



## **AURORA'S ENGINEERING COLLEGE**

Bhuvanagiri, Nalgonda Dist.-508116.



## **ENGINEERING CHEMISTRY**

### **LAB MANUAL**

### **B.Tech. 1<sup>st</sup> Year**

*(As per 2013-14 Academic Regulation)*

*Department of*

**HUMANITIES & SCIENCES**

**COMMON TO**

**CSE, ECE, EEE, IT, MECH, CIVIL**

### **CODE OF CONDUCT**

1. Students should report to the concerned lab as per the time-table schedule.
2. Students who turn up late to the labs will in no case be permitted to perform the experiment scheduled for the day.
3. After completion of the experiment, certification of the concerned staff in-charge in the observation book is necessary.
4. Students should bring a note book of about 100 pages and should enter the readings / observations into the note book while performing the experiment.
5. The record of observations along with the detailed experimental procedure of the experiment performed in the immediate last session should be submitted and certified by the staff member in-charge.
6. The group-wise division made in the beginning should be adhered to, and no mix up of students among different groups will be permitted later.
7. The components required pertaining to the experiment should be collected from the lab assistants.
8. When the experiment is completed, students should clean the apparatus, carefully dispose the leftover chemicals and disconnect the setup made by them, and should return all the components/instruments taken for the purpose.
9. Any damage to apparatus that occurs during the experimentation, should be brought to the notice of lab in-charge, consequently, the cost of the repair or new apparatus should be brought by the students.
10. Students should be present in the labs for the total scheduled duration.
11. Students are required to prepare thoroughly to perform the experiment before coming to Laboratory.
12. Procedure sheets/data sheets provided to the students' groups should be maintained neatly and to be returned after the experiment.

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**GENERAL PRECAUTIONS TO BE FOLLOWED IN THE  
LABORATORY**

1. Never work in the laboratory unless a demonstrator or teaching assistant is present. A minor mishap that may be of little consequence if someone is at hand to help you, may be serious if you are alone.
2. Do not throw waste such as match sticks, filter papers etc., into the sink. They must be thrown into the waste jars.
3. Keep the water and gas taps closed except when these utilities are needed.
4. Never taste any chemical unless instructed to do so and do not allow chemicals to come in contact with your skin.
5. While working with obnoxious gases, conduct the experiment in a fume hood.
6. Sodium metal should be used with extra care and scraps of sodium should never be thrown into the sink or waste jar and may be destroyed by treating with alcohol. Sodium should be kept covered with kerosene or toluene.
7. Keep all the doors and windows open while working in the laboratory.
8. Bromine, acetyl chloride, benzoyl chloride, phosphorus trichloride etc. are obnoxious materials and they should be handled carefully in the hood.
9. Do not add porcelain pieces or any solids to a boiling liquid; add only after allowing the liquid to cool a little.
10. You should know about the hazards and properties of every chemical which you are going to use for the experiment. Many chemicals encountered in analysis are poisonous and must be carefully handled.
11. Sulphuric acid must be diluted only when it is cold. This should be done by adding it slowly to cold water with stirring, and not vice versa.
12. Apparatus for which no further immediate use is envisaged should be returned to the cupboard, but if it will be needed at a later stage, it may be placed at the back of the work bench.
13. Reagent bottles must never be allowed to accumulate on the work bench. They should be placed back in the shelves as and when used.
14. Containers in which reaction to be performed a little later should be labeled.
15. Working space must be neat and clean and any spillages should be cleaned immediately.

**PRECAUTIONS TO BE FOLLOWED IN THE ORGANIC LAB**

1. Those reactions which involve heating should be carried out by heating on water bath. No direct flame must be used because the chemicals are highly flammable.
2. The organic solvents / substances must not be inhaled as they are toxic to health.
3. Fuming cupboard must be used for handling the reactions involving, corrosive chemicals like acids, volatile compounds having foul odour, and organic chemicals.
4. While performing the experiments safety goggles, gloves, apron and shoes must be worn.

*Experiment No.1***CONDUCTOMETRIC TITRATION OF  
STRONG ACID VS. STRONG BASE****AIM**

To determine the strength of HCl solution by titrating against standard sodium hydroxide solution conductometrically.

**APPARATUS**

1. Conductivity meter
2. Conductivity cell with known cell constant
3. Burette
4. Beaker
5. Glass rod
6. Pipette

**CHEMICALS**

1. HCl solution
2. NaOH solution
3. Standard oxalic acid solution
4. Phenolphthalein indicator

**PRINCIPLE**

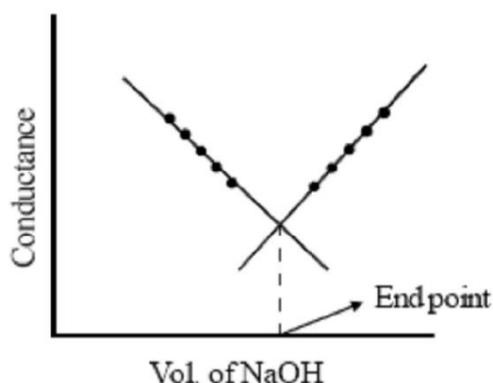
Conductometric titration is the volumetric analysis, based upon the measurement of the conductance during the titration. The number of ions, charge on the free ion and mobility of the ions effect the conductance of a solution. When one electrolyte is added to another electrolyte, the change in number of free ions causes a change in the conductance.

**For example NaOH Vs HCl:** Before adding NaOH solution to HCl, HCl has high conductivity due to highly mobile H<sup>+</sup> ions. When NaOH is added, the conductivity of HCl decreases due to neutralization of highly mobile H<sup>+</sup> ions by OH<sup>-</sup> ions of NaOH.



The conductivity of solution decreases until an equivalent point is reached. Further addition of NaOH increases the conductivity of solution due to increase in the highly mobile OH<sup>-</sup> ions.

The graph plotted between the conductance of solution against the volume of NaOH solution added will give the end point of the titration. The graph is as shown:



## PROCEDURE

### Standardisation of NaOH solution

Fill the burette with NaOH solution. Pipette out 20 ml of Oxalic acid into a conical flask and add 2-3 drops of phenolphthalein indicator and titrate the colourless solution against NaOH till pale pink colour is obtained as end point. Repeat the titration to get concurrent values.

| S.No. | Vol. of Oxalic acid | Burette reading |       | Vol. of NaOH |
|-------|---------------------|-----------------|-------|--------------|
|       |                     | Initial         | Final |              |
|       |                     |                 |       |              |

$$N_1V_1 = N_2V_2$$

(Oxalic acid) = (NaOH)

### Measurement of Conductivity

Pipette out 20 ml of HCl solution into a 100 ml beaker and dip the conductivity cell in it. Connect the conductivity cell to the conductometer. Set the function switch to check the position. Display must read 1000, if not set to 1000. Adjust the function switch to cell constant and set the cell constant value. Adjust the temperature control to the actual temperature of the solution. Set the function switch to "conductivity" and read the value. This is exact conductivity of solution. Note down this value in the table. Now add 1.0 ml of NaOH solution to HCl in the beaker, stir the solution and record the conductance. Repeat this step of adding 1.0 ml sample of NaOH and every time record the conductance. Take about 30 readings. By plotting the graph we can get the amount of NaOH required for the neutralization of HCl taken in the beaker (i.e., the end point).

**SAMPLE TABLE**

| S.No. | Vol. of NaOH | Conductance |
|-------|--------------|-------------|
| 1.    |              |             |
| 2.    |              |             |
| 3.    |              |             |
| 4.    |              |             |
| 5.    |              |             |
| 6.    |              |             |
| 7.    |              |             |
| 8.    |              |             |
| 9.    |              |             |
| 10.   |              |             |

$$N_2V_2 = N_3V_3$$
$$(NaOH) = (HCl)$$

$$\begin{aligned} \text{Strength of HCl} &= N_3 \times \text{Equivalent weight of HCl} \\ &= N_3 \times 36.5 \text{ g/l} \end{aligned}$$

**RESULT**

Strength of HCl \_\_\_\_\_ g/L

**REFERENCE BOOKS**

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

*Experiment No.2*

**CONDUCTOMETRIC TITRATION OF MIXTURE OF  
STRONG ACIDS VS. STRONG BASE**

**AIM**

To investigate the conductometric titration of a mixture of strong and weak acid against strong base.

**APPARATUS**

Conductivity bridge,

conductivity cell,

magnetic stirrer,

burette,

measuring cylinder,

beaker etc.

**CHEMICALS**

0.01N (approximate) HCl,

0.01N(approximate) CH<sub>3</sub>COOH,

0.05 N (exact) NaOH solution,

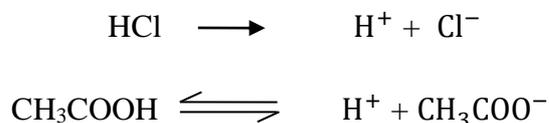
conductivity water.

**PRINCIPLE**

Electrolytic conductivity is a measure of the ability of a solution to carry electric current. Solutions of electrolytes conduct an electric current by the migration of ions under the influence of an electric field. During ionic reactions, the conductance may either increase or decrease depending upon the nature of electrolyte involved. The substitution of ions of one conductivity by the ions of other conductivity is the basis of conductometric titrations.

