

**ENGINEERING CHEMISTRY    LABEXPERIMENT-1**  
**CHY1701L**

NAME: P.NITYASREE

REGNO: 17MIS1007

DATE:11-12-17

**Titration I :**

Standardization of Sodium Thiosulphate

S. No.	Volume of $K_2Cr_2O_7$ (mL)	Burette reading (mL)		Volume of sodium thiosulphate ( $V_1$ , mL)
		Initial	Final	
1.	20	0	20.4	20.4
2.	20	0	20.4	20.4
3.	20	0	20.4	20.4
Concordant value				20.4

Calculations:

Volume of potassium dichromate ( $V_1$ )      =    20mL

Strength of potassium dichromate ( $N_1$ )      =    0.01N

Volume of sodium thiosulphate ( $V_2$ )      =    20.4 mL

Strength of sodium thiosulphate ( $N_2$ )      =

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 N_1 / V_2$$

Strength of sodium thiosulphate =  $N_2$

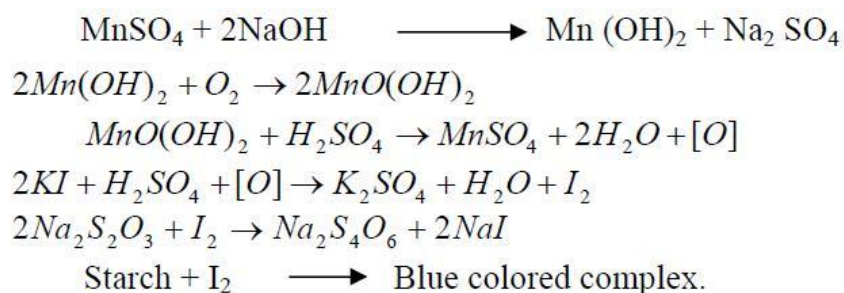
$$= \frac{20 \times 0.01}{V_2}$$

$$= \underline{0.009N}$$

## DISSOLVED OXYGEN (DO) ANALYSIS IN WATER BY WINKLER'S METHOD

### Principle:

Dissolved oxygen is an important factor in corrosion. Oxygen is poorly soluble in water. The solubility of oxygen decreases with increase in conc. of the salt under a pressure of one atmosphere, the solubility is less in saline water. The estimation of dissolved oxygen in water is useful in studying corrosion effect of boiler feed water and in studying water pollution. *Dissolved oxygen* is usually determined by Winkler's method. It is based on the fact that dissolved oxygen oxidized potassium iodide (KI) to iodine. The liberated iodine is titrated against standard sodium thiosulphate solution using starch indicator. Since dissolved oxygen in water is in molecular state. It as such cannot oxidize KI. Hence Manganese Hydroxide is used as an oxygen carrier to bring about the reaction between KI and Oxygen. Manganese hydroxide, in turn, is obtained by the action of NaOH on MnSO<sub>4</sub>.



The liberated iodine (I<sub>2</sub>) is titrated against standard sodium thiosulphate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) solution using starch as indicator.

### Apparatus:

Conical flask, Burette, Measuring flask, Beakers

## Titration : II

### Estimation of dissolved oxygen

S. No.	Volume of water sample (V <sub>1</sub> / mL)	Burette reading (mL)		Volume of sodium thiosulphate (V <sub>2</sub> / mL)
		Initial	Final	
1	100	0	11.1	11.1
2	100	0	11.1	11.1
3	100	0	11.1	11.1
Concordant value				11.1

### Calculations :

Volume of sodium thiosulphate V<sub>2</sub> = 11.1 mL

Strength of sodium thiosulphate N<sub>2</sub> = 0.009 N

Volume of water sample taken V<sub>1</sub> = 100 mL

Strength of given water sample N<sub>1</sub> = ?

$$V_1 N_1 = V_2 N_2$$

$$N_1 = \frac{V_2 \times N_2}{V_1}$$

$$= \underline{0.00099}$$

Amount of dissolved oxygen (ppm) = normality × equivalent weight of O<sub>2</sub> × 1000 mg/L  
of the given water sample.

$$= 0.00099 \text{ N} \times 8 \times 1000 \text{ mg/L}$$

$$= \underline{7.992 \text{ ppm}}$$

### Reagents:

Standard Potassium dichromate, sodium thiosulphate solution, potassium iodide solution, Alkali Iodide solution: (KI + NaOH in water), conc.  $\text{H}_2\text{SO}_4$ , Manganese sulphate, starch solution as indicator

## **PROCEDURE:**

### **TITRATION 1: STANDARDIZATION OF SODIUM THIOSULPHATE**

The burette is washed and rinsed with sodium thiosulphate solution. Then the burette is filled with given sodium thiosulphate solution. 20 mL of 0.01N potassium dichromate solution is pipette out into a clean conical flask. To this 5 mL of sulphuric acid and 10 mL of 10% potassium iodide are added. This is titrated against sodium thiosulphate solution, when the solution become straw yellow colour, starch indicator is added and then the titration is continued. The end point is disappearance of bluish brown colour. The titration is further repeated twice or thrice to get the concordant value.

### **TITRATION 2: ESTIMATION OF DISSOLVED OXYGEN**

100 mL of water sample is taken in a conical flask, 2 mL of manganese sulphate and 2mL of alkali iodide solution are added and shaken well for the rough mixing of the reagents. The flask is left aside for few minutes to allow the precipitate to settle down and then 2mL of conc. Sulphuric acid is added for the complete dissolution of the precipitate. Then it is further titrated against standard sodium thiosulphate solution. When the solution becomes light yellow, starch indicator is added. The end point is disappearance of bluish brown colour. The titration is repeated twice or thrice to get the concordant value. From the titre value the strength of dissolved oxygen is calculated and hence the amount of dissolved oxygen in the water sample is calculated.

RESULT:

Amount of dissolved oxygen in the given water sample = 7.992 ppm.

**Evaluation of result**

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded

END

## **Experiment 2**

### **IRRIGATION WATER-SULPHATE ION ANALYSIS BY CONDUCTOMETRY**

**NAME : P.NITYASREE**

**REGNO : 17MIS1007**

**Date:18-12-17**

#### **Principle:**

Electrolyte solutions conduct electricity due to the presence of ions in solution. In case of precipitation titration between  $\text{BaCl}_2$  and  $\text{Na}_2\text{SO}_4$ , the conductance decreases slowly due to the replacement of  $\text{Na}^+$  ion by  $\text{SO}_4^{2-}$  upto the equivalence point.

After the equivalence point the conductance increases rapidly due to the excess addition of  $\text{BaCl}_2$  which remains in solution as  $\text{Ba}^{2+}$  and  $\text{Cl}^-$ .

This makes detection of neutralization point easy from the conductance trend plotted as a graph. This is the principle is used in the estimation of  $\text{SO}_4^{2-}$  from an effluent sample.

#### **Apparatus required:**

TDS-Conductivity bridge, conductivity cell, burette, pipette, volumetric flasks, glass rod and 100 mL beaker.

#### **Chemicals required:**

$\text{BaCl}_2$ ,  $\text{Na}_2\text{SO}_4$ , unknown sulphate solution and distilled water.

**Procedure:****Calibration of TDS-Conductivity meter:**

Place a freshly prepared 0.01 N KCl solution (given in bottle) in a 100 mL beaker and dip the conductivity cell in this solution and connect to the TDS-Conductivity meter. Press "CAL" button and complete the internal calibration of the instrument

**Standardization of BaCl<sub>2</sub>:**

Standard flask A contains Na<sub>2</sub>SO<sub>4</sub> which after making up to 50 mL will result in a solution containing 0.96 mg/mL of sulphate ions. Pipette out 20 mL of this solution and add 20 mL of distilled water to it in a 100 mL beaker.

The conductivity cell is dipped into the beaker and connected to TDS conductivity meter. Fill the burette with BaCl<sub>2</sub> solution (Solution B). Record the conductivity of the sulphate solution as 0<sup>th</sup> reading.

Add 0.5 mL portion of known concentration of BaCl<sub>2</sub> into the beaker, stir with glass rod and note down the conductance. Continue the addition of BaCl<sub>2</sub> (0.5 mL each time) and note the conductance after each addition. Continue the titration beyond the equivalence point for about 3mL.

The conductance will decrease till complete precipitation of BaSO<sub>4</sub> and then starts increasing on continuing the addition of BaCl<sub>2</sub>.

A graph is now drawn by plotting conductance Vs volume of BaCl<sub>2</sub> added and the intersection point from the graph gives the volume of BaCl<sub>2</sub> required for precipitating the sulphate from the known sample.

