

# R. V. COLLEGE OF ENGINEERING

(Autonomous Institution affiliated to VTU, Belagavi)  
BENGALURU – 560 059



## Department of Chemistry

Engineering Chemistry (18CH12/22) Manual

## **LABORATORY SAFETY RULES AND GENERAL INSTRUCTIONS**

- Follow Dress Code**

Follow the dress code of the college. Wear white lab coat and shoes while in the laboratory.  
Take Proper care while performing the experiments.

- Don't Taste or Sniff Chemicals**

Don't smell or taste chemicals as they may harm you.  
Use fuming chemicals in the fuming hood.

- Don't Dispose the Chemicals Down the Drain**

Don't dispose all chemicals into the sink.  
Follow the directions of the instructor for chemicals which cannot be put into the sink.  
Flush the sink with water after completing the experiment.

- Don't Eat or Drink in Laboratory**

Don't eat or drink inside the laboratory.

- Collect the Data During Lab experiments**

Directly record the readings in the work book using pen and get them attested by the teacher

- Report all accidents and near-miss incidents to the instructor, immediately.**

To avoid accidents follow the guidelines below

1. Note the location of safety equipment in the lab (e.g. first aid kits, fire extinguishers).
2. Always read the upcoming experiments carefully and thoroughly. Understand the procedure, toxicity of the chemicals before entering the lab and follow the instructions.
3. Perform authorized experiments. Know what you are supposed to be doing and ask if you are not sure.
4. Keep your work area clean and notify the instructor if there is a spill or breakage. Clean up any spills as per the advice of the instructor.
5. Before you leave the lab, check that your work area is clean and that all utilities are shutoff.
6. Wash your hands with soap before you leave the laboratory.

## **GENERAL INSTRUCTIONS TO BE FOLLOWED WHILE CARRYING OUT VOLUMETRIC & INSTRUMENTAL METHODS OF ANALYSIS**

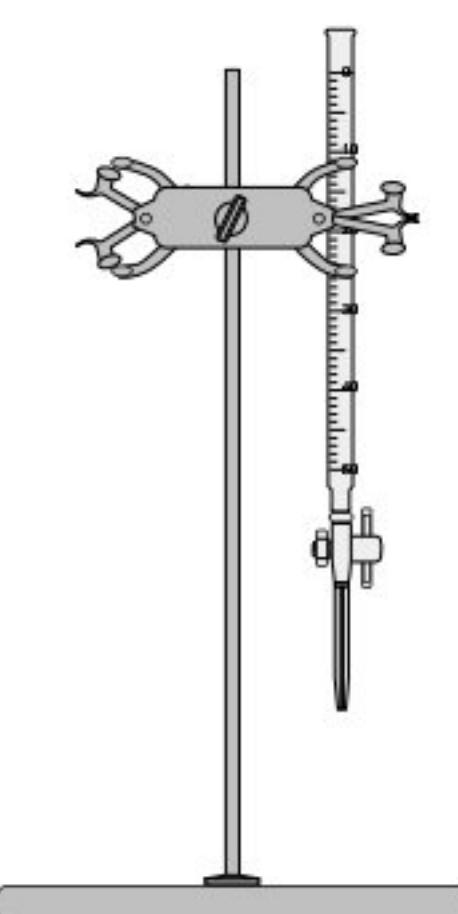
### **Pipette:**

1. Wash the Pipette minimum three times with tap water.
2. Rinse it once with small quantity of distilled water.
3. Now rinse the pipette with about  $10\text{ cm}^3$  of the solution to be pipetted out and reject the solution to the sink.
4. Draw the solution into the pipette a little above the mark by holding it almost vertically. Close the upper end of it using **INDEX FINGER** only. Adjust the lower meniscus of the liquid to the mark by rotating the pipette using other fingers and holding the pipette vertical.
5. While transferring the solution from the pipette to the conical flask, hold the pipette vertically till the solution drains out completely. Now touch the tip of the pipette 2-3 times gently to the empty bottom of the conical flask.
6. While transferring the solution do not blow air through the pipette.
7. Now rinse the pipette with about  $10\text{ cm}^3$  of the solution to be pipetted out and reject the solution to the sink.
8. Draw the solution into the pipette a little above the mark by holding it almost vertically. Close the upper end of it using **INDEX FINGER** only. Adjust the lower meniscus of the liquid to the mark by rotating the pipette using other fingers and holding the pipette vertical.
9. While transferring the solution from the pipette to the conical flask, hold the pipette vertically till the solution drains out completely. Now touch the tip of the pipette 2-3 times gently to the empty bottom of the conical flask.
10. While transferring the solution do not blow air through the pipette.



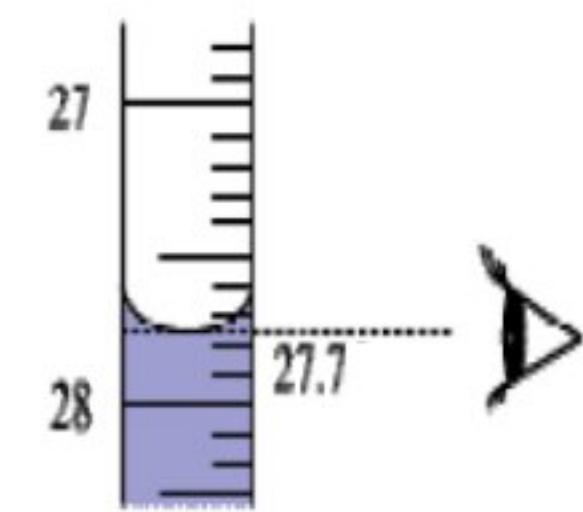
### **Burette:**

1. Wash the burette minimum three times with tap water.
2. Rinse it once with small quantity of distilled water.
3. Now rinse it once with about  $10\text{ cm}^3$  of the solution to be filled and reject the solution.
4. Fill the burette beyond zero mark using a funnel and adjust the



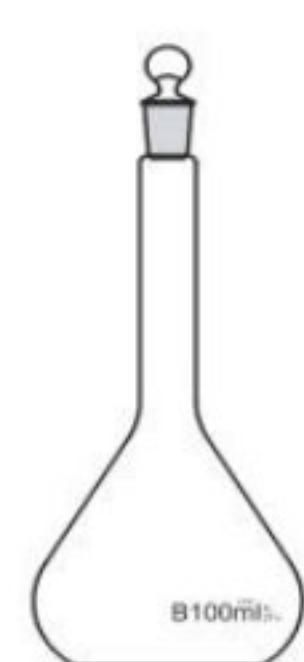
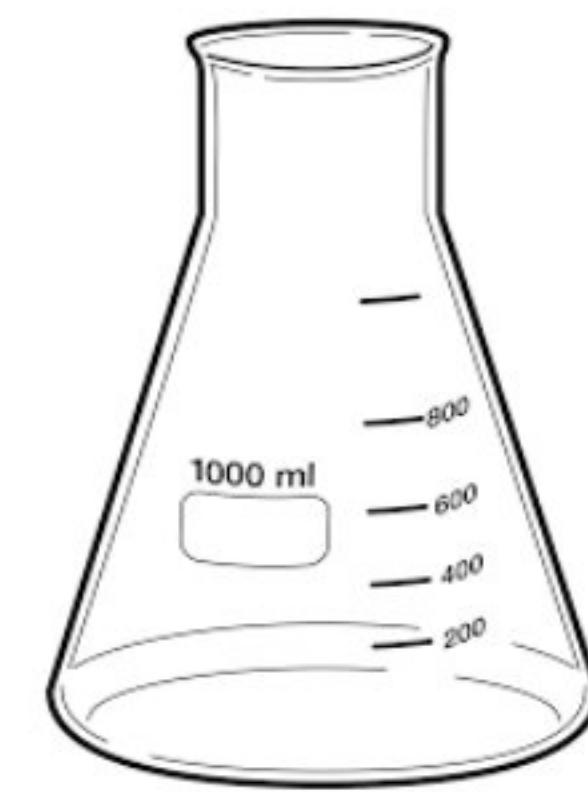
meniscus of the solution to zero mark by running down the solution through the nozzle (see that the nozzle part of the burette is completely filled with the solution)

5. Hold the burette vertically and the meniscus to your eye level, while reading the burette.



#### Conical Flask:

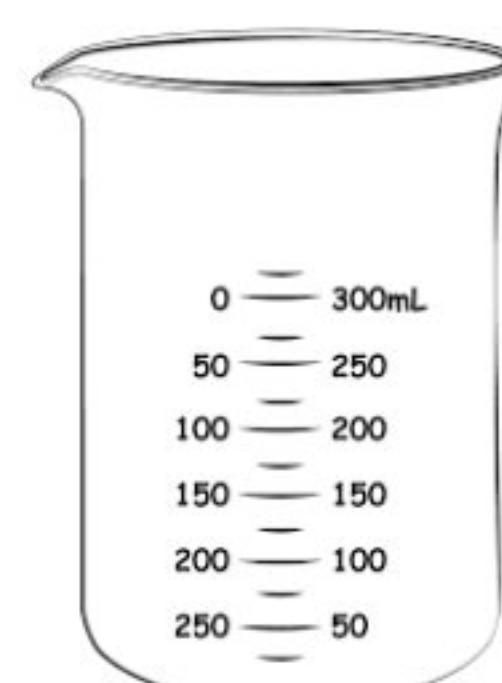
1. Wash the conical flask minimum three times with tap water.
2. Rinse it once with small quantity of distilled water.
3. Do not rinse it with any of the solutions.
4. Reject the contents of the conical flask to the sink after every trial and wash it as mentioned in 1 & 2 (under burette).



**Standard Flask or  
Volumetric Flask**



**Wash Bottle**



**Beaker**



**Measuring Cylinder**

#### Cleaning of apparatus:

- Clean the electrodes and other glassware after the experiment
- Keep the electrodes in distilled water after use.
- Do not spill any chemical or water on the instruments. If happens, immediately dry it using tissue paper.

## SOME TERMS USED IN VOLUMETRIC ANALYSIS

**Titrant:** The standard solution of accurately known strength.

**Titrate:** The solution containing the substance whose concentration/strength is to be determined.

**Equivalence point:** The exact or theoretical point at which the reaction is complete.

**End-point:** The point, at which a physical change is observed during titration.

**Indicator:** The substance used for visual detection (by colour change) of completion of reaction at the end-point.

**Types of Indicator:** The following types of indicators are used in volumetric analysis:

- **Acid-base Indicators:** Indicators which are sensitive to pH.
- **Redox Indicators:** Indicators whose oxidised form has a colour different from that of the reduced form.
- **Precipitation Indicators:** Indicators which indicates the endpoint by forming coloured precipitates.
- **Complexometric Indicators:** Indicators which undergo a change in colour on forming complex with metal ions.

**Strength of solution:** The concentration of solution or the amount of substance dissolved in 1 dm<sup>3</sup> of solution. It is usually expressed in terms of normality, molarity etc.

**Normality (N):** The number of gram equivalent weight of a substance dissolved in 1dm<sup>3</sup> of solution.

**Mass of the substance (in 1 dm<sup>3</sup>) = Normality X Gram equivalent weight of the substance**

**Molarity (M):** The number of moles of solute present in 1 dm<sup>3</sup> of solution.

**Mass of the substance (in 1 dm<sup>3</sup>) = Molarity X Gram equivalent weight of the substance**

**Molality** = Number of moles of solute present in 1 kg of a solvent.

**Parts per million (ppm):** It denotes amount of a substance per 1,000,000 or 10<sup>6</sup> parts of solution regardless of the units of measure used as long as they are same.

**Equivalent weight:** The number of parts by weight of the substance which combines with or displaces 1 part by weight of Hydrogen, 8 parts by weight of Oxygen or 35.5 parts by weight of Chlorine.

**Equivalent weight of an acid:** The ratio of molecular weight of acid to basicity of acid or the ratio of molecular weight of acid to the number of Hydrogen atoms replaced in one molecule of acid during the reaction.

**Equivalent weight of a base:** The ratio of molecular weight of base to acidity of base or the ratio of molecular weight of base to the number of OH<sup>-</sup> groups replaced in one molecule of base during the reaction.

**Equivalent weight of an oxidizing agent or reducing agent:** The ratio of molecular weight of the substance to the number of electrons gained or lost by one molecule during a redox reaction.

**Standard solution:** The solution whose concentration is known accurately.

### **Primary standard solution**

A substance which is available in highly pure state, neither hygroscopic nor efflorescent and is stable under the conditions of storage is called primary standard substance. A solution prepared using a primary standard is called primary standard solution.

Example: K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, Oxalic acid, AgNO<sub>3</sub>, KCl, anhydrous Sodium Carbonate, PHP, etc.

### **Secondary standard solution**

A substance which is not available in highly pure state, is hygroscopic or efflorescent and is not stable under the conditions of storage is called secondary standard substance. A solution prepared using a secondary standard is called secondary standard solution.

During the preparation of secondary standard solution an approximate weight of such a substance is dissolved in water and then the strength of this solution is determined by titrating against a primary standard solution this is known as standardization.

Example: NaOH, KMnO<sub>4</sub>, HCl etc.