Let us consider the titration of a mixture of strong and weak acid (HCl + CH<sub>3</sub>COOH) with strong base (NaOH).

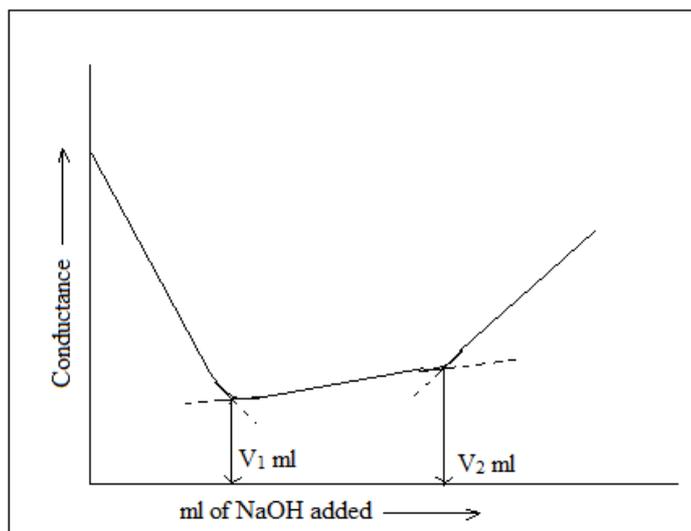
Hydrochloric acid is a strong acid which in an aqueous solution dissociates completely while acetic acid being weak acid dissociate to less extent in an aqueous solution. When a mixture of hydrochloric acid and acetic acid is titrated with sodium hydroxide, the HCl will be neutralizing first. The partial neutralization can be explained as follows:



The common ion effect of  $\text{H}^+$  ions suppresses the dissociation of acetic acid i.e. the equilibrium lies to the left side, keeping it in the undissociated state. This will continue till  $\text{H}^+$  of HCl are completely neutralized. Then neutralization of acetic acid starts with NaOH. After complete neutralization of mixture of strong and weak acid, the added NaOH will remain unreacted in the flask and the concentration of the  $\text{OH}^-$  ions increases with addition of NaOH. This trend of the process is utilized to know the mixture of HCl and  $\text{CH}_3\text{COOH}$  by conductometric titration.

In the initial stage of titration, the mixture of acid solution has high conductance due to presence of highly mobile  $\text{H}^+$  ions of HCl. As titration proceeds, the conductance falls due to replacement of highly mobile  $\text{H}^+$  ions by less mobile  $\text{Na}^+$  ions, until entire HCl is neutralized, then conductance rises as the acetic acid is neutralized to form its salt (as the acetate ions have conducting power), then finally conductance rises more steeply after the equivalence point as the excess NaOH is added. The excess NaOH contains unreacted highly mobile  $\text{OH}^-$  ions, which causes steep increase in conductance of the solution.

The plot of conductance vs volume of NaOH will have two breaks because of three intersecting straight lines. First one corresponds to the equivalence point of strong acid (HCl) component and second to that of another weak acid ( $\text{CH}_3\text{COOH}$ ). Component. as shown the figure given below:



### PROCEDURE

Take 0.1N NaOH solution and 0.01N  $\text{CH}_3\text{COOH}$  and 0.01N HCl solution in a beaker. Fill the micro-burette with 0.1N NaOH solution. Place the conductivity cell in distilled water and adjust the display to pH 10 with calibration nob. Take a beaker and add 20ml of  $\text{CH}_3\text{COOH}$  and 20ml of HCl. Titrate the mixture against 0.1N NaOH solution. Now measure initial conductance of solution. Now measure initial conductance of solution. Then 0.1 ml of NaOH is added every time from burette into the solution and stirred well each time. Note down the conductance values till the conductance values decrease and increase considerably.

### TABLE

| S.NO | Volume of NaOH added(ml) | Conductance(mhos) |
|------|--------------------------|-------------------|
| 1    |                          |                   |
| 2    |                          |                   |
| 3    |                          |                   |
| 4    |                          |                   |
| 5    |                          |                   |
| 6    |                          |                   |

**RESULT**

Volume of NaOH used for neutralization of strong acid from graph = -----ml

Volume of NaOH used for neutralization of weak acid from graph = -----ml

**REFERENCE BOOKS**

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

*Experiment No. 3***DETERMINATION OF DISSOCIATION CONSTANT (K<sub>a</sub>) OF A WEAK ACID BY pH METRIC METHOD****AIM**

To determine the dissociation constant of given weak acid by pH metric method.

**APPARATUS**

1. pH meter
2. Beaker
3. Burette

**CHEMICALS**

1. Standard NaOH solution
2. Weak acid (acetic acid) solution

**PRINCIPLE**

On addition of alkali to an acid, the concentration of H<sup>+</sup> ions will be decreased and hence pH will increase. In the beginning the pH increases slowly but at the neutralization point it changes rapidly due to the rapid increase in the fraction of the H<sup>+</sup> ions removed by the addition of constant volume of alkali. After neutralization point the pH increases slowly.

According to Henderson's equation  $\text{pH} = \text{pK}_a + \log \frac{[\text{Salt}]}{[\text{Acid}]}$

At half neutralization point  $[\text{Salt}] = [\text{Acid}]$  and hence pH at half neutralization point is equal to pK<sub>a</sub> of the given weak acid.

**PROCEDURE**

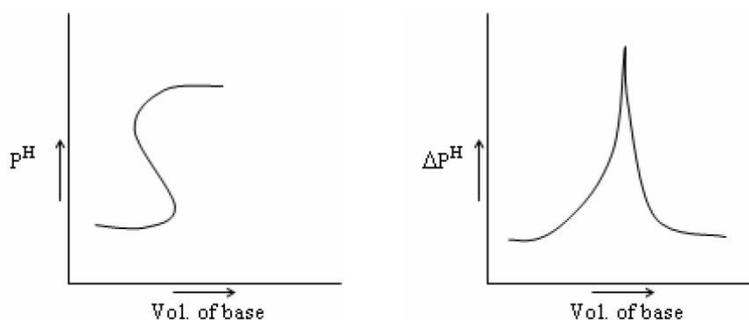
Calibrate the pH meter with the solution of the known pH. Wash the electrode with distilled water. Pipette out 20 ml of acetic acid in to 100 ml beaker and dip the electrode in it. Measure the pH of solution. Then add 0.5 ml of standard NaOH solution from the burette and measure the pH. Continue the process of adding 0.5 ml samples of NaOH and measuring pH after each addition, till a large increase in pH is observed. Take several readings well above the neutralization point. Plot pH or ΔpH Vs Volume of alkali added. From the graph neutralization point is determined.

**OBSERVATION AND CALCULATIONS**

| S.No. | Volume of NaOH added (ml) | pH value | $\Delta$ pH |
|-------|---------------------------|----------|-------------|
|       |                           |          |             |

**GRAPH**

Plot a graph by taking pH or  $\Delta$ pH on Y-axis and volume of base on X-axis.

**RESULTS**

The concentration of the given acetic acid solution is found to be \_\_\_\_\_

At half neutralization point  $\text{pH} = \text{pKa}$

Dissociation constant  $K_a = 10^{-\text{pKa}}$  or  $10^{-\text{pH}}$

The dissociation constant ( $K_a$ ) of acetic acid is found out to be \_\_\_\_\_

**REFERENCE BOOKS**

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

## Experiment No. 4

## ESTIMATION OF FERROUS IRON IN CEMENT BY COLORIMETRIC METHOD

### AIM

To estimate the amount of ferrous iron present in the given sample of cement by colorimetry, using ammonium thiocyanate as the reagent.

### APPARATUS

1. Standard flask
2. Burette
3. Pipette
4. Colorimeter
5. Beaker

### CHEMICALS

1. Ferrous Ammonium sulphate
2. H<sub>2</sub>SO<sub>4</sub>
3. Dil. KMnO<sub>4</sub>
4. HNO<sub>3</sub>
5. Conc<sup>n</sup> HCl
6. 40% ammonium thiocyanate

### PRINCIPLE

Generally, colorimeter works on Lambert Beer's law, the mathematical expression of which is given by:

$$\log \frac{I_0}{I_t} = ECl \quad \text{and} \quad \frac{I_t}{I_0} = T$$

$I_0$  = Incident light;  $I_t$  = transmitted light

$\log I_0/I_t$  = Absorbance (A)

C = Concentration

E = molar absorption coefficient

l = thickness of the absorbing medium

T = transmittance

(ECl) = Optical Density

So Absorbance (A) = Optical Density (ECl)

Therefore:

$$A = ECl = \log \frac{I_0}{I_t} = \log \frac{1}{T} = -\log T$$

Ammonium thiocyanate yields a blood red colour with ferric iron and the colour produced is stable in nitric acid medium. Its optical density is measured in a photo colorimeter and the concentration of ferric iron is found from the standard calibration curve.

