

**SIR C. R. REDDY
COLLEGE OF ENGINEERING
ELURU -- 534007**



First Year Engineering Department

APPLIED/ENGINEERING CHEMISTRY LAB MANUAL

For

**I/IV B.E /B.Tech. COURSES
SEMESTER - I/II A.Y : 2021- 22**

NAME: -----

BRANCH: ----- SECTION: -----

REGD.NO: -----

1/1V B .Tech Applied/Engineering chemistry Laboratory

Credits :1.5;Examination ;3Hrs

Lab Hrs/Week --3 ; Sessional marks - 15M ; Exam marks - 35M ; Total marks -50M

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SIR C. R. REDDY COLLEGE OF ENGINEERING, ELURU - 534007
First Year Engineering Department
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	2.
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INTRODUCTION

The chemical analysis is broadly divided into qualitative and quantitative analysis.

Qualitative analysis deals with the detection and identification of the constituents of substances or constituents present in the given solution.

Quantitative analysis deals with the determination of the amount of the substances present in the given solution.

Quantitative analytical methods can be broadly divided into three groups.

1. Gravimetric Analysis
2. Volumetric Analysis
3. Instrumental Analysis

Any person desirous of analysing a substance by any one of these methods should be acquainted not only with the way of analysis is carried out but also with the techniques and principles involved in these methods. Such knowledge is essential for an engineer with an analytical mind. Hence keeping in view of the importance of the analysis, an introduction to principles and techniques involved in the volumetric analysis are described below.

Volumetric analysis:- Volumetric methods of analysis are generally simple, more rapid, require less apparatus and frequently provide greater accuracy than other analytical methods. Volumetric analysis involves the measurement of volume of the solutions containing the reacting substances.

Definitions and standard terms

Titration:- The process of finding out the volume of one of the solution required to react completely with a definite volume of other solution.

Titrant:- The solution of known strength generally taken in the burette.

Titrate:- The solution being titrated (whose concentration is to be determined generally taken in the conical flask)

Standard solution:- The solution whose concentration is exactly known.

Primary standard solution:- Solutions whose concentrations do not vary on standing. Generally they are prepared by direct weighing and dissolving known quantity of the solute in definite volume of the solvent. Examples:- Solutions of Oxalic acid, Potassium dichromate etc.

Secondary standard solution:- Solutions whose concentrations can be known by titrating with a primary standard solution. Examples:- Solutions of Sodium hydroxide, Potassium permanganate etc.

Indicator: - Substance used to indicate end point of the titration. The indicator indicates the completion of reaction by change in the colour at the end point.

End point:- The point at which the completion of the titration is judged by a visual Colour change

Strength:- Concentration of the solution per unit volume. Strength of the solution can be expressed by the following methods.

Normality;- The number of the gram equivalents of the solute present in one litre of the solution and is represented by N.

$$\text{Normality} = \frac{\text{Wt of the substance}}{\text{Gm.Eq.weight}} \times \frac{1000}{\text{Volume of the solution}}$$

Molarity:- Number of the moles of the solute present in one litre of the solution and is represented by M.

$$\text{Molarity} = \frac{\text{Wt of the substance}}{\text{Mol. weight}} \times \frac{1000}{\text{Volume of the solution}}$$

Equivalent weight:- Number of parts by weight of it that combines with or displace one part by weight of hydrogen or eight parts by weight of oxygen.

$$\text{Equivalent weight of a metal} = \frac{\text{Atomic Weight}}{\text{Valency}}$$

$$\text{Equivalent weight of an acid} = \frac{\text{Molecular Weight of an acid}}{\text{Basicity of an acid}}$$

$$\text{Equivalent weight of a base} = \frac{\text{Molecular Weight of a base}}{\text{Acidity of a base}}$$

Acidity:- Number of replaceable hydroxide ions of a base

Basicity:- Number of replaceable hydrogen ions of an acid

Volumetric analysis can be classified in to the following four types

1. Neutralization titration
2. Redox Titration
3. Complexometric Titration
4. Precipitation titration

Neutralization titration:

S.No	Titration	Indicator	Colour change
1.	Strong acid Vs Strong base HCl Vs NaOH	Methyl orange	Orange yellow in basic media Orange red in acidic media
		Phenolphthalein	Pink in basic media Colourless in acidic media
2.	Weak acid Vs Strong base Oxalic acid Vs NaOH	Phenolphthalein	Pink in basic media Colourless in acidic media
3.	Strong acid Vs Weak base HCl Vs NH ₄ OH	Methyl orange	Orange yellow in basic media Orange red in acidic media
4.	Weak acid Vs Weak base	No indicator is suitable	-----

Redox titrations

S.No	Titration	Indicator	Colour change
1.	<i>Permanganometry</i>	Self indicator	Colourless to pink
	Oxalic acid Vs KMnO_4	Self indicator	Colourless to pink
	Mohr's Salt Vs KMnO_4	Self indicator	Colourless to pink
2.	<i>Dichrometry</i>	Diphenylamine	Green to Violet
	Mohr's Salt Vs $\text{K}_2\text{Cr}_2\text{O}_7$		
3.	<i>Idometry</i>		
	$\text{K}_2\text{Cr}_2\text{O}_7$ Vs Sodium thiosulphate	starch	Blue to light green
	CuSO_4 Vs Sodium thiosulphate	starch	Blue to light green

Equivalent weights of the substances used in volumetric analysis

S.No	Substance	Chemical formula	Equivalent weight
1	Sodium Carbonate	Na_2CO_3	53.06
2	Hydrochloric acid	HCl	36.5
3	Sulphuric Acid	H_2SO_4	49.04
4	Oxalic acid	$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$	63.03
5	Ferrous Ammonium sulphate(Mohr's salt)	$\text{FeSO}_4 (\text{NH}_4\text{SO}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$	392.14
6	Potassium permanganate	KMnO_4	31.06
7	Potassium dichromate	$\text{K}_2\text{Cr}_2\text{O}_7$	49.03
8	Sodium thiosulphate	$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$	248.19
9	Iron	Fe	55.85
10	Chromium	Cr	17.3
11	Manganese	Mn	27.5
12	Copper	Cu	31.8

Complexometric Titrations :

S.No.	Titration	Indicator	Color Change
1.	Hardness of Water (EDTA)	EBT	Wine red to Blue
2.	Zinc Vs EDTA	EBT	Wine red to Blue
3.	Copper Vs EDTA	Fast Sulphone Black F	Pale blue to bright green

Precipitation titrations :

S.No.	Titration	Indicator	Color Change
1.	Chlorides of water Vs AgNO ₃	Potassium chromate	Yellow to Brick Red
2.	Sodium Chloride Vs AgNO ₃	Potassium chromate	Yellow to Brick Red

Equivalent Weight : The equivalent weight of a substance is defined as that weight of the substance which is chemically equivalent to one gram of hydrogen, or 35.5 grams of chlorine or 8 grams of oxygen.

Acids : Equivalent weight of an acid = Formula weight / basicity (basicity is the number of replaceable hydrogen atoms present in the acid.)

S.No.	Acid Formula	Basicity	Formula Wt.	Eq. Wt
1.	HCl	1	36.5	36.5
2.	H ₂ SO ₄	2	98.0	49.0
3.	H ₂ C ₂ O ₄ ·2H ₂ O	2	126.0	63.0
4.	CH ₃ COOH	1	60.0	60.0
5.	H ₃ PO ₄	3	98.0	32.7

Bases : Equivalent weight of a base = Formula weight / Acidity (Acidity is the number of replaceable hydroxyl groups present in the base.)

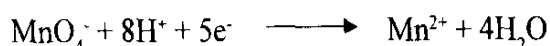
S.No.	Base Formula	Acidity	Formula Wt.	Eq. Wt
1.	NaOH	1	40	40
2.	Ca(OH) ₂	2	74	37
3.	CaO	2	56	28

REDOX REAGENTS (Electron Concept) :

OXIDISING AGENT : An oxidising agent is one that gains electrons and gets reduced to a lower oxidation state.

Eq. Wt. of oxidant = Formula Wt. / No. of electrons gained by one species.

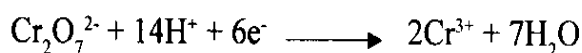
Eg. 1. KMnO₄ : The partial equation of KMnO₄ for the reduction process may be written as follows :



The number of electrons gained is five

Eq. Wt. of KMnO_4 = Formula Wt./5 = $158/5 = 31.6$

Eg. 2. $\text{K}_2\text{Cr}_2\text{O}_7$: The partial equation is given below



The number of electrons gained is six.

Eq. Wt. of $\text{K}_2\text{Cr}_2\text{O}_7$ = Formula Wt./6 = $294.18/6 = 49.03$

Eq. Wt. of Cr(VI) = Formula Wt./3 = $51.9/3 = 17.3$

REDUCING AGENT : A reducing agent is one that loses electrons and gets oxidised to a higher oxidation state.

Eq. Wt. of a reducing agent = Formula Wt./No. of electrons lost by one molecule.

Eg. 1. Mohr's salt (Ferrous ammonium sulphate) :

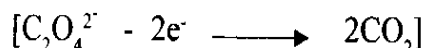


The no. of electrons lost is one.

Eq. Wt. of Mohr's Salt = Formula Wt./1 = $392.1/1$

Eq. Wt. of Ferrous iron = $55.85/1 = 55.85$

Eg. 2. Oxalic acid : The partial equation is as follows :



The no. of electrons lost is two.

Eq. Wt. of oxalic acid = Formula Wt. of oxalic acid / 2 = $126/2 = 63$

Precautions :

1. Students are required to go through the experimental procedure and come prepared for the experiment.
2. First clean your apparatus thoroughly with tap water and finally rinse with distilled water.
3. Rinse the pipette and burette with the solution before taking it into them.
4. No air bubble should be entered while filling the pipette or burette.
5. Never blow the pipette to expel the last drop (it is calibrated by leaving that drop in the pipette)
6. Note the reading of burette or pipette by keeping your eye exactly at the same level to avoid parallax error.
7. Take upper meniscus for colored solution and lower meniscus for colorless solution in the burette.
8. End point must be sharp (color change must be obtained by the addition of a single drop)
9. Wash your apparatus thoroughly before leaving the laboratory.
10. Girl students are advised to wear aprons while doing the experiments.

PREPARATION OF STANDARDED SODIUM CARBONATE SOLUTION

W_1 = Weight of the weighing bottle + substance = gms

W_2 = Weight of the weighing bottle after transferring the substance = gms

Weight of the substance transferred ($W_1 - W_2$) = gms

$$\text{Normality of the Sodium carbonate solution} = \frac{\text{Weight of the substance} \times 1000}{\text{Gram equivalent weight} \times 100}$$

$$= \frac{\text{X 1000}}{53.06 \times 100}$$

ESTIMATION OF HYDROCHLORIC ACID

S.No	Volume of Std Sodium carbonate solution taken (ml)	Burette Readings		Volume of hydrochloric acid solution run down (ml)
		Initial (ml)	Final (ml)	
1				
2				
3				

Std Sodium carbonate solution	Hydrochloric acid solution
N_1 : Normality of std Sodium carbonate solution =N	N_2 : Normality of Hydrochloric acid Solution = ...?.....N
V_1 : Volume of std Sodium carbonate solution =ml.	V_2 : Volume of Hydrochloric acid solution =ml.

