

**RAJALAKSHMI ENGINEERING COLLEGE (AUTONOMOUS)**

**THANDALAM – 602105.**



**RAJALAKSHMI  
ENGINEERING COLLEGE**

**DEPARTMENT OF HUMANITIES AND SCIENCES – CHEMISTRY**

**CHEMISTRY LABORATORY MANUAL**

**CY23131- CHEMISTRY FOR ELECTRONICS ENGINEERING**

**R - 2023**

**FOR FIRST SEMESTER B.E. ECE, EEE & BME**



## **General instructions to the students**

### **I. Laboratory safety**

The following points are important and must be followed to avoid any kind of accidents inside the laboratory:

- a) Do not smoke, drink and eat inside the laboratory.
- b) Wear laboratory coat and safety goggles.
- c) Do not perform any unauthorized experiments without the permission of the instructors.
- d) For any kind of chemical burn, wash the area with plenty of water and inform your instructor promptly.
- e) Do not pipette out liquids by mouth use suction bulbs.
- f) Do not try to smell, taste any chemicals without the instructions of the concerned person.
- g) Do not add water to the concentrated acids.
- h) Do not touch any unknown chemicals by hands.
- i) Take help from lab instructors for electrical connections and circuits.
- j) Before leaving the laboratory, wash your hands properly.
- k) Know the location of the exits, safety showers, fire extinguishers and first aid kit.

### **II. General rules**

- a) Try to understand the experiment before you start it. If you have any query, discuss with the instructor.
- b) If some reagent/apparatus is not found, ask your laboratory attendant.
- c) Finish the experiments within the specified time period.
- d) Regular submission of the laboratory note book is mandatory. Late submission will not be entertained for evaluation.
- e) Never use mobile phones inside the laboratory.



## CONTENTS

S.No.	Experiment	Page No.
<b>1</b>	<b>Construction and determination of EMF of simple electrochemical cells and concentration cells</b>	<b>3</b>
<b>2</b>	<b>Estimation of acids by pH metry</b>	<b>5</b>
<b>3</b>	<b>Determination of corrosion rate on mild steel by weight loss method</b>	<b>9</b>
<b>4</b>	<b>Estimation of mixture of acids by conductometry</b>	<b>13</b>
<b>5</b>	<b>Estimation of extent of corrosion of iron pieces by potentiometry</b>	<b>17</b>
<b>6</b>	<b>Estimation of copper / ferrous ions by spectrophotometry</b>	<b>21</b>
<b>7</b>	<b>Estimation of DO by using sensors</b>	<b>25</b>
<b>8</b>	<b>Estimation of concentration of sulphate/chloride ions in the given sample solution.</b>	<b>29</b>
<b>9</b>	<b>Determination of molecular weight of a polymer by viscometry method.</b>	<b>33</b>
<b>10</b>	<b>Synthesis of nanomaterials by simple precipitation method</b>	<b>37</b>
	<b>Viva – Voce questions</b>	<b>39</b>

## Observations

Electrode system	Electrolyte concentration(M)	Observed cell potential (mV)	Ecalomel(mV)	Potential of electrode system(mV)

## Calculations:

$$E^{\circ}_{\text{Cell}} = E^{\circ}_{\text{R}} - E^{\circ}_{\text{L}}$$

$$\text{Standard Electrode Potential } E^{\circ} =$$

# **CONSTRUCTION AND DETERMINATION OF EMF OF SIMPLE ELECTROCHEMICAL CELLS AND CONCENTRATION CELLS**

**Exp. No.**

**Date:**

**Aim:** To determine the standard electrode potential of copper /zinc /silver electrode system.

## **Principle:**

The standard electrode potential of an electrode system can be determined by coupling with the reference electrodes such as calomel electrode. The influence of the concentration of the electrolyte on electrode potential can be studied by measuring the potential at different concentrations. The emf of concentration cell can be determined by constructing a cell with either zinc or copper electrodes immersed in different concentrations of the respective electrolytes.

## **Procedure:**

Prepare 100ml of 0.01N, 0.005, 0.0025M of  $\text{ZnSO}_4/\text{AgNO}_3/\text{CuSO}_4$  solution. Standardize the potentiometer using Weston-cadmium cell (E). Set up a galvanic cell by dipping Cu/Zn/Ag electrode in 0.1M  $\text{CuSO}_4/\text{ZnSO}_4/\text{AgNO}_3$  solution respectively, kept in a beaker and the reference electrode (Calomel) through a salt bridge. Measure the emf of the cell using different concentrations of  $\text{CuSO}_4/\text{ZnSO}_4/\text{AgNO}_3$  electrolytes.

## **Result:**

Electrode potentials of the three systems under study are as follows:

Electrode system	Electrolyte concentration	Potential (mv)

## SHORT PROCEDURE

CONTENTS	Estimation of Acid Std. NaOH Vs Acid	MODELGRAPH
Burette solution	Std. NaOH	
Pipette solution	20ml Acid	
Additional solution	30ml distilled water	
Electrode/Cell used	Ion selective electrode/pH electrode/combined electrode	
Instrument used	Digital pH meter	
End point	Sudden/rapid increase in pH value	

## Std. NaOH Vs HCl

S.No	Volume of NaOH (ml)	pH	$\Delta p\text{H}$	$\Delta V$	$\Delta p\text{H}/\Delta V$
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					
21.					
22.					
23.					
24.					
25.					
26.					
27.					
28.					
29.					

## ESTIMATION OF ACID BY pH METRY

**Exp. No. 2**

**Date:**

### **AIM**

To determine the strength and amount of given HCl by pH metry. A standard solution of -----N NaOH is provided.

### **PRINCIPLE**

The pH of a solution is related to  $H^+$  ions concentration by the following formula.

$$pH = -\log_{10} [H^+]$$

When NaOH is added slowly to HCl,  $H^+$  ions get neutralized by  $OH^-$  ions. The pH increases slowly.



When all  $H^+$  ions of HCl are neutralized at the end point, addition of NaOH causes high increase in pH because of the excess  $OH^-$  ions.

### **PROCEDURE**

Fill the given Standard Sodium hydroxide solution in a clean burette. Transfer the given solution of HCl carefully into a 100ml std.flask and make it up to the mark using distilled water. Now pipette out 20 ml of the HCl solution into a clean beaker. Then dilute it to 50ml with distilled water. Dip a glass electrode into the solution and connect to a pH meter.