**Table. 2 Estimation of unknown sulphate solution**

Volume of BaCl <sub>2</sub>	Conductance	Volume of BaCl <sub>2</sub>	Conductance
0.0	0.8	5.5	0.74
0.5	0.79	6.0	0.73
1.0	0.78	6.5	0.73
1.5	0.77	7.0	0.73
2.0	0.76	7.5	0.72
2.5	0.76	8.0	0.72
3.0	0.75	8.5	0.72
3.5	0.75	9.0	0.72
4.0	0.75	9.5	0.72
4.5	0.74	10.0	0.73
5.0	0.74	10.5	0.75
		11.0	0.78
		11.5	0.81
		12.0	0.83
		12.5	0.85
		13.0	0.87
		13.5	0.91



		14.0	0.94
		14.5	0.97
		15	0.99

**Table. 1 standardization of barium chloride**

Volume of BaCl <sub>2</sub>	Conductance	Volume of BaCl <sub>2</sub>	Conductance
0.0	0.46	5.5	0.5
0.5	0.46	6.0	0.52
1.0	0.45	6.5	0.54
1.5	0.45	7.0	0.56
2.0	0.45	7.5	0.58
2.5	0.45	8.0	0.63
3.0	0.44	8.5	0.66
3.5	0.44	9.0	0.69
4.0	0.44	9.5	0.72
4.5	0.46	10	0.75
5.0	0.48	10.5	0.78

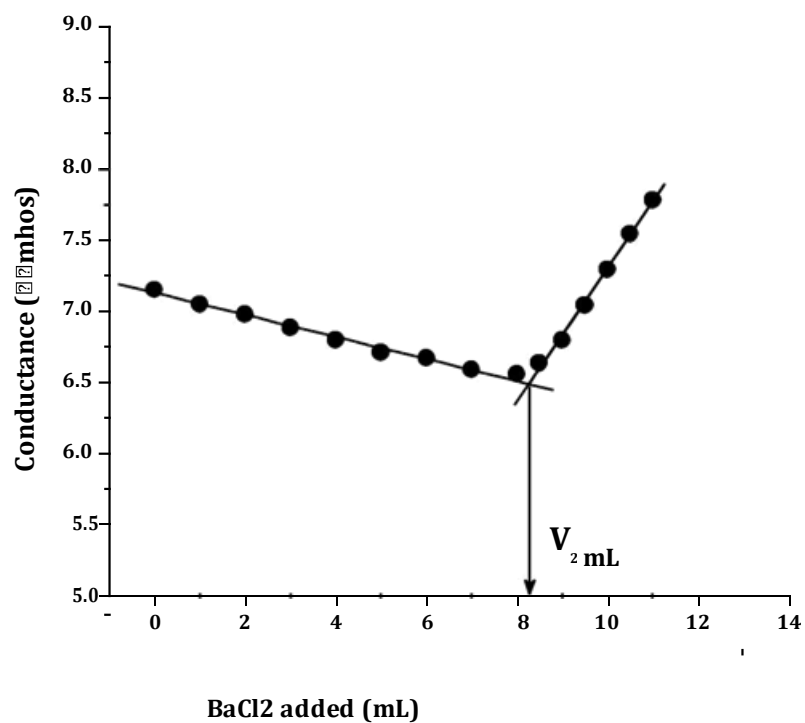
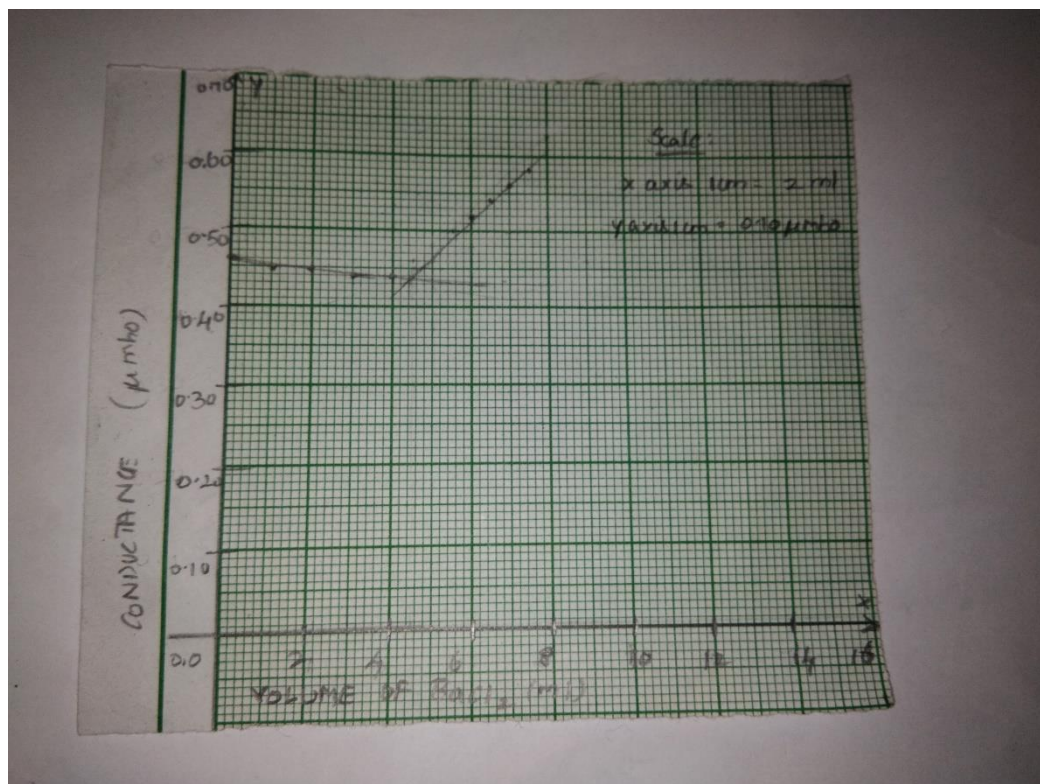
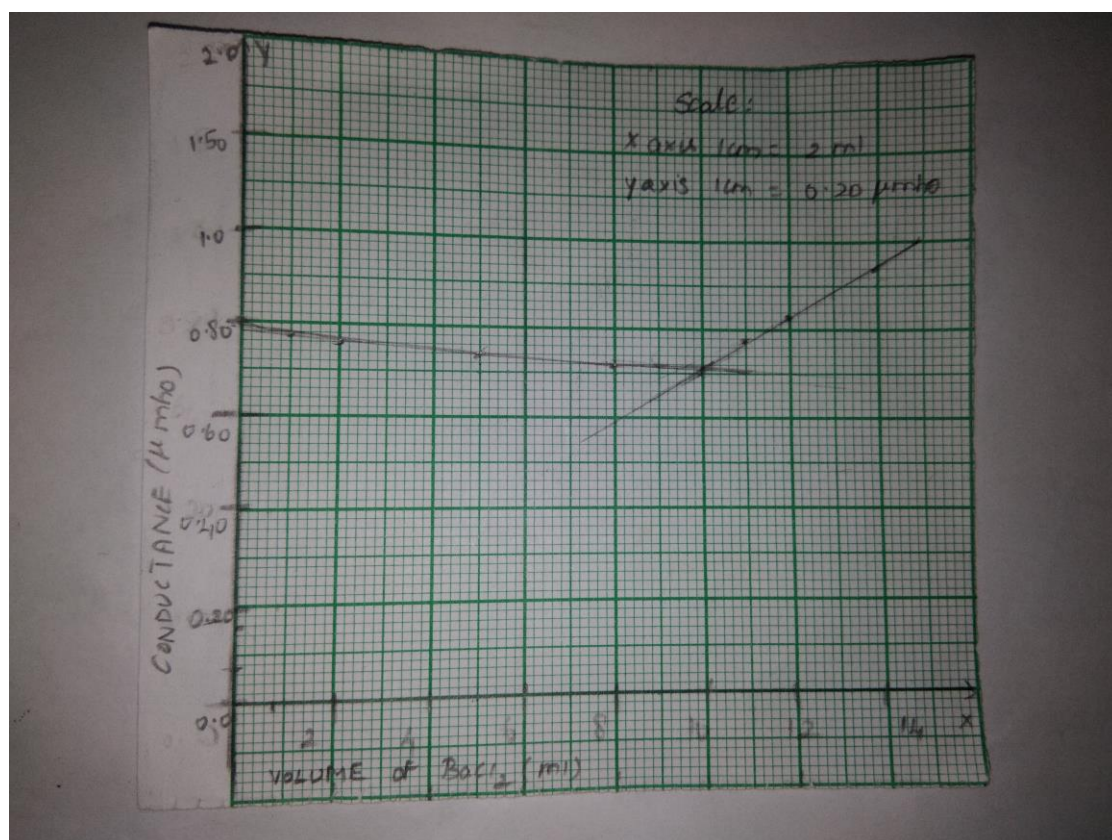


Fig. Model graph-2 for TDS-Conductometric estimation of unknown sulphate sample





### Estimation of unknown sulphate in the given solution:

Make up the unknown sulphate solution given in standard flask B up the 50 mL mark. Pipette out 20 mL of this solution into a 100 mL beaker and add 20 mL distilled water to it. Dip the conductivity cell and repeat the above procedure with the unknown sulphate solution and determine the amount of  $\text{BaCl}_2$  required for precipitating the unknown sulphate in the sample.

From the two titrations carried out, calculate the amount of sulphate present in the effluent sample.

#### Calculations:

##### A) Standardization of $\text{BaCl}_2$ :

From the first plot,

20 mL of known sulphate solution required 10  $V_1$  mL of  $\text{BaCl}_2$  for complete precipitation of sulphate.

Sulphate present in 20 mL of the solution used = 19.2 mg

Therefore, 1 mL of  $\text{BaCl}_2$  will precipitate  $19.2/V_1$  = 1.92 mg of sulphate (Y)

##### B) Estimation of unknown sulphate:

From the second plot,

20 mL of unknown sulphate consumed 4.5  $V_2$  mL of  $\text{BaCl}_2$  for complete precipitation of sulphate.

Sulphate present in 20 mL of the unknown solution =  $V_2 \times Y$  = 8.64 mg (Z)

Therefore, Sulphate present in the unknown sample =  $Z / 20 \times 1000$  = 432 ppm

## Result

Amount of sulphate present in the given sample = .....432.....ppm.