Normality of Hydrochloric acid solution $N_2 = \frac{N_1 V_1}{V_2} =$

= N

Amount of hydrochloric acid present in the given 100 ml. of the hydrochloric acid solution

$$= \frac{\text{Normality of hydrochloric acid Solution } (N_2) \times \text{Equivalent weight of hydrochloric acid } (36.5) \times 100}{1000}$$

= gms

Expt.No:

Date:

Determination of HCl using standard Na₂CO₃ solution.

Aim:- To estimate the amount of HCl using standard Sodium carbonate solution

Chemicals required: 1) Standard sodium carbonate solution 2) Hydrochloric acid

Indicator: Methyl orange

Theory: Determination of an acid by using a standard alkali solution is called alkali metric method. The amount of hydrochloric acid present in the given sample can be estimated by titrating with a standard Sodium carbonate solution using methyl orange as an indicator. It is Neutralization (Acid - Base) titration. During titration the following neutralisation reaction takes place.



PART -A: PREPARATION OF STANDARDED SODIUM CARBONATE SOLUTION

Procedure: - About 5.5 grams of Sodium carbonate is placed in a clean, dried weighing bottle. The weighed substance is transferred in to 100 ml volumetric flask. After transferring the substance, weight of the weighing bottle is determined

The substance in the volumetric flask is diluted by adding distilled water up to the mark. The solution is then thoroughly mixed to get homogeneous solution

PART -B: ESTIMATION OF HYDROCHLORIC ACID

Procedure:-10 ml of standard sodium carbonate solution is taken in a clean conical flask, 2-3 drops of methyl orange indicator are added and then the solution is titrated against the hydrochloric acid solution present in the burette till the colour changes from orange yellow to orange red. It is the end point. The titration is repeated until two successive constant values are obtained. The results are reported in a neat tabular form.

Result: - Amount of hydrochloric acid present in the given 100ml of the hydrochloric acid solution =

TABLE-1: STANDADIZATION OF HYDROCHLORIC ACID

S.No	Volume of Std Sodium carbonate solution taken (ml)	Burette Readings		Volume of Hydrochoric acid solution rundown (ml)
		Initial (ml)	Final (ml)	
1				
2				
3				

Std Sodium carbonate solution	Hydrochoric acid solution
N ₁ : Normality of the Std Sodium carbonate solution taken = N	N ₂ : Normality of the Hydrochloric acid Solution rundown = N
V ₁ : Volume of Std Sodium carbonate Solution taken = ml.	V ₂ : Volume of Hydrochloric acid Solution rundown = ml.

$$\text{Normality of hydro choric acid } N_2 = \frac{N_1 \times V_1}{V_2}$$

TABLE -2: ESTIMATION OF THE ALKALINITY

S.No	Volume of water sample taken(V ₃) (ml)	Burette Readings		Volume of Hydrochoric acid solution rundown (ml)
		Initial (ml)	Final (ml)	
1				
2				
3				

Normality of Std. Hydrochloric acid solution N₂ = N
 Volume of Hydrochloric acid consumed = V₃ ml
 Volume of the Water sample taken = V₄ ml

Alkalinity of the given sample

$$= \frac{\text{Normality of HCl Solution (N}_2) \times \text{Vol. of HCl Solution (V}_3) \times \text{Equiv. Wt. of CaCO}_3 (50) \times 1000}{\text{Volume of Water Sample Taken (V}_4)}$$

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Date:

Determination of Alkalinity of a sample containing Na_2CO_3 and NaOH

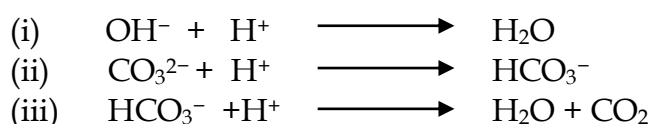
AIM: - To estimate the alkalinity of the given water sample containing Na_2CO_3 and NaOH

Requirements: - 1) Std Sodium Carbonate Solution 2) Hydro chloric acid 3) water sample

Indicator:- Methyl orange

Theory:-

. Alkalinity of the water sample can be estimated by titration of the water sample against a standard hydrochloric acid solution methyl orange as indicator. This is a Neutralization Titration and it involves the following chemical reactions.



The titration of the water sample against a standard acid solution using methyl orange indicator marks the completion of all the above three reactions.

The acid solution is to be standardized with standard sodium carbonate solution using methyl orange indicator.

PART - A: Standardisation of hydrochloric acid solution:

10 ml of the standard sodium carbonate solution is pipetted out in to a clean conical flask 2-3,drops of methyl orange indicator is added and then the solution is titrated against hydro chloric acid present in the burette until the colour changes from orange yellow to orange red. It indicates the end point. The titration is repeated until two successive constant values are obtained

PART - B: ESTIMATION OF THE ALKALINITY OF THE GIVEN WATER SAMPLE

10 ml of the water sample is pipette out in to a clan conical flask . 2-3,drops of methyl orange indicator is added and then the solution is titrated against hydro chloric acid present in the burette until the colour changes from orange yellow to orange red. It indicates the end point. The titration is repeated until two successive constant values are obtained

Result:- Alkalinity of the given water sample =

Preparation of the standard Oxalic acid solution

W_1 = Weight of the weighing bottle + substance =

W_2 = Weight of the weighing bottle after transferring the substance =

Weight of the substance transferred ($W_1 - W_2$) =

$$\text{Normality of the oxalic acid solution} = \frac{\text{Weight of the substance} \times 1000}{\text{Gram equivalent weight of Oxalic acid} \times 100}$$

Gram equivalent weight of Oxalic acid = 63.03 gms

DETERMINATION OF POTASSIUM PERMANGANATE

S. No.	Volume of Std. oxalic acid Solution taken (ml.)	Burette Readings		Volume of potassium permanganate Solution rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				
3				
4				

Std Oxalic acid solution	Potassium permanganate solution
N_1 : Normality of Std oxalic acid Solution = N	V_2 : Volume of potassium permanganate solution = ml.
V_1 : Volume of Std oxalic acid solution = ml.	N_2 : Normality of potassium permanganate Solution = ...?... N

$$\text{Normality of potassium permanganate Solution } N_2 = \frac{N_1 \times V_1}{V_2}$$

=

Amount of the Mn(II) present in given 100 ml. of the $KMnO_4$ Solution

$$\frac{\text{Normality of } KMnO_4 \text{ Solution } (N_2) \times \text{Equivalent Weight of Mn } (27.5) \times 100}{1000}$$

=

=gms

Expt No:

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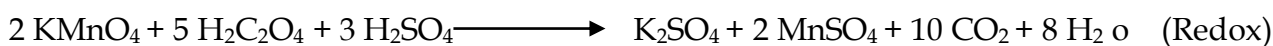
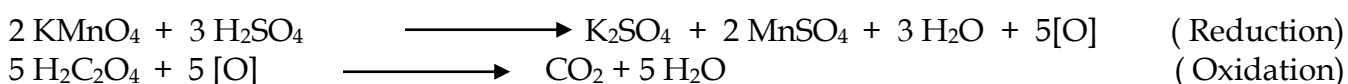
. Determination of Mn (II) using standard Oxalic acid solution

AIM: - To determine the normality and estimate the amount Mn (II) present in the given 100 ml of the solution by using Oxalic acid solution.

Chemicals required: 1) Oxalic acid solution 2) Potassium permanganate solution
3) 6N Sulphuric acid

Indicator: Potassium permanganate (self indicator)

THEORY: - The amount of Mn (II) present in the given sample can be estimated by titration with a Standard Oxalic acid solution. It is a redox type of titration. as it involves both reducing agent and oxidising agent. Here Mn (II) solution acts as an oxidizing agent and oxalic acid act as reducing agent.



Role of sulphuric acid is to provide acidity. Here, no indicator is used because potassium permanganate solution itself acts as a self-indicator.

PART -A: Preparation of the standard Oxalic acid solution

Procedure: - About 6.5 grams of oxalic acid is placed in a clean, dried weighing bottle. The weighed substance is transferred in to 100 ml volumetric flask. After transferring the substance, weight of the weighing bottle is determined

The substance in the volumetric flask is diluted by adding distilled water up to the mark. The solution is then thoroughly mixed to get homogeneous solution

PART B: DETERMINATION OF POTASSIUM PERMANGANATE

10 ml of the Standard oxalic acid solution is pipetted out in to a clean conical flask, 5 ml of 6N sulphuric acid is added to this solution and then the solution is heated to 60 - 70°C. Now the hot solution is titrated against potassium permanganate present in the burette drop by drop till to get permanent pale pink colour. It is the end point. The titration is repeated until two successive constant values are obtained.

Result:- Amount of the Mn (II) present in the given 100ml of the KMnO₄ solution =

Preparation of the standard Potassium dichromate solution

W_1 = Weight of the weighing bottle + substance =

W_2 = Weight of the weighing bottle after transferring the substance =

Weight of the substance transferred ($W_1 - W_2$) =

$$\text{Normality of the potassium dichromate solution} = \frac{\text{Weight of the substance} \times 1000}{\text{Gram equivalent weight of } K_2Cr_2O_7 \times 100}$$

Gram equivalent weight of $K_2Cr_2O_7$ = **49.03 gms**

ESTIMATION OF FERROUS IRON

S. No.	Volume of Ferrous iron sample Solution Taken (ml.)	Burette Readings		Volume of Standard Potassium Dichromate Solution Rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				
3				
4				

Std. Potassium Dichromate Solution
N_1 : Normality of Std. Potassium Dichromate solution = N
V_1 : Volume of Std. potassium dichromate Solution Rundown =ml

Ferrous iron sample
N_2 : Normality of Iron sample Solution = ?.. N
V_2 : Volume Iron ore sample Solution takenml

$$\text{Normality of the Ferrous Iron sample Solution} = \frac{N_1 \times V_1}{V_2}$$

$$= \dots\dots\dots N$$

Amount of Ferrous iron present in 100 ml. of the given Mohr's Salt Solution

$$= \frac{\text{Normality of Iron sample Solution } (N_2) \times \text{Equiv. weight of iron}(55.85) \times 100}{1000}$$

=

= **gms**

Expt.No:

Date;

ESTIMATION OF FERROUS IRON - DICHROMETRY

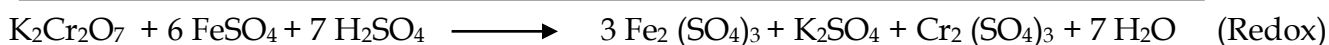
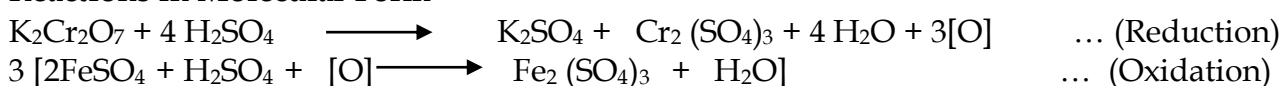
Aim:- To determine the normality and estimate the amount ferrous iron present in the given 100 ml ferrous iron sample by using Standard $K_2Cr_2O_7$ solution.

