

## ESTIMATION OF HARDNESS OF WATER BY EDTA METHOD

Experiment No. ....

Date:.....

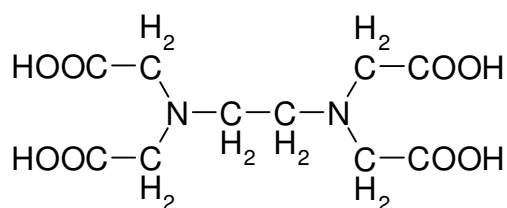
**Aim:** To estimate the amount of total hardness present in the given sample of water by EDTA titration method.

**Apparatus required:** 50 ml Burette, 20 ml Pipette, 250 ml Conical flask, 100 ml Beaker, 250 ml beaker, Glass funnel.

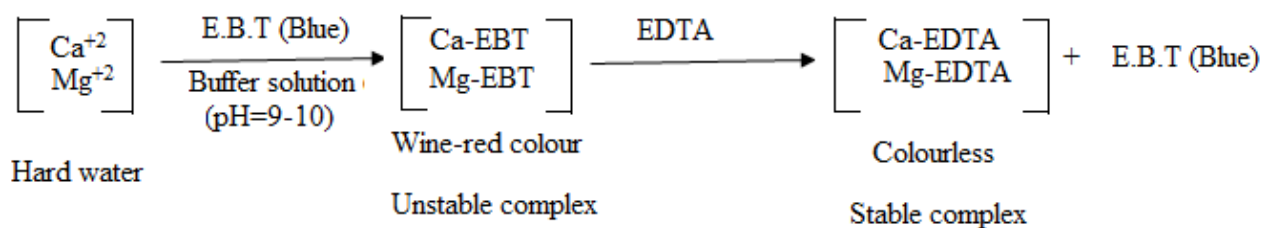
**Reagents:** EDTA solution, Standard  $\text{CaCO}_3$  solution, Eriochrome Black-T indicator, Buffer solution.

**Theory:** EDTA (Ethylenediamine tetra acetic acid) forms colorless stable complexes with  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions present in water at pH = 9-10. To maintain the pH of the solution at 9-10, buffer solution ( $\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$ ) is used. Eriochrome Black-T (E.B.T) is used as an indicator.

The sample of hard water must be treated with buffer solution and EBT indicator which forms unstable, wine-red colored complexes with  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  present in water.



Structure of EDTA



## Procedure

### 1. Standardization of EDTA

- (i) Pipette out 20 ml of standard hard water into a conical flask.
- (ii) Add 5 ml of buffer solution and few drops of Eriochrome Black-T. The indicator, which is originally blue color would acquire a wine-red color.
- (iii) Titrate with EDTA solution taken in the burette, till the wine red color changes to blue which is the end point. Let the burette reading of EDTA be  $V_2$  ml.

### 2. Determination of Total hardness

Repeat the above titration method for sample hard water instead of standard hard water. Let the burette reading of EDTA be  $V_3$  ml.

### 3. Determination of Permanent hardness

Take 100 ml of sample hard water in 250 ml beaker. Boil it to remove temporary hardness to about half of this volume and cool to room temperature. Filter through filter paper to remove insoluble  $\text{CaCO}_3$  and  $\text{MgCO}_3$ . Make up the volume to the original 100 ml by adding distilled water. Now pipette out 20 ml of this solution into a clean conical flask. Then repeat the process of titration steps as mentioned above. Let the burette reading of EDTA be  $V_4$  ml.

## Observations

### 1. Standardization of EDTA

S.No	Vol. of Hard water taken (ml)	Burette Reading		Vol. of EDTA Consumed ( $V_2$ ml)
		Initial	Final	
1.				
2.				
3.				

**2. Determination of Total hardness**

S.No	Vol. of Hard water taken (ml)	Burette Reading		Vol. of EDTA Consumed (V <sub>3</sub> ml)
		Initial	Final	
1.				
2.				
3.				

**4. Determination of Permanent hardness**

S.No	Vol. of Hard water taken (ml)	Burette Reading		Vol. of EDTA Consumed (V <sub>4</sub> ml)
		Initial	Final	
1.				
2.				
3.				

**Calculations****1. Standardization of EDTA**

$$M_1V_1 = M_2V_2$$

Where, M<sub>1</sub> = Molarity of standard hard water

V<sub>1</sub> = Volume of standard hard water in conical flask

M<sub>2</sub> = Molarity of EDTA

V<sub>2</sub> = Volume of EDTA consumed (burette reading)

## 2. Determination of Total hardness

$$M_2V_2 = M_3V_3$$

Where,  $M_3$  = Total hardness of sample water

$V_1$  = Volume of sample hard water in conical flask

## 3. Determination of Permanent hardness

$$M_2V_2 = M_4V_4$$

Where,  $M_4$  = Permanent hardness of sample water

$V_4$  = Volume of sample hard water in conical flask

*Note:* Multiply  $M_3$  and  $M_4$  with  $10^5$  to convert hardness into parts per million (ppm).

## 4. Determination of Temporary hardness

Temporary hardness = Total hardness – Permanent hardness

**Result:** The hardness of the given water sample has been found to be as follows:

Total hardness = ----- ppm

Permanent hardness = ----- ppm

Temporary hardness = ----- ppm

**ESTIMATION OF MANGANESE DIOXIDE IN PYROLUSITE**

Experiment No. ....

