume of copper(II) solution pipetted out, mL (V_1)	Burette Readings		Volume of hypo solution rundown from burette, mL (V_2)
		Initial	Final	
1. 2.				

CALCULATIONS: The concentration of hypo is calculated using the formula,

$$N_1V_1 = N_2V_2$$

where N_1 = Normality of standard Cu(II) solution;

V_1 = Volume of Cu(II) solution pipetted out = 10.00 mL;

V_2 = Volume of hypo solution run down from burette and

N_2 = Normality of hypo solution.

Therefore, the concentration of hypo, $N_2 = N_1V_1 / V_2$ = N

PART – B

ESTIMATION OF AVAILABLE CHLORINE CONTENT IN THE BLEACH USING STANDARD HYPO

The given bleaching powder sample solution in 100 mL volumetric flask is shaken thoroughly for complete homogenization. The burette is filled with the hypo solution up to the zero mark without any air bubbles and clamped to burette stand. The pipette is rinsed with bleaching powder sample solution and 10 mL of the same solution is pipetted out into a clean 250 mL conical flask. To this 10 mL of glacial acetic acid and 10 mL of 10% KI solution

are added followed by 40 mL of distilled water down the inner wall of the conical flask. The contents of the conical flask are mixed well by swirling the conical flask. (Liberation of iodine from potassium iodide by active chlorine is almost instantaneous.) The dark brown solution in the conical flask is titrated against hypo solution taken in the burette until the colour of the solution changes from dark brown to light yellow. At this stage 2 mL of 1% starch indicator is added. The solution turns blue and the titration of the blue coloured solution with hypo is continued until the blue colour of the solution disappears. The readings are entered in Table-B. The above process is continued taking fresh 10 mL aliquots of the bleach sample solution until two successive concurrent titers are obtained and the readings are recorded in Table-B and the available chlorine content is calculated as shown under it.

Table-B: Estimation of available chlorine content in bleach

S.No	Volume of bleach solution pipette, mL (V_3)	Burette Readings		Volume of hypo rundown from burette, mL (V_2)
		Initial	Final	
1. 2. 3.				

CALCULATION: The concentration of available chlorine is calculated using the equation,

$N_2V_2 = N_3V_3$ where

N_2 = Normality of hypo solution =

V_2 = Volume of hypo rundown from burette = mL

V_3 = Volume of bleach sample solution pipetted out = 10.00 mL and

N_3 = Normality of 'Available Chlorine' in bleaching powder sample solution, which is calculated as $N_3 = \frac{N_2V_2}{V_3}$ =

Normality of available chlorine content in bleaching powder sample solution, N_3 =

Available Chlorine present in 100 mL bleaching powder sample solution,

$\mathbf{X} = \frac{N_3 \times \text{Eq. Wt. of Cl} \times 100}{1000}$

$= N_3 \times 35.45 / 10 = 3.545 \times N_3$

Amount of bleach sample taken , **Y =** g

% of available Chlorine in bleaching powder = $\frac{\mathbf{X} \times 100}{\mathbf{Y}}$ =

Table: C Percentage Error Table

Roll/Regd. No.	Flask No.	% of Available chlorine		% Error
		Given	Reported	

REPORT: Percentage of available chlorine content in bleaching powder =

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11. DETERMINATION OF HARDNESS OF A WATER SAMPLE

AIM: To determine the total hardness of an underground water sample using standard EDTA solution.

APPARATUS : 50 mL burette, stand and clamp, glazed tile,10 mL pipette, 250 mL beakers, 50 mL measuring jar, 250 mL conical flask, 100 mL volumetric flask , wash bottle, etc.

CHEMICALS REQUIRED: Standard zinc sulphate solution, EDTA solution, pH-10 buffer and water sample.

INDICATIOR: EBT (Eriochrome Black – T)

END POINT: Wine red to blue.

THEORY: Hardness, soap consuming capacity, of water is mainly due to the presence of bicarbonates, carbonates, sulphates, chlorides, and nitrates of calcium and magnesium. When these metals are present as their bicarbonates in water, they cause temporary hardness to water. When water containing these bicarbonates is boiled, the bicarbonates are converted into their corresponding insoluble carbonate or hydroxides, as the case may be, and hence can be removed by sedimentation or filtration. Sulphates, chlorides and nitrates of calcium, magnesium, zinc, etc., cause permanent hardness to water which cannot be removed by mere boiling. Conventionally, water hardness is expressed in terms of $CaCO_3$ in parts per million (p p m). Hardness of water can be determined using standard EDTA solution as titrant and EBT as indicator. NH_4OH – NH_4Cl buffer solution is added to maintain a pH of 9-10, as EDTA forms stable complexes with calcium and magnesium at this pH. EBT functions as a metal ion indicator to locate the end point by forming a relatively less stable complex with metal ion compared to that formed by EDTA. In the present context, at the equivalence point, once all the free calcium and magnesium ions in water are completely removed by complexation with EDTA, the calcium and magnesium ions in Ca/Mg- EBT complex get preferably complexed by EDTA as the latter is stronger ligand than EBT and the EBT indicator is set free imparting blue color to the solution.

$$Ca^{+2} / Mg^{+2} + \underset{\textit{Blue}}{EBT} \xrightarrow{\text{pH-10}} \underset{\textit{(Wine-red coloured less stable complex)}}{Ca^{+2} / Mg^{+2}\text{- EBT}} \xrightarrow[EDTA]{\text{At end point}} \underset{\textit{(Colourless \& more stable complex)}}{(Ca/Mg - EDTA)} + \underset{\textit{Blue}}{EBT}$$

PART – A

STANDARDISTATION OF EDTA USING STANDARD ZINC SULPHATE SOLUTION

All the given glass apparatus are washed with tap water first and then rinsed with distilled water. Burette is rinsed with EDTA solution and filled with the same solution up to the zero mark without any air bubbles and then clamped to the burette stand. Pipette is first rinsed with standard Zn(II) solution and 10 mL of it is pipetted out into a clean 250 mL

conical flask. To this solution, 30 mL of distilled water and 1 mL of pH - 10 Buffer solution, followed by 2 drops of EBT indicator are added. The colour of the solution is wine red at this stage. The solution is titrated against EDTA solution taken in the burette until the colour of the solution turns from wine red to blue. The burette readings are noted down to the nearest 0.05 mL in Table-A. The titrations are repeated taking fresh 10 mL aliquots of zinc(II) solution and following the above procedure until two successive concordant titers are obtained and the readings are recorded in Table-A.

