



## Course Outcomes

**Course Name – Chemistry of Smart Materials and Devices**

**Course Code – 22CHY12A**

- CO1** Identify the materials, conventional & non-conventional energy systems for engineering applications.
- CO2** Investigate chemical properties of materials for various technological applications.
- CO3** Apply the knowledge of material property and energy to analyze environmental issues.
- CO4** Develop solutions in the areas of applied materials and energy systems for sustainable engineering application.



## Department of Chemistry

### Certificate

This is to certify that Pranav Venkatesh Jambur, bearing USN PVC23BCS021 has satisfactorily completed the experiments in Chemistry of Smart Materials and Devices for Computer Science Engineering Under Graduate programme during the academic year 2023-2024

*[Signature]*  
Signature of Head of the Department

*[Signature]*  
Signature of Faculty in-charge

LAB SEAT No:

SPARK-2-03

~~19~~

## PERSONAL DETAILS

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CHEMISTRY OF SMART MATERIALS AND DEVICES PRACTICE (22CHY12A)

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2	Estimation of copper from PCB	9	20/10/23	12	24	4	40
3	Determination of total acid content of the soft drinks using pH sensors	14	17/11/23	16	20	4	40
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10	Synthesis of metal oxide nanomaterials using solution combustion synthesis	49					
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Signature of Lab Teachers	Experiment Marks	10
	Lab Internal Marks	
	Lab EL Marks	
	Total Lab Marks	

312  
320

## 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.

- **Do not Taste or Sniff Chemicals**

Do not smell or taste chemicals as they may harm you.  
Use fuming chemicals in the fuming hood.

- **Do not Dispose the Chemicals Down the Drain**

Do not 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.

- **Do not eat or drink inside the laboratory.**

Do not eat or drink inside the laboratory.

- **Collect the data while performing Lab experiment.**

Directly record the readings in the workbook 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 given 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 chemical methods of analysis.

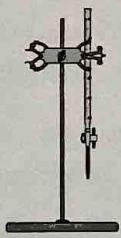
### Pipette:

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.



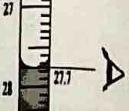
### Burette:

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



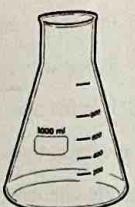
completely filled with the solution)

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.

## EXPERIMENT NO – 1

### Aim: Introduction to Volumetric analysis

#### 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.

**Endpoint:** 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 endpoint.

**Types of Indicators:** 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  $1 \text{ dm}^3$  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  $1 \text{ dm}^3$  of solution.

**Mass of the substance (in  $1 \text{ dm}^3$ ) = Normality \* Gram equivalent weight of the substance**

**Molarity (M):** The number of moles of solute present in  $1 \text{ dm}^3$  of solution.

**Mass of the substance (in  $1 \text{ dm}^3$ ) = Molarity \* 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  $10^6$  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  $\text{OH}^-$  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:  $K_2Cr_2O_7$ , Oxalic acid,  $AgNO_3$ ,  $KCl$ , 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:  $NaOH$ ,  $KMnO_4$ ,  $HCl$  etc.

## EXPERIMENT NO – 2

#### Aim: Estimation of copper from PCB

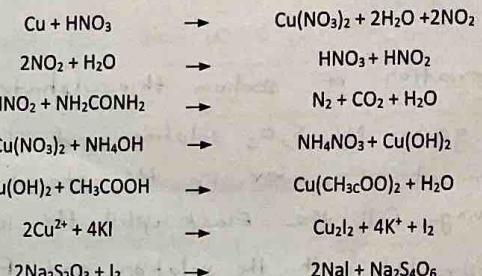
##### Theory:

Printed circuit board is an essential part of all electrical and electronic equipment's. It is composed of polymers, ceramics and metals which all are come under Hazardous and non-hazardous categories. About 40% heavy metals present in it which are turned into hazardous residue if the discarded pcbs are not disposing properly. Metal compositions in PCBs are varies according to the manufacturer and the year of its manufacturing and technology

Table 1 shows the composition of PCB.

Metals	Availability (wt%)
Copper	30.57
Aluminium	11.69
Zinc	1.86
Tin	7.3
Nickel	1.58
Iron	15.21
Lead	6.71

A solution of copper is made by dissolution of the PCB 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 Sodium-thiosulphate consumed is a measure of the amount of copper present. The principle of this experiment is iodometric titration.



##### Description:

##### Part-A: Preparation of standard Sodium thiosulphate solution

Weigh accurately the given  $Na_2S_2O_3$  and transfer it into a  $100\text{ cm}^3$  standard flask using a funnel. Add small amount of distilled water to dissolve the salt and make it up to the mark by adding some more distilled water. Shake it well to get uniform concentration. Rinse the burette and fill the burette with the prepared standard sodium thiosulphate solution.

$$\text{Normality of } Na_2S_2O_3 = (\text{weight of } Na_2S_2O_3) / \text{gram equivalent weight of } Na_2S_2O_3 (248.17)$$

##### Part-B: Preparation of copper solution and estimation of copper

Weigh exactly the given sample of PCB into a clean  $250\text{ cm}^3$  conical flask. Add Conc. Nitric acid drop by drop until the Brass dissolves completely and filter it into a  $250\text{ cm}^3$  volumetric flask. Make it up to the mark using distilled water.

Take  $25\text{ cm}^3$  of the prepared brass solution. Add one spatula urea and 1 tt of distilled water to the solution and heat it to boiling and cool it. 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-B two more times. Calculate the percentage of copper present in the Brass samples by taking one titer value.

#### Part-C: Calculation

Weight of PCB sample dissolved in  $250\text{ cm}^3$  of solution = 'W' g ( $W = 5.0\text{ g}$ )

Normality of sodium thiosulphate = 'Y'

Volume of sodium thiosulphate = 'X'  $\text{cm}^3$  (from titration)

$1000\text{ cm}^3$  1N sodium thiosulphate = 63.54g of copper

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

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

$250\text{ cm}^3$  of prepared PCB solution contains = '10A' g of Cu

**Model Procedure:** Procedure of this experiment is divided into two parts.

#### Part A: Preparation of sodium thiosulphate (solution)

- 1) Weigh the given  $\text{Na}_2\text{S}_2\text{O}_3$  solution and dissolve it in minimal amount of water in the standard flask.
- 2) After dissolving fill the flask upto the  $100\text{ cm}^3$  mark
- 3) Rinse the burette with the solution and fill it till the zero mark (lower meniscus touching zero)

#### Part B: Preparation of copper solution for titration

- 1) Already prepared copper solution (Brass + conc.  $\text{HNO}_3$ ) was taken in a clear conical flask ( $25\text{ cm}^3$ )
- 2)  $\frac{1}{2}$  table spoon of urea was added with one test tube of distilled water and was dissolved with the help of heating.
- 3) 2 drops of  $\text{NH}_4\text{OAc}$  and  $\frac{1}{4}$  th testube of  $\text{CH}_3\text{COOH}$  was added

- 4) This mixture was treated with KI and water keeping in mind  $\text{I}_2$  doesn't escape.
- 5) Started the process of titration until we achieved buff yellow colouration
- 6) Two drops of starch were added followed by titration till the dark blue solution turned white.

#### Model Calculation:

Weight of PCB sample dissolved in  $250\text{ cm}^3 \times 4 = 1000\text{ cm}^3$  of solution = 5g ( $W = 5.0\text{ g}$ )

Normality of sodium thiosulphate = 0.05589 N (Y)

Volume of sodium thiosulphate =  $100\text{ cm}^3$  (X)

$1000\text{ cm}^3$  1N  $\text{Na}_2\text{S}_2\text{O}_3$  = 63.54g of copper

$$\text{Therefore: } X \text{ cm}^3 \text{ of } Y \text{ N } \text{Na}_2\text{S}_2\text{O}_3 = \frac{63.54 \times Y}{100} \text{ g of Cu}$$

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

Since value was calculated for  $25\text{ cm}^3$  of PCB sol. (PCB titrate)

Weight of Cu in  $250\text{ cm}^3$   $\text{Na}_2\text{S}_2\text{O}_3$  sol = '10A' g (PCB titrate)

$\therefore$  Weight of Cu in  $1000\text{ cm}^3$   $\text{Na}_2\text{S}_2\text{O}_3$  sol = '40A' g

#### Observation and Calculation:

##### Part-A: Preparation of standard Sodium thiosulphate solution

Weight of the Weighing Bottle + $\text{Na}_2\text{S}_2\text{O}_3$	6.927	g
Weight of the Weighing Bottle	5.540	g
Weight of $\text{Na}_2\text{S}_2\text{O}_3$	1.387	g

$$\text{Weight of } \text{Na}_2\text{S}_2\text{O}_3 \text{ dissolved in } 100\text{ cm}^3 \times 10 = \frac{1.387 \times 10}{248.17}$$

$$\text{Normality of } \text{Na}_2\text{S}_2\text{O}_3 \text{ solution} = \frac{\text{Gram equivalent weight of } \text{Na}_2\text{S}_2\text{O}_3 \text{ salt (248.17)}}{248.17}$$

$$= 0.05589 \text{ N}$$

Part-B: Estimation of Copper in Brass solution

Burette readings (in cm <sup>3</sup> )	Trial-I	Trial-II	Trial-III	Expected Value by teacher
Final	7.1	7.3	7.1	
Initial	0.0	0.0	0.0	7.2
Volume of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> run down	7.1	7.3	7.1	

Part-C: Calculation

Hence expected concordant value = 7.1 cm<sup>3</sup>

1000 cm<sup>3</sup> of 1N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> = 63.54 g of copper

Normality of sodium thioglycolate sol = 0.05589 N

Volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> after titration = 7.1 cm<sup>3</sup> (X)

∴ 7.1 cm<sup>3</sup> of 0.05589 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> gives:  $\frac{63.54 \times X}{1000}$

$$\Rightarrow A = \frac{63.54 \times 7.1 \times 0.0559}{1000} = 0.02521 \text{ g of Cu} = \underline{\underline{Ag}}$$

But For 250 ml of PCB contains 40 g of Cu

∴ For 1000 ml of PCB contain 40A g of Cu

$$\Rightarrow 40A = 40 \times 0.02521 = \underline{\underline{1.0084 \text{ g of copper}}}$$

Inference: From the following experimentation, we can observe:

- 1) Yield: 1000 cm<sup>3</sup> of PCB yields around 1.0084 g of copper
- 2) Atom efficiency: It has quite a low atom efficiency due to formation of multiple by products.
- 3) Indication of end point: Additional auxiliaries are not added which vary the pH, but the reaction has a self indication cycle.

Relevance to Society & Environment: Certain points are essential while indicating the extraction of copper from PCB:  
 1) Safe by-products: Following principles of green-chemistry, by products formed in the reaction are non toxic and contained.  
 2) Recycling: Copper is a valuable metal in several industries hence this extraction process can increase availability.  
 3) Economical: Study shows for every amount utilised in manufacturing copper induced devices, 4% of that money can be saved by extraction.

Report: Amount of Cu present in the given 5 g of PCB sample =

Weight of Cu in 1000 ml solution containing  
5g e-waste:  $\boxed{1.0084 \text{ g}}$

Components	Marks	
	Max	Obtained
Model Procedure & Calculation	7+5	12
Burette Reading & Execution	20+4	24
Inference & Societal Relevance	2+2	4
Total	40	40
Signature of Teacher		26/10/23.

### EXPERIMENT NO – 3

**Aim:** Determination of total acid content of the soft drinks using pH sensor

**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  $pK_a$  value is coined. If  $K_a$  is the dissociation constant of a weak acid, then

$$pK_a = -\log_{10} K_a$$

The pH of a weak acid solution and  $pK_a$  are related as per Henderson - Hasselbalch equation:

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

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.,  $[\text{salt formed}] = [\text{acid unreacted}]$ . Therefore, equation (1) reduces to  $\text{pH} = pK_a$ . This indicates that at half equivalence point the  $pK_a$  of the weak acid is equal to pH. Therefore, the  $pK_a$  of a weak acid can also be defined as the pH at half equivalence point.

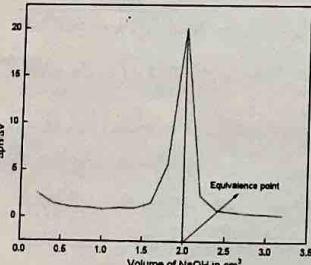
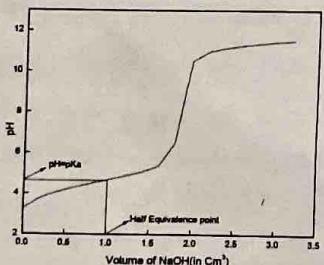
During an acid-base titration, initially the pH value of the solution increases gradually and increases more rapidly thereafter till the equivalence point is reached. Once the equivalence point is crossed, pH increases by small amounts. The variation of pH is measured using combined glass electrode.

#### Description:

Pipette out  $50 \text{ cm}^3$  of the given soft drinks (weak 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  $pK_a$  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



#### Calculation:

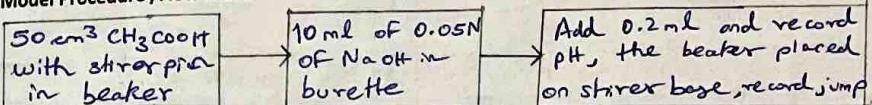
The experiment is divided into two parts.