Date:.....

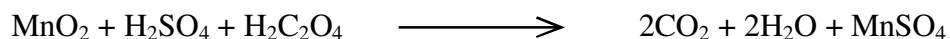
**Aim:** To estimate the amount of manganese dioxide (MnO<sub>2</sub>) present in the given sample of pyrolusite

**Apparatus required:** 50 ml Burette, 20 ml Pipette, 250 ml Conical Flask, Funnel, 250 ml Beaker, 100 ml Measuring cylinder.

**Reagents required:** Manganese dioxide, Oxalic acid (N/10), Dil. H<sub>2</sub>SO<sub>4</sub>, KMnO<sub>4</sub> (N/10).

**Theory:** Manganese dioxide occurs in nature in the form of pyrolusite. Pyrolusite is not only used as a source of manganese but also as an oxidizing agent in many industrial processes. For such purposes, the ore is graded on the basis of its available oxygen content rather than its percentage of manganese since in many cases the pyrolusite contains less available oxygen than that corresponding to the formula MnO<sub>2</sub>.

The MnO<sub>2</sub> present in the pyrolusite sample is reduced to a known excess of standard oxalic acid as acidic medium. The unreacted oxalic acid is titrated against the standard solution of KMnO<sub>4</sub>.



**Procedure:** Weigh about 0.25g finely powdered dry pyrolusite sample into a 250 ml conical flask and add 40 ml of standard N/10 oxalic acid. Add 40 ml of 10% H<sub>2</sub>SO<sub>4</sub> and place a small funnel over the conical flask. Boil the contents of the flask gently until no black particles are visible in the flask. Allow it to cool about 60-70°C and titrate with standard 0.1N KMnO<sub>4</sub> solution. End point is indicated by appear in a pale pink color.

**Observations**

S.No	Volume of oxalic acid (ml)	Burette Reading		Vol. of KMnO <sub>4</sub> consumed (ml)
		Initial	Final	
1.				
2.				
3.				

**Calculations**

Weight of the sample = W gm

Volume of N/10 oxalic acid added = 40 ml

Volume of N/10 oxalic acid used against W gm of pyrolusite = (40-V) ml

Weight of MnO<sub>2</sub> in W gm of the sample = (40-V) ml of 0.1N solution

$$\% \text{ of Mn} = \frac{(40 - V) \times \text{Normality of oxalic acid} \times \text{Atomic Wt. of Mn} \times 100}{1000 \times \text{Sample weight}}$$

$$\% \text{ of Mn} = \frac{(40 - V) \times 0.1 \times 54.94 \times 100}{1000 \times \text{Sample weight}}$$

Where, V = Burette reading

**Result:** The percentage of manganese present in the given pyrolusite sample is .....

## DETERMINATION OF PERCENTAGE OF COPPER IN BRASS

Experiment No. ....

Date:.....

**Aim:** To determine the percentage of copper in the given brass sample.

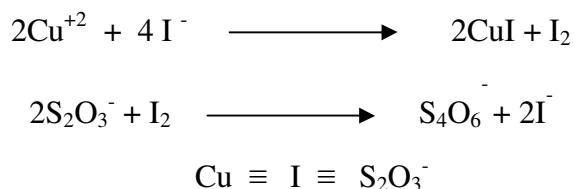
**Apparatus required:** 50 ml Burette, 20 ml Pipette, 250 ml Conical Flask, 100 ml Measuring cylinder.

**Reagents required:** Standard hypo solution (N/10), Conc. H<sub>2</sub>SO<sub>4</sub>, Phosphoric acid, Ammonia solution, Potassium iodide, Starch indicator, Brass sample, Potassium thiocyanate.

**Theory:** Brass is an alloy of copper and zinc. It may also contain a small amount of lead, tin or Aluminum. The composition of brass is: Cu (60-80%) and Zn (20-40%) along with small amounts of Pb (0-2%); Sn (0-6%) and Fe(0-1%).

This Experiment consists of dissolving a known quantity of brass in nitric acid, removing the nitrate by fuming with sulphuric acid. Adjusting the P<sup>H</sup> by ammonium hydroxide, complexing the iron present using phosphoric acid and finally titrating the copper ions with hypo (sodium thiosulphate-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) by iodometric method.

When the brass sample is dissolved in nitric acid, the copper present is brought into solution in the form of cupric ions. At the same time, tin is converted to metastannic acid (SnO<sub>2</sub>.4H<sub>2</sub>O) lead and zinc ions are oxidized to their respective soluble divalent ions, while iron is converted into ferric ions. During the treatment with sulfuric acid nitrate is eliminated, the meta-stannic acid is re-dissolved and lead ions are precipitated as PbSO<sub>4</sub> while all other metals go into solution as their respective sulphates. Out of all the metal ions present in solution, only Cu<sup>+2</sup> and Fe<sup>+3</sup> are reducible by iodide. The interference by iron is eliminated by complexing it with phosphate ion. Then, the Cu<sup>+2</sup> ions can be determined by iodometric method



**Procedure:** A piece of clean and dry brass sample is cut into small pieces. About 0.25g of the sample is accurately weighed and transferred into a conical flask. About 10ml of 6N HNO<sub>3</sub> is added and the flask is gently heated until the brass sample is completely dissolved. Then 10 ml of Conc. H<sub>2</sub>SO<sub>4</sub> is added and the solution is evaporated on a water bath. The mixture is then allowed to cool and 20 ml of water is added very carefully from the sides of the flask. The solution is boiled and then cooled. Now liquor ammonia is added drop wise while shaking the flask until blue cupric ammonium complex appears. Then add 6N H<sub>2</sub>SO<sub>4</sub> slowly until the dark blue color just disappears and cool it followed by 2 ml of phosphoric acid. The solution is cooled and transferred into 100 ml volumetric flask . The volume is made up to the mark by adding distilled water.

