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## DEPARTMENT OF CHEMISTRY

Chemistry is one of the vital branches of science without which life does not exist. Engineering is the application of basic sciences and it may be noted that all engineering branches originated from basic sciences. Science without technology is useless and technology without science is blind.

Department of Chemistry has been established during the year 1962 and staffs pattern of the Department have one Professor, one Associate Professor and three Asst. Professors. Among them three had Ph.D. degree and rest of them M.Sc: degree holders. Department of Chemistry have well equipped laboratory with area of about 4500 sq. ft.

Course title: **Engineering Chemistry lab**

Course code: **P18CHL 17 / 27**

Scheme of valuation with effect from **2018** Examination onwards

<b>Distribution of Marks</b>	<b>Max. Marks</b>	<b>Part (A + B) Marks</b>
Procedure writing	<b>10</b>	<b>05 + 05</b>
Conduction of experiments	<b>24</b>	<b>12 + 12</b>
Calculation, graph and results	<b>10</b>	<b>05 + 05</b>
Viva – Voce	<b>06</b>	<b>03 + 03</b>
<b>Total</b>	<b>50</b>	<b>25 + 25</b>

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## VOLUMETRIC METHOD OF CHEMICAL ANALYSIS

### PART - A

#### Instructions to the students:

- ☞ Read the procedure before commencement of the experiment and do the experiment according to the procedure.
- ☞ Handle the glass wares with care.
- ☞ Apparatus used: Burette, Pipette, Standard flask, conical flask, Beaker, Funnel, washing bottle for volumetric analysis.
- ☞ Wash the all apparatus with tap water and rinse them with distilled water before commencement of the experiment.
- ☞ Wash all the apparatus with tap water again after the experiment is completed and return them to the instructor.

**Note: Students are require to checkup all the glass wares and equipments before starting of the experiment. If any broken or damage bring the notice of concern teachers or Lab instructors.**

#### Definitions

**Standard solution:**-Solution whose normality is known

**Standardization:** - Determination of the strength of solution using standard solution.

**Estimation:** - Determination of the weight of a given substance present in a known volume of a solution.

**Normality:** - Number of gm equivalent weight of the substance present in one liter of its solution.

**Molarity:** - Number of gm molecular weight of the substance present in one liter of its solution.

**Indicator:** - It is a substance, which shows the end point of the reaction by its color change.

**Oxidation:** - It is process of loss of electrons by atoms or ions.

**Reduction:** - It is process of gain of electrons by atoms or ions.

**Oxidizing agent:**-Substance which gain of electrons. eg.  $K_2Cr_2O_7$ ,  $KMnO_4$  etc.

**Reducing agent:**-Substance which loss of electrons. eg. FAS,  $FeCl_2$ , hypo etc.

**Observations and calculations:****I. Preparation of standard potassium dichromate solution:**

Weight of weighing bottle +  $K_2Cr_2O_7$  crystals  $w_1 = \text{-----} \text{ g}$

Weight of empty weighing bottle  $w_2 = \text{-----} \text{ g}$

$\therefore$  Weight of  $K_2Cr_2O_7$  crystals taken  $(w_1 - w_2) = \text{-----} \text{ g}$

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{Weight of } K_2Cr_2O_7 \times 4}{\text{Equivalent weight of } K_2Cr_2O_7}$$

$$N \text{ } K_2Cr_2O_7 = \frac{\text{-----} \times 4}{49} = \text{-----} \text{ (a) N}$$

**II. Estimation of hypo:**

Burette : Hypo solution

Conical flask: 25 ml of  $K_2Cr_2O_7$  solution +  $\frac{1}{4}$  t.t concentrated HCl + 10ml  
of 10% KI + shake well + one tt of distilled water

Indicator : 2 ml of freshly prepared starch.

End point : Disappearance of blue colour.

Burette readings	I	II	III
Final burette readings			
Initial burette readings			
Volume of hypo run down in mls			

$\therefore$  Volume of hypo solution required =  $V = \text{-----} \text{ ml}$

**PART – A****Volumetric methods of chemical analysis****Expt. No. : 1****Date:****Estimation of hypo using potassium dichromate crystals.****Theory:**

The commercial name of sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) is hypo. It is obtained in a solid state and it is affected by moisture and sunlight. It is standardized by potassium dichromate. It is very good reducing agent and its equivalent weight and molecular weight is 248. The solution of hypo is affected with all oxidizing agents. Hypo is estimated using potassium dichromate solution along with potassium iodide in presence of acid medium. The need of adding potassium iodide to liberate Iodine in the reacting mixture. Any titration is carried out with hypo, the iodine must be in the reacting mixture and starch used as a indicator to predict the end point. Hence this type of titration is known as iodometric titration. Starch is added towards the endpoint to avoid the formation of solid starch iodide complex which cannot decompose during the titration. Potassium dichromate is a good oxidizing agent and its equivalent weight is 49. It liberates nascent oxygen in presence of acid.



Nascent oxygen is the responsible to oxidize the potassium iodide to iodine.

**Procedure:****a) Preparation of standard potassium dichromate solution.**

Weigh about 0.5 g of potassium dichromate crystals accurately using an electronic balance. Transfer the crystals of potassium dichromate into a cleaned 250 ml standard flask using a glass funnel. Dissolve the crystals by adding distilled water and make up the solution to the mark by adding distilled water. Mix well for uniform concentration and calculate the normality of potassium dichromate solution.

Note: Equivalent weight of hypo = 248

1000 ml of 1 N of  $K_2Cr_2O_7$  = 1000 ml of 1 N of hypo = one eq. wt of hypo

$$\therefore (N \times V) \text{ hypo} = (N \times V) K_2Cr_2O_7$$

$$N \text{ hypo} = \frac{(N \times V) K_2Cr_2O_7}{V \text{ hypo}} =$$

$$N \text{ hypo} = \dots\dots\dots N$$

$\therefore$  Weight per liter of hypo = N hypo x eq. wt of hypo

$$= \dots\dots\dots \times 248 = \dots\dots\dots \text{gms}$$

Weight per 250ml of hypo = Weight per liter of hypo /4

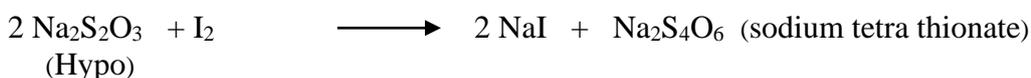
$$= \dots\dots\dots / 4$$

$\therefore$  Weight per 250ml of hypo = ----- gms

## II. Estimation of hypo

Pipette out 25 ml of the standard potassium dichromate solution into a cleaned 250 ml conical flask.  $\frac{1}{4}$  t.t concentrated hydrochloric acid and 10 ml of 10% potassium iodide solution. Shake well and add one t.t of distilled water. Titrate the liberated iodine against hypo solution taken in a burette using starch as indicator towards the end point. (Add starch when yellow colour solution changes to greenish yellow colour). Disappearance of blue colour is the end point. Note down the volume of hypo added and repeat the experiment for concordant values. Calculate the normality, weight per 250 ml and weight per litre of hypo.

### Reactions:



**Applications:** Hypo acts as a good reducing agent and it is used as medicine for skin diseases. It is also used to the estimation of iodine in the solution, copper in brass, arsenic in sodium arsenate, manganese dioxide in pyrolysite ore and available chlorine in bleaching power etc.

**Observations and calculations:****I. Preparation of Mohr's salt or FAS solution:**

Weight of weighing bottle + FAS crystals  $w_1 =$  \_\_\_\_\_ g

Weight of empty weighing bottle  $w_2 =$  \_\_\_\_\_ g

$\therefore$  Weight of FAS crystals taken  $(w_1 - w_2) =$  \_\_\_\_\_ g

$$\text{Normality of FAS} = \frac{\text{Weight of FAS} \times 4}{\text{Equivalent weight of FAS}}$$

$$N_{\text{FAS}} = \frac{\text{_____} \times 4}{392} = \text{_____} \text{ (a) N}$$

**II. Estimation of COD or back titration**

Burette : Standard FAS solution

Conical flask : 25 ml of waste water +25 ml of standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution  
+ 10 ml of 1:1  $\text{H}_2\text{SO}_4$  + one or two boiling chips + reflux  
the mixture for about 20 minutes + wash the funnel with  
distilled  $\text{H}_2\text{O}$  + cool the solution to room temperature.

Indicator : 4-6 drops of ferroin indicator

End point : Blue green to reddish brown colour

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of FAS run down in mls			

$\therefore$  Volume of FAS solution required for back titration =  $V_1 =$  \_\_\_\_\_ ml

**III. Blank titration or (FAS > <  $\text{K}_2\text{Cr}_2\text{O}_7$ )**

Burette : Standard FAS solution

Conical flask : 25 ml of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution + 10 ml of 1:1  $\text{H}_2\text{SO}_4$

Indicator : 4-6 drops of ferroin indicator

End Point : Blue green to reddish brown color

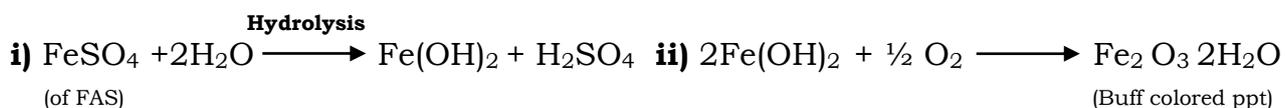
**Expt. No. : 2**

**Date:**

**Determination of chemical oxygen demand of the industrial waste water.**

**Theory:**

Mohr's salt is a double salt containing ferrous sulphate and ammonium sulphate  $[\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}]$ . It is a good reducing agent in presence of acid. Mohr's salt solution is prepared by adding two t. t full of dilute sulphuric acid and little water. The need of adding dilute  $\text{H}_2\text{SO}_4$  in order to prevent hydrolysis of Mohr's salt.



The chemical oxygen demand (COD) test is extensively employed for the measurement of the pollution strength of industrial wastes. Chemical oxygen demand is a measure of the total quantity of oxygen required for oxidation of organic and inorganic compounds of waste water to carbon dioxide and water by a strong oxidizing agent. This parameter is particularly valuable in surveys designed to determine the control losses to sewer systems. Results may be obtained within a relatively short time and measures taken to correct error on the day they occur.

Waste water contains organic impurities which include straight chain aliphatic and aromatic compounds, such as alcohols, acids, amines, pyridine and other oxidisable materials. They are oxidizing more effectively when silver sulphate is added as a catalyst. But silver sulphate reacts with chlorides in the waste water to form precipitates which are oxidized partially by this procedure. This difficulty is overcome by adding mercuric sulphate to the sample.

**Ill effects of waste water:** Acidity and basicity of soil increases and hence fertility of soil decreases.  $\therefore$  It is not used in agriculture. Waste water is not used for drinking and washing purpose. It is not used in industries to generate steam.

**Procedure:**

**I. Preparation of standard Mohr's salt or FAS solution.**

Weigh about 2.5 g of Mohr's salt crystals accurately using an electronic balance transfer the crystals into a clean 100 ml beaker. Dissolve the crystals by adding two t.t. of dilute sulphuric acid and little distilled water. Transfer the solution of Mohr's salt into a cleaned 250ml standard flask and makes it up to the mark by

adding distilled water. Mix well for uniform concentration and calculate the normality of Mohr's salt solution.

∴ Volume of FAS solution required for blank titration:

$$V_2 = \text{Final B.R} - \text{Initial B.R} = \text{-----} - \text{-----} = \text{-----} \text{ ml}$$

∴ Volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  required for the oxidation of organic compounds of waste water =  $V_2 - V_1 = \text{-----} - \text{-----} = \text{-----} \text{ ml}$

1000 ml of 1 M of FAS = 1000 ml of 1M  $\text{K}_2\text{Cr}_2\text{O}_7$  solution = 6x8 g of oxygen  
[If Molar solution used, use this relation to calculate COD of waste water]

**OR**

1000 ml of 1 N of FAS = 1000 ml of 1N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution = 8 g of oxygen  
[If Normality solution used, use this relation to calculate COD of waste water]

$$V_2 - V_1 \text{ ml of (a) N FAS} = X = \frac{8x(a) x V_2 - V_1}{1000} =$$

$$= \frac{8 x}{1000} = \text{-----} \text{ g of oxygen}$$

∴ Amount of  $\text{O}_2$  required to oxidize organic compounds present in 25 ml of waste water =  $X = \text{-----} \text{ g}$

∴ Amount of  $\text{O}_2$  required to oxidize organic compounds present in 1000ml of waste water

$$Y = \frac{X x 1000}{25} = \frac{x 1000}{25} = \text{-----} \text{ g}$$

$$\therefore \text{COD of waste water} = Y x 1000 = \text{-----} x 1000$$

$$\therefore \text{COD of waste water} = \text{-----} \text{ mg /dm}^3 \text{ or ppm.}$$

**COD:** The amount of oxygen is required to oxidize both organic & inorganic compounds present in the wastewater using strong oxidizing agent for a period of 1½ hours is called COD.