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## VOLUMETRIC ANALYSIS & PREPARATION

### EXPERIMENT NO – 1

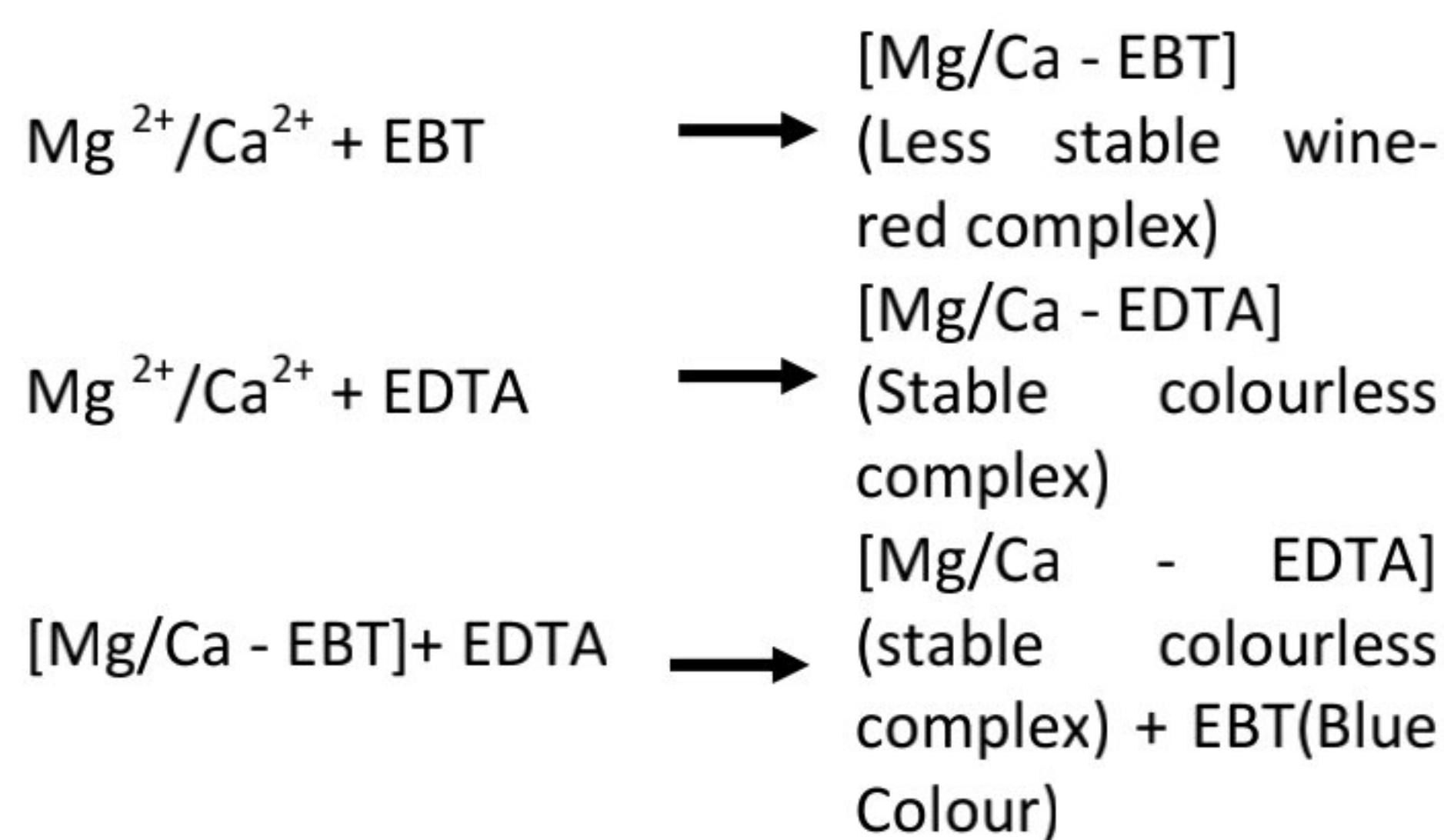
#### Aim:

Evaluation of quality of water in terms of total hardness by Complexometric titration

#### Theory:

Hardness of water is mainly due to the presence of Calcium and Magnesium salts in it. Total hardness is the sum of temporary hardness (due to Bicarbonates of Calcium and Magnesium) and permanent hardness (due to Chlorides, Sulphates etc., of Calcium and Magnesium). Ethylene diamine tetra Acetic acid (EDTA) is a reagent, which reacts with metal ions like  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  forming complexes. Therefore, this reagent can be used to determine the concentration of hardness causing substances. The amount of standard EDTA solution used during titration is a direct measure of the hardness.

The completion of the reaction (end point of the titration) is identified using Eriochrome black-T indicator. It is a blue coloured organic dye. It also forms relatively less stable complexes with bivalent metal ions of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  etc., which are wine red in colour. Therefore, addition of the indicator to hard water produces wine red colour. When EDTA is added to hard water, it first reacts with free metal ions and then attacks the metal ion-indicator complex. The reaction can be represented as



So, at the end point a change from **wine red to blue** colour is observed. Since the reaction involves the liberation of  $\text{H}^+$  ions and the indicator is sensitive to the concentration of  $\text{H}^+$  ions (pH) of the solution, a pH of around 10 has to be

maintained. For this purpose ammonia-ammonium Chloride buffer is used.

#### Procedure:

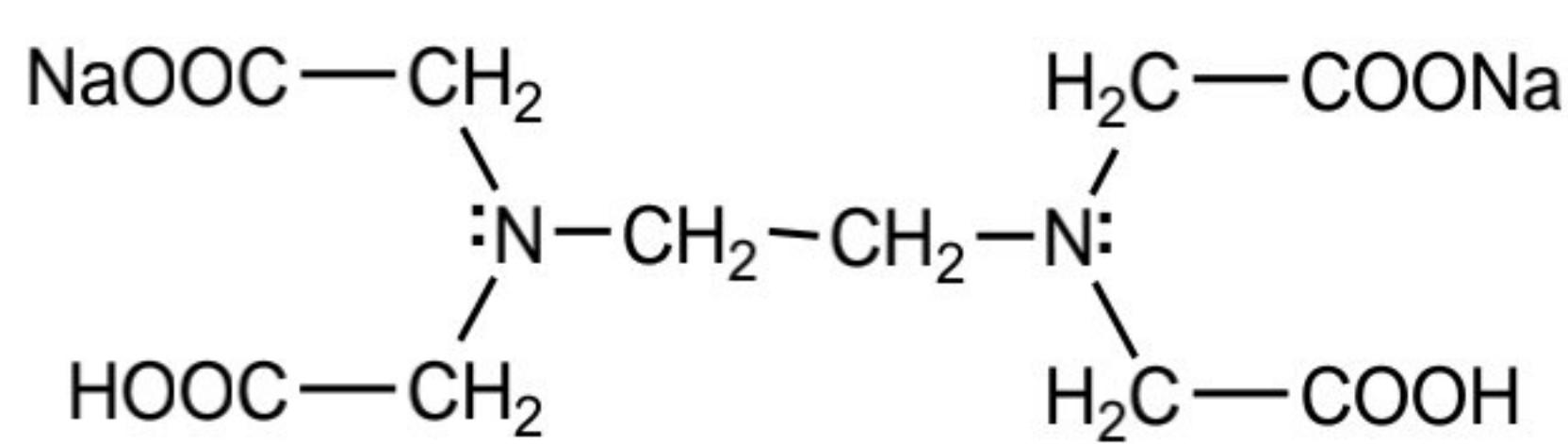
##### Part-A: Preparation of standard EDTA solution:

Weigh the Weighing Bottle containing disodium salt of EDTA (dissolves fast and completely in water) accurately and transfer the salt onto the funnel placed on a  $100 \text{ cm}^3$  volumetric flask. Weigh the Bottle again. The difference between the two weights will give the amount of EDTA transferred. Add about  $2 \text{ cm}^3$  of Ammonia followed by water. Dissolve the salt by adding about  $20-30 \text{ cm}^3$  of de-ionized water. Make up the solution to the mark and shake well for uniform concentration.

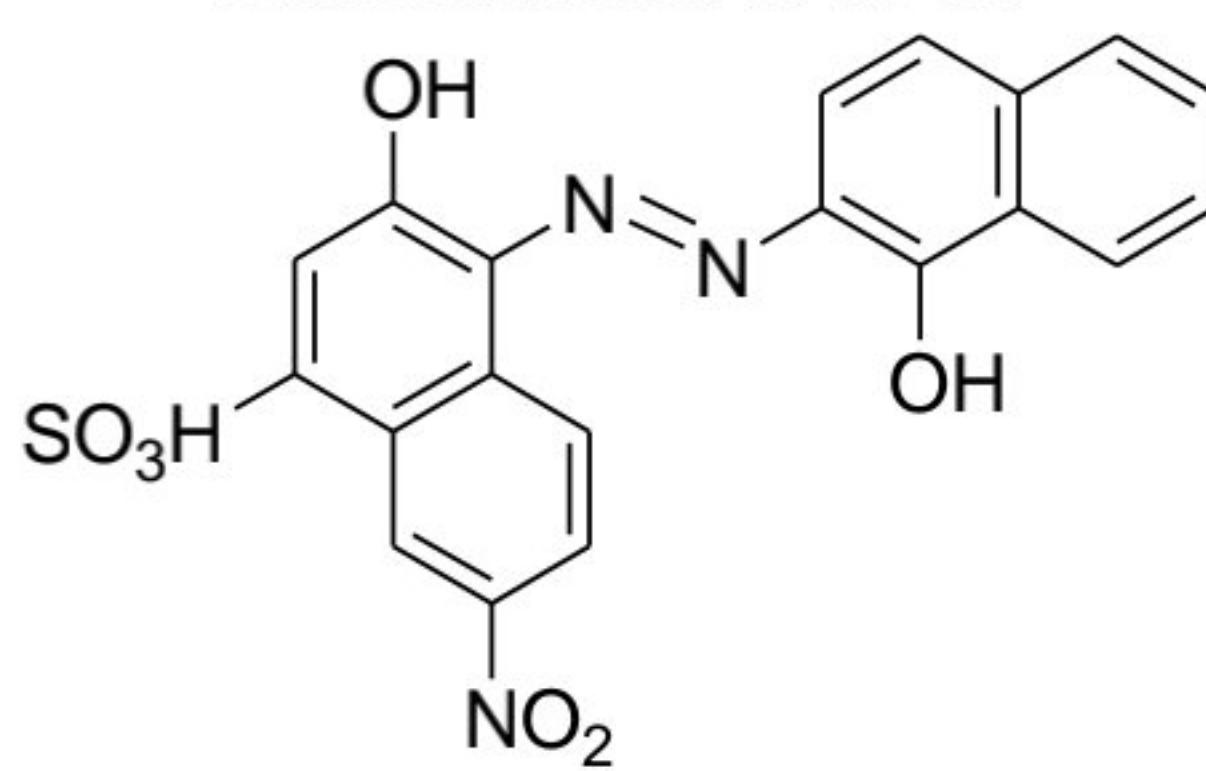
##### Part-B: Estimation of hardness of water:

Pipette out  $25 \text{ cm}^3$  of the given sample of hard water into a clean conical flask. Add  $3 \text{ cm}^3$   $\text{NH}_3\text{-NH}_4\text{Cl}$  buffer followed by a pinch of Eriochrome black-T indicator. Titrate this against EDTA taken in a burette till the colour changes from wine red to pure blue. Note down the burette reading and repeat the titration to get concordant values.

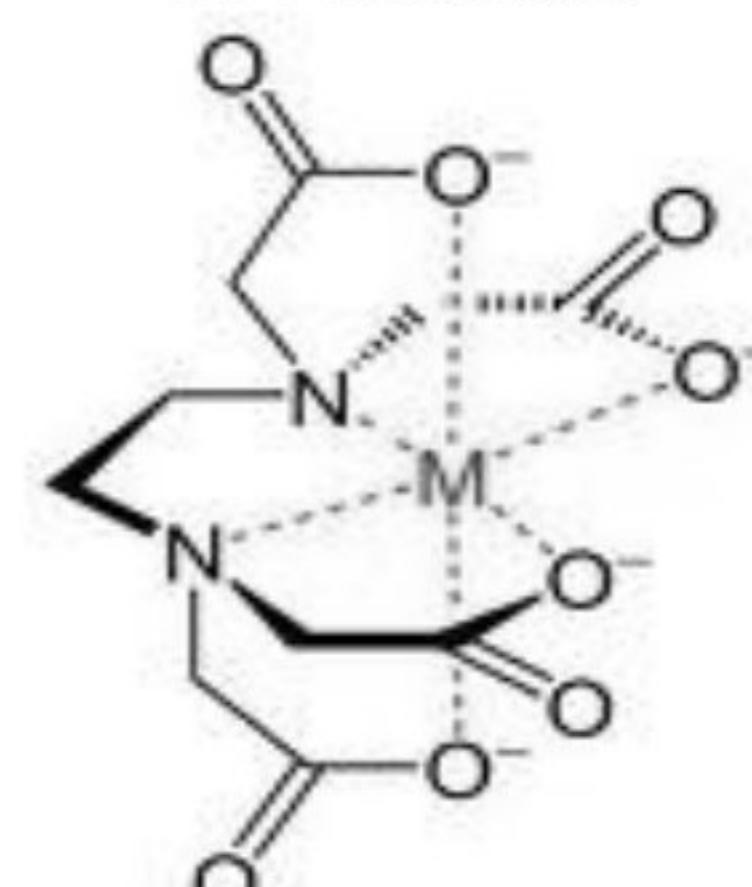
#### Structure of compounds:



Disodium salt of EDTA



EBT indicator



Metal Complex of EDTA

## VIVA QUESTIONS

- 1 What is Hard water?
- 2 How are the waters classified based on the degree of Hardness?
- 3 How is Hardness of water caused?
- 4 How temporary hardness is removed?
- 5 How do you express the total Hardness of water?
- 6 What is EDTA?
- 7 Write the structural formula for EDTA.
- 8 Why is disodium salt of EDTA preferred to EDTA?
- 9 Why is Ammonia solution added while preparing EDTA solution?
- 10 2.0746 g of EDTA was dissolved in 100 cm<sup>3</sup> of water. What is the molar concentration of EDTA?
- 11 What is buffer solution?
- 12 Why is Ammonia-Ammonium Chloride buffer added?
- 13 What is the pH of the buffer used? How is it prepared?
- 14 Why is the indicator Eriochrome Black (EBT) shows wine red color at the beginning and blue color at the end?
- 15 Name a metal ion sensitive indicator.
- 16 Why is the titration involving EDTA carried out slowly towards the end point?
- 17 Why is the degree of Hardness expressed in terms of CaCO<sub>3</sub>?
- 18 What is the application of Hardness data in environmental engineering practice?
- 19 How do you explain the color change of solution at the end point of titration?

**Observation and Calculation:****Part-A: Preparation of standard EDTA solution**

Weight of the Weighing Bottle + EDTA	=	g
Weight of the Weighing Bottle	=	g
Weight of the EDTA salt transferred	=	g

$$\text{Molarity of EDTA solution} = \frac{\text{Weight of EDTA dissolved in } 100 \text{ cm}^3 \times 10}{\text{Gram molecular weight of EDTA salt (372.24)}}$$

=

**Part-B: Estimation of hardness of water**

Burette readings(in $\text{cm}^3$ )	Trial-I	Trial-II	Trial-III
Final			
Initial			
Volume of EDTA run down			

**Part-C: Calculation**

$$1000 \text{ cm}^3 \text{ of } 1\text{M EDTA} = 100 \text{ g of } \text{CaCO}_3$$

$$\text{Therefore 'V' cm}^3 \text{ of 'X' M EDTA} = \frac{100 \times V \times X}{1000} \text{ g of } \text{CaCO}_3$$

$$= Y \text{ grams of } \text{CaCO}_3$$

$$25 \text{ cm}^3 \text{ of hard water has hardness equivalent to 'Y' g of } \text{CaCO}_3$$

$$\text{Therefore } 10^6 \text{ cm}^3 \text{ of hard water has hardness equivalent to } \frac{Y \times 10^6}{25} \text{ g of } \text{CaCO}_3$$

$$\text{Report: Total hardness of water} = \text{ppm}$$

## EXPERIMENT NO – 2

### Aim:

**Estimation of percentage of Copper in Brass by Iodometric titration**

### Theory:

The chief constituents of Brass alloy are Copper and Zinc. It also contains small quantities of Tin, Lead and Iron. The percentage composition of a typical Brass is Copper: 50-90, Zinc: 20-40, Tin: 0-6, Lead: 0-2, Iron: 0-1.

A solution of Brass is made by dissolution of the sample in Nitric acid. Oxides of Nitrogen are destroyed by boiling with Urea. Excess of acid is neutralized by adding Ammonia. The solution is changed to weak acidic medium by adding Acetic acid. Potassium Iodide is added. Iodine is liberated by the cupric ions. Then the solution is titrated against Sodiumthiosulphate solution using starch as an indicator. The amount of Sodiumthiosulphate consumed is a measure of the amount of Copper present.



### Procedure:

#### Part-A: Preparation of Brass solution

Weigh exactly the given sample of Brass into a clean  $250 \text{ cm}^3$  conical flask. Add Conc. Nitric acid drop by drop until the Brass dissolves completely. Add 2 tt of distilled water and about 1 g of Urea. Boil for about 1 minute to destroy oxides of Nitrogen. Cool the mixture.