20 ml of this solution is pipetted out into a 250 ml conical flask and 10 ml of potassium iodide solution is added. The flask is shaken and the solution is immediately titrated with standard thiosulphate solution until the solution assumes a faint yellow color of iodine. About 3 ml of freshly prepared starch solution is added and titrate with hypo until the blue color disappears (milky white is formed). The titration is repeated for three concurrent values.

### Observations

Sl.No	Volume of solution taken (ml)	Burette Reading		Vol. of thiosulphate consumed (ml)
		Initial	Final	
1.				
2.				
3.				



**Calculations:**

$$\% \text{ of Copper} = \frac{\text{Burette reading} \times \text{Normality of hypo} \times \text{At. Wt. of Cu} \times 100 \times 100}{1000 \times \text{Sample weight} \times 20}$$

$$\% \text{ of Copper} = \frac{\text{Burette reading} \times 0.1 \times 63.54 \times 100 \times 100}{1000 \times \text{Sample weight} \times 20}$$

**Result:** The percentage of Cu present in the given brass sample is .....



**DETERMINATION OF FERROUS IRON IN CEMENT**

Experiment No. ....

Date:.....

**Aim:** To estimate the amount of ferrous iron present in the given sample of cement by colorimetry using ammonium thiocyanate as the reagent.

**Apparatus required:** 50 ml Burette, 20 ml Pipette, 250 ml Beaker, 10 ml Measuring cylinder, 100 ml Volumetric flask, Photo colorimeter.

**Reagents required:** Cement sample, Conc. HCl, Conc. HNO<sub>3</sub>, 40% Potassium thiocyanate (KSCN).

**Theory:** The quantitative calorimetry is based on two principles of photometry namely Lambert's law and Beers law.

**Lamberts law:** When a beam of monochromatic light passes through absorbing medium, the rate of decrease in intensity with the thickness of the medium is proportional to the intensity of the light.

**Beers law:** The intensity of the light emitted decreases exponentially as the concentration of the light absorbing component increased.

**Optical density:** It is the chemical analysis through the measurement of the light radiation in visible region of spectrum.

Ammonium thiocyanate yields a blood red color with ferric iron which is stable in nitric medium. Its optical density is measured in a calorimeter.

**Procedure:** Weigh about 0.1g of the cement sample and transfer it into a 250 ml beaker. Add 5 ml of distilled water followed by 5 ml of Conc. HCl. Heat the solution till the sample get dissolves and evaporate till dryness to expel acid. Cool the beaker and add 20 ml of distill water to dissolve the contents. Transfer the solution into a 100 ml volumetric flask and add 1 ml of

Conc. HNO<sub>3</sub> followed by 5 ml of 40% KSCN. Make up this solution with distilled water up to the mark. Measure the optical density with the help of a calorimeter.

### Calculations

#### *Standard values for calibration curve*

Conc. (in mg)	Optical Density
0.05	0.11
0.10	0.22
0.15	0.32
0.20	0.40
0.25	0.50
0.30	0.62
0.35	0.70
0.40	0.80

$$\% \text{ of Fe} = \frac{\text{Optical density in mg} \times 100}{2 \times 1000 \times \text{Sample weight}}$$

**Result:** The percentage of iron present in the given cement sample is .....

**TITRATION OF STRONG ACID Vs STRONG BASE BY POTENTIOMETRY**

Experiment No. ....

Date:.....

**Aim:** To titrate HCl against standard NaOH potentiometrically.**Apparatus required:** Saturated Calomel electrode, Platinum electrode, potentiometer, 100 ml Beaker, 50 ml Burette, 20 ml pipette, Stirrer and Magnetic bead.**Reagents required:** 0.1M NaOH, HCl**Theory:** The neutralization of acids and bases are always accompanied by the change in concentration of  $H^+$  or  $OH^-$ , it is evident that hydrogen electrode may be employed in these titrations. The reference electrode used in this case is a Calomel electrode.

A known volume of the acid to be titrated is kept in a beaker having an automatic stirrer and a standard hydrogen electrode. It is connected to a calomel electrode through a salt bridge. The hydrogen and calomel electrodes are connected to a potentiometer. By adding a known volume of base for 10-12 times and E.M.F of the solution is recorded after each addition of the base from the burette into a beaker. The value of E.M.F are plotted against the volume of base added and is obtained as shown in the figure-1.