**BOD:** The amount of oxygen is required to oxidize only biologically oxidisable organic compounds present in the wastewater using mild oxidizing agent for a period of 5 days is called BOD.

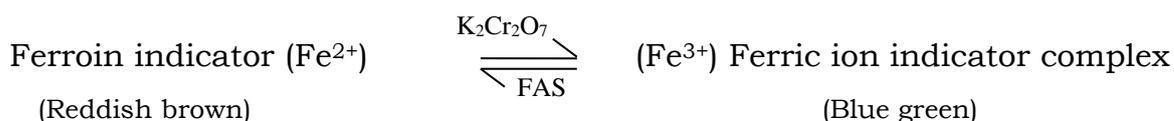
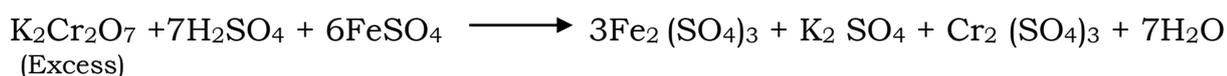
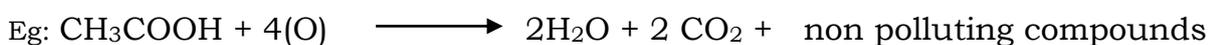
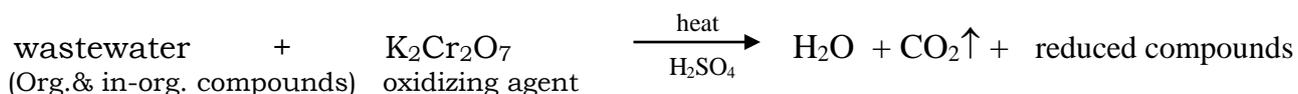
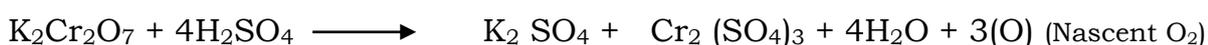
## II. Determination of COD of waste water or back titration

Pipette out 25 ml of the industrial wastewater into a clean conical flask and add 25ml of standard potassium dichromate solution (0.025N). Add 10 ml of 1:1 sulphuric acid containing silver sulphate and one or two pieces of boiling chips. Cover the mouth of the conical flask with a glass funnel and reflux the mixture for about 20 minutes. Wash the glass funnel with little distilled water and cool the mixture to room temperature. Add 4 - 6 drops of ferroin indicator and titrate against standard Mohr's salt solution taken in a burette until blue-green color changes to reddish-brown. Note down the volume of Mohr's salt solution added for un-reacted potassium dichromate solution ( $V_1$ ) and repeat the experiment to get concordant values. Using blank titre values determine the volume of potassium dichromate required to oxidize organic compounds and calculate the COD of the given industrial waste water.

### I. Blank titration or Mohr's salt > < $K_2Cr_2O_7$

Pipette out 25ml of standard potassium dichromate solution into a clean conical flask and add 10ml of 1:1 sulphuric acid containing silver sulphate. Add 4-6 drops of ferroin indicator and titration this against Mohr's salt solution taken in a burette until blue-green color changes to reddish brown. Note down the volume of Mohr's salt solution required for blank titration ( $V_2$ ).

#### Reactions:



**Applications:** From COD experiment to measure pollution strength and to prevent the pollution of waste water.

### Observations and calculations:

#### I . Preparation of brass solution:

Weight of brass foils taken = W = \_\_\_\_\_ g

#### II . Estimation of copper

Burette : Standard hypo solution

Conical flask : 25 ml brass solution + NH<sub>4</sub>OH drop wise till a bluish white ppt. is formed. Dissolve the ppt. by adding dilute acetic acid + ¼ th t.t. of acetic acid in excess + one t.t. of 10% KI + shake well + one t.t. of distilled water.

Indicator : 2 ml of freshly prepared starch.

End point : Disappearance of blue colour

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of hypo run down in mls			

∴ Volume of hypo required = V = \_\_\_\_\_ ml

Note: 1. Normality of hypo = .....(a) N

2. One gm equivalent weight of copper = 63.54

∴ 1000 ml of 1 N of hypo = one gm equivalent weight of copper ( 63.54 )

$$\therefore V \text{ ml of } (a) \text{ N hypo} = X = \frac{Vx(a)Nx63.54}{1000} = \frac{63.54 x}{1000}$$

$$X = \text{_____ g of copper}$$

∴ Weight of copper present in 25 ml of brass solution = X = \_\_\_\_\_ g

**Expt. No. : 3****Date:****Determination of percentage of copper in brass****Theory :**

The chief constituents of brass alloy are 50 – 90 % copper and 20 – 40 % zinc. It also contains small quantities of tin, lead and iron. Brass is used for making statues, utensils, conducting wire etc., A solution of brass is made by dissolving the sample in minimum amount of nitric acid. The dissolved oxides of nitrogen in the solution are expelled by boiling with urea. The solution is neutralised by ammonium hydroxide and made in to slight acidic medium with acetic acid. Potassium iodide solution is added and the cupric ion present in the brass solution oxidise potassium iodide to iodine. The iodine liberated is titrated against sodium thiosulphate using starch as indicator. The volume of sodium thiosulphate consumed is measure of the amount of copper present in the solution and sodium thiosulphate is commonly known as hypo.

**Procedure:****I. Preparation of brass solution:**

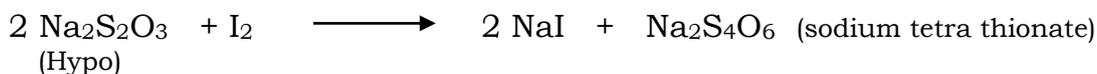
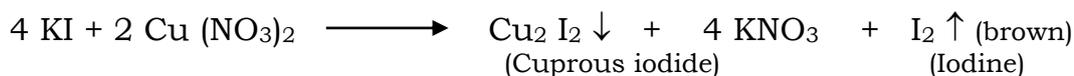
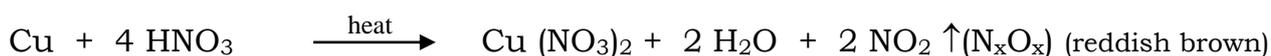
Weigh about 0.5 g of the given brass pieces accurately using an electronic balance and transfer it into a clean conical flask. Add two t.t. of 1:1 nitric acid and cover the mouth of the flask with a glass funnel. Warm gently to dissolve the brass piece completely and add two t.t of distilled water. Add about two gms of urea and continue the heating until the reddish brown fumes of oxides of nitrogen are expelled completely. Cool the solution to room temperature and transfer the solution of brass in to a clean 250 ml standard flask. Make up the solution to the mark by adding distilled water and mix well for uniform concentration.



## II. Estimation of copper:

Pipette out 25 ml of brass solution into a clean conical flask and add dilute ammonium hydroxide drop by drop until bluish white precipitate is formed. Dissolve the ppt. by adding dilute acetic acid drop by drop and then add  $\frac{1}{4}$  t.t. of acetic acid in excess. Add one t.t. of 10% potassium iodide solution and shake well. Titrate the liberated iodine against standard sodium thiosulphate solution taken in a burette using starch as indicator towards the end point. (Add starch when brown solution changes to whitish yellow). Disappearance of blue colour is the end point. Note down the volume of hypo added and repeat the experiment for concordant values. Calculate the percentage of copper in the given sample of brass.

### Reactions:



Some important copper alloys and their applications are:

Alloys	Compositions	Applications
<b>Brass</b>	<b>80% Cu &amp; 20%Zn</b>	<b>Statues, Utensils, Conducting wire, coins etc;</b>
Bell Metal	80% Cu and 20% Sn	Utensils, cast of bells, functional bells for producing sounds.
Gun metal	88% Cu, 10% Tn and 2% Zn	Foundry works, Hydraulic fittings, Heavy load bearings, Steam plants, water fittings, Marline pumps.
Stainless steel	50% Fe, 30% Cr and 20% Ni	Chemical equipments, Automobile parts, House hold utensils, Decorative pieces, Sinks, Surgical instruments.
Gold	90% Au & 10% Cu	Ornamental, In ayurvedic treatments.
German silver	50% Cu, 20%Zn and 25%Ni	Utensils, Table wares, Bolts, Screws, Coins, Ornamentals.

### Observations and calculations:

#### I. Preparation of standard potassium dichromate solution:

Weight of weighing bottle +  $K_2Cr_2O_7$  crystals  $w_1 = \text{----- g}$

Weight of empty weighing bottle  $w_2 = \text{----- g}$

$\therefore$  Weight of  $K_2Cr_2O_7$  crystals taken  $(w_1 - w_2) = \text{----- g}$

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{Weight of } K_2Cr_2O_7 \times 4}{\text{Equivalent weight of } K_2Cr_2O_7}$$

$$N \text{ } K_2Cr_2O_7 = \frac{\text{-----} \times 4}{49} = \text{----- (a) N}$$

#### II . Estimation of iron

Burette : Standard  $K_2Cr_2O_7$  solution

Conical flask : 25 ml of haematite ore solution +  $\frac{1}{4}$  t.t concentrated HCl  
+ heat +  $SnCl_2$  drop wise till yellow colour changes to  
colorless + 5.6 drops of  $SnCl_2$  in excess + cool the  
solution to room temperature + 10 ml of saturated  $HgCl_2$   
+ shake well + one t.t. of distilled  $H_2O$

Indicator : potassium ferricyanide as external indicator

End point : Failure to observe blue colour when a drop of reacting  
mixture is brought in contact with the indicator placed  
on paraffined paper

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of $K_2Cr_2O_7$ run down in mls			

$\therefore$  Volume of  $K_2Cr_2O_7$  required =  $V = \text{----- ml}$

**Expt. No. :4****Date:****Determination of iron in a given sample of haematite ore solution****Theory:**

Haematite is an important ore of iron containing mainly, ferric oxide:  $\text{Fe}_2\text{O}_3$  and small amount of silica ( $\text{SiO}_2$ ). Some other iron ores are: Limonite:  $\text{Fe}_2\text{O}_3 \cdot x \text{H}_2\text{O}$ , Magnetite:  $\text{Fe}_3\text{O}_4$ , Siderite:  $\text{FeCO}_3$ , Pyrites:  $\text{FeS}_2$ , etc., A known weight of the ore is digested with hydrochloric acid and the insoluble residue mainly containing silica is removed by filtration. The filtrate is diluted to 250 ml and is supplied for the estimation. Iron is present in the solution as  $\text{Fe}^{3+}$  (ferric) ions and it is reduced to  $\text{Fe}^{2+}$  (ferrous) ions using stannous chloride ( $\text{SnCl}_2$ ) in hot condition in presence of concentrated HCl. The excess of stannous chloride is oxidized by adding with mercuric chloride solution. The ferrous ions in the resulting solution are titrated against standard potassium dichromate using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense deep blue colour with ferrous ions due to the formation of ferriferrocyanide complex. Iron is extracted from ore by metallurgical process.

**Procedure:****a) Preparation of standard solution of potassium dichromate**

Weigh about 0.5 g of potassium dichromate crystals accurately using an electronic balance. Transfer the crystals of potassium dichromate into a clean 250ml standard flask using a glass funnel. Dissolve the crystals by adding distilled water and make up the solution to the mark by adding distilled water. Mix well for uniform concentration and calculate the normality of potassium dichromate solution.

**b) Estimation of Iron**

Pipette out 25ml of the haematite ore solution into a clean conical flask. Add  $\frac{1}{4}$  t.t of concentrated hydrochloric acid and heat the solution nearly to boiling. Add stannous chloride drop by drop taken in a separate burette till yellow colour solution becomes colourless and then add 6-8 drops of stannous chloride in excess. Cool the solution to lab temperature and add 10ml of saturated mercuric chloride at once. Shake well and silky white precipitate is formed. (If no precipitated or black precipitated is formed, reject and repeat the experiment).

