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**National Institute of Technology Jamshedpur**  
**Department of Chemistry**

**Lab manual**  
**Engineering Chemistry Lab CH1102 & CH1202**  
**Department of Chemistry**

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## **Experiment No.01**

### **Aim:**

To find the unknown concentration of a given glycerol solution with the help of known glycerol concentrated solutions by using Ostwald's viscometer.

### **Apparatus and reagents required:**

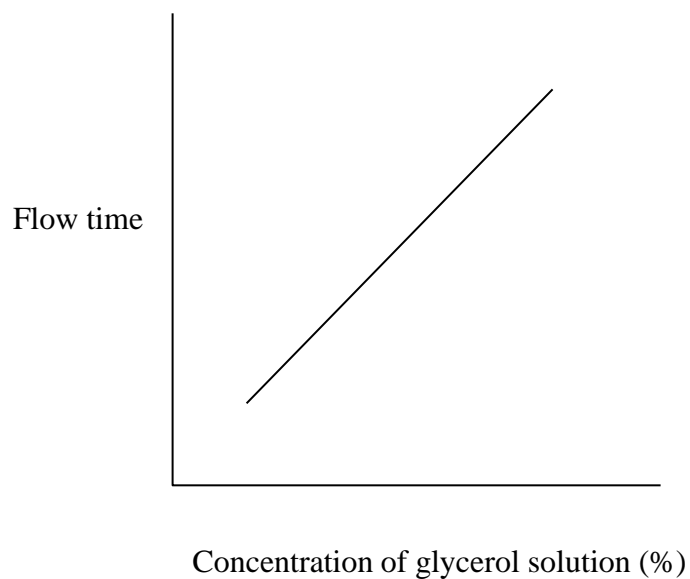
Glycerol solution, Ostwald's viscometer, beakers, pipette, stop watch, stand with clamp.

### **Procedure**

- Wash and clean and Ostwald viscometer and then clamp the right-hand limb of the viscometer vertically in a clamp stand
- Introduce 25 ml of distilled water in right side limb with the help of a pipette.
- Suck up the water in the left-hand limb with the help of rubber tubing till the level of the water reaches a little above the upper mark on this limb.
- Release the pressure on the rubber tubing and as soon as water level passes the upper mark start the stopwatch.
- As soon as the distilled water reaches the lower mark on this limb stop the stop watch.
- Record the time of flow of distilled water passes through the capillary tube from upper mark to lower mark on the left-hand limb of the viscometer. This process has to be done at least three times.
- Now repeat the same process with the supplied series of known concentrated glycerol solution at least three times to determine the average flow time  $t$  of every known concentrated glycerol solution passes through the capillary tube from upper mark to the lower mark on the left-hand limb of the viscometer.
- Now first wash the viscometer, pipette and breaker thoroughly with the distilled water then repeat the above-described process with the unknown concentrated glycerol solution.
- Now, plot the graph of glycerol solution with concentration against the flow time.
- Draw a straight line such that it covers maximum numbers of points.
- Now draw a straight line perpendicular to the X-axis from plotted line corresponding to point of time of flow of unknown glycerol solution to get the concentration of unknown glycerol solution.

## Observation Table

Concentration of glycerol solution (%)	Flow time of Glycerol solution			
	1 <sup>st</sup> time	2 <sup>nd</sup> time	3 <sup>rd</sup> time	Average
0				
1				
2				
4				
6				
8				
Unknown 1				
Unknown 2				



## Result

The unknown concentrations of glycerol solution are:

Unknown 1.....

Unknown 2.....

## Experiment No.02

### **Aim**

To determine the viscosity of given organic liquid at room temperature by using Ostwald's viscometer.

### **Apparatus and reagents required**

Reference liquid, experimental liquid, Ostwald's viscometer, beakers, pipette, stop watch, stand with clamp.

### **Theory**

In this method a fixed volume of liquid is allowed to flow from one limb to another limb of viscometer and time is counted. This method is based on Poiseuille equation.