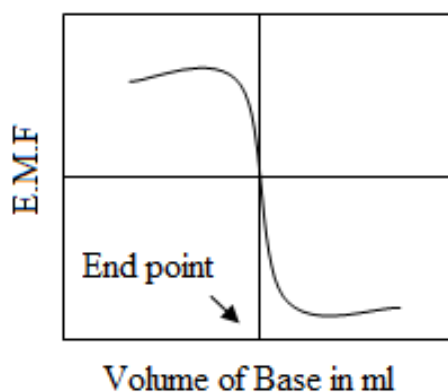


Figure-1

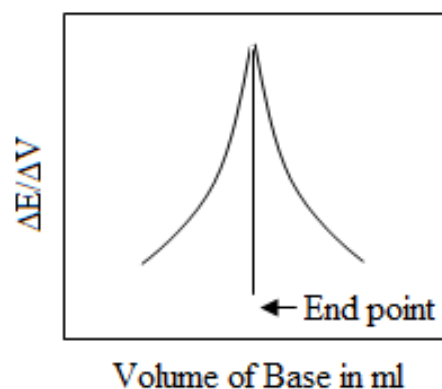


Figure-2

The point of inflexion in the curve (the point where the curve changes its curvature) gives the equivalent point when the titration curve does not exhibit a sharp inflexion point, it becomes to locate the exact point of inflexion. The more accurate method is the differential method where  $\Delta E/\Delta V$  i.e change in EMF resulting from the successive additions of the reagent is plotted against the volume of the reagent added. The maximum of the curve so obtained corresponds to the equivalent point of the titration.

**Cell representation:** (-) Pt / Hg<sub>(l)</sub>, Hg<sub>2</sub>Cl<sub>2</sub> (s) / KCl<sub>(sat)</sub> // H<sup>+</sup> (unknown)/ Pt (+)

**Procedure:** Titrate 20 ml of HCl with the alkali solution using phenolphthalein as the indicator so as to have an idea of correct distribution of readings to be made. Let the titer reading be 20 ml.

Calibrate the instrument before starting experiment. 20 ml of acid (HCl) solution into a 100 ml beaker and set up the following cell by combining Hydrogen and Calomel electrodes. Now the Calomel electrode and Hydrogen electrode dipped into the solution and the burette filled with the standard NaOH solution immediately above the beaker. Connect the Hydrogen and Calomel electrode to the potentiometer and measure the E.M.F. of the cell. In the neighbourhood of equivalent point, small additions of the base should be made.

Plot a graph between  $\Delta E/\Delta V$  values against the titer readings as abscissa. The maximum of the curve represents the equivalence point.

**Calculations:**  $M_{\text{NaOH}} \times V_{\text{NaOH}} = M_{\text{HCl}} \times V_{\text{HCl}}$







**CONDUCTOMETRIC TITRATION OF STRONG ACID Vs STRONG BASE**

Experiment No. ....

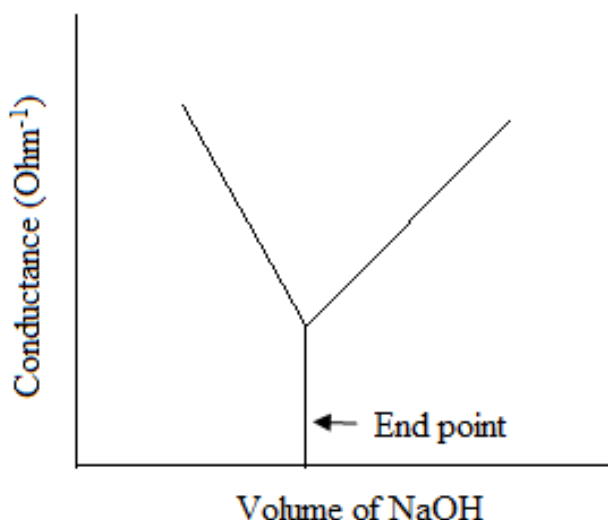
Date:.....

**Aim:** To estimate the amount of HCl present in the given volume of test solution using conductometer.

**Apparatus required:** Volumetric flask, Pipette, conical flask, 50 ml Burette, 100 ml Beaker, conductivity meter, conductivity cell.

**Reagents required:** 0.1M HCl, 0.1M KCl, 1M NaOH

**Theory:** An acid-base conductometric titration using HCl and NaOH can be performed using a conductivity cell and the change of conductance followed as the base is added. The behavior of this reaction depends on high ionic conductance of  $H^+$  and  $OH^-$  ions compared with salt ions of  $Na^+$  and  $Cl^-$ . Initially the  $H^+$  concentration is large and as the titration proceeds, the concentrations of  $H^+$  decreases up to the equivalence point and is replaced by less conducting  $Na^+$  ions. At the equivalence point, for strong acid and strong base the concentration of both  $H^+$  and  $OH^-$  ions are only  $10^{-7}$ , the conductance is due to the  $Na^+$  and  $Cl^-$  ions. As further base is added, the  $OH^-$  concentration builds up and conductance increases. The equivalence point (point of neutralization) can be conveniently taken as the intersection of the two straight lines that can



be drawn. Minimum value of the conductance in the graph will correspond to the equivalence point.



### Procedure

1. Standardize the conductivity meter using 0.1N KCl solution at the room temperature.
2. Rinse a clean 100 ml beaker with HCL test solution and pipette out 20 ml of the HCl solution in it. Dip the conductivity cell in it.
3. Connect the conductivity cell to the conductivity meter and measure the conductance.
4. Add NaOH solution from the burette to the HCl test solution in the beaker in 1.0 ml . increments. After the addition of each increment of NaOH gently mix the solution with a glass rod and measure its conductance value and note down the same. As NaOH is gradually added  $\text{H}^+$  of acid combine with  $\text{OH}^-$  of base and will form unionized water where as the concentration of slow moving  $\text{Na}^+$  ions will increase. On adding more NaOH , the conductance will go down decreasing until the whole acid has been neutralized by the base. Further addition of NaOH beyond the equivalence point will increase conductance due to free and fast moving  $\text{OH}^-$  ions.
5. Plot a graph between volume of NaOH added (in ml) on X-axis against conductance ( $\text{Ohm}^{-1}$ ) on Y- axis. The intersection of both the straight lines is the equivalence point ( $V_2$ ) of the titration.