Note: 1. Weight of haematite ore in 250ml =  $W = \text{----- g}$

2. Equivalent weight of iron = 55.85

1000 ml of 1 N of  $K_2Cr_2O_7$  = one gm equivalent weight of iron ( 55.85 )

$$\therefore V \text{ ml of (a) N } K_2Cr_2O_7 = X = \frac{55.85 \times (a) \times V}{1000}$$

$$X = \frac{55.85 \times}{1000} = \text{----- g of iron}$$

$\therefore$  Weight of iron present in 25 ml of haematite solution =  $X = \text{----- g}$

$\therefore$  Weight of iron present in 250 ml of haematite solution

$$Y = \frac{X \times 250}{25} = \frac{\text{-----} \times 250}{25} = \text{----- g}$$

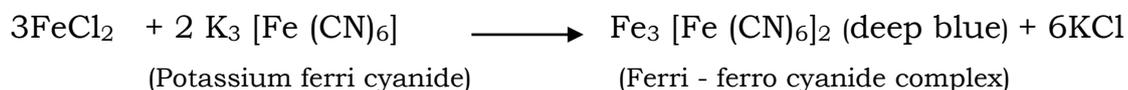
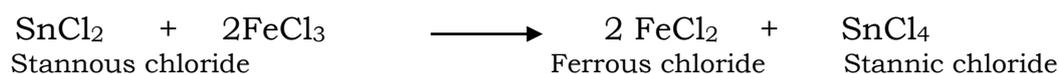
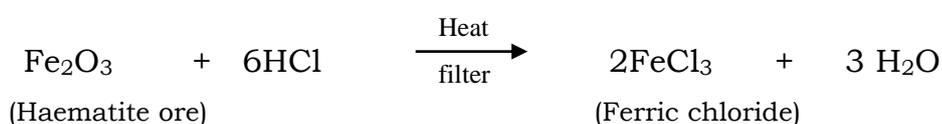
$\therefore$  Percentage of iron in the given haematite solution,

$$= \frac{Y \times 100}{\text{Weight of haematite ore in 250 ml (W)}} = \frac{\text{-----} \times 100}{\text{-----}}$$

Percentage of iron in the given haematite solution =  $\text{----- \%}$

Add one t.t. of distilled water and titrate this against standard potassium dichromate solution taken in a burette, using potassium ferri-cyanide as external indicator. The end point is failure to observe blue colour when one drop of the titrated mixture is brought in contact with a drop of the indicator placed on paraffin paper. Note down the volume of potassium dichromate added and repeat the experiment for concordant values. Calculate the percentage of iron in the given sample of haematite ore solution.

**Reactions:**



**Applications:** Iron used for the construction of building, tanks, roads, reservoirs, bridges, alloys, tunnels, dams, etc.,

## Observations and calculations

### I. Preparation of standard solution of disodium salt of EDTA

Weight of weighing bottle + EDTA salt  $w_1 =$  \_\_\_\_\_ g

Weight of empty weighing bottle  $w_2 =$  \_\_\_\_\_ g

$\therefore$  Weight of EDTA salt taken  $(w_1 - w_2) =$  \_\_\_\_\_ g

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA Salt}}{\text{Molecular weight of EDTA}} \times 10$$

$$M_{\text{EDTA}} = \frac{\quad \times 10}{372.24} = \text{_____ (a) M}$$

### II . Determination of total hardness of water

Burette : Standard EDTA solution

Conical flask : 25 ml of hard water + 5ml of  $\text{NH}_4\text{OH} - \text{NH}_4\text{Cl}$  buffer  
+ one t.t of distilled water

Indicator : Pinch of Eriochrome black – T

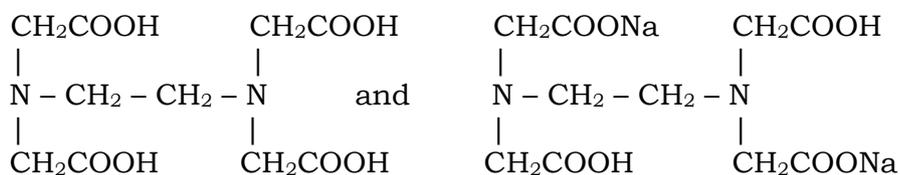
End point : Wine red to clear blue color

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of EDTA run down in mls			

$\therefore$  Volume of EDTA required =  $V =$  \_\_\_\_\_ ml

**Expt. No: 5****Date:****Determination of total hardness of water****Theory:**

Hardness of water is due to the presence of dissolved salts of calcium and magnesium. The total hardness of water is determined using ethylene di-amine tetra acetic acid (EDTA) which form complex with  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions. The EDTA molecule has two easily replaceable hydrogen atoms and hence the EDTA form complex with metal ion present in water. Reaction can be carried out quantitatively at a pH of 10 using Eriochrome black-T as indicator. Since the reaction involves the liberation of  $\text{H}^+$  ions, a buffer mixture has to be used to maintain a pH of 10. The buffer mixture used in the titration is ammonium hydroxide and ammonium chloride. The total hardness of the water is usually expressed in terms of ppm (i.e., parts per million) of  $\text{CaCO}_3$ . EDTA is sparingly soluble in water and hence disodium salt of EDTA is used as reagent for preparing the solution. The structures of EDTA ( $\text{H}_4\text{Y}$ ) and disodium salt of EDTA ( $\text{Na}_2\text{H}_2\text{Y}$ ) are given below.

**EDTA ( $\text{H}_4\text{Y}$ )****Disodium salt of EDTA ( $\text{Na}_2\text{H}_2\text{Y}$ )****Procedure:****1. Preparation of standard solution of disodium salt of EDTA:**

Weigh about 0.5 g of di-sodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA in to a cleaned 100 ml standard flask using a glass funnel. Dissolve the crystals by adding  $\frac{1}{2}$  t.t. of ammonium hydroxide and little distilled water. Make up the solution to the mark by adding distilled water and mix well for uniform concentration. Calculate the molarity of EDTA solution.

Note: Molecular Weight of  $\text{CaCO}_3 = 100$

1000 ml of 1M of EDTA = one gm molecular weight of  $\text{CaCO}_3$  (100)

$$\therefore V \text{ ml of (a) } M_{\text{EDTA}} = X = \frac{V(a)100}{1000} = \underline{\hspace{2cm}}$$

$$\therefore V \text{ ml of (a) } M_{\text{EDTA}} = \underline{\hspace{1cm}} \text{ mg of } \text{CaCO}_3$$

$\therefore$  Weight of  $\text{CaCO}_3$  present in 25 ml of hard water =  $X = \underline{\hspace{1cm}}$  mg

$\therefore$  Weight of  $\text{CaCO}_3$  present in one million or  $10^6$  ml of hard water

$$Y = \frac{X \times 10^6}{25} = \frac{\hspace{1cm} \times 10^6}{25} = \underline{\hspace{1cm}} \text{ mg}$$

Thus total hardness of water in terms of ppm =  $Y = \underline{\hspace{1cm}}$  ppm

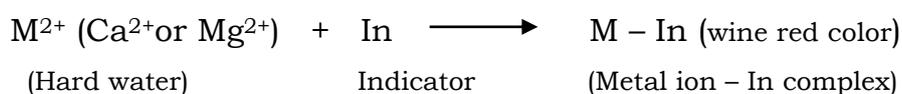
**Note:** Parts per million: ppm: - is the parts of  $\text{CaCO}_3$  equivalent hardness present per million or  $10^6$  parts of water.

**i e:** 500 ppm = 500 part of  $\text{CaCO}_3$  equivalent hardness present per million or  $10^6$  parts of water.

## II. Determination of total hardness of water

Pipette out 25ml of the given water sample into a cleaned conical flask and add 5 ml of ammonium hydroxide and ammonium chloride buffer solution. Add a pinch of Eriochrome black-T indicator and one t.t. of distilled water. Titrate this against EDTA solution taken in a burette until the wine red colour changes to purple blue. Note down the volume of EDTA added and repeat the experiment for concordant values. Calculate the total hardness of water in terms of ppm.

### Reactions:



### Note:

1. Rinse the Burette, Pipette, Measuring flask and conical flask using 5.0 ml of distilled water only.
2. Avoid wastage of water and conserve it for future generation, because it is life saving compound
3. Water is universal solvent.
4. Water is liquid because of hydrogen bonding.
5. No life without water.
6. Do not pollute the water.

## Observations and calculations

### I. Preparation of standard solution of disodium salt of EDTA

Weight of weighing bottle + EDTA salt  $w_1 =$  \_\_\_\_\_ g.

Weight of empty weighing bottle  $w_2 =$  \_\_\_\_\_ g

∴ Weight of EDTA salt taken  $(w_1 - w_2) =$  \_\_\_\_\_ g

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA salt}}{\text{Molecular weight of EDTA}} \times 10$$

$$M_{\text{EDTA}} = \frac{\quad \times 10}{372.24} = \text{_____ (a) M}$$

### II . Estimation of CaO in cement solution

Burette : Standard EDTA solution

Conical flask : 25 ml of cement solution + 5ml of 1:1 glycerol + 5ml of di-ethyl-amine + 10 ml of 2N NaOH + 1 t.t of distilled water and shake well

Indicator : Pinch of Pattern - Reeder's indicator

End point : Wine red to clear blue color

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of EDTA run down in mls			

∴ Volume of EDTA required =  $V =$  \_\_\_\_\_ ml

**Expt. No. : 6****Date:****Determination of calcium oxide in cement solution by rapid EDTA method.****Theory:**

Cement contains oxides of calcium, aluminum, magnesium, iron and small amount of silica. Calcium oxide is a prime constituent of cement and the general composition of Portland cement is given below.

CaO = 60 – 66%, SiO<sub>2</sub> = 17 – 25%, Al<sub>2</sub>O<sub>3</sub> = 3 – 8%, Fe<sub>2</sub>O<sub>3</sub> = 2 – 6%, MgO = 0.1 - 5.5%, SO<sub>3</sub> = 1 – 3 % and Na<sub>2</sub>O and K<sub>2</sub>O = 0.5 – 1.5%

In the estimation of calcium in cement, the given cement sample is treated with concentrated hydrochloric acid. The insoluble silica is filtered off and the filtrate which contains calcium ions is titrated against EDTA at a pH of 12 – 14 using Pattern-Reeder's indicator. The latter is used in this titration because Eriochrome black – T indicator forms a very weak complex with calcium ions. Diethylamine is added to maintain a pH of about 12.5 and glycerol is added to get a sharp end point. This method facilitates the determination of calcium only although Mg<sup>+2</sup> ions are present in the cement solution which can readily form complexes with EDTA (i.e., Mg<sup>+2</sup> ions react with NaOH gives magnesium hydroxide which is stable white precipitated and do not react with EDTA in the above conditions)

**Procedure:****I. Preparation of standard solution of disodium salt of EDTA.**

Weigh about 0.5 g of disodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA into a clean 100 ml standard flask using a glass funnel. Dissolve the crystals by adding ½ t.t. of ammonium hydroxide and little distilled water. Make up the solution to the mark by adding distilled water and mix well for uniform concentration. Calculate the molarity of EDTA solution.

Note: 1. Weight of cement in 250 ml = W = \_\_\_\_\_ g

2. Molecular weight of CaO = 56.08

1000 ml of 1M of EDTA = one gm molecular weight of CaO (56.08)

$$\therefore V \text{ ml of } (a) \text{ M EDTA} = X = \frac{56.08 \times (a) \times V}{1000} = \frac{56.08 \times}{1000}$$

$$X = \text{_____ g of CaO}$$

\(\therefore\) Weight of CaO present in 25 ml of cement solution = X = \_\_\_\_\_ g

$$\therefore \text{Weight of CaO present in 250 ml of cement solution} = Y = \frac{250 \times}{25}$$

$$Y = \text{_____ g}$$

$$\therefore \text{Percentage of CaO in the cement solution} = \frac{Y \times 100}{\text{Weight of cement in 250 ml (W)}}$$

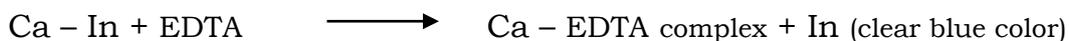
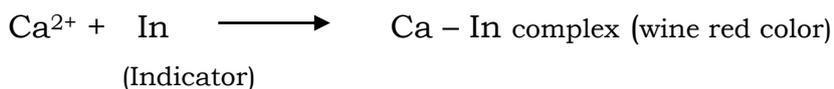
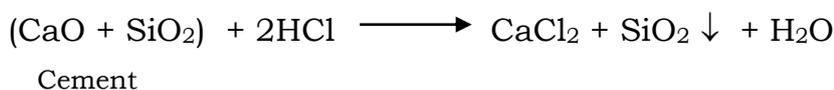
$$= \frac{\quad \times 100}{\quad}$$

\(\therefore\) Percentage of CaO in the cement solution = \_\_\_\_\_ %

## II. Estimation of calcium oxide

Pipette out 25ml of the given cement solution into a cleaned conical flask. Add 5ml of 1:1 glycerol, 5ml of diethyl amine and 10ml of 2N sodium hydroxide. Add a pinch of Pattern- Reeder's indicator and one t.t of distilled water. Shake well and titrate this against EDTA solution taken in a burette until wine red color changes to purple blue. Note down the volume of EDTA added and repeat the experiment for concordant values. Calculate the percentage of calcium oxide in the given cement solution.