1. Determination of equivalence point
2. Determination of  $pK_a$ . (Using table-1 corresponding to  $pK_a$  find out which weak acid is present)

Table-1

Acid	pKa	Gram equivalent weight
CH <sub>3</sub> COOH (acetic)	4.7	60/1 = 60
HCOOH (formic)	3.7	46/1 = 46
H <sub>2</sub> CO <sub>3</sub> (carbonic)	6.3	62/2 = 31
H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> (oxalic)	1.2	126/2 = 63

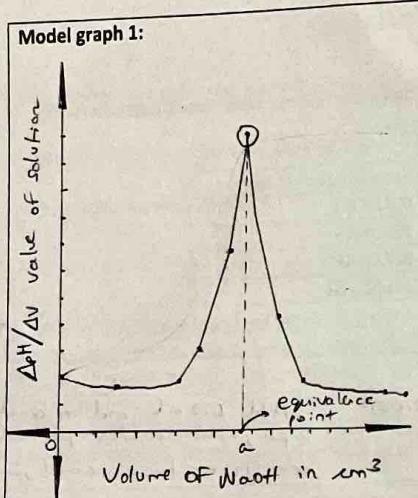
#### Model Procedure / Flow Chart:



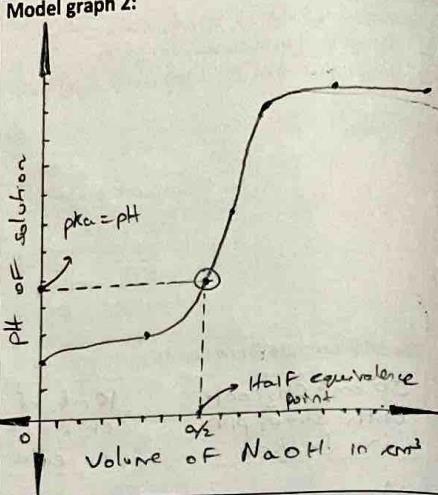
- Measuring the pH:
- ① Take  $50 \text{ cm}^3$  of  $\text{CH}_3\text{COOH}$  in a beaker and drop a stirring pin inside.
  - ② Insert the glass platinum electrode inside the given beaker and place it on the stirrer base.
  - ③ Take  $10 \text{ ml}$  of  $\text{NaOH}$  in a burette and mount it over the given beaker, on the iron stand.
  - ④ Ensure pH meter is set to pH mode, switch on the stirrer, ensure it doesn't collide with electrodes.
  - ⑤ Measure the initial reading, slowly add  $0.2 \text{ ml}$  of  $\text{NaOH}$  drop-by-drop and measure the pH.
  - ⑥ Continue till  $3.6 \text{ cm}^3$  of  $\text{NaOH}$  is added, record the jump in pH, and the following should be plotted.

- Graphical procedure:
- ① With the given pH values, find  $\Delta \text{pH}$  and  $\Delta V$  values for change in pH and volume of  $\text{NaOH}$  at every iteration. ( $\Delta V_{\text{NaOH}} = 0.2 \text{ cm}^3$ )
  - ② Except for the iteration, calculate  $\Delta \text{pH}/\Delta V$  value.
  - ③ plot  $\Delta \text{pH}/\Delta V$  vs  $V_{\text{NaOH}}$  graph, get the equivalence point value. Calculate the half equivalence point.
  - ④ plot  $\Delta \text{pH}$  vs  $V_{\text{NaOH}}$  graph, final pH at half equivalence point. At this point:  $pK_a = \text{pH}$ .

Model graph 1:



Model graph 2:



## Model Calculation:

For every iteration of  $0.2 \text{ cm}^3$  of NaOH, we need to measure change in pH =  $\Delta \text{pH}_{(k)}$  =  $\text{pH}(K+1) - \text{pH}(K)$

Change in Volume of NaOH =  $\Delta V = 0.2 \text{ cm}^3$  (constant)

Relative change of pH wrt Volume =  $\Delta \text{pH}/\Delta V$  for every iteration  
 $\Rightarrow \Delta \text{pH}/\Delta V = \frac{\Delta \text{pH}_{(k)}}{0.2}$  where  $K \neq 1$ .

Through the equivalence point, if get it as  $\psi \text{ cm}^3$  in the  $\Delta \text{pH}/\Delta V$  vs  $V_{\text{NaOH}}$  graph: equivalence point =  $\psi \text{ cm}^3$

Then half equivalent point =  $\psi/2 \text{ cm}^3$

plot the same  $\psi/2 \text{ cm}^3$  point in the pH vs  $V_{\text{NaOH}}$  graph, and identify the pKa value of the acid.

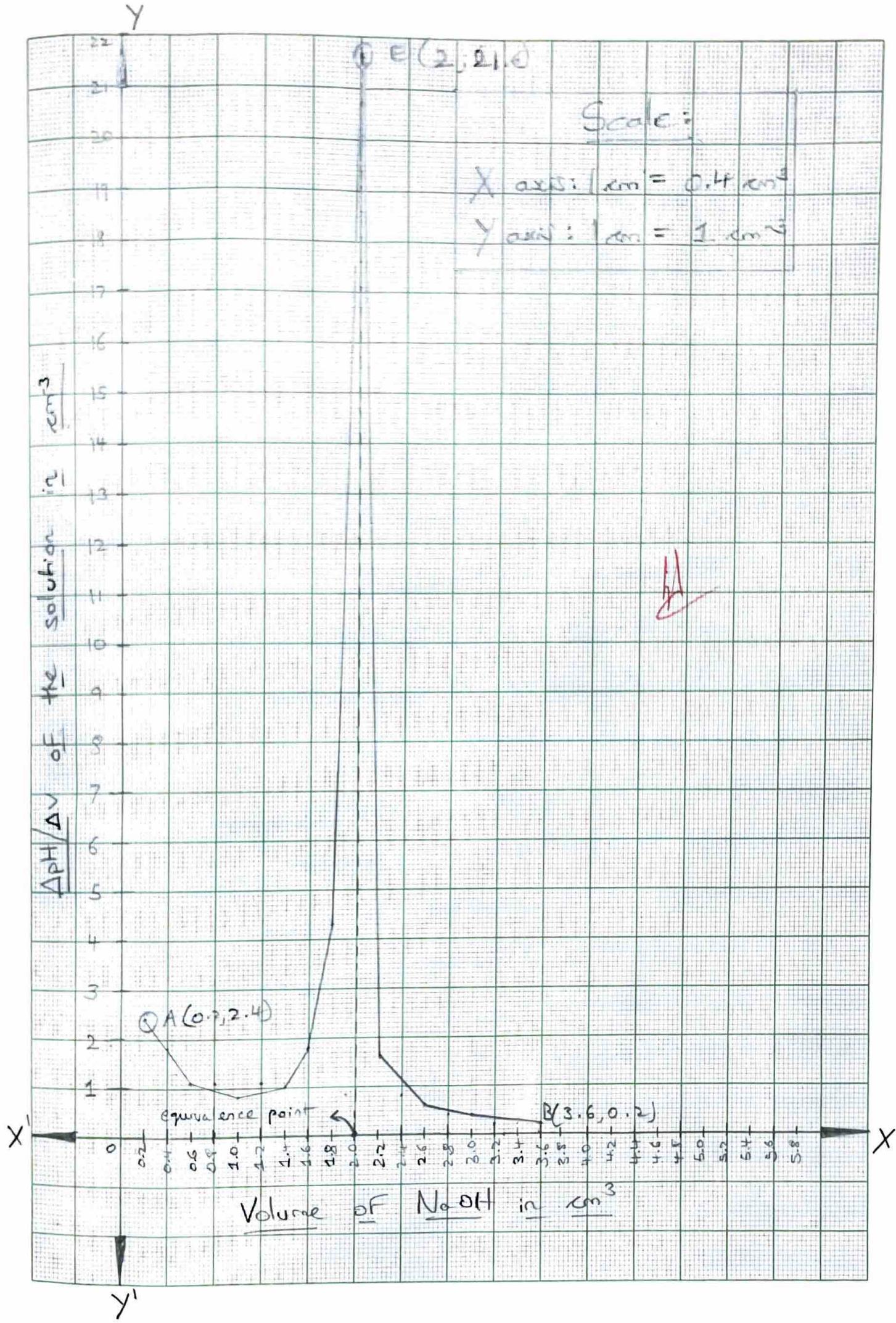
This according to the Henderson - Hasselbach Equation:

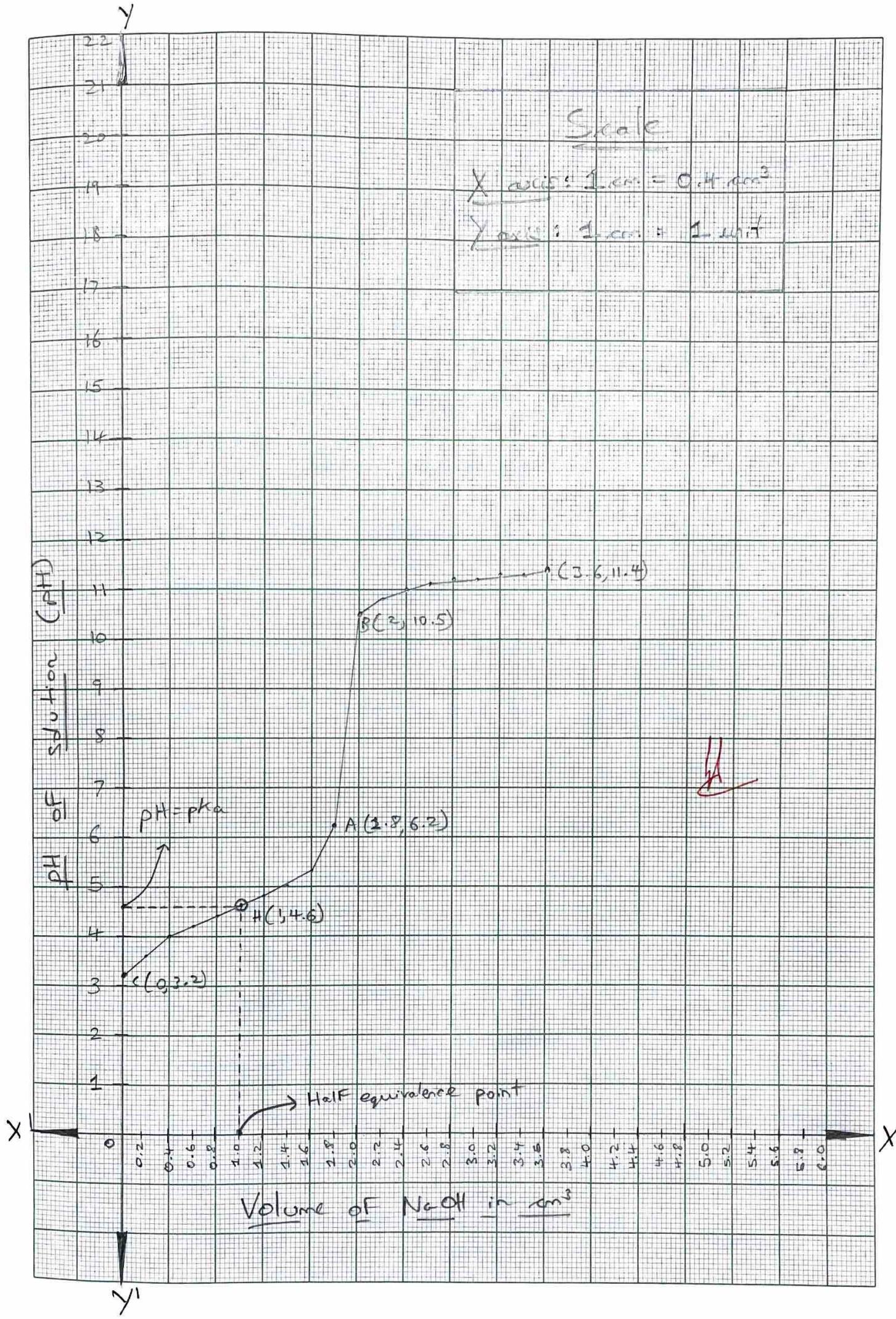
$$\text{pH} = \text{pka} + \log_{10} \frac{[\text{salt}]}{[\text{acid}]} \quad \text{where } \text{pka} = -\log_{10} \text{ka}$$

Hence at half equivalence point,  $\log_{10} \frac{[\text{salt}]}{[\text{acid}]} = 0$   
 as  $[\text{salt}] = [\text{acid}]$ . Hence at this point:  $\boxed{\text{pH} = \text{pka}}$

Tabulation:

Volume of NaOH added. in $\text{cm}^3$	pH	$\Delta \text{pH}$	$\Delta V$	$\frac{\Delta \text{pH}}{\Delta V}$
0.0	3.15	—	—	—
0.2	3.62	0.47	0.2	2.35
0.4	3.97	0.35	0.2	1.75
0.6	4.18	0.21	0.2	1.05
0.8	4.39	0.21	0.2	1.05
1.0	4.54	0.15	0.2	0.75
1.2	4.76	0.22	0.2	1.10
1.4	4.96	0.20	0.2	1.00
1.6	5.34	0.36	0.2	1.80
1.8	6.20	0.86	0.2	4.30
2.0	10.51	4.31	0.2	21.55
2.2	10.83	0.32	0.2	1.60
2.4	10.98	0.15	0.2	0.75
2.6	11.10	0.12	0.2	0.60
2.8	11.17	0.07	0.2	0.35
3.0	11.24	0.07	0.2	0.35
3.2	11.28	0.04	0.2	0.20
3.4	11.34	0.06	0.2	0.30
3.6	11.37	0.03	0.2	0.15

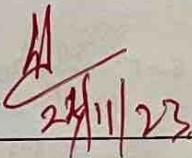




- Inference: Experimental inference:
- Using Henderson-Hasselbalch equation, we can find out pKa of an acid without other instrument.
  - The jump in pH is observed due to weak acid-strong base interaction. At this stage,  $\text{pH} = -\frac{1}{2} [\log K_a - \log K_b + \log C]$  at 298K.
  - Stronger the acid, lower is the pKa and higher is the  $K_a$ . As  $\text{pKa} = -\log(K_a)$ ,  $\text{pK}_b = -\log(K_b) \Rightarrow \boxed{\text{pKa} + \text{pK}_b = 14}$
  - At half equivalence point,  $[\text{salt}] = [\text{acid}]$  hence  $\text{pKa} = \text{pH}$ .
  - Calomel electrode used as reference, glass electrode as inert.