Chemicals required:- 1). Std potassium dichromate solution 2). Mohr's salt solution
3). Sulphuric acid 4). Phosphoric acid

Indicator: - Di-phenyl Amine

THEORY:-The amount of ferrous iron present in the given sample can be estimated by titrating against a standard $K_2Cr_2O_7$ solution. It is a redox type of titration. In this titration chromium (VI) Solution act as an Oxidizing agent and Mohr's salt solution act as reducing agent.

Reactions in Molecular Form



Role of sulphuric acid, in this titration is to provide acidity. Indicator used is the diphenyl amine. The titration of iron with diphenyl amine indicator must be performed in the presence of phosphoric acid because phosphoric acid increases the efficiency of the indicator

PART -A: Preparation of the standard Potassium dichromate solution

About 0.49 grams of potassium dichromate acid is placed in a clean, dried weighing bottle. The weighed substance is transferred in to 100 ml volumetric flask. After transferring the substance, weight of the weighing bottle is determined

The substance in the volumetric flask is diluted by adding distilled water up to the mark. The solution is then thoroughly mixed to get homogeneous solution

PART B: - ESTIMATION OF FERROUS IRON

The given unknown Mohr's Salt Solution present in the volumetric flask is diluted up to the mark by adding distilled water and then the solution is thoroughly mixed in order to get homogeneous solution. 10 ml of the standard Mohr's salt solution is pipetted out in to a clean conical flask. 10 ml of dilute sulphuric acid, 5 ml of phosphoric acid and 2-3 drops of diphenyl amine indicator are added to the Mohr's salt solution present in the conical flask. This colourless solution is titrated against the potassium dichromate solution, taken in the burette until to get green colour, bluish green colour and finally bluish violet colour at the end point.

RESULT :-Amount of ferrous iron present in the given 100 ml of the solution

= gms

TABLE 1:-STANDARDISATION OF SODIUM THIOSULPHATE SOLUTION

S. No.	Volume of Std. copper Sulphate Solution Taken (ml.)	Burette Readings		Volume of Sodium Thiosulphate Solution rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				
3				

Std. potassium dichromate Solution
N_1 : Normality of Std. Copper sulphate Solution = N
V_1 : Volume of Std Copper Sulphate Solution =ml

Sodium Thiosulphate Solution
N_2 : Normality of Sodium Thiosulphate Solution =?... N
V_2 : Volume of Sodium Thiosulphate Solution =ml

Normality of the Sodium Thiosulphate Solution $N_2 = \frac{N_1 \times V_1}{V_2}$

=

TABLE 2:- ESTIMATION OF COPPER

S. No.	Volume of Copper Sulphate Solution Taken (ml.)	Burette Readings		Volume of Std. Sodium Thiosulphate Solution rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				
3				

Std. Sodium Thiosulphate Solution
N_3 : Normality of Std. Sodium Thiosulphate Solution = ...
V_3 : Volume of Std. Sodium Thiosulphate Solution =ml

Unknown Copper Sulphate solution
N_4 : Normality of unknown copper Solution = ... N
V_4 : Volume of unknown copper Solution =

Normality of the Copper Sulphate Solution $N_4 = \frac{N_3 \times V_3}{V_4}$

=

Amount of Copper present in 100 ml. of the given Copper Sulphate Solution

= $\frac{\text{Normality of Copper Sulphate Solution } (N_4) \times \text{Equivalent weight of Copper } (31.8) \times 100}{1000}$

1000

= g

Expt.No:

Date;

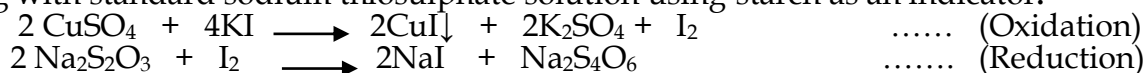
Determination of copper (II) using standard Hypo solution

AIM:- To determine the normality and estimate the amount of copper present in the given 100 ml of the copper solution by using hypo solution

Chemicals required:- 1).Std Copper sulphate solution 2) Sodium thiosulphate solution
4) Potassium iodide 4) Unknown copper solution

Indicator: - Starch

Theory:-The amount of copper present in the given unknown copper solution can be estimated by titrating with standard sodium thiosulphate solution using starch as an indicator.



The above equations show that each copper ion (Cu^{++}) gains an electron from the iodide ion (I^-) and is reduced to mono valent cuprous ion (Cu^+), which is then precipitated in the form of the sparingly soluble cuprous iodide (Cu I) with liberation of copper equivalent amount of iodine. The liberated free iodine is then titrated against the standard sodium thiosulphate solution, using starch as an indicator. The sodium thiosulphate solution must be standardized by titrating it against standard copper sulphate solution using starch as indicator.

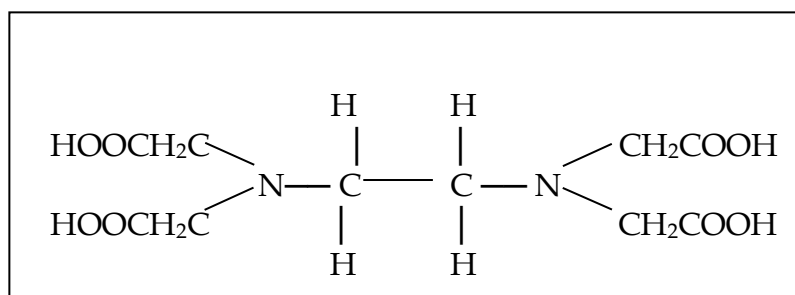
PART A:-STANDARDISATION OF SODIUM THIOSULPHATE SOLUTION

10 ml of the Standard Copper sulphate solution is pipetted out in to a clean conical flask. 10 ml. of 10% potassium iodide solution is added to this solution. The flask is covered with a watch glass for about 2-3 minutes in order to prevent the escape of the liberated iodine. Then, the watch-glass is removed out and then the solution is titrated against sodium thiosulphate solution present in the burette until the colour changes to pale yellow. At this stage add two ml of starch indicator. As it results the colour changes to blue. Continue the titration with sodium thiosulphate present in burette until the blue colour disappears. It indicates the end point. The titration is repeated until two successive constant values are obtained

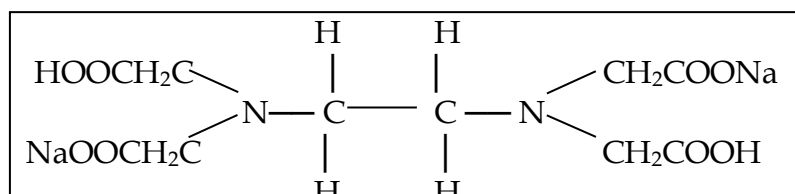
PARTB:- ESTIMATION OF COPPER

The given unknown Copper solution present in the volumetric flask is diluted up to the mark by adding distilled water and then the solution is thoroughly mixed in order to get homogeneous solution. 10 ml of this solution is pipette out in to a clean conical flask, 10 ml. of 10% potassium iodide solution is added to the copper solution. The flask is covered with a watch glass for about 2-3, minutes in order to complete the liberation of iodine. Then, the watch-glass is removed out and then the solution titrated against sodium thiosulphate present in the burette until the colour changes from reddish brown to pale yellow. At this stage add two ml of starch indicator. As it results the colour changes to blue. Now the solution is titrated against sodium thiosulphate present in burette until the blue colour disappears. It indicates the end point. The titration is repeated until two successive constant values are obtained

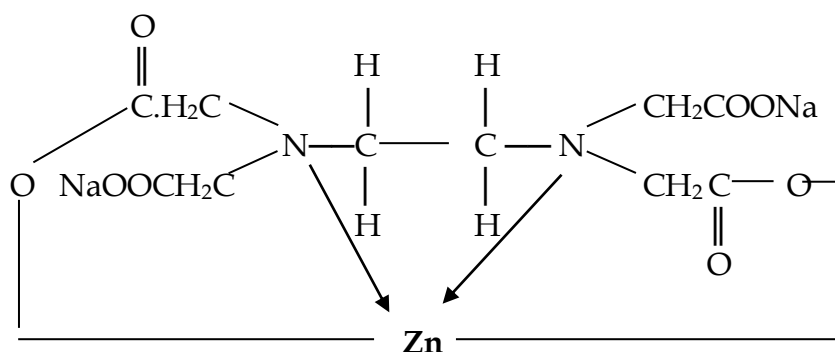
Result:- Amount of the copper present in the given 100ml of the sample =



Structure of E.D.T.A.(H₄EDTA)



Disodium Salt of E.D.T.A. (Na₂H₂EDTA)



METAL EDTA COMPLEX

TABLE: 1-STANDARDISATION OF E. D. T. A. SOLUTION

S. No.	Volume of Std. Zinc Sulphate Solution Taken (ml.)	Burette Readings		Volume of E. D. T. A. Solution rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				
3				

Std. Zinc Sulphate Solution
N ₁ : Normality of Std. Zinc Sulphate Solution =N
V ₁ : Volume of Std. Zinc Sulphate Solution =ml

E. D. T. A. Solution
N ₂ : Normality of E. D. T. A. Solution =?...N
V ₂ : Volume of E. D. T. A. Solution =ml

Normality of the E. D. T. A. Solution, N₂ = $\frac{N_1 \times V_1}{V_2}$

=N

Expt.No :

Date :

DETERMINATION OF TEMPORARY AND PERMANENT HARDNESS

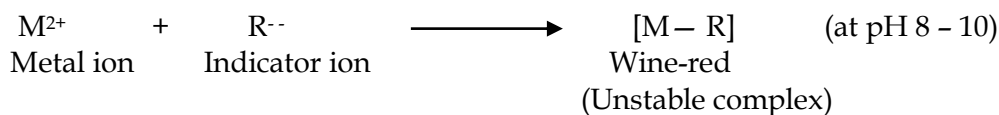
AIM:-To determine the temporary and permanent hardness of the given water sample by using the E.D.T.A. solution.

Chemicals required: - 1) Std Zinc solution 2) E.D.T.A. solution 3) (NH₄Cl + NH₄OH) Buffer solution 4) Water sample.