### Reactions:



**Applications:** Cement is used for construction of buildings, tunnels, dams, roads, reservoirs. etc.,

### Observation and calculations

#### Estimation of alkalinity:

Burette : Standard H<sub>2</sub>SO<sub>4</sub>

Conical flask : 25 ml sample water

Indicator : First add Phenolphthalein (P) and add Methyl orange (M)

End Point : First disappearance of pink color and then orange yellow to red

Burette reading	1		2		3	
	Up to P	Up to M	Up to P	Up to M	Up to P	Up to M
Final Burette reading						
Initial Burette reading						
Volume of acid Run down (ml)	V <sub>1</sub> =	V <sub>2</sub> =	V <sub>1</sub> =	V <sub>2</sub> =	V <sub>1</sub> =	V <sub>2</sub> =

Volume of water sample taken = 25 ml

N H<sub>2</sub>SO<sub>4</sub> = \_\_\_\_\_ N

Vol. of acid consumed for Phenolphthalein end point = V<sub>1</sub> ml = \_\_\_\_\_ ml

Volume of acid used for methyl orange end point ( V<sub>2</sub> - V<sub>1</sub> ) ml = \_\_\_\_\_ ml

1000 ml of 1N H<sub>2</sub>SO<sub>4</sub> = 1gm Eq. wt of CaCO<sub>3</sub> = 50 g of CaCO<sub>3</sub>

$$\therefore V_1 \text{ ml of } N \text{ H}_2\text{SO}_4 = \frac{V_1 \times N \text{H}_2\text{SO}_4 \times 50}{1000} \quad X = = \frac{50x}{1000} \text{-----}$$

$$X = \text{-----} \text{ g of CaCO}_3$$

$\therefore$  Phenolphthalein alkalinity in 25 ml of water = X = \_\_\_\_\_ g

$\therefore$  Phenolphthalein alkalinity in ppm or 10<sup>6</sup> ml of H<sub>2</sub>O = P =  $\frac{X \times 10^6}{25}$  =

Phenolphthalein alkalinity in ppm or 10<sup>6</sup> ml of H<sub>2</sub>O = P = \_\_\_\_\_ ppm

## Determination of total alkalinity of given water sample

**Expt No.**

**Date:**

### Theory :

The alkalinity of water is due to the presence of those substances in which water have tendency to increase the concentration of  $\text{OH}^-$  ions either by hydrolysis or dissociation in water.

The salts of weak organic acids undergo hydrolysis and consume  $\text{H}^+$  ions of . As a result concentration of  $\text{OH}^-$  ions increases in water and it become alkaline. Also alkalinity is due to the presence of  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$ ,  $\text{OH}^-$  from  $\text{NaOH}$  and  $\text{KOH}$ ,  $\text{CO}_3^{2-}$  from  $\text{Na}_2\text{CO}_3$  &  $\text{K}_2\text{CO}_3$  and  $\text{HCO}_3^-$  from  $\text{NaHCO}_3$  and  $\text{KHCO}_3$  or  $\text{Ca}(\text{HCO}_3)_2$  and  $\text{Mg}(\text{HCO}_3)_2$  are responsible for alkalinity

The following reaction were taken place when titrated with acid



The titrations of water with standard acid up to Phenolphthalein end point make the completion of (1) and (2) reaction. The amount of acid consumed corresponds to complete neutralization of  $\text{OH}^-$  and half neutralization of  $\text{CO}_3^{2-}$  i.e., neutralization  $\text{CO}_3^{2-}$  up to  $\text{HCO}_3^-$  ions.

Titration of water sample with the same acid up to methyl orange endpoint marks the completion of all the three reactions. Hence the amount of acid used after the Phenolphthalein endpoint corresponds to half neutralization of  $\text{CO}_3^{2-}$  and full neutralization of  $\text{HCO}_3^-$  present in the water sample.

### Procedure:

Pipette out 25 ml of water sample in a conical flask and add 3-4 drops of Phenolphthalein indicator. Titrate this against standard sulphuric acid solution taken in a burette till disappears of red colour. Note down the volume of sulphuric acid solution added ( $V_1$ ). Then add 2-3 drops of Methyl orange indicator to the same solution and continue the titration against the same sulphuric acid solution till orange yellow colour just changes to orange red colour. Note down the volume of sulphuric acid solution added ( $V_2$ ). Repeat the experiment for concordant values.

1000 ml of 1N H<sub>2</sub>SO<sub>4</sub> = 1gm Eq. wt of CaCO<sub>3</sub> = 50 g of CaCO<sub>3</sub>

$$\therefore (V_2 - V_1) \text{ ml of } N \text{ H}_2\text{SO}_4 = \frac{V_2 - V_1 \times N \times \text{Eq. wt of CaCO}_3}{1000} \quad Y = \frac{50x}{1000} \text{ -----}$$

$$Y = \text{-----} \text{ g of CaCO}_3$$

$\therefore$  Methyl Orange alkalinity in 25 ml of water = Y = ----- g

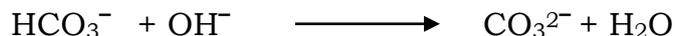
$$\therefore \text{Methyl Orange alkalinity in ppm or } 10^6 \text{ ml of H}_2\text{O} = M = \frac{Y \times 10^6}{25} =$$

Methyl Orange alkalinity in ppm or 10<sup>6</sup> ml of H<sub>2</sub>O = M = \_\_\_\_\_ ppm

$\therefore$  The total alkalinity of water sample = Alkalinity of (P+M) =

$$= \text{-----} \text{ ppm}$$

**Note:**  $\text{HCO}_3^-$  and  $\text{OH}^-$  do not exist simultaneously because they react to form  $\text{CO}_3^{2-}$  and  $\text{H}_2\text{O}$ .



### Identification of alkalinity of water

If  $P = 0$ , no hydroxide, no carbonate and alkalinity only bicarbonate.

If  $P = M$ , no carbonate, no bicarbonate and alkalinity only hydroxide.

If  $P = \frac{1}{2}M$ , no hydroxide, no bicarbonate and alkalinity only carbonate.

If  $P > \frac{1}{2}M$ , no bicarbonate, alkalinity only hydroxide and carbonate.

If  $P < \frac{1}{2}M$ , no hydroxide, alkalinity only carbonate and bicarbonate.

Alkalinity	$\text{OH}^-$ ppm	$\text{CO}_3^{2-}$ ppm	$\text{HCO}_3^-$ ppm	Total alkalinity
$P = 0$	0	0	M	M
$P = M$	M	0	0	P or M
$P = \frac{1}{2}M$	0	2P	0	2P
$P > \frac{1}{2}M$	$2P - M$	$2(M - P)$	0	$(2P - M) + 2(M - P)$
$P < \frac{1}{2}M$	0	2P	$M - 2P$	$2P + (M - 2P)$

**Result:** The total alkalinity of given water sample. =..... ppm.

**Applications:** Alkalinity test is used to measure carbonates, bicarbonates, hydroxyl ions in sea water, waste water, ponds, lake, swimming pools, spa etc., and alkalinity serves as an index of the admixture of river water with sea water. It helps to measurement of blood alkalinity.



# ***PART-B***



Instrumental

Methods of Analysis

## **PART – B**

### **Instrumental methods of chemical analysis**

#### **Instructions to the students**

- ☞ Read the procedure before commencement of experiment and do the experiment according to the procedure.
- ☞ Instruments used for Part-A experiments are:  
pH meters, Potentiometers, Colorimeters, Conductivity meters, Viscometers and Flame Photometers.
- ☞ Wash the micro burette, pipette and beaker with tap water and rinse with distilled water before commencement of the experiments.
- ☞ Wash the electrodes or cells with distilled water and wipe them with tissue paper before commencement of the experiments.
- ☞ Switch off the instruments after experiment is completed.
- ☞ Wash the electrodes or cells with distilled water and dip them in distilled water taken in a 100 ml beaker after the experiments is completed.
- ☞ Wash the micro burette, pipette, standard flask and beaker with tap water again after the experiment is completed and return them to the instructor.

**Note: Students are require to checkup all the glass wares and equipments before starting of the experiment. If any broken or damage bring the notice of concern teachers or lab instructors.**

**Observations and calculations**

Volume of NaOH added in ml	pH in moles per liters of H <sup>+</sup> ions	$\Delta\text{pH} / \Delta V$
0.0		-----
0.5		
1.0		
1.5		
2.0		
2.5		
3.0		
3.5		
4.0		
4.5		
5.0		
5.5		
6.0		
6.5		
7.0		
7.5		
8.0		
8.5		
9.0		
9.5		
10.0		
10.5		
11.0		
11.5		
12.0		
12.5		
13.0		
13.5		
14.0		

## Instrumental Methods of Chemical Analysis

**Expt. No. :7**

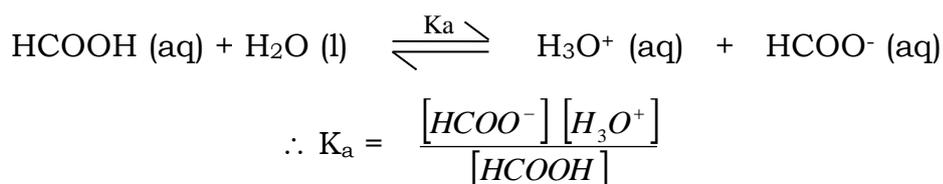
**Date:**

### Determination of pKa value of a weak acid using pH meter.

**Theory:**

The strength of an acid is experimentally measured by determining its equilibrium constant or disassociation constant ( $K_a$ ). Strong acids are completely ionized in aqueous solution and hence it is impossible to determine the disassociation constant of strong electrolytes. But in case of weak electrolytes are partially ionized in aqueous solution and hence the equilibrium constant of weak electrolytes can be experimentally determined.

Let us consider weak acid like formic acid, which ionizes, freely in aqueous solution as,



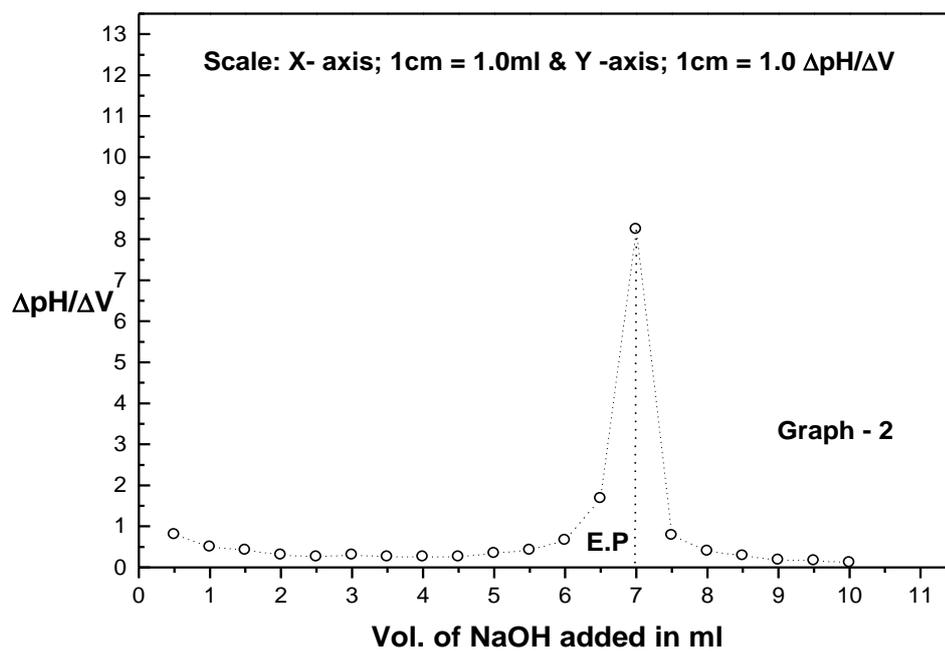
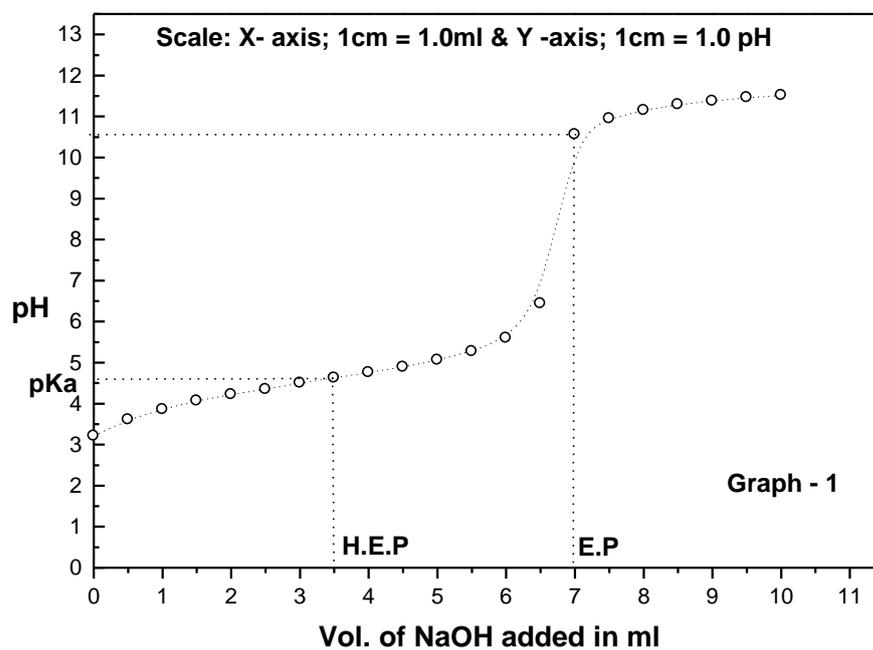
pKa is a modern method of expressing the strength of weak acid ,

$$\text{ie., } \text{pKa} = -\log_{10} K_a$$

pKa value is determined by measuring the changes in pH of acid solutions at different amount of the base added. In the titration of an acid with a base, the pH of the solution rises gradually at first and then more rapidly. At the equivalence point there is a very sharp increase in pH for a very small quantity of base added. Beyond the equivalence point, the pH increases slightly on addition of base. The titration curve is obtained by plotting changes in pH against different volume of the base added and from the graph, determined the equivalence point of an acid. The pKa value of an acid can be calculated using Henderson equation,

$$\text{pH} = \text{pKa} + \frac{0.0591}{n} \log_{10} \frac{[\text{Salt}]}{[\text{Acid}]}$$

At half equivalence point [salt] = [acid] and hence pH at half equivalence point gives the pKa value of weak acid, i.e. **pH = pKa.**



**Note:** Do not write the nature of the graphs in the record.

**Procedure:**

Pipette out 25 ml of weak acid in to a cleaned 100 ml beaker followed by adding two t.t. of distilled water and stir well using a glass rod.