## PROCEDURE

### To obtain standard calibration curve

Dissolve the given ferrous ammonium sulphate (0.7022 gms) in 100 ml of water and add 5 ml of 1 : 5 H<sub>2</sub>SO<sub>4</sub> followed by dil. KMnO<sub>4</sub> solution through burette until light-pink colour appears. Dilute the solution to 1 L such that 1 ml of solution contains 0.1 mg of Fe<sup>+3</sup>. From the above solution, take separately 1,2,3,4,5 ml into separate 100 ml standard volumetric flasks. Add 1 ml of nitric acid and 5 ml of 40% ammonium thiocyanate solution to all the above samples to get blood-red colour and make up the solutions to the mark by adding distilled water. Now measure the optical densities of all the solutions using photocolorimeter and plot a graph by taking amount of ferrous iron on x-axis and optical density on the y-axis. The curve obtained is called standard calibration curve.

### Dissolution of sample

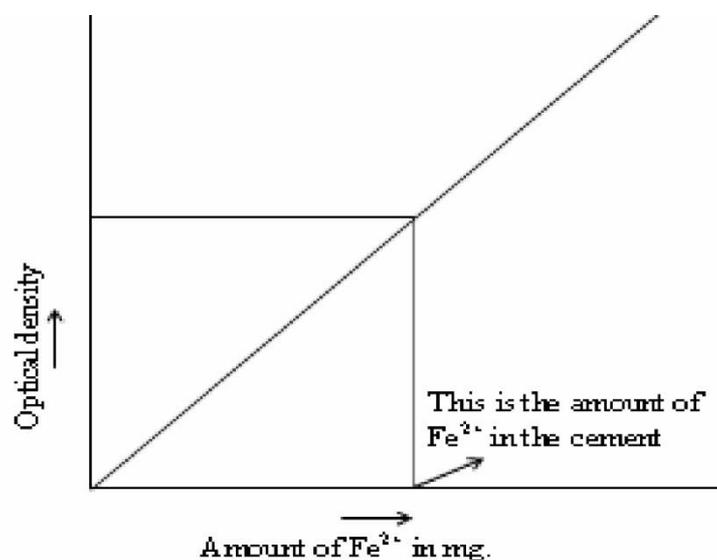
Weight out 0.1 gm of the cement sample into a beaker. Add 5 ml of water and stir with a stirrer. Add few drops of conc<sup>n</sup> HCl through the walls of the beaker and stir again. Heat the mixture until the moisture is evaporated. Then add 20 ml of distilled water and dissolve the content and make up the solution with distilled water to 100 ml. Shake well for uniform concentration.

### Development of Colour

Pipette out 10 ml of above solution, into 100 ml std. flask add 1 ml of conc<sup>n</sup> HNO<sub>3</sub> and then add 5 ml of 40% NH<sub>4</sub>SCN with a Burette. Make up the solution upto the mark with distilled water. Now take the sample solution into colorimeter tube and measure the O.D. (optical density) using the photo colorimeter. From the O.D. we can measure the concentration of Fe<sup>2+</sup> from the calibration curve. The calibration curve is as follows:

## STANDARD VALUES FOR CALIBRATION CURVES

| Conc <sup>n</sup> of Fe <sup>2+</sup> is Mg | Optical density |
|---|-----------------|
| 0.05  | 0.11            |
| 0.10  | 0.22            |
| 0.15  | 0.32            |
| 0.20  | 0.40            |
| 0.25  | 0.50            |
| 0.30  | 0.62            |
| 0.35  | 0.75            |
| 0.40  | 0.80            |



## RESULT

The optical density of the given solution is \_\_\_\_\_

The amount of  $\text{Fe}^{2+}$  present in the given solution of cement solution is \_\_\_\_\_ mg

## REFERENCE BOOKS

1. Practical Engineering Chemistry by K. Mukkanti, et al, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

## Experiment No. 5

## POTENTIOMETRIC TITRATIONS OF STRONG ACID Vs. STRONG BASE

**AIM**

To determine the strength of HCl solution by titrating against standard sodium hydroxide solution potentiometrically.

**APPARATUS**

1. Potentiometer
2. Hydrogen electrode
3. Calomel electrode
4. Salt bridge
5. Burette
6. Beaker
7. Glass rod

**CHEMICALS**

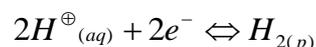
1. HCl solution
2. Standard NaOH solution

**PRINCIPLE**

Since the neutralization of acids and bases are always accompanied by change in the concentration of  $H^+$  &  $OH^-$ , it is evident that hydrogen electrode may be employed in these titrations. The reference electrode used in this case is a calomel electrode.

**Hydrogen Electrode**

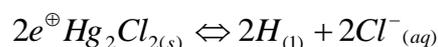
A std. hydrogen electrode consists of 1M HCl, a glass jacket with platinum wire having platinized Pt foil that makes contact with outer circuit. Pure and dry  $H_2$  is passed through glass jacket at a pressure of one atm. The reaction that takes place in std. hydrogen electrode is



The electrode potential of this electrode is taken as zero volt.  $E^{\ominus}_{H_2/2H^{\oplus}} = 0.00 \text{ volts}$

**Calomel Electrode**

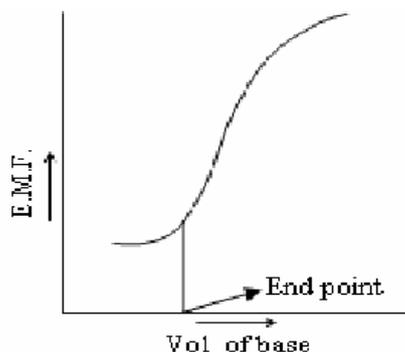
This constitutes of mercury-mercurous chloride electrode. It consists of mercury in contact with a std. solution of KCl and  $Hg_2Cl_2$ . The potential of this electrode depends upon conc<sup>n</sup> of KCl solution. When std. KCl is used it comes to be +0.2458 volts at 25<sup>0</sup>C on hydrogen scale. The electrode reaction taking place in this half cell is:



The principle involved in this type of titrations are

$$\text{EMF} = E_{\text{right}} - E_{\text{left}}$$

A known volume of acid to be titrated is taken in a beaker having a standard hydrogen electrode (SHE). It is connected to a calomel electrode through a salt bridge. Both electrodes are now connected to a potentiometer. Now add a known volume of NaOH for 30 times (1ml each time) and the EMF of the solution is recorded after each addition of the base into the beaker. The values of EMF are plotted against the volume of NaOH added, from this the end point can be known.



### Quinhydrone Electrode

This is a redox electrode at which a reversible reaction occurs. The quinhydrone electrode has a platinum wire dipped in a solution of unknown pH. The solution is made saturated with quinhydrone. For determining the pH value of this half cell, it is combined with another reference electrode, usually a saturated calomel electrode. The quinhydrone electrode gives pH values as accurately as the hydrogen electrode.

### PROCEDURE

Pipette out 20 ml of the acid solution in a 100 ml beaker and set up the following cell by combining hydrogen and Calomel electrodes.



The Calomel electrode may be dipped directly into a solution (or) through the KCl-salt-bridge. Now fill the burette with standard NaOH solution.

Standardise the potentiometer. Connect the electrodes to the potentiometer and measure the EMF of the cell. Then by adding 1 ml of NaOH solution record the EMF of the cell and continue for 30 readings and record the EMF of the cell every time.

**SAMPLE TABLE**

| S. No. | Total volume of NaOH | EMF/ millivolts or pH |
|--------|----------------------|-----------------------|
| 1.     |                      |                       |
| 2.     |                      |                       |
| 3.     |                      |                       |
| 4.     |                      |                       |
| 5.     |                      |                       |
| 6.     |                      |                       |
| 7.     |                      |                       |
| 8.     |                      |                       |
| 9.     |                      |                       |
| 10.    |                      |                       |

$$N_1V_1 = N_2V_2$$

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

$N_1 = 0.01\text{N NaOH};$

$V_1 = \text{Volume of NaOH at the end point (from the graph)}$

$N_2 = \text{Normality of HCl (To be calculated)}$   $V_2 = \text{Volume of HCl (20 ml)}$

$$\text{Strength of HCl} = \text{Normality of HCl} \times \text{Equivalent weight of HCl}$$

$$= N_2 \times 36.5 \text{ g/l}$$

**RESULT**

Strength of HCl \_\_\_\_\_ g/L

**REFERENCE BOOKS**

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

## Experiment No.6

**ESTIMATION OF HARDNESS OF WATER BY EDTA METHOD****AIM**

To estimate the total, permanent and temporary hardness of water by complexometric (EDTA) method.