Now add NaOH gradually from the burette to HCl solution taken in the beaker. Note the pH of the solution for each addition of NaOH. Continue the titration until at least 5 readings after the endpoint. Plot a graph between  $\Delta pH/\Delta V$  and the volume of Sodium hydroxide.

Draw a graph as depicted in the diagram. The volume corresponding to the peak  $\Delta pH/\Delta V$  is the sharp Equivalence Point and it is equal to the volume of NaOH exactly required for neutralization of the strong acid.

**Calculation:**

Volume of NaOH (V<sub>1</sub>) = ----- ml (from graph)

Strength of NaOH (N<sub>1</sub>) = ----- N

Volume of HCl (V<sub>2</sub>) = 20 ml

Strength of HCl (N<sub>2</sub>) =  $\frac{(V_1 \times N_1)}{V_2}$

=

Strength of HCl = ----- N

Amount of HCl present = Strength of HCl × Equivalent weight of HCl.

in the given solution

= ----- N × 36.5

= ----- g/l

## **RESULT**

Strength of the given HCl solution = ----- N

Amount of HCl present in the given solution = -----g/l

**Table-1: Corrosion rate – Weight loss method**

S.No	Initial Weight w <sub>1</sub> g	Final Weight w <sub>2</sub> g	Weight loss (w <sub>1</sub> - w <sub>2</sub> ) g	Concentration of HCl (N)	Time of Exposure (Minutes)	Area (cm) <sup>2</sup>
1.						
2.						

$$\text{Rate of corrosion} = \frac{(W_1 - W_2)}{DAT} \text{ cm / year}$$

$$T = \frac{\text{Time of Exposure in minutes}}{60 \times 24 \times 365}$$

$$= \text{-----} \text{ cm /year}$$

Where,

w<sub>1</sub> = Initial weight of the specimen

w<sub>2</sub> = Final weight of the specimen

D = Density of the specimen (For mild steel, D 7.85 g/cm<sup>3</sup>)

T = Time of exposure in minutes

$$T = [\text{Time of Exposure in minutes} / (60 \times 24 \times 365)]$$

$$A = \text{Area of the specimen in (cm)}^2$$

As the corrosion is a slow process, the other units used for rate of corrosion are

- (a) millimeter per year (mpy)
- (b) inch per year (ipy)
- (c) mg per dm per day (mdd)

# **DETERMINATION OF CORROSION RATE ON MILD STEEL BY WEIGHT LOSS METHOD**

**Exp. No. 3**

**Date:**

## **AIM**

Determine the rate of corrosion of mild steel specimen in hydrochloric acid of different concentrations at room temperature by weight loss method

## **MATERIALS REQUIRED**

1. Mild steel pieces
2. Electronic balance
3. Abrasive (sand) paper
4. Air drier
5. Stop watch
6. Hydrochloric acid

## **PRINCIPLE**

Almost all metals undergo corrosion. Corrosion is an undesirable and slow process. The cost of replacement of materials and equipments lost through corrosion is estimated to be more than one lakh crore rupees per year. Thus, corrosion is regarded as a cancer that destroys economy and equipments. Generally metal surfaces are covered with impurities like rust and scales. These impurities at the time of coating, will produce porous and discontinuous coating. In order to obtain an adherent cohesive, smooth and uniform coating, the impurities must be completely removed by dipping them in an acid. This is known as acid cleaning or *pickling*.

Pickling is generally employed to remove the scale by oxidation of metal.



Dilute hydrochloric acid is used as pickling solution. By dipping the metal plate in the acid, the metal ions along with rust get dissolved. Such dissolution is called *corrosion*. It is observed that as the time of dipping in acid increases and the acid concentration increases, the rate of corrosion also increases.

## **PROCEDURE**

Mild steel specimens of known dimensions are taken and are washed thoroughly with distilled water and are air dried. The initial weight of the specimens is noted ( $w_1$  g). Different concentrations (0.5, 1 N, 2 N, 3 N, 4 N, 5 N) of hydrochloric acid solutions are prepared. They are known as *pickling solutions*. The mild steel specimens are dipped in 100 mL of various



hydrochloric acid solutions for about 30 minutes. After that they are taken out and are washed with distilled water thoroughly and are air dried. Again the specimens are weighed. The final weights are noted ( $w_2$  g). From the weight loss ( $w_1 - w_2$ ), the rate of corrosion can be calculated using the formula.

## **RESULT**

- (i) Rate of corrosion (.....N) = -----cm/year
- (ii) Rate of corrosion (.....N) = -----cm/year

## SHORT PROCEDURE

CONTENTS	Estimation of SA and WA Std. NaOH Vs SA+WA	MODELGRAPH
Burette solution	Std. NaOH	
Pipette solution	20ml acid mixture (SA+WA)	
Additional solution	30ml distilled water	
Electrode/Cell used	Conductivity cell	
Instrument used	Digital Conductivity meter	
End point	1. Decrease to increase of conductance 2. slow to rapid increase of conductance	

### Std. NaOH Vs Strong acid

S.No.	Volume of NaOH added (ml)	Observed conductance ( $10^{-3}$ mho or m mho)
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		
21.		
22.		
23.		
24.		
25.		
26.		

# CONDUCTOMETRIC TITRATION OF MIXTURE OF ACIDS

Exp. No. 4

Date:

## AIM

To determine the strength and amounts of strong acid (SA) and weak acid (WA) present in the given acid mixture. A Standard solution of NaOH of strength ..... N is provided.

## PRINCIPLE

Solution of electrolytes conducts electricity due to the presence of ions. Since Specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured continuously during the titration, to find the end point.



For example , when a mixture of HCl and CH<sub>3</sub>COOH is titrated with NaOH the fast moving hydrogen ions are progressively replaced by slow moving sodium ions to form salt and water. As a result, conductance of the solution decreases. This decrease will take place until the HCl neutralisation point is reached. Further addition of alkali results in formation of sodium acetate. Since sodium acetate is a stronger electrolyte than acetic acid, conductance slowly increases until all acetic acid is completely neutralized(II-Neutralisation). Furthermore addition of alkali increases the conductance sharply due to the addition of fast moving OH<sup>-</sup> ions.

