GAYATRI VIDYA PARISHAD COLLEGE OF ENGINEERING FOR WOMEN

(Approved by AICTE New Delhi & Affiliated to JNTUK, Kakinada)

Kommadi, Madhurawada, Visakhapatnam- 530048

Department of Basic Science and & Humanities

LABORATORY MANUAL

FOR

APPLIED CHEMISTRY-LAB

(Common to all branches)

REGULATION: R-20 **YEAR&SEMESTER:** I– II

SUBJECTCODE: 1R201116 **NBA COURSECODE**

Department of Basic Science and & Humanities

Institute Vision: Produce competitive experience instied with ethical and social responsibiities to deal with the technoogical chaenges in the fied of electronics and communication engineering

Institute Mission: Facilitate value -based educational environment education that provides value based education

Provide opportunities for developing creative, innovative and leadership skills

Department Vision: To develop the basic experimental skills for under graduate students of all branches of Engineering

Department Mission: The course attempts to learn the quantitative aspects & other chemical techniques and the students can apply this learning in their respective areas of expertise.

Course Outcomes

Course Outcomes		***Experiment No.***
CO1	Make use of experimental skills for quantitative analysis in acid-base titrations using indicators	1& 2
CO2	Apply the principals of redox titrations like Mn(II), ferrous (II) and vitamin-C in different samples	3,4 & 14
CO3	Experiment with complexometric titrations to determine hardness of water and estimate the amount of copper (II) using hypo solution by iodometric method	5 & 6
CO4	Perform experiments with instruments such as conductometter, P^H ⁻meter & colorimetric methods to acquire skills of different methods of chemical analysis	7,8,9, 10 & 11
CO5	Estimate the amount of Mg^{2+} present in an antacid, adsorption of acetic acid by charcoal and $CaCO_3$ present in egg shell	11-17

Mapping of Course Outcomes (COs) with Program Outcomes (POs)

Course Outcomes		**Program Outcomes (POs)**													
		PO 1	**PO 2**	**PO 3**	**PO 4**	**PO 5**	**PO 6**	**PO 7**	**PO 8**	**PO 9**	**PO 10**	**PO 11**	**PO 12**	**PSO 1**	**PSO 2**
NBA Course Code (C117)		**2**	**-**	**-**	**2**	**-**	**2**	**-**	**-**	**2**	**2**	**-**	**-**	**-**	**-**
		2	**2**	**2**	**1**	**-**	**2**	**1**	**-**	**1**	**-**	**-**	**-**	**-**	**-**
		2	**2**	**2**	**2**	**-**	**2**	**2**	**-**	**-**	**-**	**-**	**-**	**-**	**-**
		2	**2**	**3**	**2**	**-**	**2**	**-**	**-**	**-**	**1**	**-**	**-**	**-**	**-**
		1	**2**	**2**	**2**	**-**	**2**	**2**	**-**	**-**	**-**	**-**	**-**	**-**	**-**

SYLLABUS

TITLE OF THE EXPERIMENTS
Introduction to Chemistry laboratory – Molarity, normality, primary, secondary standard solutions, volumetric titrations, quantitative analysis
1. Determination of HCl using standard Na_2CO_3solution.
2. Determination of alkalinity of a sample containing $Na_2CO_3$and NaOH.
3. Determination of Mn^{+2}using standard oxalic acid solution.
4. Determination of ferrous iron using standard $K_2Cr_2O_7$solution.
5. Determination of Cu^{+2} using standard hypo solution.
6. Determination of temporary and permanent hardness of water using standard EDTA solution.
7. Determination of Fe^{+3} by a colorimetric method.
8. Determination of the concentration of acetic acid using sodium hydroxide (pH-metry method).
9. Determination of iso-electric point of amino acids using pH-metry method/conductometric method.
10. Determination of the concentration of strong acid vs strong base (by conductometric method)
11. Determination of strong acid vs strong base (by potentiometric method).
12. Determination of Mg^{+2} present in an antacid.
13.Determination of $CaCO_3$present in an egg shell.
14.Estimation of Vitamin C.
15.Determination of phosphoric content in soft drinks.
16.Adsorption of acetic acid by charcoal.
17.Preparation of nylon-6, 6 and Bakelite (demonstration only).

Outcomes: The students entering into the professional course have practically very little exposure to lab classes. The experiments introduce volumetric analysis; redox titrations with different indicators; EDTA titrations; then they are exposed to a few instrumental methods of chemical analysis. Thus at the end of the lab course, the student is exposed to different methods of chemical analysis and use of some commonly employed instruments. They thus acquire some experimental skills.

LAB RUBRICS

Internals	Category	Points			
Day to Day Performance	**Attendance (1)**	Attended and completed on the same day	Attended and partially completed on the same day	Attended but completed in the extra lab	Not attended but completed in the extra lab
	Understanding of the Experiment (2)	Complete understanding of the experiment with learning objectives	Partial understanding of the experiment with learning objectives	Most of the experiment misunderstood	Complete misunderstanding of the experiment
	Implementation with result analysis (5)	Complete implementation with result analysis and interpretation	Complete implementation with result analysis only	Complete implementation with result analysis and interpretation in extra lab	Complete implementation with result analysis only in extra lab
	Observation submission on time (2)	Submission of the observation on time	Submission of the observation almost on time	Submission of the observation immediately after the extra lab	Submission of the observation after the extra lab
Record	**Comprehensiveness & Legible (3)**	Write all the elements of the experiments which can be easily readable	Write all the elements of the experiments with poor handwriting	Some elements are missing but presented clearly	Some elements are missing and poor handwriting
	Timely Submission (2)	Submission of the record on time	Submission of the record almost on time	Submission of the record immediately after the extra lab	Submission of the record after the extra lab

Internals	Category	Points			
Internals	**Aim of the experiment (1)**	Complete understanding of the learning objectives and outcomes	Complete understanding of the learning objectives only	Partial understanding of the learning objectives	Misunderstanding of the learning objectives
	Write up (3)	Write all the elements of the experiments which can be easily readable	Write all the elements of the experiments with poor handwriting	Some elements are missing but presented clearly	Some elements are missing and poor handwriting
	Implementation & result analysis (4)	Complete implementation with result analysis and interpretation	Complete implementation with result analysis only	Partial implementation with result analysis only	Partial implementation only
	Viva- Voce (2)	Experiment and subject knowledge with good oral presentation	Experiment and subject knowledge with poor oral presentation	Partial experiment knowledge with poor oral presentation	Partial subject knowledge with poor oral presentation

ENGINEERING CHEMISTRY PRACTICAL MANUAL CUM RECORD

GAYATRI VIDYA PARISHED COLLEGE OF ENGINEERING FOR WOMEN

Dr. P. SRINIVASA RAO M.Sc., Ph.D.

APPLIED CHEMISTRY

I B.TECH
(COMMAN TO ALL BRANCHES)

CERTIFICATE

DEPARTMENT OF APPLIED CHEMISTRY

Certified that this bonafide record of the work done in this Laboratory by Miss_____of_____class during the year _____

The total number of Experiments certified by :_____

In charge of the faculty

Submitted for the University examination held on

Internal examiner *External examiner*

INDEX

Regd.No._____

Batch No._____

S.No.	Date	Name of the experiment	Page. No.	Remark

CHEMISTRY-LAB

(Common to all branches)

Course Code: 1R201116

I Year –I &II Semester		L	T	P	C
		0	0	3	1.5
APPLIED CHEMISTRY LAB (BS1103)					

Aim: The aim of the course is to develop the basic experimental skills for under graduate students of all branches of Engineering.

Objectives: The course attempts to learn the quantitative aspects & other chemical techniques and the students can apply this learning in their respective areas of expertise.

CONTENTS

Outcomes: The students entering into the professional course have practically very little exposure to lab classes. The experiments introduce volumetric analysis; redox titrations with different indicators; EDTA titrations; then they are exposed to a few instrumental methods of chemical analysis. Thus at the end of the lab course, the student is exposed to different methods of chemical analysis and use of some commonly employed instruments. They thus acquire some experimental skills.

ENGINEERING CHEMISTRY LAB

(Introduction)

Chemical analysis is the resolution of a chemical compound into it's proximate or ultimate parts. This is done in a chemistry laboratory. It is divided into two types.

(i) **Qualitative analysis :** It deals with identification and conformation ofthe nature of the substance or impurities present in a given sample.

(ii) **Quantitative analysis :** It deals with the determination of how much of each component or of specified components is present. Quantitative chemical analysis is further divided into two types

(i) **Volumetric analysis :** It involves the accurate measurement of volume of liquids, thoughone or two weighing may also be needed

(ii) **Gravimetric analysis :** Gravimetric analysis is more accurate but volumetric analysis is readily carried out. However volumetric analysis is no that accurate. If an experienced worker carries out the analysis the error should not exceed 0.2%

Essential conditions for accurate titrametry :

(i) The equation governing the reaction must be known
(ii) The reaction must go to completion and there must be no complicating side reaction.
(iii) Their must be some means to asses the completion of reaction (usually an indicator is employed)
(iv) The standard solution must not alter in it's strength during the period of experimentation. The solution must be stable to light, atmosphere and must not react with the solvent.

Terms used in volumetric analysis :

(a) **Titration :** It is a process of adding one solution from the burette to another in a conical flask in order to complete the chemical reaction. Out of the two solutions one must be standard.

(b) **Titrant :** The reagent of known concentration from the burette is called titrant

(c) **Titrate :** The substance being titrated is termed as titrate

(d) **Equivalence point or end point :** It is the point at which the amount of titrant and titrate being determined are chemically equivalent or it is the exact stage at which the chemical reaction involved in the titration is just completed

(e) **Indicator** : The substance which helps in the visual detection of the completion of the reaction is known as indicator.

(f) **End point** : The point at which the colour change of the indicator is apparent to the eye is called the end point.

(g) **Titration Error :** Ideally, the visible end point and the equivalence point should coincide but in practice there is a always a difference between the two.This difference is called the titration error. It is because the colour change of indicator takes place just before or after the equivalence point. Errors do occur in instrumental analysis also. These errors occur due to analyst or instrument or method or reagent, hence most probable value is taken as the result.

Standard solution : A solution of accurately known strength is called as standard solution. e.g. N/10 sodium hydroxide is a standard solution. Standard solution is prepared by dissolving a definite amount of a substance, called primary substance in a definite volume of solvent.

Primary standard substance should satisfy the following requirements :

(a) Easy to obtain high purity and to preserve in a pure state.

(b) The substance should maintain its composition unchanged during storage and unaltered in air during weighing; it is neither oxidized by air nor affected by carbon dioxide.

(c) It should neither be hygroscopic nor efflorescent

(d) The total amount of impurities should not in general, exceed 0.01 – 0.02%

(e) It should have a high equivalent weight so that the weighing errors may be negligible.

(f) The substance should be readily soluble under the condition in which it is employed. In practice, an ideal primary standard is difficult to obtain and a compromise between the above ideal requirements is usually necessary.

Commonly employed primary standard substance are:

In acidimetric and alkalimetric : sodium carbonate (Na_2CO_3), Borax ($Na_2B_4O_7$) and potassium hydrogen phthalate ($KHC_8H_4O_4$) etc.

In precipitation titrations : silver, silver nitrate, sodium chloride, potassium chloride etc.
In oxidation – reduction titration : potassium dichromate($K_2Cr_2O_7$), potassium bromate (KBrO3), potassium iodate (KIO_3), potassium iodate ($KH(IO_3)_2$), iodine (I_2), sodium Oxalate ($Na_2C_2O_4$), arsenic oxide(As_2O_3) etc.

Secondary standard substance :

Those substances which do not lose water of crystallization, such as borax ($Na_2B_4O_7.10H_2O$), oxalic acid ($H_2C_2O_4.2H_2O$) and copper sulphate ($CuSO_4.5H_2O$) are considered to be secondary standard substances.

A standard solution prepared by dissolving a primary standard substance is, known as primary standard solution and by dissolving a secondary substance is known as secondary

standard solution. In this case the number of atoms/ molecules/ions are same as reactants side (LHS) and product side (RHS) such reaction is termed as stoichiometrically balanced reaction.

Ex, $H_2 + ½\ O_2 \rightarrow H_2O$

We all know very well that atoms, ions and molecules react in definite proportions. But one cannot know that how many numbers of atoms, ions or molecules are participated in a reaction.

Ex : $H^+ + Cl^- \rightarrow HCl$

Hence chemists talk about the relative masses instead of number of atoms ions or molecules reacting. One can calculate the relative mass (weight) of an atom or ion or molecule.

Formula weight of HCl is 36.5 mass units**.**

To simplify the calculations mole was introduced. Mole is the atomic, molecular or formula weight
of a substance expressed in grams.

36.5 g of HCl = 1 mole X g of HCl = ... Mole

A mole of any substance contains equal number of atoms, ions or molecules i.e., equal to Avogadro number.

Avogadro number = $6.023 \text{ X } 10^{23}$

Concentration of a solution is generally expressed in the following chemical units:

1. **Molarity (M) :** The molarity of a solution is defined as the number of gram molecules of solute present in one litre of solution.

$$\text{Molarity (M)} = \frac{\text{Number of gram-molecular weight}}{\text{Grams of solute for 1000 ml of solute}}$$

Normality (N) : The normality of a solution is defined as the number of gram equivalent of solute present in one litre of solution.

$$\text{Normality} = \frac{\text{Number of gram-equivalents of solute}}{\text{Number of litres of solution}}$$

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

Example: Equivalent weight of oxalic acid= Equivalent weight of oxalic acid/ Basicity of acid

i.e., 126/2=63

Equivalent weight of some Acids

Name of molecule	Formula of acid	Basicity	Equivalent weight
Hydrochloric acid	HCl	1	36.1/1=36.1
Nitric acid	HNO_3	1	63/1=63
Sulphuric acid	H_2SO_4	2	98/2=49
Acetic acid	CH_3COOH	1	60/1=60
Phosphoric acid	H_3PO_4	2	98/3=32.66
Oxalic acid	$H_2C_2O_4$	3	126/2=63

Equivalent weight of base = $\frac{\text{Molecular weight of the base}}{\text{Replaceable } OH^- \text{ atoms in one molecule of the base}}$

$= \frac{\text{Molecular weight of base}}{\text{Acidity of the base}}$

Example: Equivalent weight of Na_2CO_3 = 106/2=53

Equivalent weight of some bases

Name of molecule	Formula of base	Acidity	Equivalent weight
Sodium hydroxide	NaOH	1	40/1=40.0
Potassium hydroxide	KOH	1	58/1=58
Ammonium hydroxide	NH_4OH	1	35/1=35
Magnesium hydroxide	$Mg(OH)_2$	2	58/2=29
Calcium hydroxide	$Ca(OH)_2$	2	74/2=37

Equivalent weight of a salt: It is the ratio of molecular weight to the total valence of cations or anions.

Equivalent weight of Salt = $\frac{\text{Molecular weight}}{\text{Total valence of cations or anions}}$

Name of molecule	Formula of salt	Total valency of cations or anions	Equivalent weight
Sodium chloride	NaCl	1	58.1/1=58.1
Sodium carbonate	Na_2CO_3	2	106/2=53
Magnesium sulphate	$MgSO_4$	2	120/2=60
Calcium carbonate	$CaCO_3$	2	100/2=50
Silver nitrate	$AgNO_3$	1	170/1=170
Copper sulphate	$CuSO_4$	2	189.2/2=59.75

Equivalent weight of a reducing agent: It is the ratio of molecular weight to the number of electrons lost.

$$E_{RA} = \frac{\text{Molecular weight}}{\text{No. of electrons lost}}$$

Equivalent weight of some reducing agents

Name of compound	Formula of compound	No. of electrons lost	Equivalent weight
Mohr's salt	$FeSO_4(NH_4)_2SO_4H_2O$	1	392/1=392
Hypo	$Na_2S_2O_3$	1	158/2=158
Oxalic acid	$H_2C_2O_42H_2O$	2	126/2=63
Ferrous sulphate	$FeSO_4\,7H_2O$	1	278/1=278

Equivalent weight of an oxidising agent: It is the ratio of molecular weight to the number of electrons gained

$$E_{OA} = \frac{\text{Molecular weight}}{\text{No. of electrons gained}}$$

Equivalent weight of some oxidising agents

Name of compound	Formula of compound	No. of electrons gained	Equivalent weight
Potassium permanganate	KMnO4	5 (in acidic medium)	158/5=31.6
Potassium permanganate	KMnO4	3 (in neutral)	158/3=52.6
Potassium dichromate	$K_2Cr_2O_7$	6	294/6=49

Equivalent weight of oxidizing or reducing agent= Molecular weight of the substance / Number of electrons lost/gain by one molecule in the redox reaction

Example: Potassium dichromate ($K_2Cr_2O_7$) oxidizes

$$Cr_2O_7^{2-} + 14\,H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

Hence, equivalent weight=294/6=49

Potassium permanganate ($KMnO_4$) oxidize

$MnO_2 + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4\ H_2O$

Hence, equivalent weight=158/5=31.6

Hence, equivalent weight of iodine =254/2=127

Hence, equivalent weight of ferrous sulphate ($FeSO_4.\ 7H_2O$)=278/1=278

Equivalent weight of some elements

Name of acid	Symbol	valence	Equivalent weight
Sodium	Na	1	23/1=23
Aluminum	Al	3	27/3=9
Magnesium	Mg	2	24/2=12
Ferrous	Fe^{+2}	2	56/2=28
Silver	Ag	1	108/1=108
Ferric	Fe^{+3}	3	53/3=18.6
Zinc	Zn	2	65.4=32.7
Copper	Cu^{+2}	2	63.5=31.75
Potassium	K	1	39/1= 39

Normal solution: A solution containing 1gram equivalent weight of the solute dissolved per litre is called a normal solution, e.g., when 40 gms of sodium hydroxide (solute) is dissolved in one litre of solution formed is known as normal solution.

Sub-normal solution: A solution containing a fraction of gram equivalent weight of the solute dissolved per litre is known as sub-normal solution. For example a solution containing 20gms (1/2 fraction of gram equivalent weight) of sodium hydroxide dissolved per litre is known as semi-normal solution; similarly a solution containing 4 gm (1/10 fraction of gm eq.wt.) of sodium hydroxide per litre is known as deci-normal solution and commonly written as N/10 or 0.1N-NaoH.

Symbol	**Significance**	**Normality**
N/2	Semi-normal	1 g eq/lit
N/10	Deci-normal	0.1 g eq/lit
N/100	Centi-normal	0.01 g. eq/lit

Molar solution: A solution containing 1 gram molecular weight of the solute per litre is known as a molar solution, e.g., the molar solution of sulfuric acid contains 98 gm/litre, while its normal solution contains 49 gm/litre. Normality varies according to the reaction in which the solute participates. Example : Reduction of Mn takes place as in the following:

In acid medium : $Mn^{+7} \rightarrow Mn^{+2} + 5e^-$

Alkaline medium : $Mn^{+7} \rightarrow Mn^{+4} + 3e^-$

No of gram equivalents

Substance	Molecular mass	Balanced ionic equation	No. of electron in equation	Moleculer weight / n
Ferrous ammonium sulphate [$FeSO_4$ $(NH_4)_2SO_4$. $6H_2O$	392	$Fe^{+2} \rightarrow Fe^{+3} + e^-$	1	392/1=392
Sodium thio sulphate Na_2SO4 .$5H_2O$	248	$2S_2O3^{2-} \rightarrow 1/2S_2O_3^{2-} + 2e^-$	1	248/1=248
Iodine(I_2)	254	$I_2 + 2e^- \rightarrow 2I^-$	2	254/2=127
Potassium dichromate $K_2Cr_2O_7$	294	$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{+3} + 7H_2O$	6	294/6=49

Further since number of gram equivalents = N X Vol. in .lit.