- Relevance to Society & Environment:
- Manufacture of wines, juice and soups require proper pH levels to be maintained to maintain uniformity.
  - Simulation of enzymes: To study the action of enzymes, under lab conditions by using proper pH liquids.
  - Manufacture of fertilisers: Appropriate acid mixtures and levels have to be maintained to manufacture various fertiliser.
  - Purification of water, honey and milk can be measured the acid content which is proportional to microorganism activity.
  - Soil pH: To maintain soil alkalinity, we can take samples and various sensors use the following application to measure.

Report: The acid content of the given soft drink = Normality of  $\text{CH}_3\text{COOH} \approx N_1 V_1 = N_2 V_2$   
 Hence we can see that  $\Rightarrow [0.00204N]$   $\boxed{\text{pKa found} = 4.6}$   
 and weight of  $\text{CH}_3\text{COOH} \approx 0.07438 \text{ g}$   $\rightarrow \boxed{\text{CH}_3\text{COOH}}$

Evaluation of experiment - 3		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation	9+7	16
Equivalence Point & Execution	16+4	19+
Inference & Societal Relevance	2+2	4
Total	40	39+ = 40
Signature of Teacher		

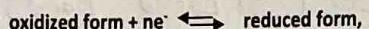
## EXPERIMENT NO - 4

Aim: Potentiometric estimation of iron

### Theory:

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

For the reaction,

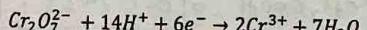
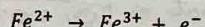


Nernst equation can be written as

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

Where  $E^\circ$  is the standard electrode potential and the terms within the brackets represent their concentrations.

It is evident from the equation that the potential depends upon the concentrations of the oxidised and the reduced species in the solution. When a reducing agent in solution is oxidized using an oxidizing agent, the concentrations and hence the potential changes. Potentiometry essentially involves the measurement of change in potential as and when a species in solution is oxidized or reduced. When a titration of a reducing agent is carried out against an oxidising agent, the potential gradually changes in the beginning but changes rapidly near the end point. A plot of change in potential against volume reveals a sudden change in potential at the equivalence point. This sudden change in the present experiment can be explained as follows. The reactions that take place when dichromate is added to ferrous solution are:



The potential of the system before the equivalence point is given by

$$E = E^\circ + \frac{0.0591}{1} \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

i.e., the potential is dependent on the concentration of  $Fe^{2+}$  and  $Fe^{3+}$  ions. The potential of the solution will be around 0.75V. At the equivalence point the potential is decided by the concentrations of  $Fe^{2+}$ ,  $Fe^{3+}$ ,  $Cr_2O_7^{2-}$  and  $Cr^{3+}$  ions. The potential at equivalence point is given by

$$E = \frac{E_{Fe^{3+}/Fe^{2+}}^\circ + E_{Cr^{6+}/Cr^{3+}}^\circ}{2}$$

$$= \frac{0.75V + 1.33V}{2} = 1.04V$$

Beyond the equivalence point as no ferrous ions exist, the potentials is determined by the concentration of  $Cr_2O_7^{2-}$  and  $Cr^{3+}$  ions and it is given by

$$E = E^\circ + \frac{0.0591}{6} \log \frac{[Cr^{6+}]}{[Cr^{3+}]}$$

$$= 1.33V + \frac{0.0591}{6} \log \frac{[Cr^{6+}]}{[Cr^{3+}]}$$

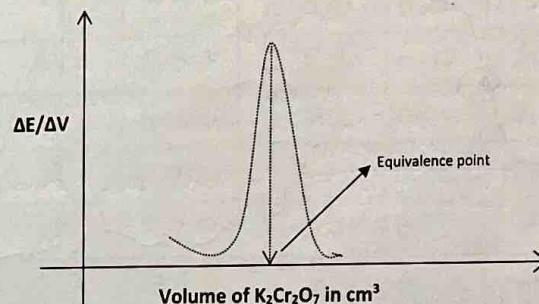
therefore, an abrupt increase in the potential is observed at the equivalence point. This increase marks the end point of the titration. Potential change at an electrode cannot be directly measured. The electrode at which the potential changes with concentration (indicator electrode) is connected to another electrode whose potential remains constant [ex:-calomel electrode (Standard reference electrode)], to form a cell.

$$\text{EMF} = E_{\text{cathode}} - E_{\text{anode}}$$

As the potential of the indicator electrode changes, the EMF of the cell also changes. It is the change in EMF that is measured during a potentiometric titration.

### Description:

FAS solution is prepared by dissolving known amount of FAS in 1 tt of dilute Sulphuric acid and little amount of water. Using the dispenser take 25 cm<sup>3</sup> of rust solution into a clean beaker. Add 1 test tube of dil. Sulphuric acid. Dip the electrode assembly into the solution and connect to a potentiometer. Measure the potential. Add 0.2 cm<sup>3</sup> of Potassium dichromate from a burette. Stir the solution well and measure the potential. Continue the process till the potential shows a tendency to increase rapidly. Now add dichromate in increments of 0.2 cm<sup>3</sup> and measure the potential after each addition. Plot a graph of  $\Delta E/\Delta V$  against volume of dichromate added as shown in the figure and find out the end point. Calculate the normality of the ferrous solution and determine the amount of iron in the given volume.



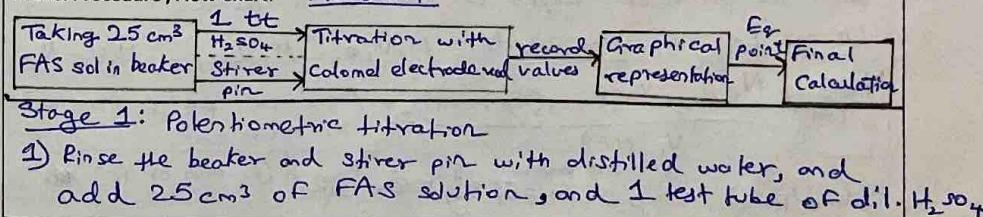
### Calculation:

Volume of  $K_2Cr_2O_7$  required for the reaction =  $V$  cm<sup>3</sup> (From graph),

$$N_{\text{Iron}} = \frac{NV_{K_2Cr_2O_7}}{V_{\text{Iron}}}$$

Amount of iron presented in 1000 cm<sup>3</sup> of its solution =  $N_{\text{iron}} \times \text{gram equivalent weight of Iron} = \dots \text{B g}$

### Model Procedure /Flow Chart: Flowchart:



- 2) Using a burette, filled with  $K_2Cr_2O_7$  sol, mount the following on iron stand, and place beaker on shaker base.  
 3) Place the calomel electrode in the beaker, and commence titration, noting down the EMF at every  $\Delta V = 0.2 \text{ cm}^3$ .

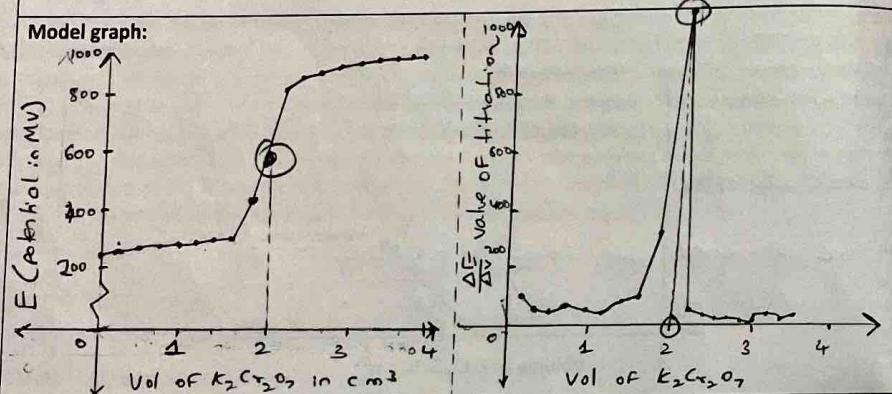
Stage 2: Graphical representation:

- 1) After noting down the values, calculate values of  $\Delta E$ ,  $\Delta E/\Delta V$  at every stage.  
 2) Plot two graphs:  $\Delta E$  vs Vol of  $K_2Cr_2O_7$ ,  $\Delta E/\Delta V$  vs vol of  $K_2Cr_2O_7$ .

Stage 3: Calculation

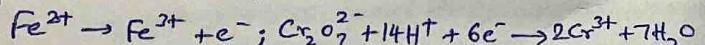
- 1) Calculate the value of iron present in  $1000 \text{ cm}^3$  of FAS.

Model graph:



Model Calculation: We know:  $N_{K_2Cr_2O_7} = 0.049 N$

The following reactions take place:



We see that in Nernst equation:

$$E = E_0 - \frac{0.0591}{n} \log \left( \frac{[M^+]}{[N^-]} \right)$$

Hence change in the EMF is measured. We find out equivalence point as peak of  $\Delta E/\Delta V$  vs Vol  $K_2Cr_2O_7$  curve.

Using Law of Equivalents:  $M_1 V_1 e_1 = M_2 V_2 e_2 \Rightarrow N_1 V_1 = N_2 V_2$

$$\frac{N_{Fe}}{(FAS)} = \frac{N_{K_2Cr_2O_7} V_{K_2Cr_2O_7} (\text{equivalent})}{V_{Fe} (\text{FAS})} \Rightarrow \text{Then we for finding mass in } 1000 \text{ cm}^3.$$

$$\text{Here: } V_{Fe} = 25 \text{ cm}^3, \text{ of Fe} = 55.85 \text{ g} \quad \frac{N_{Fe}}{(FAS)} \times (E_{eq, \text{mass of Fe}})$$

Tabulation:

Volume of $K_2Cr_2O_7$ added in $\text{cm}^3$	Potential (E) in mV	$\Delta E$	$\Delta V$	$\Delta E/\Delta V$
0.0	240	—	—	—
0.2	264	24	0.2	120
0.4	277	13	0.2	65
0.6	288	11	0.2	55
0.8	300	12	0.2	60
1.0	311	11	0.2	55
1.2	321	10	0.2	50
1.4	336	15	0.2	75
1.6	357	21	0.2	105
1.8	429	72	0.2	360
2.0	807	378	0.2	1890
2.2	820	13	0.2	65
2.4	828	8	0.2	40
2.6	832	4	0.2	20
2.8	836	4	0.2	20
3.0	838	2	0.2	10
3.2	839	1	0.2	5
3.4	841	2	0.2	10
3.6	842	1	0.2	5

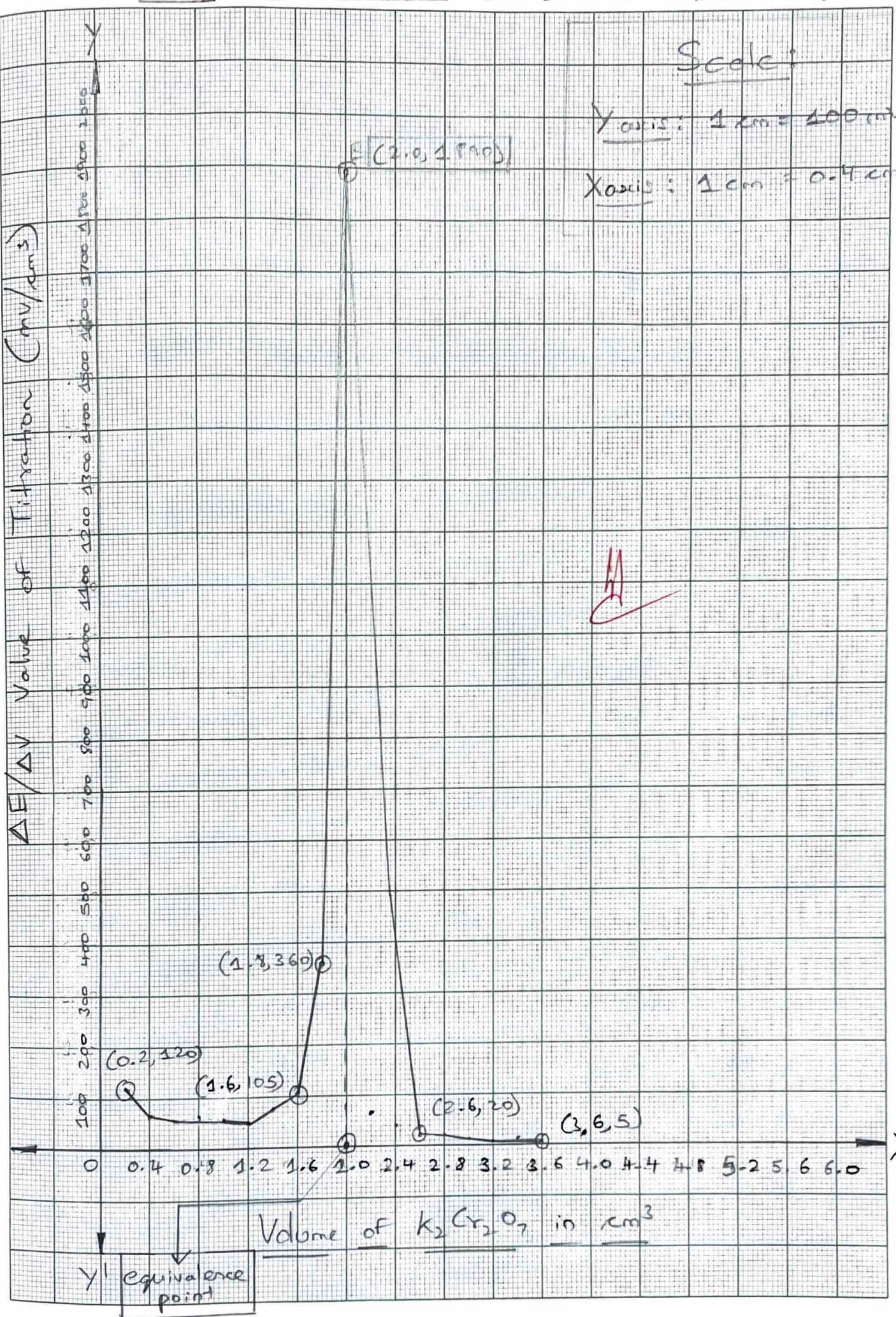
Graph of  $\Delta E/\Delta V_{(PA)}$  vs Volume of  $k_2Cr_2O_7$