## PROCEDURE

Fill up the burette with NaOH solution up to zero mark. Make up the given unknown solution (mixture of a weak & a strong acid) to 100ml in a standard flask with distilled water. Pipette out exactly 20ml of the made up solution into a clean 100ml beaker and dilute it to 50ml using distilled water. Introduce a conductivity cell into the solution and connect the terminals to a conductivity meter.

Add NaOH from the burette to the Acid mixture in the beaker in terms of 0.5ml increments. Stir the solution using a glass rod, and observe the conductance values for every addition. Continue the titration after the endpoint, for a minimum of 3ml of the burette solution. The conductance values decrease initially, then gradually increase and finally show a steep increase. The titration hence shows two endpoints (ie) I neutralization for (strong acid) & II neutralization for (weak acid).

Calculate the strength and amount of the acids using the endpoints obtained from the graph.

**(i) Calculation of strength of Strong Acid**Volume of NaOH (V<sub>1</sub>) = ----- (a) ml (from graph)Strength of NaOH (N<sub>1</sub>) = ----- NVolume of HCl (V<sub>2</sub>) = 20 mlStrength of HCl (N<sub>2</sub>) = -----?

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

=

Strength of HCl = ----- N

Amount of HCl present in the solution = Strength of HCl × 36.5 (eq.wt of HCl)

= ----- g/l.

**(ii) Calculation of strength of Weak Acid**Volume of NaOH (V<sub>1</sub>) = ----- (b-a) ml (from graph)Strength of NaOH (N<sub>1</sub>) = ----- NVolume of CH<sub>3</sub>COOH (V<sub>2</sub>) = 20 mlStrength of CH<sub>3</sub>COOH (N<sub>2</sub>) = -----?

$$N_2 = (V_1 \times N_1) / V_2$$

=

Strength of CH<sub>3</sub>COOH = ----- NAmount of CH<sub>3</sub>COOH present in the solution =Strength of CH<sub>3</sub>COOH × 60 (eq.wt of CH<sub>3</sub>COOH)

= g/l.

## **RESULT**

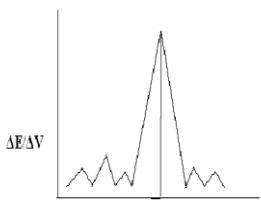
(i) The strength of acids present in the given unknown solution are

a) Strong acid = ----- N    b) Weak acid = ----- N

(ii) The amount of Strong acid present in the given solution = ----- g/l

(iii) The amount of Weak acid present in the given solution = ----- g/l

## SHORT PROCEDURE

CONTENTS	Estimation of Acid Std. $K_2Cr_2O_7$ Vs $FeSO_4$	MODELGRAPH
Burette solution	Std. Potassium dichromate	<p>MODEL GRAPH</p>  <p>Volume of Potassium dichromate</p>
Pipette solution	20ml Ferrous ion solution	
Additional solution	10ml dil. $H_2SO_4$ and 20ml distilled water	
Electrodes	Platinum-Indicator electrode Calomel- $2^{\circ}$ reference electrode	
Instrument used	Digital potentiometer	
End point	Sudden/rapid increase in emf Value	

## Std. $K_2Cr_2O_7$ Vs $FeSO_4$

S.No	Volume of Potassium Dichromate in ml	Emf $10^{-3}$ Volt	$\Delta E$ in mV ( $E_2 - E_1$ )	$\Delta V$ in ml ( $V_2 - V_1$ )	$\Delta E/\Delta V$ in mV/ml
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					
21.					
22.					
23.					
24.					
25.					
26.					
27.					
28.					
29.					

# **ESTIMATION OF EXTENT OF CORROSION OF IRON SPECIMEN BY POTENTIOMETRY**

**Exp. No. 5**

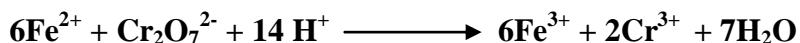
**Date:**

## **AIM**

To estimate the amount of extent of corrosion by estimating the ferrous ions present in the whole of the given solution by potentiometric titration using standard decinormal potassium dichromate.

## **PRINCIPLE**

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



During this titration,  $\text{Fe}^{2+}$  is oxidized to  $\text{Fe}^{3+}$  and its concentration increases. At the end point, there will be a sharp rise in the emf value. This is called “Sweeping potential”. It indicates complete formation of  $\text{Fe}^{3+}$  ions at the end of redox reaction.

The cell is set up by connecting this  $\text{Fe}^{2+}/\text{Fe}^3$  redox electrode with a calomel electrode as shown:



A graph is drawn between emf measured against the volume of potassium dichromate added. The end point is noted from the graph.

## **PROCEDURE**

### **Pipette Solution**

Iron pieces are taken and washed thoroughly and immersed in conc. Sulphuric acid overnight. This is then made upto 100 mL. Transfer the given ferrous solution into a 100ml standard flask and make it upto the mark using distilled water. Fill in standard potassium dichromate solution into the burette upto the mark.

Pipette out 20ml of ferrous solution into a 100ml clean beaker. Add 10ml of dil.  $\text{H}_2\text{SO}_4$  and 20ml of distilled water to it. Dip in a platinum electrode and a calomel electrode into this solution and connect to a potentiometer. Then add Potassium dichromate in terms of 0.5ml/addition from the burette to the solution in the beaker and stir it well. For each addition of the solution from the burette note the emf of the cell using the potentiometer. Continue the titration atleast six reading after the “Sweeping potential”.

## CALCULATIONS

Volume of Potassium dichromate  $(V_1)$  = ----- ml (from graph)

Strength of Potassium dichromate  $(N_1)$  = ----- N

Volume of Ferrous sulphate  $(V_2)$  = ----- ml

Strength of Ferrous sulphate  $(N_2)$  =  $\frac{(V_1 \times N_1)}{V_2}$

=

= ----- N

Amount of Ferrous ion present = Strength of ferrous  $\times$  55.85(eq.wt of Fe)

1000ml of the solution solution

=

= ..... g/l

Plot a first derivative graph by taking  $\Delta E/\Delta V$  versus volume of  $K_2Cr_2O_7$ . The strength of ferrous solution and the amount of ferrous ion present are calculated from the end point.