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

Where,  $\eta$  = Coefficient of viscosity

$P$  = pressure,  $r$  = radius of capillary tube,  $l$  = length of capillary tube

$V$  = volume of liquid,  $t$  = time of flow

Moreover, liquid flowing under its own hydrostatic pressure  $P = h\rho g$  where,

$h$  = height of liquid column

$\rho$  = density of liquid

$g$  = acceleration due to gravity

$$\text{So that } \eta = \frac{\pi h \rho g r^4 t}{8 V l}$$

For equal volume ( $V$ ) of two liquids flowing from the same height( $h$ ) so that length( $l$ ) and radius ( $r$ ) are the same. If  $\eta_1$  and  $\eta_2$  are the coefficient of viscosity of two liquids and  $\rho_1$  and  $\rho_2$  are their densities and  $t_1$  and  $t_2$  are respective time of flow.

$$\frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2}$$

Thus measuring the time of flow of two liquids knowing their densities and knowing the coefficient of viscosity of one of the liquids, we can calculate the viscosity of the other liquid.

### **Procedure**

- Clean the viscometer and test bottles.
- Attach the piece of clean rubber tube to the upper end of the viscometer and clamp the viscometer.
- Introduce suitable volume of reference liquid to the one of the bulb of viscometer.
- With the help of rubber tube suck up the reference liquid in the capillary of the viscometer until the meniscus of the liquid is above the upper mark, allow the liquid to flow back by releasing the pressure, start the stop-watch when meniscus passes the upper mark and stop when it reaches the lower mark thus measure the time of flow from the

two mark.

- Repeat the counting process twice.
- Fill it with exactly equal volume of test liquid and determine flow time as done before.
- Record all the observation in the observation table.

### Observation Table

Liquid	Flow time	Mean	Density ( $g/cm^3$ )	Viscosity (mili poise)
Liquid 1	1.			
	2.			
	3.			
Liquid 2	1.			
	2.			
	3.			

### Calculations

We know the equation;-  $\frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2}$

$$\eta_2 = \frac{\eta_1 \rho_2 t_2}{\rho_1 t_1}$$

So that  $\eta_2$ =..... milli poise

### Result

The relative viscosity of given organic liquid is found to be ..... milli poise

### **Experiment No. 03**

#### **Aim**

To determine the surface tension of given organic liquid at room temperature by drop number method.

#### **Theory**

In this method a fixed volume of liquid is delivered as freely flowing drop from the end of a capillary tube and the number of drops form is counted. If the same volume of experimental liquid is allowed to flow through the capillary and the numbers of drops is delivered, then from original equation.

$$mg = 2\pi r\gamma$$

then,

$$\frac{\gamma_1}{\gamma_2} = \frac{m_1 g}{m_2 g} = \frac{v_1 \rho_1}{v_2 \rho_2} \quad \dots\dots\dots 1$$

Where  $\gamma_1$  and  $\gamma_2$  are the surface tension of the experimental liquid and reference liquid  $m_1$  and  $m_2$  mass of the drop.  $v_1$  and  $v_2$  are volume of single drop &  $\rho_1$  and  $\rho_2$  are their density. If  $v$  is the volume of each liquid delivered  $n_1$  and  $n_2$  are the number of drops counted for liquid 1 & 2 respectively.

$$\frac{\gamma_1}{\gamma_2} = \frac{(v/n_1)*\rho_1}{(v/n_2)*\rho_2} = \frac{n_2 \rho_1}{n_1 \rho_2} \quad \dots\dots\dots 2$$

Thus, determination of number of drops of a certain volume of a given liquid & a reference liquid & to calculate the surface tension of the organic liquid if the surface tension of the reference liquid is known.

#### **Apparatus and reagents required**

Stalagmometer, reference liquid, experimental liquid

#### **Procedure**

- Thoroughly clean stalagmometer & test bottles.
- Attach a piece of clean rubber tube to the upper end of the stalagmometer with the screw pinch cock.
- Fill the stalagmometer with distill water.
- Close the pinch cock and clamp the stalagmometer in a vertical hand.
- Open the pinch cock gently. So that liquid flows out slowly.

- Count the numbers of drops obtained when a fixed volume of liquid flows between the two marks i.e. upper mark and lower mark.
- Now thoroughly wash the stalagmometer & dry it.
- Fill it exactly equal volume of experimental liquid and determine the number of drops between the two marks is upper & lower marks.
- Repeat counting process thrice & record the observation in the table.
- Calculate the surface tension of a given organic liquid by using equation no. 2.

### Observation Table

Liquid	No. of drops in a given time		Density ( $\text{g/cm}^3$ )	Surface tension (dyne/cm)
	No. of time	Mean		
Liquid 1	1.			
	2.			
	3.			
Liquid 2	1.			
	2.			
	3.			