**Observations**

S.No	Vol. of NaOH (ml)	Conductance ( $\text{ohm}^{-1}$ )

**Result:** The amount of hydrochloric acid present in the given test solution found to be ..... g/100ml.



**DETERMINATION OF VISCOSITY OF LUBRICANTS BY REDWOOD VISCOMETER**

Experiment No. ....

Date:.....

**Aim:** To determine the viscosity of lubricant oil by means of Redwood viscometer.

**Apparatus required:** Redwood viscometer, Stop watch.

**Reagents:** Lubricating Oil

**Theory:** The absolute viscosity of a oil can be determined by measuring the rate of flow of oil through a capillary tube kept at a uniform temperature. But in case of lubricating oil specific viscosity is generally determined by measuring the time taken for a given quantity of oil to flow through an orifice or jet of standard dimension under standard conditions.

The redwood apparatus measures viscosity of Redwood viscometers (i.e) Red wood No.1 & No.2. The Red wood No.1 will correctly indicate the viscosity for a liquid having flow time between 30 sec. to 2000 sec. If the flow time measured with this apparatus for any oil exceeds 2000 seconds, the test should be repeated with Redwood viscometer No.2. The two viscometers RW-1 & RW-2 are identical in principle, shape and mode of testing. They differ only in dimensions of orifice.

Viscometer	Diameter	Length of the jet
RW- 1	1.62 mm	10mm
RW-2	3.8 mm	50mm

RW-1 is commonly used for light or thin oil (kerosene, mustard oil etc)

RW-2 is used for highly viscous oils (fuel oils, mobile oil, glycerol etc.)

**Description of viscometer:** The Redwood viscometer consists of the standard oil cup which is opened at the upper end. It is fitted with an agate jet at the base. The diameter of orifice is different for Redwood viscometer 1 & 2. The jet can be kept closed or opened by a ball valve which permits the flow of oil being stopped or started. The cup is provided with a pointer which indicates the level

up to which the oil should be filled in the cup. The lid of the cup is provided with an arrangement to fix a thermometer to indicate the oil temperature. The oil cup is surrounded by a cylindrical copper vessel containing water which serves as a water bath for maintaining the desired oil temperature with the help of electrical heating coils. A stirrer with four blades is provided in the water bath to maintain uniform temperature in the bath. A thermometer is also provided in the bath to know the temperature of water. A spirit level used for leveling the apparatus and a 50ml flask for receiving the oil from the jet outlet are also provided.

**Procedure:** The viscometer cup should be cleaned with the help of a suitable solvent like  $\text{CCl}_4$ , ether, Benzene or petroleum spirit and properly dry to remove any trace of the solvent. The instrument should be leveled with the help of the leveling screws on the tripod. The water bath is filled with water for determining viscosity at  $80^\circ\text{C}$  and below. For higher temperature the bath should be filled with an oil having suitable viscosity at the test temperature. A brass ball is kept in position to seal the orifice. The oil under test should be carefully poured into the oil cup up to the tip of the indicator. 50 ml standard flask is kept in position below the jet. Now a thermometer is inserted and a stirrer is covered with a lid. By stirring the water in the bath and oil in the oil cup the temperatures are adjusted until the oil attains the desired constant temperature. When the temperature of oil has become quite steady in the oil cup and shows a constant reading lift the ball valves and simultaneously start the stop watch, allow the oil to fill in the flask upto 50 ml mark. Stop the stop watch and note down the time in seconds. Replace the ball valve in position to seal the cup to prevent over flow of the oil. Refill the oil up to the indicator tip of the oil. Repeat the experiment to get nearly reproducible results. Report the mean value as redwood 1 viscosity at  $1^\circ\text{C} = t$  seconds. This is the viscosity at room temperature  $T^\circ\text{C}$ . Repeat the experiment at five elevated temperature, say  $45^\circ\text{C}$ ,  $55^\circ\text{C}$ ,  $65^\circ\text{C}$ ,  $75^\circ\text{C}$  &  $85^\circ\text{C}$ .

**Observations**

S.No.	Water Temp.	Oil Temp.	Time (sec)	KV in stokes	AV in stokes	Density ( $\rho$ )
1.						
2.						
3.						
4.						

**Calculations**

$$V = At - B/t$$

Where, V = Kinematic viscosity of the oil in centistokes.

t = Time of flow in seconds.

A & B are instrument constants.