**APPARATUS**

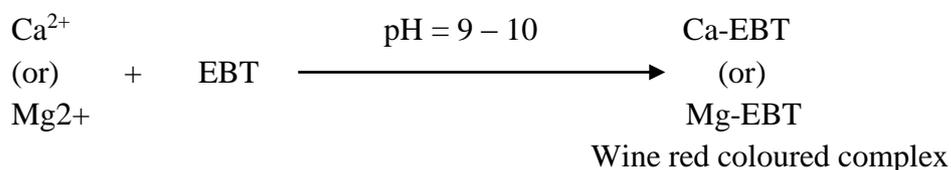
1. Conical flask
2. Burette
3. Pipette
4. Burette stand
5. Spatula

**CHEMICALS**

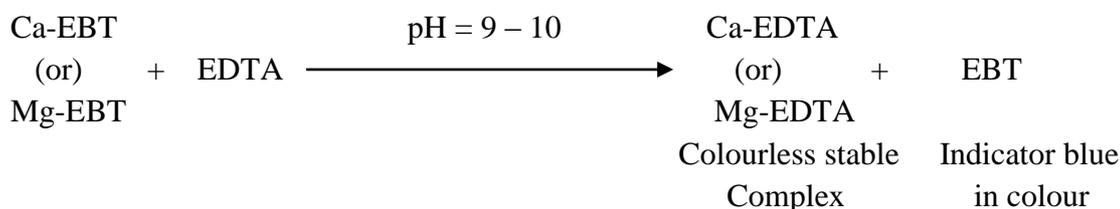
1. Buffers solution
2. Eriochrome black-T indicator (EBT)
3. EDTA Solution

**PRINCIPLE**

EDTA forms colorless, stable complexes with  $\text{Ca}^{2+}$  &  $\text{Mg}^{2+}$  ions present in water at pH 9-10. To maintain pH at 9-10,  $\text{NH}_4\text{Cl}$  &  $\text{NH}_4\text{OH}$  buffer is used. Eriochrome Black-T (EBT) is used as Indicator. Hard water sample with EBT indicator forms unstable, wine red coloured complexes with  $\text{Ca}^{2+}$  &  $\text{Mg}^{2+}$  present in water.



The metal indicator complex is less stable than metal EDTA complex. So this metal indicator complex is titrated with std. EDTA solution. Now colourless metal-EDTA complex is formed by releasing EBT indicator which is blue colour. So the colour change from wine red to blue indicates the end point.



One mole of EDTA reacts with one mole of complex, hence equimolar reaction.(n=1).

**PROCEDURE****Prep<sup>n</sup> of std. hard water**

Dissolve 1 gm of CaCO<sub>3</sub> (dry) in minimum (2-3 drops) quantity of dil. HCl and evaporate the solution on water bath. Dissolve this Sol<sup>n</sup> in small amount of distilled water and then transfer into 1000 ml std. flask. Make up the sol<sup>n</sup> upto the mark and shake well for uniform concentration.

**Standardisation of EDTA Solution**

Pipette out 20 ml of std. hard water solution into a conical flask, add 2 ml of buffer sol<sup>n</sup> and 2-3 drops of EBT indicator and titrate the wine red colour complex sol<sup>n</sup> with EDTA sol<sup>n</sup> taken in the burette, till the colour changes to blue colour. Note the reading. Repeat the titration to get concurrent values.

| S.No. | Vol. of std. hard water | Burette reading |       | Vol. of EDTA required |
|-------|-------------------------|-----------------|-------|-----------------------|
|       |                         | Initial         | Final |                       |
|       |                         |                 |       |                       |

$$\frac{M_1 V_1}{n_1} = \frac{M_2 V_2}{n_2} \quad (\text{Note that } n_1 = n_2 = 1)$$

$$M_1 V_1 (\text{Hard water}) = M_2 V_2 (\text{EDTA sol}^n)$$

**Estimation of Total Hardness**

Pipette out 50 ml of hard water sample (tap water) into 250 ml conical flask, add 2 ml of buffer sol<sup>n</sup> and 3 drops of EBT-Indicator. Titrate the wine red coloured sol<sup>n</sup> with EDTA, till the blue colour is obtained. Note the reading. Repeat the titration to get concurrent values.

| S.No. | Vol. of std. hard water | Burette reading |       | Vol. of EDTA required |
|-------|-------------------------|-----------------|-------|-----------------------|
|       |                         | Initial         | Final |                       |
|       |                         |                 |       |                       |

$$\frac{M_3 V_3}{n_3} = \frac{M_2 V_2}{n_2} \quad (\text{Note that } n_3 = n_2 = 1)$$

$$M_3V_3 \text{ (Tap water)} = M_2V_2 \text{ (EDTA solution)}$$

$$\text{Total Hardness} = M_3 \times 100 \times 1000 \text{ ppm}$$

### Estimation of Permanent Hardness

Pipette out 100 ml of tap water and boil the solution till the volume is reduced to half i.e. 50 ml (All bicarbonates of  $\text{Ca}^{2+}$  &  $\text{Mg}^{2+}$  decomposes to  $\text{CaCO}_3$  and  $\text{Mg(OH)}_2$  respectively). Cool the solution and filter the water into conical flask. Now add 2 ml of buffer sol<sup>n</sup> and 3 drops of EBT-indicator, till the blue colour is obtained. Note the readings. Repeat the titration to set concurrent values.

| S.No. | Vol. of std. hard water | Burette reading |       | Vol. of EDTA required |
|-------|-------------------------|-----------------|-------|-----------------------|
|       |                         | Initial         | Final |                       |
|       |                         |                 |       |                       |

$$\frac{M_4V_4}{n_4} = \frac{M_2V_2}{n_2} \quad (\text{Note that } n_4 = n_2 = 1)$$

$$M_4V_4 \text{ (Heated tap water)} = M_2V_2 \text{ (EDTA solution)}$$

$$\text{Permanent hardness} = M_4 \times 100 \times 1000 \text{ ppm}$$

$$\text{Temporary hardness} = \text{Total hardness} - \text{Permanent hardness}$$

### RESULT

Temporary hardness of water = \_\_\_\_\_ ppm

Permanent hardness of water = \_\_\_\_\_ ppm

Total hardness of water = \_\_\_\_\_ ppm

### REFERENCE BOOKS

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

## Experiment No. 7

**PREPARATION OF ASPIRIN****AIM**

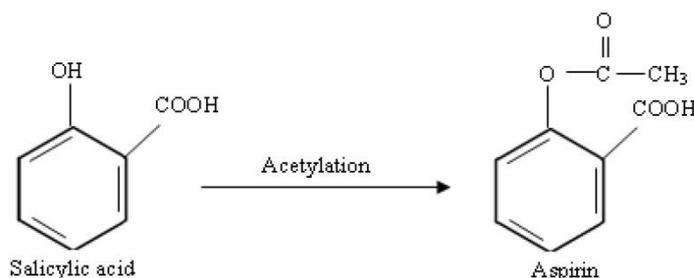
To prepare a pure sample of Aspirin

**APPARATUS**

1. Measuring jar
2. Beaker
3. Round bottom flask
4. Glass rod
5. Watch glass
6. Funnel

**CHEMICALS**

1. Salicylic acid
2. Acetic anhydride
3. Acetic acid
4. Ethanol

**PRINCIPLE**

Acetylation mixture used for the above reaction is acetic anhydride in presence of acetic acid.

**PROCEDURE**

Add 3 gms of salicylic acid to the clean R.B. flask. Mix 5 ml of acetic anhydride and 5 ml of acetic acid in a beaker, add this mixture to the R.B. flask containing salicylic acid. Heat the mixture for about 20 min. on the water bath. Then add this hot mixture in to the ice cold water taken in a beaker and stir vigorously. Crude solid sample of aspirin separates out. Filter solid aspirin using funnel. The yield is about 3.5 gm.

**RECRYSTALLISATION**

Crude aspirin consists of some impurities. To remove these impurities, take the crude aspirin solid into the beaker and add 10 ml of ethanol and 25 ml of water, warm the mixture until aspirin dissolves. Remove the heat and cool the beaker. Now needle like crystals of aspirin separate out.

**RESULT**

Yield of pure sample of aspirin is \_\_\_\_\_ gms

## **REFERENCE BOOKS**

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

## Experiment No. 8

**DETERMINATION OF THE VISCOSITY OF THE GIVEN LIQUIDS****AIM**

Determination of the Viscosity of given liquids.

**APPARATUS**

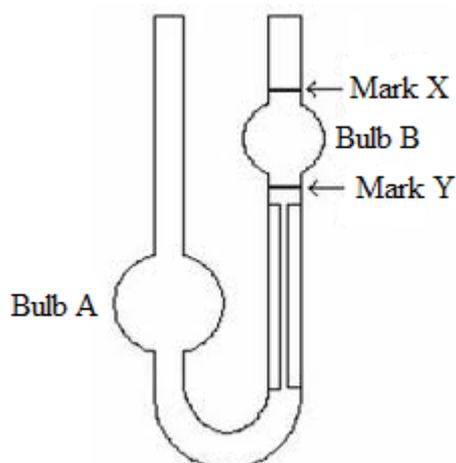
1. Ostwald Viscometer
2. Stop Clock

**CHEMICALS**

1.  $C_6H_6$  (Benzene)
2.  $C_6H_5CH_3$  (Toluene)
3.  $CH_3COOCH_3$  (Methyl Acetate)
4.  $CH_3COOH$  (Acetic Acid)

**PROCEDURE**

1. Take a clean and dry Ostwald Viscometer and set it vertically on a stand.
2. Close the viscometer with finger on the top of Bulb A side and introduce water through Bulb B side until the water level is just above the Mark X. By lifting the finger on Bulb A side slowly adjust the water level exactly at Mark X.
3. Remove the finger from the top of Bulb A side (Simultaneously start the stop clock) to allow the water level to drop from Mark X to Mark Y. Note down the time taken for water to drop down from Mark X to Mark Y in the table..
5. Repeat steps 2 and 3 for each of the given organic solvents and note down the time of flow for each liquid in the table.



| S.No | Liquid (Density<br>g/cm <sup>3</sup> )               | Time flow (in Sec) |         | Time (average) | Viscosity<br>(milliopoise) |
|------|--|--------------------|---------|----------------|----------------------------|
|      |  | Trial 1            | Trial 2 |                |                            |
| 1    | Water (1.00)   |                    |         |                | 10.2                       |
| 2    | C <sub>6</sub> H <sub>6</sub> (0.87)                 |                    |         |                |                            |
| 3    | C <sub>6</sub> H <sub>5</sub> CH <sub>3</sub> (0.86) |                    |         |                |                            |
| 4    | CH <sub>3</sub> COOCH <sub>3</sub> (0.93)            |                    |         |                |                            |
| 5    | CH <sub>3</sub> COOH (1.05)                          |                    |         |                |                            |

## CALCULATIONS

Use the following formula to calculate the viscosity of each organic solvent ( $\eta_2$ ) and record the answers in the table.