Switch on the pH meter and immerse the combined electrode (glass + calomel) in to the acid taken in a beaker. Measure the pH of the acid by pressing 'READ' button only. Add 0.5 ml of sodium hydroxide solution at a time taken in a micro burette in to the acid, stir well and measure the pH of the acid and continue the addition of sodium hydroxide until the sudden raise in the pH of the acid occur. Then take few more readings and plot the graph, pH against the volume of sodium hydroxide added (graph No1). Take the difference in the pH of acid ( $\Delta\text{pH}$ ) and the volume of sodium hydroxide added ( $\Delta V$ ). Plot the graph,  $\Delta\text{pH}/\Delta V$  against the volume of sodium hydroxide added (graph No.2) and from the graph 2, determine the equivalence point of the acid. Determine the half equivalence point that gives the pKa value of weak acid by the graph 1.

**Apparatus used:** pH meter consists of combined electrode made up of glass and calomel electrode. Hence glass electrode is used as ion selective electrode for  $\text{H}^+$  ions and calomel electrode used as secondary reference electrode. Two electrodes are required for construction of a cell.

**Applications:** It is used to determination of strength of weak acid (pKa) , pH of solutions, Dissociation constant (Ka), neutralization point of acid,...etc;  
 $\text{pH} = -\log_{10}[\text{H}^+]$ . Range of pH Scale is 0 to 14.  $\text{pH} < 7$  , acidic pH ;  $\text{pH} > 7$ , basic pH and  $\text{pH} = 7$ , Neutral pH.

**Results:**

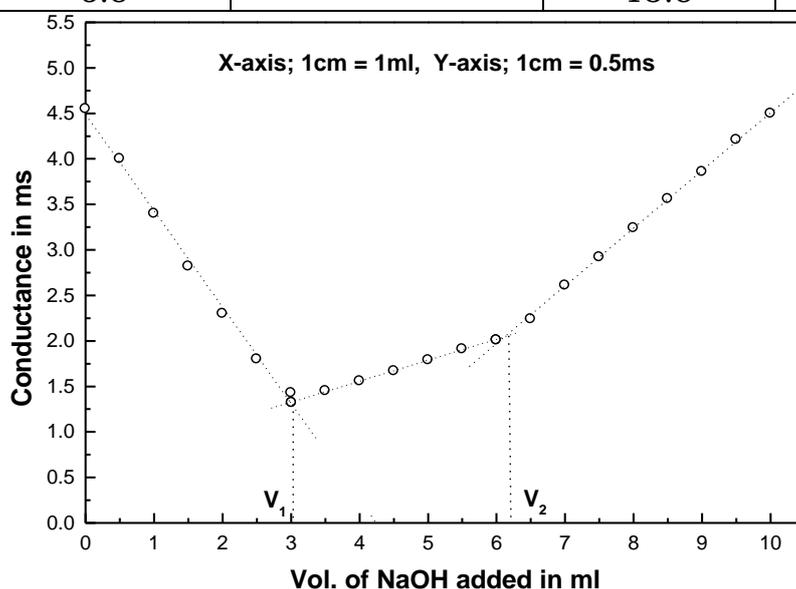
1. Equivalence Point = \_\_\_\_\_ml
2. Half Equivalence Point = \_\_\_\_\_ml
3. pH at Equivalence Point = \_\_\_\_\_ moles per liter of  $\text{H}^+$  ions
4. pH at Half Equivalence Point = \_\_\_\_\_ moles per liter of  $\text{H}^+$  ions
5. pKa of the given acid = \_\_\_\_\_

**Note:** i) Switch off the instrument after experiment is completed.

ii) Calibrate the pH meter using buffer solutions of 4 and 9 pH.

### Observations and calculations

Vol. of NaOH in ml	Conductance ohm <sup>-1</sup> or ms	Vol. of NaOH in ml	Conductance Ohm <sup>-1</sup> or ms
0.0		7.0	
0.5		7.5	
1.0		8.0	
1.5		8.5	
2.0		9.0	
2.5		9.5	
3.0		10.0	
3.5		10.5	
4.0		11.0	
4.5		11.5	
5.0		12.0	
5.5		12.5	
6.0		13.0	
6.5		13.5	



Volume of NaOH required to neutralize HCl,  $V_1 = \text{----- ml}$

$V_{\text{NaOH}}$  required to neutralize  $\text{CH}_3\text{COOH}$  ( $V_2 - V_1$ ) =  $\text{-----} = \text{----- ml}$

- Note:** 1. Do not write the nature of the graph in the record  
 2. Flat the graph at the center of graph sheet.

**Expt. No. : 8****Date:****Determination of Acid mixture (HCl + CH<sub>3</sub>COOH) by Conductometric Method****Theory:**

Measurement of conductance can be employed to determine, the end point in acid base titrations. In conduct metric titration's, there is a sudden change in conductance of the solution near the endpoint. The end point is determined graphically by plotting conductance in millisiemens (ms) against volume of sodium hydroxide added in ml.

Conductometric titration may be applied for the determination of acids present in a . In the titration of a mixture of a weak acid (acetic acid) and a strong acid (hydrochloric acid) with a strong base (sodium hydroxide), the conductance decreases upon adding sodium hydroxide to acid mixture at the initial stages owing to the replacement of highly mobile H<sup>+</sup> ions (mobility: **350 ms**) by the less mobile Na<sup>+</sup> and Cl<sup>-</sup> ions (mobility: **50 ms**). This trend continues till all the H<sup>+</sup> ions hydrochloric acid replaced (i.e., the strong acid is neutralized). Continued addition of sodium hydroxide raises the conductance moderately, as the weak acid; acetic acid is converted into its salt, sodium acetate. Further addition of sodium hydroxide raises the conductance due to the presence of free OH<sup>-</sup> ions (mobility: **200 ms**). The titration curves in the graph given depict the location of the equivalence points.

**Procedure:**

Make up the acid mixture given in a 100 ml standard flask to the mark by adding distilled water and mix well for uniform concentration. Pipette out 50 ml of the given acid mixture into a clean 100 ml beaker and stir well using a glass rod.

Dip the conductivity cell in the acid mixture and measure the conductance of the acid by using conductivity meter by pressing "**CAL**" button only. Add 0.5 ml of sodium hydroxide solution at a time taken in a burette. After each addition, stir the solution gently and note down the conductance. As the titration proceeds, the conductance first gradually decreases and then increases slowly and finally increases sharply. Plot the graph conductance versus volume of sodium hydroxide added. From the graph determined the volume of sodium hydroxide is required to neutralize hydrochloric acid [V<sub>1</sub>] and acetic acid [V<sub>2</sub> - V<sub>1</sub>] in an acid mixture.

Note:

1. Normality of NaOH solution = 0.2 N
2. Equivalent weight of HCl = 36.5

### I. Estimation of HCl

Volume of NaOH required to neutralize HCl =  $V_1 = \text{----- ml}$

$$\therefore \text{Normality HCl} = \frac{N_{\text{NaOH}} \times V_{\text{NaOH}}}{V_{\text{HCl}}} = \frac{0.2 \times \text{-----}}{25} = \text{----- N}$$

Weight per liter of HCl,  $X = N_{\text{HCl}} \times \text{equivalent weight of HCl}$

$$X = \text{-----} \times 36.5 = \text{----- g}$$

$$\text{Weight per 100 ml of HCl} = \frac{X}{10} = \frac{\text{-----}}{10} = \text{----- g}$$

### II. Estimation of CH<sub>3</sub>COOH

Volume of NaOH required neutralizing CH<sub>3</sub>COOH =  $V_2 - V_1 = \text{-----} = \text{---- ml}$

$$\therefore N \text{ of CH}_3\text{COOH} = \frac{N_{\text{NaOH}} \times (V_2 - V_1)_{\text{NaOH}}}{V_{\text{CH}_3\text{COOH}_3}} = \frac{0.2 \times \text{-----}}{25} = \text{-- N}$$

Weight per liter of CH<sub>3</sub>COOH =  $X = N_{\text{CH}_3\text{COOH}} \times \text{eq. weight of CH}_3\text{COOH}$

$$X = \text{-----} \times 60 = \text{----- g}$$

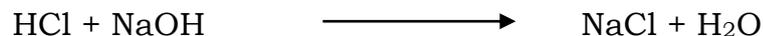
$$\text{Weight per 100 ml of CH}_3\text{COOH} = \frac{X}{10} = \frac{\text{-----}}{10} = \text{----- g}$$

### III. Estimation of acid mixture

$$\text{Normality of acid mixture} = \frac{N_{\text{NaOH}} \times V_{2 \text{ NaOH}}}{V_{\text{acid mixture}}} = \frac{0.2 \times \text{-----}}{50} = \text{-----}$$

$$\text{Normality of acid mixture} = \text{----- N}$$

Calculate the weight per liter and weight per 100 ml of hydrochloric acid and acetic acid respectively



**Apparatus used: Conductometer.**

**Conductance** =  $1 / \text{Resistance Ohm}^{-1}$       **or**

**Conductance** = Specific conductance / Cell constant  $\text{Ohm}^{-1}$  **or** ms

**Specific conductance** = Conductance x cell constant  $\text{Ohm}^{-1} \text{cm}^{-1}$

Conductivity cell made up of two Pt electrodes fixed at a distance '**l**' cm apart with area of cross section '**a**'  $\text{cm}^2$ .

**∴ Cell constant** =  $1/a \text{ cm}^{-1}$  or  $\text{m}^{-1}$

**Cell constant** = Specific conductance of 0.1N KCl / Conductance of 0.1N KCl  $\text{cm}^{-1}$

Hence cell constant is used for determination of conductance of solution.

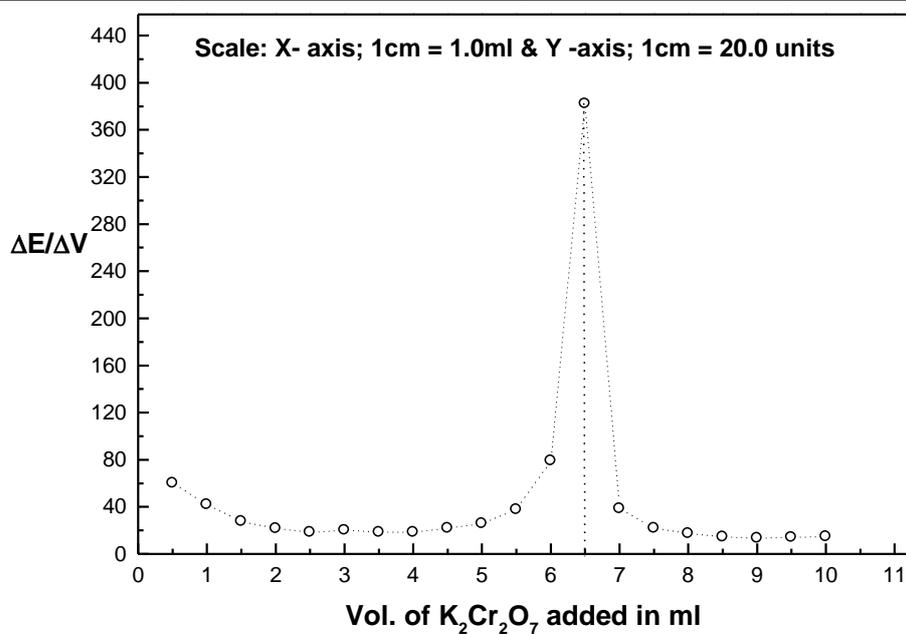
**Resistance** is the opposes the flow of current through the solution.

**Applications:** It is used to measure the conductance of polar solutions, conductivity of +ve and -ve ions, specific conductance, cell constant and strength of acid.