Table-A: Standardisation of EDTA solution using standard zinc(II) solution.

S.NO	Volume of standard zinc(II) solution pipetted out, mL (V_1)	Burette Readings		Volume of EDTA solution rundown from burette, mL (V_2)
		Initial	Final	
1 2 3				

CACULATIONS: As zinc(II) and EDTA react in 1 : 1 mole ratio, the concentration of EDTA is calculated using the formula,

$$V_1 M_1 = V_2 M_2$$

where,

V_1 = Volume of Zn(II) solution pipette out = 10.00 mL

M_1 = Molarity of Zn(II) solution =

V_2 = Volume of EDTA solution rundown from the burette

and M_2 = Molarity of EDTA solution

Therefore, Molarity of EDTA solution is calculated as, $M_2 = V_1 M_1 / V_2$

PART – B

DETERMINATION OF TOTAL HARDNESS OF A WATER SAMPLE USING STANDARD EDTA SOLUTION

The standardized EDTA solution in the burette is filled with the beaker containing EDTA solution and the burette is washed with tap water and then rinsed with distilled water. The burette is now rinsed with newly prepared standard 0.01 M EDTA solution and is filled with the same solution up to the zero mark without any air bubbles and clamped to the burette stand. 50 mL of water sample is taken into a clean 250 ml conical flask by means of 50 mL measuring jar. To this 1 mL of buffer solution followed by 2 drops of EBT indicator is added. The colour of the solution is wine red at this stage. This solution is titrated against 0.01 M EDTA taken in the burette until the colour of the solution turns from wine red to clear blue and the burette readings are noted down to the nearest 0.05 mL in Table-B. The above process is continued with fresh 50 mL portions of water sample until two

successive concurrent titers are obtained and the readings are recorded in Table-B. The hardness concentration in water sample is calculated and reported as shown under Table-B.

Table-B: Determination of hardness of a water sample using 0.01M EDTA solution

S.NO.	Volume of water sample, mL (V_4)	Burette Readings		Volume of 0.01 M EDTA solution rundown from burette, mL (V_3)
		Initial	Final	
1. 2. 3.	50 50 50			

CALCULATIONS: According to law of equivalence

$$V_3 M_3 = V_4 M_4$$

where,

V_3 = Volume of 0.01 M EDTA rundown from the burette,

M_3 = 0.01 M

M_4 = Molarity of hardness in water and

V_4 = Volume of water sample taken = 50 mL

Therefore hardness concentration in water, $M_4 = \frac{0.01 \times V_3}{50}$

Finally the total hardness of water sample is calculated using the formula,

M_4 x Mol.Wt.of $CaCO_3$x 1000 = M_4 x 100 x 1000 = ppm

REPORT: The total hardness of water sample is ppm.

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12. DETERMINATION OF VISCOSITY OF A LIQUID

AIM: To determine the viscosity of the given liquid using Ostwald's viscometer

APPARATUS: Ostwald's viscometer, stop watch, specific gravity bottle, pipette, clean flexible rubber tubing, pinch cock, etc.

REAGENTS: Distilled water and organic solvent.

THEORY: Determination of viscosity using Ostwald's viscometer is based on Poiseuille's equation, which relates the rate of flow of a liquid through a capillary tube with the coefficient of viscosity. The Poiseuille's equation for viscosity can be expressed as:

$$\eta = \frac{\rho \pi r^4 t}{8 v l} \qquad (1)$$

where v is volume of the liquid (of viscosity η) flowing in time 't' through a capillary tube of radius 'r', and length ι, when a pressure difference 'ρ' is maintained between the two ends of the tube.

The determination of absolute viscosity by means of Poiseuille's expression requires a knowledge of v, r, t, ι and ρ. But in practice this method of determination of η is tedious. Hence a simpler method of comparing the viscosities of two liquids can be followed. If t_1 and t_2 are the flow times required for equal volume of two liquids to flow through the same length of capillary tube, then

$$\eta_1 / \eta_2 = \rho_1 t_1 / \rho_2 t_2 \qquad (2)$$

As $\rho = h d g$ and h and g are constant under the conditions, equation (2) becomes

$$\eta_1 / \eta_2 = d_1 t_1 / d_2 t_2 \qquad (3)$$

where d_1 and d_2 are the respective densities of the two liquids, and can be readily measured or obtained.

PROCEDURE:

The viscometer (Fig. 9) is cleaned first with chromic acid, followed by tap water and then with distilled water. It is finally washed with alcohol and ether and then dried. A piece of clean rubber tubing is attached to the end 'a' and the viscometer is clamped to the stand vertically. Sufficient volume of distilled water is introduced in bulb 'e' so that the bent portion of the tube and half or a little more than half of bulb 'e' are filled up. With the help of the rubber tube attached to the upper arm "a", water is sucked until it raises much above the upper mark "c" and is allowed to flow under its own weight. The time required for the

flow of water from ‘c ’ to ‘d ’ is noted using a stopwatch. The same procedure is repeated three times, the data are entered in Table-1 and the mean value is calculated. The viscometer is cleaned with alcohol and ether and dried. The same procedure is repeated with an identical volume of the given liquid and the time of flow of liquid is measured and the values are recorded in Table-1. The relative density of the given liquid is determined using pycnometer.