Scale:

Y-axis:  $1 \text{ cm} = 100 \text{ mV/cm}^3$

X-axis:  $1 \text{ cm} = 0.4 \text{ cm}^{-1}$

H



Calculation: By the following graph we infer that equivalence point occurs at  $25 \text{ cm}^3$  of  $\text{K}_2\text{Cr}_2\text{O}_7$ .  $\Rightarrow [2 \text{ cm}^3]$

Using law of equivalents:  $N_{\text{Fe}} \cdot V_{\text{Fe}} = N_{\text{K}_2\text{Cr}_2\text{O}_7} \cdot V_{\text{K}_2\text{Cr}_2\text{O}_7}$

We know:  $N_{\text{K}_2\text{Cr}_2\text{O}_7} = 0.049 \text{ N}$ , Eq point  $V_{\text{K}_2\text{Cr}_2\text{O}_7} = \frac{2}{3} \text{ cm}^3 + 2 \text{ cm}^3$

$V_{\text{K}_2\text{Cr}_2\text{O}_7}$  used =  $3.6 \text{ cm}^3$ ,  $V_{\text{Fe(FAS)}} = 25 \text{ cm}^3$ .

$$\text{Hence: } N_{\text{Fe(FAS)}} = \frac{N_{\text{K}_2\text{Cr}_2\text{O}_7} \cdot V_{\text{K}_2\text{Cr}_2\text{O}_7}}{V_{\text{Fe(FAS)}}} = \frac{(0.049)(2)}{(25)}$$

$$= 0.00392 \text{ N}$$

Amount of iron present in  $1000 \text{ cm}^3$  of FAS

$$= N_{\text{Fe(FAS)}} \times \text{Eq. Weight(Fe)} = 0.00392 \times 55.85$$

$$\therefore \boxed{\alpha} = \boxed{0.21893 \text{ g}}$$

$$\alpha = \frac{\beta \gamma}{8}$$

where:  $N_{\text{Fe(FAS)}} = \alpha$ ,  $V_{\text{K}_2\text{Cr}_2\text{O}_7} = \beta$  Iron present =  $\gamma$

$N_{\text{K}_2\text{Cr}_2\text{O}_7} = \gamma$ ,  $V_{\text{Fe(FAS)}} = \delta$  Eq. weight =  $\eta$  of Fe

$$\gamma = \alpha \eta$$

- Inference: Experimental titration inference:
- ① A sudden increase of EMF value is noted at a point called equivalence point.
  - ② This peak helps determine exact mass of iron in the FAS solution. (keeping in mind the addition of little sulphuric acid in order to prevent oxidation of FAS).

- Observational inference:
- ① We see that sudden jump indicates maximum electric current flow through the medium.
  - ② By calculating normality and equivalence point, we can determine the accurate mass of Fe in solution upto 4 decimals.

Relevance to Society & Environment: OE-waste management: A given sample can be tested using this experiment, in order to commercially extract Iron to the highest quality.

③ Drug sampling using blood: Blood contains Fe complex surrounded by EDTA Ligands, which can determine RBC count per 1cc blood. Similarly drugs which contain iron complexes are required to be at a maximum normality, which could be serious if exceeded.

3) Degree of Galvanisation: The extent upto which minimum  $R_{\text{ext}}$  can be generated can be tested using the following experiment, under various anti-rust agents like Zn coating.

4) Iron-ore detection: Using soil sample at a location, test Fe content, in order to detect presence of ore.

Report: Amount of iron present in  $1000 \text{ cm}^3$  of FAS solution = 6 Eq point  $\rightarrow [2 \text{ eq}]$   
Iron present:   $1000 \text{ cm}^3$  OF FAS solution  
 Iron present is: 0.21893 g

Evaluation of experiment - 4		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation	9 + 7	14 + 1
Equivalence Point & Execution	16 + 4	18 + 1
Inference & Societal Relevance	2 + 2	3 + 1
Total	40	35 + 2 = 38 + 1
Signature of Teacher	A 10/10/23	(58) 40

## EXPERIMENT NO - 5

Aim: Conductometric estimation of strong acid with strong base

### Theory:

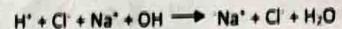
Conductance of any solution (C) is the ease with which current flows through it. It is the reciprocal of resistance (R) offered by the solution.

$$C = 1/R$$

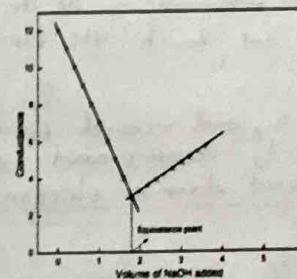
Conductance of a solution depends on number of ions, charge on the ions and also on the mobility of the ions. During titration, the conductance of the solution may change due to any of the above reasons. Such titrations in which conductance measurements are made use of in determining the equivalence 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 is the maximum, before the addition of NaOH. 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. After the neutralization of all hydrogen ions of strong acid, sodium hydroxide solution will neutralize hydrogen ions of acetic acid. 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 vs. volume of NaOH (added) is given below.



The intersection of two lines corresponds to the equivalence point corresponding to strong acid. From the volume of NaOH required to neutralize HCl, the amount of HCl is calculated.

### Description:

Pipette out  $50 \text{ cm}^3$  of acid into a clean  $200 \text{ cm}^3$  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  $0.2 \text{ cm}^3$  of NaOH solution into the beaker and shake well. Note down the conductance. Note down the conductance by

adding  $0.2 \text{ cm}^3$  of NaOH each time to the beaker. Conductance decreases in the beginning and finally starts increasing by large value. Take around 8-10 readings on the increasing side. Draw a graph of conductance against volume of NaOH added. The equivalence point in the graph corresponds to the neutralization of HCl. Calculate the normality and amount of HCl present in  $1000 \text{ cm}^3$  of its solution.

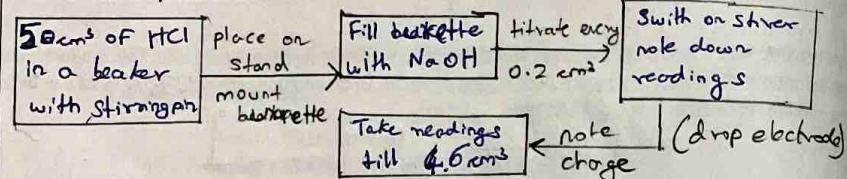
**Calculation:**

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

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

Amount of HCl or  $\text{CH}_3\text{COOH}$  present in  $1000 \text{ cm}^3$  of its solution =  $N_{\text{acid}} \times$  gram equivalent weight of acid

**Model Procedure / Flow Chart:**

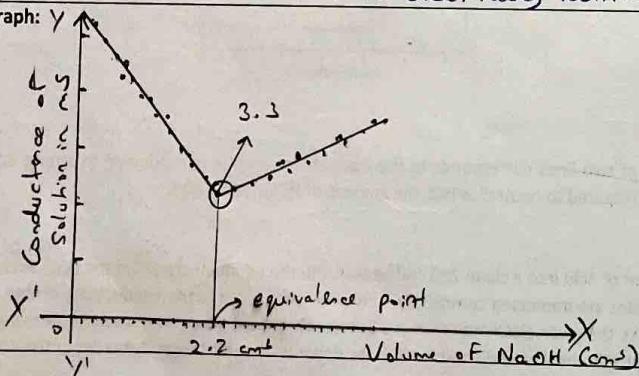


- Arrangement of apparatus:
- ① In a  $500 \text{ ml}$  beaker, take  $50 \text{ cm}^3$  HCl and drop a stirring pin inside.
  - ② Take a burette, and fill it with  $100 \text{ cm}^3$  of NaOH
  - ③ Place the beaker on the magnetic stirrer stand, place the mounted burette just above the beaker.
  - ④ Place the platinum electrodes inside the beaker, ensure that stirrer does not touch the electrode

Reading:

- ① Switch on the stirrer, and record first reading, slowly add NaOH drop-by-drop, and record after  $0.2 \text{ cm}^3$
- ② Ensure NaOH doesn't drop on electrode, continue till  $4.6 \text{ cm}^3$

Model graph:



**Model Calculation:** Using law of Equivalents, we know:  $M_1 V_1 e_1 = M_2 V_2 e_2$

$$\text{Hence, } N_1 V_1 = N_2 V_2 \Rightarrow N_{\text{HCl}} V_{\text{HCl}} = N_{\text{NaOH}} V_{\text{NaOH}}$$

$$\alpha_H f_H = \alpha_N f_N \Rightarrow \alpha_H = \frac{\alpha_N f_N}{f_H}$$

(we know:  $N_{\text{NaOH}} = \alpha_N = 0.051 \text{ N}$ )  
 $f_H = f_N = 25 \text{ cm}^3 \times 2$

Using this: Amount of HCl in  $1000 \text{ cm}^3$  =  $W_{\text{HCl}} = N_{\text{HCl}} \times \text{GEW}_{\text{HCl}}$

$$W_{\text{HCl}} = \alpha_H \times \gamma_H = \frac{\alpha_N f_N}{f_H} \times 36.461 \quad \left[ N_{\text{HCl}} = \frac{0.051 \times V_{\text{NaOH (eq)}}}{25 \times 2} \right]$$

(we know:  $\gamma_H = \text{Gram equivalent weight of HCl} = 36.461 \text{ g/mol}$ ) Amount<sub>HCl</sub> =  $N_{\text{HCl}} \times 36.461$

**Tabulation:**

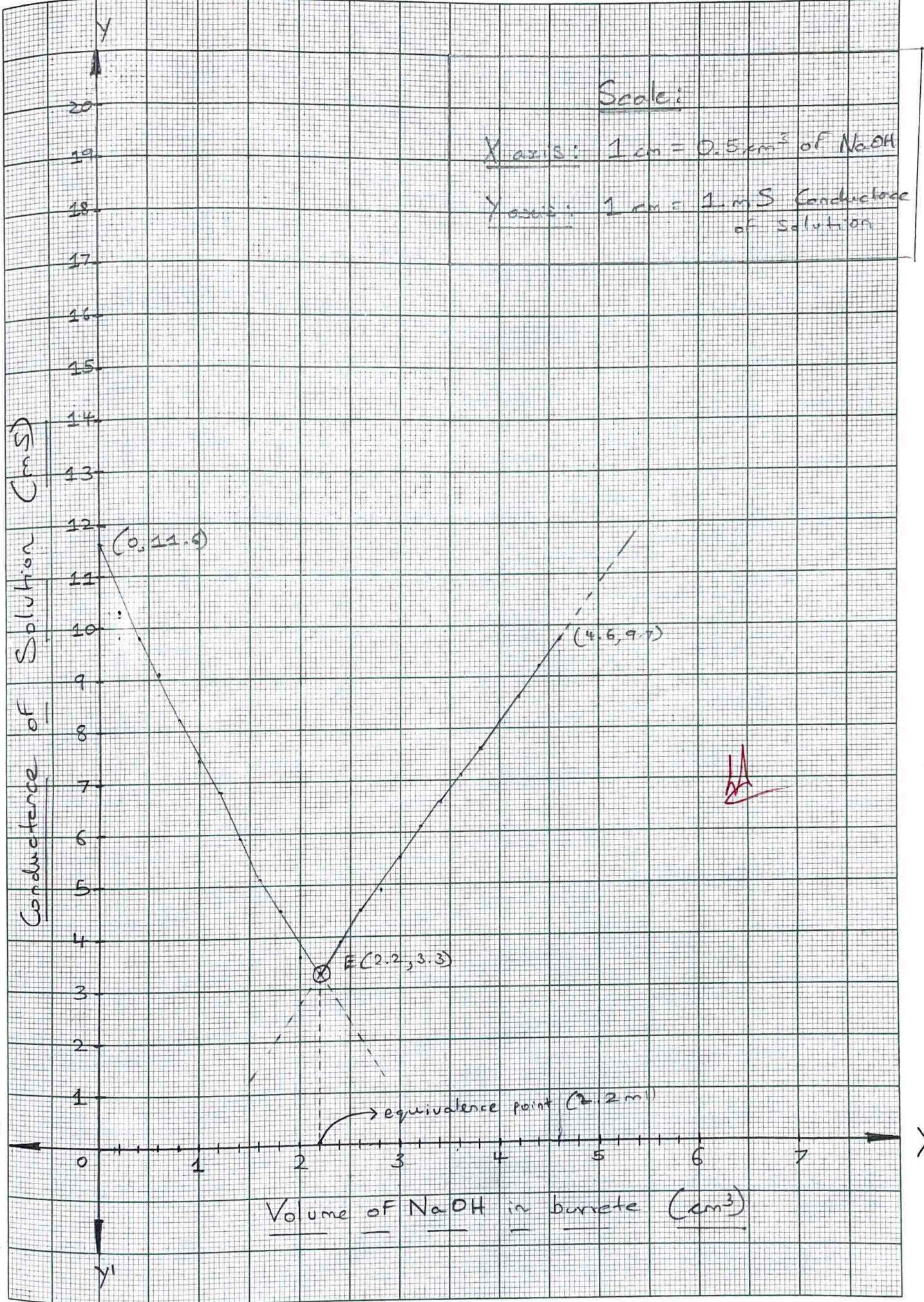
Volume of NaOH in $\text{cm}^3$	Conductance in mS
0.0	11.5
0.2	10.3
0.4	9.8
0.6	9.1
0.8	8.2
1.0	7.4
1.2	6.8
1.4	5.9
1.6	5.1
1.8	4.5
2.0	3.6
2.2	3.3
2.4	3.9
2.6	4.5
2.8	4.9
3.0	5.5