## **RESULT**

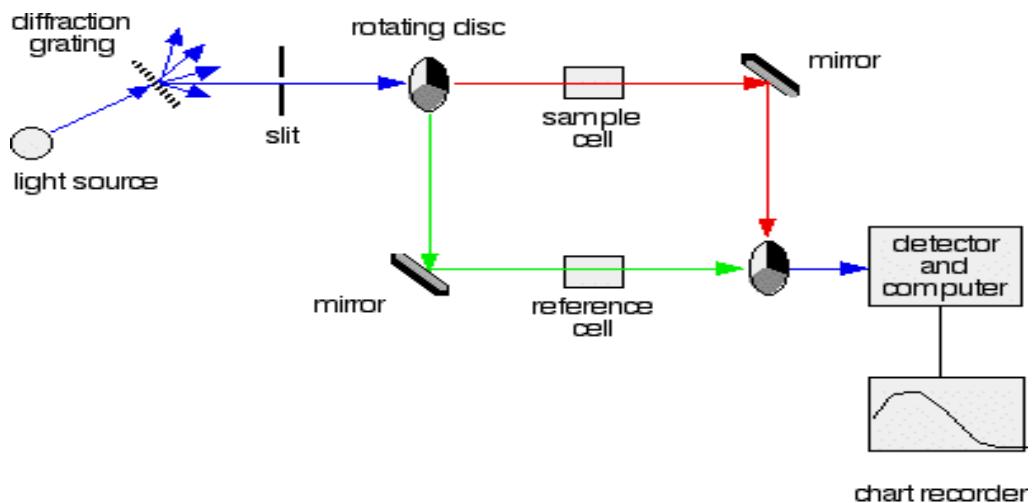
Strength of Ferrous sulphate = ----- N

The amount of Ferrous ion present in the given solution is = ----- g/l.

## SHORT PROCEDURE

CONTENTS	Estimation of Iron	MODELGRAPH
Cuvette Solution	Ferrous solution	
Additional solution	Potassium permanganate & Potassium thiocyanate	
Instrument used	Spectrophotometer	
End point	The absorbance values of every ppm of ferrous solution are noted	

## LAYOUT OF SPECTROPHOTOMETER



# ESTIMATION OF FERROUS IONS BY SPECTROPHOTOMETRY

Exp.No: 6

Date:

## AIM

To determine the amount of iron dissolved in the given sample by spectrophotometry

## PRINCIPLE

When a monochromatic light passes through a homogeneous coloured solution, a portion of incident light is reflected, a portion is absorbed and the remaining is transmitted.

$$I_o = I_r + I_a + I_t$$

Where,  $I_o, I_r$  = intensity of light entering solution and Intensity of light reflected

$I_a, I_t$  = intensity of light absorbed and Intensity of light transmitted

$I_r$  is usually eliminated and hence  $I_o = I_t + I_a$ . The mathematical statement of Lambert – Beer's law is given by

$$T = I / I_o = 10^{-\epsilon Cl} \text{ (or)} \quad A = \log I_o / I = \epsilon Cl$$

Where  $T$  = transmittance of solution

$I$  = intensity of light leaving solution (transmitted light)

$\epsilon$  = molar absorption co-efficient

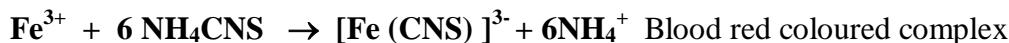
$c$  = concentration of the solution in moles/litre

$l$  = Path length of light through the solution in cm

$A$  = absorbance or optical density of the solution.

When a ray of monochromatic light passes through an absorbing medium, its intensity decreases exponentially as the concentration of the absorbing substance and the path length. Keeping the path length constant, say  $l=1$  cm, the variation in absorbance is with respect to only concentration, C.

$Fe^{3+}$  ions in low concentrations does not give any colour in solution. However, it develops a red colour when it reacts with  $NH_4CNS$  solution.



Further, this colour is in the blue region, ( $\lambda = 480$  nm). Spectrophotometer has a wide range of adaptability that allows selection of monochromatic light of any wavelength in the visible spectrum. Therefore spectrophotometry is used to find very low concentrations of  $Fe^{3+}$  ion solutions.

**Table – 1****Preparation of various concentration of Fe<sup>2+</sup> solution**

S.No	Volume of stock solution(mL)	Volume of dil. HNO <sub>3</sub>	Volume of 1% NH <sub>4</sub> SCN (mL)	Volume of distilled water	Concentration of Fe <sup>3+</sup>	
					ppm	N
1						
2						
3						
4						
5						
6						

**Table-2 Measurement of absorbance****Blank solution zero absorbance ,  $\lambda=480\text{nm}$** 

S.No	Concentration of Fe <sup>2+</sup> ions (ppm)	Absorbance
1		
2		
3		
4		
5		
6		

## **PROCEDURE**

### **a) Preparation of Stock solution:**

A stock solution of ferric iron is prepared by dissolving 0.0838g of ferrous ammonium sulphate with 1mL of con. $\text{HNO}_3$  and made up to 1 L in a standard flask using distilled water. This solution will be containing 10 ppm of iron 1 mL solution.

$$\text{i.e., } 1 \text{ mL stock solution} = 10 \text{ ppm of iron}$$

From this stock solution as given in the table-1 various concentrated (1, 2, 3, 4, 5 and 6) solutions are prepared.

Instead of various ppm  $\text{Fe}^{3+}$  solutions, various normal solutions (say 0.01, 0.02, 0.03, 0.04, 0.05 and 0.06) are prepared from 0.1 N  $\text{Fe}^{3+}$  stock solutions. This stock solution is prepared by dissolving 39.2g of ferrous ammonium sulphate in 1mL of conc. $\text{HNO}_3$  and made up to 1L in a standard flask using distilled water.

### **b) Estimation of iron content:**

The various standard solutions (0.4, 1.2, 2.0, 2.8, 3.6 ppm) and unknown concentration of the given ferric iron solution is taken in the given 25ml volumetric flask to which 1ml of 4N nitric acid and 1ml. of potassium thiocyanate solution are added to develop a red colour and such a solution is made up to the mark. It is shaken well to make it homogeneous in concentration. It is allowed to stand for 5 minutes to attain stability in colour.