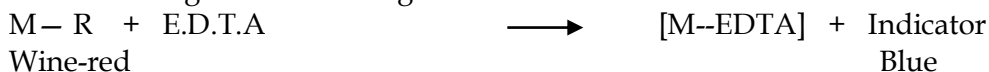
Indicator: - Eriochrome Black-T

THEORY: - Total hardness is the sum of temporary hardness and permanent hardness. Temporary hardness is due to bicarbonates of calcium and magnesium. The permanent hardness is due to chlorides and sulphates of calcium and magnesium. The total hardness of the given water sample can be estimated by titration with standard E.D.T.A. solution using Eriochrome Black-T as an indicator. It is a complexometric titration as it involves the formation of a soluble complex.

In this determination, the water sample is made as alkaline by the addition of (NH₄OH + NH₄Cl) buffer solution in order to maintain the pH in the range 10, because the complex formation takes place at this pH only. When indicator is added to the buffered water sample, then it forms less stable metal - indicator complexes, which are wine red in colour.



When the wine-red coloured solution is titrated with a standard E.D.T.A. solution, then the less stable Metal-indicator [M - R] complexes are decomposed to more stable Metal - EDTA complexes, with the liberation of the indicator ions (R⁻) in the solution. At the equivalence point, the wine-red colour of the solution changes to blue owing to accumulation of the indicator ions.



The E.D.T.A. solution can be standardized using standard zinc solution and Eriochrome Black-T indicator.

PART A:-STANDARDISATION OF E. D. T. A. SOLUTION

10 ml of standard zinc solution is pipetted out into a clean conical flask. 2 ml of the buffer solution and 3-4 drops of the of the E.B.T. indicator are added. The solution is now titrated against the E.D.T.A. solution until the colour of the solution is changed from wine red to blue at the end point. The titration is repeated until two successive constant values are obtained.

PART B:-ESTIMATION OF TOTAL HARDNESS

10 ml of the water sample is pipette out into a clean conical flask. 2 ml of the buffer solution and 3-4 drops of the of the E.B.T. indicator are added. The solution is now titrated against the std E.D.T.A. solution until the colour of the solution is changed from wine red to blue at the end point. The titration is repeated until two successive constant values are obtained.

TABLE-2:-ESTIMATION OF TOTAL HARDNESS

S. No.	Volume of Water Sample Taken (ml.)	Burette Readings		Volume of Std. E. D. T. A. Solution rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				
3				

Water Sample
V ₃ : Volume of Water Sample =ml

Std. E. D. T. A. Solution
N ₄ : Normality of Std. E. D. T. A. Solution =N
V ₄ : Volume of Std. E. D. T. A. Solution =ml

Total Hardness of the given Water Sample

$$= \frac{\text{Normality of E.D.T.A. Solution}(N_4) \times \text{Vol. of E.D.T.A. Solution } (V_4) \times \text{Equiv. Wt. of CaCO}_3 (50) \times 1000}{\text{Volume of Water Sample Taken } (V_3)}$$

=

= ----- P.P.M

TABLE-3:-ESTIMATION OF PERMANENT HARDNESS

S. No.	Volume of Filtrate Water Sample Taken (ml.)	Burette Readings		Volume of Std. E. D. T. A. Solution rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				

Water Sample
V ₅ : Volume of Water Sample =ml

Std. E. D. T. A. Solution
N ₄ : Normality of Std. E. D. T. A. Solution =N
V ₅ : Volume of Std. E. D. T. A. Solution =ml

Permanent Hardness of the given Water Sample

$$= \frac{\text{Normality of E.D.T.A. Solution}(N_4) \times \text{Vol. of E.D.T.A. Solution } (V_5) \times \text{Equiv. Wt. of CaCO}_3 (50) \times 1000}{\text{Volume of Water Sample Taken } (V_4)}$$

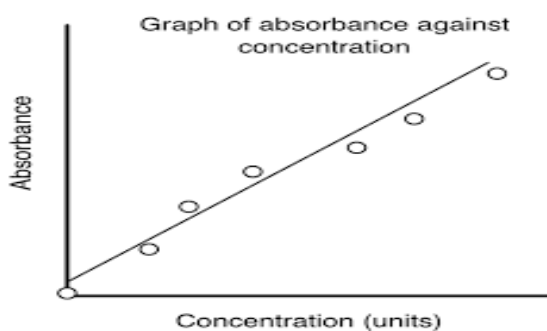
=

= ----- P.P.M

Observation Table:-

S.No	Concentration of Fe ³⁺ (PPM)	Absorbance/Optical density
1		
2		
3		
4		
5		
6		
7		

Standard calibration curve



Beer-Lambert Law

$$A = \epsilon c l$$

- A** = absorbance (no units).
- ϵ** = Molar extinction coefficient ($M^{-1} cm^{-1}$).
- c** = Concentration (M).
- l** = pathlength (cm).

Result:- The concentration of Fe³⁺ in the given sample =

Expt.No:

Date:

DETERMINATION OF IRON BY A COLORIMETRIC METHOD

AIM: - To determine the amount of Iron present in the given 100 ml of the Iron solution by a calorimetric method using thiocyanate as reagent.

Apparatus:- Spectrophotometer, Burette, Pipette, Cuvette

Chemicals required: - 1) Stock iron solution ,2). 20%Pottasium thiocyanate solution,
3) 4M HNO₃

Theory: When a monochromatic light of intensity I_0 is incident on a transparent medium, a part I_a is absorbed, a part of I_r is reflected and the remaining part I_t is transmitted.

$$I_0 = I_a + I_r + I_t$$

For a glass - air interface I_r is negligible. Therefore

$$I_0 = I_a + I_t$$

$I_t / I_0 = T$ called Transmittance. $\log I_0 / I_t$ is called the absorbance or optical density.

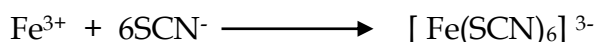
The relationship between absorbance A , Concentration C (expressed in mol/lit) and path length t (expressed in cm) is given by Beer- Lambert's law

$$A = \log I_0 / I_t = E c t$$

Where e is the molar extinction coefficient, E is a constant for a given substance at a given wave length. If the path length is (cell thickness) kept constant, then $A \propto c$.

Hence a plot of absorbance against concentration gives a straight line as shown in figure (Calibration curve). The colour is measured using a Spectrophotometer at a wave length where the absorbance is maximum.

Ferric iron forms a red coloured complex with thiocyanate ion, $[\text{Fe}(\text{SCN})_x]$ in acid medium. Beer- Lambert's law obeyed in dilute solution. The colour of the complex is not stable and hence should be measured within 5 minutes after producing colour. The optical densities are measured by a photocolorimeter and the concentration of ferric iron is obtained from a standard curve.



Procedure:-

Preparation of the standard calibration curve.

Dissolve the given ferrous ammonium sulphate in 100ml of water and 5ml of 1:5 H₂SO₄ dilute the solution through KMnO₄ present in the burette until light pink colour appears. Dilute the solution to 1 litre such that 1 ml of solution contains 0.1 mg of Fe³⁺. From the above solution take separately 1, 2, 3, 4, 5 ml in to five 100ml standard volumetric flasks. Add 1 ml of 4M nitric acid and 5 ml of 40% potassium thiocyanate solution to all the samples to get blood red colour and made up to the mark by adding distilled water. Now measure the optical densities of all five samples using Photo colorimeter. Plot a graph by taking amount of ferrous iron on X - axis and optical densities on Y- axis. The curve obtained is called calibration curve. Concentration of the sample is determined from the graph.

Table:-Determination of the P^H of the given solution of 50 ml 0.1N CH₃COOH with 0.1 N NaOH

S.No	Volume of the sodium hydroxide	P ^H
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

P^H of the given sample solution is =

Expt.No:-

Date:-

Determination of the concentration of acetic acid using Sodium hydroxide

pH metry method

Aim: - To determine the strength of the acetic acid solution by titrating against NaOH solution by using pH meter.

Requirements: - Beakers (500ml), Burette, Glass electrode, Reference electrode, Stirrer
Chemicals:- 0.1N NaOH solution ; 0.1N acetic acid , Buffer Tablets pH 4 & 7. Distilled water

Theory: - The concentration of solutions expressed in terms of normality. However to express very small concentrations of H⁺ ions, pH scale is used. pH can be measured directly with the help of pH meter. The acidic, alkaline or neutral nature of solution depends on the pH value. pH is defined as negative logarithm of hydrogen ion concentration.

$$pH = -\log H^+$$



Example: Titration of 50.0 mL of 0.100 N CH₃COOH with 0.100 N NaOH.



Equivalence point pH > 7 •

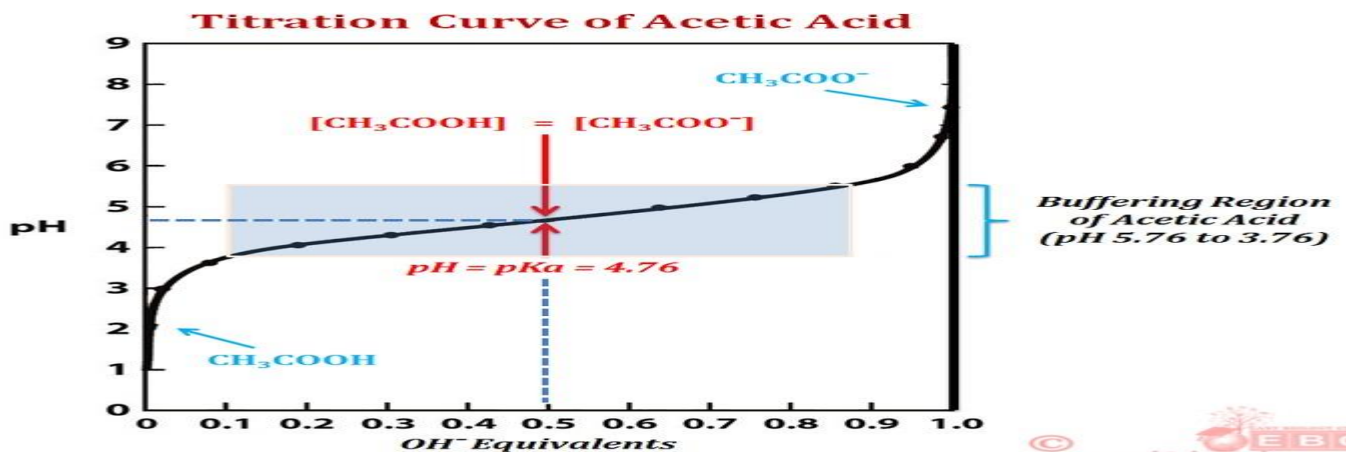
Smaller change in pH near equivalence point •

Before equivalence point the pH is nearly constant (buffer) •

After equivalence point the pH depends on excess of standard base

Procedure: -

1. Glass electrode and reference electrode are washed with water and then dipped in distilled water
2. Turn 'ON' the pH meter & Calibrate the pH meter by using standard buffer solutions of pH 4 & 7.
3. 10 ml of the acid solution is pipetted out in to a clean beaker.
4. Turn 'ON' the pH meter and note down the pH of the solution.
5. Add ml of the NaOH solution and note down the pH of the solution
6. Continue the process by adding NaOH solution and note down the pH of the solution
7. Then concentration of the solution is calculated.