## Evaluation of result

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded

END

## Experiment-3

NAME: P.NTYASREE

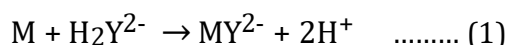
REGNO: 17MIS1007

### SOFTENING OF WATER THROUGH ION EXCHANGE RESIN – ASSESSMENT OF TOTAL HARDNESS USING EDTA METHOD

Date:8-1-18

#### Principle:

Hardness of water is due to the presence of calcium and magnesium salts in water. Ethylene diamine tetraacetic acid (EDTA) forms complexes with a large number of cations including  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions depending upon pH of the solution. Hence, it is possible to determine the total hardness of water using EDTA solution. EDTA in the form of its sodium salt ( $\text{H}_2\text{Y}^{2-}$ ) is more often used in place of EDTA in complexometric titration for estimation of metal ions because pure EDTA ( $\text{H}_4\text{Y}$ ) is sparingly soluble in water. EDTA has six binding sites (the four carboxylate groups and the two amino groups) providing six pairs of electrons. The resulting metal-ligand complex, in which EDTA forms a cage-like structure around the metal ion, is very stable at specific pH. All metal-EDTA complexes have a 1:1 stoichiometry. The  $\text{H}_2\text{Y}^{2-}$  form complexes with metal ions as follows.



where, M is  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  present in water. Reaction (1) can be carried out quantitatively at pH of 10 using Eriochrome Black T (EBT) indicator. EBT forms a wine-red complex with  $\text{M}^{2+}$  ions which is relatively less stable than the metal-EDTA complex. On titration EDTA first reacts with free  $\text{M}^{2+}$  ions and then with the metal-EBT indicator complex. The end point of the reaction is change of colour from wine red to steel blue.

#### Requirements

**Reagents and solutions:** Standard hard water (0.01M or 1000ppm), EDTA solution, EBT indicator, hard water sample and  $\text{NH}_3\text{-NH}_4\text{Cl}$  buffer solution.

**Apparatus:** Burette, pipette, conical flask, standard flask and burette stand

## Procedure

### Titration I - Standardisation of EDTA

Pipette out 20 mL of the standard hard water containing 1mg/mL of calcium carbonate (1000 ppm) into a clean conical flask. Add one test tube full of ammonia buffer ( $\text{NH}_4\text{OH} - \text{NH}_4\text{Cl}$ ) solution to maintain the pH around 10. Add three drops of Eriochrome Black – T (EBT) indicator and titrate it against the given EDTA solution taken in the burette. The end point is change of colour from wine red to steel blue. Repeat the titration for concordant titre values. Let 'V<sub>1</sub>' be

the volume of EDTA consumed.

S. No.	Volume of standard hard water (mL)	Burette reading (mL)		Volume of EDTA (V <sub>1</sub> , mL)
		Initial	Final	
1	20	0	21	21
2	20	0	21	21
3	20	0	21	21
Concordant titre value				21

### Calculations:

$$\begin{aligned} 20\text{mL of standard hard water} &= 21 \text{ mL of EDTA} \\ \Rightarrow 20 \times 1\text{mg of CaCO}_3 &= 21 \text{ mL of EDTA} \\ \Rightarrow 1\text{mL of EDTA} &= (20/V_1) \text{ mg of CaCO}_3 \text{ eqvt.} \\ &= 0.95 \text{ mg of CaCO}_3 \text{ eqvt.} \end{aligned}$$

*This relation will be used in other two titrations*

## Titration II - Estimation of Total hardness of hard water sample

Pipette out 20 mL of the given sample of hard water into a clean conical flask. Add one test tube full of ammonia buffer ( $\text{NH}_4\text{OH} - \text{NH}_4\text{Cl}$ ) solution and three drops of Eriochrome Black-T (EBT) indicator. Titrate this mixture against standardized EDTA solution taken in the burette. The end point is the change of color from wine red to steel blue. Repeat the titration as carried out earlier for concordant titre value. Let 'V<sub>2</sub>' be the volume of EDTA consumed.

S. No.	Volume of standard hard water (mL)	Burette reading (mL)		Volume of EDTA (V <sub>2</sub> , mL)
		Initial	Final	
1	20	0	8.5	8.5
2	20	0	8.5	8.5
3	20	0	8.5	8.5
Concordant titre value				8.5

### Calculations:

$$20\text{mL of given hard water} = \frac{8.5}{V_1} \text{ mL of EDTA} = \frac{V_2 \times 20}{V_1} \text{ mg of CaCO}_3 \text{ eqvt.}$$

$$\therefore 1000\text{mL of given hard} = \frac{V_2 \times 20 \times 1000}{V_1 \times 20} \text{ mg of CaCO}_3 \text{ eqvt}$$



$$= \frac{V_2 \times 1000}{V_1} \text{ mg / L}$$

3

$$\text{Total Hardness} = \frac{V_2}{V_1} \times 1000 = \dots 404.7 \dots \text{ ppm}$$

**Removal of hardness using ion exchange method: (This should be the first step in the lab)**

Arrange the ion exchange column on to a burette stand and place a clean funnel on top of the column. Take 50 mL of the hard water sample in a beaker and slowly pass the water sample through the ion exchange column. Place a clean beaker under the column and collect water passing through the column over a period of 15 minutes. Adjust the valve of the column to match the duration of outflow.

From the water collected through the column, pipette out 20 mL into a clean conical flask and repeat the EDTA titration as carried out above. Note down the volume of EDTA consumed as 'V<sub>3</sub>'.

### TITRATION 3:

Volume of standard soft water	Burette (ml)		Volume of EDTA V3(ml)
	Initial	final	
20	0	5	5
20	0	5	5
	Concordant value		5

### Calculations:

$$20\text{mL of given treated hard water (softened)} = 5 \text{ mL of EDTA}$$

$$= \frac{V_3 \times 20}{V_1} \text{ mg of CaCO}_3 \text{ eqvt.}$$

$$1000 \text{ mL of given boiled hard water} = \frac{V_3 \times 20 \times 1000}{V_1 \times 20} \text{ mg of CaCO}_3 \text{ eqvt.}$$

$$\text{Residual hardness} = \frac{V_3}{V_1} \times 1000 = 238.095 \text{ mg / L or ppm}$$

### Result:

1. Total hardness of the water sample = 404.7 ppm
2. Residual hardness in the water sample = 238.095 ppm
3. Hardness removed through the column = 166.6 ppm

END

NAME:P.NITYASREE  
NAME:P.NITYASREE  
REGNO:17MIS1007

#### Experiment-4

### CONSTRUCTION AND WORKING OF AN ELECTROCHEMICAL CELL

Date:29-1-18

#### Principle :

The electromotive force (emf) of an electrochemical cell is measured by means of a potentiometer. An electrochemical cell ( $E_{\text{cell}}$ ) is considered as a combination of two individual single electrodes. The potential difference between the two single electrode potentials is a measure of emf of the cell ( $E_{\text{cell}}$ ). In order to measure the potential difference between electrodes in contact with electrolyte containing the same cation, it is necessary to have another electrode in contact with electrolyte of same cation, both the half-cells connected through a salt bridge. Saturated calomel electrode (SCE;  $E_{\text{calomel}}$ ) whose potential is known, is used as a reference electrode and it is coupled with the metal electrode for which the potential is to be determined.

**Hg / Hg<sub>2</sub>Cl<sub>2</sub> (s), saturated KCl || (N/10) electrolyte of the metal / Metal**

From the emf of the cell involving saturated calomel electrode and metal electrode dipped in its solution of 0.1 N electrolyte, electrode potential of the metal electrode is readily calculated using the standard potential of calomel electrode as;

$$E_{\text{cell}} = E_{\text{M/M}^+} - E_{\text{calomel}}$$

$$E_{\text{M/M}^+} = E_{\text{cell}} + E_{\text{calomel}}$$

$E_{\text{cell}}$  is the total emf of the cell. The electrode potential of the metal electrode is given by Nernst equation as;

$$E_{\text{M/M}^+} = E^{\circ}_{\text{M/M}^+} + \frac{RT}{nF} \ln a_{\text{M}^{n+}}$$

$$E^{\circ}_{\text{M/M}^+} = E_{\text{M/M}^+} - \frac{RT}{nF} \ln a_{\text{M}^{n+}}$$

$$E^{\circ}_{\text{M/M}^+} = E_{\text{M/M}^+} - \frac{0.0595}{n} \log a_{\text{M}^{n+}}$$

**Table 1: EMF measured for various concentrations of  $M/M^{n+}$  system**

Electrode/ Electrolyte	Concentration of Electrolyte (N)	$E_{\text{cell}}$ (V) (EMF of the cell)	$E_{M/M^+} = E_{\text{cell}} + E_{\text{calomel}}$
$\text{Zn}/\text{Zn}^{2+}$	0.01 N	-0.942	-0.698
	0.05 N	-0.948	-0.704
	0.1 N	-0.959	-0.715
$\text{Cu}/\text{Cu}^{2+}$	0.01 N	0.031	0.275
	0.05 N	0.032	0.276
	0.1 N	0.037	0.281

**Table 2: Individual activity coefficients of  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$  in water at 25°C**

Metal ion system ( $\text{Cu}^{2+}/\text{Zn}^{2+}$ )	0.001	0.002	0.005	0.01	0.02	0.05	0.1	0.2
Activity coefficient ( $\gamma_c$ )	0.905	0.870	0.809	0.749	0.675	0.570	0.485	0.405

Solution Temperature (T) = °C

The potential of saturated calomel electrode (SCE) = 0.244 + 0.0007 (25°C)

$$E^\circ_{\text{M}/\text{M}^+} = E_{\text{M}/\text{M}^+} - \frac{0.0595}{1n} \log [\gamma_c \times C] \text{ -----}$$

where  $E^\circ$  is the standard electrode potential of the metal electrode;  $a_{\text{M}^{n+}}$  is the activity of metal ions in the solution ( $a_{\text{M}^{n+}} = \gamma_c[C]$ );  $\gamma_c$  is the activity coefficient (Table 2) and C is the concentration of the electrolyte solution.