The value of

(A) = 0.264 and (B) = 190, when t = 40 to 85 sec

(A) = 0.247 and (B) = 65, when t = 85 to 2000sec

Unit conversions: 100 centistokes = 1 stoke

Stoke X density = poise (Absolute density)

1/poise = centipoises.

$$\text{Density}(\rho) = \frac{\text{Oil weight}}{\text{Volume}} \text{ or } \frac{\text{Massweight}}{\text{Volume}}$$

Weight of empty flask = .....gm

Weight of flask with oil = .....gm

**Result:** The viscosity of an oil sample with the help of redwood viscometer at different temperatures are tabulated.





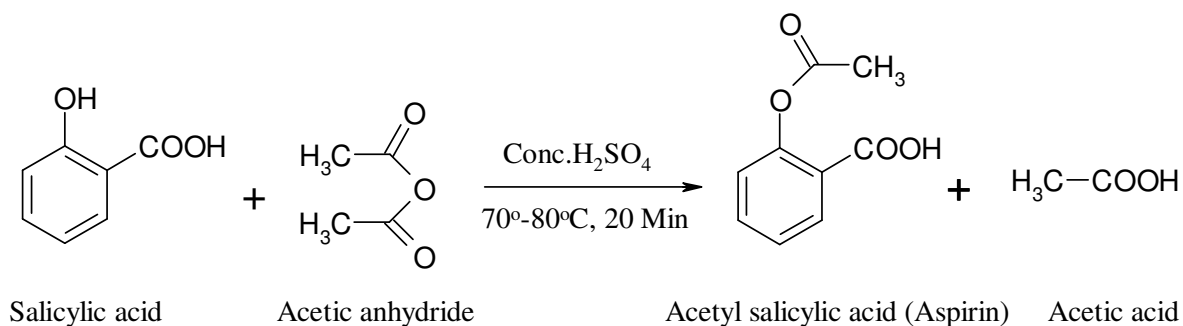
**PREPARATION OF ASPIRIN**

Experiment No. ....

Date:.....

**Aim:** To prepare the sample of aspirin by acetylation reaction method.**Reagents required:** Salicylic acid, Acetic anhydride, Conc. H<sub>2</sub>SO<sub>4</sub>, Ethanol.**Apparatus required:** Dropper, 250 ml Beaker, 10 ml Measuring cylinder, Filter paper, Buchner Funnel, Electric water bath, Hot air oven.

**Theory:** Aspirin (acetyl salicylic acid) is a weak monoprotic acid which was synthesized in 1893 by Felix Hofmann. This compound had the medicinal properties of salicylic acid, an extract of willow bark. Aspirin contains the functional groups of an organic ester and carboxylic acid. It is used extensively in medicine as a pain killer (analgesic) and as a fever reducing drug (antipyretic). It is prepared by acetylation reaction which proceeds by an electrophilic substitution mechanism. Aspirin (molar mass = 180.2g/mol) is prepared by reacting salicylic acid (molar mass = 138.1g/mol) with acetic anhydride (molar mass = 102.1 g/mol).



**Procedure:** Weigh out 2.0g of salicylic acid and transfer it into a clean 250 ml beaker. Add 5 ml of acetic anhydride followed by five drops of Conc. H<sub>2</sub>SO<sub>4</sub> to the mixture. Shake the solution well and the temperature of the reaction mixture rises to 70-80°C. Maintain the temperature of the reaction mixture at 70-80°C for about 20 minutes by heating on a water bath. Allow the solution to cool to room temperature. Add 100 ml of cold water to the reaction mixture with stirring. Scratch the side

walls of the beaker with glass rod to induce crystallization and stir the solution for complete precipitation. Filter the solution on a buchner funnel and wash with 10 ml of acetone. Dry the sample in a hot air oven and weigh the sample.

**Result:** Weight of the Aspirin sample is ..... gm

## PREPARATION OF THIOKOL RUBBER

Experiment No. ....

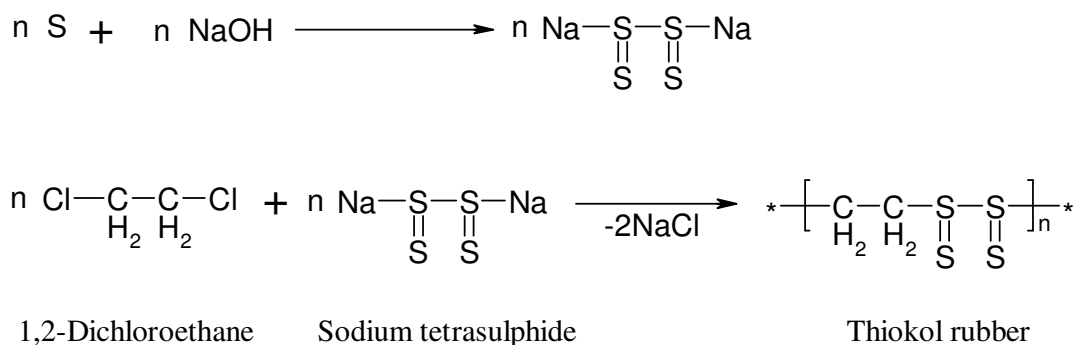
Date:.....

**Aim:** To prepare the sample of thiokol rubber by co-polymerization.

**Apparatus required:** 100 ml Breaker, 100 ml Measuring cylinder, glass rod.

**Reagents required:** 1,2-Dichloro ethane (ethylene dichloride), Sodium hydroxide, Powdered sulfur.

**Theory:** Thiokol is an elastomer which is obtained by co-polymerization of 1,2-dichloroethane and sodium tetra sulphide. This rubber cannot undergo vulcanization and it cannot form a hard rubber. It possess extremely good resistance to mineral oils, fuels, oxygen, solvents, ozone, sunlight and possess low abrasion resistance.