**Note:** Switch off the instrument after experiment is completed.

### Observations and calculations

Vol. of $K_2Cr_2O_7$ added in ml (V)	E.M.F (E) in mv	$\frac{\Delta E}{\Delta V}$	Vol. of $K_2 Cr_2 O_7$ added in ml (v)	E.M.F (E) in mv	$\frac{\Delta E}{\Delta V}$
0.0		-----	7.5		
0.5			8.0		
1.0			8.5		
1.5			9.0		
2.0			9.5		
2.5			10.0		
3.0			10.5		
3.5			11.0		
4.0			11.5		
4.5			12.0		
5.0			12.5		
5.5			13.0		
6.0			13.5		
6.5			14.0		
7.0			14.5		



$\therefore$  Volume of  $K_2Cr_2O_7$  required = V  $K_2Cr_2O_7$  =            ml

**Note:** Do not write the nature of the graph in the record.

**Expt. No. : 9****Date:****Potentiometric estimation of Mohr's salt or FAS.****Theory:**

When the electrode is dipped in the solution, the potential of an electrode depends on the concentration of the ions in the electrolyte. Therefore determination of maximum potential can serve as an indicator in potentiometric analysis. Determination of change of potential with concentration gives more accurate results than volumetric analysis.

When the given electrode is combined with saturated calomel electrode and dipped in the given electrolyte, an electro chemical cell is formed. The emf of this cell is measured using potentiometer. During the titration, the change in emf of the cell or concentration of ions in the electrolyte can be measured. Then  $\Delta E/\Delta V$  is plotted against the volume of the titrant, there will be a peak corresponding to the endpoint of the reaction.



$\therefore$  emf of the cell is calculated using Nernst's equation,

$$E_{\text{cell}} = E^{\circ}_{\text{cell}} - \frac{0.0591}{n} \log Q \quad \text{or} \quad E_{\text{cell}} = E^{\circ}_{\text{cell}} - \frac{0.0591}{n} \log \frac{[\text{Fe}^{3+}]}{[\text{Fe}^{2+}]}$$

Where  $E_{\text{cell}}$  = emf of the cell,  $E^{\circ}_{\text{cell}}$  standard emf of the cell,  $n$  = number of electrons involved in the reaction.

The potential of a cell after adding  $\text{K}_2\text{Cr}_2\text{O}_7$  due to the formation of  $\text{Fe}^{2+}/\text{Fe}^{3+}$  before the end point.

ie Cal. electrode /  $\text{Fe}^{2+}/\text{Fe}^{3+}$  / Pt. electrode, hence the formal potential of this cell is 0.7v.

After the end point, steep increase the emf of cell due to the formation of  $\text{Cr}_2\text{O}_7^{2-} / \text{Cr}^{3+}$  ions. ie Cal. electrode /  $\text{Cr}_2\text{O}_7^{2-} / \text{Cr}^{3+}$  / Pt. electrode. Hence the formal potential of this cell is 1.4v and thus double the emf of cell is take place.

**Note:**

$$1. \text{ Normality of } K_2Cr_2O_7 = N \text{ } K_2Cr_2O_7 = 0.1 \text{ N}$$

$$2. \text{ Equivalent weight of Mohr's salt} = 392$$

$$\therefore \text{ Normality of Mohr's salt solution} = \frac{NK_2Cr_2O_7 \times VK_2Cr_2O_7}{V \text{ Mohr's salt}}$$

$$N \text{ Mohr's salt} = \frac{\quad}{25} = \text{-----} \text{ N}$$

Weight per litre of Mohr's salt crystals,

$$X = N \text{ Mohr's salt} \times \text{equivalent weight of Mohr's salt}$$

$$X = \text{-----} \times 392 = \text{-----} \text{ g}$$

$$\text{Weight per 100 ml of Mohr's salt} = \frac{X}{10} = \frac{\quad}{10} = \text{-----} \text{ g}$$

**Procedure:**

Make up the Mohr's salt solution given in 100 ml standard flask to the mark by adding distilled water and mix well for uniform concentration. Pipette out 25 ml of Mohr's salt solution in to a clean 100 ml beaker followed by adding two t.t. of dilute sulphuric acid and stir well using a glass rod.

Switch on the potentiometer and immerse the Platinum & Calomel electrodes in to the Mohr's salt solution taken in a beaker. Measure the emf of the solution by pressing both **'READ'** and **mV** buttons and calibrate the instrument by adjusting the emf of 130 – 140 mV using **"CAL"** knob only. Add 0.5 ml of potassium dichromate solution at a time taken in a micro burette in to the Mohr's salt solution, stir well and measure the emf of the solution and continue the addition potassium dichromate added until the sudden raise in the emf of solution occur. Then take few more readings and take the difference in the emf of solution ( $\Delta E$ ) and the volume of potassium dichromate added ( $\Delta V$ ). Plot the graph,  $\Delta E/\Delta V$  against the volume of potassium dichromate added. From the graph, determine the volume of potassium dichromate required to rapid increase in the emf of the solution. Calculate the weight per liter and weight per 100 ml of Mohr's salt.



**Apparatus used:** Potentiometer is made up of Platinum and calomel electrodes. The Pt electrode is used to measure the emf of the cell and calomel electrode is used as secondary reference electrode.

**Applications:** It is used to measure the emf of the cell by using the emf, the strength or the concentration of the solution can be determined without using an indicator.

**Note:** Switch off the instrument after experiment is completed.

### Observations and calculations

Volume of ferric alum in ml	Concentration of ferric alum in mg	Concentration of iron in mg	Absorbance (A) or Optical density
5	$0.087 \times 5 = 0.435$	$0.01 \times 05 = 0.05$	
10	$0.087 \times 10 = 0.870$	$0.01 \times 10 = 0.10$	
15	$0.087 \times 15 = 1.305$	$0.01 \times 15 = 0.15$	
20	$0.087 \times 20 = 1.740$	$0.01 \times 20 = 0.20$	
25	$0.087 \times 25 = 2.175$	$0.01 \times 25 = 0.25$	
Test solution V =            ml	$0.087 \times \dots\dots =$	$0.01 \times \dots\dots =$	

**Note:** 1. Molecular weight of ferric alum =  $\text{Fe}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O} = 964$ .

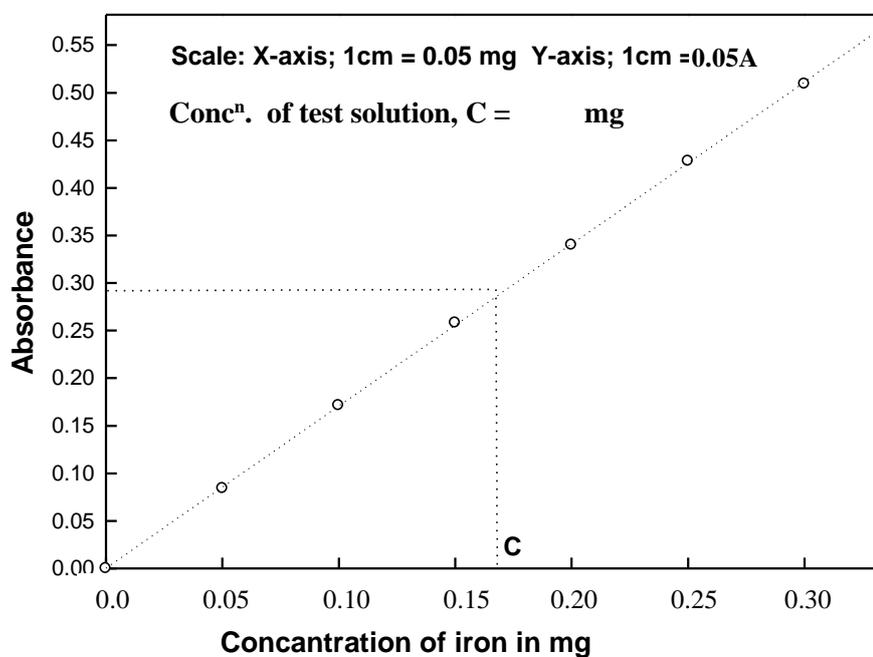
2. Atomic weight of iron = 55.85

1.0 ml of stock ferric alum solution = 0.087 mg of ferric alum = 0.01 mg of iron

964 g of ferric alum = 55.85 x 2 g of iron

$\therefore 0.087 \text{ mg of ferric alum} = \frac{111.7 \times 0.087}{964} = 0.01 \text{ mg of iron.}$

964



Hence, the concentration of Fe in test solution given = C =    \_ \_ \_ mg

Note: 1. Do not write the nature of the graph in the record  
2. Flat the graph at the center of graph sheet.

**Expt. No. : 10****Date:****Determination of iron by colorimetric method.****Theory:**

When a monochromatic light of intensity ( $I_0$ ) is incident on a transparent medium, a part of light is absorbed by the media ( $I_a$ ), a part of light is reflected ( $I_r$ ) and the remaining part of light is transmitted ( $I_t$ ).

$$\therefore I_0 = I_a + I_t + I_r \quad (\text{for a glass - air interface, } I_r \text{ is negligible}) \quad \therefore I_0 = I_a + I_t$$

**Lambert's law:** statement: The intensity of transmitted light ( $T$ ) decreases exponentially or geometrically as the path length ( $l$ ) increases arithmetically. ie  $T = I_t / I_0 = 10^{-k \cdot l}$ , where  $T$  is called transmittance and is defined as  $T = I_t / I_0$  and where  $k$  is a proportional constant.

**Beer's law:** statement: The intensity of transmitted light ( $T$ ) decreases exponentially or geometrically as the concentration ( $C$ ) of the solution increases arithmetically. ie  $T = I_t / I_0 = 10^{-k' \cdot C}$  (where  $k'$  is a proportional constant). Combining of these two laws called as,

**Beer's - Lambert's law:** statement: The intensity of transmittance ( $T$ ) decreases exponentially or geometrically as the path length ( $l$ ) and concentration ( $C$ ) of the transparent solution increases arithmetically.

$$\text{i.e. } T = I_t / I_0 = 10^{-\epsilon Cl} \quad (\text{where } \epsilon \text{ is a combined constant of } k \text{ \& } k').$$

$$\therefore \text{The absorbance (A) is defined as } A = -\log T = \log 1/T = \log (1/10^{-\epsilon Cl}),$$

$$A = \log_{10} (10^{\epsilon Cl}) = \epsilon Cl \log_{10} 10. \quad \therefore \mathbf{A = \epsilon Cl} \quad \because \log_{10} 10 = 1$$

Here  $\epsilon$  is the molar extension coefficient which is a constant for a given substance at a given wavelength and ( $l$ ) is the path length is also constant for given cell tube. The absorbance ( $A$ ) is dependence on only concentration ( $C$ ) of the solution.  $\therefore \mathbf{A \propto C}$ . "Beer's-Lambert's law also states that the absorbance is directly proportional to the concentration of the solution". Hence plot the graph, absorbance ( $A$ ) against concentration ( $C$ ) of the solution gives a straight line.

A series of standard solution of iron salt and test solution is treated with 5ml of 4N nitric acid and 5ml of 2% potassium thiocyanate solution and is diluted to a definite volume. The absorbance of each of these solution and test solution is measured at **480 nm** since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against concentration of iron to

Concentration of iron in test solution = C = .....mg

Since, 1ml of ferric alum = 0.01mg of Fe

V ml of ferric alum = C mg of Fe

$$\therefore V \text{ ml of ferric alum} = V = \frac{C}{0.01} = \frac{\quad}{0.01} = \text{.....ml}$$

Hence, the volume of iron in test solution = V = .....ml

### Results:

1. The volume of test solution given = \_\_\_\_\_ ml

2. Absorbance of test solution = \_\_\_\_\_

3. Concentration of iron in test solution = \_\_\_\_\_ mg

4. Concentration of ferric alum solution = \_\_\_\_\_ mg

get a calibration curve and concentration of iron in test solution is determined from the graph.

**Procedure:** Transfer the given ferric alum solution (stock solution) to a burette and draw out 5, 10, 15, 20 and 25 ml of the solution into 50 ml standard flasks. Add 5 ml of 4N nitric acid and 5ml of 2% potassium thiocyanate solution to each of them and also to the test solution given in a separate 50 ml standard flask. Make up the solution to the mark by adding distilled water and mix well for uniform concentration.

Switch on the colorimeter and warm it for 5 minutes. Using the filter knob, select the wave length of  $\lambda_{\text{max}} = 480 \text{ nm}$  for ferric alum solution. Take the blank solution in the cell tube and keep it in the colorimeter and adjust zero absorbance for 100% transmittance using the knob. Then measure the optical density or absorbance for each standard solution and test solution using a photo electric colorimeter. A straight line graph is obtained by plotting optical density or absorbance against the concentration of iron. From the graph, determine the concentration of iron in the test solution and calculate the volume of ferric alum in the given test solution.



**Apparatus used:** Colorimeter and photoelectric cell tube

**Applications:** It is used to determination the concentration of the colored metal ions, optical density (OD), intensity of absorbed light ( $I_a$ ), transmitted light ( $I_t$ ), reflected light ( $I_r$ ) transmittance (T), molar absorption coefficient ( $\epsilon$ ), path length (l). Verification of Beer's and Lambert's law.