#### Part-B: Estimation of Copper in Brass solution

Add 1 tt of distilled water to the solution obtained in part-A. Add Ammonium Hydroxide drop by drop until a pale blue precipitate is obtained. Dissolve the precipitate by adding dil. Acetic acid drop by drop, followed by  $\frac{1}{4}$  tt of Acetic acid and 1 tt of 10% KI solution. Add a test tube of distilled water. Swirl the conical flask thoroughly for about 30 seconds. Titrate the liberated iodine against standard Sodiumthiosulphate solution taken in the burette until the solution becomes pale yellow or cream colored. Add about  $2 \text{ cm}^3$  of freshly prepared starch solution as an indicator. Continue the titration by adding Sodiumthiosulphate solution **strictly drop by drop** until the dark blue coloration disappears, leaving behind a white ppt. Repeat Part-A and B to conduct a duplicate. Calculate the percentage of Copper present in the Brass samples.

## VIVA QUESTIONS

- 1 What are the constituents of Brass?
- 2 How is Brass solution prepared?
- 3 What is the purpose of adding Urea?
- 4 What are Iodometric titrations?
- 5 Why is Ammonium Hydroxide added to the Brass solution?
- 6 What is the bluish white precipitate formed after adding Ammonia solution?
- 7 Why is the solution acidified with Acetic acid after the addition of Ammonium Hydroxide?
- 8 Why is Acetic acid added?
- 9 How is liberated Iodine estimated?
- 10 What is the reaction that occurs between iodine and Sodiumthiosulphate?
- 11 Write the reaction between acidified Brass solution and Potassium Iodide.
- 12 Why is starch indicator added towards the end point?
- 13 What is the white precipitate produced at the end point?

### Observation and Calculation:

#### Part-A: Preparation of Brass solution

	Brass sample- 1	Brass sample –2
Weight of Brass + Weighing Bottle	g	g
Weight of empty Weighing Bottle	g	g
Weight of the Brass taken	g	g

#### Part-B: Estimation of Copper in Brass solution

Burette readings(in cm <sup>3</sup> )	Trial-I	Trial-II
Final		
Initial		
Volume of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> run down		

#### Part-C: Calculation

Normality of sodium thiosulphate = 'Y'

Volume of sodium thiosulphate = 'X' cm<sup>3</sup>

1000 cm<sup>3</sup> 1N sodium thiosulphate = 63.54g of copper

$$\text{Therefore } 'X' \text{ cm}^3 \text{ of } 'Y' \text{ N sodium thiosulphate} = \frac{63.54 \times X \times Y}{1000} \text{ g of copper}$$

$$= 'A' \text{ g of Cu}$$

Weight of brass taken = 'W' g

'W' g of brass contains 'A' g of copper

$$\text{Therefore } 100 \text{ g brass contains } \frac{A \times 100}{W} \text{ g of copper}$$

$$= 'B' \text{ g Copper}$$

#### Report:

Trial No.	Weight of brass sample	Percentage of Copper in the given brass sample
1		
2		

## EXPERIMENT NO – 3

### Aim:

**Estimation of Iron in the given sample of Haematite ore solution by Redox titration**

### Theory:

Haematite is an important ore of Iron containing mainly  $\text{Fe}_2\text{O}_3$  and Silica. Estimation of Iron involves the dissolution of the ore in Hydrochloric acid, reducing the ferric ( $\text{Fe}^{3+}$ ) ions in the solution to ferrous ( $\text{Fe}^{2+}$ ) ions using a reducing agent such as Stannous Chloride and the estimation of Ferrous ions so obtained by titrating against an oxidizing agent like Potassium Dichromate. Potassium Ferricyanide which produces intense blue color with  $\text{Fe}^{2+}$  ions and no colour with  $\text{Fe}^{3+}$  ions is used as an external indicator

### Procedure:

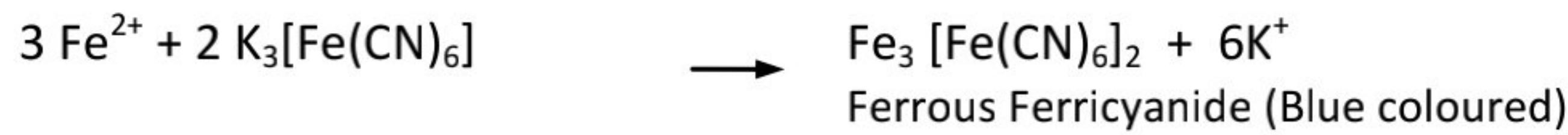
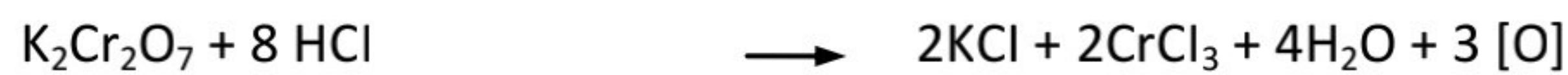
#### Part - A: Preparation of Potassium Dichromate solution

Weigh accurately the given Potassium Dichromate crystals and transfer onto the funnel placed on a  $100 \text{ cm}^3$  volumetric flask. Dissolve by adding deionised water and make it up to the mark. Shake well to get uniform concentration.

#### Part - B: Estimation of Iron

Pipette out  $25 \text{ cm}^3$  of the given Haematite solution into a clean conical flask. Add  $1/4$  test tube of concentrated Hydrochloric acid. Heat the solution nearly to boiling. Add Stannous Chloride drop by drop to the hot solution until the solution becomes colourless. Add 2-3 drops of Stannous Chloride in excess. Add 2 tt of demineralized water and cool the solution to room temperature, followed by  $3 \text{ cm}^3$  of Mercuric Chloride at a stretch. A silky white precipitate is formed. Reject the contents of the flask and repeat the experiment if no precipitate or greyish precipitate is formed.

Titrate the solution against standard Potassium Dichromate taken in the burette, using Potassium Ferricyanide as an external indicator. In the beginning, take out a drop of the reaction mixture using a clean glass rod and mix it with a drop of the indicator arranged on paraffined paper. The colour of the drop of indicator changes to blue. Take out a drop of the reaction mixture after every addition of  $\text{K}_2\text{Cr}_2\text{O}_7$  and mix it with a fresh drop of the indicator. Appearance of blue or green colour indicates that the end point is not reached. At the end point, a drop of the reaction mixture fails to give either blue or green colouration. Note down the burette reading and repeat the experiment for concordant values.



## VIVA QUESTIONS

- 1 What is the main constituents of Haematite ore?
- 2 In what form does Iron exist in Haematite?
- 3 What is the oxidation number of Iron in Haematite?
- 4 Give the others form of Iron ore.
- 5 What is the role of Stannous Chloride?
- 6 Why is Mercuric Chloride added and at a stretch?
- 7 What happens when the excess of Stannous Chloride is not removed?
- 8 What is the indicator used?
- 9 Why the color of the indicator drop remains the same at the end point?
- 10 Name any two oxidizing and reducing agents used in the experiment.
- 11 What is the reaction that occurs during the titration?
- 12 Write down the equations for the
  - a. Reactions between hot acidified ore solution and Stannous Chloride.
  - b. Addition of saturated mercuric Chloride to the solution in a.
  - c. Addition of Potassium Dichromate to the solution obtained in b.
- 13 Why is it necessary to add only very small excess of Stannous Chloride during the reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$ ?
- 14 Why grey ppt. is obtained some time?
- 15 Why Stannous Chloride is added while the solution is hot?

**Observation and Calculation:****Part-A: Preparation of Potassium Dichromate solution**

Weight of $K_2Cr_2O_7$ + Weighing Bottle	g
Weight of empty Weighing Bottle	g
Weight of the $K_2Cr_2O_7$ transferred	g

Normality of  $K_2Cr_2O_7$  =  $\frac{\text{Weight of } K_2Cr_2O_7 \text{ crystals dissolved in}}{\text{Gram equivalent weight of } K_2Cr_2O_7 \text{ (49.03)}}$

=

**Part-B: Estimation of Iron**

Burette readings(in cm <sup>3</sup> )	Trial-I	Trial-II	Trial-III
Final			
Initial			
Volume of $K_2Cr_2O_7$ run down			

**Part-C: Calculation**

1000 cm<sup>3</sup> of 1N  $K_2Cr_2O_7$  = 1 equivalent of iron  
= 55.85 g of Iron

Therefore 'Y' cm<sup>3</sup> of 'x' N  $K_2Cr_2O_7$  =  $\frac{55.85 \times X \times Y}{1000}$  g of iron = 'Z' g of iron.

25cm<sup>3</sup> of Haematite ore solution contains 'Z' g of iron

250cm<sup>3</sup> of Haematite ore solution contains '10xZ'g of iron

Weight of Haematite ore dissolved in 250cm<sup>3</sup> of the solution = 'W'g

'W' g of haematite ore contains '10 xZ' g of iron.

Therefore, 100g of haematite ore contains =  $\frac{10Z \times 100}{W}$  g of iron = 'B' g of iron

**Report: Percentage of iron in a given sample of haematite ore = B**

## **EXPERIMENT NO – 4**

### **Aim:**

**Determination of Chemical Oxygen Demand (COD) of the given industrial waste water sample**

**“COD is defined as the amount of Oxygen in mg, required by the waste materials (both organic and inorganic) present in 1 dm<sup>3</sup> of the given waste water sample for their oxidation using an oxidizing agent like acidified K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>”**

COD determination involves treating a known amount of waste water with a known excess of an oxidizing agent and finding the leftover amount of it after oxidation of impurities, by titrating against a standard reducing agent, using a red-ox indicator like Ferroin.

### **VIVA QUESTIONS**

- 1 What is chemical Oxygen demand?
- 2 What are the constituents which increase the COD of sewage?
- 3 What general group of organic compounds is not oxidized in the COD test?
- 4 What is the role of Silver Sulphate?
- 5 What is the role of Mercuric Sulphate?
- 6 Explain the role of acidified Potassium Dichromate.
- 7 What are the products formed after COD analysis?
- 8 Why is Sulphuric acid added during the preparation of standard FAS solution?
- 9 Why can't we use Nitric acid instead of dilute Sulfuric acid?
- 10 What is the composition of Ferroin?
- 11 What is the unit of COD?
- 12 Mention a few application of COD test in environmental engineering practice.
- 13 What is the limitation of COD?
- 14 What conclusion can be drawn from the COD measurement?

**Observation and Calculation:****Part-A: Preparation of FAS solution**

Weight of Weighing Bottle + FAS crystals	g
Weight of Weighing Bottle	g
Weight of the FAS crystals transferred	g

$$\text{Normality of FAS} = \frac{\text{Weight of FAS}}{\text{Gram mol wt of FAS}}$$

**Part – B: Back Titration**

Burette readings(in cm <sup>3</sup> )	Trial-I	Trial-II	Trial-III
Final			
Initial			
Volume of FAS run down			

**Part-C: Blank titration**

Burette readings(in cm <sup>3</sup> )	Trial-I	Trial-II	Trial-III
Final			
Initial			
Volume of FAS run down			

**Part-D: Calculation**Blank titre value = 'X' cm<sup>3</sup>Back titre value = 'Y' cm<sup>3</sup>Amount of dichromate (in terms of FAS) that has reacted with water sample = (X – Y) cm<sup>3</sup>.1000 cm<sup>3</sup> of 1N FAS solution = 1 equivalent of oxygen = 8 g of oxygen.

$$(X-Y) \text{ cm}^3 \text{ of 'Z' N FAS solution} = \frac{8(X-Y)xZ}{1000} \text{ g of oxygen} = 'A' \text{ g of oxygen}$$

25 cm<sup>3</sup> of waste water requires 'A' g of oxygen

$$\begin{aligned}\text{Therefore, } 1000 \text{ cm}^3 \text{ of waste water requires} &= \frac{AX1000}{25} \text{ g of oxygen} = 'B' \text{ g of oxygen} \\ &= 'C' \text{ mg of oxygen}\end{aligned}$$

**Report:** COD of the given sample of water = 'C' mg / dm<sup>3</sup>

## EXPERIMENT NO – 5

### Aim:

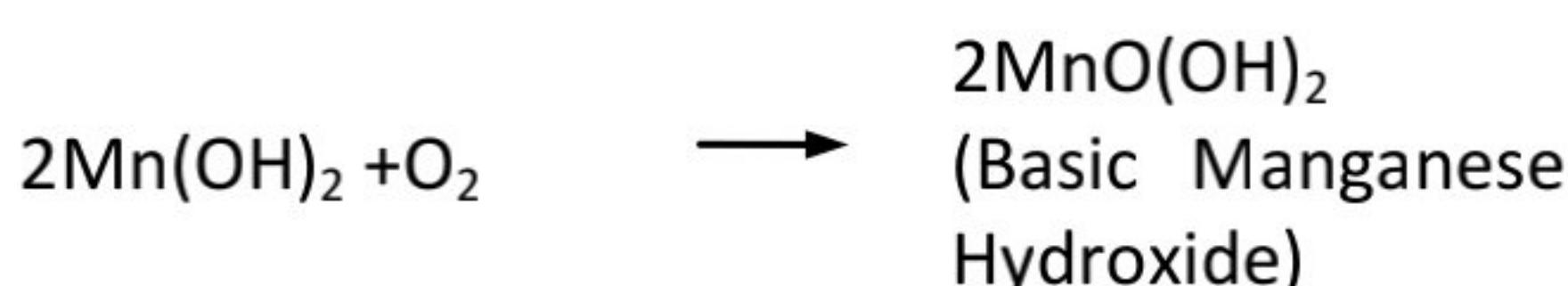
Determination of Dissolved Oxygen in the given water sample by Winkler's method

### Theory:

Dissolved Oxygen is needed for aquatic organisms for their survival. It is also important for precipitation and dissolution of inorganic substances in water. Dissolved Oxygen is an important factor in corrosion studies as Oxygen is responsible for corrosion at many places including boilers. DO test helps to decide the method (mechanical, physical or chemical) that can be used to control the amount of Oxygen in water for any specific purpose. This test helps to assess raw water quality and to keep a check on stream pollution. Dissolved Oxygen levels in any sample water would depend on the physical, chemical or biochemical activities taking place in it. DO test is the basis of B.O.D. The analysis for DO is a key activity in water pollution and waste water management..

Oxygen is poorly soluble in water. The solubility of Oxygen decreases with increase in concentration of salts. Under a pressure of one atmosphere, the solubility of Oxygen of air in distilled or fresh waters with low solid concentrations varies from 14.5 mg/lit. at 0 °c to about 7.5 mg/lit. at 30 °c. The solubility is less in saline waters and at a given temperature; it decreases with increase in the concentration of impurities.

The experiment is based on the principle of iodometric titration. Upon addition of Manganese Sulphate solution to water in alkaline condition, a precipitate of basic Manganese Hydroxide is formed as per the reaction below



Basic Manganese Hydroxide acts as an Oxygen carrier. In presence of Iodide and acid, the Manganese again reverts back to divalent state ( $\text{Mn}^{+2}$ ) oxidizing Iodide to Iodine. The amount of Iodine is equivalent to the dissolved Oxygen content.