According to the law of equivalency, $N_1V_1 = N_2V_2$

CLASSIFICATION OF TITRIMRTRIC REACTIONS

Volumetric titrations are classified into four classes depending upon the type of reaction taking place during the titration.

1. **Acid – Base Titration (Neutralization Titrations)**

Titrations based on neutralization reaction are called as neutralization titrations or acid–base titrations. In neutralization reactions, an acid and base react with each other and both become neutral.

$$HCl + NaOH \rightarrow NaCl + H_2O$$
Acid Base Salt Water

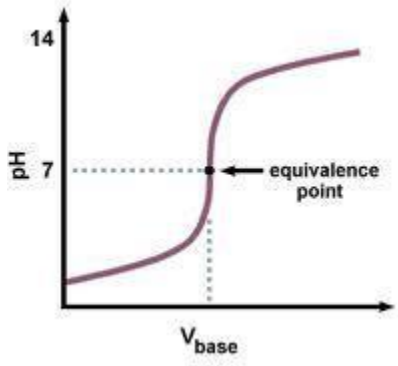
In aqueous solutions, both acids and bases are ionized to give H^+ and OH^- ions respectively, which then react to form water molecule, hence these reaction are merely (nothing but) combination of H^+ and OH^- Ions to form neutral water.

$$H^+ + OH^- \rightarrow H_2O$$

i. Strong Acid against a Strong Base:

Let us consider the titration of HCl and NaOH. The pH values of different stages of titration shows that, at first the pH changes very slowly and rise to only about 4. Further addition of such a small amount as 0.01 mL of the alkali raises the pH value by about 3 units to pH 7. Now the

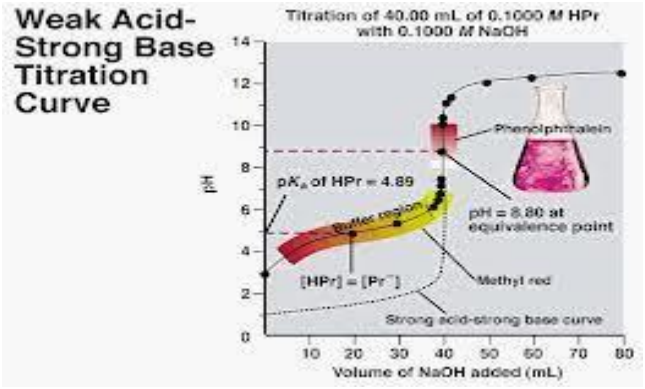
acid is completely neutralized. Further of about 0.01 mL of 0.1 M NaOH will amount to adding hydrogen ions and the pH value will jump to about 9. Thus, near the end point, there is a rapid increase of pH from about 4 to 9. Can be used An indicator is suitable only if it undergoes a change of colour at the pH near the end point. Thus the indicators like ***methyl orange, methyl red and phenolphthalein*** can show the colour change in the ph range of 4 to 10. Thus, in strong acid- strong base titrations, any one of the above indicators. This titration is graphically represented in the following way.



Strong acid vs strong base titration curve

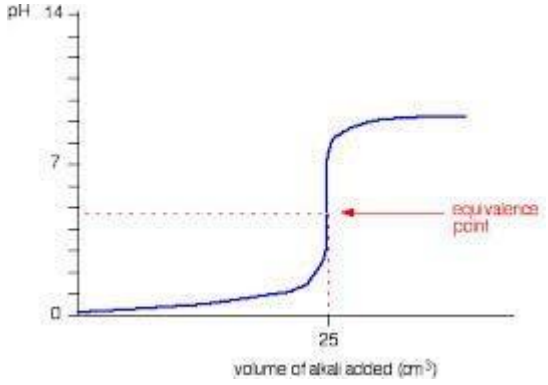
ii. Weak Acid against Strong Base:

Let us consider the titration of acetic acid against NaOH. The titration shows the end point lies between pH 8 and 10. This is due to the hydrolysis of sodium acetate formed. Hence ***phenolphthalein*** is a suitable indicator as its pH range is 8-9.8. However, methyl orange is not suitable as its pH range is 3.1 to 4.5.



iii. Strong Acid against Weak Base:

Let us consider the titration ammonium hydroxide against HCl. Due to the hydrolysis of the salt, NH_4Cl, formed during the reaction, the pH lies in the acid range. Thus, the pH at end point lies in the range of 6 to 4. Thus ***methyl orange*** is a suitable indicator while phenolphthalein is not suitable.



Strong acid vs weak base titration curve

StrongAcids	StrongBases	WeakAcids	WeakBases
HCl	NaOH	Acetic acid	Ammonia
HNO_3	KOH	Hydrocyanic acid	Magnesium hydroxide
HBr	etc	HF	Pyridine
H_2SO_4		Oxalic acid	Sodium carbonate
HI		Ethanoic acid	Potassium carbonate
$HClO_4$		etc	etc

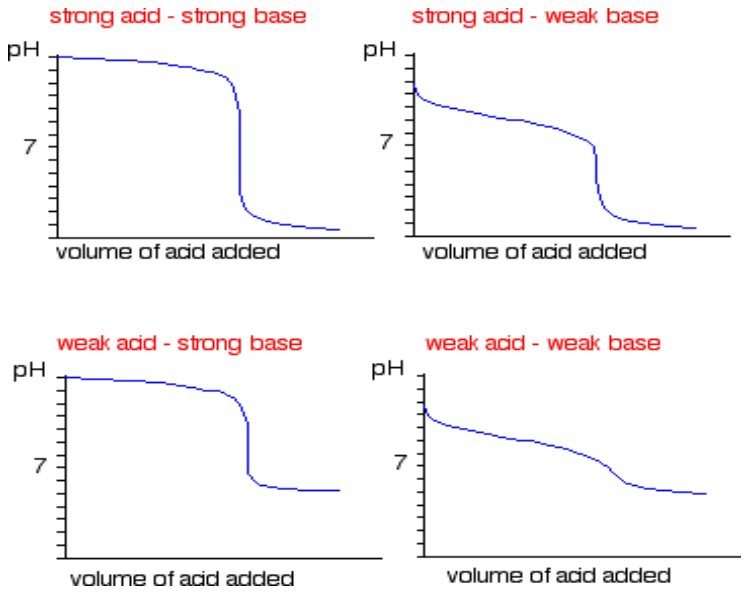
An acid – base indicator is a substance which has one colour in acidic medium and a completely different color in the base medium. In fact the change in color of an acid – base indicator does not take place at a particular pH value but takes with in a small range of pH (usually about 2 p^H units). This range of p^H is known as "color change interval " of the indicator. This is different indicators.

Example : pH range of phenolphthalein is 8.3-10. it means the color of the indicator is colorless in solution of pH range is 8.3 or less i.e., the indicator gives white color in acid, while in solution of pH 10 or above the indicator gives pink color in base. In solution having pH between 8.3 and 10, phenolphthalein gives a color intermediate between colorless and pink. Thus phenolphthalein can be used as an indicator in the pH range 8.3 - 10

Table 1 : **Range of commonly used indicators**

Indicator	pH range	Color in acidic solution	Color in basic solution
Methyl orange	3.1-4.5	Red	Yellow
Methyl red	4.2-6.3	Red	Yellow
Litimus Phenol red	4.5-8.3 6.4-8.2	Red Yellow	Blue Red
Phenolphthalen	8.3-10.0	Colourless	Pink

Acid – Base Titration curves:



Theory of indicators:

There are two theories which explain the change of color of the acid-base indicators at the end point.

Ostwald Theory (Ionic theory)

Essential features of the theory are :

(a) Neutralization indicators are weak acidic or weak basic organic compounds. Hence they ionize partly in the aqueous solution and are in equilibrium between the ions and the unionized molecules.

$$\underset{\text{weak acid}}{HIn} \leftrightarrow H^+ + In \text{ indicator}$$

$$\underset{\text{weak base}}{InOH} \leftrightarrow In^+ + OH \text{ indicator}$$

(b) The ions produced in the solution have different colors than that of unionized molecule.

Working indicators :

Let us explain the working of litmus, phenolphthalein and methyl orange on the basis of the above
theory.

Working litmus is a weak acidic substance represented by HIn. The unionized from is red, while the
dissociated ions are blue in color.

$$HIn \leftrightarrow H^+ + In^-$$

Thus in an aqueous solution of litmus, un dissociated molecules as well as dissociated ions are present together and hence its colour will be an intermediate one i.e., In- violet. If the litmus solution is made acidic, the added H^+ ions combine with most of In- anions present in the solution to form un dissociated HIn molecules and hence the equilibrium shifts to left, thus the solution turns to red. On the other hand, if an alkali is added to litmus solution, the equilibrium shifts to right (H^+ ions of the indicator are removed by OH^- ions of the alkali) and thus, the amount of In^- anoins are increased with the result the solution turns to blue. Working of phenolphthalein : it is represented by HPh, is a weak acidic substance . its unionized form is colorless, while its anion ph- is pink in colour.

$$HPh \leftrightarrow H^+ + Ph^-$$

Colourless pink

In acidic medium, due to high concentration of H^+ ions the equilibrium lies towards left and imparts colorless appearance to the solution. On the other hand , in alkaline medium the equilibrium lies to the right and imparts pink color to the solution. In case of weak alkali (Ex :NH_4OH),sufficient ions are not produced easily to shift the equilibrium to right and hence a large amount of NH_4OH is required for this purpose. Hence end point is not sharp. This explains why phenophthalein is not a suitable indicator in the titration of a strong acid with weak base . Alternatively this can be explained as below: Ammonium ion(NH_4^+) is produced by the ionization of NH_4OH combines with Ph^- ion to form NH_4Ph which is readily hydrolyzed in the following manner. Thus the resulting solution contains unionized molecule (HPh) which hinder in changing colour to pink. Working of methyl orange : it is a weak basic indicator is usually represented by MeOH. Its unionized form is yellow, while its ionized form is red in color.

$$MeOH \leftrightarrow Me^+ + OH\ In$$

presence of alkali, the OH^- concentration is high and hence the equilibrium shifts towards left thus solution acquires yellow color. On the other hand in presence of acid the equilibrium shift towards right hence the solution acquires red color. The Ostwald theory of ionization explains that methyl orange is not a suitable indicator for the titration of a weak acid with strong base. The weak acid (CH_3COOH) ionize to a small extent and thus not capable of providing sufficient amount of H^+ ions.

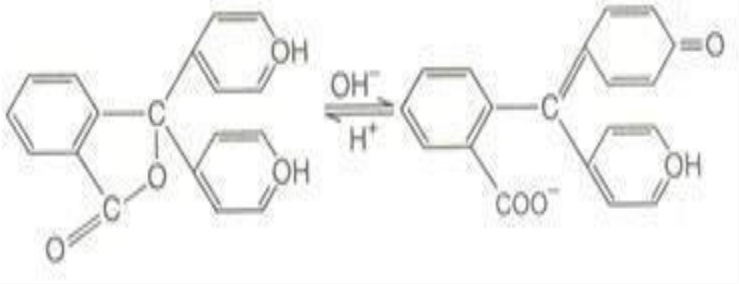
Essential features of the theory are :

(a) Acid – Base indicators are complex aromatic compounds, capable of existing in two tautomeric forms namely benzenoid and quinonoid

(b) The two forms have different colors generally the benzenoid is lighter in color than quinonoid form.

(c) One of the two forms is stable in acidic medium and other in alkaline medium. Thus change of medium form acidic to basic or vice versa changes the structure form one form to other leading to change in color. Let us illustrate this by taking example of phenolphthalein and metylorange.

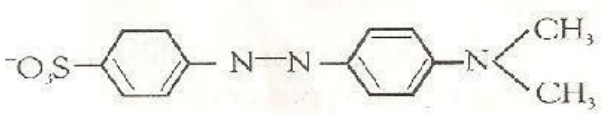
(a) **phenolphthalein :** In the acidic medium, phenolphthalein exists in the benzenoid form which is colorless. However as soon as the medium becomes alkaline the benzenoid structure is converted into quinonoid structure which possesses pink color.



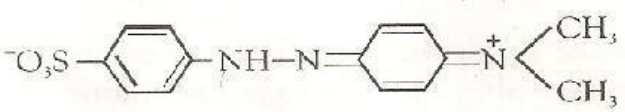
benzenoid form(colorless) quinonoid form(Pink color)

acidic medium alkaline medium

(b) **Methyl orange :** The two tautomeric forms of methyl orange capable of existing in the basic and acidic medium are drawn below.

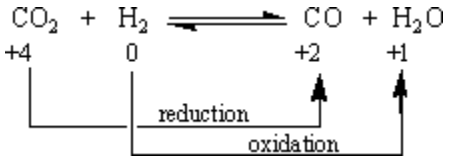


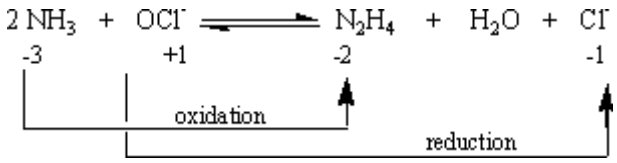
Benzenoid form (yellow)
(stable in alkaline medium)



Quinonoid form (red)
(stable in acidic medium)

2.Oxidation-reduction reaction Or (Redox reactions)*:* In such titrations, oxidation(loss of electrons) and reduction(gain of electrons) necessarily occur together, one substance being oxidized and another is reduced.





Reactions described as oxidations are usually those in which the oxidizing agent (i.e., $KMnO_4$, acidic $K_2Cr_2O_7$, I_2, Cu^{+2}, etc.) forms the standard solution, while in those classified as reduction a standard solution of the reducing agent (e.g., ferrous salts, oxalic acid, oxalate, As_4O_6,$S_2O_3^{-2}$,I^- etc.)

For example consider the reduction of potassium permanganate by ferrous sulphate in acid medium.

$$MnO_4 + 8\ H^+ + 5e^- \rightarrow Mn^{2+} + 4\ H_2O \text{ (reduction)}$$
$$Fe^{+2} \rightarrow Fe^{3+} + 5\ Fe^{3+} + 4\ H_2O \text{ (oxidation)}$$
$$MnO_4 + 8\ H^+ + 5e^- \rightarrow Mn^{2+} + 5\ Fe^{3+} + 4\ H_2O$$

Titrations based upon oxidation-reduction reactions are called redox titrations. However , sometime redox titrations are specifically name after the name of the reagent Viz., iodometric, permanganometric, dichrometric titrations, etc.

Indicators for Redox titrations:

These are three types:

i) Self indicator
ii) External indicator
iii) Redox indicator (internal indicator)

Self indicator:

In some cases one of the reactants of the reaction in the titration acts as an indicator. Such reactant is called self indicator. During the reaction of potassium permanganate (taken in a burette) against sodium oxalate (taken in conical flask) the solution of sodium oxalate remains colorless till the reaction is complete. However, as soon as reaction becomes complete, i.e., when whole of the oxalate present in conical flask is completely oxidized with the added permanganate solution. Further addition of single drop of permanaganate from the burette imparts pink color to the solution of conical flask. This indicates the end point. Since here potassium permanganate itself acting as indicator, it is known self indicator.

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(O)$$

$$2\,H_2C_2O_4 + 5(O) \rightarrow 10CO_2 + 5H_2O$$

$$2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$$

External indicators:

Sometime completion of the reaction is indicated by the use of substance outside in the reaction mixture. Such a substance is called external indicator. For example, potassium ferricyanide is used as an external indicator during the titration of ferrous salt solution with potassium dichromate solution in acidic medium.

The use of potassium ferricyanide as indicator is based upon the fact that the Fe^{2+} ions from ferrous salt (taken in conical flask) react with ferricyanide ions forming deep blue color ferro-ferricyanide complex.

$$Fe^{2+} + [Fe(CN)_6]^{3-} \rightarrow [Fe\,Fe(CN)_6]^-$$

Or $FeSO_4 + K_3[Fe(CN)_6] \rightarrow K[FeFe(CN)_6] + K_2SO_4$

Potassium ferro-ferricyanide (deep blue color)

However, when whole of the Fe^{2+} ions present in conical flask are oxidized by the progressive addition of dichromate solution from the burette Fe^{3+} ions, the indicator will not give blue color with the solution

Redox indicators:

Redox indicator is a compound which can undergo oxidation and reduction reversibly and the oxidized and reduced forms have different colors. In general

$$I_{Ox} + ne^- \leftrightarrow I^{ne-}_{red}$$

For example, diphenylamine is used as a redox indicator during the titration of ferrous ion with dichromate. The reagent has blue-violet color in the oxidized form and is colour less in the reduced form.

3.Precipatitaon reactions

Precipitation processes of volumetric analysis are based the formation of insoluble precipitates when the reacting solutions are brought together. Thus when a solution of silver nitrate is treated with a sodium chloride or potassium thiocyanate, a white precipitate of silver chloride or silver thiocyanate is obtained.

$AgNO_3 + NaCl \rightarrow AgCl + NaNO_3$

$AgNO_3 + KCNS \rightarrow AgCNS + KNO_3$

Titrations based upon such reactions are called precipitation reactions. sometimes the method is called after the reagent used, e.g., titration involving the use of silver nitrate as titrant are called argentometric titrations.

(i) Ferric alum in titrations of silver nitrate with ammonium(or) potassium) thiocyanate (*Volhard's method*)

(ii) Potassium chromate in titrations of silver nitrate with sodium chloride (*Mohr's method*)

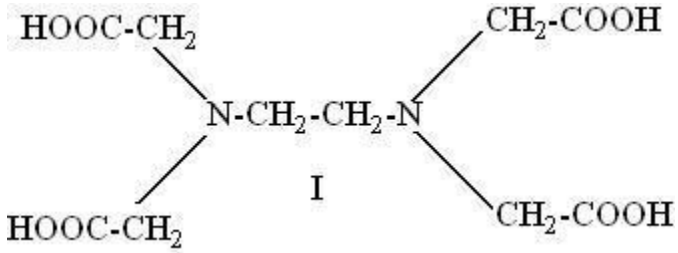
(iii) Eosin in titrations of silver nitrate with potassium bromide.

4. Complexometric reactions:

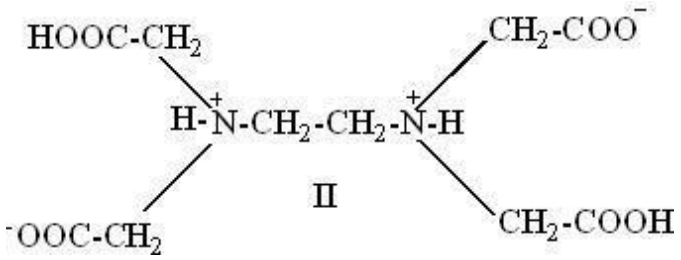
There are certain reactions in which complex compounds are called complexometric titrations. The most important examples are: EDTA generally react with metal ions like Mg^{2+}, Zn^{2+}, Ca^{2+}, etc.

Principle of Complexometric Titration:

Complexometric titrations are particularly useful for determination of a mixture of different metal ions in solution. Ethylenediamine tetra acetic acid (EDTA), is a very important reagent for complex formation titrations. EDTA has been assigned the formula II in preference to I since it has been obtained from measurements of the dissociation constants that two hydrogen atoms are probably held in the form of zwitter ions.