- Note:** i) Preparation of blank solution: - Blank solution can be prepared by adding 5.0 ml of 2N nitric acid and 5ml of 2% potassium thiocyanate solution in 50 ml standard flask and distilled water to the mark. Mix well for uniform concentration.
- ii) Switch off the instrument after experiment is completed.



**Expt. No. : 11****Date:****Determination of copper by colorimetric method.****Theory:**

When a monochromatic light of intensity ( $I_0$ ) is incident on a transparent medium, a part of light is absorbed by the media ( $I_a$ ), a part of light is reflected ( $I_r$ ) and the remaining part of light is transmitted ( $I_t$ ).

$$\therefore I_0 = I_a + I_t + I_r \quad (\text{for a glass - air interface, } I_r \text{ is negligible}) \quad \therefore I_0 = I_a + I_t$$

**Lambert's law:** statement: The intensity of transmitted light ( $T$ ) decreases exponentially or geometrically as the path length ( $l$ ) increases arithmetically. ie  $T = I_t / I_0 = 10^{-kl}$ , where  $T$  is called transmittance and is defined as  $T = I_t/I_0$  and where  $k$  is a proportional constant.

**Beer's law:** statement: The intensity of transmitted light ( $T$ ) decreases exponentially or geometrically as the concentration ( $C$ ) of the solution increases arithmetically. ie  $T = I_t / I_0 = 10^{-k'C}$  (where  $k'$  is a proportional constant). Combining of these two laws called as,

**Beer's -Lambert's law:** statement: The intensity of transmitted light ( $T$ ) decreases exponentially or geometrically as the path length ( $l$ ) and concentration ( $C$ ) of the transparent solution increases arithmetically.

$$\text{i.e. } T = I_t / I_0 = 10^{-\epsilon Cl} \quad (\text{where } \epsilon \text{ is a combined constant of } k \text{ and } k^*).$$

$\therefore$  The absorbance ( $A$ ) is defined as  $A = -\log T = \log 1/T = \log (1/10^{-\epsilon Cl})$ ,

$$A = \log_{10} (10^{\epsilon Cl}) = \epsilon Cl \log_{10} 10. \quad \therefore \mathbf{A = \epsilon Cl} \quad \because \log_{10} 10 = 1$$

Here  $\epsilon$  is the molar extension coefficient which is a constant for a given substance at a given wavelength and ( $l$ ) is the path length is also constant for given cell tube. The absorbance ( $A$ ) is dependence on only concentration ( $C$ ) of the solution.  $\therefore$  **A $\propto$ C**. "Beer's-Lambert's law also states that the absorbance is directly proportional to the concentration of the solution". Hence plot the graph, absorbance ( $A$ ) against concentration ( $C$ ) of the solution gives a straight line.

A series of standard solution of copper salt and test solution is treated with ammonia to get blue cupra-ammonium complex and is diluted to a definite volume. The absorbance of each of these solution and test solution is measured at **620 nm** since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against concentration of copper get a

Concentration of Cu in test solution =  $C = \dots\dots\dots$ mg

Since, 1ml of  $\text{CuSO}_4 = 1\text{mg}$  of Cu

V ml of  $\text{CuSO}_4 = C$  mg of Cu =  $\dots\dots\dots$ ml

Hence, the volume of Cu in test solution =  $V = \dots\dots\dots$ ml

**Results:**

1. The volume of test solution given =  $\dots\dots\dots$  ml

2. Absorbance of test solution =  $\dots\dots\dots$

3. Concentration of copper in test solution =  $\dots\dots\dots$  mg

4. Concentration of copper sulphate solution =  $\dots\dots\dots$  mg

calibration curve and concentration of copper in test solution is determined from the graph.

**Procedure:** Transfer the given copper sulphate solution (Stock solution) to a burette and draw out 5, 10, 15, 20 and 25 ml of the solution into 50 ml standard flasks. Add 5 ml of 1:1 ammonia solution to each of them and also to the test solution given in a separate 50 ml standard flask. Make up the solution to the mark by adding distilled water and mix well for uniform concentration.

Switch on the colorimeter and warm it for 5 minutes. Using the filter knob (B), select the wave length of  $\lambda_{\text{max}} = 620 \text{ nm}$  for copper sulphate solution. Take the blank solution in the cell tube and keep it in the colorimeter and adjust zero absorbance for 100% transmittance using the knob (A). Then measure the optical density or absorbance for each standard solution and test solution using a photo electric colorimeter. A straight line graph is obtained by plotting optical density or absorbance against the concentration of copper. From the graph, determine the concentration of copper in the test solution and calculate the volume of copper sulphate in the given test solution.

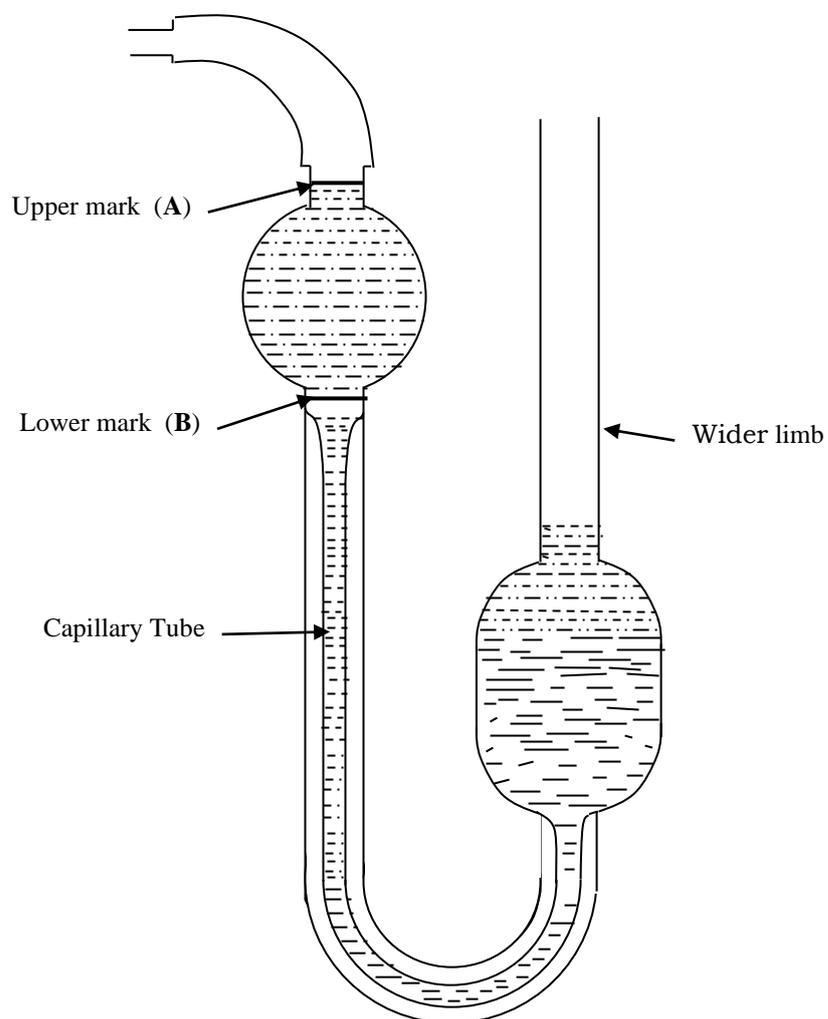


**Apparatus used:** Colorimeter and photoelectric cell tube

**Applications:** It is used to determination the concentration of the colored metal ions, optical density (OD), intensity of absorbed light ( $I_a$ ), transmitted light ( $I_t$ ), reflected light ( $I_r$ ) transmittance (T), molar absorption coefficient ( $\epsilon$ ), path length (l). Verification of Beer's and Lambert's law.

**Note:** i) Preparation of blank solution: - Blank solution can be prepared by adding 5.0 ml of 1:1 ammonia and distilled water in 50 ml standard flask. Mix well for uniform concentration.

ii) Switch off the instrument after experiment is completed.



**Ostwald's Viscometer**

**Note:** Do not write the Viscometer figure in the record.

**Expt. No. : 12****Date:****Determination of viscosity coefficient of liquid using Ostwald's viscometer.**

**Theory:** Viscosity arises due to internal friction between moving layers of molecules. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. A layer close to the surface is almost stationary while that at the axis of tube moves faster than any other intermediate layer. A slow moving layer exerts a drag or friction on its nearest moving layer backwards. This property of a liquid by which it retards or opposes motion of a liquid between the layers is called viscosity. The coefficient of viscosity is defined as the tangential force per unit area required maintaining a unit velocity gradient between any two successive layers of a liquid situated unit distance apart. The coefficient of viscosity of a liquid is given by Poiseuille's formula,

$$\eta = \frac{\pi P r^4 t}{8 V l} \quad \text{Where, } P = h d g, \quad \eta = \frac{\pi h d g r^4 t}{8 V l}$$

Where  $\eta$  is the coefficient of viscosity of the liquid,  $V$  = volume of the liquid,  $r$  = radius of the tube,  $l$  = length of the tube,  $P$  is the pressure difference between the two ends of the tube,  $h$  = height of the capillary tube,  $d$  = density of the liquid, and  $g$  = gravitational force. Co-efficient of viscosity of the given liquid is determined by comparison method by taking water as standard liquid. If equal volumes of two different liquids are allowed to flow through the same tube under identical conditions, then,

$$\frac{\eta_w}{\eta_L} = \frac{\pi h g r^4 t_w d_w}{8 V l} \times \frac{8 V l}{\pi h g r^4 t_L d_L} = \frac{t_w d_w}{t_L d_L}$$

Where,  $\eta_w$  = co efficient of viscosity of the standard liquid (water), and  $\eta_L$  = co efficient of viscosity of the given liquid which can be determined.  $t_w$  = time taken by the standard liquid(Water),  $t_L$  = time taken by the given liquid,  $d_w$  = density of standard liquid (water),  $d_L$  = density of given liquid. Viscosity is a temperature dependent property and is inversely proportional to the temperature ( $T$ ), i.e.  $\eta \propto 1/T$ . Hence the measurements are carried out at constant temperature.

### Observations and calculations

Trial No	I	II	III	Average time in seconds
Time taken by water in sec [ $t_w$ ]				
Time taken by Liquid in sec [ $t_L$ ]				

∴ Bottle No = .....

Wet temperature = ..... °C

$$\eta_L = \frac{\eta_w t_L d_L}{t_w d_w}$$

Where  $t_w$  = time taken by water = \_\_\_\_\_ sec

$t_L$  = time taken by liquid = \_\_\_\_\_ sec

$d_w$  = density of water = \_\_\_\_\_ g / cc

$d_L$  = density of liquid = \_\_\_\_\_ g / cc

$\eta_w$  = co-efficient of viscosity of water = \_\_\_\_\_ milli poise

$$\eta_L = \frac{\eta_w t_L d_L}{t_w d_w} = \frac{\text{_____}}{\text{_____}} = \text{_____} \text{ milli poise}$$

∴  $\eta_L$  = coefficient of viscosity of liquid = \_\_\_\_\_ milli poise

Correct value of  $\eta_L$  = \_\_\_\_\_ milli poise

$$\% \text{ error of } \eta_L = \frac{\text{Differences of determined value \& correct value of } \eta_L \times 100}{\text{Correct value of } \eta_L} = \text{_____} \times 100 = \text{_____}$$

∴ % error of  $\eta_L$  = ..... %

**Procedure:**

1. Transfer a known volume (say 15 ml) of distilled water taken in a burette into the cleaned and dried viscometer which is fixed in to the stand. Suck the distilled water above the upper mark 'A' in the viscometer and allow it to flow freely through a capillary. Start the stop clock when the water level just crosses the upper mark 'A' and stop it when the water level just crosses the lower mark 'B' in the viscometer. Note down the time of flow of water in seconds ( $t_w$ ) and repeat the experiment to get agreeing values.
2. Pour out the distilled water and rinse the viscometer in acetone again. Dry it in an electric oven and cool to the room temperature.
3. Transfer the known volume (say 15 ml) of the given organic liquid taken in a separate burette into a viscometer. Note down the time of flow of organic liquid ( $t_L$ ) as similar to the water.
4. Using the values of  $\eta_w$ ,  $d_w$  and  $d_L$ , determine the viscosity co-efficient of the given organic liquid using the relation,

$$\eta_L = \frac{\eta_w t_L d_L}{t_w d_w}$$

**Note:** -Transfer the organic liquid into the bottle after the experiment is completed and rinses the viscometer with acetone and keep it in an electric oven.