### Result

The surface tension of given organic liquid is. .... dyne/cm.

### Precautions

1. Before the use of the stalagmometer, it should be clean and dry.
2. No air bubbles should be formed while sucking to liquid into stalagmometer.



## **Experiment no.04**

### **Aim**

To determine iodometrically the strength in (g/L) of a given unknown Potassium dichromate ( $K_2Cr_2O_7$ ) solution using KI as a source of iodine and N/10 standard solution of Sodium Thiosulphate( $HypoNa_2S_2O_3$ ).

### **Theory**

When Potassium iodide is added to Potassium dichromate it becomes brick red, an equivalent amount of iodine is liberated.



The liberated iodine is titrated against standard sodium thiosulphate in presence of starch solution as indicator. The blue colour appears due to adsorption of iodine on starch. The iodine immediately reacts with sodium thiosulphate. When the end point reaches the blue colour of starch complex suddenly disappears and pure green solution appears  $Cr_2(SO_4)_3$



(Sodium tetrathionate) colourless

### **Apparatus and reagents required**

Burette, pipette, conical flask Sodium Thiosulphate solution, Potassium dichromate solution, Potassium iodide solution, Dilute Sulphuric acid, Starch solution.

### **Procedure**

- Wash the apparatus with distilled water.
- Rinse pipette with  $K_2Cr_2O_7$  and transfer 10 ml of it to conical flask.
- Add 10 ml of dilute sulphuric acid
- Add 10 ml of KI solution, brick red colour solution is obtained due to iodine
- Start adding sodium thiosulphate solution from burette, the brown orange colour fades on fading as the hypo soln. is added.
- When only a faint yellow colour remains add 2 drops of starch solution, the solution becomes dark blue. To avoid excess adsorption of iodine on starch, indicator is added when solution is faint yellow.
- Add more sodium thiosulphate from burette till appearance of light green/blue colour which indicate the endpoint.
- Repeat the titration until concordant reading is obtained.

**Observation table:**

Titration between  $\text{Na}_2\text{S}_2\text{O}_3$  solution & unknown  $\text{K}_2\text{Cr}_2\text{O}_7$  solution.

S.No.	Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution (mL)	Burette reading (mL)		Volume of $\text{Na}_2\text{S}_2\text{O}_3$ solution consumed(mL)	Concordant volume (mL)
		Initial	Final		
1					
2					
3					

**Calculations**

A. To calculate strength of unknown  $\text{K}_2\text{Cr}_2\text{O}_7$  solution.

$$N_1 V_1 = N_2 V_2$$

Strength of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution,  $N_1 =$

Strength of  $\text{Na}_2\text{S}_2\text{O}_3$  solution,  $N_2 =$

Volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution,  $V_1 =$

Volume of  $\text{Na}_2\text{S}_2\text{O}_3$  solution,  $V_2 =$

$$N_1 = N_2 V_2 / V_1$$

We have to determine  $N_1 =$

Strength in g/L of unknown  $\text{K}_2\text{Cr}_2\text{O}_7$  solution = Normality X Equivalent weight(49)

**Result**

The strength of given unknown potassium dichromate solution  $\text{K}_2\text{Cr}_2\text{O}_7 =$

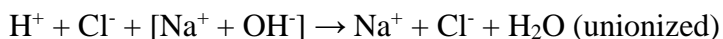
## **Experiment No.05**

### **Aim**

To find out the concentration of NaOH solution in a strong acid (HCl) and strong base (NaOH) titration Conductrometrically.

### **Theory**

Initially the conductance is mainly due to the presence of hydrogen & chloride ions. As alkali (NaOH) is added gradually, the hydrogen ions are replaced by slow moving sodium ions, as represented below:



Hence, on continued addition of NaOH, the conductance will go on decreasing until the acid has been completely neutralized. Any subsequent addition of alkali will result in introducing fast moving hydroxyl ions. The conductance, therefore, after reaching a certain minimum value, will begin to increase. On plotting the conductance against the volume of alkali added, the points will lie on two straight lines. The point of intersection of these lines gives the volume of alkali can be obtained as:

$$N_1 \times V_1 \text{ (acid)} = N_2 \times V_2 \text{ (base)}$$

### **Apparatus and reagents required**

Conductivity meter, magnetic stirrer, burette, pipette, beaker etc.