I

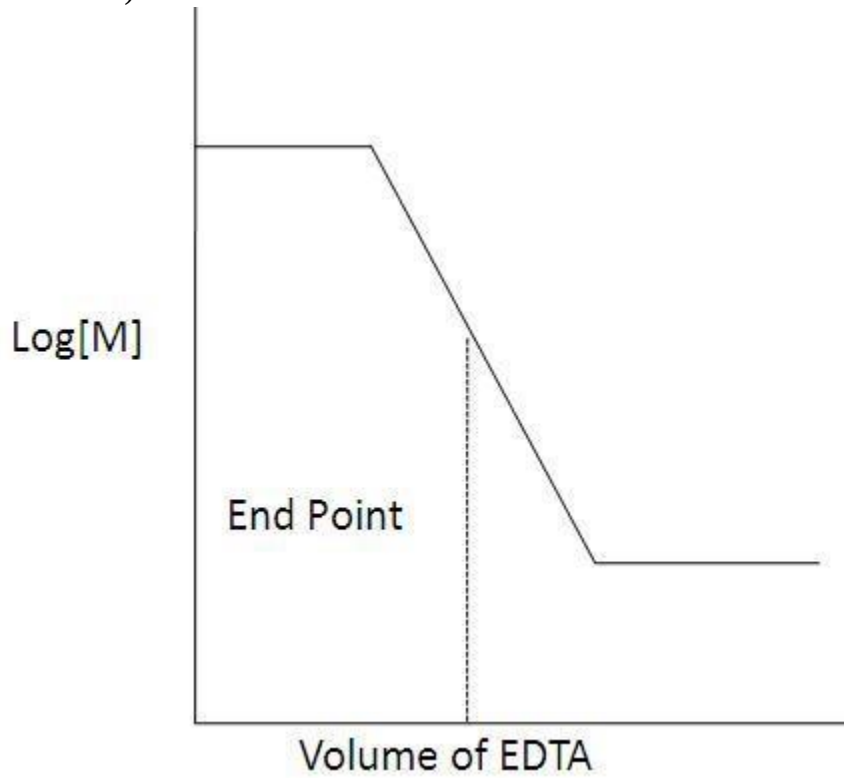


II

EDTA behaves as a dicarboxylic acid with two strongly acidic groups. For simplicity EDTA may be given the formula H_4Y, the disodium salt is therefore Na_2H_2Y and it has the complex forming ion H_2Y^{2-} in aqueous solution. The reactions with cations may be represented as;

$$M^{2+} + H_2Y_2^- \rightarrow MY^{2-}$$
$$+2H^+ \; M^{3+} + H_2Y_2^- \rightarrow MY^-$$
$$+2H^+ \; M^{4+} + H_2Y_2^- \rightarrow MY + 2H^+$$

One gram ion of the complex-forming ion H_2Y^{2-} reacts in all cases with one gram ion of the metal. EDTA forms complexes with metal ions in basic solutions. In acid-base titrations the end point is detected by a pH sensitive indicator. In the EDTA titration metal ion indicator is used to detect changes of pM. It is the negative logarithm of the free metal ion concentration, i.e., pM = -log $[M^{2+}]$. Metal ion complexes form complexes with specific metal ions. These differ in colour from the free indicator and a sudden colour change occurs at the end point. End point can be detected usually with an indicator or instrumentally by potentiometric or conductometric (electrometric) method.



There are three factors that are important in determining the magnitude of break in titration curve at end point.

- **The stability of complex formed:** The greater the stability constant for complex formed, larger the charge in free metal concentration (pM) at equivalent point, more clear would be the end point.

- **The number of steps involved in complex formation:** Fewer the number of steps required in the formation of the complex, greater would be the break in titration curve at equivalent point and clearer would be the end point.
- **Effect of pH:** During a complexometric titration, the pH must be constant by use of a buffer solution. Control of pH is important since the H^+ ion plays an important role in chelation. Most ligands are basic and bind to H^+ ions throughout a wide range of pH. Some of these H^+ ions are frequently displaced from the ligands (chelating agents) by the metal during chelate formation.
- Equation below shows complexation between metal ion and H^+ ion for ligand:

$$M_2^+ + H_2\text{-EDTA} \rightarrow \text{M-EDTA} + 2H^+$$

Thus, stability of metal complex is pH dependent. Lower the pH of the solution, lesser would be the stability of complex (because more H^+ ions are available to compete with the metal ions for ligand). Only metals that form very stable complexes can be titrated in acidic solution, and metals forming weak complexes can only be effectively titrated in alkaline solution.

Mechanism of action of indicator:

During an EDTA titration 2 complexes are formed: i) M-EDTA complex and ii) M-indicator complex. The metal-indicator complex must be less stable than the metal-indicator complex.

$$\text{M-In} + \text{EDTA} \rightarrow \text{M-EDTA} + \text{In}$$

Erichrome black T is a metal ion indicator. In the pH range 7-11 the dye itself has a blue colour. In this pH range addition of metallic salts produces a brilliant change in colour from blue to red.

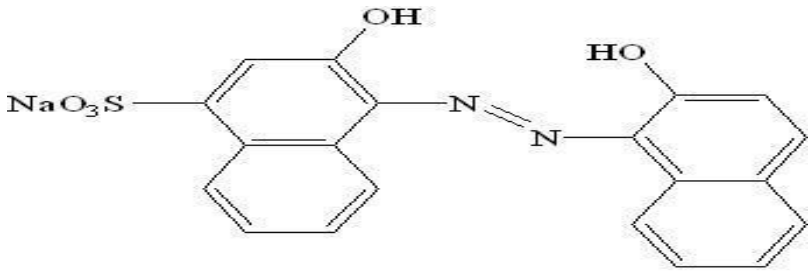
$$M^{2+} + \underset{\text{(Blue)}}{HIn^{2-}} \rightarrow \underset{\text{(Red)}}{MIn^-} + H^+$$

This colour change can be obtained with the metal ions. As the EDTA solution is added, the concentration of the metal ion in the solution decreases due to the formation of metal-EDTA complex. At the end point no more free metal ions are present in the solution. At this stage, the free indicator is liberated and hence the colour changes from red toblue.

Indicators used in complexometric titrations are as follows:

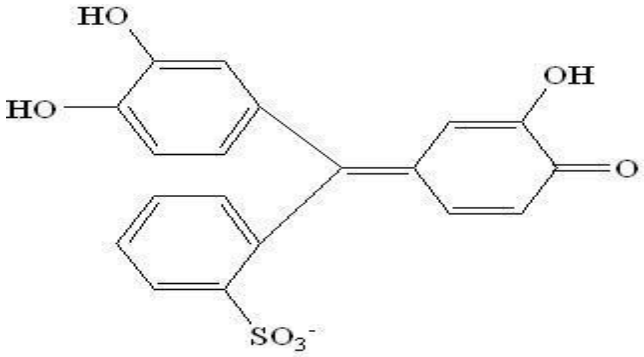
S.No.	Name of indicator	Colour change	pH range	Metals detected
1	Mordant black II	Red to	6-7	Ca,Ba

	Eriochrome black T	Blue		Mg,Zn,Cd,Mn,Pb,Hg
	Solochrome black T			
2	Murexide or Ammonium purpurate	Violet to Blue	12	Ca,cu,Co
3	Catechol-violet	Violet to Red	8-10	Mn,Mg,Fe,Co,Pb
4	Methyl Blue	Blue to Yellow	4-5	Pb,Zn,Cd,Hg
	Thymol Blue	Blue to Grey	10-12	
5	Alizarin	Red to Yellow	4.3	Pb,Zn,Co,Mg,Cu
6	Sodium Alizarin sulphonate	Blue to Red	4	Al, Thorium
7	**Xylenol range**	**Lemon to Yellow**	**1-3**	**Bi, Thorium**
			4-5	Pb, Zn
			5-6	Cd, Hg

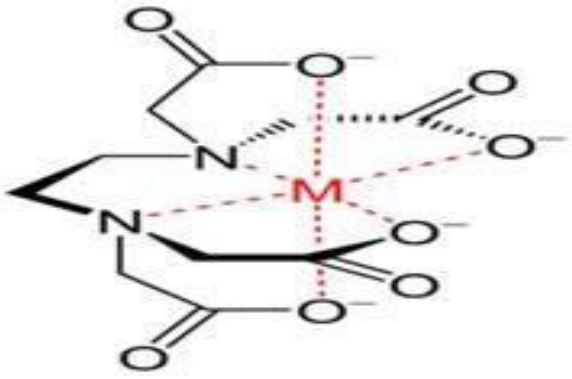


Calcone (mordant black 17)

Eriochrome Blue Black R



Catechol - violet



(Metal-EDTA complex)

Applications of Complexometric titration:

- Complexometric titration is widely used in the medical industry because of the micro litre size sample involved. The method is efficient in research related to the biological cell.
- Ability to titrate the amount of ions in a living cell.
- Ability to introduce ions into a cell in case of deficiencies. Complexometric titration involves the treatment of complex ions such as magnesium, calcium, copper, iron, nickel, lead and zinc with EDTA as the complexing agent.
- Complexometric titration is an efficient method for determining the level of hardness of water.

CONDUCTOMETRY

Conductometric analysis is based on the measurement of the electrical conductivity of the solution. The electrical conductivity is entirely due to the movement of ions. The conductometry is used in direct and indirect methods of physico-chemical analysis. It is widely used in complexometric titrations, chemical kinetics, precipitation titrations and plant laboratories.

Conductance :

In case of electrolytes, the term conductance (C) is generally used. The ease with which the current flows through a conductor. Thus, the conductance is the reciprocal of resistance. Mathematically, It is expressed in units of reciprocal ohms or mhos.

Ohm's law:

Ohm's Law is obeyed by a metallic as well as by electrolytic conductors. According to this law, "the strength of current (I) flowing through a conductor is directly proportional to the potential difference (E) applied across the conductor and inversely proportional to the resistance (R) of the conductor". Mathematically, the law can be represented as The current is measure in amperes, potential difference in volts and electrical resistance in ohms.

$$I = E/R$$

Where,

I= current in amperes

E= potential difference(Volts)

R= Resistance in ohm's

Specific Conductance:

Specific conductance can be defined as " the conductivity offered by a solution of length 1 cm and area of 1sq.cm cross section." It is expressed in mhos/cm. The specific conductance of an conductor is the reciprocal of specific resistance and is denoted by k .Mathematically,

Specific conductance= conductance X cell constant

$$K_v = 1/\rho$$

Equivalent conductance:

Equivalent conductance can be defined as the conductivity offered by a solution containing 1 gm equivalent weight of solute in it.

Molar conductivity (μ_v): The conductivity of a solution containing one molecular weight of solute between electrodes, 1cm apart and 1 sq.cm surface area

Molar conductivity= Specific conductivity X vol. of solution containing one molecular weight of the electrolyte

Specific resistance (ρ): It is the resistance offered by unit volume of an electrolyte solution(1 cm^3) substance 1 cm length and 1 sq.cm surface area. Units of measurement is mhos cm^{-1}

Relation between conductivity & resistivity: Conductivity is reciprocal and is expressed in mhows or Siemens

$$C = 1/R$$

Where, C mhos, R in ohms

1 Mho= 1 Siemens

Conductivity cell: It consists of two platinum foils fixed flush with insulating container which serves to isolate a portion of the liquid. The electrodes are coated with spongy platinum black, which increase the effective surface area and reduce the polarization of the exposed electrode surface. This coating can be easily deposited and is resistance to mechanical or chemical abrasion

Conductometric measurements :

1. Primary standard KCl solution ,at 25 ℃, 7.419g of KCl in 1000g of solution has a specific conductivity of 0.01286 Ω^{-1} /cm
2. Electrodes two parallel platinized Pt. foil electrodes or Pt. black electrodeposited on Pt foils . which increase the surface area of the electrodes and further reduce faradaic polarization..
3. Conductivity cell , Avoid the change of temperature during determination .
4. Wheat stone bridge

Factors affecting conductivity:

Size of ions,
Temperature ,
Number of ions and
Charge of ions

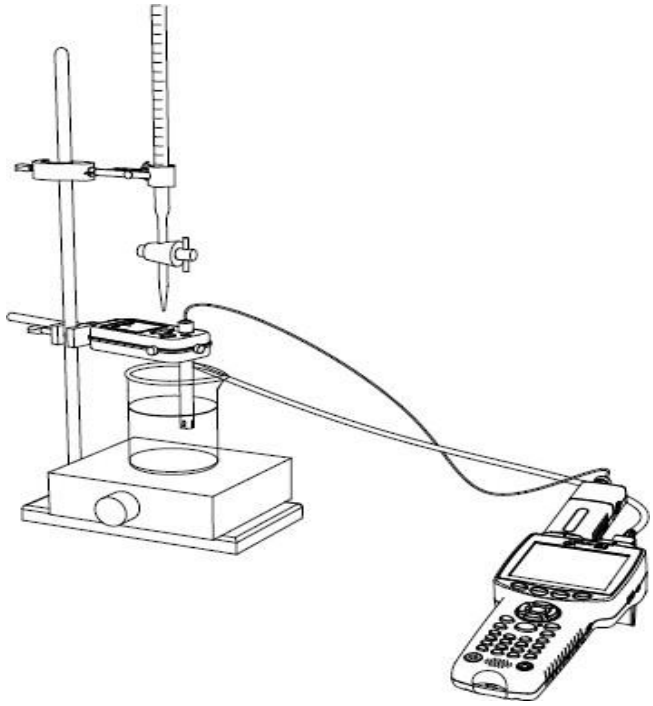
Applications of conductometry:

It can be used for the determination of, solubility of sparingly soluble salts, ionic product of water, basicity of organic acids, salinity of sea water (oceanographic work), chemical equilibrium in ionic reactions, degree of dissociation of weak electrolyte and degree of Hydrolysis

Conductometric titration: Introduction conductometric Titration (measurements) provide a convenient means for locating end point in Titration, Sufficient measurements are needed to define the Titration curve. After being corrected for volume change, the conductance data are plotted as a function of titrant volume. The two linear portions are then extra plotted , the point of intersection taken as equivalent point (or) end point. The conduct metric end point is completely non-specific although it is potentially Adaptable to all type of volumetric reaction. definition It is defined as that the determination of the end point of a titration with the help of conductivity measurement is termed as conductometric titration.

Apparatus for conductometric titarions:

Apparatus for conductometric titarions Measuring Circuit: In order to prevent concentration change due to reactions at electrodes the conductivity of solution generally measured with alternating currents at frequencies from 60 to 1000cps. Most of the circuits used are of the Wheatstone bridge type. commercial apparatus : The commercially available equipment is accurate enough for routine conductometric titarions . The important advantage of this instrument is that it is convenient and can be set up and used without the knowledge of electronic or electricity.



Conductometter

Types of conductometric titrations :

Types of conductometric titrations acid-base titration replacement titration precipitation titration redox -titration complexometric titration

Acid-base titration:

(a) strong acid with strong base e.g. HCl with NaOH
(b) strong acid with weak base e.g. HCl with NH_4OH
(c) weak acid with strong base e.g. CH_3COOH with NaOH
(d) weak acid with weak base e.g. CH_3COOH with NH_4OH :

(e) Replacement titration of strong acid with Sodium salt of weak Acid e.g. HCl with CH_3COONa :

Precipitation titration :

The precipitation titration can't be carried out so effectively as the acid-base titration. This is so because the acid-base titrations are characterized by sharp breaks because both the hydrogen and hydroxyl ions have very high equivalent conductance's. Consider the titration between silver nitrate and potassium chloride.

Redox (Oxidation-reduction) titration:

In case of oxidation – reduction titrations, there is a decrease in hydrogen ion concentration . The redox reactions give good result only when the initial concentration of acid is low.

Complexometric titration:

Complexometric Titration have been used in the study of formulae of complex compounds. Penta ammino cobalt sulphate $[Co(NH_3)_5H_2O]_2(SO_4)_3$ with Barium hydroxide and obtained two breaks in the titration curve.

Cell constant and its determinations: If c is the conductance of a solution measured by a cell with electrode of cross sectional area a cm^2 and l cm apart, the specific conductivity of the solution is given by

$$K = C \times l/a$$

Where,

$$l/a = K/c \qquad (l/a = K)$$

The cell constant, the ratio l/a cannot be obtained from the geometrical dimension of the cell for both l and a are accurately known. It is therefore necessary to calibrate the cell with a solution of known specific conductivity, usually 0.1 or 0.01 KCl solution is used.

Cell constant (K) = Specific conductivity/ conductance

Procedure Prepare 0.1M KCl solution and transfer the solution in 100ml beaker. Wash the conductivity cell with distilled water and wipe (electrode) with tissue paper. Dip the electrode in the KCl solution taken in a beaker and measure the conductance.

Calculations

Specific conductivity of KCl ; units ohm^{-1} cm^{-1}

0.1 N specific conductance is 0.01224

0.01 N specific conductance is 0.001412 at 25^0C

0.1 = Wt /74.5 x 1000/100

To prepare 0.1 N KCl in 100 ml wt(x) is

X = 0.1 x 74.5/10=0.745 g.

To prepare 0.01 N KCl in 100 ml from the above solution is $M_1V_1 = M_2V_2$

0.1 x V1 = 0.01 x 100

V_1 = 0.01 x 100/0.1 =10 ml (10 ml of the stock has to be diluted to 100ml)

10^6 μΩ =1Ω

1 μΩ =10^{-6} Ω

Conductivity = 13.44 μΩ

= 13.44 x1000

=13440 μΩ= 13440 x 10^{-6} Ω

K = 0.01224/0.01344 =0.91 cm^{-1}

Method of calibration:

(i) Prepare KCl solution and allow is at attain room temperature. The solution so prepared is temperature dependant

(ii) Dip the cell K =1 in the solution

(iv) Check that the std. cond. Switch is in the upward position

(v) Turn the standardize shaft with a screwdriver till the display reads the correct conductance of the solution . The temperature effect has to be considered.

Advantages of conductometric titrations:

Temperature is maintained constant throughout the titration. End point can be determined accurately and errors are minimized as the end point is being determined graphically. They can be used in the case of colored liquids where ordinary indicators cannot work. They can be used for the analysis of dilute solutions and also for very weak acids .

Recent developments:

In refinery industries. In the estimation of poly electrolytic solution. In biotechnology. In Micro biosensors for environmental monitoring.

Potentiometer

Potentiometer: It is a device or circuit used for comparing potential sources.

Principle of potentiometric titration:

The principle involved in potentiometric titration is the measurement of the emf between two electrodes, an indicator electrode and a reference electrode. In these titrations, measurement of emf are made while the titration is in progress. The equivalence point of the reaction is revealed by a sudden change in potential in the plot of emf readings against the volume of titrant.

Potentiometric tritration:

The determination of the equivalent point of acid-base titrations on the basis of potential measurements is called potentiometric titration.

Single electrode potential: The potential that is developed when an element is in contact with a solution containing its own ions is called single electrode potential.

Standard electrode potential: The potential that is developed when an element is in contact with a solution containing its own ions of 1M concentration at 298 K is referred to as standard electrode potential. If the gasses are involved , they must be passed at a partial pressure of 1 atmosphere.

EMF: It is the potential difference required to drive a current across the electrodes.

E.M.F = E cathode - E anode

Electrodes used in potentiometric titration:

The indicator electrode used is the platinum electrode (acts as an anode) and the reference electrode used is the calomel electrode (acts as a cathode).