Table - 1 : Determination relative viscosity of a liquid

	Water	Given liquid
Trial No.	Time of Flow, t_1 Seconds	Time of Flow, t_2 Seconds
1. 2. 3. Mean value	----------	----------

CALCULATIONS: Finally the relative viscosity of the given liquid, η_2 is calculated by substituting the known values of d_1, d_2 and η_1 in the equation

$$\eta_2 = \eta_1 d_2 t_2 / d_1 t_1$$

where

η_1 = Viscosity of water = poise

η_2 = Viscosity of the given liquid = poise

d_1 = Density of water= gm/cm^3

d_2 = Density of the given liquid = gm/cm^3

t_1 = Mean value of time of flow of water= sec

t_2 = Mean value of time of flow of the given liquid= sec

Precautions

1. The viscometer should be thoroughly cleaned.
2. Viscometer must be kept in vertical position
3. Same volumes of liquid and water are to be taken while performing the experiment

REPORT: The relative viscosity of the given liquid with respect to water at room temperature is ---------- poise.

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13. DETERMINATION OF SURFACE TENSION OF A GIVEN LIQUID

AIM: To determine the surface tension of the given liquid at room temperature using a Stalagmometer.

APPARATUS: Stalagmometer, beaker, clean rubber tubing, pinch cock, relative density bottle and thermometer.

REAGENTS: Distilled water and an organic solvent.

THEORY: Surface tension is a manifestation of the forces of attraction that hold the molecules together in the liquid state; thus, liquid droplets tend to become spheres – the form of least surface area – because of the mutual cohesion of the molecules. When a liquid is allowed to flow down through a capillary tube, a drop is formed at its lower end. The drop increases to a certain size and then falls down. The size of the drop formed depends on the radius of the capillary and the surface tension of the liquid. The surface tension acting along the circumference of the capillary tube supports the drop in the upward direction.

The measurement of surface tension of a liquid is based on the fact that the drop of the liquid formed at the lower end of capillary falls down when the weight of the drop becomes equal to the surface tension. The surface tension of the given liquid γ_2 is determined relative to that of water γ_1 at room temperature by using stalagmometer. The number of drops formed for the same volume of water and the given liquid are counted and let they be n_1 and n_2 respectively. Now if the densities of water and the given liquid at room temperature are determined separately using a specific gravity bottle, then the surface tension γ_2 of the given liquid can be calculated using the relationship

$$\gamma_1/ \gamma_2 = n_2 d_1/ n_1 d_2$$

where, d_1 and d_2 are the densities of water and the given liquid respectively.

PROCEDURE:

The stalagmometer is cleaned thoroughly first with chromic acid solution, tap water and finally with distilled water, and then dried. The upper end of the stalagmometer is fitted with a rubber tube and pinch cock set. The lower end of the stalagmometer is immersed in a beaker containing distilled water. The distilled water is sucked into the stalagmometer until the level rises a little above the mark 'C' and the pinchcock screw is tightened. The lower end of the stalagmometer is raised much above the level of the distilled water in the beaker and the stalagmometer is fixed to its stand vertically. By careful manipulation of the screw of the pinch cock, the water in the stalagmometer is allowed to flow down at a rate of 15 – 20 drops per three minutes. Counting of the drops is started when the meniscus just reaches the upper mark 'C' and stopped when the meniscus just passes the lower mark 'D'

and the number of drops fallen down is noted in Table 1.The same procedure is repeated thrice, the number of drops in each case and the mean of the three values is also recorded in Table 1. The stalagmometer is once again cleaned and dried. The entire procedure is repeated with the given sample liquid and the corresponding data also entered in Table1. The density of the given liquid sample d_2 is determined using a specific gravity bottle.

Table 1: Determination of surface tension of a liquid

Trial. No	Distilled Water	Given liquid
	No. of drops, n_1	No. of drops, n_2
1. 2. 3. Mean value	----------	----------

CALCULATIONS: As γ_1 , n_1 , d_1 and d_2 are known, the surface tension of the given liquid γ_2 , is calculated using the formula,

$$\gamma_2 = \gamma_1 \, n_1 \, d_2 \, / \, n_2 \, d_1$$ where

γ_1 = Surface tension of water = …….. dynes/cm

γ_2 = Surface tension of the given liquid =
n_1 = Mean value of No. of drops of water =
n_2 = Mean value of No. of drops of the given liquid =
d_1= density of water = g/cm^3
d_2 = density of the given liquid = g/cm^3

Precautions:

1. The stalagmometer and specific gravity bottle should be cleaned properly and dried before use.
2. The stalagmometer should be fixed to its stand vertically
3. The number of drops formed must be 15 to 20 per 3 minutes

REPORT: The relative surface tension of the given liquid with respect to water at room temperature is_____Dynes/cm

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14. DETERMINATION OF IRON(II) PRESENT IN MOHR'S SALT BY POTENTIOMETRIC METHOD

AIM: To determine the amount of iron(II) present in Mohr's salt solution by titration with standard potassium dichromate solution using potentiometric method.

APPARATUS: Potentiometer, calomel electrode, platinum electrode, beaker, (KCl salt bridge), spirit lamp and plastic stirrer.

REAGENTS: Standard (N/20) potassium dichromate solution and Mohr's salt solution.