**Requirements:**

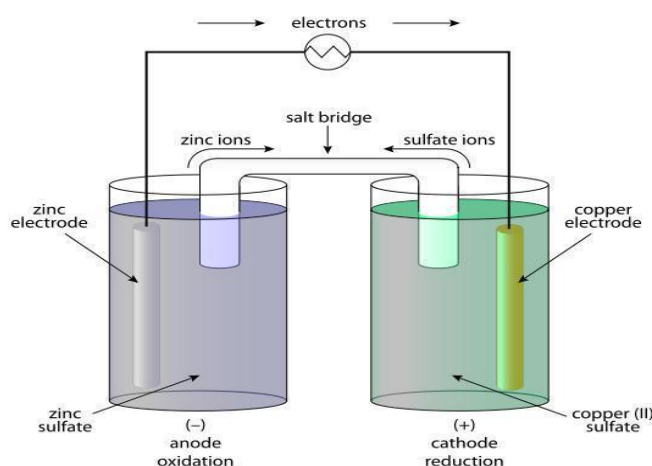
**Reagents and solutions:** Copper sulphate stock solution (0.1N), zinc sulphate stock solution (0.1N), KCl salt.

**Apparatus:** Digital potentiometer, copper electrode, zinc electrode, calomel electrode, 100 mL beaker, burette, standard flask.

**Procedure:**

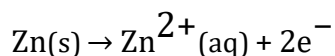
Calibrate the digital potentiometer with the help of the wires to display 1.018 V. The metal electrode is sensitized by dipping in a small quantity of 1:1 nitric acid containing a small quantity of sodium nitrite until effervescence occurs. Then the electrode is washed well with distilled water. 50 mL of the given concentration of the electrolyte solution is taken in a beaker and its corresponding metal electrode is introduced. This is connected with the saturated calomel electrode (half-cell) by means of a salt bridge. The metal electrode is connected to the positive terminal of and the calomel electrode is connected to the negative terminal of the potentiometer. The emf of the cell ( $E_{\text{cell}}$ ) is measured and noted in Table 1. The standard electrode potential [ $E^\circ \text{M}/\text{M}^{2+}$ ] is computed using Nernst equation (eqn. no. 1).

**Construction of Daniel cell and measurement of its voltage with three different concentrations of Copper and Zinc Solutions:**

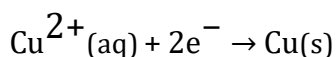


**Fig.1.** Daniel cell

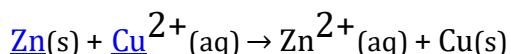
In the Daniel cell, copper and zinc electrodes are immersed in the equimolar solution of copper(II)sulfate and zinc(II)sulfate respectively. At the anode, zinc is oxidized as per the following half-reaction:



At the [cathode](#), copper is reduced as per the following reaction:



The overall reaction is:



Construct Daniel cell using the following concentrations of Copper and Zinc solutions and record the voltage of the cells and enter into the Table 3.

**Table 3: EMF of Daniel Cell observed from three different concentrations of zinc and copper solutions**

Metal	Concentration (N)	Metal	Concentration (N)	EMF observed (E <sub>cell</sub> / V)
Zn/Zn <sup>2+</sup>	0.01 N	Cu/Cu <sup>2+</sup>	0.01 N	0.981
	0.05 N		0.05 N	0.994
	0.1 N		0.1 N	1.011

**Result:**

(a) The single electrode potential of zinc and copper systems are \_\_\_-0.698\_\_\_ and \_\_\_0.275\_\_\_ respectively for 0.01N

(b) The single electrode potential of zinc and copper systems are \_\_\_-0.704\_\_\_ and \_\_\_0.276\_\_\_ respectively for 0.05N

(c) The single electrode potential of zinc and copper systems are \_\_\_-0.715\_\_\_ and \_\_\_0.281\_\_\_ respectively for 0.1N

1. Potentials (V) of Daniel cell: \_\_\_0.981\_\_\_ for 0.01N

2. Potentials (V) of Daniel cell: \_\_\_0.994\_\_\_ for 0.05N

(f) Potentials (V) of Daniel cell: \_\_\_1.011\_\_\_ for 0.1N

**Evaluation of result:**

Sample No.	Experimental Value	Actual Value	Percentage of error	Marks awarded

END



## 5. ESTIMATION OF $\text{Fe}^{2+}$ BY POTENTIOMETRIC TITRATION

Expt. No.: 5

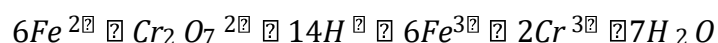
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### Aim

To estimate the amount of ferrous ion present in the whole of the given solution by potentiometric redox titration.

### Principle

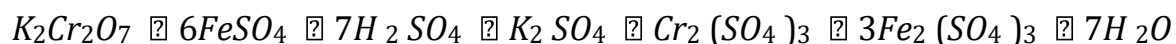
Potentiometric titrations depend upon the measurement of emf between the reference electrode and an indicator electrode. When a solution of ferrous ion is titrated with a solution of potassium dichromate, the following redox reaction takes place.



During this titration  $\text{Fe}^{2+}$  is converted into  $\text{Fe}^{3+}$ , whose concentration increases. At the end point, there will be a sharp change due to removal of all  $\text{Fe}^{2+}$  ions.

In this method a cell is set up which consists of calomel electrode as reference and an oxidation reduction system as indicator electrode. In this case the oxidation-reduction system consists of excess of  $\text{Fe}^{2+}$  ions which in turn is oxidized to  $\text{Fe}^{3+}$  ions by potassium dichromate during the course of titration.

**Hg /  $\text{Hg}_2\text{Cl}_2$  (s), saturated KCl // salt bridge //  $\text{Fe}^{3+}$ ,  $\text{Fe}^{2+}$  / Pt**



This reaction changes the activity of  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  ions in the solution. Thus change in electrode potential of  $\text{Fe}^{3+} / \text{Fe}^{2+}$  system takes place accordingly. Since the emf of the cell depends on electrode potential of indicator electrode, the emf measurement can be used to determine the equivalence point of titration of  $\text{Fe}^{2+}$  against dichromate solution.

The oxidation potential of this redox system is given by

$$E = E^0 + (RT/F) \ln [\text{Fe}^{3+}] / [\text{Fe}^{2+}]$$

Where  $E^0$  is the redox potential of the ferrous - ferric system R

$= 8.314 \text{ J/K/mol}$ ,

$F = 96500 \text{ coulombs}$

The potential difference between two electrodes which causes the flow of current from higher oxidation potential to lower oxidation potential is called emf of the cell.

When  $\text{K}_2\text{Cr}_2\text{O}_7$  is added to the system,  $\text{Fe}^{2+}$  is converted into  $\text{Fe}^{3+}$  whose concentration increases with the progressive addition of  $\text{K}_2\text{Cr}_2\text{O}_7$ . The observed emf will gradually increase. At the end point, there will be a sharp increase due to sudden removal of all  $\text{Fe}^{2+}$  ions. A plot between emf measured E Vs volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  added and  $\Delta E / \Delta V$  Vs average volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  added is drawn and the end point is determined graphically.

### **Calibration of potentiometer:**

Switch on the potentiometer and connect the standard cell terminals to either channel A (move channel switch to position A) or channel B (move the channel switch to position B). The meter should read 1.018 V. In case it is not 1.018 V, adjust the std. knob to obtain that value.

### **Procedure:**

20 mL of made up  $\text{Fe}^{2+}$  solution is pipetted out into a clean 100 mL beaker and a platinum electrode is inserted. A cell is formed by connecting this with saturated calomel electrode, through a salt bridge and connected to the potentiometer. One mL of potassium dichromate solution is added to the beaker, stirred well and emf is measured. The titrant is added in steps by 1 mL and corresponding emf is measured. When the rise of emf is rapid the amount is reduced to 0.2 mL per step and the measurements are continued till the rise is more gradual. The titrant added is increased to 1mL per step beyond the equivalence point. Record the emf reading after each addition.

### **Plot two graphs by taking**

1. EMF in y-axis and volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  added in x-axis,
2.  $\Delta E / \Delta V$  in y-axis and average volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  added in x-axis.

From the graph the end point is determined and from the endpoint the strength and hence the amount of  $\text{Fe}^{2+}$  ion present in one litre of the given solution is calculated.