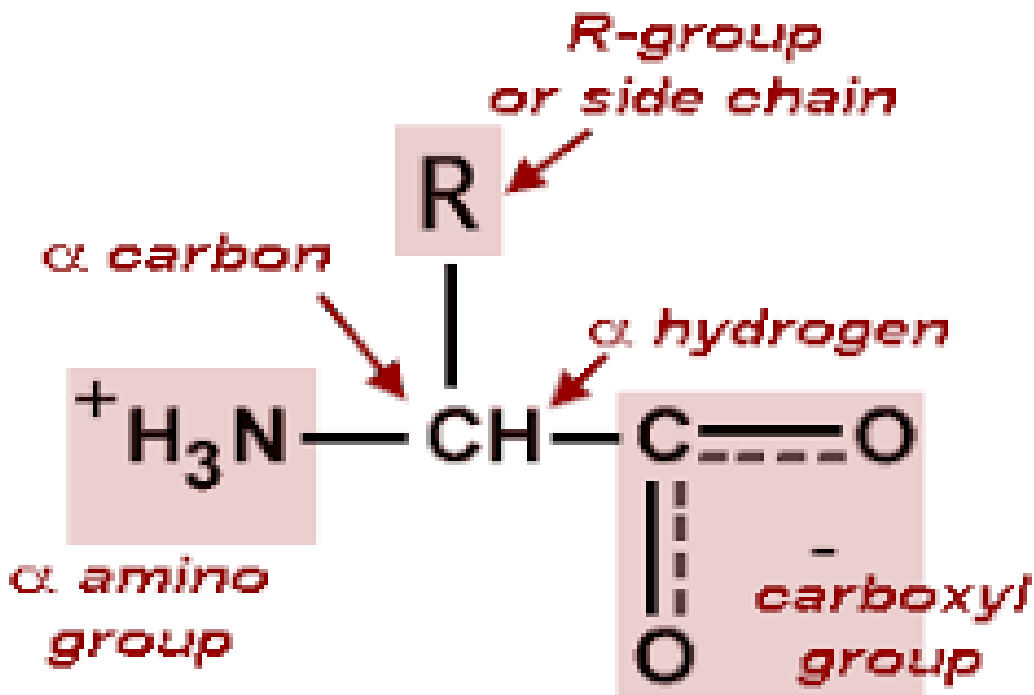


Determination of Isoelectric point of Amino acid by PH metric method

To determine isoelectric point (pI) of Amino acid by Ph metric method

Titration stand, Burette, Ph meter,

Amino acids consist of: a basic amino group ($-\text{NH}_2$) & an acidic carboxyl group ($-\text{COOH}$)
a hydrogen atom ($-\text{H}$), a distinctive side chain ($-\text{R}$).



Amino acids are Amphoteric which means it can react as an acid (donate a proton) as well as a base (accept a proton) •

Amphoteric properties of amino acids are due to the presence of their ionizable α -amino and α -carboxylic group can act sometimes as acids and sometimes as bases depending on the pH of their media. •

pH at which amino acid has no net charge is called isoelectric point. Amino acid at this point has Zwitter ionic form

Each amino acid have a different isoelectric point (pI) •

pI: It is the pH value at which the positive charge equals the negative charge (i.e. the net charge of this molecule equals zero) (zwitter ion)

Amino acids have more than one pKa, because it is polyprotic (contain more than one ionizable groups).

1. At a very low pH (acidic) both groups are fully protonated where the solution predominantly contains:
2. When the pH is raised, the $-\text{COOH}$ group starts to be deprotonated and the proportion will be
3. $\text{pH} = \text{pK}_a1$, where it will act as a buffer and the solution will contain an equal amount of
4. Further increase in pH, the solution will predominantly contain zwitterion and the pH at this point is equal to pI.
5. As the pH increases, the second group $-\text{NH}_3^+$ will be deprotonated
6. After that, $\text{pH} = \text{pK}_a2$ where it will act as a buffer and the solution will contain an equal amount of
7. the NH_3^+ group will dissociate and at the same time the glycine fully dissociates in end point

Procedure

- Pipette 10 ml of glycine solution (0.1 M) into a 50 ml breaker.
 - Add 0.5 ml of (0.1 M) HCl from the burette and determine the pH of the solution after each addition.
 - Continue adding acid until pH falls to about 1.3.
 - Wash the electrode in distilled water titrate a further 10 ml of glycine solution with 0.1 M NaOH until pH reaches 12.5.
 - Plot a titration curve for glycine (pH versus titrant in ml).
 - Record the titration table and Plot a Curve of pH versus ml of NaOH and HCl added.
 - Determine the pKa and pI values from your curves and compare them with the standard values.
 - For glycine: $\text{pK}_1 = 2.34$, $\text{pK}_2 = 9.69$, $\text{pI} = 6.01$.
- Titration curve of amino acid (glycine)

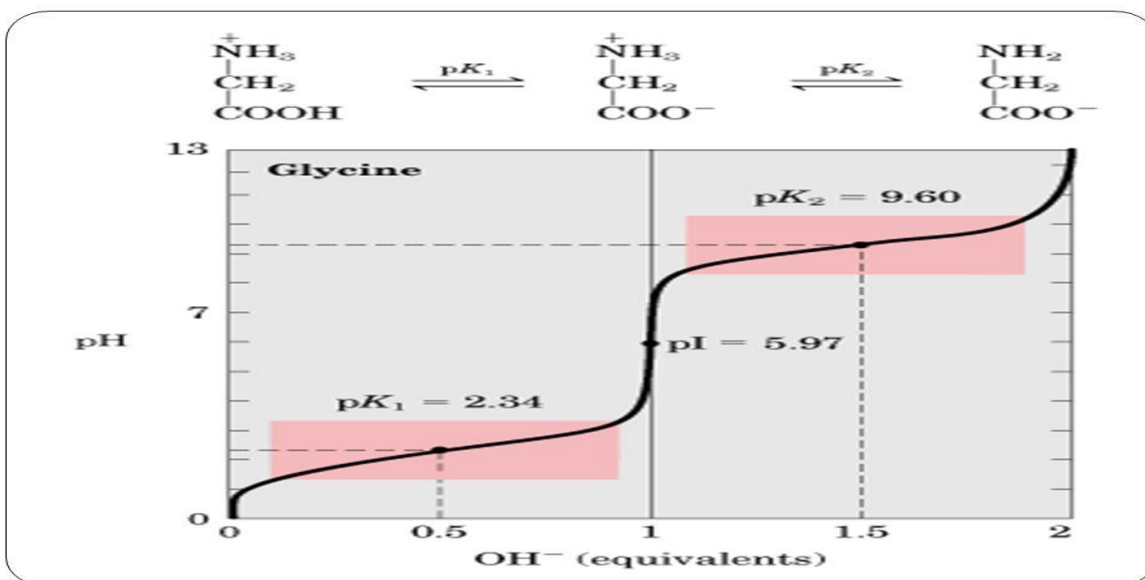


Table:- Titration between HCl and 0.1N NaOH solution.

S.No	Volume of NaOH solution added in ml	Conductance(ms) C	Corrected conductance (50+V)) $C \times \frac{\text{-----}}{50}$
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			

Calculations:

Normality of HCl $N_{HCl} = ?$

Volume of HCl $V_{HCl} = \text{-----}$

Normality of NaOH $N_{NaOH} = 0.1N$

Volume of NaOH from graph $V_{NaOH} =$

Acid Base
 (HCl) (NaOH)

$$N_{HCl} \times V_{HCl} = N_{NaOH} \times V_{NaOH}$$

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

$$= 0.1 \times \text{----} / 10$$

Result:- Normality of the given Hydrochloric acid solution =

Expt.No:-

Date:-

CONDUCTOMETRIC TITRATION STRONG ACID VERSES STRONG BASE

Aim: - To determine the strength of the Hydrochloric acid solution by titrating against NaOH solution conductometrically.

Requirements:- Conductivity meter, Conductivity cell , Beakers (500ml), Burette, pipette, stirrer

Chemicals:- 0.1 NaOH solution ; HCl sample, distilled water,

Theory:-The electrical conductance of the solution depends upon the number of the ions and their mobilities. When sodium hydroxide solution is added to the Hydro chloric acid solution, their conductivities are decreased .It is due to replacement of highly mobile H⁺ ions by less mobile Na⁺ ions. Conductivity keeps on decreasing until to reach the end point. At the equivalent point solution contains Na⁺ and Cl⁻ ions. Further addition of NaOH increases the conductivity, which is due to high momilities of the OH⁻ ions.



Procedure:-

1. Take 10 ml of the given HCl solution in a 100 ml beaker.
Add 40ml of distilled water and stir it.
2. Wash the conductivity cell with distilled water and then rinse it with the given HCl solution. Dip the cell in to the solution taken in a beaker.
3. Initial conductance of the solution is measured.
4. Take 0.1N NaOH solution in a burette and fixed to stand.
5. Add 0.5ml of NaOH into the beaker containg HCl, stir well and measure their conductivity.
6. Continue the procedure by adding 0.5ml of NaOH and note down the corresponding conductivity in the Table.
7. Initially, the conductivity decreases, reaches the minimum value and then increases.
At least 5 or 6 readings must be taken after the minimum value.
9. Plot the graph between conductances on Y- axis Vs volume of NaOH added on X- axis
10. The point of interaction will give the amount of alkali required for neutralization of the acid.

Modal graph

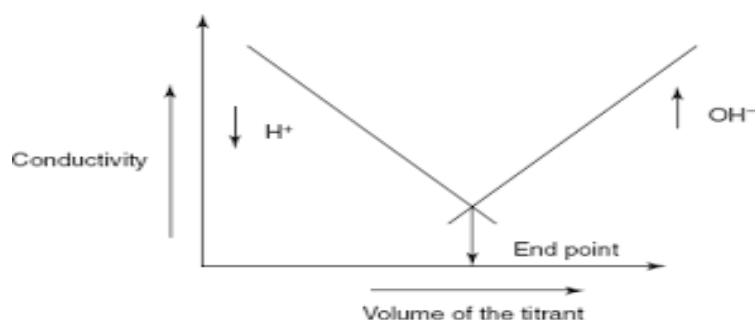


Table:- Potentiometric Titration between Strong Acid Vs weak base

S.No	Volume of the alkali added	EMF (mv) of the cell
01		
02		
03		
04		
05		
06		
07		
08		
09		
10		
11		
12		
13		
14		
15		

Calculations:

Normality of HCl $N_{HCl} = ?$

Volume of HCl $V_{HCl} = \text{-----}$

Normality of NaOH $N_{NaOH} = 0.1N$

Volume of NaOH from graph $V_{NaOH} =$

$$\begin{array}{ccc}
 \text{Acid} & & \text{Base} \\
 \text{(HCl)} & & \text{(NaOH)} \\
 N_{HCl} \times V_{HCl} & = & N_{NaOH} \times V_{NaOH}
 \end{array}$$

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

$$= 0.1 \times \text{----} / 10$$

Result:- Normality of the given Hydrochloric acid solution =

Expt.No:-

Date:-

POTENTIOMETRIC TITRATION BETWEEN STRONG ACID AND STRONG BASE

Aim: - To determine the strength of the Hydrochloric acid solution by titrating against NaOH solution potentiometrically.