### **Procedure**

- Take 10 ml of given acid (HCl) solution in a beaker and dilute to 50 ml by double distilled water.
- In this beaker insert the conductivity cell, which is connected to the conductivity bridge.
- Now switch on for the direct conductance measurement.
- Titration is to be started as by gradual addition of alkali solution from the burette into the beaker and after each addition the conductivity is to be noted down.
- A plot is to be drawn between conductance vs volume of alkali added.
- A V shaped graph gives the end point for this strong acid vs strong base titration.
- By employing normality equation calculate the strength of alkali solution.

### **Observation table**

S.No.	Volume of alkali added (mL)	Conductance (mS)

**Result**

The normality of the given sodium hydroxide solution is .....

(This experiment can provide more accurate result (i.e., the end point value) as compared to the usual indicator type titration)

**Precautions:**

- 1) Solutions should be prepared in double distilled water.
- 2) Conductivity cell should be neat and clean by double distilled water.
- 3) Careful and small addition of NaOH is to be preferred.
- 4) Stirring should be proper. Prefer the use of magnetic stirrer.

## **Experiment No: 06**

### **Aim**

To estimate the strength (in g/L) of potassium permanganate solution by titrating against standard ferrous ammonium sulphate solution.

### **Apparatus and reagents required**

Burette (25ml), Pipette (10ml), conical flask (100ml), ferrous ammonium sulphate (FAS), potassiumpermanganate, dilute sulphuric acid

### **Theory**

Ferrous ammonium sulphate or Mohr's Salt  $\text{Fe}(\text{SO}_4)_2(\text{NH}_4)_2 \cdot 6\text{H}_2\text{O}$  is a stable double salt of ammonium sulphate and ferrous sulphate. Its active constituent is ferrous sulphate which is oxidized to ferric sulphate by  $\text{KMnO}_4$  in presence of dil.  $\text{H}_2\text{SO}_4$  at room temperature.

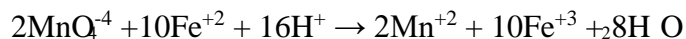
Reduction:  $2\text{KMnO}_4 + 3\text{H}_2\text{SO}_4 \rightarrow \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 + 3\text{H}_2\text{O} + 5[\text{O}]$

Oxidation:  $2\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O} + \text{H}_2\text{SO}_4 + [\text{O}] \rightarrow \text{Fe}_2(\text{SO}_4)_3 + 2(\text{NH}_4)_2\text{SO}_4 + 13\text{H}_2\text{O}$

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$$2\text{KMnO}_4 + 10[\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}] + 8\text{H}_2\text{SO}_4 \rightarrow 5\text{Fe}_2(\text{SO}_4)_3 + 10(\text{NH}_4)_2\text{SO}_4 + \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 + 8\text{H}_2\text{O}$$

The ionic reaction may be written as:



At the end point permanent light pink is developed in the solution

### **Procedure**

- Wash the apparatus with distilled water.
- Rinse the burette with  $\text{KMnO}_4$  then fill the burette with  $\text{KMnO}_4$  solution. (Note down the burette's initial readings.)
- Rinse the pipette with FAS solution, pipette out 10 mL solution and transfer it to conical flask.
- Add 10 mL of dilute  $\text{H}_2\text{SO}_4$  into the FAS solution in conical flask.
- Titrate FAS solution present in conical flask with the  $\text{KMnO}_4$  solution filled in burette, continue addition of  $\text{KMnO}_4$  solution drop by drop till the color of the solution just change to permanent pink color indicates the end point.
- Note down the volume of  $\text{KMnO}_4$  used (burette reading).
- Repeat the titration to get concordant reading.

**Observation table**

Titration between standard FAS and  $\text{KMnO}_4$  solution.

S.No.	Volume of FAS solution taken (mL) $V_1$	Burette reading(mL)		Volume $\text{KMnO}_4$ solution consumed (mL)	Concordant volume (mL) $V_2$
		Initial	Final		
1					
2					
3.3					

**Calculations**

To calculate the strength of  $\text{KMnO}_4$  solution

$$N_1 V_1 = N_2 V_2$$

Strength of FAS solution taken,  $N_1 = \text{_____}$  N

Volume of FAS solution taken,  $V_1 = \text{_____}$  mL

Strength of  $\text{KMnO}_4$  solution taken,  $N_2 = \text{_____}$  N

Volume of  $\text{KMnO}_4$  solution,  $V_2 = \text{_____}$  mL (from table)

Strength (g/L) of unknown  $\text{KMnO}_4$  solution = Normality ( $N_2$ )  $\times$  equivalent weight (31.6)

**Result:**

The strength of a given unknown  $\text{KMnO}_4$  solution = ... g/L

## Experiment No. 07

### Aim

To estimate the strength in g/L of a given unknown solution of potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) by titrating it with known Ferrous ammonium sulphate (FAS) solution using Diphenyl amine as the indicator.