The iodine liberated is then titrated against standard Sodiumthiosulphate. The sample of water may contain interfering species like Nitrites ( $\text{NO}_2^-$ ). To minimize the effect of nitrites, Sodium Azide is added. Sodium Azide converts nitrites into Nitrogen and Nitrous Oxide.

### Procedure:

#### Estimation of Dissolved Oxygen

Transfer 900 cm<sup>3</sup> of the water sample to BOD Bottle. Add about 6.0 cm<sup>3</sup> of alkali Iodide-Azide reagent by means of pipette (**Keep the tip of the pipette well below the surface of the solution**). Pipette out 6.0 cm<sup>3</sup> of 48%  $\text{MnSO}_4$  solution and add it to the BOD Bottle. Stopper the Bottle and remove the bubbles if formed and mix the contents of the Bottle by gently inverting it a few times. Allow the precipitate to settle down. Now add 6.0 cm<sup>3</sup> of conc.  $\text{H}_2\text{SO}_4$  and stopper the Bottle and invert it several times, till the precipitate dissolves completely. Iodine is liberated.

Pipette out 50 cm<sup>3</sup> of the reaction mixture into a conical flask. Add 2.0 cm<sup>3</sup> of freshly prepared starch indicator to it. Titrate the solution immediately against standardized Sodium-thiosulphate till the colour changes from blue to colourless.

## VIVA QUESTIONS

1. What is meant by Dissolved Oxygen?
2. What is the importance of dissolved Oxygen in water?
3. Differentiate between dissolved Oxygen and COD?
4. Explain the principle involved in the Winkler's method.
5. What are Iodometric titrations?
6. What is the function of Sodium Azide in the estimation of water?
7. Upon what factors does dissolution of Oxygen in water depend?
8. Write down the reaction taking place when
  - (i)  $\text{MnSO}_4$  and alkaline Iodide-Azide is added to water.
  - (ii) Conc.  $\text{H}_2\text{SO}_4$  is added to the above mixture
  - (iii) Iodine reacts with Sodiumthiosulphate.
9. How does the presence of  $\text{NO}_2^-$  affect the DO results?
10. What is the effect of oxidisable impurities on the dissolution of Oxygen?
11. When do you say the sample of water is polluted badly?
12. Justify:  $1000 \text{ cm}^3$  of 1N Sodiumthiosulphate is equivalent to 8 g of Oxygen.
13. How do you prepare  $2 \text{ dm}^3$  of 0.0246 N Sodiumthiosulphate?
14. The reaction is carried out in a BOD Bottle and not in a conical flask. Why?
15. Define BOD and COD of water

**Observation and Calculation:****Part-A: Estimation of Dissolved Oxygen**

Burette reading(in cm <sup>3</sup> )	Trial - I	Trial - II	Trial - III
Final reading			
Initial reading			
Volume of Sodiumthiosulphate rundown in cm <sup>3</sup>			

**Part-B: Calculation**Normality of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution = Y50 cm<sup>3</sup> of reaction mixture (49 cm<sup>3</sup> of water sample) requires X cm<sup>3</sup> of Y N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution.1000 cm<sup>3</sup> of 1.0 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> = 8.0 g of Oxygen1.0 cm<sup>3</sup> of 1.0 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> = 8.0 mg of OxygenX cm<sup>3</sup> of 'Y' N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> = 8.0 × X × Y = Z mg of oxygen49 cm<sup>3</sup> of water sample contains 'Z' mg of Oxygen1000 cm<sup>3</sup> of water sample contains  $\frac{Z \times 1000}{49}$  mg of oxygen = 'A' mg of Oxygen = A ppm**Report: Amount of dissolved oxygen in given sample of water = A ppm**

## **EXPERIMENT NO – 6**

### **Aim:**

**Preparation of Polystyrene by Bulk Polymerization method**

### **Theory:**

Suspension polymerization is a process in which a liquid monomer is suspended in water in the form of fine droplets. The droplets are stabilized by using suitable water soluble protective colloids, surface active agents and by stirring. The size of the monomer droplets formed depends on the monomer to water ratio, nature of stabilizing agents and also on the type and speed of agitation employed. An initiator soluble in monomer is added and polymerization is started. Polymerization occurring in the droplets changes them in to beads finally. Polystyrene can be prepared by suspension polymerization method, using monomer soluble initiator (Ex.DBP). Styrene monomer can be suspended by using water as media.

### **Chemicals Required:**

Styrene, Polyvinyl Alcohol (PVA-suspending agent), Benzoyl Peroxide (initiator), medium (water), 1 % NaOH solution.

### **Apparatus Required:**

100 ml three necked round-bottom flask, thermometer, dropping funnel, Nitrogen gas cylinder, hot plate cum magnetic stirrer and water cooled condenser.

### **Procedure:**

About 30 ml of styrene monomer is freed from stabilizer by washing three times with 10 ml of 1% NaOH solution followed by three washings (20ml each) with distilled water. The monomer is then dried over anhydrous  $\text{CaCl}_2$ .

A Mixture of 50 ml of distilled water and 1 g of PVA is taken in a 100 ml round bottom flask and placed on a hot plate at  $80\pm2$  °C. To the washed 10 ml of Styrene, 0.5 g of benzoyl peroxide (BPO) is added. The Styrene containing Benzoyl-Peroxide is added into the round bottom flask by means of a dropping funnel over a period of 5 hour. The prepared Polystyrene (PS) pearls or beads are filtered off, washed with 2% HCl and water, and then dried. Note down the appearance and yield of Polystyrene.

## **VIVA QUESTIONS**

1. Pearl or bead shaped products are obtained in Suspension Polymerization? Give reasons.
2. Explain Suspension Polymerization Process?
3. What type of initiators are used in Suspension Polymerization?
4. Give two advantages of Suspension Polymerization technique?
5. Give two uses of Polystyrene?
6. Give the names of two more initiators used in Polymerization process
7. Write the mechanism of Polymerization in the present case
8. What is a free radical and what is its use in Polymerization?

**Report:** Report the yield, colour and confirmation tests for Polystyrene.

## EXPERIMENT NO – 7

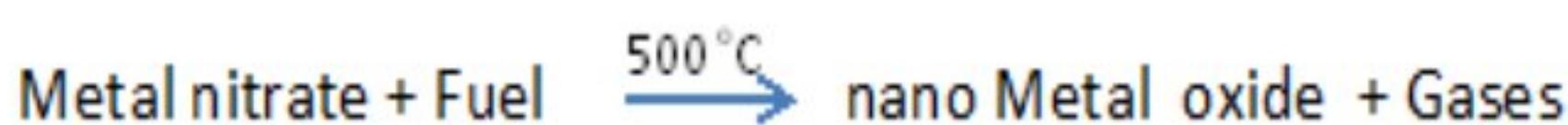
**Aim:**

**Preparation of ZnO by Solution Combustion method**

**Theory:**

Combustion method is a low temperature, time saving, energy efficient, self-propagating method, involving spontaneous exothermic redox reaction between metal nitrate and organic fuel, used to prepare nanomaterials.

**Principle:** The method is based on the utilization of heat energy produced during the exothermic spontaneous redox reaction between an oxidizer (metal nitrate) and a reducing agent (organic fuel). The oxidizer can be any metal nitrates and reducing agents may be organic fuels, such as Glycine, Oxalic acid, Urea, Hexamine, Sugar, EDTA, Dextrose etc.



The amount of fuel can be calculated in such way that the total oxidizing valency of fuel should match with the total reducing valency of metal nitrate.

**Chemicals Required:**

Zinc nitrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , (Mol. Wt=297.46) any organic fuel (say Urea;  $\text{NH}_2\text{CONH}_2$ ; Mol. Wt = 60) and distilled water.

**Apparatus Required:**

High Temperature Furnace, 250 ml Borosil glass beaker, Tong, Spatula, Weighing balance, Magnetic stirrer.

**Calculation of fuel:**

The valency of  $= 2(0) + 4(+1) + 1(+4) + 1(-2)$   
Urea ( $\text{N}_2\text{H}_4\text{CO}$ )  $= 6$

The valency of  $= 1(+2) + 2(0) + 6(-2) + 6(0)$   
 $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} = 10$

The amount of Urea for 5.0 g of  $\text{Zn}(\text{NO}_3)_2$  is calculated as follows.

$$\frac{\text{Wt of Urea}}{\text{Mol wt of Urea}} \times \text{Valency of Urea} = \frac{\text{Wt of MgNO}_3}{\text{Mol Wt of MgNO}_3} \times \text{Val of MgNO}_3$$

$$\text{Wt of Urea} = (5 \times 10 \times 60) / (297.46 \times 6) \text{ g} \\ = 1.68 \text{ g}$$

**Procedure:**

Weigh exactly 5.0 g of Magnesium Nitrate and 1.95 g of Urea (1:1 stoichiometry) in 250 ml beaker and dissolve the salts by adding minimum quantity of distilled water and stirring on a magnetic stirrer for 10 min. The uniformly dissolved redox mixture is kept in a pre-heated furnace maintained at  $500^\circ\text{C}$ . The redox solution undergoes evaporation and finally results in viscous-gel containing uniformly mixed Magnesium Nitrate and Urea. After some time it catches fire with the liberation of gases and heat energy, finally results in highly porous, white powder of nano MgO.

### VIVA QUESTIONS

- What is the formula for the calculation of amount of fuel?
- What is the principle behind Solution Combustion method?
- What are the advantages of Solution Combustion method?

**Report:** Report the yield, colour and confirmation tests for nano ZnO.

## INSTRUMENTAL METHODS OF ANALYSIS

### EXPERIMENT NO – 8

#### Aim:

Determination of pKa of a weak acid using pH meter

#### Theory:

The dissociation constant of a weak acid is denoted as  $K_a$  and it is given by Ostwald's dilution law.



$$K_a = \frac{[\text{H}^+][\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]}$$

The strength of the acid is indicated by its dissociation constant. Dissociation constant is also a measure of the tendency of an acid to donate proton. Generally,  $K_a$  value of weak acids are very small. In order to avoid negative power appearing as  $K_a$  value, a new term called  $\text{p}K_a$  value is coined. If  $K_a$  is the dissociation constant of a weak acid, then

$$\text{p}K_a = -\log_{10} K_a$$

The pH of a weak acid solution and  $\text{p}K_a$  are related as per Henderson - Hasselbalch equation:

$$\text{pH} = \text{p}K_a + \log_{10} \frac{[\text{salt}]}{[\text{acid}]} \dots\dots\dots (1)$$

Addition of a strong base (Sodium Hydroxide) to a weak acid (Acetic acid) produces salt (Sodium Acetate). At equivalence point (end point) all the acid is neutralized. At half equivalence point, half of the acid is neutralized, i.e., [salt formed] = [acid unreacted]. Therefore, equation (1) reduces to  $\text{pH} = \text{p}K_a$ . This indicates that at half equivalence point the  $\text{p}K_a$  of the weak acid is equal to pH. Therefore, **the  $\text{p}K_a$  of a weak acid can also be defined as the pH at half equivalence point.**

During an acid-base titration, initially the pH value of the solution increases gradually and increases more rapidly thereafter till the equivalence point is reached (Fig. 2). Once the equivalence point is crossed, pH increases by

small amounts. The variation of pH is measured using combined glass electrode

The experiment is divided into two parts.

- (1) Determination of end point or equivalent point (fig. 1) and
- (2) Determination of  $\text{p}K_a$  (fig. 2).

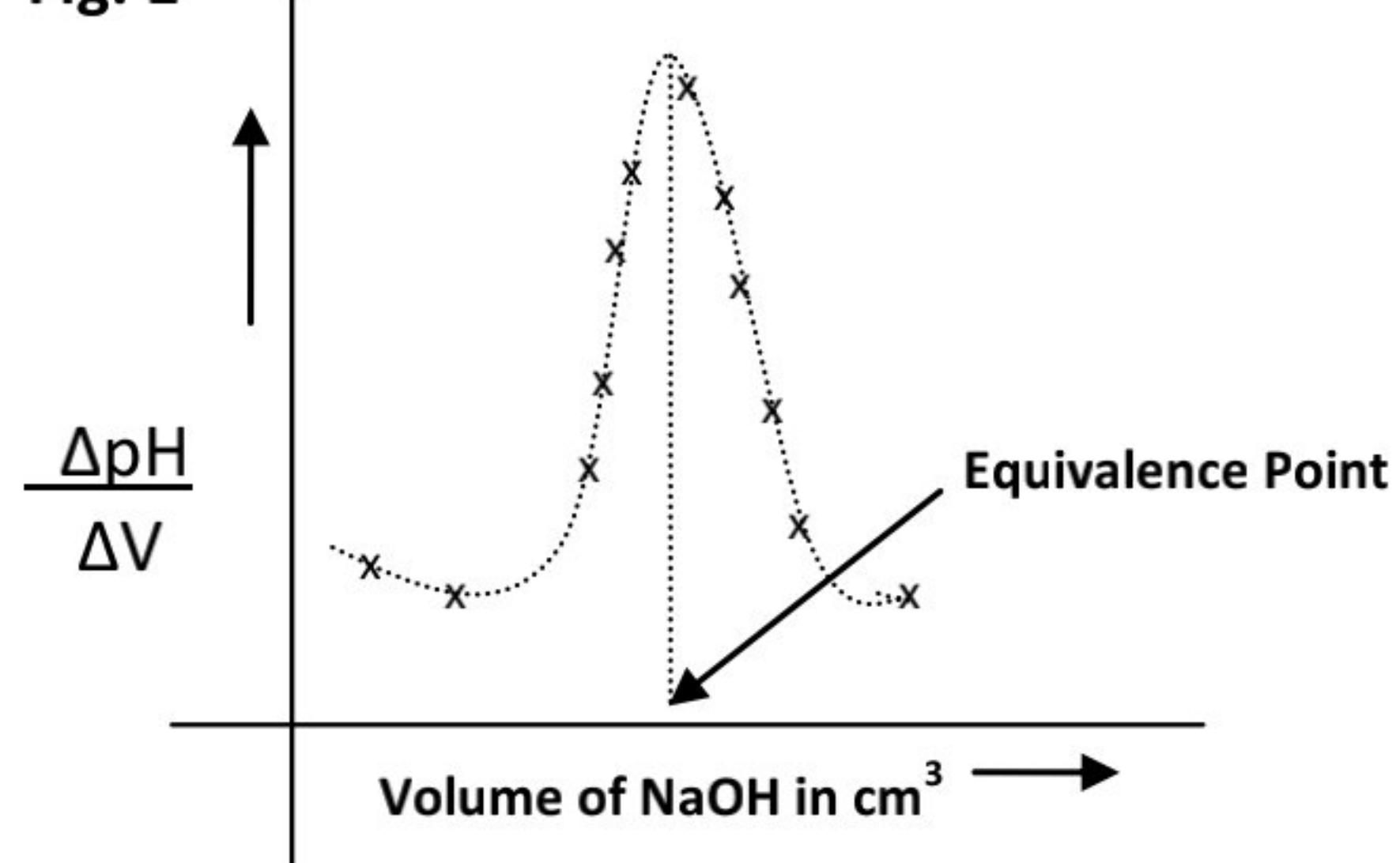
#### Procedure:

Pipette out  $50 \text{ cm}^3$  of the given weak acid (Acetic acid) into a clean  $250 \text{ cm}^3$  beaker. Insert glass electrode - calomel electrode assembly into it and connect it to pH meter. Measure the pH of the acid. Fill the burette with the given Sodium-Hydroxide. In the beginning add  $0.2 \text{ cm}^3$  of NaOH at a time and stir the mixture. Measure the pH. Continue adding  $0.2 \text{ cm}^3$  of NaOH & measure the pH regularly after each addition until the increase in pH is comparatively large. Take a few more reading by adding  $0.2 \text{ cm}^3$  of NaOH.