THEORY: The principle behind potentiometric titration of iron(II)solution with potassium dichromate { Cr(VI)} solution can be explained using modified Nernst equation, viz.,

$$E = E^{o}_{Oxd/Red} + \frac{0.0591}{n} \log_{10} \frac{[Oxd]}{[Red]} \qquad (1)$$

where E, E^{o}, [Oxd], [Red] and 'n' are the Experimental electrode potential at room temperature, Standard reduction potential, concentration of Oxidised form, concentration of Reduced form and number of electrons involved in the redox process of the system respectively. Considering the present titration, the two systems of relevance, viz., Fe(III)/Fe(II) and Cr(VI)/Cr(III) have E^{o} values of 0.76 V, 1.36 V respectively, with a large potential difference of 0.6 V and hence, quantitative oxidation of Fe(II) to Fe(III) by Cr(VI) should be possible. Basing on equation (1), the expressions for the experimental potentials (EMFs) of the two systems at room temperature may be written as

$$E_{Fe(III)/Fe(II)} = E^{o}_{Fe(III)/Fe(II)} + \frac{0.0591}{1} \log_{10} \frac{[Fe(III)]}{[Fe(II)]} \ , \ E^{o}_{Fe(III)/Fe(II)} = 0.76 \text{ V} \qquad (2)$$

$$E_{Cr(VI)/Cr(III)} = E^{o}_{Cr(VI)/Cr(III)} + \frac{0.0591}{3} \log_{10} \frac{[Cr(VI)]}{[Cr(III)]} \ , \ E^{o}_{Cr(VI)/Cr(III)} = 1.36 \text{ V} \qquad (3)$$

At the beginning of the titration, only Fe(III) and Fe(II) species are present in the titration vessel and the potential displayed by the meter is mainly due to that of Fe(III)/Fe(II) system only. As the titration progresses, more of Fe(III) is formed, Fe(III)/Fe(II) ratio increases and there will be slow but steady increase in the potential. Once all the Fe(II) is quantitatively oxidized, to Fe(III) by Cr(VI) at the equivalence point, the next added drop of Cr(VI) causes a sudden large jump in potential (EMF) as the response of the potentiometer is now mainly due to that of Cr(VI)/Cr(III) system (1.36 V).

The titration is accomplished using an inert indicator electrode (Platinum electrode), a Reference electrode (Calomel electrode) and a digital potentiometer (Milli-Volt meter) (Fig. 12). The relevant electrochemical cell reaction of the reductant may be represented as

Pt/Fe(III)/Fe(II) *soln*., 1N H_2SO_4 // KCl *(sat)*,Hg_2Cl_2 (*s*)/ Hg(l)

PROCEDURE:

10 mL of the given Mohr's salt solution is pipetted out into a clean 100 mL beaker. 5 mL of 1:1 sulphuric acid and 35 mL of distilled water are added to it using a measuring jar. The end terminals of the indicator electrode (platinum) and reference electrode (calomel) are connected to the proper terminals of the potentiometer after placing the electrodes in the titration vessel (Fig.12). The contents of the beaker are then titrated against standard dichromate solution taken in the burette. A pilot titration is carried out by adding 1.00 mL portions of dichromate solution each time into the reaction vessel. The solution is thoroughly mixed after each addition of the titrant ($K_2Cr_2O_7$) solution, making use of a plastic stirrer and the corresponding burette readings and emf (potential) values, as displayed by the potentiometer, are recorded in Table 1. After reaching a certain stage in the titration process, a sudden and large change in potential will be noticed, indicating overstepping of the equivalence point. The process up to this stage, which locates the approximate equivalence point, is known as ***pilot titration***. An ***Accurate titration***, on similar lines to the pilot titration, is carried out until a volume 1 mL before the equivalence point is reached. After this stage, the titration is continued by adding 0.1 mL increments of dichromate solution (instead of 1 mL), until the equivalence point is crossed. The procedure is continued until 3 more mL of the titrant is added after the equivalence point, adding fractions of 1 ml increments of the titrant. All these observations are recorded in Table 2. A graph is then drawn between volume of dichromate solution taken on X-axis and the corresponding potentials on Y–axis, for the accurate titration. From the graph, the correct equivalence point is ascertained and the concentration of iron(II) calculated as shown under table 2.

Pilot titration of Mohr's salt solution with standard solution of potassium dichromate

10.0 mL Mohr's Salt Solution + 5 mL of 1:1 H_2SO_4 + 35 mL of distilled water = 50 mL

Table 1: Readings of Pilot titration

S.No	Volume of dichromate solution, mL	Potential, mV
1 2 --- n		

Accurate titration of Mohr's salt solution with standard solution of potassium dichromate

10.0 mL of Mohr's Salt Solution + 5 mL of 1:1 H_2SO_4 + 35 mL of distilled water = 50 mL

Table 2. Readings of Accurate titration

S.No	Volume of dichromate solution, mL	Potential, mV
1 2 --- n		

CALCULATIONS: The concentration of iron(II) in Mohr's salt solution is calculated using the formula $V_1N_1 = V_2 N_2$ where,

V_1 = Volume of Mohr's salt solution pipetted = 10.00 mL

N_1 = Normality of iron in Mohr's salt solution = ?

V_2 = Volume of dichromate solution = mL

N_2 = Concentration of dichromate solution = N (will be given)

As V_1, V_2 and N_2 are known, Normality of iron(II) in Mohr's salt solution, N_1 may be calculated as

$$N_1 = V_2 N_2 / V_1$$

Amount of Iron(II) present in Mohr's salt per 1 liter = N_1 x Eq. wt. of iron(II)
= N_1 x 55.845 = g

Therefore, amount of iron(II) present in 100 ml = $\frac{N_1 \times 55.845}{10}$ = N1 x 5.584 = g.