Requirements:- potentiometer, calomel and platinum electrodes, Beakers (500ml), Burette, pipette, stirrer

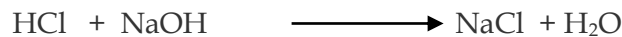
Chemicals:- 0.1 NaOH solution ; HCl sample solution , distilled water

Theory:- Potentiometric titration is a volumetric method in which the potential between two electrodes are measured as a function of added reagent volume. In this titration, indicator electrode (the electrode which responds to change in concentration of ions in solution) is combined with reference electrode to form a complete cell. During the titration EMF of the cell is measured.

$$\text{EMF of the cell} = E_{\text{indicator}} - E_{\text{reference}} + E_{\text{solution}}$$

As the concentration of the ions (H^+ or OH^-) around the electrode changes, E_{cell} also changes correspondingly in acid base titrations.

The EMF of the cell changes gradually till the end point and rapidly at very close to the end point and then gradually changes after the end point.



Procedure:-

1. The 10ml of the HCl solution is pipette out in to a clean 100ml beaker provided with a stirrer.
2. The platinum and calomel electrodes immersed in the solution are connected to negative and positive terminals of the potentiometer.
3. Initial EMF of the solution is noted.
4. 0.1N NaOH solution is taken in burette and added 0.5 ml additions to the beaker and the corresponding potential is noted after stirring.
5. Continue the titration by adding 0.5ml of alkali solution. Take three to four readings after the equivalent point. Record the volume of alkali added and EMF of the solution.
6. The same experiment is repeated with addition of 0.1ml of NaOH solution.
7. A graph is plotted between the emf or E/ V on Y- axis and the volume of the solution on X - axis.

The maximum of the curve obtained from the graph represents the end point

Model graph

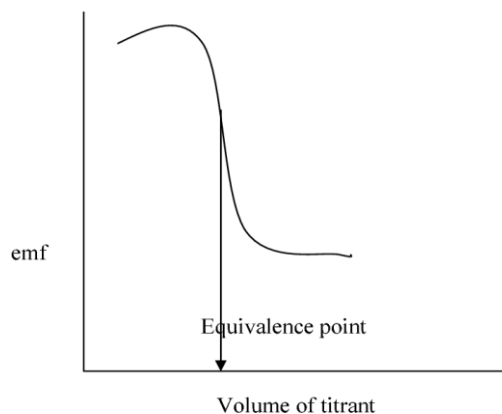


Table:- Titration of the egg solution with NaOH solution

S.No	Volume of eggshell solution taken (ml)	Burette readings		Volume of NaoH rundown (ml)
		Initial (ml)	Final (ml)	
01				
02				
03				
04				

Volume of eggshells solution taken = 25 ml				
Volume of NaOH used =				

Expt.No:-

Date:-

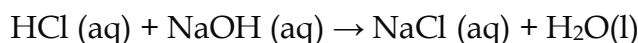
Determination of Calcium carbonate in eggshells - Back Titration

The major component of eggshells is calcium carbonate (CaCO_3). This analysis is done by reacting the calcium with acids. Calcium carbonate is insoluble in pure water but will dissolve in acid.



Back Titration Technique

This reaction cannot be used directly to titrate the CaCO_3 because it is very slow when the reaction is close to completion (endpoint). Instead, the determination is achieved by adding an excess of acid to dissolve all of the CaCO_3 and then titrating the remaining excess HCl with NaOH solution to determine the amount of acid that did not react with the calcium carbonate. The difference between amounts of the acid (HCl) initially added and the amount left over after the reaction is equal to the amount that is used by the CaCO_3 . From this, the amount of CaCO_3 in the sample can be calculated. The reaction used to determine the leftover acid is



Method

1. Wash the egg shell with deionized water and peel off all of the membranes from the inside of the shell. If you leave the membranes, the proteins in the membrane will react with the sodium hydroxide and give poor results.
2. Dry the shell for about 10 minutes in the oven and grind the shell to a fine powder.
4. Weigh 1 g of the dried shell and place into flask.
5. Pipet 25 mL of 1 M HCl to the flask containing eggshell.
7. Heat the solution on a hotplate until it just begins to boil and then allow it to cool.
8. Add a few drops of bromophenol blue indicator to the flask. (Bromophenol blue indicator is yellow in acidic solution and blue in basic solution).
9. Titrate the solution with a standardised solution of sodium hydroxide.

The end point is recorded.

Table:-Determination of Vitamin - C

S.No	Volume of Ascorbic acid	Burette readings		Volume of Iodine solution
		Initial	Final	
1	20 ml			
2	20 ml			
3	20 ml			

20 ml of standard vitamin - c required = ml of iodine solution
 100 ml of standard vitamin c contains = 0.250 gms of vitamin c
 20 ml of standard vitamin c contains = ?

$$\text{Amount of vitamin c in 20 ml of standard vitamin c solution} = \frac{0.25 \times 20}{100} = 0.05 \text{ gms}$$

For 0.05 grams of vitamin -C required x ml of iodine solution.

For titration 20 ml of sample solution requires y ml of iodine solution.

$$\text{Amount of vitamin -C in sample solution} = \frac{0.05 \times y}{x} = Z \text{ gms}/100\text{ml}$$

Amount of vitamin c in present in 1000ml of the sample solution = 10 x Z

$$= 10 \times \dots\dots\dots / \text{gms}/1$$

Expt.No:

Date:

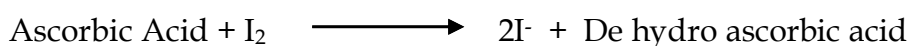
DETERMINATION OF VITAMIN - C

Aim :- Estimation of Vitamin -C (Ascorbic acid)

Chemical required: - Vitamin-c tablet, 0.05 M Iodine solution, KI and Iodine, 3M sulphuric acid

Indicator :- Starch

Theory:- Vitamin-c is also called as ascorbic acid, an important Antioxidant. It can easily Oxidised to de hydro ascorbic acid. When iodine is added during the titration, the acid is oxidised to de hydro ascorbic acid, while the iodine is reduced to Iodide ions.



Iodine is reduced to Iodide as long as Ascorbic acid is present, when all the Ascorbic acid is Oxidised, then the excess iodine is turn to react with starch and forms Blue - black starch Iodide complex. This is the end point of titration.

Sample preparations:-

Vitamin C- :-Dissolve 0.250 gms of Vitamin -c in 100 ml of distilled water.

Iodine Solution:-

Dissolve 2 grams of KI and 1.03 g of Iodine in 200 ml distilled water. Ad 30 ml of 3M sulphuric and made the solution up to 1 litre by adding distilled water.

Standardization of Iodine solution with vitamin C standard solution

Pipette out 20 ml of Vitamin C solution into a 250 ml conical flask .add 10 drops of 1% starch solution. Rinse the burette with iodine solution and fill the burette with iodine solution. Then titrate the vitamin C standard solution with iodine solution till blue back colour is obtained. Let the titre value be x ml.

Estimation of ascorbic acid

Pipette out 20 ml of the sample in to 250 ml conical flask and add 150 ml of distilled water and 1 ml of the starch indicator. Then titrate the sample solution against 0.005 molar Iodine solution present in the burette till persistent Blue - Black colour at the point is due to the formation of starch - iodide complex. Repeat the titration until to get two same values. Let the titre value be y ml.

Then we can calculate how much amount of vitamin-c is present in the given sample.

PHENOL FORMALDEHYDE

Aim: To prepare phenol formaldehyde resin

Requirements:

- (i) **Apparatus:** 500 ml. Beaker(glass), Glass-Rod and Measuring jar.
- (ii) **Chemicals:** Acetic acid(glacial), Formaldehyde, Phenol and Conc. HCl

Theory: Phenolic Resins are the condensation products of phenol or phenolic derivatives and aldehydes. These resins are also called phenol-plasts. Bakelite was the earliest thermosetting resin named after its discoverer, Dr. L.H. Bakeland who patented it in 1909. It was prepared by condensation of phenol and formaldehyde. Today, there are several types of phenol formaldehyde resins which are extensively used in a variety of applications such as water-soluble adhesives, lamination adhesives, varnish and lacquer resins and thermosetting moulding powders. The nature of the product formed depends upon:

- (i) The proportion of the reactants, Phenol and Formaldehyde, and
- (ii) The nature of the catalyst used, i.e. acidic or basic.