Determine the  $\text{p}K_a$  of the given weak acid from the two graphs:

- (i)  $\frac{\Delta \text{pH}}{\Delta V}$  against volume of NaOH added.
- (ii) pH against volume of NaOH added.

Fig. 1

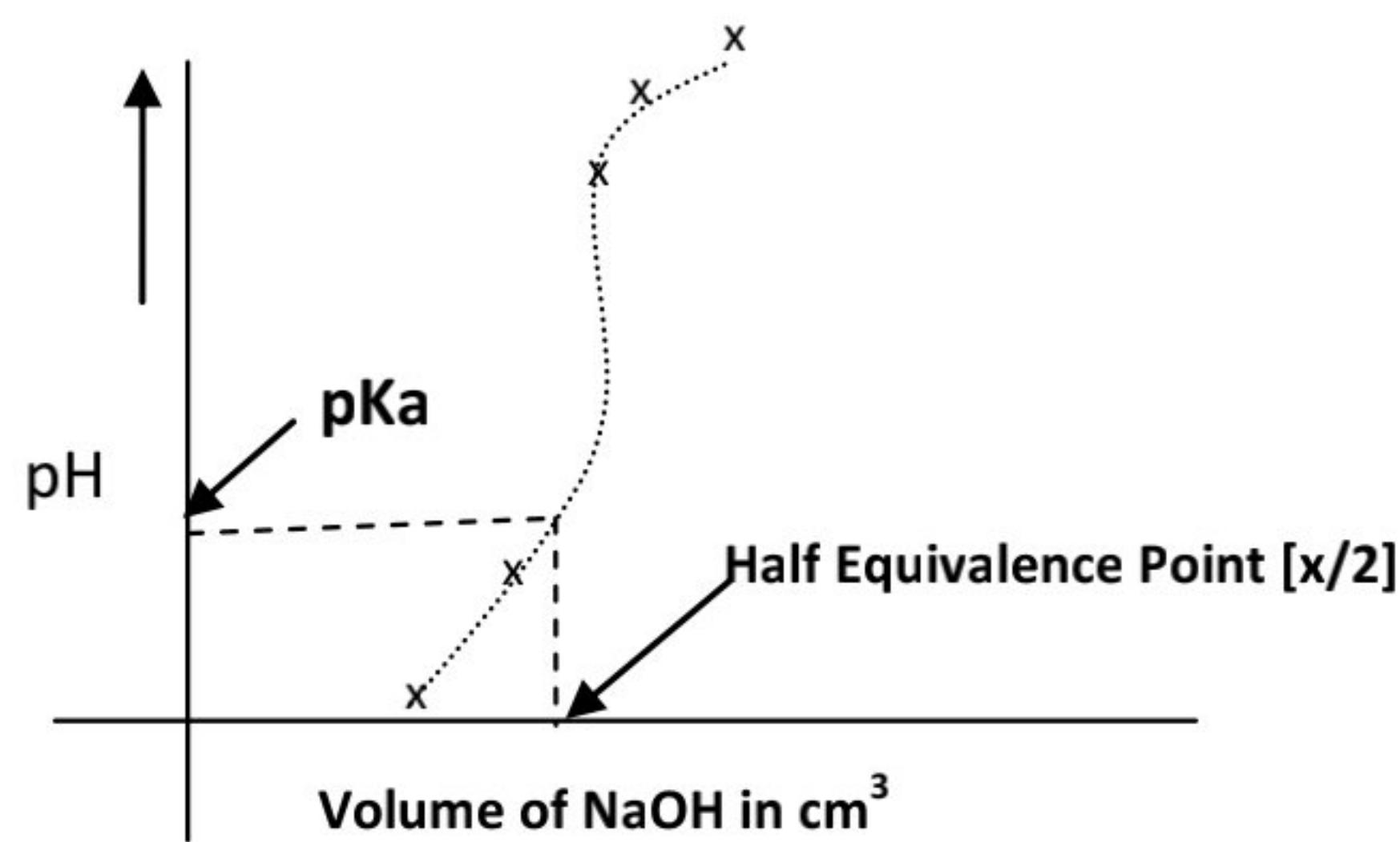


Equivalence point = 'x'  $\text{cm}^3$

Half Equivalence point = 'x/2'  $\text{cm}^3$

pH Corresponding to half equivalence point = 'y' =  $\text{p}K_a$

**Fig. 2**



### VIVA QUESTIONS

1. Define pKa of a weak acid.
2. pK<sub>a</sub> values of two acids are 2.8 and 3.2. Which one of these acids is stronger?
3. Why should we compute the pKa values of a weak acid?
4. Define pKa of a weak acid in terms of pH of the solution
5. Account for the following:
  - a) pH of the medium changes enormously at its equivalence point.
  - b) At half equivalence point, pH is equal to pKa.
6. Write down Henderson-Hasselbalch equation.
7. pKa values of Acetic acid and Formic acid are 4.7 and 3.7 respectively at a given temperature. Which one of the two acids is stronger?
8. Henderson-Hasselbalch equation holds good for buffer of weak acid and its salt with strong base. How do you justify the use of this equation in the experiment?
9. What is a buffer solution? Give any two examples.
10. What are the applications of Henderson-Hasselbalch equation?
11. Find out the ratio in which the acid and its salt are to be dissolved in 1 dm<sup>3</sup> such that the resultant buffer would offer a P<sup>H</sup> of 5.95. Given pKa = 4.0.
12. 1.0 mole of sodium acetate and 0.5 mole of Acetic acid were dissolved in 1 dm<sup>3</sup> of water. Calculate the pH of the buffer. Given, dissociation constant of Acetic acid at 298 K is 10<sup>-5</sup>
13. Why half equivalence point is taken into account for assigning pKa?

**Tabulation:**

Volume of NaOH added in cm <sup>3</sup>	pH	ΔpH	ΔV	$\frac{\Delta pH}{\Delta V}$
0.0				
0.2				
0.4				
0.6				
0.8				
1.0				
1.2				
1.4				
1.6				
1.8				
2.0				
2.2				
2.4				
2.6				
2.8				
3.0				
3.2				
3.4				
3.6				

**Report: pKa of the given weak acid =**

## EXPERIMENT NO– 9

**Aim:**

**Estimation of Iron present in 1750 cm<sup>3</sup> solution of FAS using Potentiometer. Given, the normality of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution is 0.0489N**

Nernst equation gives the relation between the potential at an electrode and the concentration of the active species in the solution.

For the reaction,



Nernst equation can be written as

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

Where E° is the standard electrode potential and the terms within the brackets represent their concentrations.

## VIVA QUESTIONS

- 1 What is a Potentiometer titration?
- 2 Give the principle of Potentiometer titration.
- 3 What are the electrodes used in Potentiometer titration?
- 4 What is determining factor in the oxidation-reduction reaction?
- 5 What is an indicator electrode?
- 6 What is the reaction that occurring between FAS and Potassium Dichromate?
- 7 What are the advantages of Potentiometric titration?

**Tabulation:**

Volume of $K_2Cr_2O_7$ added in $cm^3$	Potential (E) In mV	$\Delta E$	$\Delta V$	$\Delta E/\Delta V$
0.0				
0.2				
0.4				
0.6				
0.8				
1.0				
1.2				
1.4				
1.6				
1.8				
2.0				
2.2				
2.4				
2.6				
2.8				
3.0				
3.2				
3.4				
3.6				

**Calculation:**

Volume of  $K_2Cr_2O_7$  required for the reaction =  $cm^3$  (from graph)

$N_{FAS}$  =

Amount of Iron presented in  $1000\ cm^3$  of its solution =  $N_{FAS} \times$  Gram equivalent weight of Iron

=

**Report: Amount of Iron present in  $1000\ cm^3$  of its solution =**

## EXPERIMENT NO– 10

### Aim:

**Estimation of Copper present in a given Copper Sulphate solution, using Colorimeter. Amount of Copper Sulphate pentahydrate in 100 cm<sup>3</sup> of solution is 55 mg**

### Theory:

When a monochromatic light of intensity  $I_0$  is incident on a medium, a part of it may be absorbed ( $I_a$ ), a part of it may be transmitted ( $I_t$ ) and the remaining part may be reflected ( $I_r$ ).

$$\text{Thus, } I_0 = I_a + I_t + I_r$$

$I_r$  can conveniently ignore when the medium is transparent like perfect glass.

$$\text{Therefore, } I_0 = I_a + I_t$$

If  $I_t/I_0$  is called transmittance then  $\log(I_0/I_t)$  is called absorbance or optical density (A). The relation between absorbance, concentration of the medium and path length or thickness of the medium is referred as Beer-Lambert's law.

$$A = \epsilon Cl$$

Where 'A' is absorbance, 'c' is concentration, 'l' is thickness and ' $\epsilon$ ' is a constant called molar extinction coefficient.  $\epsilon$  is constant for a given substance.

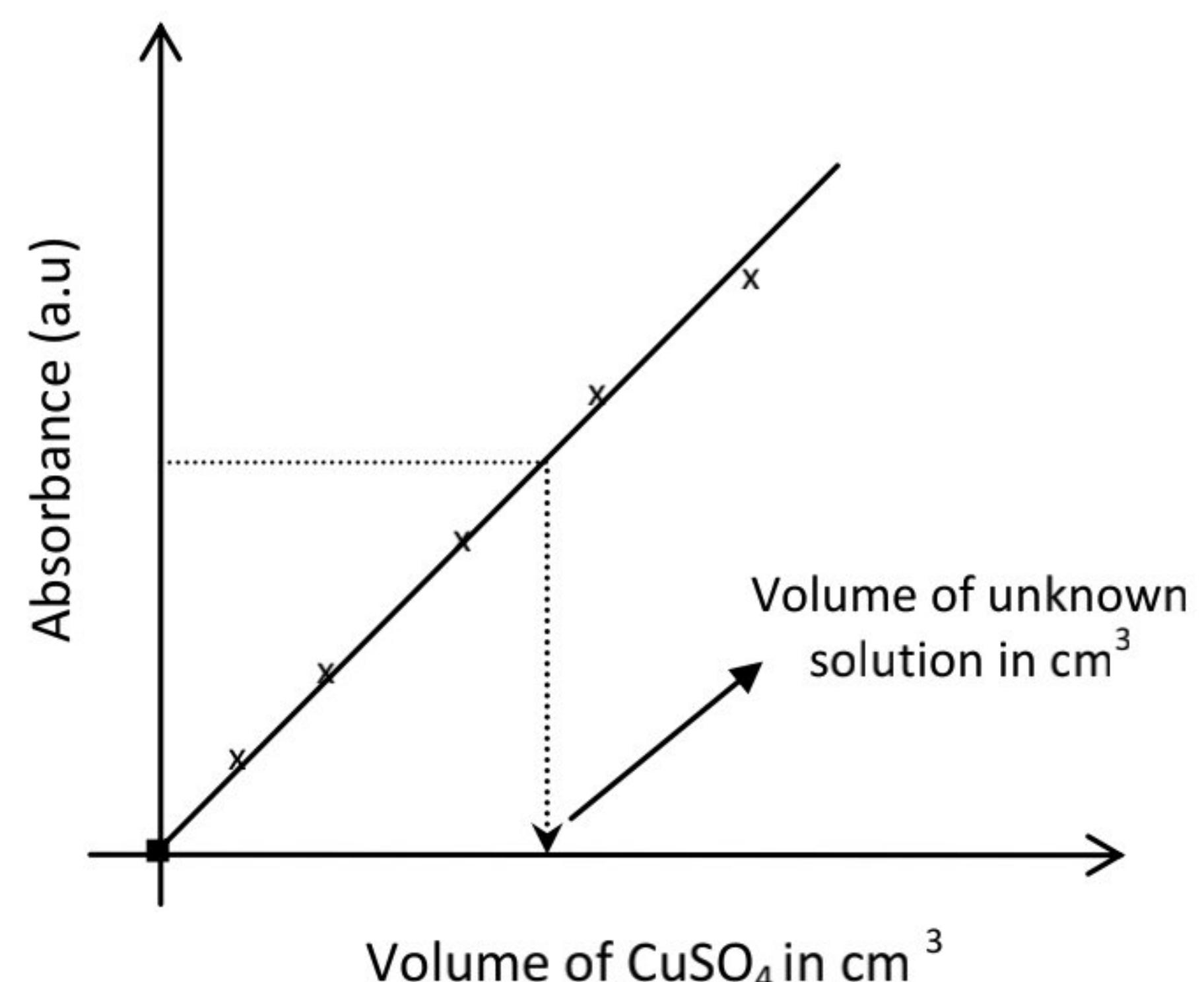
Chemical analysis through measurement of absorption of light of a particular wavelength is known as colorimetry. The absorbance of light of a particular wavelength by a substance in solution varies directly with its concentration and the thickness of the solution. When the thickness of the medium is kept constant, the absorbance directly depends upon the concentration.

### Procedure:

Transfer 2, 4, 6, 8 and 10 cm<sup>3</sup> of given Copper Sulphate solution into different 25 cm<sup>3</sup> volumetric flask from a burette. Add 2.5 cm<sup>3</sup> of Ammonia to all the flasks and makeup all the solutions up to the mark using distilled water. Stopper the flasks shake well to get uniform concentration.

Prepare a blank solution by taking 2.5 cm<sup>3</sup> of Ammonia in another 25 cm<sup>3</sup> volumetric flask. Make up that solution also up to the mark using distilled water and shake well. To the given unknown solution also add 2.5 cm<sup>3</sup> of Ammonia, make it up to the mark and shake well. After 10 minutes, set the instrument to zero reading using blank solution and measure the absorbance of all solutions, by using 620 nm filter of a Colorimeter.

Note down the optical density of all solutions and tabulate the readings. Draw a calibration curve by plotting absorbance against volume of Copper Sulphate taken. Using the calibration curve, find out the unknown volume of Copper Sulphate solution given and calculate the amount of Copper present in it.



## VIVA QUESTIONS

- 1 What is Colorimetry?
- 2 What forms the basis for Colorimetric determination?
- 3 What is photoelectric Colorimeter?
- 4 What are filters? Why are they used?
- 5 What is wave length?
- 6 What is frequency?
- 7 Why is estimation of Copper done at 620 nm wave length?
- 8 What is wave number?
- 9 State Beer's law.
- 10 State Lambert's law.
- 11 State Beer-Lambert's law.
- 12 Upon what factors the absorbance of a solution depends?
- 13 What is calibration curve?
- 14 What is meant by transmittance?
- 15 Why a filter of 620 nm is used in the estimation of Copper?
- 16 Mention a few important criteria for satisfactory Colorimetric analysis.
- 17 Mention a few advantages of Photoelectric Colorimetric determination.
- 18 What is Blank solution?
- 19 What is the basic requirement of a compound for its estimation by Colorimetric method?
- 20 Why is Ammonia added? Why is that same amount of Ammonia added?