Advantages of potentiometric titrations:

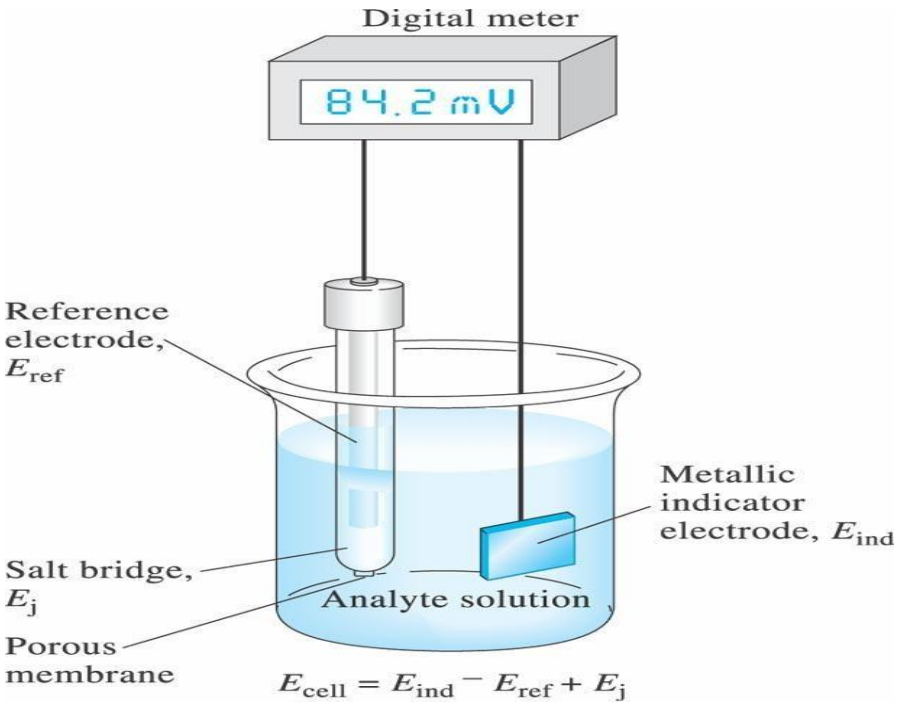
1) Turbid, fluorescent, opaque or coloured solutions can be titrated.
2) Mixture of solutions or very dilute solutions can be titrated.
3) The results are more accurate because the actual end point is determined graphically.

Potentiometric Measurements: A reference electrode, an indicator electrode, and a potential-measuring device based on measuring the potential of electrochemical cells without drawing

appreciable current. Used to– locate end points in titrations. Determine ion concentrations with ion-selective membrane electrodes measure the pH
determine thermodynamic equilibrium constants such as K_a ,K_b and K_{sp}

General Principles for potentiometric analysis:

Potentiometric Analysis:



- Reference electrode:

A half-cell with an accurately known electrode potential, *E*ref, that is independent of the concentration of the analyte or any other ions in the solution– Always treated as the left-hand electrode• Indicator electrode– which is immersed in a solution of the analyte, develops a potential, *E*ind, that depends on the activity of the analyte. Is selective in its response• Salt bridge preventing components of the analyte solution from mixing with those of the reference electrode . A potential develops across the liquid junctions at each end of the salt bridge Potassium chloride is a nearly ideal electrolyte for the salt bridge because the mobilities of the K^+ ion and the Cl^- ion are nearly equal. Reference Electrodes Ideal reference electrode has a potential that is accurately known, constant and
completely insensitive to the composition of the analyte solution calomel reference electrodes:

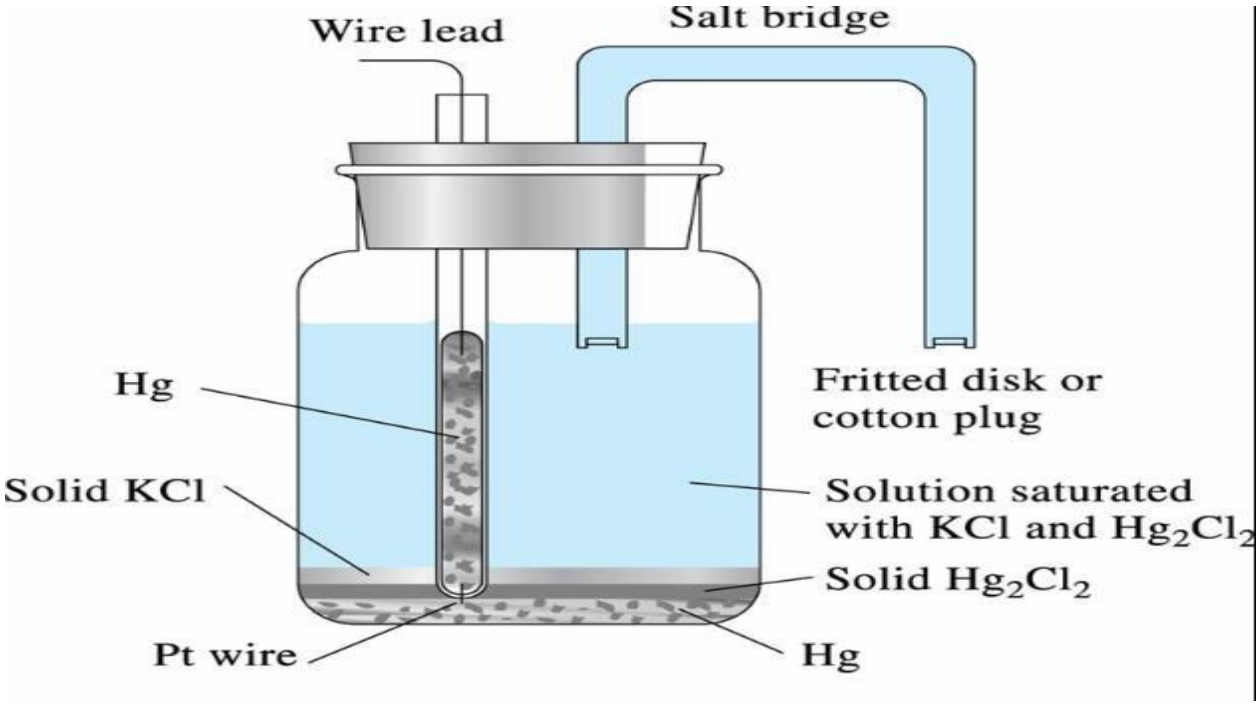
$$Hg \mid Hg_2Cl_2(sat'd),\ KCl(x\ M) \parallel$$

where *x* represents the molar concentration of potassium chloride in the solution. Concentrations of potassium chloride that are commonly used in calomel reference electrodes are 0.1 M, 1 M, and saturated(about 4.6 M).

The saturated calomel electrode (SCE) is the most widely used because it is easily prepared. The potential is 0.2444 V at 25°C. The electrode reaction in calomel half-cell:

$$Hg_2Cl_2(s) + 2e^- \rightleftharpoons 2Hg(l) + 2Cl^-(aq)$$

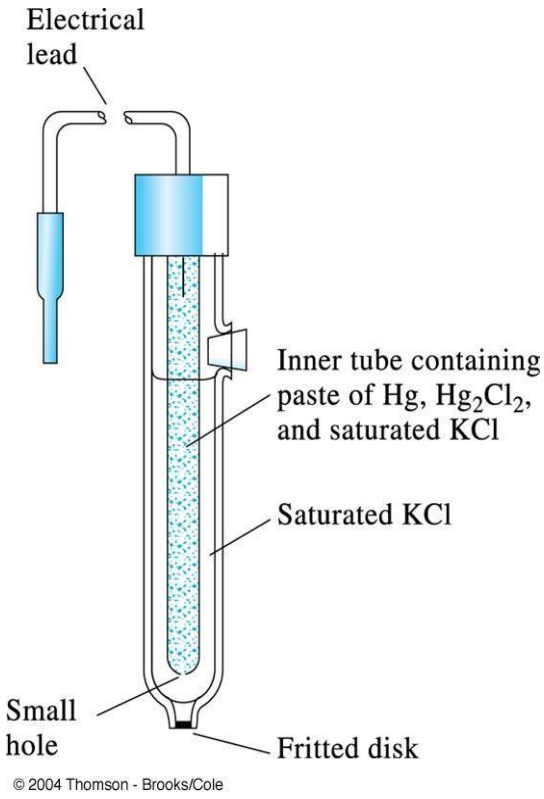
Commercial Saturated Calomel Electrode:



Liquid junction consisting of a 1 M HCl solution that is in contact with a solution that is 0.01 M HCl Both H^+ and Cl^- ions tend to diffuse across the inert porous barrier H^+ ions diffuse more rapidly than Cl^- ions, and a separation of charge results the potential difference resulting from this charge separation is the junction potential

The magnitude of the liquid-junction potential can be minimized by placing a salt bridge between the two solutions.

The mobilities of the negative and positive ions are nearly equal a saturated solution of potassium chloride is good from both standpoints. The net junction potential with such a bridge is typically a few millivolts



Electrodes of the First Kind:

A pure metal electrode that is in direct equilibrium with its cation in the solution:

$$X^{n+}(aq) + ne^- \rightleftharpoons X(s)$$

For which,

$$E_{ind} = E^0_{X^{n+}/X} - \frac{0.0592}{n} \log \frac{1}{a_{X^{n+}}} = E^0_{X^{n+}/X} + \frac{0.0592}{n} \log a_{X^{n+}}$$

Electrodes of the First Kind: A pure metal electrode that is in direct equilibrium with its cation in the solution, For which,

$$E_{ind} = E^0_{X^{n+}/X} + \frac{0.0592}{n} \log a_{X^{n+}} = E^0_{X^{n+}/X} - \frac{0.0592}{n} pX$$

Where, *E*ind is the electrode potential of the metal electrode and a x^{n+} is the activity of the ion (or, in dilute solution, approximately its molar concentration, $[Xn^+]$). **Or**

$$E_{ind} = E^0_{X^{n+}/X} + \frac{0.0592}{n} \log a_{X^{n+}}$$

Electrodes of the Second Kind:

- Metal electrode respond to the activities of anions that form sparingly soluble precipitates or stable complexes with such cations.

$$AgCl(s) + e^- \rightleftharpoons Ag(s) + Cl^-(aq) \qquad E^0_{AgCl/Ag} = 0.222\ V$$

The Nernst expression for this process at 25ºC is:

$$E_{ind} = E^0_{AgCl/Ag} - 0.0592 \log a_{Cl^-} = E^0_{AgCl/Ag} + 0.0592\ pC$$

Electrodes of the Second Kind

Mercury serves as an indicator electrode of the

$$HgY^{2-} + 2e^- \rightleftharpoons Hg(l) + Y^{4-} \qquad E^0 = 0.21\ V$$

Second kind for the EDTA anion Y4-:

$$E_{ind} = 0.21 - \frac{0.0592}{2} \log \frac{a_{Y^{4-}}}{a_{HgY^{2-}}}$$

The Nernst expression for this process:

$$E = K - \frac{0.0592}{2} \log a_{Y^{4-}} = K + \frac{0.0592}{2}\ pY$$

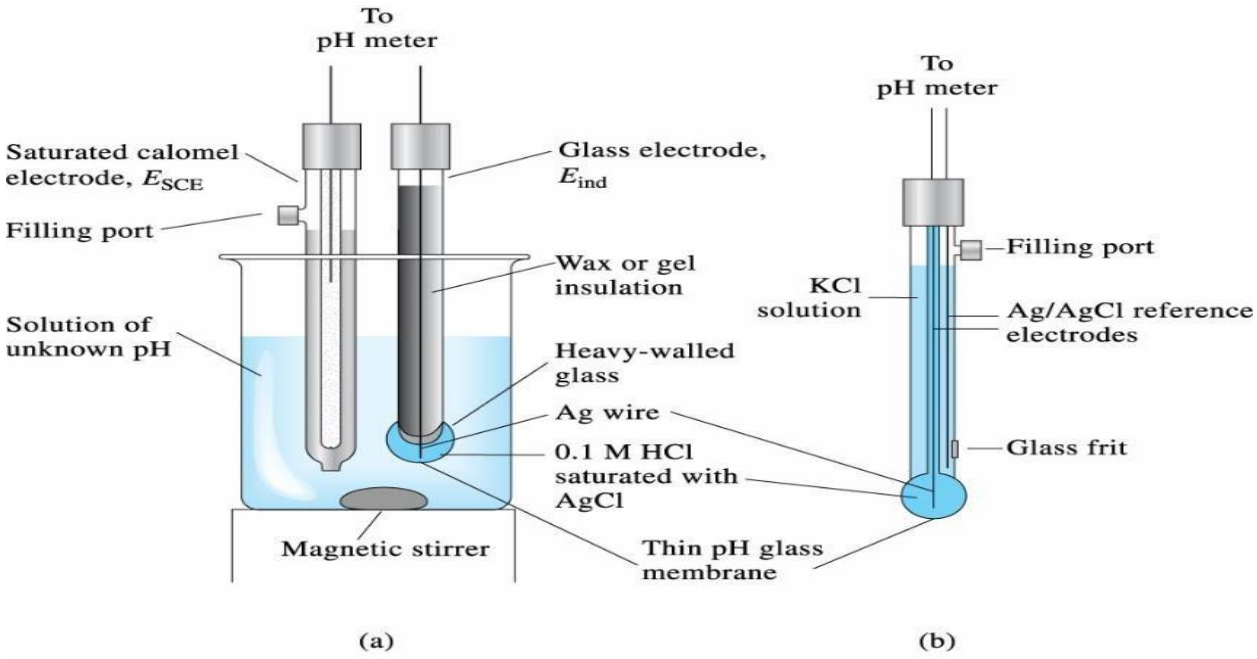
Inert metallic electrodes for redox systems: Several inert conductors can be used to monitor redox systems. Such as platinum, gold, palladium, and carbon Ex:

Platinum electrode immersed in a solution containing cerium (III) and cerium (IV):

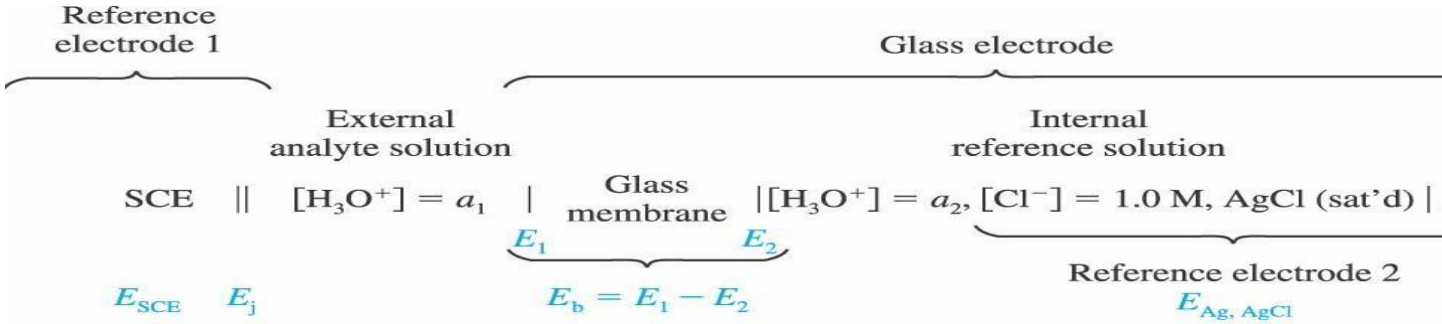
$$E_{ind} = E^0_{Ce^{4+}/Ce^{3+}} - 0.0592 \log \frac{a_{Ce^{3+}}}{a_{Ce^{4+}}}$$

a convenient indicator electrode for titrations involving standard cerium(IV) solutions

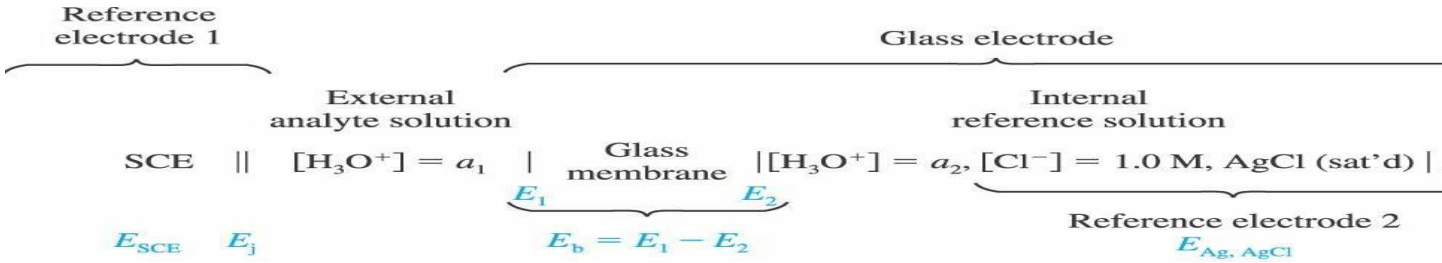
pH Sensitive glass membrane electrode:
A glass electrode system contains two reference electrodes: the external calomel electrode (Immersed in a solution of unknown pH) and the internal silver/silver chloride electrode the internal electrode consists of
a thin, pH-sensitive glass membrane sealed onto one end of a heavy-walled glass or plastic tube a small volume of dilute hydrochloric acid saturated with silver chloride is contained in the tube. A silver wire in this solution forms a silver/silver chloride reference electrode. It is the thin glass membrane bulb at the tip of the electrode that responds to pH. Typical electrode system for measuring pH



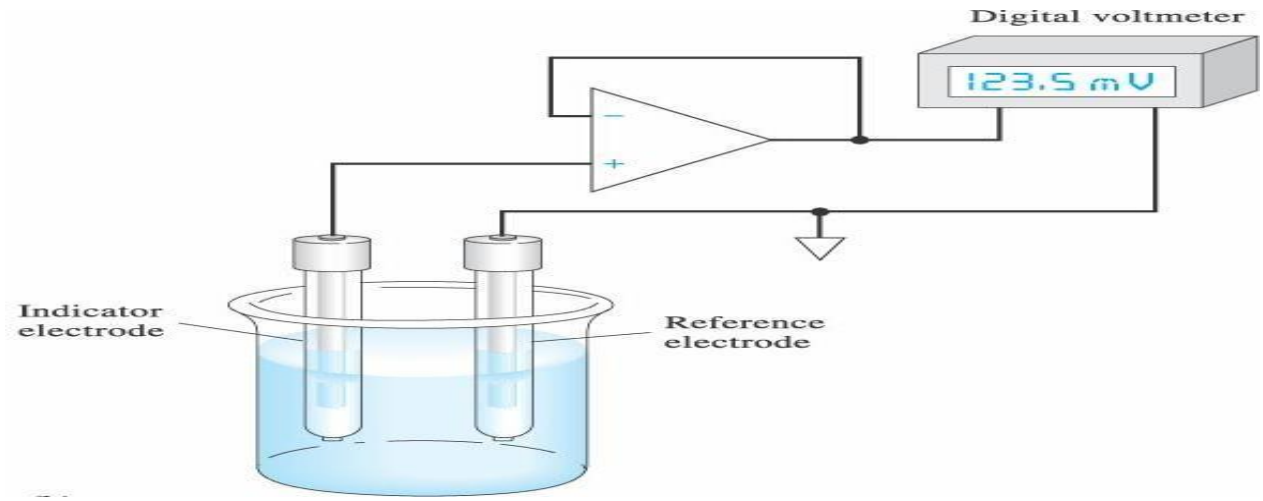
pH Meter Whenever there is a charge imbalance across any material, there is an electrical potential across the material the concentration of protons inside the membrane is constant, and the concentration outside is determined by the concentration, or activity, of the protons in the analyte solution. This concentration difference produces the
potential difference that we measure with a pH meter cell potentials The potentials of the two reference electrodes
depend on the electrochemical characteristics of their respective redox couples. The potential across the glass membrane depends on the physicochemical characteristics of the glass and its response to ionic concentrations on both sides of the membrane