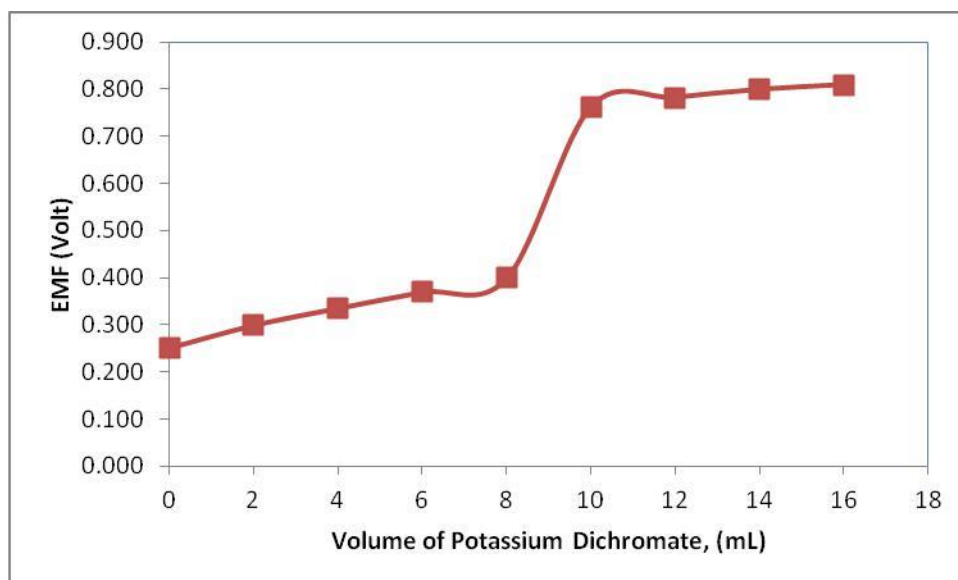
Equivalent Weight of ferrous ammonium sulphate is 392.

Equivalent Weight of  $\text{Fe}^{2+}$  ion is 55.85.

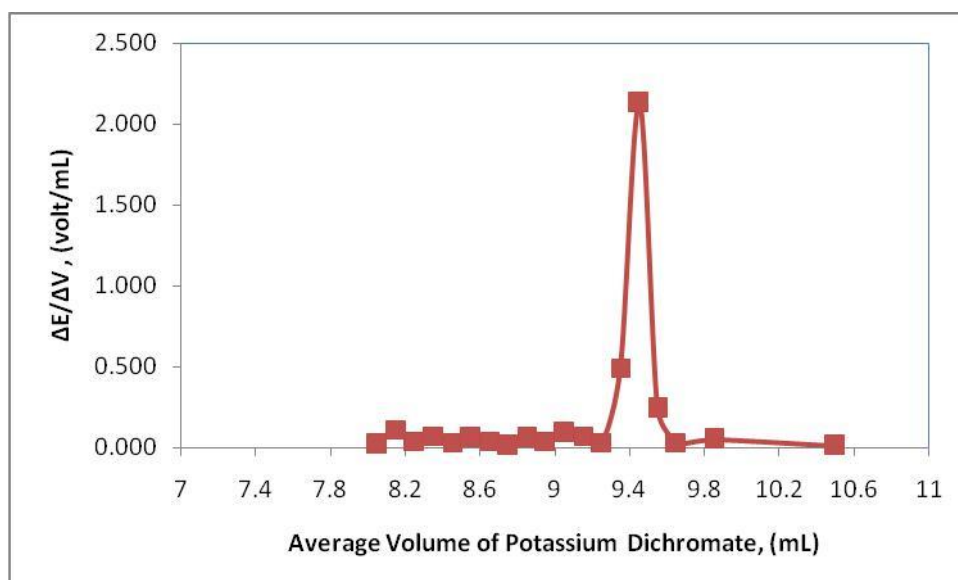
**Result:**

1. Equivalence point determined from the graph = 9.3mL.
2. The amount of  $\text{Fe}^{2+}$  ion present in one litre of the given solution = 1.298 g

**A graph between volume Vs EMF**



**A graph between  $V_{avg}$  Vs  $\Delta E / \Delta V$**



**Observation and Calculation:****Rough titration**

Strength of Standard Potassium dichromate = 0.01 N

Volume of FAS solution = 20 mL.

S. No.	Volume of K <sub>2</sub> CrO <sub>4</sub> (mL)	EMF (volt)
1	0	305
2	1	344
3	2	367
4	3	379
5	4	391
6	5	401
7	6	412
8	7	424
9	8	440
10	9	465
11	10	553
12	11	563
13	12	565
14	13	570

--	--	--

### Fair Titration

Strength of Standard Potassium dichromate = 0.01 N

Volume of FS solution = 20 mL.

Sl. No.	Volume of K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ( mL)	EMF (Volt)	$\Delta E$ (Volt)	$\Delta V$ (mL)	$\Delta E/\Delta V$ (Volt/mL)	Average Volume (mL)
1	8.4	441	5	0.2	25	8.5
2	8.6	446	7	0.2	35	8.7
3	8.8	453	6	0.2	30	8.9
4	9	459	7	0.2	35	9.1
5	9.2	466	124	0.2	620	9.3
6	9.4	590	3	0.2	15	9.5
7	9.6	593	3	0.2	15	9.7
8	9.8	596	3	0.2	15	9.9
9	10	599	3	0.2	15	10.1
10	10.2	602	3	0.2	15	10.3
11	10.4	605		0.2		

### CALCULATIONS:

Volume of  $K_2Cr_2O_7$  (from derivative graph) ( $V_1$ ) = 9.3 mL.

Normality of  $K_2Cr_2O_7$  ( $N_1$ ) = 0.05 N.

Volume of  $Fe^{2+}$  solution ( $V_2$ ) = 20 mL.

Normality of  $Fe^{2+}$  solution ( $N_2$ ) = ?

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V_1N_1}{V_2}$$

$$= 0.02325 \text{ N.}$$

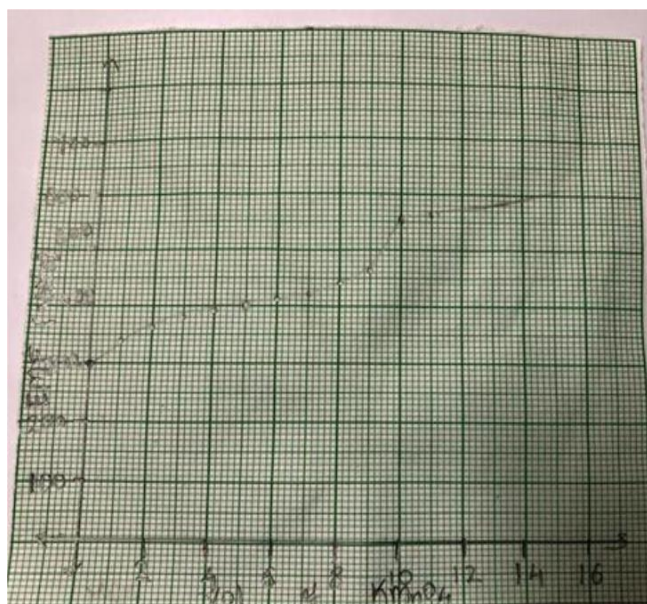
Amount of  $Fe^{2+}$  present in 1 litre of the given solution = Eq. wt. (55.85) X Normality

$$= 1.2985 \text{ g}$$

Amount of  $Fe^{2+}$  present in whole of the given solution =

$$[\text{Eq. Wt. (55.85) X Normality}] / 10$$

$$= 0.1298 \text{ g}$$



EXP-6

NAME: NITYASREE

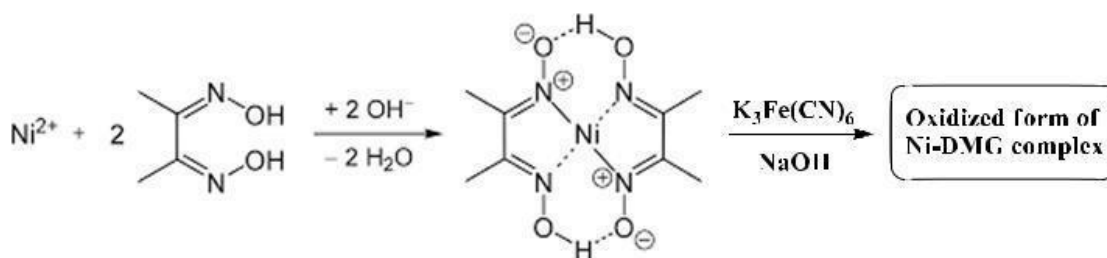
REGNO: 17MIS1007

**ESTIMATION OF NICKEL IN A NI-PLATED MATERIAL FOR CORROSION PROTECTION BY COLORIMETRY**

Date:

**Principle:**

Dimethylglyoxime reacts with nickel ions and forms a pink colored  $\text{Ni}(\text{dmg})_2$  complex in alkaline medium. Nickel forms a brown-red, water soluble oxidized nickel-dimethylglyoxime complex with potassium ferricyanide in alkaline medium as shown in the scheme below. The absorption spectrum of the oxidized complex shows absorption maximum at a wavelength of 440 nm (Figure 1). After drawing calibration graph the determination of nickel concentration in a given unknown sample can be done by colorimetry applying Beer-Lambert law.



Scheme

**Solutions required**

1. **Steel sample containing Nickel (II) solution** – Steel sample is prepared by dissolving steel containing 1 mg/mL of Ni(II) (1000 ppm).
2. **Potassium Ferricyanide solution** –  $\text{K}_3\text{Fe}(\text{CN})_6$  is prepared by dissolving 1.5 g of  $\text{K}_3\text{Fe}(\text{CN})_6$  in a 100 mL volumetric flask with distilled water.
3. **Dimethylglyoxime in NaOH solution** – 2.0 g of dimethylglyoxime and 8 g of NaOH are dissolved together in 25 mL distilled water and then made up to 100 mL volumetric flask with distilled water.