Volume of NaOH in $\text{cm}^3$	Conductance in mS
3.2	6.1
3.4	6.6
3.6	7.1
3.8	7.6
4.0	8.1
4.2	8.6
4.4	9.2
4.6	9.7
4.8	10.2
5.0	10.7
5.2	11.2
5.4	11.7
5.6	12.1
5.8	12.6
6.0	13.1

Scale:

X axis: 1 cm =  $0.5 \text{ cm}^3$  of NaOH

Y axis: 1 cm = 1 mS Conductance  
of Solution



Calculation: Using the given the formulae:  $N_{HCl} = \frac{0.051 \times V_{NaOH}(\text{cc})}{25 \times 2}$

$$N_{HCl}(\text{acid}) = \frac{0.051 \times 2.2}{50} = 0.00224 \text{ N}$$

We know,  $N_{HCl}(\text{acid}) = 0.00224 \text{ N}$

Amount of HCl present  $\text{in } 1000 \text{ cm}^3 \text{ sol} = N_{HCl}(\text{acid}) \times 36.461$

Amount (HCl)  $\text{in } 1000 \text{ cm}^3 = 0.00224 \times 36.461 = 0.08167 \text{ g}$

Hence we got. Normality of HCl = 0.00224 N

and Amount of HCl present = 0.08167 g  
in 1000 cm<sup>3</sup> solution

Inference: Experimental and graphical inference:

- ① By observing the graph, we see that conductance and volume of NaOH are linearly dependent. (Slope =  $\Delta S / \Delta V$ )
- ② The graph continuous and the minima of the following function gives the equivalence point.  $E(V) = \begin{cases} m_1 V + c_1 & \rightarrow \text{continuous but} \\ m_2 V + c_2 & \text{non-differentiable} \end{cases}$

Observational inference:

- ① Amount of HCl can be found out using, law of equivalents, which also corresponds to Debye-Hückel-Onsager equation:  $A_m = A_m^0 - AVC$
- ② As HCl is a strong acid, it has a linear relation with volume, and by experimentation, it turns out to be: 0.01% w/v solution.

**Relevance to Society & Environment:** ① Water purification: presence of high acidic levels can be accurately measured using conductivity. Drinking water should be between 7-8 pH.

② To predict conductivity: To find out conductivity of a given liquid, conductivity can be used to measure accurately at certain conditions such as temperature, cell constant.

③ Cyclic Voltammetry: In the process cyclic voltammetry, estimation of levels of Iron-glycine complex, conductivity is used.

④ Iso-electric point in protein: Manufacturing drugs require conductivity as it will maintain safe level of drug intake.

**Report:**

1. Normality of HCl = ..... N → 0.00224 N

2. Amount of HCl present in 1000 cm<sup>3</sup> of its solution = ..... g → 0.08167 g

Evaluation of experiment - 5		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation	9 + 7	<u>16</u>
Equivalence Point & Execution	16 + 4	<u>19 + 1</u>
Inference & Societal Relevance	2 + 2	<u>4</u>
Total	40	<u>39/40</u>
Signature of Teacher	<u>H</u> <u>17/11/23</u>	

## EXPERIMENT NO – 6

**Aim:** Conductometric estimation of acid mixture

**Theory:**

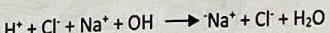
Conductance of any solution (C) is the ease with which current flows through it. It is the reciprocal of resistance (R) offered by the solution.

$$C = 1/R$$

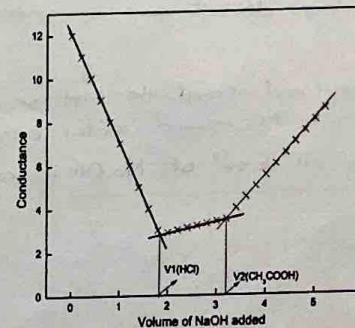
Conductance of a solution depends on number of ions, charge on the ions and also on the mobility of the ions. During the course of a titration, the conductance of the solution may change due to any of the above reasons. Such titrations in which conductance measurements are made use of in determining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

Among all Conductometric titrations, reaction involving strong acid (HCl)/ weak acid (CH<sub>3</sub>COOH) 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 is the maximum, before the 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. After the neutralization of all hydrogen ions of strong acid, sodium hydroxide solution will neutralize hydrogen ions of acetic acid. 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 vs. volume of NaOH (added) is given below.



The break in the curve corresponds to the two equivalence points corresponding to two acids. From the volume of NaOH required to neutralize HCl or CH<sub>3</sub>COOH, the amount of HCl or CH<sub>3</sub>COOH is calculated.

**Description:**

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

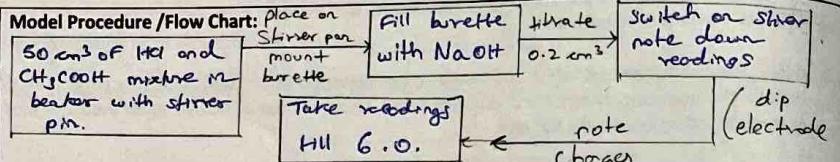
NaOH solution into the beaker and shake well. Note down the conductance. Note down the conductance by adding 0.2 cm<sup>3</sup> of NaOH each time to the beaker. Conductance decreases in the beginning, increases slightly and finally starts increasing by large value. Take around 8-10 readings on the increasing side. Draw a graph of conductance against volume of NaOH added. 1<sup>st</sup> equivalence point in the curve corresponds to the neutralization of HCl and the difference of two equivalence point corresponds to the neutralization of CH<sub>3</sub>COOH acid. Calculate the normality and amount of HCl and CH<sub>3</sub>COOH present in 1000 cm<sup>3</sup> of its solution.

#### Calculation:

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

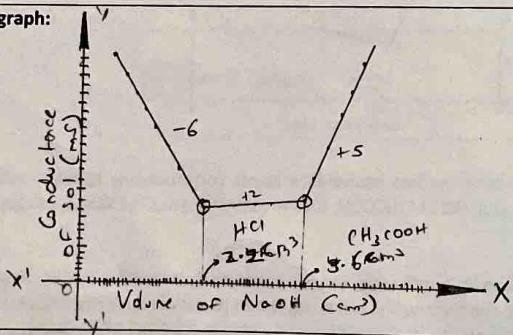
$$N_{\text{acid}} = \frac{(NV)_{\text{NaOH}}}{V_{\text{acid}}}$$

Amount of HCl or CH<sub>3</sub>COOH present in 1000 cm<sup>3</sup> of its solution = N<sub>acid</sub> × gram equivalent weight of acid



- Arrangement of Apparatus
- ① In a 500 ml beaker, take 50 cm<sup>3</sup> mixture of HCl and CH<sub>3</sub>COOH and drop a stirring pin
  - ② Take a burette, and fill it with 100 cm<sup>3</sup> NaOH!
  - ③ Place the beaker on the magnetic stirrer stand mounted with the NaOH filled beaker.
  - ④ Place the platinum electrode inside the beaker, ensure that stirrer or the NaOH drops do not come in contact with the electrode.
- Reading:
- ① Switch on the stirrer, and record the first reading, slowly drop NaOH into the mixture and record after every 0.2 cm<sup>3</sup>.
  - ② Continue the following till 6 cm<sup>3</sup> of NaOH is consumed.

#### Model graph:



**Model Calculation:** Using law of equivalents:  $M_1 V_1 e_1 = M_2 V_2 e_2 \Rightarrow N_1 V_1 = N_2 V_2$

Here we have two acids: HCl and CH<sub>3</sub>COOH. Let N<sub>acid</sub> =  $N_{\text{NaOH}} V_{\text{NaOH}}$

Let N<sub>acid</sub> of HCl =  $N_{\text{HA}} = \alpha_{\text{HA}}$  (V<sub>NaOH</sub> = β<sub>H</sub> = 2.2 cm<sup>3</sup> (equivalence))

Let N<sub>acid</sub> of CH<sub>3</sub>COOH =  $N_{\text{CA}} = \alpha_{\text{CA}}$  (V<sub>NaOH (CH₃COOH)</sub> = β<sub>C</sub> = 3.6 cm<sup>3</sup> (equivalence))

$N_{\text{NaOH}} = \alpha_N = 0.051 \text{ N}$ ,  $V_{\text{acid}} = \beta_A = 80 \text{ cm}^3$

$$N_{\text{HA}} = \alpha_{\text{HA}} = \frac{\beta_{\text{H}} \times \alpha_N}{\beta_A} = \frac{0.051 \times \beta_{\text{H}}}{50}$$

$$N_{\text{CA}} = \alpha_{\text{CA}} = \frac{\beta_{\text{C}} \times \alpha_N}{\beta_A} = \frac{0.051 \times \beta_{\text{C}}}{50}$$

Amount of HCl = γ<sub>H</sub> = N<sub>HA</sub> × GEW<sub>HCl</sub>

$$\gamma_H = \alpha_{\text{HA}} \times 36.461$$

Amount of CH<sub>3</sub>COOH = γ<sub>C</sub> = N<sub>CA</sub> × GEW<sub>CH₃COOH</sub>

$$\gamma_C = \alpha_{\text{CA}} \times 60.05$$

#### Tabulation:

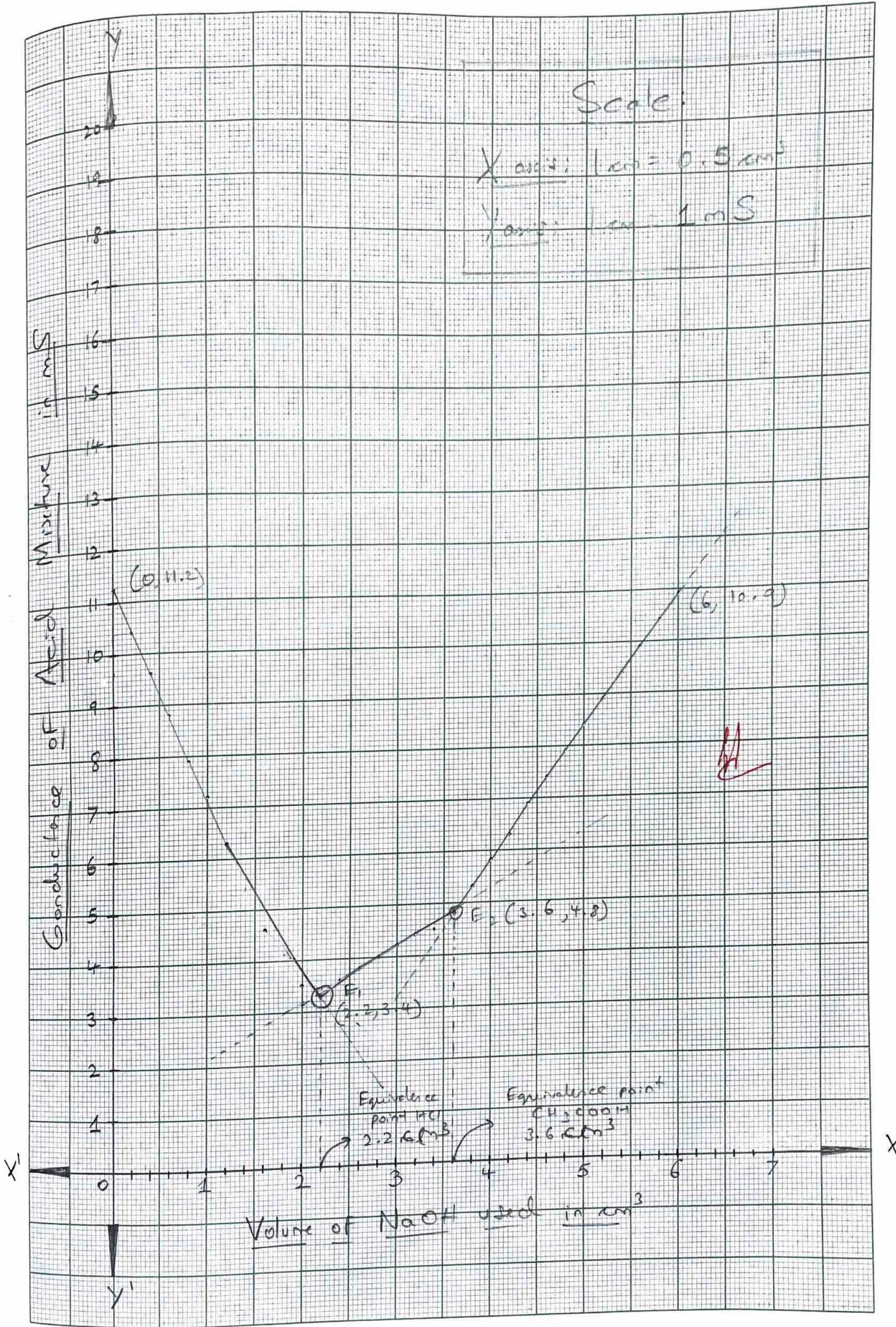
Volume of NaOH in cm <sup>3</sup>	Conductance in mS
0.0	11.2
0.2	10.4
0.4	9.6
0.6	8.8
0.8	8.0
1.0	7.2
1.2	6.3
1.4	5.4
1.6	4.6
1.8	4.1
2.0	3.5
2.2	3.4
2.4	3.6
2.6	3.8
2.8	4.0
3.0	4.2

Volume of NaOH in cm <sup>3</sup>	Conductance in mS
3.2	4.4
3.4	4.5
3.6	4.8
3.8	5.3
4.0	5.8
4.2	6.3
4.4	6.9
4.6	7.4
4.8	7.9
5.0	8.4
5.2	8.9
5.4	9.4
5.6	9.9
5.8	10.4
6.0	10.9

Scale.