### Apparatus and reagents required

Burette, Pipette, conical flask, volumetric flask, Ferrous ammonium sulphate, Diphenylamine indicator, dilute sulphuric acid.

### Theory

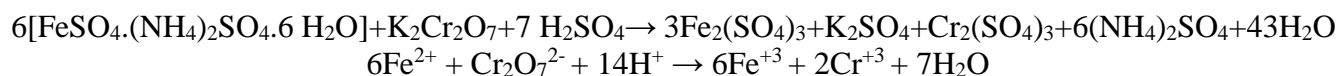
The oxidizing agent  $\text{K}_2\text{Cr}_2\text{O}_7$  in presence of dil. sulphuric acid liberates three atoms of oxygen.



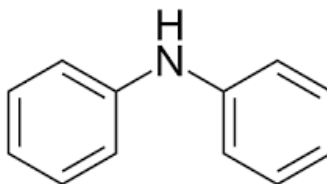
The liberated  $[\text{O}]$  oxidizes ferrous ion ( $\text{Fe}^{+2}$ ) to ferric ion ( $\text{Fe}^{+3}$ ).



The complete reaction is,



The colour exhibited by the diphenylamine changes from **Greenish to Purple** at the end point.



(Diphenylamine)

### Procedure

- Wash the apparatus with distilled water.
- Rinse the Burette with  $\text{K}_2\text{Cr}_2\text{O}_7$  solution and fill the burette with  $\text{K}_2\text{Cr}_2\text{O}_7$  solution.
- Rinse the Pipette with FAS solution. With the help of the pipette measure 10ml of FAS solution transfer it to the conical flask.
- Add 10ml. of dil.  $\text{H}_2\text{SO}_4$  into the FAS solution.
- Add 2-3 drops of indicator solution.
- Add  $\text{K}_2\text{Cr}_2\text{O}_7$  solution from burette into FAS solution with constant shaking &

observe the colour change. **The appearance of purple colour indicates the end point.** Note down the Burette readings.

- Repeat the Titration till concordant reading obtained.

### Observation table

Titration between FAS solution & unknown  $K_2Cr_2O_7$  solution.

S.No.	Volume of FAS solution (mL) $V_1$	Burette reading (mL)		Volume of $K_2Cr_2O_7$ solution consumed (mL)	Concordant volume (mL) $V_2$
		Initial	Final		
1					
2					
3					

### Calculation

To calculate the strength of unknown  $K_2Cr_2O_7$  solution.

$$N_1 V_1 = N_2 V_2$$

Strength of FAS solution,  $N_1 =$

Volume of FAS solution,  $V_1 =$

Strength of  $K_2Cr_2O_7$  solution,  $N_2 =$

Volume of  $K_2Cr_2O_7$  solution,  $V_2 =$

We have to determine  $N_2 =$

Strength in g/L of unknown  $K_2Cr_2O_7 =$  Normality of  $K_2Cr_2O_7$  solution  $\times$  Equivalent weight of  $K_2Cr_2O_7$

### Result

The strength of a given unknown  $K_2Cr_2O_7$  solution = .....



## Experiment No. 08

### Aim

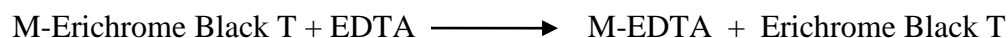
To determine the total hardness of the laboratory tap water sample by EDTA method.

### Apparatus and reagents required

Beaker, conical flask, Test tube, Burette, Pipette & Stand with Clamp.