Table 3: Report Table

Roll No. / Regd.No.	Flask No.	Amount of iron(II) present in 100 mL of Mohr's salt solution, g		Percentage error
		Given	Reported	

REPORT: Amount of iron(II) present in 100 mL of the given Mohr's salt solution

Precautions

1. The platinum electrode should be activated by strong heating using spirit lamp before starting the titration.

2. The solution should be thoroughly stirred with a plastic stirrer after each addition of the titrant.

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15. DETERMINATION OF STRENGTH OF HYDROCHLORIC ACID BY pH-METRIC METHOD

AIM: To determine the strength of hydrochloric acid using standard sodium hydroxide by pH - metric method.

APPARATUS: pH meter, combined electrode/glass electrode and calomel electrode, 100 mL beaker, 10 mL micro burette and plastic stirrer.

REAGENTS: Distilled water, standard 0.1N NaOH solution, HCI sample solution, standard Buffer solutions of pH 4 and 9.2

THEORY: For many purposes, especially when dealing with small concentrations, it is cumbersome to express concentrations of H^+ and OH^- ions in terms of gram equivalents per liter. A very convenient method to express the concentrations of these ions was proposed by S.P.L. Sorensen in 1909. He introduced the H^+ ion exponent, pH, defined by the relationships:

$$pH = - \log_{10} [H^+] = \log_{10} 1/[H^+] \text{ or } [H^+] = 10^{-pH}$$

The quantity of pH is thus the logarithm (to base 10) of the reciprocal of the H^+ ion concentration, $[H^+]$, or is equal to the logarithm of the $[H^+]$ with negative sign. This method of expression has the advantage that all states of acidity and alkalinity between 1N in $[H^+]$ to 1N in $[OH^-]$ can be expressed by a series of positive numbers between **zero** and **14**. Thus, a neutral solution with $[H^+] = 10^{-7}$ has a pH of 7; a solution 1N in $[H^+]$ has a pH of zero; and a solution 1N in $[OH^-]$ possesses a pH of 14.

The common lab pH meter (Fig. 13) is an electronic digital milli voltmeter, scaled to read pH directly with a resolution of 0.01 pH unit and an accuracy of ± 0.01 unit. The reading displayed by the meter is a direct measure of the potential difference registered between a glass electrode and calomel electrode (reference electrode) immersed in the test solution. The basic electrochemical cell involving glass electrode (Indicator electrode), calomel electrode (reference electrode) and test solution may be represented as:

Ag/AgCl(*s*), HCl/glass /Test solution// | KCI (*sat*), Hg_2Cl_2 (*s*) |Hg(l)

The strength of given Hydrochloric acid solution can be determined using standard sodium hydroxide solution following the pH - metric method.

PROCEDURE:

The given hydrochloric acid sample solution in 100ml volumetric flask is made up to the mark with distilled water and homogenised. 10 mL of the solution is pipetted out in to a clean 100 mL beaker. 40 mL of distilled water is added to this solution. Meanwhile the pH meter is switched on and is allowed to stabilize for about 10 minutes. The instrument is calibrated with standard buffers of pH 4.0 and 9.2, after connecting the combined electrode, or else, glass electrode calomel electrode combination to the pH meter. The cleaned glass

and calomel electrodes or the combined electrode is dipped into the HCL solution in the 100 mL beaker and the solution is stirred and the reading displayed by the pH - meter noted. The contents of the beaker are then titrated against standard sodium hydroxide solution taken in the burette. A pilot titration is carried out by adding 1.00 mL increments of the titrant (sodium hydroxide) solution each time. The solution is thoroughly mixed after each addition of the titrant, making use of a plastic stirrer and the corresponding pH values as displayed by the pH - meter are recorded in Table 1. After reaching a certain stage in the titration process, a sudden and large change in pH will be noticed indicating overstepping of the equivalence point. The process up to this stage, which locates the approximate equivalence point, is known as ***pilot titration***. An ***Accurate titration***, on similar lines to the pilot titration, is carried out until a volume 1 mL before the equivalence point is reached. After this, the titration is continued by adding 0.1 mL increments of the titrant solution until the equivalence point is crossed. The process is continued until 3 more mL of the titrant is added, after the equivalence point. All the relevant observations are recorded in Table 2. A graph is then drawn between volume of titrant (sodium hydroxide) solution taken on X-axis and the corresponding pH values on Y–axis, for the accurate titration. From the graph, the correct equivalence point is ascertained and the concentration of hydrochloric acid in the sample is calculated as shown under table 2.

Pilot titration of hydrochloric acid solution with standard sodium hydroxide solution

10 mL of hydrochloric acid + 40 mL of distilled water = 50 mL

Table 1: Pilot titration of HCl with NaOH

S.No	Volume of NaOH Solution added, mL	pH
1 2 -- n		

Accurate titration of hydrochloric acid solution with standard sodium hydroxide solution

10 mL of hydrochloric acid + 40 mL of distilled water = 50 mL

Table 2: Accurate titration of HCl with NaOH

S.No	Volume of NaOH Solution added, mL	pH
1 2 -- N		

CALCULATIONS: The concentration of HCl in the given sample solution is calculated using the formula $V_1N_1 = V_2N_2$ where,

V_1 = Volume of sodium hydroxide = mL

N_1 = Normality of sodium hydroxide = (will be given)

V_2 = Volume of hydrochloric acid = 10.00 mL

N_2 = Normality of hydrochloric acid = ?

As V_1, V_2 and N_1 are known, Normality of HCl in the given sample solution, N_2 , may be calculated as

$$N_2 = V_1 N_1 / V_2$$

Amount of hydrochloric acid present in 100 ml of the given solution is equal to

$$\frac{\text{Normality of HCl x Eq. Wt. of HCl(36.45)}}{10}$$

$$= N_2 \times 3.645$$

Amount of Hydrochloric acid present in 100 ml of the given solution is: –----- g

Table: 3 Report Table

Roll No. / Regd.No.	Flask No.	Amount of Hydrochloric acid present in 100 ml of the solution, g		Percentage error
		Reported	Given	

REPORT: Amount of hydrochloric acid present in 100 mL of the given solution

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16. ESTIMATION OF ZINC IN ZINC ORE BY EDTA METHOD

Aim: Estimation of Zinc present in Zinc ore solution by titrating against a standardized EDTA solution using Eriochrome Black- T as indicator.