Cell Potentials:



4 potentials:
*E*Ag,AgCl and *E*SCE, are reference electrode potentials that are constant.– A 3rd potential is the junction potential *E*j across the salt bridge that separates the calomel electrode from the analyte solution.– The 4th and most important potential is the **boundary potential***E*b, *which varies with the pH of the analyte solution*.Instruments for measuring



Cell potential the voltmeter should have an electrical resistance that is several orders of magnitude greater than the resistance of the cell being measured: Numerous high-resistance, direct reading digital voltmeters with

internal resistances of > 1011 ohms are available. These meters are commonly called pH meters, pIon meters or ion direct potentiometry

The potential of the cell:

$$\mathrm{pA} = \frac{(E_{\mathrm{cell}} - K)}{0.0592/n} = \frac{n(E_{\mathrm{cell}} - K)}{0.0592}$$

$$E_{\mathrm{cell}} = E_{\mathrm{ind}} - E_{\mathrm{ref}} + E_{\mathrm{j}}$$

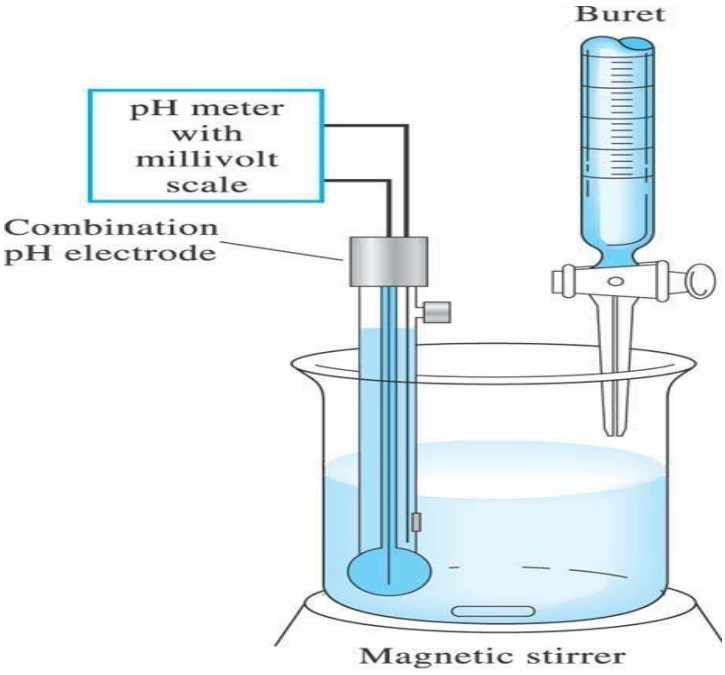
For the cation X^{n+} at 25°C, the electrode response takes the general *Nernstian* form:

$$E_{\mathrm{ind}} = L - \frac{0.0592}{n}\,\mathrm{pX} = L + \frac{0.0592}{n}\log a_{\mathrm{X}}$$

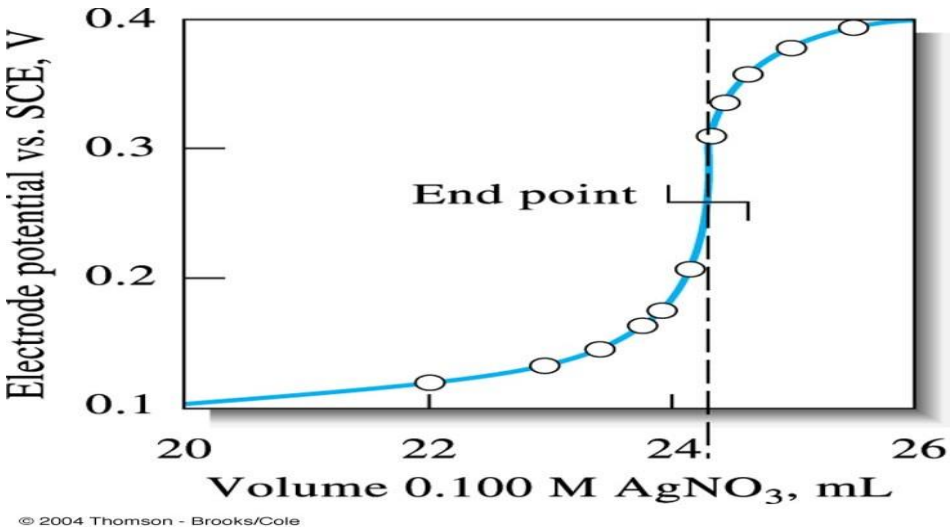
$$\mathrm{pX} = -\log a_{\mathrm{X}} = -\frac{(E_{\mathrm{cell}} - K)}{0.0592/n} = -\frac{n(E_{\mathrm{cell}} - K)}{0.0592}$$

Potentiometric Titrations:

A potentiometric titration involves measurement of the potential of a suitable indicator electrode as a function of titrant volume. The measurement is base on the titrant volume that causes a rapid change in potential near the equivalence point. Potentiometric titrations provide data that are more reliable than data from titrations that use chemical indicators. They are particularly useful with colored or turbid solutions and for detecting the presence of unsuspected species. Potentiometric titrations are not dependent on measuring absolute values of Ecell. Potentiometric titration results depend most heavily on having a titrant of accurately known concentration



Detecting the end point: A direct plot of potential as a function of reagent volume the inflection point
in the steeply rising portion of the curve, and take it as the end point.



EXP. NO:

DATE:

VOLUMETRIC ANALYSIS (EXPERIMENTES)

ESTIMATION OF SODIUM CARBONATE
(Acid-Base titration**)**

Aim: To estimate the amount of Sodium carbonate present in 100 ml of the given unknown solution using standard hydrochloric acid solution.

Apparatus: 100 ml standard flask, Burette, 250ml conical flask, 20ml pipette, funnel, measuring jar & simple balance.

Chemicals required:

Hydrogen chloride (HCl), Sodium carbonate (Na_2CO_3), Methyl orange indicator

Distilled water solution

Experimental : Preparation of reagents

0. 1 N Hydrogen chloride (HCl) solution: Dissolve **8.50** ml of (11.6 M lab strength) and 36% conc. HCl (Mol.Wt.36.5) to 1000ml with distilled water.

0.05 N Sodium carbonate solution: Weigh accurately about **5.3000** gms. of pure A.R grade pure anhydrous sample of sodium carbonate is transferred into 1000 ml volumetric flask. Dilute to the mark of distilled water

$$\text{Weight} = \frac{Normality X Gram molecular weight}{Given volume of solution in ml}$$

Methyl orange indicator: dissolve **0.2 g**. of the dye stuff in100 ml of distilled water

Principle: Sodium carbonate reacts with hydrochloric acid in the following equation

$2HCl + Na_2CO_3 \rightarrow 2NaCl + H_2O + CO_2$ (1 mole of Na_2CO_3 = 2 mole of HCl)

Hydrochloric acid is reacted with sodium carbonate in the basis of 2;1 ratio.

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$$

Preparation of standard hydrogen chloride solution: (Part-a)

Weigh out accurately the given pure sample of sodium carbonate and transfer into conical flask, dissolve the content with distilled water, and make up to the mark. The content in

the flask are shaken well for uniform concentration. Pipette out 20 ml of standard Sodium carbonate solution into a 250 ml conical flask, add 20 ml of distilled water with measuring jar and add few drops (1 or 2 drops) of Methyl orange indicator. Titrate the solution against hydrogen chloride solution until the end point appear to yellow color changes to pink colour. Repeat the titrations for concurrent values and note down the burette readings. Calculate the normality of hydrogen chloride solution.

S.No	Vol of Na_2CO_3 solution (ml)	Burette readings		Consumed Vol. of HCl (ml)
		Initial (ml)	Final (ml)	
1	20.00			
2	20.00			
3	20.00			

Formula:

$$\frac{N_1V_1}{n_1} = \frac{N_2V_2}{n_2}$$

Where,

HCl Solution Na_2CO_3 Solution

N_1= Normality of HCl Solution ? ; N_2= Normality of Na_2CO_3 Solution

V_1= Vol of HCl Solution (from table); V_2= Volume of Na_2CO_3 Solution 20.0 ml

n_1 = No . of moles of HCl= 2 n_2 = No . of moles of NaOH= 1

$$N_1 = \frac{N_2V_2n_1}{V_1n_2} =$$

Standardized HCl solution normality(Known) = ...N

Determination of sodium Carbonate solution: (Part-b)

Carefully dilute the unknown sample in the100 ml volumetric flask to the mark with de ionized water and mix thoroughly, results the prepared unknown solution. Pipette out 20 ml of standard Na_2CO_3 solution into a 250 ml conical flask, add 20.00 ml of distilled water with measuring jar and few ml of methyl orange indicator solution. This content is titrate against HCl solution until

yellow color changes to pink colur. Repeat the titrations for concurrent values and note down the burette readings. Calculate the normality of unknown Na_2CO_3 solution.

S.No	Vol.of Na_2CO_3 solution (ml)	Burette readings Initial (ml)	Final (ml)	Consumed Vol. of HCl (ml)
1	20.00			
2	20.00			
3	20.00			

Calculations: $\frac{N_3V_3}{n_3} = \frac{N_4V_4}{n_4}$ Where,

HCl Solution Unknown solution (Na_2CO_3)

N_3= Normality of HCl Solution (From standard); N_4= Normality of unknown Na_2CO_3 ?

V_3= Vol of HCl Solution (From table); V_4= Volume of unknown Na_2CO_3 20.0 ml

n_3 = No . of moles of HCl= 2 (From equation) n_4= No . of moles of NaOH= 1

Normality of unknown (Na_2CO_3) $N_4 = \frac{N_3V_3n_4}{V_4n_3}$ = ... N

The amount of 100 ml unknown Na_2CO_3 present in your given solution =

$$\frac{Normalityxmolecularweight}{10} = \text{---} \quad \text{gms.}$$

(Molecular weight of Na_2CO_3 = 105.9885 g/mol)

$$\% \text{ Error formula} = \frac{Givenvalue - \text{Re}portedvalue}{Givenvalue} \times 100$$

Final report: S.No.	Given value (gms)	Reported value (gms)	% error
1.			

Signature of the faculty

EXP. NO:

DATE:

Determination of alkalinity of a sample containing Na_2CO_3 and NaOH (Acid-Base titration)

Aim: To estimate the strength of Na_2CO_3 and NaOH in a given alkali mixture

Apparatus: 100 ml standard flask, Burette, 250ml conical flask, 20ml pipette, funnel, measuring jar & simple balance.

Chemicals required:

Hydrogen chloride (HCl), Sodium carbonate (Na_2CO_3), Methyl orange indicator

Distilled water solution

Experimental : Preparation of reagents

0. 1 N Hydrogen chloride (HCl) solution: Dissolve **8.50** ml of (11.6 M lab strength) and 36% conc. HCl (Mol.Wt.36.5) to 1000ml with distilled water.

N Sodium carbonate solution: Weigh accurately about **5.3000** gms. of pure A.R grade pure anhydrous sample of sodium carbonate is transferred into 1000 ml volumetric flask. Dilute to the mark of distilled water

$$\text{Weight} = \frac{Normality X Grammolecularweight}{Givenvolumeofsolutioninml}$$

Methyl orange indicator: dissolve **0.2 g**. of the dye stuff in100 ml of distilled water

Principle: Sodium carbonate reacts with hydrochloric acid in the following equation

$2HCl + Na_2CO_3 \rightarrow 2NaCl + H_2O + CO_2$ (1 mole of Na_2CO_3 = 2 mole of HCl)

Hydrochloric acid is reacted with sodium carbonate in the basis of 2;1 ratio.

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$$

$$NaOH + HCl \rightarrow NaHCO_3 + NaCl$$

Procedure:

i) The burette is rinsed and filled with given std.HCl solution
ii) 10 ml of mixture solution is pipette into a 100 ml.flask. Add 1-2 drops of phenolphathalein to it.
iii) Titrate the above mixture wit HCl solution till solutions turns colourless from pink colour.
iv) Add 2-3 methyl orange to the solution to make it orange

v) Titrate it with standard HCl solution til solution turns red from orange
vi) Note down the volume of HCl consumed with each indicator
vii) Repeat two more times, to get a consistant reading.

Observations:

S.NO.	Burette reading phenolphthalein			Burette reading vol. HCl with methyl orange			X-Y ml	2 y ml
1								
2								
3								

Calculation: **Estimation of NaOH** N_1= Normality of NaOH

V_1= Volume of NaOH

N_2= Normality of HCl

V_2= Volume of HCl

NaOH VS HCl

$$\mathbf{N_1V_1=N_2V_2}$$

$$N_1 = \frac{N2_1V2_1}{V1} = 0.1 \text{ x (X-Y)}$$

Strength of NaOH= Normality X 40 g/l =……..

Estimation of Na_2CO_3: N_1= Normality of Na_2CO_3

V_1= Volume of Na_2CO_3

V_2= Volume of HCl=2y ml

N2= Normality of HCl=0.1 N

$$N_1 = \frac{N\ 2\ V\ 2}{V1}$$

$$= 0.1 \text{ X } 2y \text{ mL}$$

Strength of Na_2CO_3== Normality X 53 g/l=……

Signature of the faculty

EXP. NO:

DATE:

ESTIMATION OF SODIUM HYDROXIDE

(Acid-Base titration)

Aim: To estimate the amount of Sodium hydroxide present in 100 ml of the solution using standard hydrochloric acid solution.

Apparatus: 100 ml standard flask, Burette, 250ml conical flask, 20ml pipette, funnel, measuring jar & simple balance.

Chemicals required:

Hydrogen chloride (HCl)

Sodium hydroxide (NaOH)

Methyl orange indicator

Distilled water solution

Experimental :

Preparation of reagents

M Hydrogen chloride (HCl) solution: Dissolve 0.86 ml of 11.6 M and 36% conc.HCl (Mol.Wt.36.5) to 100ml with distilled water.

0.1 M Sodium hydroxide solution: Weigh accurately about 4.00 gms. of pure A.R grade pure anhydrous sample of sodium hydroxide is transferred o 1000ml volumetric flask. Dilute to the mark of distilled water

$$\text{Weight} = \frac{Molarity X molecular weight}{Given volume of solution in ml}$$

Methyl orange indicator: dissolve 0.2 g. of the dye stuff in100 ml of distilled water

Principle: Sodium hydroxide reacts with hydrochloric acid in the following equation

$$HCl + NaOH \rightarrow NaCl + H_2O$$ (1 mole of NaOH = 1 mole of HCl)

Hydrochloric acid is reacted with sodium hydroxide in the basis of 1:1 ratio.

Preparation of standard hydrogen chloride solution: (Part-a)

Weigh out accurately the given pure sample of sodium hydroxide and transfer into conical flask, dissolve the content with distilled water, and make up to the mark. The content in the flask are shaken well for uniform concentration. Pipette out 20 ml of standard Sodium hydroxide solution into a 250 ml conical flask, add few drops (1 or 2 drops) of Methyl orange indicator. Titrate the solution against Hydrogen chloride solution until the end point appear to yellow color changes to pale pink. Repeat the titrations for concurrent values and note down the burette readings. Calculate the molarity of hydrogen chloride solution.

S.No	Vol of NaOH solution (ml)	Burette readings		Consumed Vol. of HCl (ml)
		Initial (ml)	Final (ml)	
1	20.00	00.00		
2	20.00			
3	20.00			

Formula: $M_1V_1 = M_2V_2$

Where,

HCl Solution NaOH Solution

M_1= Molarity of HCl Solution ? ; M_2= Molarity of NaOH Solution

V_1= Vol of HCl Solution (from table); V_2= Volume of NaOH Solution 20.0 ml

$$M_1 = \frac{M_2V_2}{V_1}$$

Standardized HCl solution Molarity = ...M

Determination of sodium hydroxide solution: (Part-b)

Carefully dilute the unknown sample in the100 ml volumetric flask to the mark with de ionized water and mix thoroughly, results the prepared unknown solution. Pipette out 20 ml of standard NaOH solution into a 250 ml conical flask, add few ml of methyl orange indicator solution. This content is titrate against HCl solution until yellow color changes to pale pink. Repeat the titrations for concurrent values and note down the burette readings. Calculate the molarity of unknown NaOH solution.

S.No	Vol.of NaOH solution (ml)	Burette readings Initial (ml)	Final (ml)	Consumed Vol. of HCl (ml)
1	20.00			
2	20.00			
3	20.00			

Calculations: $M_3V_3 = M_4V_4$

Where,

HCl Solution Unknown solution (NaOH)

M_3= Molarity of HCl Solution (from standard); M_4= Molarity of unknown NaOH ?

V_3= Vol of HCl Solution (from table); V_4= Volume of unknown NaOH 20.0 ml

$$M_4 = \frac{M_3V_3}{V_4}$$

Molarity of unknown (NaOH) =.....M

The amount of 100 ml unknown NaOH present in your given solution =

$$\frac{\text{Molarity X Molecular weight of NaOH}}{10} = \text{--------gms.}$$

(Molecular weight of NaOH =40 gL^{-1})

$$\% \text{ Error formula} = \frac{Given value - Reported value}{Given value} x100$$

Final report:

S.No.	Given value (gms)	Reported value (gms)	% error
1.			

Signature of the faculty

EXP. NO:

DATE:

DETERMINATION OF TOTAL HARDNESS OF WATER

(Complexometric Titration)

Aim :

To determine the total hardness of a given sample of water.

Apparatus :

Burette, Pipette, Burette stand, Conical flask.

Solutions required:

0.01 N EDTA Solution:

Disslove **0.7306** gms of AR disodium dihydrogen ethylenediamine tetra acetate dehydrate in water and dilute it to 250 ml in a volumetric flask with distilled water.

Ammonium chloride ammonium buffer:

Dissolve 17.5 gms of ammonium chloride in 142 ml of liquor ammonia and dilute it to 250 ml in a volumetric flask. This will maintain a P^H-10.

Erichrome black –T indicator:

The indicator solution is prepared by dissolving 0.2 gms of the dyestuff in 15 ml of triethanolamine and 5 ml of rectified spirit. Here triethanolamine serves the purpose of masking the interfering cations. Alternatively 0.4% solution of pure dyestuff in ethanol also can be made it stable for at least one month.

Theory:

The hardness of water is due to dissolved calcium and magnesium salts and may be determined by complexometric titration. Both calcium and magnesium form 1:1 complexes with EDTA. The total hardness includes permanent and temporary hardness.

Procedure:

Take 100ml tap water from burette into a 250 ml conical flask. To this add 2ml of buffer (pH-10) and two drops of eriochrome black-T indicator and titrate with 0.01 EDTA solution until the color changes from wine red to pale blue. The experiment is repeated till the concurrent readings are obtained. Then the readings are entered in the tabular form.