**Tabulation:**

Sl. No.	Vol. of $\text{CuSO}_4$ in $\text{cm}^3$	Optical Density	Wt. of Copper (mg)
1.	0 [ Blank]	.....	.....
2.	2		
3.	4		
4.	6		
5.	8		
6.	10		
7.	Unknown		

**Calculation:**

From the graph,

Volume of  $\text{CuSO}_4$  corresponds to optical density of unknown solution (test solution) = Z  $\text{cm}^3$ Weight of copper sulphate pentahydrate present in  $100 \text{ cm}^3$  of the given solution = 'X' mgWeight of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in  $1 \text{ cm}^3$  of its solution =  $X/100 \text{ mg} = \frac{\text{'Y'}}{100} \text{ mg}$  = 'Y' mgWeight of copper present in  $1 \text{ cm}^3$  of its solution =  $\frac{\text{'Y'}}{100} \times 63.54$   
 $\frac{63.54}{249.54}$ Weight of copper present in Z  $\text{cm}^3$  of its solution =**Report:****Volume of unknown solution = ....  $\text{cm}^3$** **Amount of Copper in the given unknown solution = ..... mg.**

## EXPERIMENT NO– 11

### Aim:

Estimation of Hydrochloric acid present in 1.5 dm<sup>3</sup> of HCl solution using Conductometer.  
Given, the normality of NaOH solution is 0.05N

### Theory:

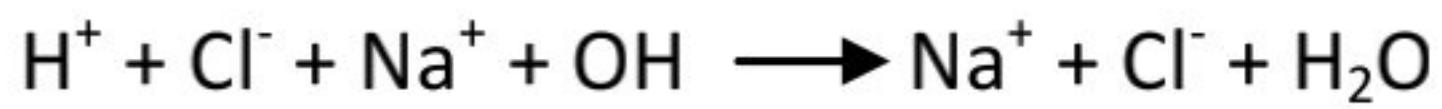
Conductance of any solution (C) is the ease with which current flows through it. It is the reciprocal of resistance (R) offered by the solution.

$$C = 1/R$$

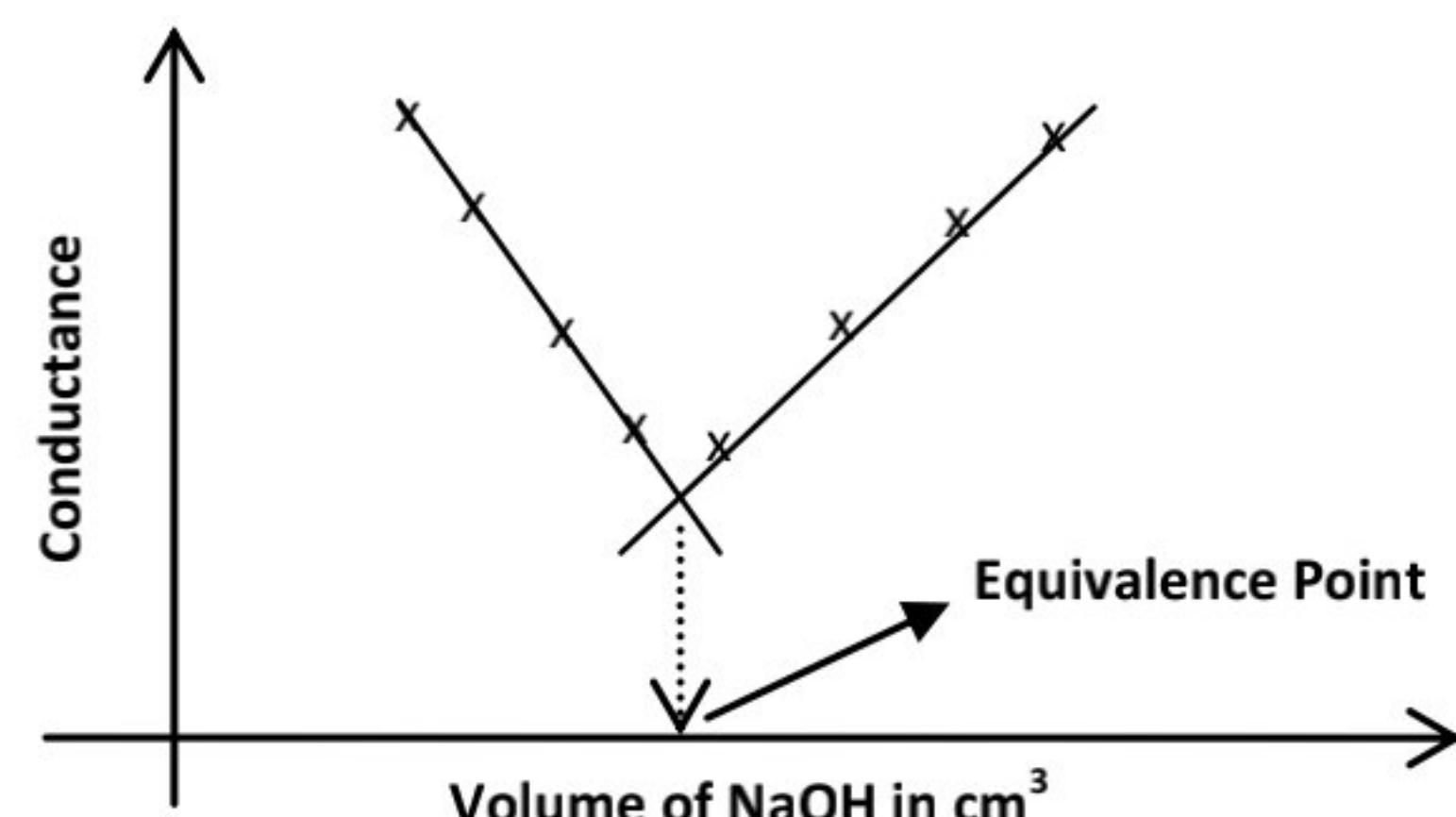
Conductance of a solution depends on number of ions, charge on the ions and also on the mobility of the ions. During the course of a titration, the conductance of the solution may change due to any of the above reasons. Such titrations in which conductance measurements are made use of in determining the equivalence point are called Conductometric titrations.

Among all Conductometric titrations, reaction involving strong acid (HCl) and strong base (NaOH) is the most common activity.

Hydrochloric acid is a strong electrolyte and would be completely in dissociated state. The conductance of this solution would be high. Upon addition of NaOH, highly mobile H<sup>+</sup> ions of the acid are replaced by less mobile Na<sup>+</sup> ions.



Hence the conductance would decrease steeply. The same trend would continue till all the H<sup>+</sup> ions available in the acid are completely reacted with the added NaOH. Now, the addition of NaOH would add more and more of Na<sup>+</sup> and OH<sup>-</sup> ions. Due to relatively higher mobility of OH<sup>-</sup> ions, the conductance of the solution increases again. A typical graph of conductance v/s volume of NaOH (added) is given below.



The break in the curve corresponds to the equivalence point of the reaction. From the volume of NaOH required to neutralize HCl solution, the amount of HCl is calculated.

### Procedure:

Pipette out 50 cm<sup>3</sup> of acid into a clean 200 cm<sup>3</sup> beaker. Dip the conductivity cell in the acid, such that the two Platinum electrodes are immersed completely. The cell is connected to the conductivity bridge. Measure the conductance. Take the given NaOH solution in a burette rinsed with the same solution. Rundown 0.2 cm<sup>3</sup> of NaOH solution into the beaker and shake well. Note down the conductance. Note down the conductance by adding 0.2 cm<sup>3</sup> of NaOH each time to the beaker. Conductance decreases in the beginning starts increasing after a certain addition of NaOH. Take around 8-10 readings on the increasing side. Draw a graph of conductance against volume of NaOH added. The point of minimum conductance (intersecting point of the lines) is called the **Equivalence point** and it corresponds to complete neutralization of HCl. Calculate the normality and amount of HCl present in 1000 cm<sup>3</sup> of its solution.

## VIVA QUESTIONS

- 1 State Ohm's law.
- 2 What is conductance?
- 3 What is the unit of conductance?
- 4 Name the SI unit of conductance.
- 5 Mention the different types of conductivities.
- 6 Which of the above conductivity measured during Conductometric titration?
- 7 What is specific conductivity?
- 8 What is equivalent conductivity?
- 9 What is molar conductivity?
- 10 What is a cell?
- 11 Define cell constant
- 12 Describe a conductivity cell.
- 13 What is the principle involved in Conductometric titration?
- 14 How is the accuracy of the method determined?
- 15 Explain the graph obtained in case of Conductometric titration involving acid mixture and NaOH.
- 16 What are the advantages of Conductometric titration over visual or potentiometric titration?
- 17 How do you account for the decrease in the conductivity at the beginning of the titration?
- 18 How would you account for the increase in the conductivity after the end point?
- 19 The second line in the graph is not as steep as the first. Explain.

**Tabulation:**

<b>Volume of NaOH in cm<sup>3</sup></b>	<b>Conductance in m S</b>
0.0	
0.2	
0.4	
0.6	
0.8	
1.0	
1.2	
1.4	
1.6	
1.8	
2.0	
2.2	
2.4	
2.6	
2.8	
3.0	
3.2	
3.4	
3.6	
3.8	

**Calculation:**

From the graph, equivalence point corresponds to Volume of NaOH = cm<sup>3</sup>

$$(NV)_{\text{HCl}} = (NV)_{\text{NaOH}}$$

$$N_{\text{HCl}} = \frac{(NV)_{\text{NaOH}}}{V_{\text{HCl}}}$$

Amount of HCl present in 1000 cm<sup>3</sup> of its solution = N<sub>HCl</sub> × Gram equivalent weight of HCl (36.5 g)

**Report:**

1. Normality of the HCl = ..... N
2. Amount of HCl present in 1000 cm<sup>3</sup> of its solution = .....g

## EXPERIMENT NO– 12

### Aim:

Determination of Viscosity Coefficient of a given liquid using Ostwald's viscometer

### Theory:

Viscosity arises due to friction between moving layers of a liquid. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. Layers close to the surface are almost stationary while that at the axis of the tube moves faster than any other intermediate layer. A slow moving layer exerts a drag or frictional force on its nearest moving layer, backwards. This property of the liquid which retards or opposes the motion between the layers is called viscosity. **The coefficient of viscosity is defined as the tangential force per unit area required to maintain a unit velocity gradient between the two successive layers of the liquid situated at unit distance apart.** The coefficient of viscosity of a liquid is given by the Poiseuille's formula

$$\eta = \frac{\pi P r^4 t}{8 V L}$$

Where 'V' is the volume of the liquid, 'r' is the radius of the tube and 'P' is the pressure between the two ends of the tube and ' $\eta$ ' is the coefficient of viscosity. If equal volumes of the two different liquids are allowed to flow through the same tube under identical conditions then,

$$\frac{\eta_1}{\eta_2} = \frac{t_1 d_1}{t_2 d_2}$$

The time 't' taken by the given liquid to travel through a certain distance in the tube is determined. The time 't' taken by standard liquid to travel through the same distance is measured. Knowing the densities of the two liquids ( $d_1$  and  $d_2$ ) and also the coefficient of viscosity of the standard liquid, coefficient of viscosity of test liquid is calculated.

### Procedure:

Take a clean and dry viscometer. Fix it vertically to a stand. Using graduated pipette, transfer

known volume (say 10 cm<sup>3</sup>) of the given liquid into the viscometer. Then it is placed in a thermostat (Water Bath) such that its big bulb is completely immersed in water. Suck the liquid above the upper mark of the small bulb of the viscometer. Allow it to flow through the capillary. When the liquid crosses the upper mark of the small bulb, start a stop watch and when the liquid crosses the lower mark, stop the watch. Note down the time of flow in seconds.

Repeat the experiment 3-4 times and calculate the average time of flow. Remove the liquid from the viscometer, clean well with acetone and dry it in oven. Cool the viscometer to room temperature. Similarly, measure the time of flow for water by taking the same volume. Calculate average time of flow for water. Note down the room temperature. Knowing the density, viscosity of water and density of the given liquid, viscosity of the liquid can be calculated.

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

If Laboratory temperature [T] K  
At 'T' K density of water =  $d_w$   
At 'T' K density of liquid =  $d_l$   
At 'T' K viscosity of water =  $\eta_w$   
At 'T' K viscosity of liquid =  $\eta_l$

### VIVA QUESTIONS

- 1 What is Viscosity Coefficient of a liquid?
- 2 What is density of a liquid?
- 3 What is specific gravity?
- 4 How are specific gravity and density related?
- 5 What is SI unit of Viscosity Coefficient?
- 6 What are the factors that affect the viscosity of a liquid?
- 7 How does the viscosity vary with temperature?
- 8 Why is acetone used for cleaning viscometer?
- 9 Why do you require laboratory temperature for viscosity determination?
- 10 How is the viscosity of liquid related to its mobility?
- 11 What is fluidity of a liquid?