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

Where  $\eta_1$  is the viscosity of water (given in the table).

$d_1$  and  $d_2$  are the densities of water and given liquid respectively.

$t_1$  and  $t_2$  are average flow times of water and given liquid respectively.

## RESULT

Viscosity of the given liquids is Benzene = -----(milliopoise)

Toluene = -----(milliopoise)

Methyl acetate = -----(milliopoise)

## REFERENCE BOOKS

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

*Experiment No. 9***DETERMINATION OF SURFACE TENSION****AIM**

To determine the effect of soap and detergent on the surface tension of water using a Stalagmometer.

**APPARATUS**

1. Stalagmometer
2. Volumetric flask

**CHEMICALS**

1. Detergent solution

**PRINCIPLE**

Surface tension is defined as the force in dynes acting on a surface at right angles to any line of unit length or per centimeter.

There are two methods of determining surface tension

1. Drop weight method and
2. Drop number method

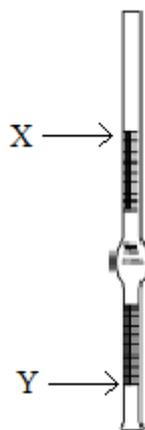
Surface tension of a liquid is measured by using an apparatus called stalagmometer. The method is based on the principle that the weight of a liquid falling from a capillary tube held vertical is approximately proportional to the surface tension of the liquid. It is more convenient to count the number of drops of the liquid than finding the weight of a single drop. The weights of equal volumes of two liquids are proportional to their densities. If  $N_1$  and  $N_2$  are the number of drops of two liquids of the same volume, then:

$$\frac{\gamma_1}{\gamma_2} = \frac{N_2 d_1}{N_1 d_2}$$

Where  $\gamma_1$  and  $\gamma_2$  are surface tensions of water (given in the table) and solution (to be calculated) respectively;  $d_1$  and  $d_2$  are the densities of water and solution (given in the table) respectively.

**DESCRIPTION OF APPARATUS**

Stalagmometer is especially used for relative methods of determination of surface tension for comparing surface tension of liquids. It consists of a capillary tube the end of which is flattened to provide a large dropping surface with a sharp boundary. The capillary tube extends upwards upward into bulb which terminates again into a capillary. On either ends of bulb are found marks X and Y the surface tension may be determined by counting of drops formed by liquid volume from X to Y. The number of drops is counted for a liquid provided with a scale the fraction of the drop may be estimated to an accuracy of 0.05 of a drop. To check the flow the capillary tube is attached to a rubber fuse filled with a pinch cock at one of the ends the rate of flows should not exceed 15 drops per minute if it is more than this it should be decreased with the help of rubber tube with pinch cock.



## PROCEDURE

- 1 Take a clean and dry Stalagmometer.
- 2 Suck distilled water into the Stalagmometer till the level reaches to the top mark above the bulb (X).
- 3 Now allow the water to flow through the capillary and count the number of drops formed till the liquid level reaches the lowest mark below the bulb (Y).
- 4 Prepare 2% detergent solution by dissolving 2gms of detergent in 100 ml distilled water.
- 5 Repeat Steps 2 and 3 with 2% detergent solution
- 6 Dilute the 2% detergent solution to different concentrations as given in the table and repeat Steps 2 and 3 with each solution.
7. Use the formula to calculate the surface tension of each liquid. Record your data and the surface tension of each liquid in the table.

| No | Solution / Water                            | Concentration of detergent Solution (Density) | Number of drops |       |      | Surface Tension Dynes/cm |
|----|---|---|-----------------|-------|------|--------------------------|
|    |   |   | Exp-1           | Exp-2 | Ave. |                          |
| 1  | Pure Water                                  | 0% (1)  |                 |       |      | 72                       |
| 2  | Pure detergent solution                     | 2%(1.04)                                      |                 |       |      |                          |
| 3  | 50 ml water + 50 ml 2% detergent solution   | 1%(1.03)                                      |                 |       |      |                          |
| 4  | 50 ml water + 50 ml 1% detergent solution   | 0.5%(1.02)                                    |                 |       |      |                          |
| 5  | 50 ml water + 50 ml 0.5% detergent solution | 0.25%(1.01)                                   |                 |       |      |                          |

## RESULT

Surface Tension of the given liquids is 2% solution = ----- Dynes/cm

1% solution = ----- Dynes/cm

0.5% solution = ----- Dynes/cm

0.25% solution = ----- Dynes/cm

## REFERENCE BOOKS

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

*Experiment No. 10***DETERMINATION OF PERCENTAGE OF COPPER IN BRASS****AIM**

To determine the amount of copper in the brass sample by iodometric titration.

**APPARATUS**

1. Burette
2. Pipette
3. Beaker
4. Watch glass
5. Water bath
6. Conical flask
7. Volumetric flask

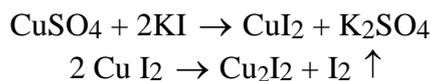
**CHEMICALS**

1. Ammonia
2. Acetic acid
3. KI
4. Hypo solution ( $\text{Na}_2\text{S}_2\text{O}_3$ )
5. Starch indicator
6. Con.  $\text{HNO}_3$
7. Con.  $\text{H}_2\text{SO}_4$
8.  $\text{H}_3\text{PO}_4$
9. KSCN
10. Standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution
11.  $\text{Na}_2\text{CO}_3$

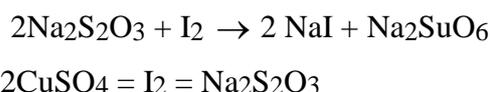
**PRINCIPLE**

Estimation of copper present in alloys and ores is widely carried by iodometry.

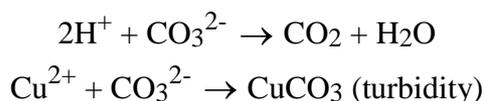
The brass sample given for estimation is dissolved in acid and treated with excess of KI making the copper solution neutral. Quantitative liberation of iodine takes place.



The liberated iodine is titrated against std. Hypo solution using starch as an indicator.



Before doing the titration, the acidic  $\text{CuSO}_4$  solution must be neutralized by the dropwise addition of 10%  $\text{Na}_2\text{CO}_3$  (1 Spatula of solid  $\text{Na}_2\text{CO}_3$ ) until a slight turbidity (or) ppt occurs.



The turbidity is removed by dropwise addition of dil.  $\text{CH}_3\text{COOH}$ .



The ppt of  $\text{Cu}_2\text{I}_2$  absorbs  $\text{I}_2$ , and releases slowly making the detection of end point difficult. To avoid this disadvantage a small amount of  $\text{KSCN}$  is added near the end point to release the absorbed  $\text{I}_2$  from  $\text{Cu}_2\text{I}_2$  ppt.

## PROCEDURE

### Preparation of Copper Solution from Brass

Weigh out 0.25 gm of brass sample into conical flask. Add 5 ml of Con.  $\text{HNO}_3$  and 5 ml of Con.  $\text{H}_2\text{SO}_4$ . Cool the solution and add this copper solution into 10 ml of  $\text{NH}_3$  solution taken in a 250 ml beaker, drop wise from the sides of the beaker by cooling the beaker from time to time. Wash the conical flask with 2 to 3 ml of distilled water and transfer the washings into the beaker. Repeat this washing process for 2 to 3 times so that all the copper solution from the conical flask is transferred into ammonia solution. Stir this copper solution till blue colour solution is formed due to formation of cupric ammonium complex. Now add 2 ml of  $\text{H}_3\text{PO}_4$  to this solution and transfer this copper solution from the beaker into a 100 ml volumetric flask. Wash the beaker with 2 to 3 ml of distilled water and transfer the washings into the conical flask. Repeat this washing process 2 to 3 times. Now make up the solution up to the mark with distilled water.