**Apparatus used:** Ostwald's viscometer

**Applications** : Viscosity play an important role on:

- 1) Separation of miscible liquids
- 2) For characterization of the texture of foods
- 3) Separation of lubricating oils and other fractions in petroleum industries.
- 4) Mixing of concrete for RCC
- 5) Flowing of liquids at different rate depending on the temperature
- 6) High viscosity play important role on adhesives

### Observations and calculations

Volume of Na in ml	Concentration of Na in ppm	Flame photometric reading (FPR)
2	2	
4	4	
6	6	
8	8	
10	10	
Test solution (V) =            ml		

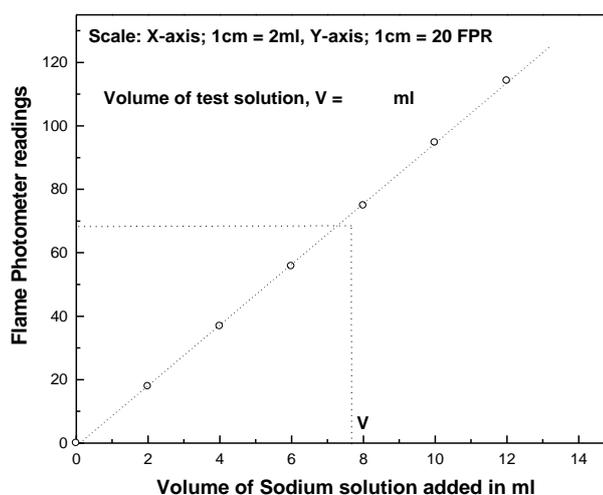
#### Note:

1. Molecular weight of NaCl = 58.5
2. Atomic weight of sodium = 23
3. Stock solution of Na = 500 ppm
4. 1 mg of sodium in one liter = 1 ppm

58.5 gms NaCl = 23 g of sodium

$$2.55 \text{ gms of NaCl} = \frac{23 \times 2.55}{58.5} = 1.0 \text{ mg of Na in one litre}$$

$\therefore$  1.0 ml of NaCl solution = 1.0 mg of Na = 2.55 mg NaCl



- Note:**
1. Do not write the nature of the graph in the record
  2. Flat the graph at the center of graph sheet.

Expt. No. : 13

Date:

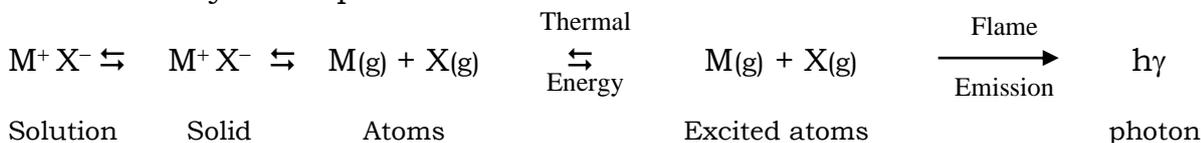
**Determination of sodium in fluid by flame photometric method.****Theory:**

Flame photometer is the main tool to measure the intensity of colored elements. Therefore sodium, potassium, calcium and lithium and other common elements impart characteristic colors with the Bunsen flame.

e.g. sodium gives golden yellow, lithium gives crimson red ...etc with flame. Hence the intensity of colored flame is directly proportional to the number of atoms present in the solution and this principle is adopted in flame photometer. When a solution containing a compound of the metal to be investigated is injected into a flame, the following process occurs.

1. Solvent evaporates leaving behind a solid residue.
2. Vaporization of the solid coupled with dissociation into its constituent atoms, which are initially in the ground state.
3. All gaseous atoms get excited by the thermal energy of the flame to the higher energy levels.
4. The excited atoms are unstable at higher energy state, quickly emit photons and return to lower energy state. (i.e. ground state)
5. Flame photometry involves the measurement of emitted radiation equal to the number of atoms present in the excited state

Schematically it is represented as:



The relationship between the ground state and excited state populations is given by the Boltzmann equation:  $N_1 / N_0 = (g_1 / g_0) e^{-\Delta E / KT}$

$N_1$  = Number of atoms in the excited state

$N_0$  = Number of atoms in the ground state

$g_1 / g_0$  = Ratio of statistical weights for ground and excited states

$\Delta E$  = Energy of excitation =  $h\nu$  (photon)

$K$  = the Boltzmann constant and  $T$  = Absolute Temperature

**Results:**

1. The volume of sodium in test solution = \_ \_ \_ \_ \_ ml.
2. Flame photometer reading of test solution = \_ \_ \_ \_ \_
3. Concentration of Na in test solution = \_ \_ \_ \_ \_ ppm

From the above equation, it is evident that the ratio  $N_1/N_0$  is dependent upon both the excitation energy  $E$  and the temperature  $T$ . An increase in temperature and a decrease in  $\Delta E$  will both result in a higher value for the ratio  $N_1/N_0$ .

**Procedure:**

1. Transfer 2, 4, 6, 8 and 10 ml of standard sodium chloride solution into different 50 ml standard flasks from the burette.
2. Make up all the solutions and the test solution to mark in 50 ml standard flasks by adding distilled water and mix well.
3. Place the sodium filter (589nm) in position.
4. Switch on the compressor and adjust the compressor air supply to 10-psi (pound per square Inch) using pressure regulator knob and also switch on the main unit.
5. Turn the gas supply on and light the gas at the burner, regulate the gas supply so that the colour of the flame completely turns to blue.
6. Dip the capillary tube in a cell containing distilled water.
7. Adjust the reading to zero by "Set Zero" knob.
8. Now feed standard solution of sodium and adjust the reading to 120 by "Set Span" knob.
9. Feed the various sodium solutions and test solution through the capillary tube and note down the flame photometer reading.
10. Plot a graph of flame photometer reading vs. volume of the solution to get the calibration curve and from the curve find out the volume of sodium in test solution.

**Apparatus used:** Flame photometer, LPG, gas burner, air and filter.

**Applications:** It is used to determination of the concentration of Na, Ca, Li, K, and other elements present in the solutions or fluids.

## Viva questions

### PART - A

1. Define molecular and equivalent weights.
2. What is an acid and base?
3. What is meant by titration?
4. What is end point?
5. What is a standard solution?
6. What is standardization?
7. What is estimation?
8. Define normality.
9. Define molarity.
10. What is an indicator? and mention the different types of indicators.
11. What is an oxidation and reduction?
12. What is an Oxidising and reducing agents?
13. What is hard water?
14. How is hardness of water caused?
15. What are the types of hard water?
16. What are the causes of temporary and permanent hardness of water?
17. How is temporary and permanent hardness of water removed?
18. How do you express the total hardness of water?
19. Name the metal ion indicator.
20. What is EDTA?
21. What are buffer solutions?
22. Mention types of buffer solutions.
23. Why is basic buffer solution is added in the determination of hardness of water?
24. What is the end point in the determination of total hardness of water?
25. What are the constituents of cement?
26. Which indicator used in the determination of CaO in cement solution?
27. Why the diethyl-amine and glycerol added to the cement solution?
28. What is an alloy?
29. What are the constituents of brass?
30. How is a brass solution prepared?
31. What is the purpose of adding urea to the brass solution?
32. Why is  $\text{NH}_4\text{OH}$  added to the brass solution in the determination of Cu?
33. Why is acetic acid added to the brass solution in the determination of Cu?
34. Why is KI added to the brass solution although copper is present in it?
35. Why is the blue solution of brass turns brown by the addition of KI solution?
36. Why is starch indicator added towards the endpoint in iodometric titrations?
37. Why is the white precipitated formed in the determination of Cu in brass?
38. What is hypo?
39. What is an ore? Give the differences between ore and mineral.
40. What is the main constituent of haematite ore?
41. What is the role of  $\text{SnCl}_2$  in the determination of iron in haematite ore?
42. Why is mercuric chloride added in the determination of iron in haematite?
43. What happens when the excess of  $\text{SnCl}_2$  is not removed in the determination iron?
44. What is the indicator used in the determination of iron in haematite ore?
45. Why is potassium ferricyanide cannot be used as an internal indicator in the analysis of haematite ore?
46. What is the colour of  $\text{K}_3[\text{Fe}(\text{CN})_6]$  with ferrous ion?

47. Why, the color of the indicator drop remains the same at the end point in the determination of iron?
48. What is Mohr's salt?
49. What are the salts present in Mohr's salt?
50. Why is dilute  $\text{H}_2\text{SO}_4$  added during the preparation of Mohr's salt solution?
51. What is a hydrolysis?
52. Which part of the Mohr's salt undergoes oxidation?
53. What is chemical oxygen demand (COD)?
54. What is biological oxygen demand (BOD)?
55. What is the role of silver sulphate in the determination of COD?
56. What is role mercuric sulphate in the determination COD?
57. What is the indicator used in COD experiment?
58. What is the colour change at the end point in the determination of COD?
59. Why COD value always greater then BOD value.
60. How does  $\text{K}_2\text{Cr}_2\text{O}_7$  act as oxidizing agent?
61. What is blank and back titration?
62. What is alkalinity?
63. Name the responsible for alkalinity of water.
64. What is the source of alkalinity in water?
65. In alkalinity of water, the presence of  $\text{OH}^-$ ,  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$  cannot be determined simultaneous, explain.
66. What alkalinities are indicated by Phenolphthalein and methyl orange endpoint?

**PART – B**

1. What is weak acid?
2. What is pKa of a weak acid?
3. Define pH.
4. Mention functions of pH and give the scale of pH.
5. What is Ka?
6. How are pH and pKa related?
7. What are the electrodes used in pH meter?
8. What is conductance?
9. What is the unit for conductance?
10. What is emf of the cell?
11. What is cell constant? Mention its unit.
12. Why, the conductance of acid mixture decreases on adding NaOH before the end point?
13. Why, the conductance of acid mixture increases on adding NaOH after the end point?
14. What is absorbance?
15. Define Beer's law.
16. Define Lambert's law.
17. Define Beer – Lambert's law.
18. What is the relation between absorbance and concentration?
19. What is a blank solution? What is meant by transmittance?
20. Name the complex formed between Cu and NH<sub>4</sub>OH in the colorimetric experiment.
21. Why is ammonia added to CuSO<sub>4</sub> solution in the colorimetric determination of Cu?
22. Why, 620 nm wavelength is used in the determination of copper in colorimeter?
23. Name the complex formed between Fe and KSCN in the colorimetric experiment.
24. Why is KSCN added to ferric alum solution in the colorimetric determination of Fe?
25. Why, 480 nm wavelength is used in the determination of iron in colorimeter?
26. What is meant by atomic emission?
27. What is the principle involved in flame photometry?
28. What are alkali and alkaline earth metals?
29. What are the various events that occur when a solution containing metal ions introduced into the flame?
30. What are different components of a flame photometer?
31. What is the flame photometric analysis of sodium is advantageous?
32. What is the role of filter in flame photometry?
33. Differentiate emission spectroscopy from atomic spectroscopy?
34. What are the factors that influence in flame photometric experiment?
35. Name the fuel and oxidant used in flame photometric experiment?
36. What is viscosity?
37. What is viscosity coefficient of a liquid?
38. What is the SI unit of viscosity coefficient?
39. How does the viscosity vary with temperature?
40. Write the expression used to calculate the viscosity coefficient of liquid.
41. Why, the same volume of water and liquid taken in the determination of viscosity Co-efficient of liquid?
42. What is an electrolyte?
43. What are types of electrolyte? And give example for each.
44. Mention electrodes used in potentiometer.
45. Why the emf of cell increases sharply in potentiometer.
46. How do you construct a cell in potentiometer?



## DEPARTMENT OF CHEMISTRY

PES COLLEGE OF ENGINEERING, MANDYA - 571401

(An Autonomous Institution affiliated to VTU, Belagavi)



B.E. I/II Sem		Course Code: P18CHL17/27	Course Code: Engineering Chemistry Lab			Academic Year : 2019-20
Sl. No	Date	Name of Experiments	Marks for conduction of each Experiment & Record [A]			
			Conduction 20 Marks	Record 05 Marks	Total 25 Marks	
1		Estimation of hypo				
2		COD of waste water				
3		% of Copper in brass				
4		% of Iron in haematite ore solution				
5		Total hardness of water				
6		% of CaO in Cement solution				
7		pKa value by pH meter				
8		Acid mixture by Conductometer				
9		FAS by Potentiometer				
10		Fe by colorimeter				
11		Cu by colorimeter				
12		Viscosity-coefficient by viscometer				
13		Alkalinity of water	Demonstration Experiment			-----
14		Sodium by Flame Photometer	Demonstration Experiment			-----
Total marks of conduction and record of all the experiments						
Average marks of conduction and record of all the experiments					[A] =	
Marks for Engineering Chemistry Lab Test						
Date	Experiment Name	Distribution of Marks for Lab Test [B]				
		Procedure 5 Marks	Conduction 12 Marks	Calculation 5 Marks	Viva- Voce 3 Marks	Total [B] 25 Marks
	Part – A or Part -B					
(Max. Marks: 50)		CIE Marks of Engineering Chemistry Lab [A + B] =				/ 50

**Note: - Student is required to verify the CIE marks of Engg, Chemistry Lab. If any discrepancy, bring the notice of Head of the Department.**

Signature of the Faculty

Signature of HOD with Seal