Theory: Zinc in a given ore sample solution can be determined by using the complexometric method, in which the Disodium salt of EDTA is employed (soluble in water) and it can be represented as follows.

(EDTA – Ethylene Diamine Tetra Acetic acid)

$HOOCH_2C$ CH_2COONa

$N\text{-}CH_2\text{ - }CH_2\text{ -}N$

$NaOOCH_2C$ CH_2COOH

EDTA forms complexes with Mn+ (Ca^{+2}/Mg^{+2}/Za^{+2} etc.,)when the pH is in the range of around 9.5 to 10.5 and to maintain the pH, a basic buffer solution is used (NH4OH + NH4Cl buffer serves pH 9.5 to 10.5). The metal-EDTA complexes are colourless, therefore it is necessary to use indicator to locate the end point. In this titration Eriochrome black – T is used as indicator, which forms an unstable wine red coloured complex with zinc. When once all the zinc ions are completely removed by EDTA, free indicator is left in the solution which imparts blue colour to the solution. So the colour change at the end point is wine red to blue.

$$Zn^{2+}(aq) + In^{2-} \xrightarrow{pH-10} [Zn\text{-}In] + EDTA^{4-}(aq) \xrightarrow{pH-10} Zn(EDTA)^{2-}(aq) + In^{2-}$$

Unstable wine red coloured complex — Stable complex — Free indicator Blue in colour

Part – I:

Standardisation of di-sodium salt of EDTA solution by titrating against a standard solution of zinc sulphate.

Procedure:

10.0 ml of standard zinc sulphate solution is pipetted out into a clean conical flask carefully. To this 2 or 3 ml of ammonia - ammonium chloride buffer solution (pH 9.5 – 10.5) and 2 or 3 drops of Eriochrome Black – T indicator are added. The burette is filled with EDTA solution, after rinsing with same and the initial reading is noted. Now the contents are titrated with EDTA solution until the colour changes from wine red to blue which is the end point of the reaction. The final reading of the burette is noted. A number of titration are carried out until 3 or 4 concurrent readings are obtained. The results are tabulated in Table– I.

Table – I

Standardization of EDTA solution with standard zinc sulphate solution

Morality of standard solution of EDTA _____ M

Indicator: Eriochrome Black – T.

Colour change at the end point: Wine red to blue

S.No.	Volume of Zinc sulphate solution taken in ml.	Burette readings		Volume of EDTA solution consumed in ml.
		Initial	Final	

Calculations:

By the law of equivalence $V_1 M_1/n_1 = V_2 M_2/n_2$

M_1 = Molarity of EDTA solution = ?

V_1 = Volume of EDTA solution = ml.

n_1 = Number of moles of EDTA =

M_2 = Molarity of zinc sulphate solution = M

V_2 = Volume of zinc sulphate solution = ml.

n_2 = Number of moles of zinc sulphate =

$\therefore M_1 = V_2 M_2 n_1 / N_2 V_1$

The Molarity of EDTA solution = _____ M

Part -II:

Estimation of Zinc present in Zinc ore solution by titrating against a standardized EDTA solution using Eriochrome Black- T as indicator.

Procedure:

The given zinc ore solution (as sulphate) is diluted up to the mark of volumetric flask with distilled water carefully. The flask is stoppered tightly and is shaken thoroughly about 3-5 minutes for complete homogenization. 10.0 ml of the zinc ore solution is pipetted out into a clean conical flask carefully. To this 2 or 3 ml of ammonia - ammonium chloride buffer solution (pH 9.5 – 10.5) and 2 or 3 drops of Eriochrome Black – T indicator are added. The burette is filled with EDTA solution, after rinsing with same and the initial reading is noted. Now the contents are titrated with EDTA solution until the colour changes from wine red to blue which is the end point of the reaction. The final reading of the burette is noted. A number of titration are carried out until 3 or 4 concurrent readings are obtained. The results are tabulated in Table– II.

Table – II

Titration of standard EDTA solution with zinc ore solution

Morality of standard solution of EDTA _____ M

Indicator: Eriochrome Black – T.

Colour change at the end point: Wine red to blue

S.No.	Volume of Zinc ore solution taken in ml.	Burette readings		Volume of EDTA solution consumed in ml.
		Initial	Final	

Calculations:

By the law of equivalence $V_3 M_3 / n_3 = V_4 M_4 / n_4$

M_3 = Molarity of EDTA solution = M

V_3 = Volume of EDTA solution = ml.

n_3 = Number of moles of EDTA =

M_4 = Molarity of zinc ore solution = ?

V_4 = Volume of zinc ore solution = ml.

n_4 = Number of moles of zinc sulphate =

$\therefore M_4 = V_3 M_3 n_4 / n_3 V_4$

The Molarity of zinc ore solution = _____ M

Molecular weight of zinc = atomic weight = 65.39

Amount of zinc present in 1 liter of solution = {Normality of zinc ore solution x molecular weight of zinc} = g

Amount of zinc present in the given 100 ml of zinc ore solution ={ amount present in 1 liter of the solution / 10 } = g

Table – III
Percentage Error Table

Roll No. / Regd. No.	Flask No.	Amount of zinc present in the given100 ml of ore solution in Grams.		Percentage of error
		Reported	Given	

Report: The amount of zinc present in the given 100 ml of an unknown solution is _____ g.

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17. DETERMINATION OF SULPHURIC ACID PRESENT IN LEAD ACID STORAGE BATTERY THROUGH ACID BASE TITRATION

Aim: Determination of % sulphuric acid present in Lead acid storage battery through acid base titration using standard sodium hydroxide solutions.