### Standardisation of Hypo

Transfer one spatula KI solid, 1 gm of  $\text{NaHCO}_3$  and 5 ml of concentration  $\text{HCl}$  into a conical flask and mix the contents. Now ppt out 20 ml of  $\text{K}_2\text{Cr}_2\text{O}_7$  into the same conical flask and keep in dark for 5 min by closing the conical flask with a paper. Now titrate the mixture against hypo solution until a light greenish yellow colour is developed in the solution. Now add 1 ml of starch indicator and continue the titration until the blue colour solution turns to green colour. Repeat the titration for concurrent values.

| S.No. | Vol. of $K_2Cr_2O_7$ | Burette readings |       | Volume of Hypo |
|-------|----------------------|------------------|-------|----------------|
|       |                      | Initial          | Final |                |
|       |                      |                  |       |                |

$$N_1V_1 = N_2V_2$$

( $K_2Cr_2O_7$ ) (Hypo solution)

### Estimation of Copper Solution

Transfer 5 ml of copper solution into clean conical flask and neutralize the copper solution by adding solid  $Na_2CO_3$  or  $Na_2CO_3$  solution drop wise until a white ppt is formed. Now add acetic acid drop wise until the ppt disappears. Now add 1 spatula of KI solid (or 20 ml of KI solution) and keep the solution in dark for 5 min. Titrate the liberated iodine with hypo till light greenish yellow is obtained. Now add 1 ml of starch solution and a pinch of KSCN and continue the titration until the blue colour solution changes to milky white.

Repeat the titration for concurrent values.

| S.No. | Vol. of copper solution | Burette readings |       | Volume of Hypo |
|-------|-------------------------|------------------|-------|----------------|
|       |                         | Initial          | Final |                |
|       |                         |                  |       |                |

$$N_3V_3 = N_2V_2$$

(Copper solution) (Hypo solution)

### CALCULATIONS

$$\begin{aligned} \text{Amount of copper in solution} &= N_3 \times \text{Equivalent weight of copper (gm/liter)} \\ &= N_3 \times 63.5/10 \text{ (gm/100 ml)} \end{aligned}$$

$$\text{Percentage of copper in brass sample} = \frac{\text{Amount of copper (gm)} \times 100}{\text{weight of the brass sample (gm)}}$$

### RESULTS

$$\text{Percentage of copper in the given brass sample} = \underline{\hspace{2cm}} \%$$

**REFERENCE BOOKS**

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

## Experiment No. 11

**ESTIMATION OF MANGANESE DIOXIDE IN PYROLUSITE****AIM**

To estimate the amount of manganese dioxide (MnO<sub>2</sub>) present in the given sample of Pyrolusite.

**APPARATUS**

1. Balance
2. Pipette
3. Burette
4. Conical flask
5. Glass rod
6. Funnel
7. Beaker

**CHEMICALS**

1. Pyrolusite
2. Standard Sodium Oxalate solution
3. Dil. H<sub>2</sub>SO<sub>4</sub>
4. KMnO<sub>4</sub> solution

**PRINCIPLE**

The MnO<sub>2</sub> present in the pyrolusite sample is reduced by a known excess of standard sodium oxalate solution and the unreacted oxalate is titrated against a std. KMnO<sub>4</sub>, where KMnO<sub>4</sub> acts as self indicator. Pale pink colour is the end point of the titration.

**PROCEDURE****Standardization of KMnO<sub>4</sub> Solution**

Fill the Burette with KMnO<sub>4</sub> solution. Pipette out 10 ml of sodium oxalate into a clean 250 ml conical flask and add 5 ml of 4N H<sub>2</sub>SO<sub>4</sub> and heat the solution, till some fumes come out of the solution. Titrate the hot solution with KMnO<sub>4</sub>, till pale pink colour is observed. Repeat till the concurrent values are observed.

| S.No. | Vol. of sodium oxalate | Burette reading |       | Vol. of KMnO <sub>4</sub> |
|-------|------------------------|-----------------|-------|---------------------------|
|       |                        | Initial         | Final |                           |
|       |                        |                 |       |                           |

$$N_1V_1 = N_2V_2$$

(Sodium oxalate) = (KMnO<sub>4</sub>)

### Estimation of Pyrolusite Sample

Weigh about 1-1.5 gm of Pyrolusite sample into a 250 ml flask, add 20 ml (excess) of sodium oxalate with the pipette, and heat the solution until the Pyrolusite is dissolved and titrate the unreacted oxalate with KMnO<sub>4</sub> solution taken in the burette until the pink colour (end point) is obtained.

| S.No. | Vol. of sodium oxalate | Burette reading |       | Vol. of KMnO <sub>4</sub> |
|-------|------------------------|-----------------|-------|---------------------------|
|       |                        | Initial         | Final |                           |
|       |                        |                 |       |                           |

### Blank Reading

Now take 20 ml of Sodium oxalate with the pipette into conical flask and heat the solution and then titrate the oxalate with KMnO<sub>4</sub> solution taken in beaker, until the pale pink colour (end point) is obtained.

| S.No. | Vol. of sodium oxalate | Burette reading |       | Vol. of KMnO <sub>4</sub> |
|-------|------------------------|-----------------|-------|---------------------------|
|       |                        | Initial         | Final |                           |
|       |                        |                 |       |                           |

$$N_3V_3 = N_2V_2$$

(Pyrolusite) = (KMnO<sub>4</sub>)

V<sub>3</sub> = Blank titrate value – Titrate value of Pyrolusite

$$\text{Amount of MnO}_2 = \frac{\text{Volume (V}_3) \times \text{Normality} \times \text{Equivalent Wt. of MnO}_2 (43.46)}{1000}$$

$$\text{Percentage of MnO}_2 = \frac{\text{Amount of MnO}_2}{\text{Wt of Pyrolusite}} \times 1000$$

## RESULT

Amount of MnO<sub>2</sub> = \_\_\_\_\_ gm

Percentage of MnO<sub>2</sub> = \_\_\_\_\_ %

## REFERENCE BOOKS

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

*Experiment No. 12***ADSORPTION OF ACETIC ACID ON CHARCOAL****AIM:**

To study the adsorption of acetic acid on activated charcoal.

**CHEMICALS**

1. activated charcoal
2. NaOH solution
3. phenolphthalein indicator

**APPARATUS:**

1. Stoppered reagent bottle
2. conical flask
3. pipette
4. burette
5. filter paper

**INTRODUCTION:**

Adsorption is a surface phenomenon of a solid due to the existence of unbalanced or residual forces on the surface of a solid. The solid attracts molecules of other species in contact either from the gas phase or from solution on its surface. There is a greater concentration of the adsorbed molecules at the surface of the solid than in the gas phase or in the bulk solution. This phenomenon is known as ADSORPTION. Adsorption depends on the specific area of the solid, the equilibrium solute concentration in solution or pressure in case of gaseous adsorption, the nature of adsorbent and temperature

**PRINCIPLE:**

1. The Freundlich isotherm may be written as;

$$X/m = k c^a$$

(OR)

$\ln(X/m) = \ln k + a \ln c$  Therefore  $\ln(X/m)$  Vs  $\ln c$  is a straight line

2. Langmuir isotherm may be written as

$$X/m = \frac{k_1 c}{1 + k_1 c}$$

$$c/(X/m) = 1/k_1 + c/k_1$$

Where  $X$  is the mass of the solute adsorbed on mass 'm' of the adsorbent and  $c$  is the equilibrium concentration of the adsorbate in the solution.

Hence, a plot of  $c/(X/m)$  Vs  $c$  will be a straight line from which the constants  $k_1$  and  $k_2$  can be calculated.

#### PROCEDURE:

Acetic acid and sodium hydroxide solutions are prepared in the usual manner. Five stoppered reagent bottles (250ml) are taken and are cleaned and dried. The bottles are numbered properly. One gram each of activated charcoal or silica gel is taken in each of them and the following solution in the bottle is prepared according to the given table

**TABLE:**

| S.NO | Bottle No<br>ml | Vol. Of acetic acid soln | Vol. Of distilled water,<br>ml | Total Volume<br>ml, |
|------|-----------------|--------------------------|--------------------------------|---------------------|
| 1    | 1               | 50                       | 0                              | 50                  |
| 2    | 2               | 40                       | 10                             | 50                  |
| 3    | 3               | 30                       | 20                             | 50                  |
| 4    | 4               | 20                       | 30                             | 50                  |
| 5    | 5               | 10                       | 40                             | 50                  |

Stopper the bottles tightly and shake them for 40 minutes. After shaking the bottles, keep them in a thermostat (or leave them at room temperature) maintained at 25°C for at least one hour. The bottle No.1 is taken and filtered through a clean and dry filter paper. The filtrate is collected after rejecting the first 5ml of it. 10ml of the filtrate is pipetted out into a conical flask and titrate against 0.1N solution of sodium hydroxide using phenolphthalein as indicator until end point is reached. Repeat the same process to the rest of the mixtures.

The time for which charcoal remains in contact with Acetic acid in different bottles is kept constant. (Adsorbent Charcoal or Silica gel)

#### CALCULATIONS:

The weight  $X$  of acetic acid adsorbed per unit mass of charcoal can be computed as  $X = (c_0 - c)MV$ .

Where,  $c_0$  = initial concentration in mol/lit. of acetic acid before adsorption

$c$  = equilibrium concentration in mol/lit. of acetic acid after adsorption

$M$  = molecular weight of acetic acid

V = volume in litres of solution.

$c_0, c, X$  are calculated for each sample and are tabulated.

### GRAPHS:

A graph may be plotted against  $\ln X$  and  $\ln c$  to test the Freundlich adsorption isotherm and  $k$  values are calculated from the straight-line plot as slope =  $a$ , and intercept =  $\ln k$ .