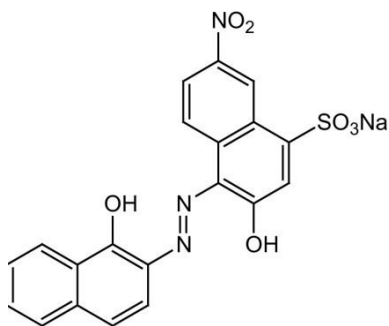
### Theory

The method is based upon the fact that the Ethylene diamine tetra acetic acid(EDTA) forms remarkably stable complex compounds with Di & Trivalent metals imparting hardness at  $P^H = 10$ . The end point is indicated by Erichrome Black-T indicator. This changes its colour from wine red to pure blue at the end point. The chemical reactions can be represented by the following equations:

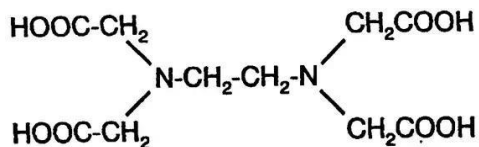


### Procedure

- Pipette out 50 mL laboratory tap water sample into a conical flask .
- Add 10 mL of buffer solution having  $p^H = 10$  (70g  $\text{NH}_4\text{Cl}$  dissolved in 570ml conc.  $\text{NH}_3$  and diluted to one ltr. With distilled water).
- 2 drops of indicator solution (0.5g Erichrome Black T in 100 ml. Methyl alcohol).
- Titrate with 0.01M EDTA solution (Dissolve 3.7275g of Disodium salt of EDTA in distilled water and then dilute the solution to one ltr. with distilled water) taken in the Burette.
- the colour changes from wine red to pure Blue.
- Note down the volume of EDTA used (burette reading).



( ERICHROME BLACK T)



(EDTA)

**Observation table:**

**Table:** Titration between standard **Tap water** and EDTA solution.

S.No.	Volume of taken solution (mL)	Burette readin		Volume EDTA solution consumed (mL) (V <sub>2</sub> )	Average volume (mL) (X)
		g(mL)			
		Initial	Final		
1.	50				
2.	50				
3.	50				

**Calculation:**

1mL 0.01M EDTA = 1mg CaCO<sub>3</sub>

X mL of 0.01M EDTA = X mg CaCO<sub>3</sub>

50 mL water sample contains hardness impurities equivalent to X CaCO<sub>3</sub>

100 mL of 0.01M EDTA = ( $\frac{X}{50} * 1000$ ) ppm

= ..... ppm

**Result:**

The total hardness of the given tap water solution = ..... ppm.

## **Experiment No.09**

### **Aim**

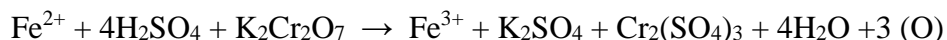
To find the amount of iron present in the given solution of ferrous ammonium sulphate by potentiometric titration with standard potassium dichromate solution.

### **Apparatus and reagents required**

Pipette, beaker, potentiometer, saturated calomel electrode, ferrous ammonium sulphate, dil. Sulphuric acid,  $\text{K}_2\text{Cr}_2\text{O}_7$  (0.1N).

### **Theory**

Potentiometric titration is the titration in which potentiometric measurements are carried out in order to fix the equivalence point. In this method, by using the standard saturated calomel electrode the change in EMF of the cell is monitored. The cell EMF is directly depends on the ratio of the ion in two different oxidation state (i.e. before equivalence point  $\text{Fe}^{2+}/\text{Fe}^{3+}$  or after equivalence point  $\text{Cr}^{3+}/\text{Cr}_2\text{O}_7^{2-}$ ). In a potentiometric titration, the change in cell e.m.f. occurs most rapidly in the neighbourhood of the equivalence point. The  $\text{Fe}^{2+} - \text{K}_2\text{Cr}_2\text{O}_7$  redox system is represented as



### **Procedure:**

- Take 25 mL of ferrous ammonium sulphate solution into the beaker.
- Add 20 mL of dil. Sulphuric acid.
- Immerse the electrode assembly into the solution in the beaker.
- Connect the electrodes to a potentiometer, and measure the potential.
- Fill the burette with potassium dichromate solution.
- Add 1 mL of potassium dichromate to the beaker. Stir the solution carefully and measure the potential after 15 seconds.
- Continue step (vi), till the potential show a tendency to increase rapidly.
- Now add  $\text{K}_2\text{Cr}_2\text{O}_7$  in increments of 0.1 mL and measure the potential after each addition.
- Plot  $\Delta E/\Delta V$  against volume and determined the end point.
- Find the normality of the ferrous solution and determine the amount of Fe.