The spectrophotometer is switched on and warmed up for 15 minutes. The spectrophotometer is adjust for  $\lambda=480\text{nm}$ . Blank solution (distilled water) is kept in the spectrophotometer and the transmittance is adjusted for 100 per cent; for which absorbance is zero. Now, the absorbance of all the standard solutions is measured. Then, absorbance of unknown solution also measured. From the absorbance of standard solutions a calibration graph is drawn and the concentration of unknown solution is obtained from the calibration graph. The equivalent weight of iron is 55.85. Using this, the amount of  $\text{Fe}^{2+}$  present in the unknown solution is calculated.

## **RESULT**

The amount of iron present in the given solution = ..... ppm



## ESTIMATION OF DO BY USING SENSORS

**Exp. No.7**

**Date:**

### **AIM**

To determine the dissolved oxygen present in given water sample.

### **Apparatus:**

Digital Dissolved Oxygen Meter.

### **Reagents:**

Potassium chloride solution (7.5%)

### **Theory:**

Oxygen dissolved in water, often referred to as DO, is a very important parameter of water quality and is an index of physical and biological processes going on in water. There are two main sources of dissolved oxygen in water i) Diffusion from air ii) Photosynthetic activity within water. Diffusion of oxygen from air to water is a physical phenomenon and depends upon solubility oxygen, which in turn, is influenced by factors like temperature, water movement, salinity etc.

### **DO Measurement Method:**

#### **Preparation of Solution**

- (i) Fill potassium chloride solution (7.5%) in the electrolyte tube. Take 7.5 gm of potassium chloride and mix it in 100 ml distilled water.
- (ii) 2% of sodium sulphite solution. Take 2 gm of sodium sulphite and mix it in 100 ml.

#### **DO probe Assembly:**

1. Fit the membrane on the lower part of the electrolyte tube. Be careful to handle its membrane.
2. Fix the ring in the electrolyte tube, covering the sides of the membrane.
3. Fill potassium chloride solution in the electrolyte tube to its top.
4. Insert the electrode in the electrolyte tube and tightly screw it up. Now the probe is ready for use.

**DISSOLVED OXYGEN SATURATION  
VALUE OF DISTILLED WATER**

<b>°C</b>	<b>Oxygen Solubility (ppm)</b>	<b>°C</b>	<b>Oxygen Solubility (ppm)</b>	<b>°C</b>	<b>Oxygen Solubility (ppm)</b>
21	8.82	25	8.06	29	7.60
22	8.67	26	8.22	30	7.50
23	8.41	27	7.90	31	7.40
24	8.36	28	7.76	32	7.30

**Table**

<b>S. No</b>	<b>Types of Water</b>	<b>Temperature (°C)</b>	<b>DO value</b>
1	Distilled Water		
2	Tap Water		
3	Waste Water		

## **Procedure**

1. Set the temperature knob to the solution temperature and CAL knob to extreme right position, and Zero knob to extreme left position.\
2. Place the DO probe in 2% sodium sulphite solution. Allow the display to attain equilibrium. Set the Zero knobs to bring the display to read 0.00 by keeping temperature knob to actual temperature of the solution. Calibrate knob should be at extreme right position.
3. Now calibrate the instrument with known value of DO solution.
4. Take 250 ml flask and fill 2/3 of it with distilled water.
5. Stopper the flask and take it for 20 seconds. Remove the stopper back and swirl the water back and repeat the procedure 4 times.
6. Measure the temperature of water and set the temp knob, knob a temp. of water in the flask.
7. Hold the probe in the flask and agitate the water. If necessary adjust the meter reading with CAL knob.
8. Now, the instrument is ready to take measurements of DO value of any unknown solution.

## **Precautions:**

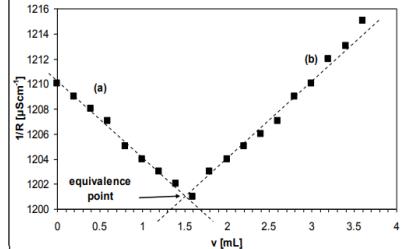
1. 7.5 % KCL solution acts as a good electrolyte in DO probe. Presence of any other salt in electrolyte solution may damage the probe.
2. The electrolyte tube should not contain any air bubble. If it is present, it will cause fluctuations in the readings.
3. Movement of the solution should be about 2 ft/sec for accurate measurement.

## **Result:**

Dissolve Oxygen present in the following water sample is

- (i) Distilled Water = ----- mg/l  
(ii) Tab water = ----- mg/l  
(iii) Waste water = ----- mg/l

## SHORT PROCEDURE

CONTENTS	Estimation of sulphate ions/Chloride ions Std. $\text{Na}_2\text{SO}_4$ Vs $\text{BaCl}_2$	MODELGRAPH
Burette solution	Std. $\text{Na}_2\text{SO}_4$	
Pipette solution	20ml $\text{BaCl}_2$	
Additional solution	30ml distilled water	
Electrode/Cell used	Conductivity cell	
Instrument used	Digital Conductivity meter	
End point	Decrease to increase of conductance	

Std.  $\text{Na}_2\text{SO}_4$ Vs  $\text{BaCl}_2$

S.No.	Volume of $\text{Na}_2\text{SO}_4$ added (ml)	Observed conductance ( $10^{-3}$ mho or m mho)
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		
21.		
22.		
23.		
24.		
25.		
26.		
27.		
28.		
29.		
30.		

# **ESTIMATION OF CONCENTRATION OF SULPHATE/CHLORIDE IONS IN THE GIVEN SAMPLE SOLUTION**

**Exp. No. 8**

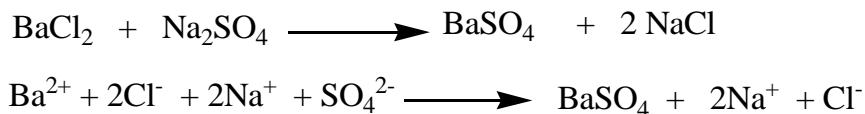
**Date:**

## **AIM**

To determine the strength and amount of BaCl<sub>2</sub> present in the given sample. A Standard solution of Na<sub>2</sub>SO<sub>4</sub> of strength ..... N is provided.