X axis:  $1 \text{ cm} = 0.5 \text{ cm}^3$

Y axis:  $1 \text{ cm} = 1 \text{ mS}$



Calculation: Using the given formula:  $N_{HCl} = \frac{0.051 \times V_{NaOH(HCl)}}{50}$

$$N_{HCl} = \frac{0.051 \times 2.2}{50} = 0.00224 N$$

Amount of HCl =  $N_{HCl} \times 36.461 = 0.00224 \times 36.461$   
in 1000 cm³ =  $\underline{\underline{0.08167 \text{ g}}} \text{ of HCl}$   
of solution

Using the given formula:  $N_{CH_3COOH} = \frac{0.051 \times V_{NaOH(CH_3COOH)}}{50}$

$$N_{CH_3COOH} = \frac{0.051 \times 1.4}{50} = 0.001428 N \rightarrow 0.001428 N$$

Amount of CH<sub>3</sub>COOH =  $N_{CH_3COOH} \times 60.050 = \underline{\underline{0.00367}} \times 60.050$   
in 1000 cm³ =  $\underline{\underline{0.22038 \text{ g}}} \text{ of CH}_3\text{COOH}$   
of solution =  $\underline{\underline{0.08575 \text{ g}}}$

Normality of HCl in solution = 0.00224 N

Amount of HCl in 1000 cm³ solution = 0.08167 g

Normality of CH<sub>3</sub>COOH in solution = ~~0.00367~~ 0.001428 N

Amount of CH<sub>3</sub>COOH in 1000 cm³ solution = ~~0.22038~~ 0.08575 g

Inference: Experimental and graphical inference: ① We see that the graph has two sharp points, indicating two equivalence points, and conductance linearly dependent on volume of NaOH used  
 (slope,  $m = \Delta S / \Delta V$ )

② The graph is continuous but not differentiable at the two sharp points, and it made up 3 linear equations:  $S(V) = \begin{cases} m_1 V + c_1 \\ m_2 V + c_2 \\ m_3 V + c_3 \end{cases}$

Observational inference: ① Equivalence point of HCl is attained first as its normality is lower compared to CH<sub>3</sub>COOH, and HCl is strong acid  
 ② The angle formed by two lines will give relative change in increase or decrease in range of conductance:  $\phi = \tan^{-1} \left( \frac{m_1 - m_2}{1 + m_1 m_2} \right)$

**Relevance to Society & Environment:** ① Purity of liquids: By determining equivalence point, we can predict the acid present, especially cleaning of water to make it drinkable between 7-8 pH.

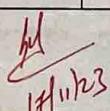
② Manufacture of drugs: Appropriate amount to be added can be calculated using conductometry.

③ Explanation of acidic organic compounds: Organic compounds can be acidic based on factors like mesomeric, hyperconjugation and electron donating or withdrawing nature.

④ Quality maintenance in industries: Industries manufacturing acids, need to maintain proper pH in liquid, conductivity is useful here.

**Report:**

1. Normality of HCl = ..... N, Normality of  $\text{CH}_3\text{COOH}$  = ..... N  
 2. Amount of HCl present in  $1000 \text{ cm}^3$  of its solution = ..... g  
 3. Amount of  $\text{CH}_3\text{COOH}$  present in  $1000 \text{ cm}^3$  of its solution = ..... g

Evaluation of experiment - 6		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation	9 + 7	16
Equivalence Point & Execution	16 + 4	19
Inference & Societal Relevance	2 + 2	4
Total	40	39
Signature of Teacher	 18/11/23	

## EXPERIMENT NO - 7

**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.

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

5.61 36.574

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

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

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

### Description:

Take a clean and dry viscometer. Fix it vertically to a stand. Using graduated pipette, transfer known volume (say  $10 \text{ cm}^3$ ) of the given liquid into the viscometer. Then it is placed in a 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 stopwatch 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.

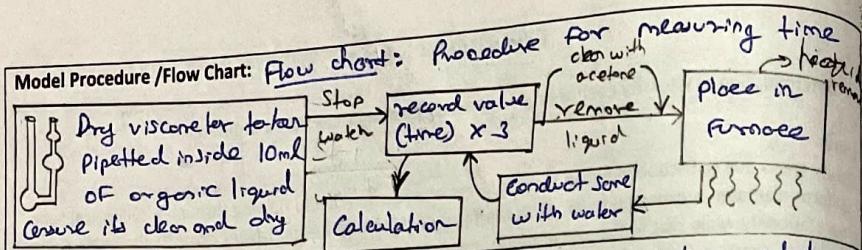
Remove the liquid from the viscometer, clean well with acetone and dry it in oven. Cool the viscometer to room temperature. Similarly, measure the time of flow of water by taking the same volume. Calculate average time of flow of water. Note down the room temperature. Knowing the density, viscosity of water and density of the given liquid, viscosity of the liquid can be calculated using the formula given below.

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

$t_w = 27.5 - 300.5$

(1 milli poise =  $10^{-4} \text{ Nsm}^{-2}$ )

$9.5 \times 0.8617 \times 54.873 \times 9.1 \times 10^{-4} \times 300.5 \times 71.667 \times 1$



- For organic solvent: ① Clean, dry viscometer was taken and placed in a room temperature water bath using an iron stand. ② 10 ml of organic liquid was pipetted and poured through one column of the device. ③ Suction apparatus (which consists of a rubber pipe and bulb) was attached to the other end while compressed, which on slow release increase the liquid column. ④ Application of atmospheric pressure pushes the liquid through the bulbs, and time taken by it to cross was measured, and same procedure repeated thrice.

Furnace use and dry: The organic liquid was removed, and acetone passed through the columns and the apparatus was placed in the furnace for about 5 min, then let cool for 5 min

- For water: ① 10 ml of water was pipetted and added to fresh, clean viscometer (keep in mind, same viscometer should be used). ② Similar procedures were followed as above, and observations made.

#### Model Calculation:

Coefficient of viscosity ( $\eta$ ) is unique for every liquid and depends on temperature and atmospheric pressure.

We can use Poiseuille's formula in order to find its value for the liquid.

From the velocity gradient formula we know:

$$\text{Hence Poiseuille's formula can be extracted as: } \eta = \frac{\pi r^4 P t}{8 L}$$

Hence for relative measurement we can

$$\text{utilise: } \frac{\eta_{\text{water}}}{\eta_{\text{liquid}}} = \frac{t_{\text{water}} d_{\text{water}}}{t_{\text{liquid}} d_{\text{liquid}}} \Rightarrow \eta_{\text{liquid}} = \frac{t_{\text{liquid}} d_{\text{liquid}} \eta_{\text{water}}}{t_{\text{water}} d_{\text{water}}} \quad \eta_{\text{water}} = \frac{\pi \beta r^2}{8L} = \frac{8.5 \times 10^{-4}}{8L}$$

where:  $\eta_{\text{liquid}} = \sigma$  = coefficient of viscosity of liquid;  $t_{\text{liquid}}$  = time of liquid  $\eta_{\text{water}}$ ;  $\eta_{\text{water}} = \sigma$  = coefficient of viscosity of water;  $t_{\text{water}}$  = time of water  $\eta_{\text{water}}$ ;  $d_{\text{liquid}} = \text{density of liquid} = 1.0$ ;  $d_{\text{water}} = \text{density of water} = 1.0$

#### Observation:

Liquid	Time of Flow		
	in min: sec	in seconds	Average in seconds
Test liquid	00:54.72	54.72 s	$t = 54.8733 s$
	00:54:91	54.91 s	
	00:54:99	54.99 s	
Water	01:11:59	71.59 s	$t_w = 71.6667 s$
	01:11:96	71.96 s	
	01:11:75	71.75	

#### Calculation:

$$\text{By the following observation we see: Average time taken by water} \\ = t_{\text{water}} = 71.6667 s$$

$$\text{Average time taken by liquid} = t_{\text{liquid}} = 54.8733 s$$

$$t_{\text{liquid}} = \frac{\sum t_i (\text{liquid})}{3} = \frac{54.72 + 54.91 + 54.99}{3} = \frac{164.62}{3} = 54.8733 s$$

$$t_{\text{water}} = \frac{\sum t_i (\text{water})}{3} = \frac{71.59 + 71.96 + 71.75}{3} = \frac{215.3}{3} = 71.6667 s$$

We know:  $\eta_{\text{water}}$  at room temperature ( $27.5^\circ C$ ) =  $8.5 \times 10^{-4} \text{ Ns m}^{-2}$

$d_{\text{water}}$  = density of water at  $27.5^\circ C \approx 0.9989 \text{ kg m}^{-3}$

$d_{\text{liquid}}$  = density of liquid at  $27.5^\circ C = 0.8617 \text{ kg m}^{-3}$

$\eta_{\text{water}}$  = coeff of viscosity of water at  $27.5^\circ C = 8.51 \times 10^{-4} \text{ Ns m}^{-2}$

Hence coefficient of viscosity of liquid =  $\eta_{\text{liquid}} = \frac{t_{\text{liquid}} \cdot d_{\text{liquid}} \cdot \eta_{\text{water}}}{t_{\text{water}} \cdot d_{\text{water}}}$

$$\Rightarrow \eta_{\text{liquid}} = \frac{(54.8733) \cdot (0.8617) \cdot (8.5 \times 10^{-4})}{(71.6667)(0.9989)} = \frac{5.61431 \times 10^{-4}}{71.6667} \text{ Ns m}^{-2}$$

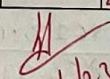
Toluene  $\text{CH}_3$  Hence:  $\eta_{\text{liquid}} = 5.6143 \times 10^{-4} \text{ Ns m}^{-2} = 5.6143 \text{ mil}$ ; Porte

- Inference:** Concept of relative measurement:
- We can measure the coefficient of viscosity of liquid using another liquid.
  - Chances of error are close to zero as, atmospheric pressure would be same for both the liquids on the surface.
- Affect of major factors:**
- A slight increase or decrease in temperature of liquid could vary the coefficient of viscosity.
  - Forces of cohesion and adhesion play a major role in the fine factor.

- Relevance to Society & Environment:**
- Determination of impurities: If it is found that viscosity of a given liquid varies, we can determine that the liquid contains impurities or adulterants.
  - Flow of liquid or gas in pipeline: streamline flow of propellants are needed inside pipelines, determined by Reynold's number  $> 1000$ .
  - Determining various components of liquid: Various expressions like terminal velocity, Stoke's law, capillary action is determined by the coefficient of viscosity. E.g.  $v_t = 2\pi r(p - \rho)/(9\eta)$  → terminal velocity

**Report:** The coefficient of viscosity of the given liquid at  $27.5^\circ\text{C}$  =  $5.6143 \times 10^{-4} \text{ Nsm}^{-2}$

Coefficient of viscosity of liquid at  $27.5^\circ\text{C}$  is  $5.6143 \times 10^{-4} \text{ Nsm}^{-2}$

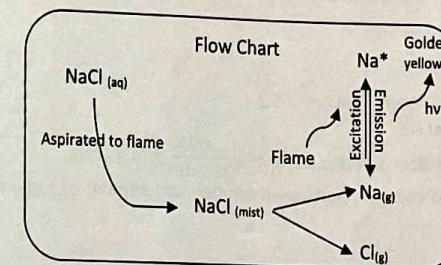
Evaluation of experiment - 7		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation	7 + 5	12
Expected Value & Execution	20 + 4	22
Inference & Societal Relevance	2 + 2	4
Total	40	37 38 11
Signature of Teacher	 10/11/23	

## EXPERIMENT NO - 8

**Aim:** Flame photometric estimation of sodium

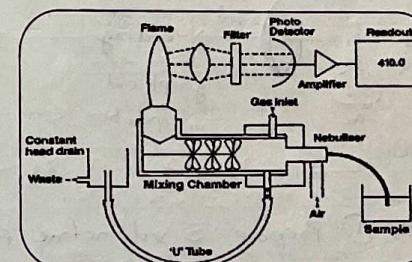
### Theory:

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 etc., by making use of thermal energy of flame. From higher energy states, these electrons will return to the ground state by emitting radiations  $(E_n - E_1) = hv$  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  $1900^\circ\text{C}$ . 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.

**Description:**  
 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 (589nm) 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.