**Sodium Hydroxide solution** – Dissolve 40 g of NaOH in 1000 mL of distilled water.

**Procedure:**

In five 100 mL standard volumetric flasks add 1.00 mL of dimethylglyoxime solution by a pipette followed by 1.00 mL of potassium ferricyanide solution through a burette. Then add 1.00, 2.00, 3.00, 4.00, and 5.00 mL of the steel containing nickel(II) solution from a burette to the flask. All flasks are shaken well and made up to 100 mL mark with 1N NaOH solution. Allow the flasks to rest for 15 minutes after the addition of all the reagents for the complete complex formation. Then the absorbance of the formed brown-red solution can be measured at 440 nm against the distilled water blank. Set the colorimeter to this wave length range using appropriate filters. Record the absorbance of each one of the solutions using water as blank and enter in Table-1. Prepare the unknown sample in a similar way and record its absorbance also. Draw a calibration graph taking concentration of nickel (II) as X-axis and absorbance readings as Y-axis and a straight line that passes through the origin is obtained as shown in Figure From the calibration plot, measure the concentration of nickel (II) in the unknown sample is calculated.

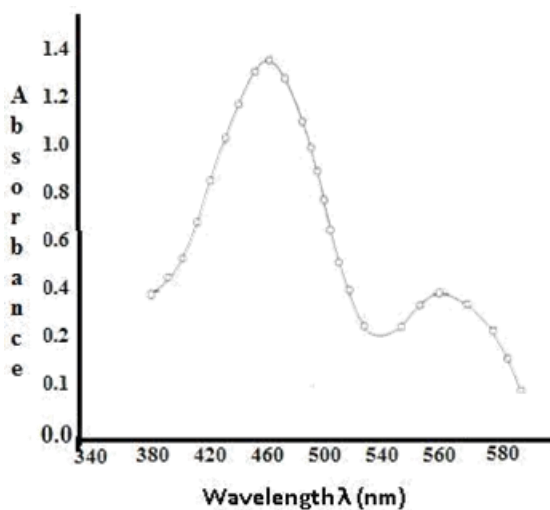


Figure 1: Absorption spectrum of oxidized Ni(II)-DMG complex showing  $\lambda_{\text{max}}$  at 440 nm

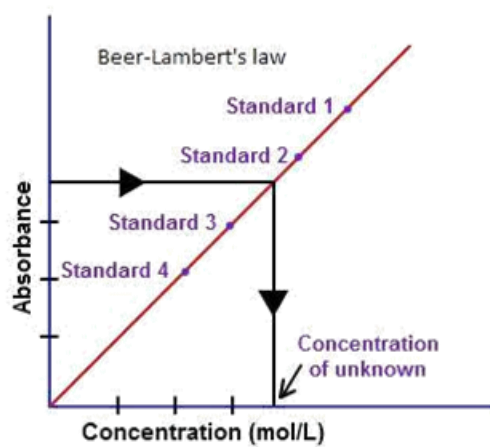
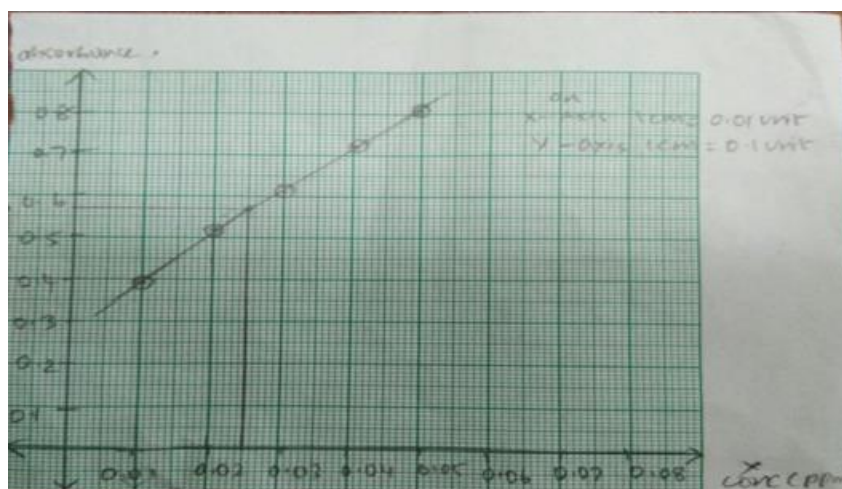


Figure 2: Model calibration curve for Ni(II) estimation

Table-1: Experimental Data

S. No	Concentration (X axis)/ppm	Absorbance (Y axis)
1.	0.01	0.39
2.	0.02	0.52
3.	0.03	0.61
4.	0.04	0.72
5.	0.05	0.80
6.	Unknown Ni(II)	0.57



### Result

Concentration of Nickel in the steel sample = 0.025 ppm (mg/L)

### Evaluation of result

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded

## **EXP-7**

**REG:17mis1007**

**NAME:P.NITYASREE**

### **DETERMINATION OF MOLECULAR WEIGHT OF A POLYMER BY VISCOSITY**

#### **METHOD**

Ex No :

Date:

#### **Aim**

To determine the average molecular weight of polyvinyl alcohol by viscosity method.

#### **Principle**

Each solvent has its own viscosity. Addition of even a very small amount of a polymer sample to a solvent of low viscosity results in a sharp increase in its viscosity. The extent of increase in its viscosity depends on the molecular weight of the polymer, in addition to other factors such as concentration, size of solute molecules.

The intrinsic viscosity  $[\eta]$  is related to the average molecular weight by the Mark-Houwink empirical equation.

$$[\eta] = K \eta M^a$$

Where K and 'a' are constants for a particular (polymer, solvent, temperature) system. From the above formula, the value of M – the molecular weight of the polymer can be determined.

#### **Reagents required**

Poly vinyl alcohol, distilled water.

#### **Glassware / Equipment Required**

Ostwald viscometer, thermometer, thermostat, stop clock, 50 mL standard

flasks.

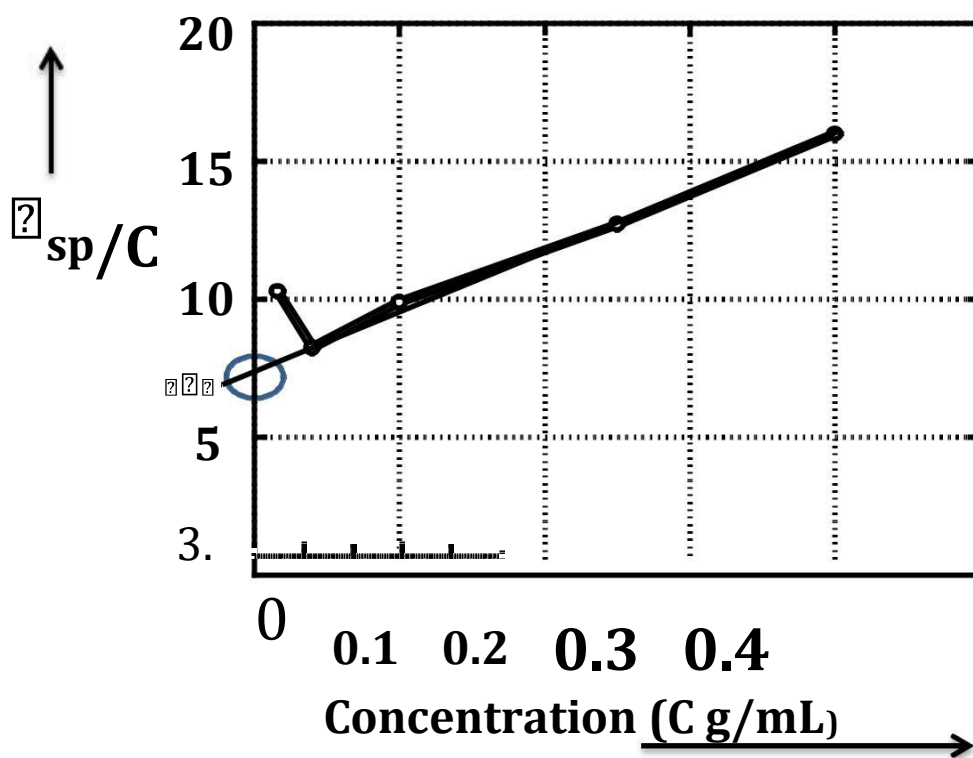
## **Procedure**

### **Preparation of various concentrations of poly vinyl alcohol in water**

Normally, a 1 w/v % solution of poly vinyl alcohol in water will be supplied. Prepare at least four dilutions viz 0.2%, 0.4%, 0.6% and 0.8% polymer in water before carrying out the experiment.

Rinse the Ostwald viscometer with a little of water. Fill it with 10mL pure water and keep it in a constant temperature thermostat maintained at 30 °C for 10 to 15 minutes so that the solvent attains the temperature of water bath (30 °C). By keeping the upper mark of the small reservoir of viscometer parallel to eyes, allow the solvent to flow down to the lower mark and note down the time in seconds. This is known as the 'efflux time'. Repeat the same procedure for at least 3 times to get the average efflux time for pure water. This time is  $t_0$ .

# OBSERVATION & CALCULATION



Volume of liquid used for each measurement = 10 mL

Solvent used = Water

Temperature = 30 °C

$K = 4.53 \times 10^{-4}$  (for PVA in water)

$a = 0.64$  (for PVA in water)

$[\eta] = K \eta M^a$

$\log [\eta] = \log K + a \log M$

$a \log M = \log [\eta] - \log K$

$\log M = \frac{\log [\eta] - \log K}{a}$

$M = \text{Anti log } \frac{\log [\eta] - \log K}{a}$

Repeat the same procedure to determine the flow rate for various diluted solutions of poly vinyl alcohol in water and note down their flow time in seconds,  $t_{s1}$ ,  $t_{s2}$ ,  $t_{s3}$  etc.