## **PRINCIPLE**

Solution of electrolytes conducts electricity due to the presence of ions. Since Specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured continuously during the titration, to find the end point. The titration of BaCl<sub>2</sub> vs Na<sub>2</sub>SO<sub>4</sub> is given as an example. In the precipitation titration, the ions are converted into insoluble precipitate which will not contribute to the conductance. When Na<sub>2</sub>SO<sub>4</sub> is added slowly from the burette to the solution of BaCl<sub>2</sub>, BaSO<sub>4</sub> gets precipitated while the chloride ions remain unchanged. The reaction between BaCl<sub>2</sub> vs Na<sub>2</sub>SO<sub>4</sub> is represented as,



The Ba<sup>2+</sup> ions in the solution are replaced by free Na<sup>+</sup> ions. Since the mobility of Na<sup>+</sup> ions are less than that of Ba<sup>2+</sup> ions, the conductance of the solution decreases.

After the end point, when all the Ba<sup>2+</sup> ions are replaced, further addition of Na<sub>2</sub>SO<sub>4</sub> increases the conductance. This is due to the increase of Na<sup>+</sup> and SO<sub>4</sub><sup>2-</sup> ions in the solution.

## **PROCEDURE**

Stock solution of Na<sub>2</sub>SO<sub>4</sub> was prepared by dissolving solid sodium sulphate in distilled water. All the working solutions were obtained by dilution from this stock solution. The solution of BaCl<sub>2</sub> was prepared also by dissolving of solid reagent in distilled water.

The burette is washed well with water and rinsed with the given Na<sub>2</sub>SO<sub>4</sub> solution. It is then filled with the same Na<sub>2</sub>SO<sub>4</sub> solution upto zero level. 20 ml of the given BaCl<sub>2</sub> solution is pipetted out into a clean 100 ml beaker. The conductivity cell is placed in it and then diluted to 50 ml by adding distilled water. The two terminals of the cell are connected with a conductivity bridge.

## Calculation of strength of sodium sulphate

Volume of  $\text{Na}_2\text{SO}_4$  ( $V_1$ ) = ----- (a)ml (from graph)

Strength of  $\text{Na}_2\text{SO}_4(N_1)$  = ----- N

Volume of  $\text{BaCl}_2(V_2)$  = 20 ml

Strength of  $\text{BaCl}_2(N_2)$  = -----?

$$N_2 = \frac{(V_1 \times N)}{V_2}$$

=

Strength of  $\text{BaCl}_2$  = ----- N

Amount of  $\text{BaCl}_2$  is present in the solution = Strength of  $\text{BaCl}_2 \times$

Eq.wt of  $\text{BaCl}_2$ )

$$= \dots \text{N} \times 122.14$$

=----- g/l.

Amount of  $\text{Cl}_2$  is present in the solution = Amount of  $\text{BaCl}_2 \times \frac{70}{122.}$

$$= \dots \text{N} \times \frac{70}{122.}$$

=----- g/l.

Now 0.5 ml of  $\text{Na}_2\text{SO}_4$  from the burette is added to the solution, taken in the beaker, stirred, and then conductivity is measured. This is continued upto the end point. (The conductivity is going on decreasing upto the end point). After the end point, again the  $\text{Na}_2\text{SO}_4$  is gradually added and few more readings are noted.

Thus, the conductivity is continuously measured for each addition of  $\text{Na}_2\text{SO}_4$  and is tabulated. Now the graph is plotted between the volume of  $\text{Na}_2\text{SO}_4$  and conductivity. The end point is the intersection of the two curves. From the volume of  $\text{Na}_2\text{SO}_4$ , the strength and the amount of  $\text{BaCl}_2$  present in given solution is calculated.

## **RESULT**

- (i) The strength of the given  $\text{BaCl}_2$  is found to be = ----- N
- (ii) The amount of the  $\text{BaCl}_2$  present in the given solution = ----- g/l
- (iii) The amount of the  $\text{Cl}_2$  present in the given solution = ----- g/l

## Observation

Solvent used = Water  
 K of the polymer solvent system = -----  
 a of the polymer-solvent system = -----  
 Volume of liquid taken for finding the flow time = 10ml  
 Flow time of the solvent ( $t_0$ ) = ----- sec

S.No.	Conc. g/dl or (%)	Flow time (t)sec	$\eta_r = t / t_0$	$\eta_{sp} = \eta_r - 1$	$\eta_{sp} / C$

S.No.	Polymer	Solvent	$K \times 10^{-5}$	a
1	Polyvinyl alcohol	Water	45.3	0.64
2	Polyvinyl pyrrolidone	Water	39.3	0.59
3	Polystyrene (atactic)	Benzene	11.5	0.73
4	Polystyrene (isotactic)	Benzene	10.6	0.735

## DETERMINATION OF MOLECULAR WEIGHT OF A POLYMER BY VISCOMETRY METHOD.

**Exp. No. 09**

**Date:**

### **AIM**

To determine the molecular weight (M) of a polymer by viscometry method.

### **PRINCIPLE**

Viscosity average method is based on the flow behavior of polymer solutions. According to Mark – Hawnik equation, the intrinsic viscosity of a polymer is given as

$$[\eta]_{\text{int}} = KM^a$$

Where,

M = molecular weight of the polymer

K & a are constants for a particular polymer – solvent system

$[\eta]_{\text{int}}$  = Intrinsic viscosity =  $[\eta_{\text{sp}}/C]_{C=0} = [\eta_r/C]_{C=0}$

$\eta_{\text{sp}}$  = specific viscosity =  $\eta_r - 1$

$\eta_r$  = relative viscosity =  $\eta/\eta_0 = t/t_0$

Since accurate measurement of absolute viscosity is a difficult task, relative viscosity is taken into account for finding the molecular weight.