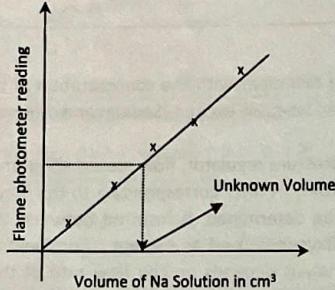
**Calculation:**

Amount of NaCl in the given 100cm<sup>3</sup> solution = 2.5 g

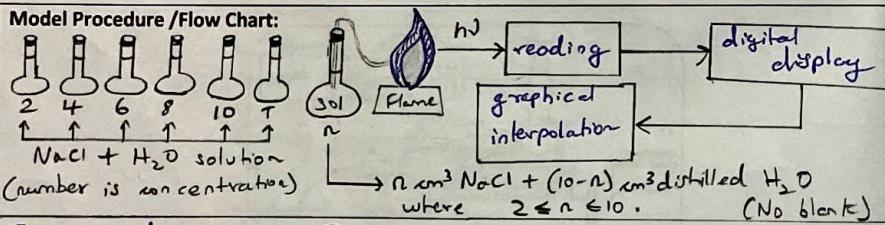
58.5 g of NaCl contains 23 g of Na.

Therefore, 1 cm<sup>3</sup> of the given stock solution contains  $\frac{wx23}{58.5 \times 100} = A' \text{g of Na}$

From the graph calculate the volume of unknown solution and amount of sodium present in the unknown solution.



**Model Procedure / Flow Chart:**



**Experimental procedure:** ① Prepare 5 volumetric flasks with 2, 4, 6, 8, 10 cm<sup>3</sup> of NaCl solution and fill it till the 10 cm<sup>3</sup> mark with distilled water.

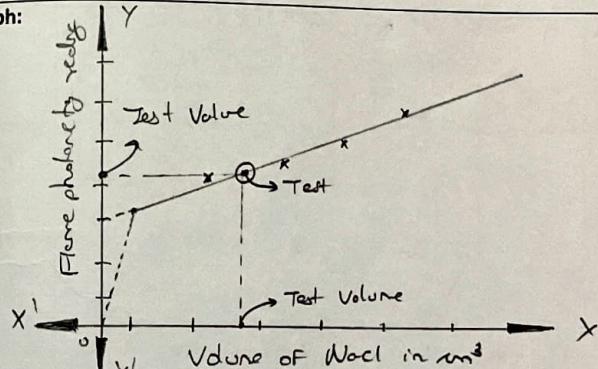
② Plot the linear graph with given data, and measure the volume of the test solution.

③ After predicting the volume, recalculate the weight of NaCl present within the unknown solution.

**Model Calculation:** We know: Amount of NaCl in the given 100cm<sup>3</sup> solution = 2.5 g = w.  
 58.5 g of NaCl contains 23 g of Na  
 Therefore 1 cm<sup>3</sup> of the solution =  $\frac{w \times 23}{58.5 \times 100} = A' \text{g of Na}$   
 $A' = \frac{2.5 \times 23}{58.5 \times 100} = 0.00983 \text{ g of Na} = 9.82905 \text{ mg}$   
 Hence given a volumetric flask having concentration of  
 As 1 cm<sup>3</sup> is 9.82905 mg,  $\phi \text{ cm}^3$  gives  $\phi \times 9.82905 \text{ mg}$

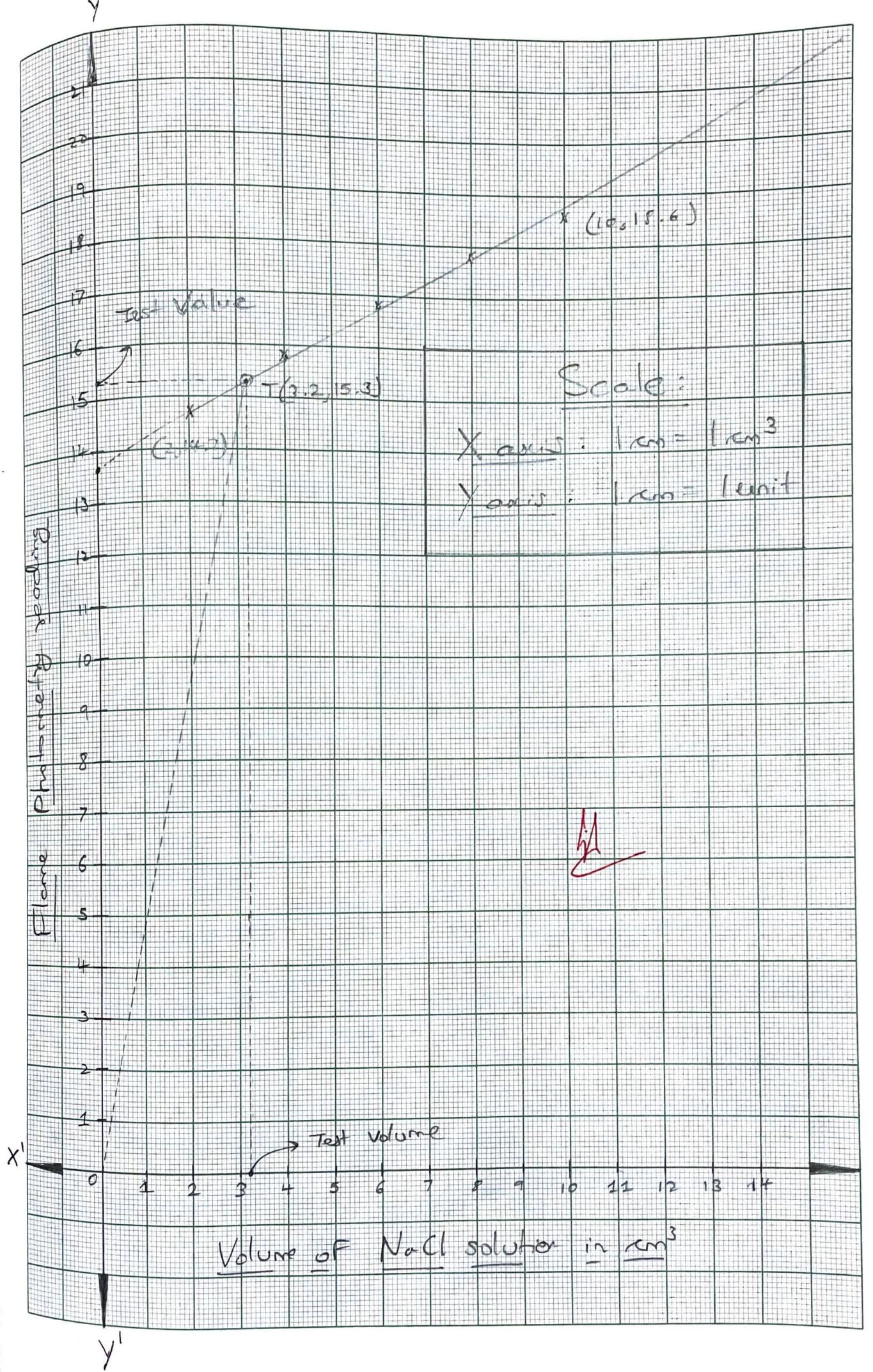
Hence weight of NaCl for that concentration:  $\phi \times 9.82905 \text{ mg}$   
 The following is also used for the test solution.

**Model graph:**



**Tabulation:**

Sl. No.	Vol. of NaCl in cm³	Flame Photometer Reading	Wt. of Sodium in mg
1.	2	14.7	<del>(144.487035/14.2) × 2 = 19.65810</del>
2.	4	15.8	<del>(155.29899/15.8) × 4 = 39.51620</del>
3.	6	16.8	<del>(165.12804/16.8) × 6 = 58.91430</del>
4.	8	17.8	<del>(174.95709/17.8) × 8 = 78.65240</del>
5.	10	18.6	<del>(182.82033/18.6) × 10 = 98.29050</del>
6. Unknown	15.3	<del>(150.38477/15.3) × 2 = 31.45296</del>	<i>(S)</i>



Calculation: To calculate individual mass of Sodium (Na) at different concentrations. Hence the following result:

①  $V_{\text{NaCl}} = 2 \text{ cm}^3$ :  $2 \times 9.82905 = 19.6581 \text{ mg of Na}$

②  $V_{\text{NaCl}} = 4 \text{ cm}^3$ :  $4 \times 9.82905 = 39.3162 \text{ mg of Na}$

③  $V_{\text{NaCl}} = 6 \text{ cm}^3$ :  $6 \times 9.82905 = 58.9743 \text{ mg of Na}$

④  $V_{\text{NaCl}} = 8 \text{ cm}^3$ :  $8 \times 9.82905 = 78.6324 \text{ mg of Na}$

⑤  $V_{\text{NaCl}} = 10 \text{ cm}^3$ :  $10 \times 9.82905 = 98.2905 \text{ mg of Na}$

Now through the plotted graph we see that volume of NaCl in test solution is  $3.2 \text{ cm}^3$ . It corresponds to 15.3 value of the flame photometry value.

Hence:  $V_{\text{NaCl}} = 3.2 \text{ cm}^3$ :  $3.2 \times 9.82905 = \boxed{31.45296 \text{ mg}}$

Through we found mass of the unknown test solution which contains NaCl solution.

Hence:  $\boxed{V_{\text{Test}} = 3.2 \text{ cm}^3 \text{ and } \text{Mass}_{\text{Na}} = 31.45296 \text{ mg}}$

Inference: Experimental inference: ① With the help of spirited flame, we can measure the weight and volume of an unknown test solution using:  $E = h\nu = hc/\lambda$ .

② Blue flame is maintained with constant gas supply, and spirit supply through a narrow capillary tube.

Observational inference: ① We see that photometric value is directly proportional to volume of NaCl in solution.

② Using linear regression we can predict the best fit line and predict values for any concentration of the similar solvents.

Relevance to Society & Environment: ① Fractional distillation: In the process of fractional distillation, we can measure properties like density and weight without disturbing flow of liquid.

② Biochemistry lab: To isolate various concentrates of biological samples which contain heavy protein amino acids, photometry can be used in processes such as: Duma's method, Kjeldahl method,

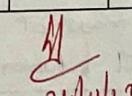
Report:

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

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

3.2

31.45296

Evaluation of experiment - 8		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation	9 + 7	16
Expected Volume & Execution	16 + 4	19
Inference & Societal Relevance	2 + 2	4
Total	40	39
Signature of Teacher	 24/11/13	

## EXPERIMENT NO - 9

Aim: Colorimetric estimation of copper from PCBs

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 ( $I_r$ ). Thus,  $I_0 = I_a + I_t + I_r$

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

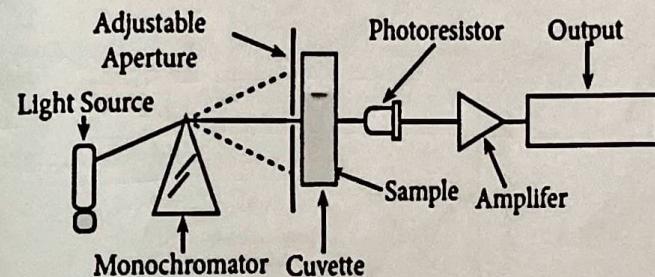
$$\text{Therefore, } I_0 = I_a + I_t$$

If  $I_0/I_a$  is called transmittance then  $\log(I_0/I_a)$  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 'E' is a constant called molar extinction coefficient. E 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.



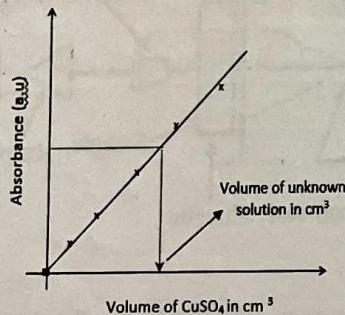
### Instrumentation of Colorimeter:

- **Light Source:** The source of light should produce energy with enough intensity to cover the entire visible spectrum (380-780 nm). Commonly, Tungsten lamps are used as a light source for measurement in the visible spectrum and near-infrared ranges. Halogen deuterium is suitable for measurement in the UV range (200-900 nm).
- **Slit:** It reduces unwanted or stray light by allowing a light beam to pass through.
- **Condensing lens:** Parallel beam of light emerges from condensing lens after the light passes through slit incidents on it.
- **Monochromator:** It filters the monochromatic light from polychromatic light, which absorbs unwanted light wavelengths and permits only monochromatic light. These are of three types: prism, grating, and glass.
  - **Prism:** It facilitates the refraction of light when it passes from one medium to another.
  - **Glass:** It selectively transmits light in certain ranges of wavelengths.
  - **Gratings:** These are made of graphite, which separates light in different wavelengths.

- Cuvette (Sample cell):** The monochromatic light from the filter passes through the colored sample solution placed in the cuvette. Their sizes range from square, and rectangle to round and have a fixed diameter of 1cm. These are of three types based on the substances these are made of: Glass, Quartz, and Plastic cuvette.
  - Glass cuvettes are cheap and absorb light of 340 nm wavelength.
  - Quartz cuvettes facilitate entry of both lights of UV and visible ranges.
  - Plastic cuvettes are cheaper, easily scratched, and have shorter lifespans.
- Photocell (Photodetector):** These photosensitive devices measure light intensity by converting light energy into electrical energy.
- Galvanometer:** The electrical signal generated in a photocell is detected and measured by a galvanometer. It displays optical density (OD) and percentage transmission.

#### Description:

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 (prepared from PCB sample) in 25 cm<sup>3</sup> volumetric flask also add 2.5 cm<sup>3</sup> of ammonia and make it up to the mark using distilled water and shake well. After 10 min. 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 solution given and calculate the amount of copper present in it.