**Procedure:** Dissolve 2g of NaOH in 50-60 ml of warm water taken in a 100 ml beaker. Boil the solution and add 4g of sulfur powder in lots with constant stirring till the yellow solution turns deep red color. Cool it to 60-70°C and add 10 ml of 1,2-dichloroethane with stirring. Stir the solution for another 20 min till the rubber polymer gets separated out as a lump. Eliminate the liquid from the beaker to obtain thiokol rubber. Wash it water under the tap and dry the sample.

**Result:** The yield of thiokol rubber obtained is .....gm.



**IDENTIFICATION OF FUNCTIONAL GROUPS PRESENT IN ORGANIC COMPOUNDS**

Experiment No. ....

Date:.....

**Aim:** To identify the functional groups in the given organic compound by qualitative analysis.**Apparatus required:** Test tubes, Test tube stand, Watch glass and spatula.**Reagents required:** 10% NaHCO<sub>3</sub>, 10% NaOH, 10% HCl, Conc. H<sub>2</sub>SO<sub>4</sub>, 2,4-DNP, Schiff's reagent, Tollens reagent, Fehling's reagent,  $\alpha$ -Naphthol and Benedict's reagent.**1. Physical characteristics**

- i. Physical state: Solid /liquid
- ii. Colour : Colored /colorless
- iii. Odour / Smell: Odour /odourless

**2. Flame test**

Experiment	Observation	Inference
Burn a small amount of the compound over the flame.	<ul style="list-style-type: none"> <li>➤ Sooty flame is observed.</li> <li>➤ Non sooty flame is observed.</li> <li>➤ Charred with sugar smell</li> </ul>	<ul style="list-style-type: none"> <li>➤ May be aromatic compound.</li> <li>➤ May be aliphatic compound.</li> <li>➤ May be carbohydrate.</li> </ul>

**3. Solubility Test**

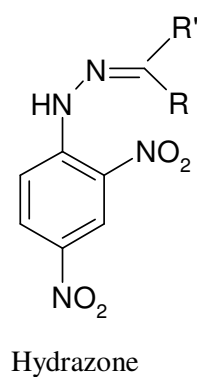
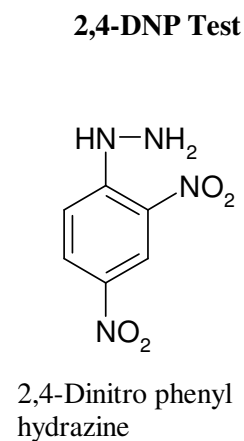
Test the solubility of the compound separately in water, ether, 10% aqueous sodium bicarbonate, 10% aqueous NaOH, Dil. HCl and Conc. H<sub>2</sub>SO<sub>4</sub>.

S. No	H <sub>2</sub> O	Ether	10NaHCO <sub>3</sub>	10%NaOH	10% HCl	Conc.H <sub>2</sub> SO <sub>4</sub>	Class of group
1.	-	+	-	-	-	+	May be carbonyl compound
2.	+	-	+	+	+	+	May be carbohydrate

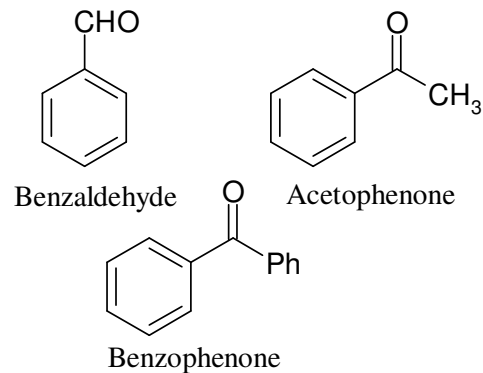
## I. Functional group analysis of carbonyl compounds

Sl.No	Experiment	Observation	Inference
1.	<b>2,4 –DNP test</b> : Add 1 ml of 2,4-DNP reagent to 2,3- drops of the compound and shake well.	An orange or yellow ppt. of 2,4-Dinitro phenyl hydrazone is formed.	May be carbonyl compound
2.	<b>Schiff's test</b> : To 1 ml of the given comp. add 5 ml of Schiff's reagent.	➤ A pink color is formed ➤ No reaction	➤ Aldehyde ➤ Ketone
3.	<b>Tollen's test</b> : To the 3 drops of the comp. add 5 ml of Tollens reagent and heat the solution.	➤ A silver mirror is formed ➤ No reaction	➤ Aldehyde ➤ Ketone
4.	<b>Fehling's test</b> : Take 1 ml of given compound and add few drops Fehling's reagent and boil for few minutes.	➤ Red color ppt is formed ➤ No reaction	➤ Aliphatic aldehyde ➤ Aromatic aldehyde

*Note:* If all the tests are negative except 2,4 – DNP test then compound may be ketone.