**Observation table**

Ferrous ammonium sulphate solution taken in beaker = 50 mL

Normality of  $K_2Cr_2O_7$  solution = 0.1N

S. No.	Volume of $K_2Cr_2O_7$ added (mL)	Potential (mV)

Let the volume of  $K_2Cr_2O_7$  added at equivalence point =  $V_1$  mL

**Calculation:**

$N_1 V_1$  of  $K_2Cr_2O_7$  solution =  $N_2 V_2$  of ferrous ammonium sulphate solution

$$0.1 * V_1 = N_2 * 50$$

$$N_2 =$$

Strength of Iron in the given solution =  $N_2 * 56.5$  = .....g/L

**Result:**

The amount of Iron in the given solution was found to be .....g/L

**Precautions:**

- Make proper concentration
- Handle electrodes very carefully.
- Before the potential measurement, stir the solution carefully.

## **Experiment No. 10**

### **Aim**

To determine the concentration of unknown  $\text{KMnO}_4$  solution spectrophotometrically.

### **Apparatus and reagents required**

Calorimeter, cuvette, test tubes, beakers, standard solution of  $\text{KMnO}_4$  with 0.5, 1, 1.5, 2, 2.5 and 3% concentration respectively,  $\text{KMnO}_4$  solution of unknown concentration.

### **Theory**

Colorimetry - The variation of the colour of the system with concentration is the basis of colorimetry. Colorimetric analysis is specially useful for systems in which substances or their solution are coloured. When a substance is colourless, then a suitable complexing agent is added to the solution so that a coloured complex is obtained. The coloured complex absorbs light in the visible region. For instance, for the estimation of cuprous ions, the complexing agent ammonium hydroxide, is used to get blue coloured solution.

Colorimeters are provided with arrangements to select appropriate wavelength of light with the help of filter and grating. A light of proper wavelength is allowed to pass through a coloured test solution. The light transmitted from this solution is made to pass through photocell and proportional to the amount of light transmitted by solution, a current is generated in the photocell. A meter is calibrated to show the fraction of light absorbed which is proportional to the concentration of the coloured substance in the test solution. From the measurements, concentration of coloured solution can be obtained by using Lambert-Beer law.

$$A = \epsilon c l$$

A- Absorbance;  $\epsilon$  - absorption coefficient; c- concentration; l- path length.

### **Procedure**

- Fix the wavelength of the spectrophotometer at  $\lambda_{\text{max}}$
- Make the standard solution of  $\text{KMnO}_4$  with 0.5%, 1%, 1.5%, 2%, 2.5% and 3% concentration respectively. (20 mL each)
- Measure the absorbance values of all the above solution at
- Plot a graph between Absorbance (along Y axis) vs concentration (along X axis). Fit all

the observation points on a straight line. Beer law is verified, if a straight line is obtained.

- Now find the absorbance value of unknown solution also at  $\lambda_{\max}$
- From the straight line of the graph of absorbance vs concentration, find out the concentration of the unknown solution.

### Observation

**Table 1.** Determination of  $\lambda_{\max}$

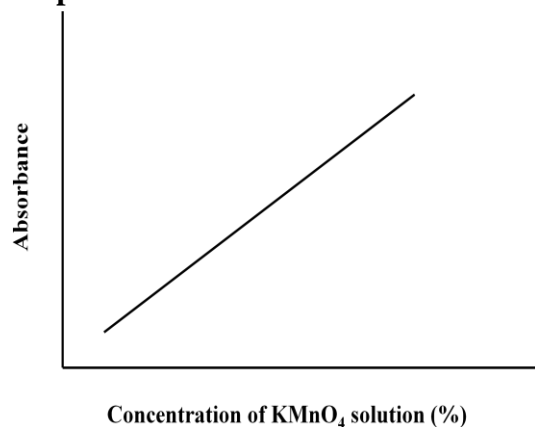
Wave length (nm)							
absorbance							

**Table 2.** Determination of concentration of  $\text{KMnO}_4$

Conc. of $\text{KMnO}_4$ (%)	0.2	0.5	1.0	1.5	2.0	2.5	3.0	unknown
Absorbance								Find it from graph

### Calculation:

#### Graph



### Results:

$\lambda_{\max}$  for  $\text{KMnO}_4$  = .....nm

The concentration of unknown  $\text{KMnO}_4$  solution = ..... %

### Precautions:

- Cuvette is fragile so use it very carefully.
- Properly clean the cuvette before and after use, and wipe it very gently with tissue paper.
- Use dilute solution of known concentrations for the preparation of standard calibration curve.