S.NO	Volume of tap water (ml)	Burette readings		Volume of EDTA rundown (ml)
		Initial	Final	
1	100 ml	0.0		
2	100 ml			
3	100 ml			

Calculations :

Normality of Hardness of water is calculated using the equation.=

$$N_1V_1 = N_2V_2$$

Where,

EDTA

WATER SAMPLE

N_1 =Normality of EDTA Solution ..... N N_2 =Normality of Hard water=?

V_1 = Vol of EDTA solution = ml V_2 = Vol. of Hard water.....ml

Normality of EDTA (N_2) = N_1V_1/V_2

Total hardness of water is calculated in terms of the amount of $CaCO_3$ present in million parts of water (ppm)

Amount of $CaCO_3$ present is100 ml. of the solution given solution

$$= \frac{N_2 \text{ X Eq.wt. of } CaCO_3 \text{ X } 1000}{\text{Water sample taken value}}$$

Another calculation: **(Or)**

Total hardness of water is calculated in terms of the amount of $CaCO_3$ present in millions parts of water (ppm)
Calcium forms 1: 1 complexes with EDTA so that we can write it as

1 ml of 0.01M EDTA = 1 mg of $CaCO_3$

Mol. Wt of $CaCo_3$ is 100.

Volume of 0.01M EDTA consumed for 100 ml tap water is x ml, then

$\frac{0.01 \times x}{100}$

is the strength of $CaCO_3$ or $0.01 \times x$ gms is present in 1 lt.

Therefore, total hardness = $1000 \times 0.01 \times x$ ppm

$= 10\ x$ ppm

Report :

The Total Hardness of given sample of water =.....ppm

Signature of the faculty

EXP. NO:

DATE:

ESTIMATION OF FERROUS IRON

(Redox-Titration)

Aim: To estimate the amount of ferrous iron solution using a standard solution of potassium dichromate solution.

Apparatus: 100 ml standard flask, Burette, 250 ml conical flask,20 ml pipette, funnel & simple balance.

Chemicals required:

Potassium dichromate ($K_2Cr_2O_7$)

Syrup phosphoric acid(H_3PO_4)

Diphenylamine indicator

Ferrous iron solution

Distilled water solution

Theory: Redox reactions are those in which the titration of reducing agent is carried out with an oxidizing agent .some of the oxidizing agents are

1. Potassium permanganate
2. Potassium dichromate
3. Cerium(IV) sulfate
4. Iodine
5. Potassium iodate
6. Potassium bromated and
7. Sodium vanadate

Some of the reducing agents such as arsenic (III) oxide and sodium thiosulphate can be titrated with an oxidizing agent like iodine. For example, in the reaction.

$$2\ FeCl_3 + SnCl_2 \rightarrow 2\ FeCl_2 + \ nCl_4$$

The oxidation number of iron is reduced from +3 to + 2 and that of tin is increased from +2 to +4 .Hence ,we say that iron undergoes reduction and tin undergo oxidation in this reaction. In such reactions ferric chloride is called as as oxidizing agent or oxidant and stannous chloride is called the reducing agent or reductant. The overall reaction is called redox reaction. Further no oxidation proceeds in absence of reduction or there cannot be reactions in which only oxidation

reduction takes place. Redox reactions play an important rule in quantitative analysis. In volumetric analysis redox titrations form a major part.

Potassium dichromate and sodium vanadate are used as oxidants in acid medium in the titration of ferrous ammonium sulphate solutions.

Potassium dichromate ($K_2C\ r_2O_7$):

Potassium dichromate is one of the most widely used oxidizing titrant. It is a mild oxidant and not as powerful as potassium permanganate. Its formal potential in 1M H_2SO_4 is 1.03V.Potassium dichromate is a stable substance and can be obtained in a pure state. It is used as a primary standard and standard solutions of potassium dichromate can be prepared by weighing an appropriate amount of the substance and dissolving in water. Unlike potassium permanganate, dichromate solutions are quite stable and do not need any special precautions in their storage.

In dilute acidic media potassium dichromate acts as an oxidizing agent in which chromium (VI) is reduced to chromium (III) involving multi de-electronation.

$$Cr_2O_7{}^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2; E = 1.03Volt$$

Oxidations with potassium dichromate also takes place by different mechanisms and initiate induced reactions. Chromium(V) and chromium(IV) species are unstable and chromium (II)salts are less stable. Freshly formed chromium (III) solutions (chlorocomplexes) are green in colour and change gradually to violet colour (hydrated complex of chromium (III)

Experimental : Preparation of solutions

0.01 N Potassium dichromate ($K_2Cr_2O_7$): Weigh accurately about **0.49** gms. of pure A.R grade pure anhydrous sample of potassium dichromate ($K_2Cr_2O_7$) is transferred into 1L dilute to the mark of distilled water. Shake the flask to mix the solution thoroughly. Equivalent weight of potassium dichromate is 49

Weight = 0.01 X equivalent weight/ Vol. of solution in ml

$[Cr_2O_7^{2-} + 14\ H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$
Hence, equivalent weight=294/6=49]

N Ferrous ammonium sulfate solution: Weigh accurately about **1.9607** gms. of pure A.R grade pure anhydrous sample of Ferrous ammonium sulphate ($(Fe_2(SO_4)_3(NH_4)_2\ SO_4\ .24\ H_2O$) mohr's salt) into a 1000 ml volumetric flask, add about 200 ml of 5 N sulphuric acid to dissolve the solid and finally make the solution up to the mark with distilled water.

1 %Diphenylamine (DPA) indicator: dissolve 1 gm. of the dye stuff in100 ml of concentrated H_2SO_4 in a 250 ml beaker and store it in a indicator bottle.

Principal: ferrous iron is oxidized to ferric iron by potassium dichromate in acid solution. the completion of oxidation reaction is marked by the appearance of blue violet color of diphenylamine, which is used as an internal indicator.

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2 + 3(O)$$

$$2FeSO_4 + H_2SO_4 + (O) \rightarrow Fe_2(SO_4)_3 + H_2O$$

$$Cr_2O_7^{-2} + 6Fe^{+2} + 14H^+ \rightarrow 6Fe^{+3} + 2Cr^{+3} + 7H_2O$$

Standardization of ferrous ammonium sulphate with potassium dichromate (Part-a)

Pipette out 20 ml of standard $FeSO_4$ solution into a 250 ml conical flask, add 20 ml of 5 N H_2SO_4 solution and 20.00 ml distilled water. To this add 3 ml syrupy phosphoric acid and four to six drops of diphenylamine indicator and titrate with potassium dichromate taken in a burette until a blue-violet color appears. Repeat the process until concordant titre values are obtained.

S.No	Vol of Fe^{+2}solution (ml)	Burette readings		Consumed Vol. of $K_2Cr_2O_7$ (ml)
		Initial (ml)	Final (ml)	
1	20.00	00.00		
2	20.00			
3	20.00			

Formula:

$$V_1N_1 = V_2N_2$$

Where, $K_2Cr_2O_7$ Solution Mohr's salt

N_1= Normality of $K_2Cr_2O_7$Solution? ; N_2= Normality of Fe^{+2} Solution

V_1= Vol of $K_2Cr_2O_7$ Solution (from table); V_2= Volume of Fe^{+2} Solution 20.0ml

Normality of $K_2Cr_2O_7$ solution $N_1 = \frac{V_2N_2}{V_1}$

Normality of potassium dichromate solution = ...N

Determination of Fe (II): (Part-b)

Make the problem solution up to the mark with distilled water and homogenize the solution. Pipette out 20 ml of standard $FeSO_4$ solution into a 250 ml conical flask, add 20 ml of 5 N H_2SO_4 solution and 20.00 ml distilled water. To this add 3 ml syrupy phosphoric acid and four to six drops of diphenylamine indicator and titrate with potassium dichromate taken in a burette until a blue-violet color appears. Repeat the process until concordant titre values are obtained.

S.No	Vol.of Fe^{+2}solution (ml)	Burette readings		Consumed Vol.of $K_2Cr_2O_7$ (ml)
		Initial (ml)	Final (ml)	
1	20.00			
2	20.00			
3	20.00			

Calculations: $V_3N_3 = V_4N_4$

Where, $K_2Cr_2O_7$ Solution Mohr's salt

N_3= Normality of $K_2Cr_2O_7$Solution ; N_4= Normality of Fe^{+2} Solution?

V_3= Vol of $K_2Cr_2O_7$ Solution (from table); V_4= Volume of Fe^{+2} Solution 20.0ml

Normality of unknown (Fe^{+2}) $N_4 = V_3N_3/V_4$ = ----------gms

The amount of 100 ml unknown Fe^{+2} present in your given solution

$$\frac{\text{Normality X Mol. Wt. Of } Fe^{2+}}{10} = \text{.....gms.}$$

(Molecular weight of Ammonium ferrous (II) Sulfate = 392.14 gL^{-1})

% Error formula= $\frac{\text{Given value-Reported values x 100}}{\text{Given value}}$

Final report	Given value (gms)	Reported value(gms)	% error
1.			

Signature of the faculty

EXP. NO:

DATE:

ESTIMATION OF POTASSIUM PERMANGANATE

(Redox titrations)

Aim: To estimate the amount of 100 ml unknown potassium permanganate solution is using standard oxalic acid solution

Apparatus: Burette, pipette, burette stand, conical flask, glazed tile etc.

Solutions required:

N Potassium permanganate ($KMnO_4$): Weigh accurately about **3.15** gms. of pure A.R grade pure anhydrous sample of potassium permanganate ($KMnO_4$) is transferred into 1L dilute to the mark of distilled water. Shake the flask to mix the solution thoroughly. Equivalent weight of potassium permanganate is 31.6

0.05N Oxalic acid: The equivalent weight of oxalic acid is 63. Dissolve **6.30** gms of oxalic acid in 1 lit solution.

Principle: Potassium permanganate is an oxidizing agent in the presence of dilute sulphuric acid. Potassium permanganate oxidizes oxalic acid to carbon dioxide and water.
The reaction takes place as follows:

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(O)$$
$$5H_2C_2O_4 + 5(O) \rightarrow 5H_2O + 10CO_2$$
$$2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$$

5 Moles of $H_2C_2O_4$ = 2 Moles of $KMnO_4$

Here the $KMnO_4$ acts as self indicator

Standardization of oxalic acid with using potassium permanganate solution: (Part-a)

Procedure:

20 ml of the oxalic acid solution is transferred into a well washed conical flask using a pipette. An equal volume of dilute sulfuric acid is added to the conical flask. The contents of the flask are heated to boiling.

The permanganate solution from the burette is run down conical flask first drop by drop then quickly while continuous shaking. At the end point the color of the solution in the flask turns to pale pink. The experiment is repeated till the concurrent readings are obtained. Then the readings

are entered in the tabular form.

S.No	Vol.of oxalic acid solution (ml)	Burette readings Initial (ml)	Final (ml)	Run down Vol. of $KMnO_4$ (ml)
1	20.00	00.00		
2	20.00			
3	20.00			

Calculations:

Normality of oxalic acid is $\frac{N_1V_1}{n_1} = \frac{N_2V_2}{n_2}$

Oxalic acid $KMnO_4$

N_1= Normality of oxalic acid=…….N ? N_2= Normality of $KMnO_4$ =…….N

V_1= Vol.of oxalic acid =20.00ml V_2= Vol.of $KMnO_4$ =…ml (burette reading)

n_1= No. moles of oxalic acid= 5 n_2= No. moles of $KMnO_4$ = 2

Normality of oxalic acid $N_1 = \frac{N_2V_2n_1 =}{V_1n_2}$

Normality of oxalic acid ..... N

Estimation of potassium permanganate solution (Part-b): (Procedure)

Make the problem solution up to the mark with distilled water and homogenize the solution. The burette is filled with the given unknown Potassium permanganate solution including the nozzle after sufficiently cleaning and rinsing it. The initial reading of the burette is noted and clamped it to the burette stand. 20 ml of the oxalic acid solution is transferred into a well washed conical flask using a pipette. An equal volume of dilute sulfuric acid is added to the conical flask. The contents of the flask are heated to boiling.

The permanganate solution from the burette is run down conical flask first drop by drop then quickly while continuous shaking. At the end point the color of the solution in the flask turns to pale pink. The experiment is repeated till the concurrent readings are obtained. Then the readings are entered in the tabular form.

S.No	Vol.of oxalic acid solution (ml)	Burette readings Initial (ml) (a)	Final (ml) (b)	Run down Vol. of $KMnO_4$ (ml)
1	20.00	00.00		
2	20.00			
3	20.00			

Calculations: Normality of $KMnO_4$ is = $\frac{N_3V_3}{n_3} = \frac{N_4V_4}{n_4}$

Oxalic acid

N_3= Normality of oxalic acid=…….N

V_3= Vol.of oxalic acid =20.00ml

n_3= No. moles of oxalic acid= 5

$KMnO_4$

N_4= Normality of $KMnO_4$ =…….N ?

V_4= Vol.of $KMnO_4$ =…ml

n_4= No. moles of $KMnO_4$ = 2

Normality of $KMnO_4$ (N_4) = $\frac{N_3V_3n_4}{V_4n_3}$ =

$KMnO_4$ normality = ...N.

The amount of potassium permanganate present in 100 ml of the solution=

$$\frac{\text{Normality x Molecular weight of } KMnO_4}{10}$$

Molecular weight of $KMnO_4$=158.033 g l^{-1}

Report:

The amount of $KMnO_4$ in 100 ml of the solution =……gms

S.No.	Given value (gms)	Reported value (gms)	% error
1.	.		

Signature of the faculty

EXP. NO:
DATE:

ESTIMATION OF CONCENTRATION FOR VITAMIN- C

(Redox-Titration)

Aim : Determination of Vitamin C Concentration by using iodine solution.

Apparatus:

Burette, Pipette, Burette stand,
Conical flask, volumetric flask.

Principle :

This method determines the vitamin C concentration in a solution by a redox titration using iodine. Vitamin C, more properly called ascorbic acid, is an essential antioxidant needed by the human body (see additional notes). As the iodine is added during the titration, the ascorbic acid is oxidised to dehydroascorbic acid, while the iodine is reduced to iodide ions.

$$I_2 + I^- \rightarrow I_3^-$$

Tri iodide oxidizes vitamin C to form dehydroascorbicacid:

Ascorbic acid + I_2 → 2 I^- + de hydro ascorbic acid

$$C_6H_8O_6 + I_3^- + H_2O \rightarrow C_6H_6O_6 + 3I^- + 2H^+$$

$$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$$

Due to this reaction, the iodine formed is immediately reduced to iodide as long as there is any ascorbic acid present. Once all the ascorbic acid has been oxidised, the excess iodine is free to react with the starch indicator, forming the blue-black starch-iodine complex. This is the endpoint of the titration. The method is suitable for use with vitamin C tablets, fresh or packaged fruit juices and solid fruits and vegetables.

Experimental :Preparation of reagents

0.005 M Vitamin C: Dissolve 0.4403 gms of (Mol.Wt.176.12) of Vitamin C dissolved in 500ml. of distilled water.

0.01M Sodium thiosulfate: Dissolve **1.581** gms of (Mol.Wt.158.10) of sodium thiosulfate dissolved in 1 lit. of distilled water.

0.005 M Iodine solution: Weigh 0.83 g of (Mol.Wt.166.00) potassium iodide into a 1000 ml beaker. Make the solution up to the 1 L mark with distilled water.

Starch indicator solution:(0.5%): Weigh 0.25 g of soluble starch and add it to 50 ml of near boiling water in a 100 ml conical flask. Stir to dissolve and cool before using.

Chemicals :

0.050 M Iodine solution, Hypo solution, sample solution (vitamin C), starch indicator.

Standardization of Sodium thiosulfate with iodine: (PART-A)

Procedure to follow: Pipette a 20 ml aliquot of the sample solution of sodium thiosulfate (hypo) into a 250 ml conical flask and add about 20 ml of distilled water and 5 ml of starch indicator solution. Titrate the sample with 0.005 mol l^{-1} iodine solution. The end point of the titrate swirling the flask, until a blue color persists for 20 seconds complex. Repeat the titration with further aliquots of sample solution until you obtain concordant results

S.No.	Volume of Hypo (ml)	Burette readings Initial Final (ml)	Rundown volume of iodine solution (ml)
1	20.00		
2	20.00		
3	20.00		

Molarity of Iodine is $\frac{M_1V_1}{n_1} = \frac{M_2V_2}{n_2}$

Iodine Sodium thiosulfate

M_1= Molarity of Iodine =…….N M_2= Molarity of Hypo =…….N
V_1= Vol.of Iodine =(burette reading) V_2= Vol.of Hypo =20.00…ml
n_1= No. moles of oxalic acid= 1 n_2= No. moles of Sodium thiosulfate=2

Molarity of Iodine $M_1 = \frac{M_2V_2n_1}{V_1n_2}$

Iodine molarity (Known) = ... M.

Determination of Vitamin –C : (PART-B)

Procedure : .Pipette a 20 ml aliquot of the sample solution into a 250 ml conical flask and add about 150 ml of distilled water and 1 ml of starch indicator solution. Titrate the sample with mol l^{-1} iodine solution. The end point of the titration is identified as the first permanent trace of a dark blue-black color due to the starch-iodine complex. Repeat the titration with further aliquots of sample solution until you obtain concordant results

S.No.	Volume of ascorbic acid (ml)	Burette readings Initial	Final (ml)	Rundown volume of iodine solution (ml)
1	20.00			
2	20.00			
3	20.00			

Calculations :

Molarity of ascorbic acid calculated using the equation. $\frac{M_3V_3}{n_3} = \frac{M_4V_4}{n_4}$

Ascorbic acid Iodine solution

M_3 = Molarity of ascorbic acid…… M_4 = Molarity of Iodine solution=

V_3 = Vol of ascorbic acid solution = 20 ml V_4 = Vol.of Iodine ..... ml(burette reading)

n_4 = No. of moles of Iodine=1

n_3 = No. of moles of ascorbic acid=1

Molarity of ascorbic acid(M_3) = $\frac{M_4V_4n_3}{V_3n_4}$

Amount of vitamin C present in the given solution= Molarity X Mol. Wt. Of Vitamin C

10

Mol. Wt. Of Vitamin C=176.12 g/mol.

Precautions:.

1. Iodine stains both skin and clothing so proper care is advised
2. Vitamin C, or ascorbic acid, is a water soluble antioxidant that plays a vital role in protecting the body from infection and disease.

3. The concentration of the prepared iodine solution can be more accurately determined by titration with a standard solution of ascorbic acid or a standard solution of potassium thiosulfate using a starch indicator. This should be done if possible as iodine solutions can be unstable.

4. The average titre volume should ideally be in the range of 10 – 30 ml. If the titre required for a 20 ml aliquot of sample solution is well outside this range then a larger or smaller aliquot volume should be chosen. If the volume of the titre is too low, dilute the standard. If the titre volume is too high, dilute the sample.

5. Ascorbic acid is susceptible to oxidation by atmospheric oxygen over time. For this reason, the samples should be prepared immediately before the titrations.

6. Identification of the endpoint in this titration is significantly affected by the coloration of the sample solution used. If the solutions are colorless or are pale in color, there is no problem identifying

Report : The amount of the ascorbic acid present in the sample solution
Is ..... gms.

S.No.	Given value (gms)	Reported value (gms)	% error
1.			