A graph may be plotted between  $c/(X/m)$  and  $c$ . A straight line indicates the validity of Langmuir adsorption isotherm.  $k_1$  and  $k_2$  are obtained from the graphs as;

Slope =  $1/k_1$  and intercept =  $1/(k_1 k_2)$ .

### CALCULATIONS:

#### 1. Standardisation of NaOH:

Normality of Oxalic acid =

Volume of Oxalic acid pipetted out =

Volume of NaOH run down =

Normality of NaOH =

#### 2. Standardisation of Acetic Acid:

Volume of Acetic Acid taken =

Volume of NaOH run down =

Normality of Acetic acid =

The Freundlich adsorption isotherm is,

$$\ln(X/m) = \ln k + a \ln c.$$

From the graph of  $\ln(X/m)$  Vs  $\ln c$

Slope,  $a =$

Intercept,  $k =$

The Langmuir adsorption isotherm is,

$$c/(X/m) = 1/(k_1 k_2) + c/k_2$$

From the graph of  $c/(X/m)$  Vs  $c$ ,

Slope,  $k_1 =$

y-intercept,  $k_2 =$

**RESULT:** The adsorption of acetic acid on activated charcoal is studied and the constants of the equations of Langmuir and Freundlich isotherms are estimated and are:

Freundlich isotherm:

$a =$  -----

$k_1 =$  -----

Langmuir isotherm:

$k =$  -----

$k_2 =$  -----

### REFERENCE BOOKS

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

## Experiment No. 13

**PREPARATION OF THIOKOL RUBBER****AIM**

To synthesise Thiokol rubber using sodium tetra sulphide with 1, 2 dichloro ethane

**APPARATUS**

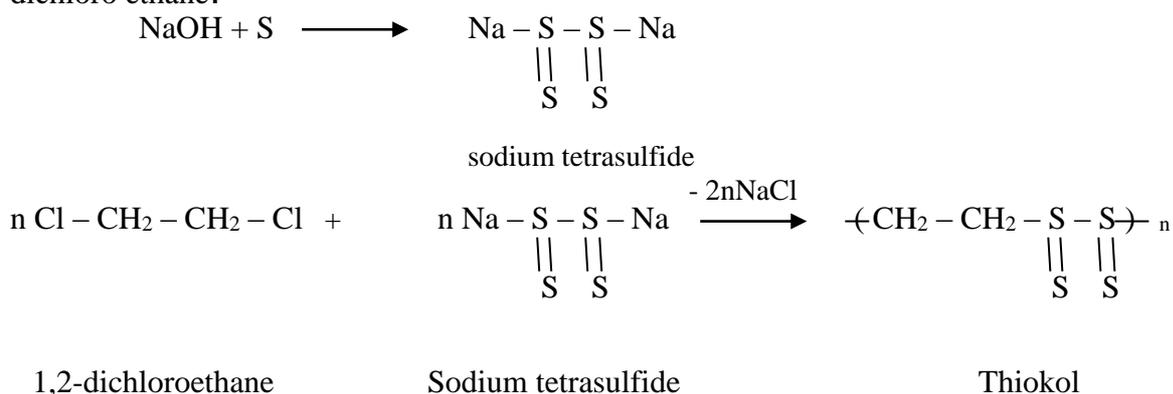
1. Beakers
2. Glass rod

**CHEMICALS**

1. NaOH
2. Powdered Sulphur
3. Ethylene chloride (1,2 dichloro ethane)

**PRINCIPLE**

It is a rubbery white substance and is obtained by treating sodium polysulfide with 1, 2-dichloro ethane.

**PROCEDURE**

1. In a 100 ml beaker dissolve 2 g NaOH in 50-60 ml of warm water.
2. Boil the solution and to this add in small lots with constant stirring 4 g of powdered sulfur. During addition and stirring the yellow solution turns deep-red
3. Cool it to 60-70 °C and add 10 ml of 1,2-dichloroethane (ethylene chloride) with stirring. Stir for an additional period of 20 minutes while rubber polymer separated out as a lump.
4. Pour out the liquid from the beaker in the sink to obtain Thiokol rubber.
5. Wash it with water under the tap.
6. Dry in the fold of filter papers. The yield is about 1.5g.

## **RESULT**

Yield obtained = ----- gms

## **REFERENCE BOOKS**

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

Experiment No. 14

## ESTIMATION OF FERROUS IRON BY DICHROMATE METHOD

### AIM

Determination of iron using potassium dichromate.

### APPARATUS

1. Balance
2. Pipette
3. Burette
4. Conical flask
5. Glass rod
6. Funnel
7. Beaker
8. volumetric flask

### CHEMICALS

1. phosphoric acid
2. sulfuric acid
3. diphenylamine sulfonate indicator.
4. iron(II) solid
5. potassium dichromate

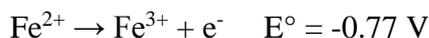
### PRINCIPLE

As an oxidant, dichromate has some advantages over permanganate, but, as it is less powerful, its use is much more limited. It is obtainable in a state of high purity and can be used as a primary standard. Solutions of dichromate in water are stable indefinitely. The half reaction for the dichromate system is:

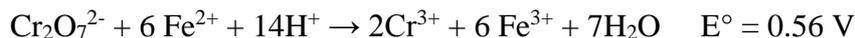


The most important application of dichromate is in its reaction with iron(II) in which it is often preferred to permanganate.

The relevant half reaction is :



and the total reaction is:



Unlike permanganate, dichromate titrations require an indicator. There are three indicators that may be used for the titration of  $\text{Fe}^{2+}$  with  $\text{K}_2\text{Cr}_2\text{O}_7$ . These are diphenylamine, diphenylbenzidine and diphenylamine sulfonate. The colour change for all three indicators is green to violet and the standard electrode potentials are all ca 0.78 V. According to Kolthoff and Sandell, this should lie between the electrode potentials of the two reduction reactions. This not being the case, phosphoric acid is added to reduce the electrode potential for the  $\text{Fe}^{3+} \rightarrow \text{Fe}^{2+}$  reaction by stabilising the ferric ion.

## PROCEDURE

Prepare a standard dichromate solution by dissolving an accurately weighed sample of about 0.4 g in water and make up to 100 ml in a volumetric flask. Into flasks or beakers weigh out accurately duplicate portions of about 0.7 g of the iron(II) solid 'M' provided. Add 30 ml of dil. sulfuric acid, 100ml of water, 7 ml of 85% phosphoric acid and 5 drops of diphenylamine sulfonate indicator. Titrate with dichromate to a purple colour. Calculate the percentage of iron in the solid 'M'.

## RESULT

The amount of iron present in a given sample is = -----mg

## REFERENCE BOOKS

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.
2. Instrumental methods of chemical analysis, Chatwal, Anand, Himalaya Publications.
3. Inorganic quantitative analysis, Vogel.

**APPENDIX – I****Atomic Masses of Elements:**

| <b>Element</b> | <b>Symbol</b> | <b>At. No.</b> | <b>At. Wt.</b> |
|----------------|---------------|----------------|----------------|
| Aluminum       | Al            | 13             | 26.98          |
| Arsenic        | As            | 33             | 74.92          |
| Barium         | Ba            | 56             | 137.34         |
| Calcium        | Ca            | 20             | 40.08          |
| Carbon         | C             | 6              | 12.011         |
| Chlorine       | Cl            | 17             | 35.453         |
| Chromium       | Cr            | 29             | 63.54          |
| Copper         | Cu            | 24             | 51.996         |
| Fluorine       | F             | 9              | 18.998         |
| Gold           | Au            | 79             | 196.967        |
| Hydrogen       | H             | 1              | 1.008          |
| Iodine         | I             | 53             | 126.904        |
| Iron           | Fe            | 26             | 55.847         |
| Lead           | Pb            | 82             | 207.19         |
| Magnesium      | Mg            | 12             | 24.312         |
| Manganese      | Mn            | 25             | 54.938         |
| Mercury        | Hg            | 80             | 200.59         |
| Nitrogen       | N             | 7              | 14.007         |
| Phosphorus     | P             | 15             | 30.974         |
| Potassium      | K             | 19             | 39.102         |
| Silver         | Ag            | 47             | 107.870        |
| Sodium         | Na            | 11             | 22.990         |
| Sulphur        | S             | 16             | 32.064         |
| Tin            | Sn            | 50             | 118.69         |
| Zinc           | Zn            | 30             | 65.37          |