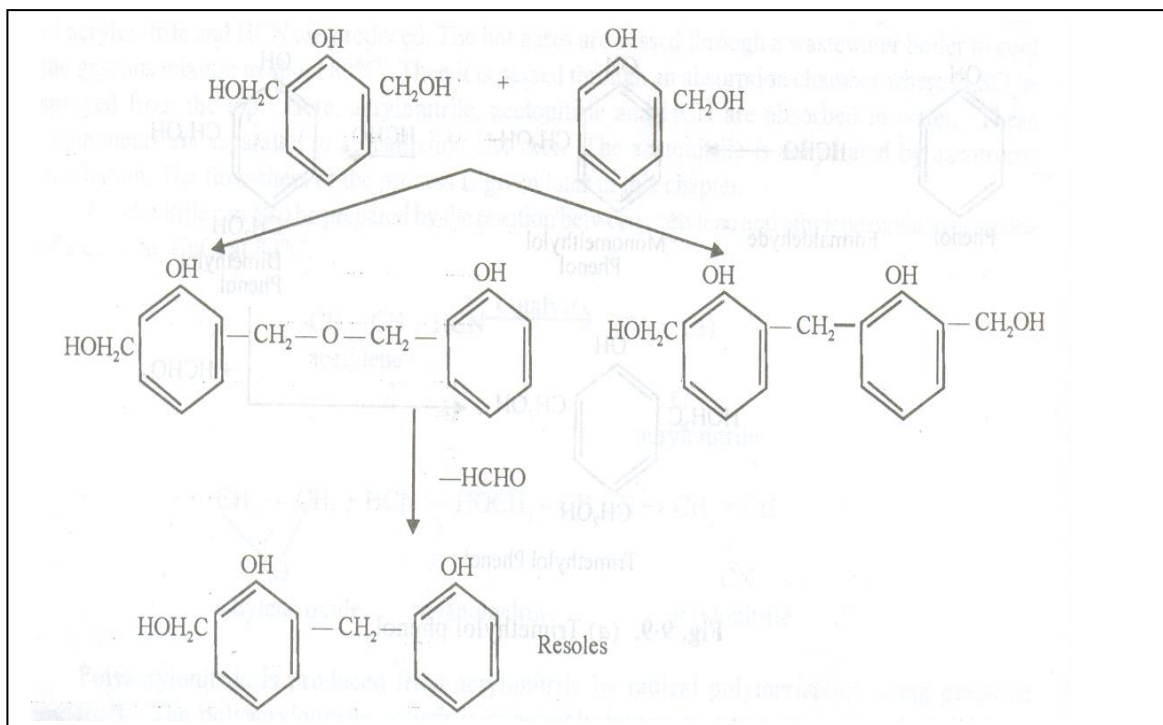
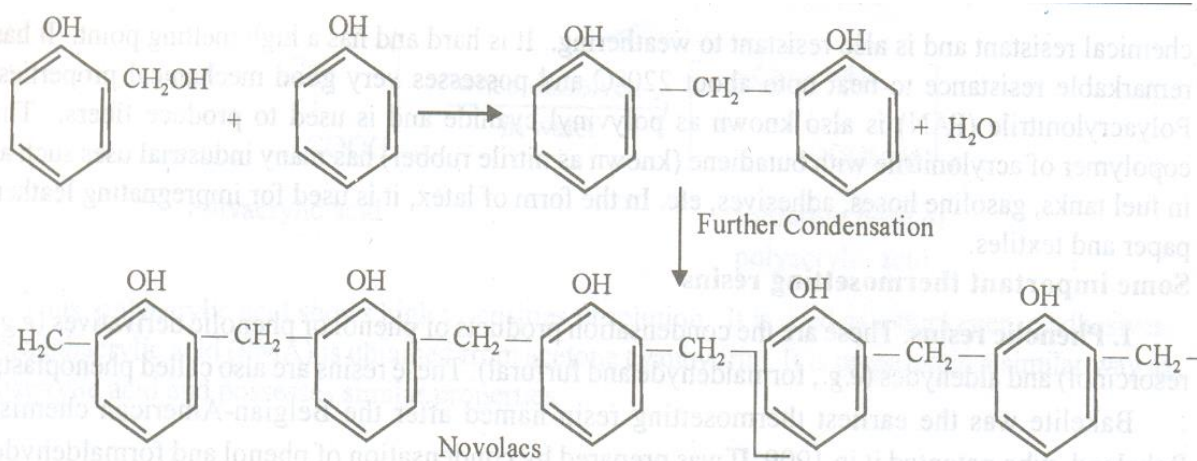
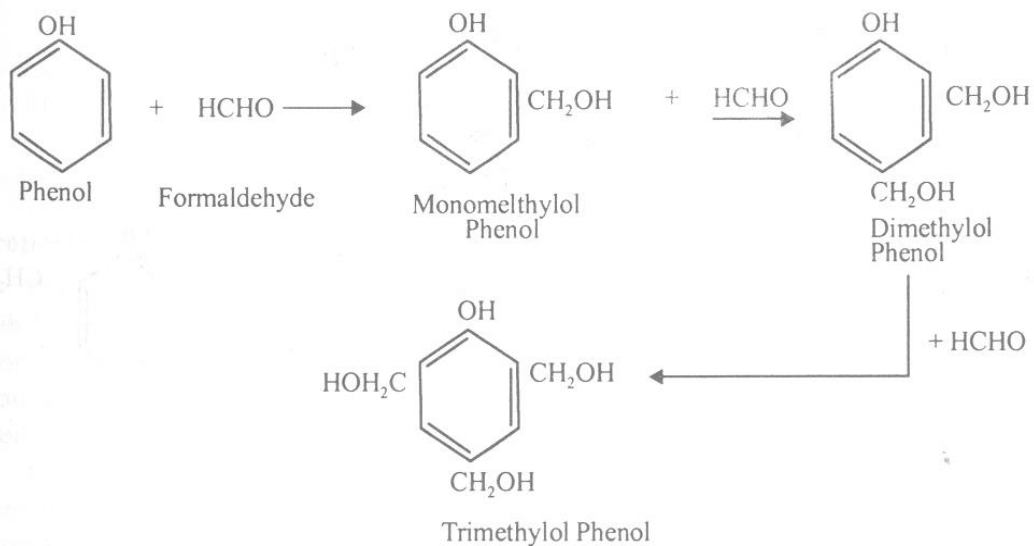
If the mole ratio of phenol to formaldehyde (P/F) is greater than one (if Phenol is in excess), the reaction proceeds in an almost linear fashion. On the other hand, if the mole ratio P/F is lesser than one (if formaldehyde is in excess) and if alkaline catalyst is present, three dimensional structure would be formed.

(a) Methylation: The first step in the reaction between phenol and formaldehyde is the formation of addition compounds known as methylol derivatives; the methylol groups get attached at ortho and para-positions. These methylol derivatives may be considered as the monomers for subsequent polymerisation. These are formed under neutral or alkaline conditions.

(b) Novolac formation: In the presence of an acid catalyst and with the mole ratio of phenol to formaldehyde greater than 1, the methylol derivatives condense with phenol to first form dihydroxy - diphenyl methane(methylene bridge formation). On further condensation, fusible and soluble linear low polymers called "Novolacs" are formed, which have the structure where ortho and para links occur at random.

(c) Resole formation: In the presence of alkaline catalyst and with more formaldehyde (i.e. P/F less than 1), the methylol phenols can condense either through methylene linkages or through other linkages. In the latter case, subsequent loss of formaldehyde may occur with methylene bridge formation. The formation of products of this type which are soluble and fusible but containing alcohol groups are called resoles. If the reactions leading to their formation are carried further, large numbers of phenolic nuclei can condense to give network structure.

PHENOL FORMALDEHYDE



Production of phenolic resins: The formation of resoles and novolacs, respectively, leads to the production of phenolic resins by one-stage and two-stage processes.

One-stage Resin: For producing a one-stage phenolic resin, all the reactants for the final polymer (i.e. phenol, formaldehyde and catalyst) are charged into resin kettle and allowed to react together. The ratio of phenol to formaldehyde is about 1:1.25 and an alkaline catalyst is used.

Two-stage Resins: These resins are prepared with an acid catalyst, and only part of the necessary formaldehyde is added to the kettle, producing a mole ratio of phenol to formaldehyde of about 1:0.8. The rest is added later in the form of hexamethylenetetramine, which decomposes in the final curing step, with heat and moisture present, to yield formaldehyde and ammonia which acts as a catalyst for curing.

Resin formation: The procedures for one-and two-stage resins are similar and the same equipment is used for both. The reaction is exothermic and cooling is necessary. The formation of a resole or a novolac is evidenced by an increase in viscosity. Water is then driven off under vacuum and a thermoplastic A-stage resin, soluble in organic solvents, remains. This is taken out of the kettle and ground to fine powder. At this stage, fillers, colorants, lubricants (and enough hexamethylene tetramine in case of two-stage resin) are added, and the mixture is rolled on heated mixing rolls, where the reactions are carried out further, to give B-stage resin. This is nearly insoluble in organic solvents but still fusible under heat and pressure. The resin is then cooled and cut into final form. The C-stage, the final stage of infusible cross-linked polymer, is reached on subsequent fabrication, e.g. by moulding.

Procedure: Place 5 ml of glacial acetic acid and 2.5 ml of 40% formaldehyde solution in a 500 ml beaker and add 2 grams of phenol. Wrap a cloth or a towel loosely round the beaker. Pass dry HCl gas (or less preferably a few ml of conc. HCl) into the mixture carefully. Within 5 minutes, a large mass of pink plastic is formed.

Precaution: The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while adding the HCl and until the reaction is complete.

TABLE 1:-STANDARDISATION OF SODIUM THIOSULPHATE SOLUTION

S. No.	Volume of Std. potassium dichromate Solution Taken (ml.)	Burette Readings		Volume of Sodium Thiosulphate Solution Rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				

Std. potassium dichromate Solution
N ₁ : Normality of Std. potassium dichromate solution N
V ₁ : Volume of Std. potassium dichromate solutionml

Sodium Thiosulphate Solution
N ₂ : Normality of Sodium Thiosulphate Solution =?... N
V ₂ : Volume of Sodium Thiosulphate Solution Rundown =ml

Normality of the Sodium Thiosulphate Solution, N₂ = _____
= _____

TABLE 2:- ESTIMATION OF CHROMIUM

S. No.	Volume of Chromium (VI) Solution Taken (ml.)	Burette Readings		Volume of Std. Sodium Thiosulphate Solution Rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				

Chromium(VI) Solution
N ₃ : Normality of chromium(VI) Solution = ?...N
V ₃ : Volume of chromium (VI) Solution Taken =ml

Std. Sodium Thiosulphate Solution
N ₄ : Normality of Std. Sodium Thiosulphate Solution = ... N
V ₄ : Volume of Std. Sodium Thiosulphate Solution = ml.

Normality of the Chromium (VI) Solution, N₃ = $\frac{N_4 \times V_4}{V_3}$

Amount of chromium present in 100 ml. of the given chromium (VI) Solution

= $\frac{\text{Normality of chromium (VI) Solution (N}_3\text{)} \times \text{Equivalent weight of chromium (17.33)} \times 100}{1000}$

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Experiments beyond the Syllabus

Expt. No:

Date:

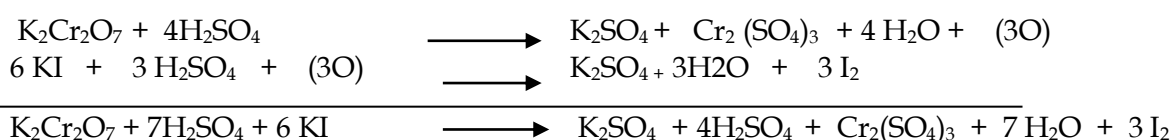
ESTIMATION OF CHROMIUM-- IODOMETRY

AIM:- To estimate the amount of chromium present in the given 100 ml of the chromium (VI) solution by Iodometry method using sodium thiosulphate solution

Chemicals required: -1) Std Potassium dichromate solution 2) Sodium thiosulphate solution
3) Potassium iodide 4) Unknown chromium (VI) solution 5) Dilute Sulphuric acid

Indicator: - Starch

Theory:-The amount of chromium present in the given unknown chromium (VI) solution can be estimated by titrating with standard sodium thiosulphate solution using starch as an indicator by Iodometric method.



The liberated iodine can be estimated by titrating with a standard sodium thiosulphate solution using starch as indicator.



Starch must be added just before the completion of the titration indicated by a faint yellow (straw) colour.