## **Experiment No. 11**

### **Aim**

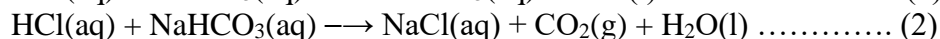
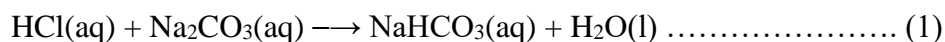
To determine the mixture of NaOH (aq) and Na<sub>2</sub>CO<sub>3</sub> (aq) by double indicator method.

### **Apparatus and reagents required**

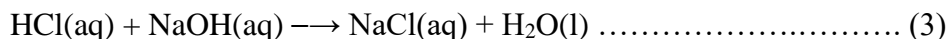
Phenolphthalein indicator, methyl orange indicator, Standard (N/10) HCl(aq), a mixture of NaOH(aq) and Na<sub>2</sub>CO<sub>3</sub>(aq), conical flask, burette and pipette, stand and burette clamp.

### **Theory**

Consider a mixture of NaOH(aq) and Na<sub>2</sub>CO<sub>3</sub>(aq). Reaction between HCl(aq) and Na<sub>2</sub>CO<sub>3</sub>(aq) takes place in two stages:-



While that between HCl(aq) and NaOH(aq) completes in only one step:-



Solution mixture of reaction (1) at the equivalence point is alkaline, that of reaction (2) is acidic and that of reaction (3) is neutral. Thus the whole titration should have three breaks in the pH curve, corresponding to the above three stages. Reactions (1) and (3) can be indicated by phenolphthalein and that of reaction (2) can be indicated by methyl orange.

### **Procedure:**

- Wash the apparatus with distilled water.
- Rinse pipette with given mixture solution and transfer 10 ml of it to conical flask.
- Rinse the burette with the provided HCl fill up burette up to the mark.
- Add 2 drop phenolphthalein indicator into the mixture titrate it until the color changes from pink to colorless.
- Note down the Burette readings.
- Then add 2 drops of methyl orange indicator into the colorless solution
- Titrate that solution against HCl until the color of solution changes from yellow to red-orange. Note the burette readings.
- Repeat the Titration 3 times and note the concordant/average value.

**Observation table:****Table 1:** Titration between standard HCl solution and NaOH in the given mixture using phenolphthalein indicator

S.No.	Volume of mixture solution (mL) $V_1$	Burette reading (mL)		Volume HCl solution consumed (mL)	Concordant volume (mL) $V_2$
		Initial	Final		
1.					
2.					
3.					

**Table 2:** Titration between standard HCl solution and  $\text{Na}_2\text{CO}_3$  in the given mixture using methyl orange indicator

S.No.	Volume of taken solution (mL) $V_1$	Burette reading (mL)		Volume HCl solution consumed (mL)	Concordant volume (mL) $V_3$
		Initial	Final		
1.					
2.					
3.					

**Calculation**

To calculate the strength of NaOH in mixture solution.

$$N_1 V_1 = N_2 V_2$$

Strength of NaOH in the mixture,  $N_{1a} =$

Volume of mixture solution taken,  $V_1 =$

Strength of HCl solution,  $N_2 =$

Volume of HCl solution,  $V_2 =$

(Put the data and calculate the normality of NaOH solution)

We have to determine  $N_{1a}$

To calculate the strength of  $\text{Na}_2\text{CO}_3$  in mixture solution.

$$N_{1b} V_1 = N_2 V_3$$

Strength of  $\text{Na}_2\text{CO}_3$  in the mixture,  $N_{1b} =$

Volume of mixture solution taken,  $V_1 =$



Strength of HCl solution,  $N_2 =$

Volume of HCl solution,  $V_3 =$

(Put the data and calculate the normality of  $\text{Na}_2\text{CO}_3$  solution)

We have to determine  $N_{1b}$

Strength of NaOH in the given mixture (g/L) = Normality of NaOH X equivalent of NaOH

Strength of  $\text{Na}_2\text{CO}_3$  in the given mixture (g/L) = Normality of  $\text{Na}_2\text{CO}_3$  X equivalent of NaOH

### **Result**

The strength of NaOH in the given mixture =

The strength of  $\text{Na}_2\text{CO}_3$  in the given mixture =