Importance of acid in lead acid storage battery

Electrochemical reactions in lead acid storage battery

$Pb+PbO_2+H_2SO_4 \longrightarrow 2PbSO_4+2H_2O$ + Electrical energy (During discharging)

$2PbSO_4+2H_2O$ + Electrical energy $\longrightarrow$ $Pb+PbO_2+H_2SO_4$ ((During charging)

The above reaction states that during the discharge of the battery it consumes sulphuric acid, the state of the discharging of the battery can be checked by measuring the density of battery. The density of sulphuric acid in healthy battery should be more than 1.2 gm/cm^3 (20-21%).

Theory: A reaction between sulfuric acid and sodium hydroxide is of an acid-base type, or is also known as a neutralization reaction. In this process, both compounds undergo a reaction to neutralize the acid and base properties. The products of this process are salt and water. The balanced equation of this reaction is:

$$H_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + 2H_2O$$

In this reaction salt and water are formed, which is an example of neutralization reaction. All the reactants and products are colourless, so phenolphthalein indicator is used to locate the end point of the reaction. The colour change of phenolphthalein is from colorless in acidic medium to pink in alkaline medium.

Procedure: The given acid solution is made up to the mark of volumetric flask with distilled water carefully. The flask is stoppered and shaken thoroughly about 2 to 3 minutes for complete homogenization. The burette is rinsed with the given acid solution and filled with same without air bubbles. 10ml standard sodium hydroxide solution is pipetted out into a clean conical flask carefully and 50.0 mL of distilled water is added with measuring jar. Two drops of phenolphthalein indicator is added directly to the contents of conical flask. The conical flask contents are titrated with given acid solution after noting the initial reading. The titration is continued till the colour changes from pink to colourless. The final reading of burette is noted. A number of titrations are repeated for getting concurrent results. The results are tabulated in table no. I.

Table – I

Titration of Sulphuric Acid solution with standard sodium hydroxide solution

S.No.	Volume of standard sodium hydroxide solution taken in ml. (V_2)	Burette reading		Volume of Sulphuric acid solution consumed in (V_1) ml.
		Initial	Final	

Calculations:

By the law of equivalence $V_1N_1 = V_2N_2$

N_1 = Normality of Sulphuric Acid solution = ?

V_1 = Volume of Sulphuric Acid solution = ml.

N_2 = Normality of Sodium hydroxide solution = N

V_2 = Volume of Sodium hydroxide solution = ml.

$\therefore N_1 = V_2N_2 / V_1$

$\therefore$ The Normality of Acid solution is_____ N.

Equivalent weight of Sulfuric acid =Molecular weight / 2=98/2=49

Amount of Sulphuric Acid present in 1000 ml of the solution = {Normality of Sulfuric acid solution X Equivalent weight of Sulphuric Acid} = gm.

Amount of Sulphuric Acid present in the given 100 ml of solution =

{Amount present in 1000 ml of solution / 10} = gm

Specific Gravity: Tare an empty dry 100 mL beaker over a balance Transfer carefully 10ml (or 25ml) of Acid solution using a volumetric pipette into the beaker. Note the volume used and weight read out. Repeat this for 2 more times to have 3 weight readings and average out the result.

Specific gravity= Mass (g)/Volume (mL) =

1 mole H_2SO_4 in 1000 mL weigh 98g

If test solution has X moles H_2SO_4 in 1000ml (XM)

The weight of H_2SO_4 molecules in 1000mL is X x 98 = Yg

Percentage Concentration [%]= (% weight of acid molecules in 100mL×100/Weight of 100 mL acid)

Report: Amount of Sulfuric acid present in the given 100 mL of solution…….gm.

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18. PREPARATION OF UREA FORMALDEHYDE

AIM: To prepare urea formaldehyde resin.

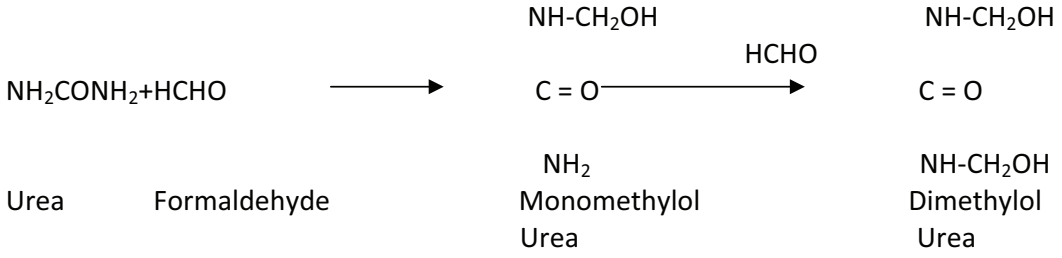
APPARATUS REQUIRED: Beaker, glass rod, funnel, filter paper and chemical balance.

CHEMICALS: Urea, formaldehyde sol., conc. H_2SO_4, distilled water.

THEORY: Amino resins are condensation products obtained by the reaction of formaldehyde with nitrogen bearing compounds such as aniline, amides for ex:- melamine formaldehyde, urea formaldehyde etc.

Urea formaldehyde is prepared by condensation reaction between urea and formaldehyde in acidic or alkaline medium.

The first product formed during the formation of resin is monomethylol and dimethylolureas.



Polymerization can take place from mono or dimethylol urea or possibly through both, with the formation of long chains

PROCEDURE:

1. Place about 5 ml of 40% formaldehyde solution in 100 ml beaker.
2. Add about 2.5 g of urea with constant stirring till saturated solution is obtained.
3. Add a few drops of conc. H_2SO_4, with constant stirring.
4. A voluminous white solid mass appears in the beaker.
5. Wash the white solid with water and dry it in the folds of filter paper.
6. Weight the yield of product

PRECAUTIONS:

1. While adding concentrated H_2SO_4, it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.
2. The reaction mixture should be stirred continuously.