Note that each time a particular solution of concentration is used; the viscometer should be cleaned with little water and then rinsed with solution of next concentration before filling viscometer with the same. Tabulate values to get value of  $\eta_{sp} / C$ . A plot of  $\eta_{sp} / C$  versus concentration  $C$  (g/100 mL) gives a straight line. Extrapolate this line to meet the y-axis to get value of  $[\eta]$ . Substitute this in the Mark-Houwink equation to get  $M$ .

**Table**

**58**

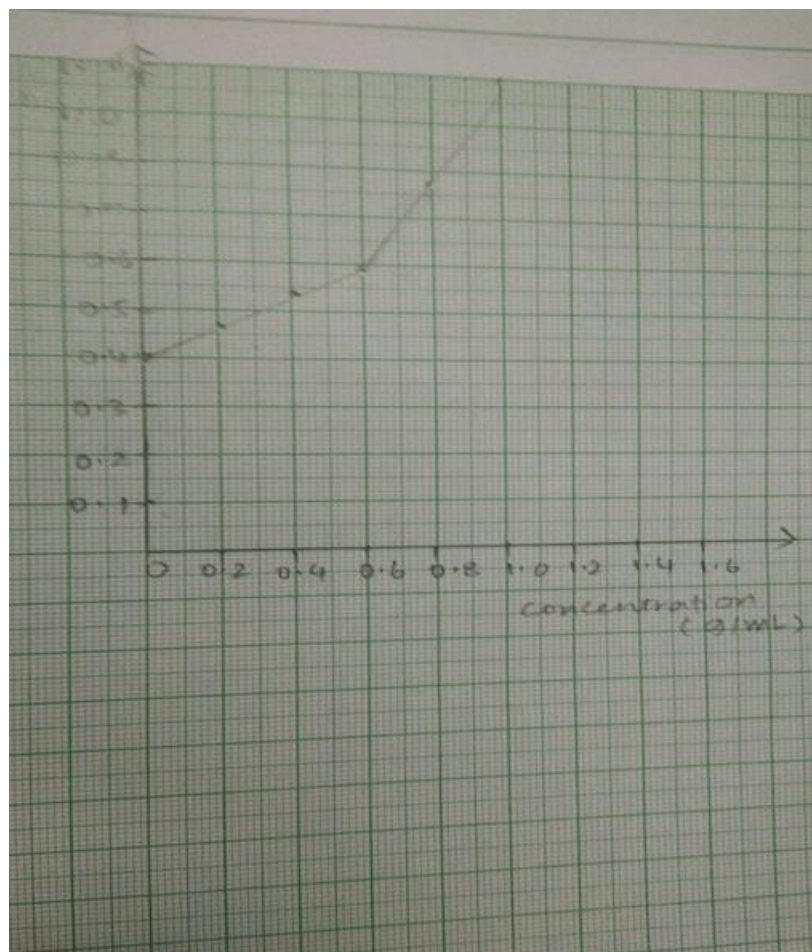
Eflux time of pure H<sub>2</sub>O (t<sub>0</sub>) = ----- Seconds

Concentration of polymer C (g/dl)	Eflux time (sec.) t <sub>s</sub>	Relative viscosity $\eta_r = t_s/t_0$	Specific viscosity $\eta_{sp} = \eta_r - 1$	Reduced viscosity $\eta_{red} = \eta_{sp}/C$
<b>0.2</b>	<b>63.4</b>	<b>1.093</b>	<b>0.093</b>	0.465
<b>0.4</b>	<b>70.6</b>	<b>1.217</b>	<b>0.217</b>	0.542
<b>0.6</b>	<b>78.4</b>	<b>1.351</b>	<b>0.351</b>	<b>0.585</b>
<b>0.8</b>	<b>93.6</b>	<b>1.613</b>	<b>0.613</b>	<b>0.766</b>
<b>1.0</b>	<b>123.2</b>	<b>2.124</b>	<b>1.124</b>	<b>1.124</b>

**Result:**

The average molecular weight of the given  
 \_\_\_\_\_40086.67\_\_\_\_g/mole\_\_\_\_\_ polyvinyl alcohol in water\_\_\_\_\_ at 30°C is  
 \_\_\_\_40086.67\_\_\_\_\_Daltons



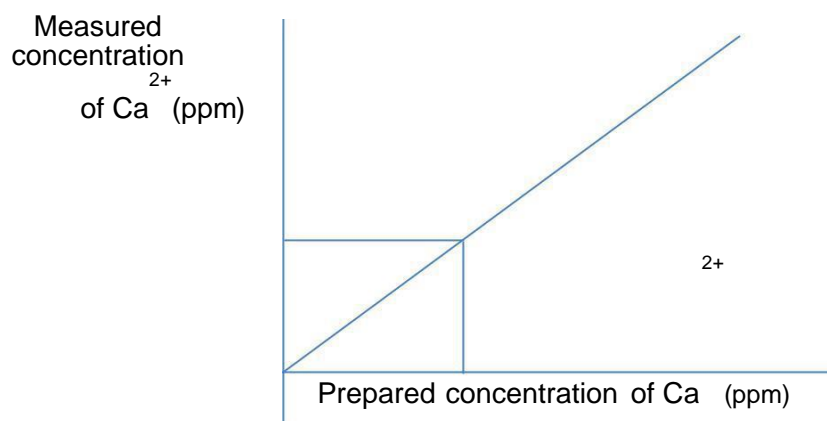


## Experiment-8

### ESTIMATION OF CALCIUM HARDNESS IN WATER BY FLAME PHOTOMETRY

Date:

#### Principle:



Express the result of calcium content in **ppm**.

**Table-1: Experimental Data**

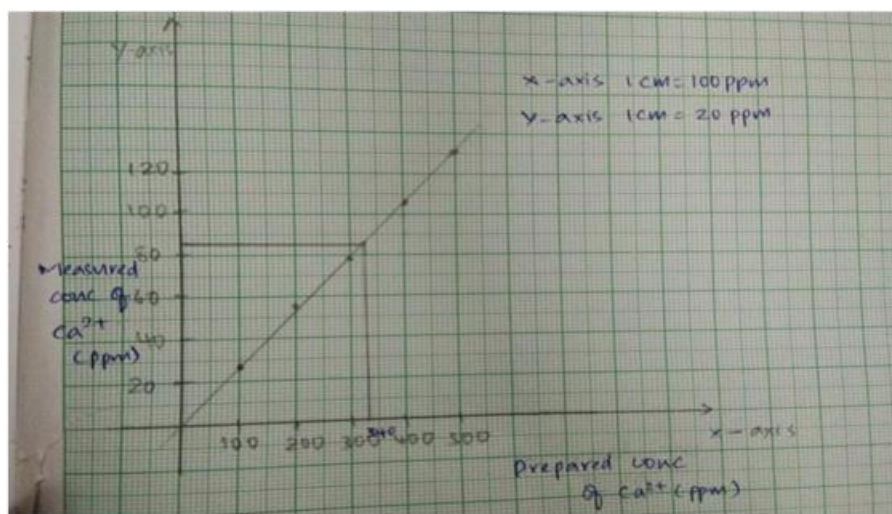
S. No	Prepared concentration of $\text{Ca}^{2+}$ (X axis)/ppm	Measured concentration of $\text{Ca}^{2+}$ (Y axis)/ppm
1.	100	28
2.	200	55
3.	300	79
4.	400	108
5.	500	125
6.	Unknown $\text{Ca}^{2+}$	85

## Result

The amount of calcium present in the given water sample = 340 ppm

## Evaluation of result

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded



**Principle:**

The estimation of calcium is based on the emission spectroscopy, which deals with the excitation of electrons from ground state to higher energy state and coming back to its original state with the emission of light. Trace amount of calcium can be determined by flame emission photometry at a wavelength of 423 nm. The intensity of light at 423 nm is proportional to the concentration of the element. After careful calibration of photometer with solution of known composition, it is possible to correlate the intensity of a spectral line of unknown solution with the amount of an element present that emits the particular radiation.

**Reagents**

2. Distilled water
3. Calcium stock solution – 1000 ppm stock (Dissolve 2.497 g  $\text{CaCO}_3$  in 1-2 mL conc. HCl and dilute to 1L accurately).
4. Prepare five different concentrations of calcium solution in the range of 20 ppm – 200 ppm in 50 mL standard flasks by making up with distilled water.
5. Along with these solutions, prepare given unknown sample in another 50 mL
6. standard flask.

**Apparatus:**

Burette, graduated pipette, beakers, rubber Bulb, standard volumetric flasks (50 mL), Flame photometer and accessories.

**Procedure:**

**Setting the instrument for estimation of Calcium**

1. Start the electrical supply and switch on the air supply. Stabilize the air and the needle should be steady at the mark to maintain about 0.5 Kg/cm<sup>2</sup> pressure.
2. Switch on the gas and maintain the gas fuel mixture so that blue flame is seen through the viewing window.
3. Adjust the flame so that the flame is seen as 5 cones each in two parallel rows.
4. Follow the operating instructions of the instrument kept near to the instrument..
5. Following the instructions seen on the screen, carry out calibration using the five diluted solutions of calcium. Stop when "CALIBRATION OVER" display comes.
6. Then select "SAMPLE" from the menu.
7. Each one of the solutions should be aspirated as sample and the concentration reading displayed by the instrument should be noted down.
8. A plot should be made as prepared concentration vs displayed concentration.
9. Simultaneously record the concentration of calcium from the given unknown by introducing unknown as sample and report the result.

**END**

**P.NITYASREE**  
**17MIS1007**

### **Experiment-9**

#### **DETERMINATION OF AROMATIC CONTENT IN DIESEL BY ANILINE POINT MEASUREMENT**

**Date:19-3-18**

#### **Principle:**

**Definition of Aniline point (AP):** AP is the lowest temperature at which the sample is homogeneous i.e. completely miscible with equal volume of aniline.