PART A:-STANDARDISATION OF SODIUM THIOSULPHATE SOLUTION

10 ml of the Standard potassium dichromate solution is pipetted out in to a clean conical flask. 10 ml. of 10% potassium iodide solution and 10 ml of dilute sulphuric acid are added to the potassium dichromate solution. The flask is covered with a watch glass for about 2-3 minutes in order to prevent the volatilisation of the liberated iodine and the solution is allowed to stand for 2-3 minutes to complete the liberation of iodine. Then, the watch-glass is removed out and then the solution is titrated against sodium thiosulphate solution present in the burette until the colour changes to pale yellow. At this stage add two ml of starch indicator and continue the titration until the colour changes from blue to green colour. It indicates the end point. The titration is repeated until two successive constant values are obtained

PART B:-ESTIMATION OF CHROMIUM

The given unknown chromium (VI) solution present in the volumetric flask is diluted up to the mark by adding distilled water and then the solution is thoroughly mixed in order to get homogeneous solution. 10 ml of this solution is pipetted out in to a clean conical flask, 10 ml. of 10% potassium iodide solution and 10 ml of dilute sulphuric acid are added to the potassium dichromate solution. The flask is covered with a watch glass for about 2-3, minutes in order to complete the reaction.. Then, the watch-glass is removed out and then the solution titrated against sodium thiosulphate present in the burette until the colour changes to pale yellow. At this stage add two ml of starch indicator and continue the titration until the colour changes from blue to green colour. It indicates the end point. The titration is repeated until two successive constant values are obtained

TABLE-1:-STANDARDISATION OF SODIUM THIOSULPHATE SOLUTION

S. No.	Volume of Std. Copper Sulphate Solution Taken (ml.)	Burette Readings		Volume of Sodium Thiosulphate Solution Rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				

Std. Copper Sulphate Solution	
N_1 : Normality of Std. Copper Sulphate Solution N
V_1 : Volume of Std. Copper Sulphate Solution Takenml l

Sodium Thiosulphate Solution	
N_2 : Normality of Sodium Thiosulphate Solution	= N
V_2 : Volume of Sodium Thiosulphate Solution Rundown	=ml

Normality of the Sodium Thiosulphate Solution, = $\frac{V_1 \times N_1}{V_2}$

=

TABLE 2:-ESTIMATION OF AVAILABLE CHLORINE.

S. No.	Volume of Bleaching powder suspension Taken (ml.)	Burette Readings		Volume of Std. Sodium Thiosulphate Solution Rundown (ml.)
		Initial (ml.)	Final (ml.)	
1				
2				

Bleaching powder suspension	
V_3 : Volume of bleaching powder suspension taken.ml l

Std. Sodium Thiosulphate Solution	
N_4 : Normality of Std. Sodium Thiosulphate Solution	= N
V_4 : Volume of Std. Sodium Thiosulphate Solution Rundown	=ml

Amount of Available Chlorine present in the given Bleaching Powder Solution

= $\frac{\text{Normality of Sod. Thiosulphate Soln}(N_4) \times \text{Vol. of Sod. Thiosulphate Soln}(V_4) \times \text{Eq. Wt. of Chlorine}(35.5) \times 1000}{\text{Volume of Bleaching Powder Solution Taken } (V_3)}$

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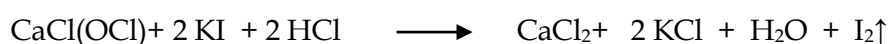
ESTIMATION OF AVAILABLE CHLORINE -- IODOMETRY

AIM: - To estimate the amount of available chlorine present in the given bleaching powder sample by Iodometry method using Sodium thiosulphate solution.

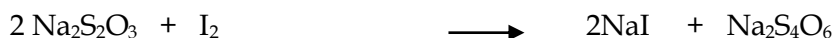
Chemicals required:- 1) Bleaching powder sample 2) copper sulphate solution 3) Sodium Thiosulphate solution 4) Sulphuric acid 5) Potassium iodide.

Indicator:- Starch

Theory:- The 'Available Chlorine' refers to the chlorine liberated by the action of dilute acids on bleaching powder. The available chlorine present in the given bleaching powder is determined by titrating the acidified bleaching powder solution against standard sodium thiosulphate solution using starch as indicator by the iodometry method.

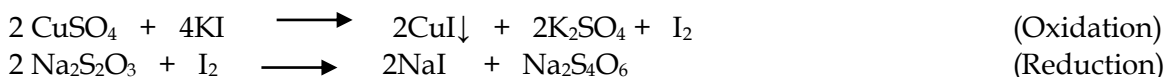


The liberated iodine, which is equivalent to the amount of available chlorine in the bleaching powder, is titrated with standard sodium thiosulphate solution using starch as indicator.



The given sodium thiosulphate solution can be standardized with standard copper sulphate solution using starch indicator.

The given sodium thiosulphate solution can be standardized with standard copper sulphate solution using starch indicator.



PART A: STANDARDISATION OF SODIUM THIOSULPHATE SOLUTION

10 ml of the Standard copper solution is pipetted out in to a clean conical flask. 10 ml. of 10% potassium iodide solution are added to the copper solution. The flask is covered with a watch glass for about 2-3, minutes in order to prevent the escape of the liberated iodine and then the solution is allowed to stand for 2-3 minutes in order to complete the reaction. Then, the watch-glass is removed out and then the solution titrated against sodium thiosulphate present in the burette until the colour changes to pale yellow. At this stage add two ml of starch indicator and continue the titration until the blue colour changes from blue to green colour. It indicates the end point. The titration is repeated until two successive constant values are obtained

PART B:-ESTIMATION OF AVAILABLE CHLORINE

10 ml of this bleaching powder suspension is pipetted out in to a clean conical flask, 10 ml of dilute HCl and 10 ml. of 10% potassium iodide solution are added to this solution. The flask is covered with a watch glass for about 2-3, minutes in order to complete the reaction. Then, the watch-glass is removed out and then the solution titrated against sodium thiosulphate present in the burette until the colour changes to pale yellow. At this stage add two ml of starch indicator and continue the titration until the blue colour disappears. It indicates the end point. The titration is repeated until two successive constant values are obtained.

VIVA -- VOCE QUESTIONS VIVA -- VOCE QUESTIONS

1. What is a Titration?
2. Define end point?
3. What is an Indicator?
4. Name the various types of Titrations.
5. What is a neutralization titration?
6. What is a redox titration?
7. What is complexometric titration?
8. What is precipitation titration?
9. What is oxidation reaction?
10. What is reduction reaction?
11. What is an Oxidizing agent?
12. What is reducing agent?
13. What is acidimetry method?
14. What is an alkalimetry method?
15. Define permanganometry?
16. Give the colour of Phenolphthalein indicator in acidic solution.
17. Give the colour of Phenolphthalein indicator in alkali solution.
18. Give the colour of Methyl orange indicator in acidic solution.
19. Give the colour of Methyl orange indicator in alkali solution.
20. Give the indicator suitable for the estimation of sodium Hydroxide by Hydro Chloric acid.
21. Give the indicator suitable for the estimation of sodium Carbonate by Hydro Chloric acid.
22. Give the equivalent weight of Sodium Hydroxide.
23. Name the oxidizing agent & reducing agent in the estimation of oxalic acid by KMnO_4 .
24. Write the redox equation in the estimation of oxalic acid by Potassium permanganate.
25. What is the indicator used in permanganometric titrations?
26. What is self indicator?
27. Why oxalic acid is heated to $60 - 70^\circ\text{C}$ in the estimation of oxalic acid by Potassium permanganate.
28. What is the colour at the end point in Permanganometric titrations?
29. Give the molecular weight & equivalent weight of Oxalic acid.
30. Write the name, formula & Molecular weight of Mohar's salt?
31. Name the oxidizing agent & reducing agent in the estimation of Iron by Potassium permanganate.
32. Write the redox equation in the estimation of Iron by Potassium permanganate.

33. Give the role of Sulphuric acid in permanganometric titrations.
34. Name the indicator used in the estimation of Chromium by using Mohr's salt solution.
35. Give the reasons for adding Sulphuric acid & Phosphoric acid in the estimation of chromium by using Mohr's salt solution.
36. Give the colour changes during the estimation of Chromium by using Mohr's salt solution.
37. Why green colour is appeared at the beginning of the Dichrometric titrations.
38. Give the Molecular weight & equivalent of Potassium Dichromate.
39. What is iodometry?
40. Give the basic principle involved in Iodometric titrations.
41. What is the indicator used in Iodometric titrations.
42. Why starch indicator is not added at the beginning of Iodometric titrations?
43. Why straw colour changes to blue with the addition of starch indicator.
44. What is the colour change at the end point of Iodometric titrations?
45. Give the differences between the estimation of Chromium by Dichrometry method and Iodometry method.
46. What is bleaching powder?
47. Give the structure of CaOCl_2 .
48. What is available chlorine?
49. What is E.D.T.A? Give the structure of E.D.T.A.
50. Give some examples of complexometric titrations.
51. Draw the structure of Metal – E.D.T.A. complex.
52. What is the indicator used in the estimation of Total hardness & Zn by Complexometric method using E.D.TA solution.
53. What is the indicator used in complexometric titrations by using E.D.TA solution?
54. Give the role of Buffer used in complexometric titrations by using E.D.TA solution.
55. Why pH of the solution should be maintained between 8 – 10 in complexometric titrations by using E.D.TA solution?
56. Give the colour changes taking place in complexometric titrations using EBT- Indicator.
57. Give the significance of viscosity index.
58. What is pH .
59. What is total alkalinity?
60. Give the possible combinations of ions causing alkalinity.
61. Give the Equivalent weight of CaCO_3 ?
62. What is buffer?
63. Give the relation between Temporary hardness & Permanent hardness.
64. Give the oxidation state of chromium in Potassium dichromate?

65. Give the effect of temperature on the dissolved oxygen content.
66. Give the difference between ferrous iron and ferric iron.
67. What is basic buffer? Give one example?
68. What is standard solution?
69. What is primary standard solution & secondary standard solution?
70. Why H_2SO_4 is most suitable when compare with HCl and HNO_3 in Permanganometric titrations.
71. What is conductometric titration?
72. What is the unit of conductance?
73. What are the factors influencing conductivity?
74. How can you identify the end point in conductometric titration?
75. What is the importance of the conductometric titration?
76. What is equivalent conductance?
77. What is effect of dilution on conductance?
78. Why ordinary water is not suitable in conductance measurement?
79. How does conductance changes when acetic acid is titrated against sodium hydroxide solution?
80. How does the conductance changes when strong acid is titrated against a strong base?
81. What are the advantages of conductometric titrations?
82. What is the effect of temperature on conductance?
83. Define electrode potential?
84. What is indicator electrode?
85. Why hydrogen electrode is not generally used in P^{H} measurements?
86. What is the effect of temperature on P^{H} ?
87. How the equivalence point in acid base titrations is determined P^{H} metrically?
88. What is the principle involved in colorimetric titrations?
89. Why do you use disodium salt of EDTA in the estimation of hardness of water?