$\eta$  = Viscosity of the polymer solution

$\eta_0$  = Viscosity of the pure solvent

t = flow time of the polymer solution

$t_0$  = flow time of the pure solvent

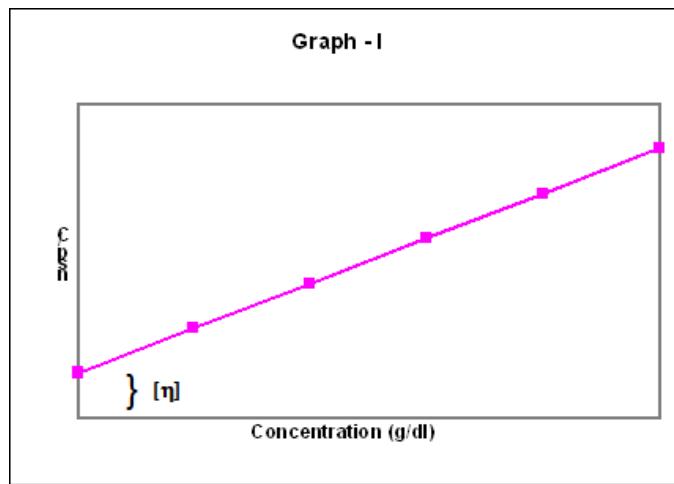
The flow time of the polymer solution (t) and that of the pure solvent ( $t_0$ ) are found experimentally and substituted to get  $\eta_{\text{sp}}$ ,  $\eta_r$  and  $[\eta]_{\text{int}}$ .

Knowing K & a, molecular weight of the polymer solution is calculated.

$DP = M/m$  (M= mol.wt of polymer, m = mol. wt of monomer)

### **PROCEDURE**

Accurately take 1g weight of polyvinyl alcohol, dissolve it in water and make up to 100ml (1dl) in a standard flask. From the solution, Prepare the polymer solutions of concentration 0.1g/dl, 0.2g/dl, 0.3g/dl, 0.4g/dl and 0.5g/dl using the relation  $V_1N_1 = V_2N_2$



$$[\eta] = KM^a$$

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

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

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

$$M = \text{antilog} \left\{ \frac{\log[\eta] - \log K}{a} \right\}$$

$$\text{Degree of polymerization} = \frac{\text{Molecular weight of the polymer}}{\text{Molecular weight of the repeating units}}$$

$$\text{Degree of polymerization} =$$

[E.g. for preparing 100ml of 0.2g/dl from 1g/dl solution x ml polymer is made up to 100ml with water where x is caculated using the relation,  $x \text{ ml} \times 1 \text{ g / dl} = 0.2 \text{ g / dl} \times 100\text{ml}$ ]

Rinse the Ostwald viscometer with water, and then fill with 10ml of distilled water. Using a rubber bulb, suck the water in the viscometer to the upper bulb. Allow the water to flow from the upper mark till the lower mark in the viscometer. Using a stop clock measure the time taken for water to flow from the upper mark to the lower mark and note the value as  $t_0$  sec.

Drain the water from the viscometer completely and take 10ml of the polymer solution of 0.1g/dl concentration in it. Note the flow time of the polymer solution as  $t$  sec. Repeat the procedure with the other solutions of the polymer.

Calculate  $\eta_{sp} / c$  from the values of  $t$  and  $t_0$ , and draw the graph taking  $\eta_{sp} / c$  Vs  $c$ . Extrapolate the straight line obtained to zero concentration. The intercept value is equal to  $[\eta]_{int}$ . Calculate the molecular weight of the polymer ( $M$ ) using the formula

$$[\eta]_{int} = KM^a \quad (\text{constant } K \text{ and } a \text{ are given in the table})$$

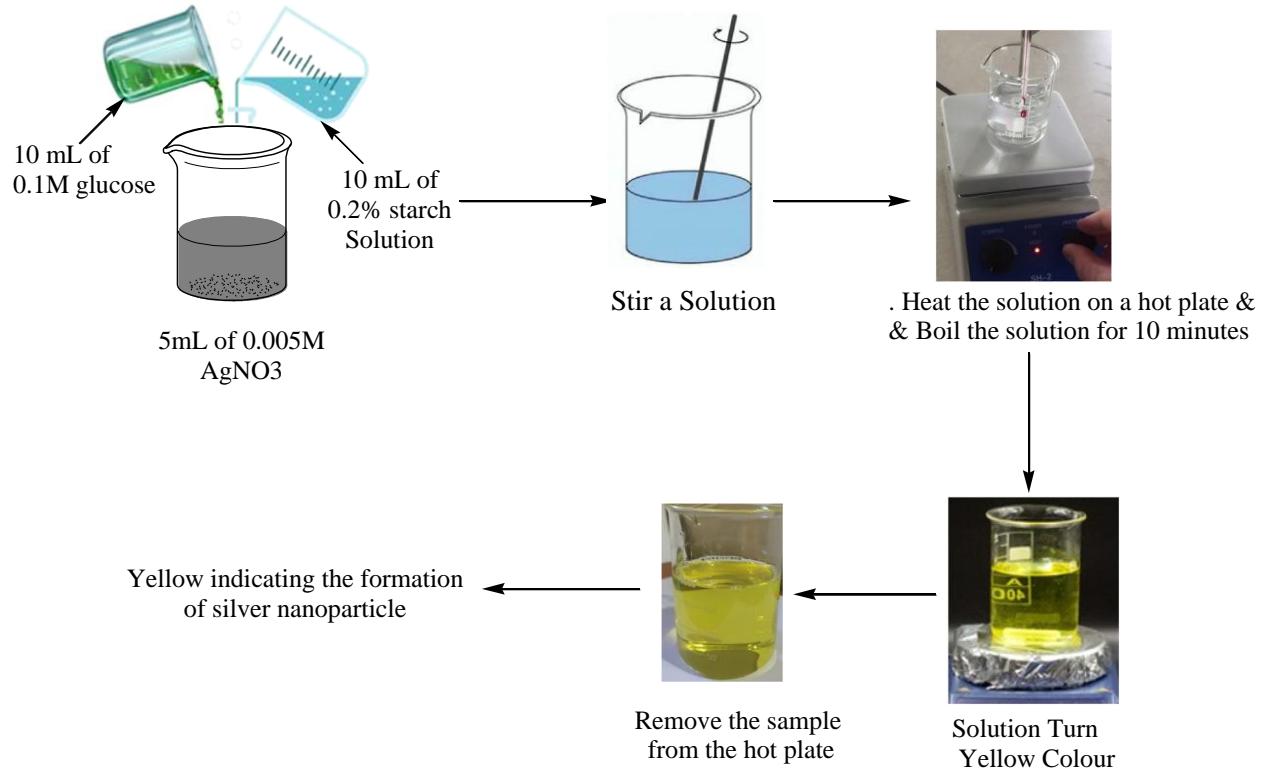
Calculate the degree of polymerization from the values of  $M$  and  $m$ .