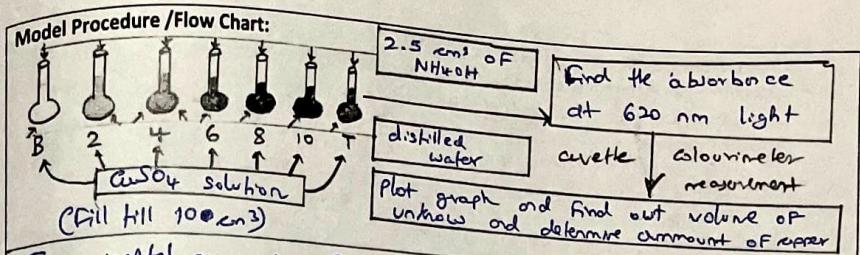


#### Calculation:

Weight of copper sulphate pentahydrate present in 100 cm<sup>3</sup> of the given solution = 55 mg

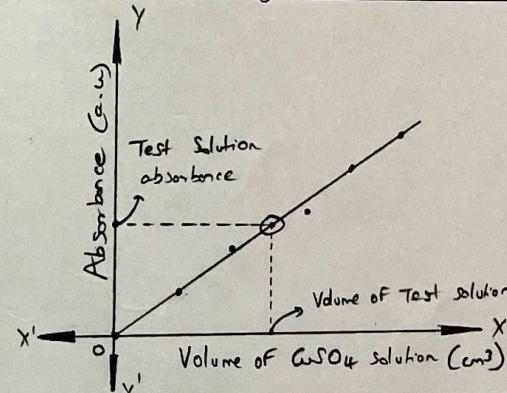
Weight of CuSO<sub>4</sub>.5H<sub>2</sub>O in 1 cm<sup>3</sup> of its solution = X/100 mg = ..... 0.55 ..... = 'Y' mg

Weight of copper present in 1 cm<sup>3</sup> of its solution =  $\frac{Y \times 63.54}{249.54}$  = 'W' mg

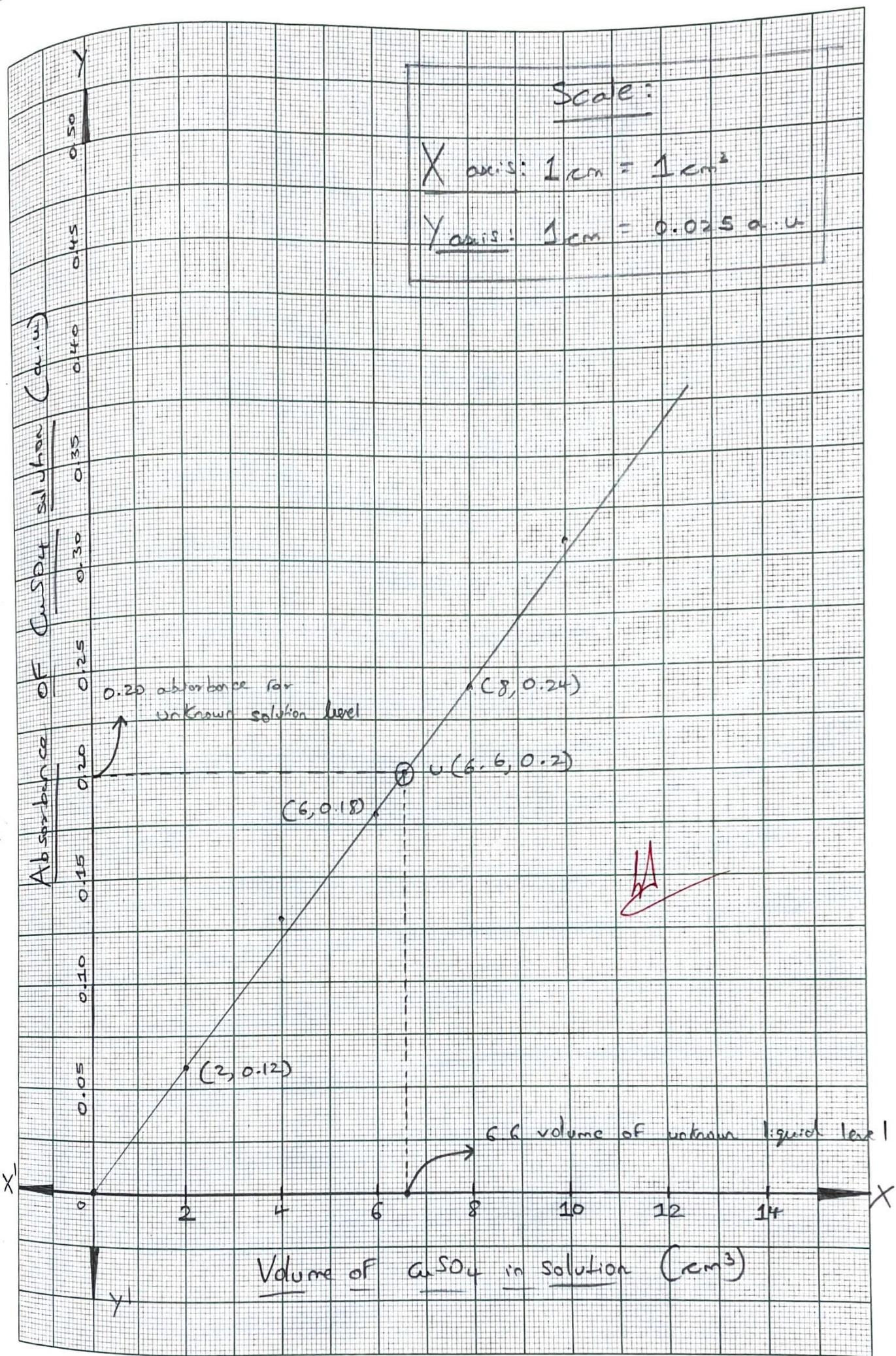


- Experimental procedure:
- In 7 empty glass volumetric flasks, add values of 2 cm<sup>3</sup>, 4 cm<sup>3</sup>, 6 cm<sup>3</sup>, 8 cm<sup>3</sup> and 10 cm<sup>3</sup> 2.5 cm<sup>3</sup> of NH<sub>4</sub>OH each, followed by CuSO<sub>4</sub> solution with corresponding
  - Add distilled water for the following flasks till the marked level. Take a small cuvette and rinse with given solution
  - Measure absorbance value for all solutions, even for the given test solution. Note down the value for 620 nm (blue)
  - After use of a given solution, clean with water (distilled only), and rinse it with the next solution.
  - Before inserting cuvette to the colorimeter, wipe it down with tissue paper to remove excess liquid. Set device 62 mode
  - Make sure liquid level in cuvette is above half mark, push cuvette into the device, to get accurate measurement. Plot the graph

Model graph:



**Model Calculation:** We know Weight of CuSO<sub>4</sub>.5H<sub>2</sub>O in 100 cm<sup>3</sup> in the given solution = 55 mg  
Weight of CuSO<sub>4</sub>.5H<sub>2</sub>O in 1 cm<sup>3</sup> of its solution =  $\frac{55}{100}$  = 0.55 mg  
Weight of copper in 1 cm<sup>3</sup> of solution =  $\frac{0.55 \times 63.54}{249.54}$  = 0.14005 = ψ



Let the weight of copper in solution be  $W_{Cu}$ .

$$W_{Cu} \text{ for } n \text{ cm}^3 = W_{Cu}(n) = n \times \psi = n \times 0.14005$$

Find the given volume using the graph given.  $[W_{Cu}(n)]$

Tabulation:

Sl. No.	Vol. of $CuSO_4$ in $cm^3$	Optical Density	Wt. of Copper (mg)
1.	0 [Blank]	0.00	0.00000
2.	2	0.06	0.28010
3.	4	0.13	0.56020
4.	6	0.18	0.84030
5.	8	0.24	1.12040
6.	10	0.31	1.40050
7.	Unknown	0.20	0.92433

Calculation: Serial number calculation  $(Vd \text{ of } CuSO_4) \times 0.14005$

$$\textcircled{1} \quad 0.00 \times 0.14005 = 0.00 \text{ mg (0 cm}^3\text{)}$$

$$\textcircled{2} \quad 2 \times 0.14005 = 0.28010 \text{ mg (2 cm}^3\text{)} \quad \text{For all}$$

$$\textcircled{3} \quad 6 \times 0.14005 = 0.84030 \text{ mg (6 cm}^3\text{)} \quad \text{for}$$

$$\textcircled{4} \quad 4 \times 0.14005 = 0.56020 \text{ mg (4 cm}^3\text{)} \quad \text{known}$$

$$\textcircled{5} \quad 8 \times 0.14005 = 1.12040 \text{ mg (8 cm}^3\text{)} \quad \text{values}$$

$$\textcircled{6} \quad 10 \times 0.14005 = 1.40050 \text{ mg (10 cm}^3\text{)}$$

Now for unknown solution:  $\boxed{\text{Volume} = 6.6 \text{ cm}^3} = V_c$

Hence weight of  $CuSO_4 \cdot 5H_2O = V_c \times \psi$

$$\Rightarrow 6.6 \times 0.14005 = \boxed{0.92433 \text{ mg}}$$

**Inference:** Experimentation and graphical inference: ① Through Beer-Lambert law,  $A = \epsilon C \lambda$ , we see that  $A \propto C \rightarrow$  concentration  $\propto$  adsorbance.

② Blue light of  $620\text{ nm}$  is used as it is good in scattering and does not cause heating of element or liquid.

**Observational inferences:** ① We can find out weight and volume of a solution without touching or disturbing the solution through linear regression.

② Slope of the graph  $\approx m = \epsilon l = \text{molar extinction} \times \text{thickness of cuvette}$ .

**Relevance to Society & Environment:** ① Identifying clean water: Using colourimetry we can compare various samples of water to figure out which one is more polluted by measuring impurities in ppm.

② Preventing Adulteration: We can identify adulterated oils and liquids like souce, juice through colourimetry which identifies added impurities. Also used for detecting dissolved drugs for smuggling.

③ Quality maintenance: Quality of a product can be maintained so as to ensure smooth flow and sale of products.

④ Glass blowing: To maintain colour of the super heated glass, colourimetry is used in order to treat it with nano-materials for robotics.

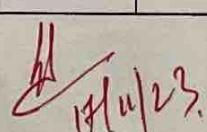
**Report:** is used in order to treat it with nano-materials for robotics

1. Volume of unknown solution = .....  $\text{cm}^3$

→ [6.6  $\text{cm}^3$ ]

2. Amount of Copper in the given unknown solution = ..... mg

→ [0.92433 mg]

Evaluation of experiment - 9		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation	9 + 7	16
Expected Volume & Execution	16 + 4	18
Inference & Societal Relevance	2 + 2	4
Total	40	38
Signature of Teacher		
 13/11/23.		

## SCHEME OF EVALUATION FOR INSTRUMENTAL ANALYSIS

VISCOSITY		COLORIMETRY & FLAMEPHOTOMETRY		WEAK ACID CONTENT		POTENTIOMETRY & CONDUCTOMETRY		
<b>20</b>		<b>16</b>		<b>16</b>		<b>16</b>		
Up to 5.0%	20	$\pm 0.2 \text{ cm}^3$	16	Up to 5 %	16	$\pm 0.2 \text{ cm}^3$	16	
$\pm 5.1 - 8.0\%$	16	$\pm 0.3 \text{ cm}^3$	14	$\pm 5.1 - 8.0 \%$	14	$\pm 0.3 \text{ cm}^3$	12	
$\pm 8.1 - 10.0\%$	14	$\pm 0.4 \text{ cm}^3$	12	$\pm 8.1 - 10.0 \%$	10	$\pm 0.4 \text{ cm}^3$	10	
$\pm 10.1 - 12.0\%$	10	$\pm 0.5 \text{ cm}^3$	10	$\pm 10.1 - 12.0 \%$	08	$\pm 0.5 \text{ cm}^3$	08	
$\pm 12.1 - 15.0\%$	06	$\pm 0.6 \text{ cm}^3$	08	$\pm 12.1 - 14.0 \%$	06	$\pm 0.6 \text{ cm}^3$	06	
> $\pm 15\%$	04	$\pm 0.7 \text{ cm}^3$	06	$\pm 14.1 - 15.0 \%$	04	$\pm 0.7 \text{ cm}^3$	04	
		$\pm 0.8 \text{ cm}^3$	04	> $\pm 15\%$	00	$\pm 0.8 \text{ cm}^3$	00	
Model Procedure & graph	07	<b>09</b>				<b>09</b>		
Calculation	05	<b>03</b>		<b>03</b>		<b>03</b>		
Graph	-	<b>04</b>		<b>02 + 02</b>		<b>04</b>		
Execution	04	<b>04</b>		<b>04</b>		<b>04</b>		
Inference	02	<b>02</b>		<b>02</b>		<b>02</b>		
Societal Relevance	02	<b>02</b>		<b>02</b>		<b>02</b>		

## SCHEME OF EVALUATION FOR VOLUMETRIC ANALYSIS

Components	Marks	Error in BR	Marks
Burette Reading (BR)	<b>20</b>	$\pm 0.2 \text{ cm}^3$	<b>10 + 10</b>
Model Procedure	<b>07</b>	$\pm 0.3 \text{ cm}^3$	<b>08 + 08</b>
Execution	<b>04</b>	$\pm 0.4 \text{ cm}^3$	<b>06 + 06</b>
Calculation	<b>05</b>	$\pm 0.5 \text{ cm}^3$	<b>04 + 04</b>
Inference	<b>02</b>	$\pm 0.6 \text{ cm}^3$	<b>02 + 02</b>
Societal Relevance	<b>02</b>	> $\pm 0.6 \text{ cm}^3$	<b>00 + 00</b>

**Note:**

1. Each experiment will be valued for 40 Marks
2. Two best readings will be considered for evaluation in volumetric analysis.
3. All the entries should be made in pen only.
4. Overwritten values will not be considered for evaluation.