OBSERVATIONS:

Mass of the beaker(W1)= ---------- g.
Mass of the beaker with urea formaldehyde(W2)=---------- g.
Therefore, mass of urea formaldehyde (W2 –W1)= ----------g.

REPORT: The yield of urea formaldehyde = ----------g

PROPERTIES:

1. They have good electrical insulating properties.
2. They are resistant to oil, grease and weak acids.
3. They are hard, resist abrasion and scratching.
4. They have good adhesive properties.

USES:

1. They are used adhesive applications for the production of plywood and laminating.
2. They are used for the manufacture of cation exchange resins.
3. These also find use in the manufacture of electrical switches, plugs and insulating foams.
4. Their applications also include the treatment of textile fibers for improving their shrink and crease resistance.

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19. PREPARATION OF PHENOL FORMALDEHYDE RESIN

AIM: To prepare phenol formaldehyde resin.

APPARATUS: Beaker, glass rod, funnel, filter paper, and chemical balance.

CHEMICALS: Phenol formaldehyde, conc.HCl, glacial acetic acid, distilled water.

THEORY: Phenolic resins are condensation polymerization of phenolic derivatives (like phenol, resorcinol) with aldehyde (like formaldehyde, furfural). Most important member of this class is bakelite or phenol formaldehyde resin.

Phenol formaldehyde is prepared by condensing phenol with formaldehyde in presence of acidic or alkaline catalyst. The initial reaction result in the formation of o- and p-hydroxy methyl phenol, which reacts to form linear polymer navalac.

During molding hexamethyline tetramine [$(CH_2)_6N_4$] is added which convert the fusible novalac in to hard infusible and insoluble solid of cross – linked structure known as Bakelite.

PROCEDURE:

1. Place 5 ml of glacial acetic acid and 2.5 ml of 40% formaldehyde solution in a 100 ml beaker.
2. Add 2 g of phenol to it.
3. Wrap a cloth loosely round the beaker. Add a few ml of conc. HCl in to the mixture carefully and heat it's lightly.
4. A large mass of plastic pink in colour is formed.
5. A residue is washed with water and filtered.
6. The product dried and yield is weighed.

PRECAUTIONS:

1. While adding conc.HCl, it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.
2. The reaction mixture should be stirred continuously.

OBSERVATIONS:

Mass of the beaker (W1)=---------- g.
Mass of the beaker with phenol formaldehyde (W2)=---------- g.
Therefore, mass of phenol formaldehyde (W2 –W1) = ----------g.

RESULT: The yield of phenol formaldehydes=---------- g

PROPERTIES:

1. Phenol formaldehyde molding resins have excellent heat resistance.
2. These have high dimensional stability.
3. Phenolic resins have good dielectric properties.
4. They have hard, rigid and scratch resistant.

USES:

1. They are used for making electric insulator parts like switches, plugs, switch board, heater, handles etc.
2. These are also used in varnishes, paints and protective coatings.
3. These are used in the protection of ion exchange resins for water softening.
4. Phenolic resins are used for improving impregnating paper, wood and other fillers.

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20. SYNTHESIS OF TITANIUM DIOXIDE NANOPARTICLES

Aim: To prepare Titanium dioxide nanoparticles

Reagents

Titanium tetrachloride ($TiCl_4$), 99%;

Titanium isopropoxide ($C_{12}H_{28}O_4Ti$), 99.8%;

Urea ($CO(NH_2)_2$), 99%;

Ammonium chloride (NH_4Cl), 99.5%;

Glacial acetic acid, 99.5%;

Methanol, 99.5%;

Ethanol 99.8%.

Compounds		**Amounts**
$TiCl_4$	:	05 mL
C_2H_5OH	:	50 mL
H_2O	:	200 mL

Procedure:

Ethanol and titanium tetrachloride are introduced into a beaker; the solution is stirred for 30 min. During this period, a yellow sol phase is formed. Double distilled water is added to form clear and colorless solution. The solution is again stirred for 30 min at room temperature and then the formed gel is dried at 50^{o}C for 24 h and characterized by SEM and TEM.

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21. SYNTHESIS AND CHARACTERIZATION OF NANO-SIZED ZnO BY PRECIPITATION METHOD

AIM: Synthesis and Characterization of Nano-sized ZnO by precipitation method

Materials Required

Zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$), 99% , sodium carbonate (Na_2CO_3), 99% and Ethanol (C_2H_5OH, 95%,).

Procedure for Preparation of ZnO nanoparticles

Two solutions are prepared

Solution A: 0.1 mol $Zn(NO_3)_2.6H_2O$ is prepared by dissolving 29.75g of Zinc nitrate hexahydrate in 200 ml distilled water; and

Solution B: 0.12 mol Na_2CO_3 is prepared by dissolving 12.72 g of sodium carbonate in 240 ml distilled water.

After that solution A is added to solution B drop wise under vigorous stirring. The white precipitate is collected by filtration and rinsed with distilled water three times. The solid is then washed with ethanol and dried at 100°C for 6 h. Finally, ZnO nanoparticles are obtained after annealing of the solid in air at 250, 300, 350, 400, 500, and 600°C for 2 h, respectively.

Characterization

- The optical properties of prepared ZnO NPs are analyzed by UV-visible Spectrophotometer (Shimadzu, UV-2450). A broad absorption peak is observed in each spectrum at 355-380 nm which is a characteristic band for the pure ZnO.
- The FT-IR shows a broad absorption band related to Zn-O vibration band at 500 cm^{-1}.
- The SEM results show the formation of spherical shaped nano particles.

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