**Observation:**

Liquid	Time of Flow		
	in min: sec	in seconds	Average in seconds
Test liquid			
			$t_l =$
Water			
			$t_w =$

**Calculation:**Laboratory temperature [T] =  ${}^{\circ}\text{C}$  = KAt 'T' K density of water =  $d_w$  =At 'T' K density of liquid =  $d_l$  =At 'T' K viscosity of water =  $\eta_w$  =At 'T' K viscosity of liquid =  $\eta_l$  =

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

(1 milli poise =  $10^{-4} \text{ Nsm}^{-2}$ )**Report:** The coefficient of viscosity of the given liquid at .....  ${}^{\circ}\text{C}$  =  $\text{Nsm}^{-2}$

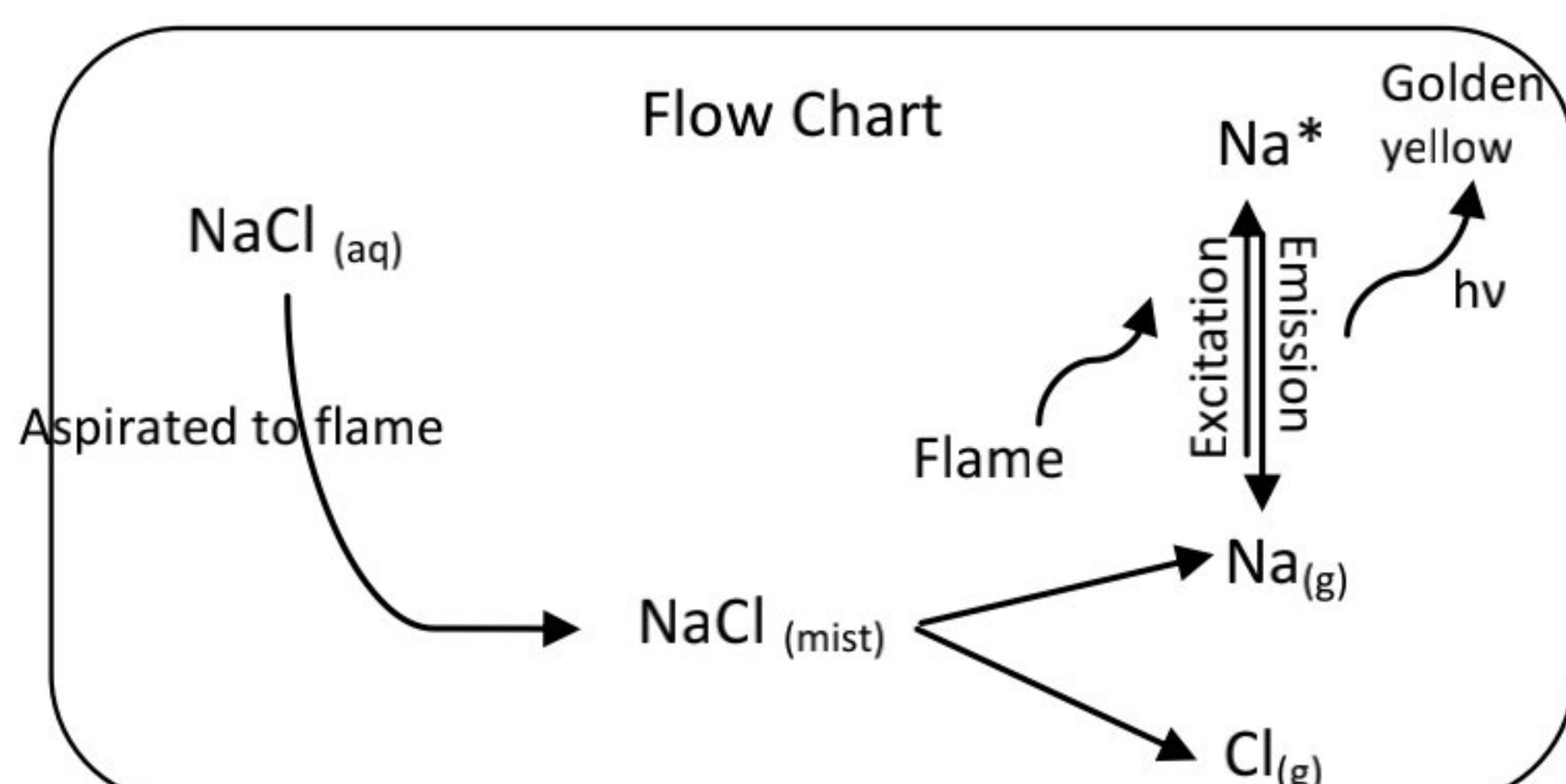
## EXPERIMENT NO– 13

### Aim:

**Estimation of Sodium present in a given NaCl solution, using Flame Photometer.** Amount of NaCl present in 100 cm<sup>3</sup> of the solution is 2.5 g

### Theory:

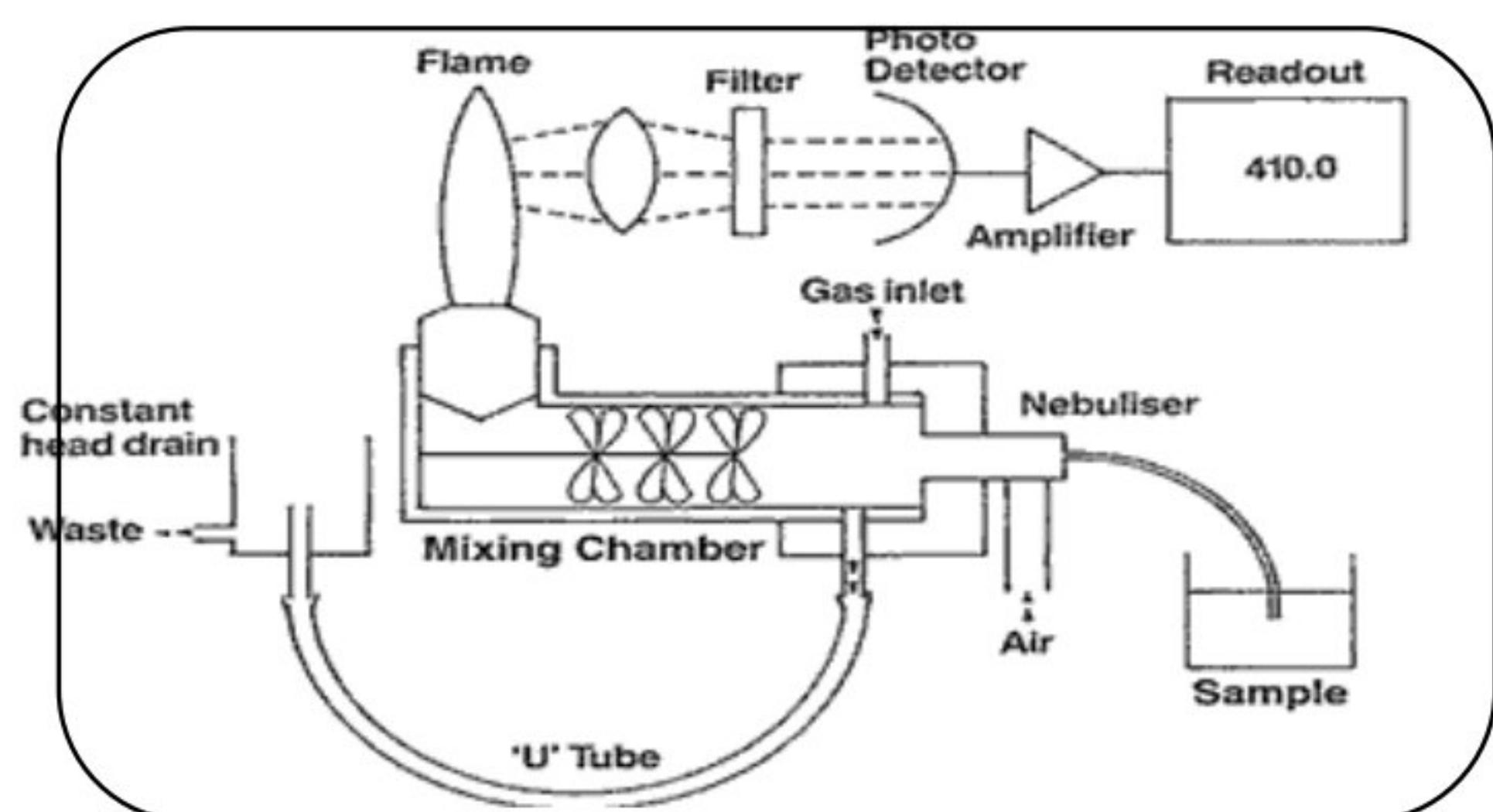
Flame Photometry is an atomic emission technique used for detection of metals. If a solution containing metallic salts is aspirated into a flame, a vapour will be formed which contains metallic atoms. The electrons from the metallic atoms are then excited from ground state ( $E_1$ ) to higher energy state ( $E_n$ ) where  $n = 2, 3, 4$ , etc., by making use of thermal energy of flame. From higher energy states, these electrons will return to the ground state by emitting radiations ( $E_n - E_1 = h\nu$  where  $n = 2, 3, 4$ , etc., which are the characteristic of each element.



Flame photometer correlates the emitted radiations with the concentration of these elements. It is simple and rapid method for the elements that can be easily excited (Sodium and other alkali metals).

A flame photometer is composed of the pressure regulator, flow meter, an atomizer, burner, optical system, photosensitive detector and output recorder. A filter corresponding to the wavelength of light emitted by the element whose concentration is to be determined, is inserted between the flame and the detector. Propane gas is used as fuel and air or Oxygen is used as oxidant. Combination of these two will give a temperature of 1900 °C. The whole

analysis depends on the flow rate of the fuel, oxidant, the rate of introduction of the sample and droplet size.



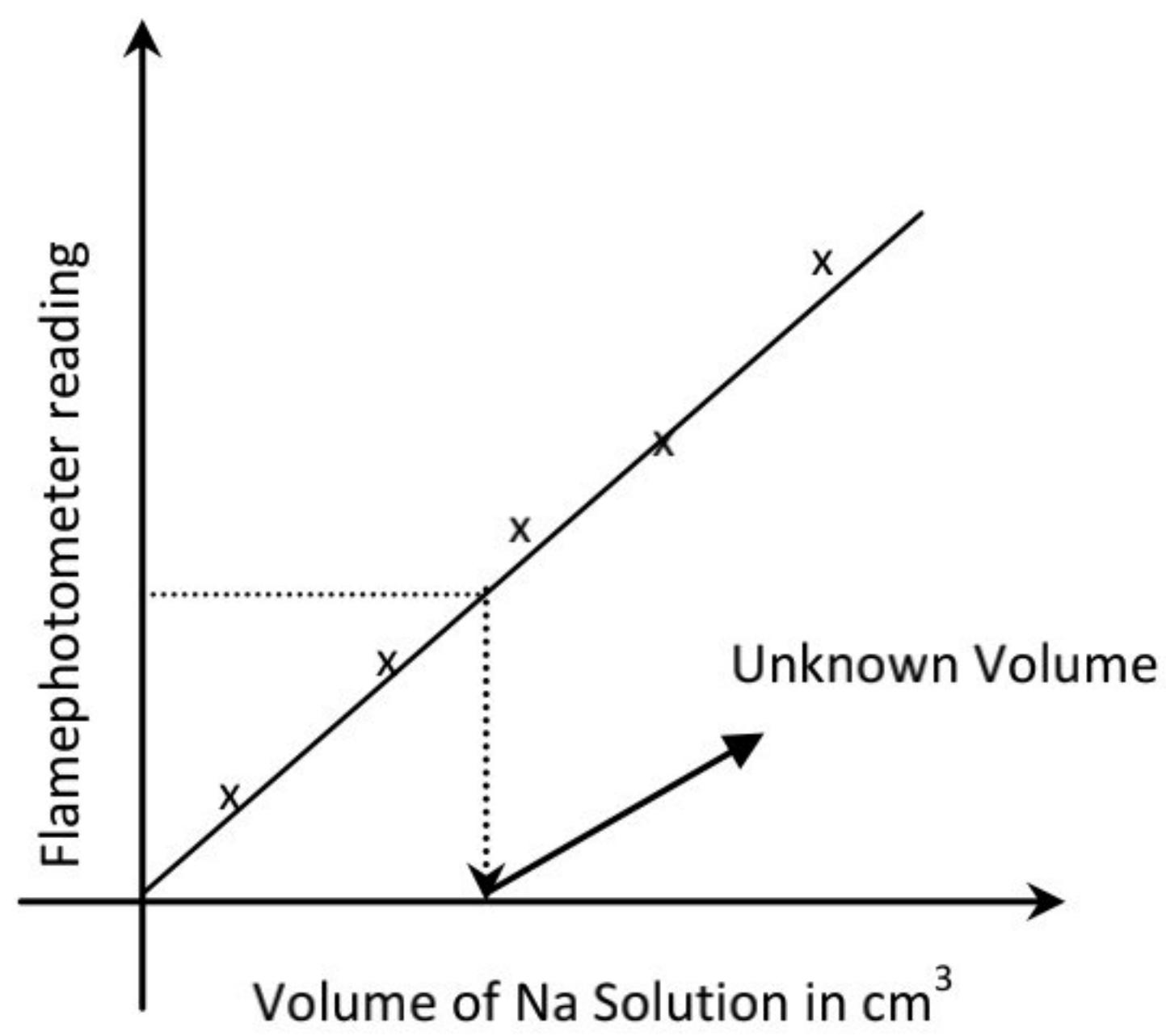
The sample containing the analyte is aspirated into the flame through nebulizer. Radiation from resulting flame is collected by the lens and allowed to pass through an optical filter, which permits only the radiation characteristic of the element under investigation into the photocell. The output from the photocell represents the concentration and nature of the element.

### Procedure:

Transfer 2, 4, 6, 8 and 10 cm<sup>3</sup> of standard Sodium solution into different 25 cm<sup>3</sup> volumetric flasks from the burette. Make up all the solutions using double distilled water. Stopper the flasks and shake well to get uniform concentration. To the given unknown solution, add double distilled water and shake well. Switch on the instrument; turn the gas supply on and light the gas at the burner. Adjust the air supply from the compressor to 10 lbs/sq inch using pressure regulator knob. Place the sodium filter (589 nm) in position. Now dip the capillary tube in a cell containing double distilled water. The stream of air atomized as a fine mist draws up the liquid. Regulate the gas supply so that the colour of the flame completely turns to blue. Adjust the flame photometer to zero by means of zero control knob. Feed the various sodium solutions prepared, through the flame one by one including the unknown solution. Note down the flame photometer readings. Plot a graph of flame photometer readings against the volume of the solution get the calibration curve. Using the curve obtained find out the volume of the

unknown solution containing sodium and calculate the amount of Sodium in it.

### Graph



### VIVA QUESTIONS

1. What is meant by atomic emission?
2. What is Flame Photometry?
3. What are alkali and alkaline earth metals?
4. What are the various events that occur when solution containing metal atom ions atomized through a flame?
5. What are different components of a Flame Photometer?
6. Why the analysis of Sodium is advantageous in Flame Photometry?
7. What is the role of filter in Flame Photometry?
8. What are the errors that occur in Flame Photometry?
9. What are the factors that influence this experiment?
10. Name the fuel and oxidant used in this experiment?

**Tabulation:**

Sl. No.	Vol. of NaCl in cm <sup>3</sup>	Flame Photometer Reading	Wt. of Sodium in mg
1.	2		
2.	4		
3.	6		
4.	8		
5.	10		
6.	Unknown		

**Calculation:**

From the graph, Volume of NaCl corresponds to Flame Photometer Reading of unknown solution (test solution) = Z cm<sup>3</sup>

Weight of Sodium Chloride present in 100 cm<sup>3</sup> of the given solution = mg

58.5 g of NaCl contains 23 g of Na.

Therefore, 1 cm<sup>3</sup> of the given stock solution contains =  $\frac{w \times 23}{58.5 \times 100}$  = 'A'g of Na

Z cm<sup>3</sup> of the given stock solution contains = (A × Z) g of Na

**Report:**

**Volume of unknown solution = .... cm<sup>3</sup>**

**Amount of Sodium in the given unknown solution = ..... mg.**

## EXPERIMENT NO– 14

### Aim:

Determination of relative and Kinematic Viscosities of given lubricating oil at different temperatures using Redwood Viscometer

### Theory:

By definition, a fluid is a substance that continues to deform so long as a shear force is acted upon it. An ideal fluid is one, which continuously deforms i.e., flow, however negligible the applied shear force may be. However, the real fluids offer some resistance before they deform. This resistance to flow is due to its property, called viscosity of the fluid. In solids the induced stress is directly proportional to the resulting strain within the elastic limit, and this is true for all types of stresses, be it tensile, compressive or shear stress. According to Newton's law of fluids, however, in fluids the shear stress,  $\tau$ , is proportional to time rate of strain,  $d\theta/dt$ . Mathematically,  $\tau \propto (d\theta/dt) \Leftrightarrow \tau = \mu (d\theta/dt)$ . Where the constant of proportionality  $\mu$  is called coefficient of dynamic viscosity and it is measured in poise. Thus, absolute or dynamic viscosity is the tangential force per unit area required to move one horizontal plane of the fluid at unit velocity with respect to another maintained at a unit distance apart by the fluid. When the force is 1 dyne/cm<sup>2</sup>, the distance between the layers or planes 1cm and the velocity gradient 1 cm/sec, the viscosity is 1poise. This being a large unit, absolute viscosity is more commonly expressed as one hundredth of poise called centipoise. The viscosity of water, for example, at 20 °C is 1.002 centipoises. The coefficient of kinematic viscosity,  $\nu$ , is defined as the ratio of dynamic viscosity of fluid to its density. Fluids, which obey Newton's law, are called Newtonian fluids and those, which do not obey, are called Non-Newtonian fluids. All gasses, water etc., are examples of Newtonian fluids while visco-elastic materials like tar, molasses, honey, blood etc., and mixtures of fluids and solids such as slurries are examples of non-Newtonian fluids.