Signature of the faculty

EXP. NO:

DATE:

ESTIMATION OF COPPER (Iodometry)

Aim: To determine the strength of $CuSO_4$ solution with the help of hypo solution

Appratures:

Funnel, Burette,
Iodometric flask,
Conical Flask,
Pipette,
Simple balance with weights

Chemicals Required: $K_2Cr_2O_7$, Hypo ($Na_2S_2O_3$), KI, Conc.HCl, dil. Acetic acid, $NaHCO_3$, H_2SO_4, $CuSO_4$, Ammonia solution, Starch & distilled water

Experimental :

Preparation of solutions

0.01 N Copper sulfate ($CuSO_4$): Weigh accurately about 2.4968 gms. of pure A.R grade pure anhydrous sample of copper sulfate is transferred into 1L dilute to the mark of distilled water. Shake the flask to mix the solution thoroughly.

Weight = 0.01 X equivalent weight/ Vol. of solution in ml

0.01 N Sodium thiosulphate solution (Hypo) : Dissolve 2.48 g of sodium thiosulphate pentahydrate ($Na_2S_2O_3.5H_2O$, 248 g/mol) in 1 liter of freshly boiled distilled water. Improve its stability by adding 0.1g of Na_2CO_3 store over night, and filter through fine grade paper, collect and store in a clean, dark glass bottle.

0.005 M Iodine solution: Weigh 1.50 g of (Mol.Wt.166.00) potassium iodide and 1.45 g of iodine transferred into a 1000 ml volumetric flask. Make the solution up to the 1 L mark with distilled water.

Starch indicator solution:(0.5%): Weigh 0.25 g of soluble starch and add it to 50 ml of near boiling water in a 100 ml conical flask. Stir to dissolve and cool before using.

Principle:

Iodometric method is used for determination of strength of CuSo4 . Copper paste reacts with KI to form white cuprous iodide which is precipitated sultaneously releasing iodine. Free iodine is titrated against standard sodium sulphate using starch as a indicator.

$$2\ CuSO_4 + 4KI \rightarrow 2CuI_2 + 2K_2SO_4 + I_2$$
$$2CuI_2 \rightarrow Cu_2I_2 + I_2$$
$$I_2 + 2Na_2S_2O_3 + \rightarrow Na_2S_4O_2 + 2NaI$$

$$2CuSO_4 = I_2 = Na_2S_2O_3$$

Liberated iodine is passed through starch solution to get blue colour starch complex.

Procedure:

Standardization of Sodium thiosulfate with iodine: (PART-A)

Procedure to follow: Pipette a 20 ml aliquot of the sample solution of sodium thiosulfate (hypo) into a 250 ml conical flask and add about 20 ml of distilled water and 5 ml of starch indicator solution. Titrate the sample with 0.005 mol l^{-1} iodine solution. The end point of the titrate swirling the flask, until a blue color persists for 20 seconds complex. Repeat the titration with further aliquots of sample solution until you obtain concordant results

S.No.	Volume of Hypo (ml)	Burette readings Initial Final (ml)	Rundown volume of iodine solution (ml)
1	20.00		
2	20.00		
3	20.00		

Molarity of Iodine is $\frac{M_1V_1}{n_1} = \frac{M_2V_2}{n_2}$

Iodine Sodium thiosulfate

M_1= Molarity of Iodine =0.005 M_2= Molarity of Hypo =…….N

V_1= Vol.of Iodine = burette reading V_2= Vol.of Hypo =20.00…ml

n_1= No. moles of Iodine = 1 n_2= No. moles of Sodium thiosulfate=2

Molarity of Sodium thiosulfate $M_1 = \frac{M_1V_1n_2}{V_2n_1}$

Sodium thiosulfate molarity (Known) =.....M.

2. Estimation of copper: (Part-B)

(i) Take 20 ml $CuSO_4$ solution in a flask
(ii) Drop wise Na_2CO_3 solution is added till solution is having blue turbidity
(iii) Add dilute acetic acid drop wise to neutralize any mineral acid present in it
(iv) 10 ml of 10 % KI solution is added and solution is mixed well
(v) With the help of a clean burette, sodium thiosulphate solution is added titrant $CuSO_4$ solution till the brown colour fades away
(vi) Add 1 ml of starch solution to get blue colour and continue to add hypo solution till the blue colour starts disappearing.
(vii) Repeat the pocedur for at least 2 mpre times

S.No	Vol.of copper(ml)	Burette readings Initial (ml)	Final (ml)	Run down Vol. of Hypo solution (ml)
1	20.00			
2	20.00			
3	20.00			

N_3 = Molarity of the Copper solution =?
V_3 = Volume of the Copper solution = 20ml
N_4 = Molarity of Hypo =0.05
V_4 = Volume of Hypo = $N_3 V_3 = N_4 V_4$

$$\text{Molarity of the Copper solution } N_3 = \frac{M_4 V_4}{V_3} \ldots\ldots\ldots\ldots\ldots\ldots N$$

Result: Amount of Copper present in the whole of the given solution (100 ml) =

Molar mass of copper sulphate= 249.685 g/mol (pentahydrate)

$$\frac{\text{Normality X Mol. Wt. Of copper}}{10} = \ldots\ldots\ldots \text{ gms.}$$

Report: The amount of the copper present in 100 ml of the solution =……gms

S.No.	Given value (gms)	Reported value (gms)	% error
1.			

Signature of the faculty

EXP. NO:

DATE:

ESTIMATION OF OXALIC ACID

(Redox titrations)

Aim: Standardization of oxalic acid using a standard sodium hydroxide solution or
To calculate the amount of oxalic acid present in 100 ml of the given solution.

Apparatus: Burette, pipette,
burette stand, conical flask,
glazed tile, wash bottle, etc.,

Solution required:

0.1 N Sodium hydroxide (NaOH): Weigh accurately about **4.00** gms. of pure A.R grade pure anhydrous sample of sodium hydroxide (NaOH) is transferred into 1L dilute to the mark of distilled water. Shake the flask to mix the solution thoroughly.

0.05 N Oxalic acid: The equivalent weight of oxalic acid is 63. Dissolve **3.15** gms of oxalic acid in 1 lit solution.

Principle: Oxalic acid reacts with Sodium hydroxide in the following equation

$$H_2C_2O_4 + 2NaOH \rightarrow Na_2C_2O_4 + 2H_2O$$

1 mole of $H_2C_2O_4$ = 2 mole of NaOH

Here the Phenolphthalein is the indicator

Procedure:

Standardization of sodium hydroxide solution: (Part-A)

20 ml of the oxalic acid solution is transferred into a well washed conical flask using a pipette. Burette is filled with sodium hydroxide solution up to the mark. The initial reading of the burette is noted and clamped it to the burette stand.. One drop of Phenolphthalein indicator is added to it. The color of the solution changes to red. Then the solution is titrated slowly with oxalic acid until a colorless solution is obtained. This is the end point. The experiment is repeated till the concurrent readings are obtained. Then the readings are entered in the tabular form.

S.No	Vol.of Oxalic acid solution (ml)	Burette readings Initial (ml)	Final (ml)	Run down Vol.of NaOH (ml)
1	20.00			
2	20.00			
3	20.00			

Normality of sodium hydroxide is $\frac{N_1V_1}{n_1} = \frac{N_2V_2}{n_2}$

Sodium hydroxide

Oxalic acid

N_1= Normality of Sodium hydroxide =…….N ? N_2= Normality of Oxalic acid
V_1 = Vol.of Sodium hydroxide =20.00ml V_2= Vol.of Oxalic acid =…ml
n_1= No. moles of sodium hydroxide =2 n_2= No. moles of Oxalic acid =1

Normality of sodium hydroxide $N_1 = \frac{N_2V_2n_1}{V_1n_2}$

Standardized sodium hydroxide normality =…..... NL^{-1}

Estimation of oxalic acid: (Part-B)

The burette is filled with the given oxalic acid solution including the nozzle after sufficiently cleaning and rinsing it. The initial reading of the burette is noted and clamped it to the burette stand.20 ml of the Sodium hydroxide solution is transferred into a well washed conical flask using a pipette. One drop of Phenolphthalein indicator is added to it. The color of the solution changes to red. Then the solution is titrated slowly with oxalic acid until a colorless solution is obtained. This is the end point.

The experiment is repeated till the concurrent readings are obtained. Then the readings are entered in the tabular form.

S.No	Vol.of oxalic acid solution (ml)	Burette readings Initial (ml)	Final (ml)	Run down Vol. of NaOH (ml)
1	20.00			
2	20.00			
3	20.00			

Calculation:

Normality of Oxalic acid is = $\frac{N_3V_3}{n_3} = \frac{N_4V_4}{n_4}$

N_3= Normality of Sodium hydroxide = (part –a) N_4= Normality of Oxalic acid =?

V_3 =Vol.of Sodium hydroxide =20.00ml

V_4= Vol.of Oxalic acid =…ml

n_3= No. moles of sodium hydroxide = 2 n_4= No. moles of Oxalic acid =1

Unknown Oxalic acid Normality = $N_4 = \frac{N_3 V_3 n_4}{V_4 n_3}$ = ... N

The amount of Oxalic acid present in 100 ml of the solution=

Molecular weight of oxalic acid = 126.07 g mol^{-1}

$$\frac{\text{Normality x Molecular weight of Oxalic acid}}{\text{Volumes in litres}}$$

Report:

The amount of the Oxalic acid in 100 ml of the solution = ...gms

S.No.	Given value (gms)	Reported value (gms)	% error
1.			

Signature of the faculty

EXP. NO:

DATE:

ESTIMATION OF SODOUM VANADATE

(Redox-Titration)

Aim: To estimate the amount of sodium vanadate solution using a standard solution of potassium dichromate solution.

Apparatus: 100ml standard flask, Burette, 250ml conical flask,20ml pipette, funnel & simple balance.

Chemicals required:

Sodium vanadate (Na_3VO_4)

Ferrous ammonium sulfate ($Fe_2(SO_4)_3(NH_4)_2\,SO_4\,.24\,H_2O$)

Potassium dichromate ($K_2Cr_2O_7$)

Diphenylamine indicator

Phosphoric acid (H_3PO_4)

5 N H_2SO_4 solution

Distilled water solution

Theory: Sodium vanadate and Potassium dichromate are used as oxidants in acid medium in the titration of ferrous ammonium sulfate solutions.

Sodium vanadate, Potassium permanganate, Potassium dichromate, Cerium(IV) sulfate, Iodine, Potassium iodate and Potassium bromated.

Sodium vanadate (Na_3VO_4):

Vanadium exists in a number of oxidation states in aqueous solution(+5, +4, +3, +2) vanadium salts are quit stable in neutral and slightly acid solutions and are colorless. Vanadium (V) gets reduced easily to vanadium(IV) which is blue in color.

$$V^{V} + e^{-} \rightarrow V^{IV}$$

The standard redox potential of V^{V}/V^{IV} is about 1.05 V very nearer to that of $Cr^{VI}/\ Cr^{III}$. Oxidations with sodium vanadate unlike those with dichromate and permanganate are free from induced reactions. Hence, sodium vanadate is preferred in the oxidimetric titration of iron(II) in the presence of oxalic acid. Not only oxalic acid but many organic reagents either do not get

oxidized with sodium vanadate or get oxidized very slowly. When the oxidants like Cr^{VI},Mn^{VII} and Ce^{IV} are used as oxidants the organic reductants also oxidized.

Preparation of solutions

0.01 N Sodium vanadate solution (Na_3VO_4): Sodium vanadate can be prepared by treating ammonium metavanadate with a slight excess of sodium carbonate and boiling the solution until all the ammonia is expelled.

$$2\ NH_4VO_3 + Na_2CO_3 \rightarrow 2NaVO_3 + H_2O + CO_2\uparrow + 2\ NH_3\uparrow$$

$$NaVO_3 + Na_2CO_3 \quad \rightarrow \quad Na_3VO_4 + CO_2\uparrow$$

Procedure: Take 1.5 gms of ammonium meta vanadate and 1.6 gms of sodium carbonate into 250 ml beaker and to it add 150 ml distilled water. Heat the mixture well until all the ammonia goes off. Cool the solution, filter and make upto 250 ml in volumetric flask. The sodium vanadate solution so prepared can be standardized with ferrous ammonium sulfate which in turn is to be standardized with a standard potassium dichromate solution**.**

0.01 N Ferrous ammonium sulfate solution: Weigh accurately about 1.00 gms. of pure A.R grade pure anhydrous sample of Ferrous ammonium sulfate (mohr's salt) into a 250 ml volumetric flask, add about 50 ml of 5 N sulphuric acid to dissolve the solid and finally make the solution upto the mark with distilled water.

0.01N Potassium dichromate ($K_2Cr_2O_7$): Weigh accurately about **0.49** gms. of pure A.R grade pure anhydrous sample of potassium dichromate ($K_2Cr_2O_7$) is transferred into 1L dilute to the mark of distilled water. Shake the flask to mix the solution thoroughly. Equivalent weight of potassium dichromate is 49

Weight = 0.01 X equivalent weight/ Vol. of solution in ml

$[Cr_2O_7^{2-} + 14\ H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H\ O_2$
Hence, equivalent weight=294/6=49]

1 %Diphenylamine (DPA) indicator: dissolve 1 gm. of the dye stuff in100 ml of concentrated H_2SO_4 in a 250 ml beaker and store it in a indicator bottle.

Standardization of potassium dichromate (Part-a)

Pipette out 20 ml of standard Na_3VO_4 solution into a 250 ml conical flask, add 20 ml of 5 N H_2SO_4 solution and 60 ml distilled water. To this add 3 ml syrupy phosphoric acid and four to six drops of diphenylamine indicator and titrate with potassium dichromate taken in a burette until a blue-violet color appears. Repeat the process until concordant titre values are obtained.

S.No	Vol of Vanadate solution (ml)	Burette readings		Consumed Vol. of $K_2Cr_2O_7$ (ml)
		Initial (ml)	Final (ml)	
1	20.00	00.00		
2	20.00			
3	20.00			

Formula:

$$V_1N_1 = V_2N_2$$

Where,

$K_2Cr_2O_7$ Solution Na_3VO_4 Solution

N_1= Normality of $K_2Cr_2O_7$Solution? ; N_2= Normality of Sodium vanadate Solution

V_1= Vol of $K_2Cr_2O_7$ Solution (from table); V_2= Volume of Na_3VO_4Solution 20.0ml

$$\text{Normality of } K_2Cr_2O_7 \text{ solution } N_1 = \frac{V_2 N_2}{V_1}$$

Normality of $K_2Cr_2O_7$ solution =..... N

Determination of sodium vanadate (Na_3VO_4): (Part-b)

Make the problem solution upto the mark with distilled water and homogenize the solution. Pipette out 20 ml of Na_3VO_4 solution into a 250 ml conical flask, and to it add 20 ml of 5 N H_2SO_4 solution and 60 ml distilled water. To this add 3 ml syrupy phosphoric acid and four to six drops of diphenylamine indicator and titrate with sodium vanadate taken in a burette until a blue-violet color appears. Repeat the process until concordant titre values are obtained.

S.No	Vol.of Na_3VO_4 solution (ml)	Burette readings Initial (ml)	Final (ml)	Consumed Vol. of $K_2Cr_2O_7$ (ml)
1	20.00			
2	20.00			
3	20.00			

Calculations: $V_3N_3 = V_4N_4$

Where,

$K_2Cr_2O_7$ Na_3VO_4

N_3= Normality of $K_2Cr_2O_7$Solution ; N_4= Normality of Na_3VO_4 Solution(Part-a)?

V_3= Vol of $K_2Cr_2O_7$ (from table); V_4= Volume of Na_3VO_4 Solution 20.0ml

Normalityof unknown (Na_3VO_4) solution (N_4) = V_3N_3/V_4 = ---------- gms

The amount of 100 ml unknown Sodium vanadate present in your given solution

$$\frac{\text{Molarity X Mol. Wt. Of sodium vanadate}}{10} = \text{..... gms.}$$

(Molecular weight of Sodium vanadate = (183.908 g/mol)

$$\% \text{ Error formula} = \frac{\text{Given value-Reported values x 100}}{\text{Given value}}$$

Final report:

S.No.	Given value (gms)	Reported value(gms)	% error
1.			

Signature of the faculty

EXP. NO:

DATE:

INSTRUMENTATION EXPERIMENTES

Determination of the concentration of acetic acid using sodium hydroxide (pH-metry method).

Theory: pH is a measure of the acidity or basicity of a solution. It is the negative logarithm of H^+ ion concentration. Definition of pH is not restricted to H^+ ion concentration but it also represents the tendency of hydrogen ions to interact with other components of the solution. Pure water with pH close to 7.0 is said to be neutral, with less than 7 is said to be Acidic and with greater than 7 is said to be Basic or Alkaline.

pH of the water is also affected by the decomposition of the aquatic material, which releases CO_2. This CO_2 combines with water to form Carbonic acid which lowers the pH of water. Changes in the pH value of water are important to many organisms. Most organisms have adopted to live in water of a specific pH and may die if it changes even slightly. Hence we need to be aware of pH of water.

Apparatus: pH meter with glass and reference electrode with temperature compensation.

A calomel or Ag / AgCl / KCl reference electrode is generally located around the glass electrode stem.

1. Beaker
2. Color comparator with discs.
3. Cuvettes (sample inserted test tubes)

Reagents: All the solutions used for pH measurement were prepared with CO_2 free water and all the buffer solutions were prepared a fresh after every four weeks.

Buffer solutions: Potassium Hydrogen Phthalate (0.05M):

Prepared by dissolving 1.0211 gm of the salt in 100ml of distilled water which shows pH of 4.00 at 25°C.

Borax buffer solution (0.05M): Prepared by dissolving 1.9608 gm of salt quantitatively in distilled water and diluted to 100ml which gives a solution of pH 9.8 at 25°C

Procedure:

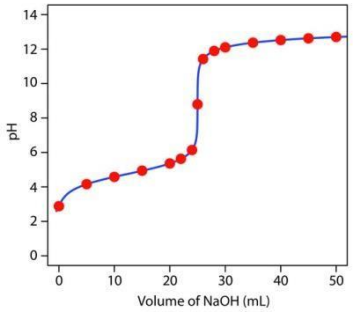
Again take 25 cm^3acetic acid in a 100 cm^3 beaker add 20-25 cm^3 of distilled water and dip the pH electrode, find the initial pH using pH meter (you pH meter should be pre-calibrated with standard buffers). Add standardized NaOH from the burette in small instalments of 1-2 cm^3 and stir well. Record the pH after each addition. Near the end point, NaOH should be added in very small instalments of 0.1-0.5 cm^3 . At the end point, you will observe sudden change in the pH and continue the titration until pH readings remain relatively constant at a pH of 10-12. Record your observation in the observation Table 3. Your pH reading should provide a smooth titration curve

Plot a graph of pH (y-axis) vs. volume NaOH added (x-axis). Determine the equivalence point from the graph. Find out the NaOH volume used in complete neutralization of acetic acid. From plotted curve find out the cm^3 of NaOH and pH at ½ at the neutralization of acetic acid. Calculate the value of Ka using Eq.pKa= pH

Observations:

S.No.	Volume of NaOH added	pH

Let's assume our analyte is acetic acid CH3_{3}3start subscript, 3, end subscriptCOOH (weak acid) and the titrant is sodium hydroxide NaOH (strong base). If we start plotting the pH of the analyte against the volume of NaOH that we are adding from the burette, we will get a titration curve as shown below



Point 1: No NaOH added yet, so the pH of the analyte is low (it predominantly contains H3_{3}3 start subscript, 3, end subscriptO+^\\text{+}+start superscript, start text, plus, end text, end superscript from dissociation of CH3_{3}3start subscript, 3, end subscriptCOOH). But acetic acid is a weak acid, so the starting pH is higher than what we noticed in case 1 where we had a strong acid (HCl).