**APPENDIX – II**  
**Equivalent weights of substances used in volumetric Analysis**

| Titration and Substances used  | Formula   | Eq. Wwights  |
|--|---|--|
| <b>ACID – ALKALI TITRATIONS:</b> <ol style="list-style-type: none"> <li>Sodium carbonate</li> <li>Sodium bicarbonate</li> <li>Potassium carbonate</li> <li>Potassium bicarbonate</li> <li>Sodium hydroxide</li> <li>Barium hydroxide</li> <li>Oxalic acid (hydrated)</li> <li>Hydrochloric acid</li> <li>Sulphuric acid</li> </ol> | $\text{Na}_2\text{CO}_3$<br>$\text{NaHCO}_3$<br>$\text{K}_2\text{CO}_3$<br>$\text{KHCO}_3$<br>$\text{NaOH}$<br>$\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$<br>$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$<br>$\text{HCl}$<br>$\text{H}_2\text{SO}_4$      | M.W./2 = 53.06<br>M.W./1 = 84.00<br>M.W./2 = 69.00<br>M.W./1 = 100.00<br>M.W./1 = 40.00<br>M.W./1 = 157.75<br>M.W./2 = 63.03<br>M.W./1 = 36.46<br>M.W./2 = 49.04 |
| <b>REDOX TITRATIONS:</b> <ol style="list-style-type: none"> <li>Potassium permanganate</li> <li>Potassium dichromate</li> <li>Ferrous sulphate (hydrated)</li> <li>Ferrous sulphate (anhydrous)</li> <li>Ferrous ammonium sulphate</li> <li>Oxalic acid (hydrated)</li> <li>Iron (ferrous)</li> </ol>                              | $\text{KMnO}_4$<br>$\text{K}_2\text{Cr}_2\text{O}_7$<br>$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$<br>$\text{FeSO}_4$<br>$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$<br>$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$<br>$\text{Fe}$ | M.W./5 = 31.606<br>M.W./6 = 49.03<br>M.W./1 = 278.00<br>M.W./1 = 152.00<br>M.W./1 = 392.12<br>M.W./2 = 63.04<br>At.W./1 = 55.84                                  |
| <b>IODINE TITRATIONS:</b> <ol style="list-style-type: none"> <li>Iodine</li> <li>Hypo (sodium thiosulphate) (hydrated)</li> <li>Anhydrous sodium thiosulphate</li> <li>Arsenious oxide</li> <li>Copper sulphate (hydrated)</li> </ol>  | $\text{I}_2$<br>$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$<br>$\text{Na}_2\text{S}_2\text{O}_3$<br>$\text{As}_2\text{O}_3$<br>$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  | M.W./2 = 126.92<br>M.W./1 = 248.19<br>M.W./1 = 158.09<br>M.W./1 = 49.45<br>M.W./1 = 249.68   |
| <b>ARGENTOMETRIC TITRATIONS:</b> <ol style="list-style-type: none"> <li>Silver nitrate</li> <li>Silver</li> <li>Sodium chloride</li> <li>Chloride ion</li> <li>Potassium sulphocyanide</li> <li>Ammonium sulphocyanide</li> </ol>  | $\text{AgNO}_3$<br>$\text{Ag}$<br>$\text{NaCl}$<br>$\text{Cl}^-$<br>$\text{KCNS}$<br>$\text{NH}_4\text{CNS}$  | M.W./1 = 169.89<br>At.W./1 = 107.88<br>M.W./1 = 58.45<br>At.W./1 = 35.46<br>M.W./1 = 97.17<br>M.W./1 = 76.12   |

## APPENDIX – III

## (A) Strength of Aqueous solutions of Concentrated Acids and Bases.

| Compound          | Molecular formula              | Molecular weight | Approximate           |                  |          | Volume for making 1 litre of approx 1M solution (ml) |
|-------------------|--------------------------------|------------------|-----------------------|------------------|----------|--|
|                   |                                |                  | % of solute by weight | Specific gravity | Molarity |  |
| Hydrochloric acid | HCl                            | 36.5             | 35                    | 1.18             | 12       | 84   |
| Nitric acid       | HNO <sub>3</sub>               | 63.0             | 70                    | 1.42             | 16       | 63   |
| Sulphuric acid    | H <sub>2</sub> SO <sub>4</sub> | 98.0             | 96                    | 1.84             | 18       | 56   |
| Acetic acid       | CH <sub>3</sub> COOH           | 60.0             | 100                   | 1.05             | 17.5     | 58   |
| Phosphoric acid   | H <sub>3</sub> PO <sub>4</sub> | 98.0             | >85                   | 1.70             | 14.7     | 68   |
| Sodium hydroxide  | NaOH                           | 40.0             | 50                    | 1.48             | 19       | 53   |
| Ammonia           | NH <sub>3</sub>                | 17.0             | 28                    | 0.90             | 15       | 67   |

## (B) Preparation of Dilute Acids and Bases

|                              |  |
|------------------------------|--|
| <b>Dil.acetic acid</b>       | Dilute 285 ml of glacial acetic acid of 1.05 sp. gr to one litre with distilled water, to give 5N solution.  |
| <b>Dil.hydrochloric acid</b> | Dilute 89 ml of conc. HCl of 1.18 sp. gr to one litre with distilled water to produce 1N solution.   |
| <b>Dil.nitric acid</b>       | Dilute 315 ml of conc. HNO <sub>3</sub> of 1.12 sp. gr to one litre with distilled water to produce 5N solution.   |
| <b>Dil.sulphuric acid</b>    | Pour 28 ml of conc. H <sub>2</sub> SO <sub>4</sub> with 1.84 sp.gr slowly and with stirring into 500 ml and dilute to one litre with distilled water to get 1N solution. |
| <b>Sodium hydroxide</b>      | Dissolve 400 gms of solid NaOH in one litre of water.  |

**APPENDIX – IV**

**Weights of substances required to prepare one litre of a solution of given normality in volumetric analysis.**

| S.No. | Substance  | Weight substance to be dissolved for |        |         |         |         |
|-------|--|--------------------------------------|--------|---------|---------|---------|
|       |  | Mol .wt.                             | Eq.wt. | N/10    | N/20    | N/30    |
| 1     | Sodium hydroxide (NaOH)  | 40                                   | 40     | 4.000   | 2.000   | 1.3333  |
| 2     | Anhydrous sodium carbonate (Na <sub>2</sub> CO <sub>3</sub> )                          | 106                                  | 53     | 5.300   | 2.6500  | 1.7667  |
| 3     | Oxalic acid (H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> .2H <sub>2</sub> O)          | 126.08                               | 63.04  | 6.3040  | 3.1520  | 2.1013  |
| 4     | Potassium dichromate (K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> )                  | 294.2                                | 49.03  | 4.9030  | 2.4515  | 1.6343  |
| 5     | Potassium permanganate (KMnO <sub>4</sub> )  | 158.03                               | 31.606 | 3.1606  | 1.5803  | 1.0535  |
| 6     | Mohr's salt or Ferrous ammonium sulphate   | 392.10                               | 392.10 | 39.2100 | 19.6050 | 13.0700 |
| 7     | Copper sulphate (CuSO <sub>4</sub> .5H <sub>2</sub> O)                                 | 249.71                               | 249.71 | 24.9710 | 12.4855 | 8.3237  |
| 8     | Sodium thiosulphate (Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .5H <sub>2</sub> O) | 248.20                               | 248.20 | 24.8200 | 12.4100 | 8.2735  |
| 9     | Silver nitrate (AgNO <sub>3</sub> )  | 169.30                               | 169.90 | 16.9900 | 8.4950  | 5.6635  |
| 10    | Ammonium thiocyanate (NH <sub>4</sub> CNS)   | 76.10                                | 76.10  | 7.6100  | 3.8050  | 2.5367  |

**APPENDIX – V****Indicators used**

| Sl. No. | Titration                       | Approximate pH range at                  | Suitable indicators                        | pH at colour change of indicator    |
|---------|---------------------------------|--|--|-------------------------------------|
| 1       | Strong acid against strong base | 4 to 10                                  | Methyl orange<br>Litmus<br>Phenolphthalein | 3.1 – 4.4<br>4.5 – 8.3<br>8.0 – 9.6 |
| 2       | Strong acid against weak base   | 3.5 to 7                                 | Methyl orange<br>Methyl red                | 3.4 – 4.4<br>4.2 – 6.3              |
| 3       | Weak acid against strong base   | 6.5 to 10                                | Phenolphthalein                            | 8.0 – 9.6                           |
| 4       | Weak acid against weak base     | No sharp inflection at equivalence point | None                                       |                                     |

**APPENDIX –VI*****Guidance for Lab Assistants:*****Preparation of stock solutions (solutions to be given to students)**

1. **Mohr's salt (Ferrous ammonium sulphate):** For estimation of  $\text{Fe}^{2+}$  by permanganometry and dichrometry = Dissolve 98.035 gms of Mohr's salt in 1000 cc of water and give 20 ml or 21 ml or 22 ml to each student in a 100 ml standard flask.
2. **Ferric Iron:** Take 40.59 gms of  $\text{FeCl}_3$  in a 1000 cc standard flask, dissolve in 10 ml of conc. HCl and make up the solution to the mark with distilled water. Give 20 ml or 21 ml or 22 ml to each student in a 100 ml standard flask.
3. **Copper sulphate:** For iodometry dissolve 62.43 gms of copper sulphate in small amount of (5ml) of conc. HCl in a 1000 cc standard flask, make up the solution to the mark with distilled water and give 20 ml, 21 ml or 22 ml to each student in a 100 ml standard flask. (for complexometry dissolve 6.5 gms of copper sulphate in 1000 cc of distilled water after dissolving in 10 ml conc. HCl)
4. **Potassium Ferrocyanide:** Dissolve 194.125 gms of potassium ferrocyanide and 3 gms. Ferricyanide in 1000 ml of distilled water in 1000 ml standard flask. Shake the solution well and give 20 ml, 21 ml or 22 ml to each student in a 100 ml standard flask.