**Mixed Aniline point (MAP):** MAP is the minimum equilibrium solution temperature of a mixture of two volume of aniline, one volume of sample and one volume of *n*-heptane of specific purity.

Aniline point (AP) is characteristic of petroleum fractions that indicate the degree of aromaticity of hydrocarbon mixtures. Lower the aniline point, the higher the aromatics content in the fuel oil. The higher the aromatics content, the lower the cetane number of the fuel. The aniline point can thus be used to indicate the probable ignition behaviour of a diesel fuel.

**Requirements:** Aniline point apparatus, thermometer, electric heating device, pipette etc.

**Procedure:**

Clean and dry the 'U' tube and arrange the apparatus desired. Now add 5 mL of distilled aniline and 5 mL of given sample in the 'U' tube, it will form two layers. Arrange the apparatus with stirrers in such way that liquid in 'U' tube and the water in the beaker are stirred simultaneously. Switch on the heater to heat the water at a controlled rate, so there is no much difference in temperature of paraffin bath and 'U' tube.

The minimum temperature at which two layers give a single phase is noted as aniline point. Now stirring of 'U' tube mixture is stopped, and mixture is allowed to cool. Temperature at which two layers are formed is also considered as aniline point.

For mixed aniline point the above procedure is repeated by taking 2.5 mL of sample, 2.5 mL of *n*-heptane and 5 mL of aniline.



## OBSERVATION AND CALCULATIONS

### Observations:

#### (A) Aniline point:

- (1) Vol. of Aniline taken = 5ml
- (2) Vol. of Kerosene/ Diesel taken = 5ml
- (3) Cloud formation temp. = 68 degree Celsius
- (4) One phase temp. (Aniline point) = 75 degree Celsius
- (5) Two phases formation temp. = 65 degree Celsius

#### (B) Mixed Aniline point:

- (1) Vol. of Aniline taken = 5ml
- (2) Vol. of *n*-heptane taken = 2.5ml
- (3) Vol. of Kerosene/ Diesel taken = 2.5ml
- (4) Cloud formation temp=  
**65 degree Celsius**
- (5) One phase temp. (Aniline point) = 68 degree Celsius
- (6) Two phases formation temp=  
**60 degree Celsius**

**Result:**

(1) Aniline point of the given sample = 75 degree celsius

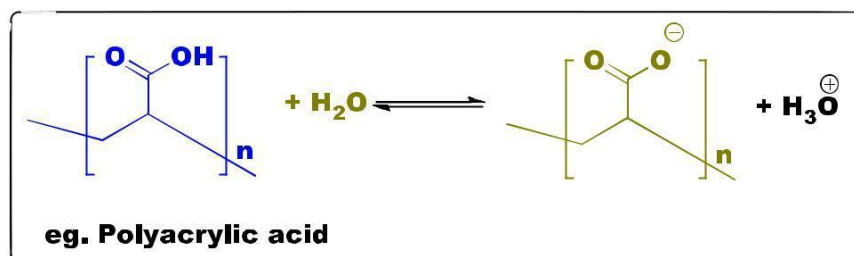
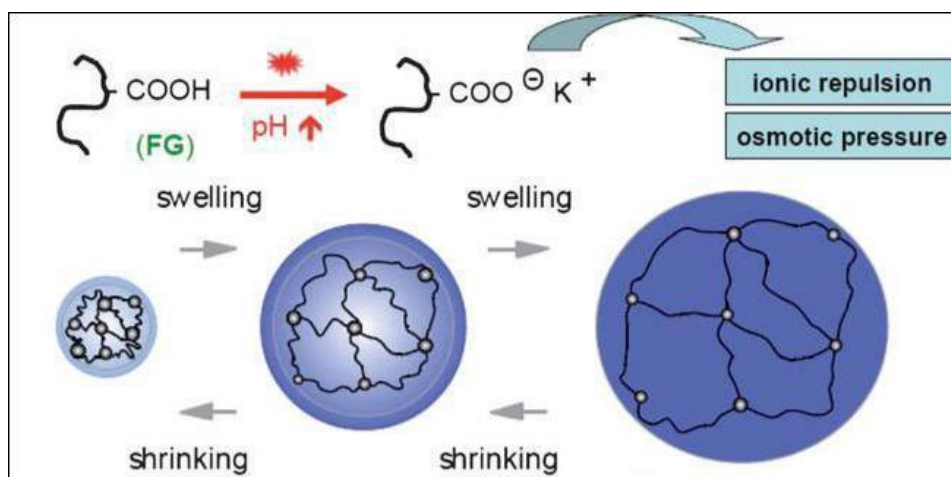
(2) Mixed Aniline point of the given sample = 68 degree celsius

**Evaluation:**

**END**

**WATER PRESERVATION THROUGH SMART MATERIALS (HYDROGEL)****Date:19-3-18****Principle:**

Hydrogels are polymeric cross-linked network structures obtained from a class of synthetic and/or natural polymers which can absorb and retain significant amount of water due to the presence of hydrophilic groups or domains (Scheme 1). Water is brought into the network through the process of osmosis and quickly is absorbed into the central part of the polymer network. This is mainly due to the affinity of these functional groups towards water (e.g., due to the protonation/deprotonation as a result of changes in pH and temperature) and leading to a reversible change in the swelling degree as shown in Scheme 1.

**Scheme 1:** Absorption of water by the polymer chain of a hydrogel**Figure 1:** Bulk hydrogels with specific functional groups (FG) – the different solvation water depending on environmental conditions (e.g., pH, or temperature) leads to large reversible changes in the degree of swelling.

Hydrogels find applications mainly in tissue engineering, pharmaceutical, and biomedical and agricultural fields. Due to their high water absorption capacity and biocompatibility they are now being used in wound dressing, drug delivery, sanitary pads as well as trans-dermal systems, dental materials, implants, injectable polymeric systems, ophthalmic applications, hybrid-type organs.

**Table-1**

Sl. No.	Wt. of polymer taken, g	Wt. of polymer after swelling, g	Amount of water absorbed, mL
1	0.5	94.645	
2	0.5	94.645	
3	0.5	94.645	
Average	0.5	94.645	94.145

Swelling index of the polymer material with deionized water = 18829%

**Table-2**

Sl. No.	Wt. of polymer taken, g	Amount of ionic compound added	Wt. of polymer after swelling, g	Amount of water absorbed, mL
1	0.5	0.1	59.118	
2	0.5	0.1	59.118	
3	0.5	0.1	59.118	
Average	0.5	0.1	59.118	58.618

Swelling index of the polymer material with ionic impurities = 11723.6 %

**Table-3**

Sl. No.	Wt. of polymer taken, g	Amount of neutral compounds added	Wt. of polymer after swelling, g	Amount of water absorbed, mL
1	0.5	1.0	87.91	
2	0.5	1.0	87.91	
3	0.5	1.0	87.91	
Average	0.5	1	87.91	87.410

Swelling index of the polymer material with neutral impurities =17482%

### **Required apparatus and chemicals**

250 mL beakers -3, Glass rods-3, Filter papers -3, Funnels -2

Deionized water, Polymer material, NaCl, Sucrose, Acetone, Methanol, Ethanol, Butanol, and Urea.

### **Procedure**

#### **Part-A:**

A small amount of [superabsorbent polymer](#) material is placed in the beaker (0.5 g, note down the exact weight of the polymer) and then 100 mL of deionized water is poured into the beaker. After 5 min the swollen polymer was separated by using a [filter paper](#) and note down the weight of the gel. By weighing the gel, one can find the swollen capacity of the polymer taken.

The swelling percentage of hydrogels was calculated based on Eq-1, which consists of the difference between the initial and the final weight of the sample divided by the initial weight. Here  $W_s$  corresponds to the weight of the swollen gels and  $W_d$  is the weight of dry polymer before immersion into the water.

$$\text{Swelling}(\%) = \frac{W_s - W_d}{W_d} \times 100$$

----Eq.1

## Part-B

The swelling behavior of polymers is determined by ionic and neutral impurities in water. A small amount of [dry polymer](#) material is placed in the beaker (0.5 g, note down the exact weight of the polymer) and then 0.1% NaCl solution (prepared by adding 0.1 g of NaCl in 100 mL of deionized water) is poured into the beaker. After 5 min the swollen polymer was separated by using a [filter paper](#) and note down the weight of the gel. By weighing the gel, one can find the swollen capacity of the polymer taken as per the following equation.

$$\text{Swelling(\%)} = \frac{W_s - W_d}{W_d} \times 100$$

## Part-C

A small amount of [dry polymer](#) material is placed in the beaker (0.5 g, note down the exact weight of the polymer taken) and then 1 % sucrose solution (prepared by adding 1 g of sucrose in 100 mL of deionized water) is poured into the beaker. After 5 min the swollen polymer was separated by using a [filter paper](#) and note down the weight of the gel. By weighing the gel, one can find the swollen capacity of the polymer using the given equation.

$$\text{Swelling(\%)} = \frac{W_s - W_d}{W_d} \times 100$$

Swelling index of the polymer material with neutral impurities =

## OBSERVATION AND CALCULATIONS

### Result

Swelling index of the polymer material with deionized water = 18829%\_\_\_\_\_

Swelling index of the polymer material with ionic impurities = 11723.6%\_\_\_\_\_

Swelling index of the polymer material with neutral impurities = 17482%\_\_\_\_\_

**END**