Some of the reasons why measurement of viscosity is vital in most engineering applications of fluids are:

- Viscosity of fuel oils influences the ease of handling, transportation and nature of storage. High viscosity fuel oil cannot be properly atomized leading to incomplete combustion and loss of fuel. Fuel oils are usually pre-heated to reduce viscosity and above 120 °C viscosity are almost constant.

Maximum viscosity for easy atomization in conventional burners is 25 centistokes (100 seconds Redwood I). A higher preheating temperature decreases the oil density causing lesser weight of oil delivered to the burner, thereby reducing burner output. Hence, accurate value of viscosity of oil is required for optimum burner output.

- High viscosity lubricating oil reduces its fluidity besides causing undue friction. Indeed, viscosity of lubricants determines the bearing friction, heat generation and fluidity under conditions of load and speed. Only lubricants of rated viscosity as per design parameters must be used.
- In case of diesel fuels, too low viscosity causes excessive leakage at the injection pistons while too high viscosity produces coarse droplets leading to incomplete combustion.
- Viscosity determines the pressure drop in pumping of fluids through open or closed channels. For easy and economic pumping the maximum viscosity should be 12 stokes (486 seconds Redwood II) in closed channels.
- Viscosity of oil blends must be experimentally determined since viscosity is neither an additive property nor can be calculated using blending charts. Though the units of kinematic and dynamic viscosity are stokes and poise respectively, the commonly used methods in engineering industry measure the viscosity in arbitrary units, using commercial viscometers. Commercial viscometers work on the following principle. When a liquid is made to flow through a capillary tube by a pressure gradient, and the time for a given volume to flow along the tube is measured, then at a constant temperature, the coefficient of viscosity,  $\mu$ , is given by Poiseuille's equation as:  $\mu = \Pi PR^4 T/8VL$  Where  $P$  = pressure difference between the ends of the tube  $L$  = length of the tube  $R$  = radius of the tube  $T$  = time in seconds  $V$  = volume of the liquid flowing through the tube in  $T$  seconds When the 'Inlet correction' and the 'kinetic energy correction' are incorporated, the above equation takes the form:  $\mu = (\Pi PR^4 T)/(8V(L + k_1R)) - (Vpk^2)/(8\Pi(L + k_1R)T)$  where  $\rho$  is the density of the liquid and  $k_1$  and  $k_2$  are constants which depend on the form of the apparatus and are determined experimentally. The driving force is provided by the head of the test oil flowing vertically down the tube and is given by:  $P = h \rho g$  where  $h$  is the mean height of the oil. The tube dimensions are so chosen that  $k_1R$  in the above equation becomes negligible as compared to  $L$ , so that  $\mu = (\Pi h \rho R^4 T/8VL) - (Vpk^2/8\Pi LT)$  for a given viscometer the head of the oil is maintained at the

same height  $h$ . The time  $T$  in seconds is measured for flow of a fixed volume of the oil through the capillary of fixed dimensions. Then  $h$ ,  $g$ ,  $R$ ,  $V$  and  $L$  all become constant and the above equation is reduced to

$$\mu = ApT - (Bp/T) \text{ where } A \text{ and } B \text{ are constants.}$$

Dividing both sides by  $\rho$ ,

$$We \ get \ \mu/\rho = At - B/t \Leftrightarrow v = At - B/t$$

The above equation is called viscometer equation because all the commercial viscometers are based on this equation. The time  $t$  in seconds measured experimentally is reported as relative viscosity and its magnitude depends on the viscometer used and the volume of the liquid that flows through the tube of the viscometer. Therefore, while reporting the viscosity of oils using commercial viscometers, the viscometer used must be specified. Most commercial viscometers express viscosity in terms of number of seconds of time required for a fixed volume of fluid at a certain temperature to flow through a standard orifice. The measured time is then converted into corresponding stokes or poise using empirical formulae i.e.,  $A$  and  $B$  in the above equation, supplied with the viscometer.

Three makes of commercial viscometers, REDWOOD, SAYBOLT and ENGLER, are popularly used in engineering applications. The time measured in seconds by these viscometers is usually post-fixed with the name of the viscometer itself. The REDWOOD viscometer is widely used in commonwealth countries including India. In REDWOOD viscometer viscosity is measured in number of seconds required for a fixed quantity of oil, usually 50 cc., to flow. Two types of REDWOOD viscometers are used, REDWOOD I viscometer is used for low viscosity oils whereas REDWOOD II viscometer is used for high viscous oils. Oil flow port area for REDWOOD II viscometer is larger than that of REDWOOD I viscometer to facilitate quicker flow of high viscous oils. Consequently the empirical formulae are different for each of them. The time required for a standard volume of oil to flow from REDWOOD II viscometer is 1/10 of that require for an equal volume of same oil at the same temperature to flow from REDWOOD I viscometer. Viscosity measured by REDWOOD viscometers are reported as "n seconds of REDWOOD I or II", as the case may be, at a given temperature. DESCRIPTION REDWOOD – I viscometer consists of a metal cup with an axially placed orifice in the base. A metal ball or a rod can be used to close the hole. The metal cup can be heated and the oil stirred to ensure uniform temperature throughout the oil.

When the ball is removed, a thin stream of oil runs into a small graduated glass flask and the time to fill the flask is recorded. This time in seconds is called "seconds REDWOOD I" and is a measure of viscosity. These seconds REDWOOD I are converted into stokes of kinematic viscosity using the empirical formula

$$v = AT - (B/T) \text{ where } A \text{ and } B \text{ are viscometer constants and } T \text{ is seconds REDWOOD I.}$$

The difference between REDWOOD I and REDWOOD II viscometers lies in the dimensions of the discharge capillary (either tube, jet or orifice) as given below: viscometer diameter of flow port length of flow port REDWOOD I 1.62 mm and 10 mm, REDWOOD II 3.8 mm and 50 mm. When an oil of unknown viscosity is given it must always be tested with REDWOOD – I viscometer. If the viscosity exceeds 2000 SECONDS REDWOOD – I then the REDWOOD – II be used.

#### Procedure:

Clean the viscometer cup properly with the help of suitable solvent e.g.  $CCl_4$ , ether, petroleum spirit or benzene and dry it to remove any traces of solvent. Level the viscometer with the help of leveling screws and fill the outer bath with water for determining the viscosity at  $80^{\circ}C$  and below. Place the ball valve on the jet to close it and pour the test oil into the cup up to the tip of indicator. Place a clean dry Kohlrausch flask immediately below and directly in line with discharging jet. Insert a clean thermometer and a stirrer in the cup and cover it with a lid. Heat the water filled in the bath slowly with constant stirring. When the oil in the cup attains a desired temperature, stop the heating. Lift the ball valve and start the stop watch, oil from the jet flows into the flask. Stop the stop watch when lower meniscus of the oil reaches the 50 ml mark on the neck of receiving flask. Record the time taken for 50 ml of the oil to collect in the flask. Repeat the experiment to get more readings.

#### VIVA QUESTIONS

1. What is Viscosity?
2. What are different types of viscosity explain them and write the units?
3. What are factors effecting viscosity?
4. Mention some applications where viscosity is considered?
5. Relation between density and viscosity?
6. Which constructional feature of viscometer varies with the viscosity of oil?

**Observation & Calculation:**Laboratory temperature = .....  $^{\circ}\text{C}$ 

Density of given liquid = ..... g/cc

Sl.No.	Temperature In ( $^{\circ}\text{C}$ )	Kinematic Viscosity(Redwood Seconds)(v)	Density( $\rho$ ) (Kg/m <sup>3</sup> )	Dynamic (or absolute) Viscosity (Redwood Seconds) ( $\mu=v * \rho$ )
1				
2				
3				
4				

**Report:** The Dynamic Viscosity of given oil sample using Redwood Viscometer no. ----- at .....  $^{\circ}\text{C}$  is \_\_\_\_\_ Red-Wood seconds.

## EXPERIMENT NO– 15

### Aim:

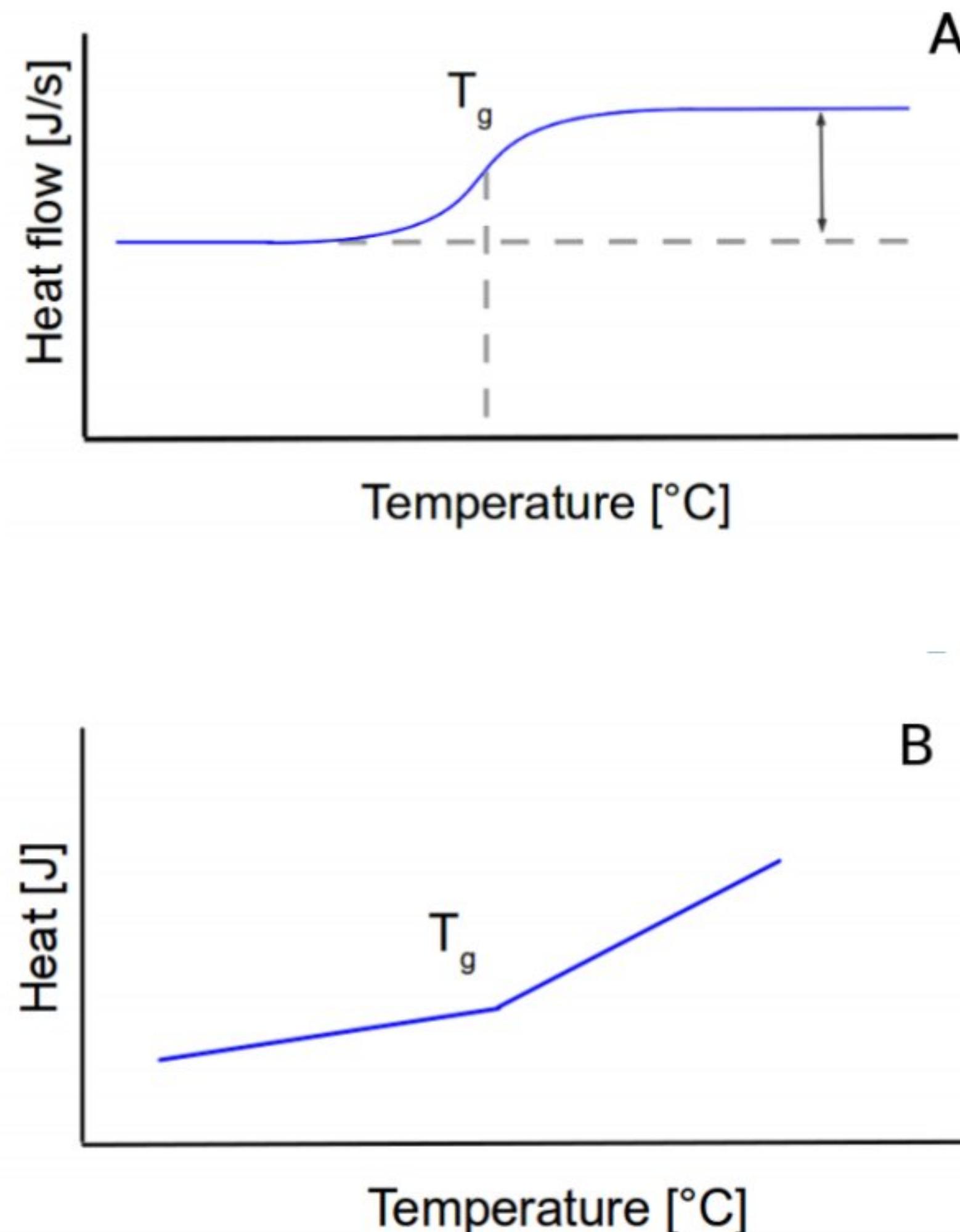
To find  $T_g$  of Polymer using DSC

### Theory:

Differential scanning calorimetry (DSC) is a technique used to investigate the response of polymers to heating. DSC can be used to study the melting of a polymer or the glass transition. If a polymer is heated, it will at some point reach its glass transition temperature ( $T_g$ ). At this point the mechanical properties of the polymer change from those of an elastic material to those of a brittle one due to changes in chain mobility. A typical example of a heat flow versus temperature plot at a glass transition temperature is shown in Figure A & B.

The glass transition results in a kink in the heat versus temperature plot due to the change in heat capacity (A). In a plot of heat flow versus temperature it is a gradual transition that occurs over a range of temperatures (B). The glass transition temperature is taken to be the middle of the sloped region.

The heat capacity of the polymer is different before and after the glass transition temperature. The heat capacity  $C_p$  of polymers is usually higher above  $T_g$ . DSC is a valuable method to determine  $T_g$ . It is important to note that the transition does not occur suddenly at one unique temperature but rather over a range of temperatures. The temperature in the middle of the inclined region is taken as the  $T_g$ . The DSC set-up is composed of a measurement chamber and a computer. Two pans are heated in the measurement chamber. The sample pan contains the material being investigated. A second pan, which is typically empty, is used as a reference. The computer is used to monitor the temperature and regulate the rate at which the temperature of the pans changes. A typical heating rate is around  $10\text{ }^{\circ}\text{C}/\text{min}$ . The rate of temperature change for a given amount of heat will differ between the two pans. This difference depends on the composition of the pan contents as well as physical changes such as phase changes.



### Procedure:

#### Sample preparation

A sample of 1-4 mg of polymer sample is placed in a  $40\text{ }\mu\text{l}$  aluminum pan. Adapt the sample size to your pan size, if different. The polymer sample should cover the bottom of the pan. The filled pan is crimped and locked with Aluminium lid. DSC Instrument is kept on for 30 minutes to attain the required temperature. The pan containing sample is placed in the heating chamber with extreme care. Give the required inputs like heating rate, sample weight, cooling time etc in the software. Run the DSC machine and get the heat versus temperature graph. The proper interpretation of the obtained graph will give you the glass transition temperature of the sample measured.

The glass transition results in a kink in the heat versus temperature plot due to the change in heat capacity (A). In a plot of heat flow versus temperature it is a gradual transition that occurs over a range of temperatures (B). The glass transition temperature is taken to be the middle of the sloped region.

## VIVA QUESTIONS

4. What is the principle of this experiment?
  5. Why Glass transition temperature is in range?
  6. Explain the interpretation of Graph A and B
1. What is Glass transition temperature?
  2. What is the significance of  $T_g$ ?
  3. What are factors effecting  $T_g$ ?

**Report:**

**The  $T_g$  of given polymer is**