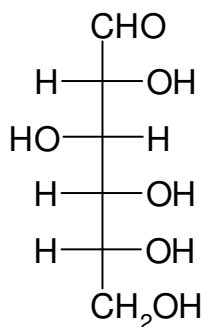
Examples of aldehydes and ketones



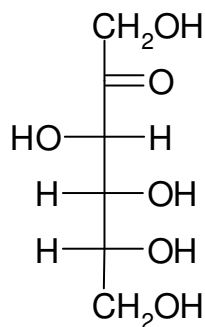
## II. Functional group analysis of carbohydrates

Sl.No	Experiment	Observation	Inference
1.	<b>Molisch test:</b> Dissolve small amount of the compound in 2 ml of water in a test tube and add 3-4 drops of 10% $\alpha$ -naphthol. Then add 2 ml of Conc. $H_2SO_4$ carefully from the sides of the test tube	A deep violet color is formed at the junction of two layers.	Carbohydrate is confirmed
2.	<b>Benedict test:</b> Take small amount of compound and add Benedict's reagent and boil for few minutes.	Yellow/brown/red ppt. is formed.	Carbohydrate is confirmed.

## Examples of carbohydrates

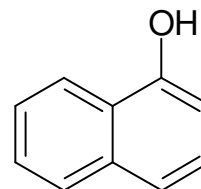


D-Glucose



D-Fructose

## Structure of alpha-Naphthol



**Result:** The given organic compound is a .....





## VIVA QUESTIONS

### 1. Estimation of hardness of water by EDTA method

- 1) What is the cause of hardness in water?
- 2) What is degree of hardness?
- 3) How does temporary hardness differ from permanent hardness?
- 4) What is the composition of Buffer solution?
- 5) Why buffer solution is added during the determination of hardness?
- 6) What is the indicator used in EDTA method?
- 7) Give the structure of EDTA?
- 8) How is temporary hardness removed?
- 9) Give the reaction for elimination of temporary hardness?
- 10) What is meant by softening of water?
- 11) Mention different units to express hardness of water and give their inter-relation?
- 12) Give the scheme for estimation of hardness of water by EDTA method?
- 13) Give the preparation of standard hard water?

### 2. Estimation of Manganese dioxide in pyrolusite

- 1) Give various industrial applications of  $\text{MnO}_2$ ?
- 2) What is pyrolusite?
- 3) How N/10  $\text{KMnO}_4$  solution will be prepared in the laboratory?
- 4) Give the reaction involved during the reaction of  $\text{MnO}_2$  with oxalic acid under acidic conditions?
- 5) What is a reducing agent?
- 6) What is the change in oxidation state of Mn in pyrolusite when it reacts with oxalic acid?

- 7) Give the structure of oxalic acid?

### **3. Estimation of percentage of copper in brass**

- 1) What is the composition of Brass?
- 2) What is the percentage of Cu in Brass?
- 3) What is an iodometric titration?
- 4) What is the chemical formula of starch?
- 5) Why should we use freshly prepared starch indicator?
- 6) What is hypo and give its formula?

### **4. Determination of ferrous iron in cement**

- 1) What is optical density?
- 2) What is photo colorimeter?
- 3) What is Beer's Lambert's law?
- 4) Give the reaction between  $\text{Fe}^{+3}$  and thiocyanate?

### **5. Titration of strong acid vs strong base by potentiometry**

- 1) What is an electro chemical cell?
- 2) In an electro chemical cell, which electrode is cathode and which electrode is anode?
- 3) What is redox reaction?
- 4) What is oxidation and reduction process?
- 5) Define electrode potential and standard electrode potential?
- 6) Define EMF of a cell?
- 7) What is a reference electrode? Give some examples?
- 8) Give the cell representation of HCl Vs NaOH by potentiometry?

- 9) Give the applications of potentiometer titrations?

### **6. Conductometric titration of strong acid vs strong base**

- 1) Define conductance and give its units?
- 2) Define specific conductance and give its units?
- 3) Define equivalent conductance and how it is effected by dilution?
- 4) Explain a saturated calomel electrode?
- 5) Define cell constant and give its units?
- 6) What is significance of conductometric titration?
- 7) Give the cell representation of HCl Vs NaOH by conductometric titration?

### **7. Determination of viscosity of lubricants by Redwood viscometer**

- 1) What are lubricants?
- 2) How does the viscosity of a liquid vary with rise in temperature?
- 3) What are units of viscosity?
- 4) What are different types of viscosities and give their formulae?
- 5) What are the various chemicals used to clean viscometer cup?

### **8. Preparation of Aspirin**

- 1) What is the chemical name of aspirin and give its structure?
- 2) Give the reaction for preparation of aspirin?
- 3) What is an acetylation reaction?
- 4) What are the functional groups present in aspirin?
- 5) What is the function of  $\text{H}_2\text{SO}_4$  in preparation of aspirin?
- 6) What is the mechanism followed for the preparation of aspirin?

**9. Preparation of thiokol rubber**

- 1) What is copolymerization?
- 2) What is vulcanization?
- 3) Why thiokol rubber does not undergo vulcanization?
- 4) Give the properties of thiokol rubber?
- 5) What is Thiokol rubber also called as?
- 6) Give the reaction for synthesis of thiokol rubber?

**10. Identification of functional groups present in organic compounds**

- 1) Name a few functional groups?
- 2) Name few carbohydrates and give their structures?
- 3) Name few compounds with carbonyl functional groups and give their structures?
- 4) Name the confirmatory tests for aldehyde functional group?
- 5) Name the confirmatory tests for carbohydrates?
- 6) What are aromatic & aliphatic hydrocarbons? How they can be identified?
- 7) Explain 2, 4-DNP test?
- 8) Explain Benedicts test, Fehlings test, Molish test, Tollens test, Schiffs test?
- 9) Explain the solubility test of aldehydes and carbohydrates?