## RESULT

The molecular weight of the given polymer =

The degree of polymerization of the polymer =

## Synthesis of Nanoparticles



# SYNTHESIS OF NANOPARTICLES

## Exp. No. 10

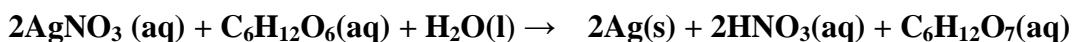
Date:

**Aim:** To synthesize Nanoscale silver from silver nitrate using D-glucose

### Principle:

Nanoparticles are made of thousands of atoms of an element and are extremely small, ranging from 1 to 100 nanometers (nm) in size. A nanometer is 1 billionth of a meter or  $1 \times 10^{-9}$ . Nanoparticles often have different properties than those associated with the element at the macro scale. For example, silver, gold, and copper interact with light differently at the nanoscale which in turn affects the color.

Silver Nanoparticles (AgNPs), or colloidal silver, will be synthesized in the presence of starch according to the following redox reaction:



In this reaction, glucose ( $\text{C}_6\text{H}_{12}\text{O}_6$ ) reduces the silver cations from the silver nitrate. As the silver metal forms, starch coats the outsides of the particles, preventing them from aggregating and forming larger particles. Nano-sized materials often have different properties than the bulk materials; for example, silver nanoparticles appear yellow

### Procedure

#### Synthesis of Ag Nanoparticles:

1. Place 5mL of 0.005M  $\text{AgNO}_3$  into a small Erlenmeyer flask or beaker.
2. Add 10 mL of 0.1M glucose, making sure that it comes into contact with the  $\text{AgNO}_3$ .
3. Invert the 0.2% starch solution several times. Add 10mL of the starch solution.
4. Heat the solution on a hot plate on a high setting until it is boiling vigorously without stirring the solution
5. Boil the solution for 10 minutes. The solution should turn yellow.
6. Remove the sample from the hot plate, and let it cool.

### Result

The colour changes to yellow indicating the formation of silver nanoparticles.



## **VIVA-VOCE – QUESTION BANK**

**Experiment: 1. Construction and determination of emf of simple electrochemical cells and concentration cells**

- 1. What is single electrode Potential?**
- 2. What is standard electrode potential?**
- 3. Define EMF**
- 4. What is a concentration cell?**
- 5. Give some examples for reference electrodes.**

**Experiment: 2 Estimation of acids by pH metry**

- 1. Define pH**
- 2. Give the principle of pH metry ?**
- 3. Name the electrodes used in the determination of pH of a solution.**
- 4. Which electrode act as anode and which acts as cathode? Why?**
- 5. What is the effect of temperature on pH?**
- 6. What is the effect of dilution on pH of an acid solution?**
- 7. What is a combined electrode?**
- 8. Brief the function of calomel electrode?**

**Experiment:3 Determination of corrosion rate on mild steel by weight loss method**

- 1. What is weight loss method?**
- 2. Define corrosion.**
- 3. What are the types of corrosion?**
- 4. What is the formula for Rate of Corrosion?**
- 5. What is Rust. Give its formula?**
- 6. What is galvanic corrosion?**

**Experiment: 4 Estimation of mixture of acids by conductometry**

- 1. Define the term conductivity.**
- 2. Give the unit of conductance.**
- 3. What is specific conductance?**
- 4. List out the advantages of conductometric titrations.**

5. Which acid will be neutralised first when a mixture of HCl and acetic acid is titrated against NaOH?
7. Why the conductance decrease until the first neutralization point?
8. Why at the second neutralization point, conductance increases slowly and then shows steep rise?

**Experiment:5 Estimation of extent of corrosion of iron pieces by potentiometry**

1. What is an electrochemical cell?
2. Give the formula to calculate EMF of a cell
3. Name the electrodes acting as cathode and anode in the experiment
4. What is redox titration?
5. Mention the redox reaction that takes place when  $\text{FeSO}_4$  is titrated against  $\text{K}_2\text{Cr}_2\text{O}_7$ .
6. Platinum electrode is a oxidation – reduction electrode in the presence of  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  Why?
7. What is calomel electrode?
8. What is the reduction potential of calomel electrode?
9. Give the advantages of Potentiometric titrations.

**Experiment: 6 Estimation of copper / ferrous ions by spectrophotometry**

1. What is the principle of spectrophotometry?
2. How is the light from a monochromatic source gets distributed on falling upon a medium?
3. What is absorbance?
4. What is a spectrophotometer?
5. State Beer-Lamberts law.
6. What is the role of KCNS in the estimation of iron by spectrophotometry?
7. Why is the monochromator adjusted for  $\lambda = 480\text{nm}$  during the estimation of iron?
8. Mention the working range of wave length of spectrophotometer?
9. What are the components of spectrophotometer?

**Experiment: 7 Estimation of DO by using sensors**

1. What is the extent of oxygen dissolution in water at  $30^\circ\text{C}$ ?
2. What is the need of determining the amount of DO in water?
3. What are the impacts of low DO levels?

**4. Which method is used for estimation of dissolved oxygen?**

**5. Why does salt affect dissolved oxygen?**

**Experiment: 8 Estimation of concentration of sulphate/chloride ions in the given sample solution.**

**1. Relate solubility, conductance and precipitation.**

**2. Write the precipitation reaction of this titration.**

**3. Why does the conductance decrease in the precipitation titration?**

**4. Give the reason for decrease in conductance when  $\text{Na}_2\text{SO}_4$  is added to the solution containing  $\text{BaCl}_2$ .**

**5. Give the reason for sudden increase in conductance after the end point.**

**Experiment: 9 Determination of molecular weight of a polymer by viscometry method.**

**1. What are polymers and monomers?**

**2. Define viscosity.**

**3. What is intrinsic viscosity?**

**4. What is specific viscosity?**

**5. How is degree of polymerization found from molecular weight of a polymer?**

**6. What is name of the viscometer used in this experiment?**

**Experiment: 10 Synthesis of nanomaterials by simple precipitation method**

**1. What are nanomaterials?**

**2. How have nanomaterials been defined?**

**3. What are the key criteria for defining nanomaterials?**

**4. What are the four stages of nanoparticle synthesis?**

**5. What factors affect the synthesis of nanoparticles?**

**6. What is precipitation synthesis method?**