Calculations:

The titre value corresponding to the point of inflection in the end point graph is ----------ml

$$\text{Strength of } CH_3OH = \frac{\text{End point titre value x Normality of NaOH}}{\text{Volume of } CH_3OH \text{ taken in beaker}} = \text{---------- N}$$

Signature of the faculty

EXP. NO:

DATE:

CONDUCTOMETRIC TITRATIONS BETWEEN STORNG ACID AND STORNG BASE

Aim : To determine the end point of the titration of strong acid and strong base by conduct metrically.

Apparatus:

Conductivity cell, Conductometer, Micro Burette, Beakers

Principle : The determination of equivalence point of a titration conductometrically is based upon the measurement of the conductance during the course of titration which varies in different manner before and after the equivalence point. Consider a titration of strong acid like hydrochloric acid Vs sodium hydroxide the equation will be

$$HCl + Na^+OH^- \rightarrow NaCl + H_2O$$

When HCl is taken in beaker as titrate, the initial conductivity is high, because strong acid completely dissociates into H^+ ions and the ionic conductivity of H^+ is 350. When NaOH is added as titrant, the OH^- and H^+ reacts to produce water and the no. of H^+ decreases and the conductivity gradually decreases after every addition. After the end point, when all the H^+ has reacted, the addition of NaOH causes increase in the no. of OH^- and hence the conductivity starts to increase (Ionic conductivity of OH^- 199) A plot conductivity vs volume of NaOH added will consist of two straight line branches intersecting at the neutralization point like V shape as shown in below graph.

Experimental : (preparation of reagents)

0.1 N HCl solution: Dissolve 8.6 ml of Conc.HCl (mol.wt 36.5) is transferred into 1000 ml litre flask into dilute up to mark with distilled water.

0.1 N NaOH: Dissolve 4.0 gm of NaOH (Mol.wt.40) in 1000 ml of distilled water

0.1 N KCl solution: Dissolve 7.456 gm of KCl in 1 liter of double distilled water of pure A.R grade pure anhydrous sample of sodium hydroxide pellets is transferred into 1L dilute to the mark of distilled water

0.1 N Sodium hydroxide (NaOH): Weigh accurately about **4.00** gms. of pure A.R grade pure anhydrous sample of sodium hydroxide (NaOH) is transferred into 1L dilute to the mark of distilled water. Shake the flask to mix the solution thoroughly.

0.05 N Oxalic acid: The equivalent weight of oxalic acid is 63. Dissolve **0.63** gms of oxalic acid in 1 lit solution.

Principle: Oxalic acid reacts with Sodium hydroxide in the following equation

$H_2C_2O_4 + 2NaOH \rightarrow Na_2C_2O_4 + 2H_2O$

1 mole of $H_2C_2O_4$ = 2 mole of NaOH

Here the Phenolphthalein is the indicator

Procedure:

(a) **Standardization of NaOH solution:** Pipette out 20 ml of prepared oxalic acid solution in a clean conical flask. Add 1 or 2 drops of Phenolphthalein indicator and titrate against standard sodium hydroxide solution. Note down the end point at which the color changes from pale yellow to pale pink. The colorless solution changes to red color.

S.No	Vol solution.of (oxalic acid (ml)	Burette readings Initial (ml)	Final (ml)	Run down Vol. of NaOH solution (ml)
1	20.00			
2	20.00			
3	20.00			

Oxalic acid

N_1= Normality of oxalic acid=…….M

V_1= Vol.of oxalic acid =(burette reading)

n_1= No. moles of oxalic acid=1

NaOH

N_2= Normality of NaOH =…….M ?

V_2= Vol.of NaOH =20…ml

n_2= No. moles of NaOH =2

$$\text{Normality of NaOH } (N_2) = \frac{N_1 V_1 n_2}{V_2 n_1}$$

Conductometric titration:

(i) Fill the burette with standard 1N NaOH solution.

(ii) Take 25 ml of the given HCl solution in a 100 ml beaker and the dip the conductivity cell in it and measure the conductance initially.

(iii) Now add NaOH from burette drop wise, i.e, 0.1 ml for each of the addition. After each of the addition, stir the solution gently by shaking and note down the change in conductance. The measured conductance are recorded and tabulated in the table.

(iv) Plot the graph between conductivity against volume of base added, the intersection of two straight lines gives the end point as shown in the above graph.

(v) Calculate the strength of the given strong acid (HCl) from the known strength of the given NaOH solution.

Calculations:

The titre value corresponding to the point of inflection in the end point graph is ----------ml

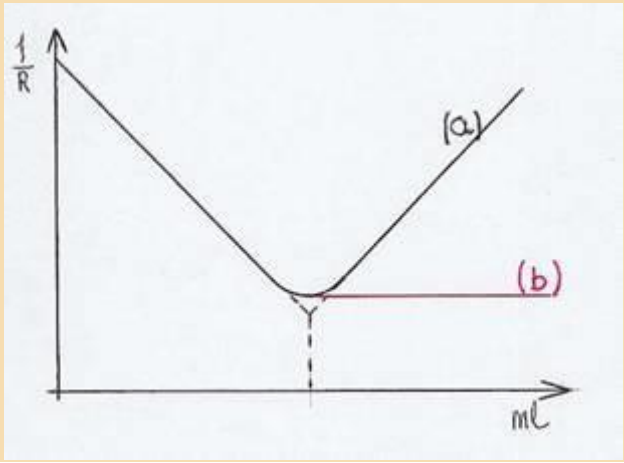
Strength of HCl = $\frac{\text{End point titre value x Normality of NaOH}}{\text{Volume of HCl taken in beaker}}$ = ---------- N

Observations:

S.No.	Volume of base added (ml)	Conductance (Siemens)

Graph

Figure shows the progress of titration of HCl with NaOH. The curve AB show the specific conductance of a mixture of acid and salt plotted against volume of NaOH added, while the curve B corresponds to a mixture of salt(Equivalence point) and excess NaOH after the end point (the point at which the two lines cross) has reached.



Precautions :

1. All precautions regarding the handling of the instrument should be observed.
1. To avoid the disturbing effects due to dilution, the concentration of the titrating solution should be5 to 10 times the concentration of the solution to be titrate.

Report:

The end point measured conductance with volume of alkali (ml) curve is----------ml
The strength of hydrochloric acid is---------- N

Signature of the faculty

EXP. NO:

DATE:

CONDUCTOMETRIC TITRATIONS BETWEEN STORNG ACID AND WEAK BASE

Aim :

To determine the strength of the hydrochloric acid against standard sodium hydroxide solution by conductometric titration.

Apparatus:

Conductivity cell, Conductometer, Micro Burette, Beakers

Principle : The determination of equivalence point of a titration conductometrically is based upon the measurement of the conductance during the course of titration which varies in different manner before and after the equivalence point. Consider a titration of strong acid like hydrochloric acid Vs ammonium hydroxide the equation will be

$$HCl + NH_4OH^- \rightarrow NH_4Cl + H_2O$$

When HCl is taken in beaker as titrate, the initial conductivity is high, because strong acid completely dissociates into H^+ ions and the ionic conductivity of H^+ is 350. When NH_4OH is added as titrant, the OH^- and H^+ reacts to produce water and the no. of H^+ decreases and the conductivity gradually decreases after every addition. After the end point, when all the H^+ has reacted, the addition of NH_4OH causes increase in the no. of OH^- and hence the conductivity starts to increase (Ionic conductivity of OH^- 199) A plot conductivity vs volume of NH_4OH added will consist of two straight line branches intersecting at the neutralization point like V shape as shown in below graph.

Experimental : (preparation of reagents)

0.1 M HCl solution: Dissolve 8.5 ml of Conc.HCl (mol.wt 36.5) is transferred into 1000 ml litre flask into dilute up to mark with distilled water.

0.1 M NH_4OH: Dissolve 72.2 ml of conc. Ammonia is dissolved in 1 lit. solution.

0.1 N ACOH: Dissolve 57.3 ml of conc. Acetic acid is dissolved in 1 lit. solution

0.1 N NaOH: Dissolve 4.00 gms NaOH dissolved in 1 lit. solution

Procedure:

Conductometric titration:

(vi) Fill the burette with standard 0.1M NH_4OH solution.

(vii) Take 25 ml of the given HCl solution in a 100 ml beaker and the dip the conductivity cell in it and measure the conductance initially.

(viii) Now add NH_4OH from burette drop wise, i.e, 0.1 ml for each of the addition. After each of the addition, stir the solution gently by shaking and note down the change in conductance.

(ix) The measured conductance are recorded and tabulated in the table.

(x) Plot the graph between conductivity against volume of base added, the intersection of two straight lines gives the end point as shown in the above graph.

(xi) Calculate the strength of the given strong acid (HCl) from the known strength of the given NH_4OH solution.

Graph:

Figure shows the progress of titration of HCl with NH_4OH. The conductance in the beginning starts falling(due to removal of H^+ ions) to from practically unionized water plus slow moving NH_4ions

$$H^+Cl^- + NH_4OH^- \rightarrow NH_4Cl + H_2O$$

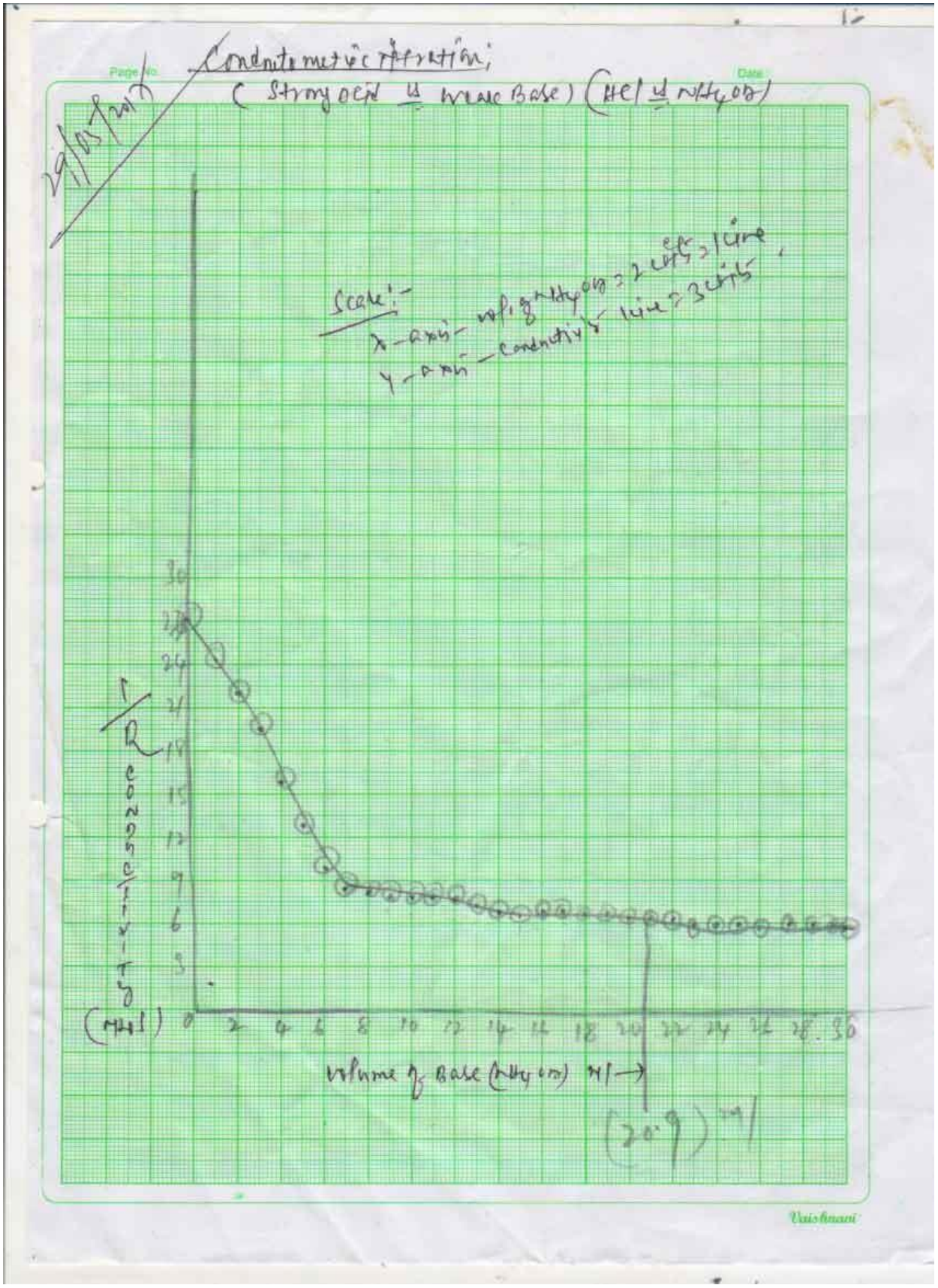
Strong acid Weak base Salt Practically unionized

However, when the entire acid is neutralized, further addition of poorly ionized ammonium hydroxide does not cause any appreciable change in the conductance. The Slope of the curve thus obtained is shown in Fig.

Calculations:

The titre value corresponding to the point of inflection in the end point graph is ----------ml

$$\text{Strength of HCl} = \frac{\text{End point titre value x Molarity of } NH_4OH}{\text{Volume of HCl taken in beaker}} = \text{---------- N}$$


Conductometric titration;
(Strong acid Vs weak Base) (HCl Vs NH_4OH)
Scale:-
x-axis - 1ml of NH_4OH = 2 cms; 1 cm = 1 ml
y-axis - conductivity 1 unit = 3 cms
1/R
CONDUCTIVITY
(mhos)
Volume of Base (NH_4OH) ml →
(20.9) ml
Vaishnavi

Observations:

S.No.	Volume of base added (ml)	Conductance (Siemens)

Precautions :

1. All precautions regarding the handling of the instrument should be observed.
2. To avoid the disturbing effects due to dilution, the concentration of the titrating solution should be5 to 10 times the concentration of the solution to be titrate.

Report:

The end point measured conductance with volume of alkali (ml) curve is----------ml
The strength of hydrochloric acid is---------- M

Signature of the faculty

EXP. NO:

DATE:

POTENTIOMETRIC TITRATIONS BETWEEN STRONG ACID AND STRONG BASE

Aim : To determine the normality of strong acid and strong base by using potentiometric method

Apparatus :

Potentiometer, pH Meter, Magnetic Stirrer, Burette, Beaker.

Chemicals :

HCl, NaOH, Distilled water.

Theory: When a solution of acid is titrated with the solution of an alkaline the change in the pH will be reflected in the change of E. When a small amount of standard alkaline is added to the acid, a little change in the EMF is produced in the beginning. The change in the electrode potential depends upon the fraction of the hydrogen ions removed. The change in the electrode potential depends upon the fraction of hydrogen ions removed. As an equivalence point reaches the fraction of the hydrogen ions removed by constant volume of standard alkali increases rapidly. There by causing a rapid change in the EMF. Above the equivalence point there is again small change in the EMF by the addition of excess of alkaline. Thus if the EMF of the cell is plotted against the volume of the standard alkali added a curve is obtained.

The point of intersection in the curve gives the equivalence point. It may be noted that the changes in the EMF is much more rapid near the equivalence point that any other region of the titration before and after the equivalence point. The maximum of the curve so obtained corresponds to the equivalence point of the titration.

H_2(Pt)/ acid solution//KCl (Aq)/ calomel electrode

The EMF of the cell is given by

$$E = E_{cal} - E_H = E^1 + 0.0591\ pH$$

Experimental :

preparation of reagents

0.1N NaOH solution: Dissolve 4.0 gm of NaOH (Mol.wt.40.0) to 100 ml with distilled water.

0.1N HCl solution: dilute 0.86 ml of 11.6 N and 36% concentrated HCl (Mol.wt.36.5) to 100 ml with distilled water.

Potassium Hydrogen Phthalate (0.05M): Prepared by dissolving 1.0211 gm of the salt in 100ml of distilled water which shows pH of 4.00 at 25°C.

|**Borax buffer solution (0.05M):** Prepared by dissolving 1.9608 gm of salt quantitatively in distilled water and diluted to 100ml which gives a solution of pH 9.8 at 25°C

Principle : The principle involved in potentiometric titration is the measurement of the emf between two electrodes, an indicator electrode and a reference electrode. In these tritations, measurent of emf are made while the tritration is in progress. The equivqlence point of the reaction is revealed by a sudden change in potentional in the plot of emf readings against the volume of titrant.

Procedure :

Calibrate the instrument before starting the experiment and wash the equipment i.e. Burette, Pipette, Conical flask using water. The micro-burette filled with the given 0.1N NaOH solution. Take 250 ml beaker add10 ml of 0.1N HCl and add around 100 ml of water of dip the electrodes properly. Keep the beaker on magnetic stirrer. Note potential initial reading and adding base slowly with continuous stirring known volumes of 0.1N NaOH solution, and note the potentials. Enter the values in a tabular form. From the data plot the graph and calculate the end point. Plot pH values or EMF values against the volume of sodium hydroxide added. Draw a smooth curve. The point of intersection gives the equivalence point. Plot another graph between $\Delta E/\Delta V$ values against the titre readings as abscissa. The maximum of the curve represents the equivalence point.

Observations:

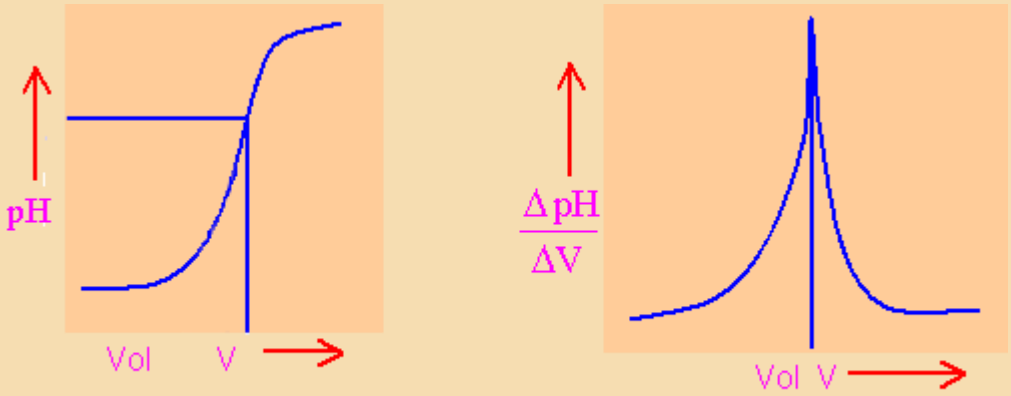
S.NO	Volume of NaOH added (ml)	Conductance (mhos)

Normality of HCl = Vol.of NaOH (equivalent point) x Normality of NaOH

Vol. of HCl taken in the beaker

Graph :

When a base is added (volume, V) to acid, pH increases and reaches to 7.0 at the inflection point. Change in pH is very fast near the inflexion point. Therefore, value of $\Delta pH / \Delta V$ is the highest at inflexion point. Hence, the end point of an acid base titration could be easily measured by a pH Meter. A representative curve for a pH-metric titration of a strong monobasic acid with strong base is shown below:



Precautions :

(i) Before starting experiment should calibrate the instrument

(ii) After completing the experiment electrode should washed and dip with distilled water.

(iii) Repeat the readings 2 – 3 times for accuracy.

Report :

The end point (Normality) of HCl by titrating with NaOH using potentiometer is _____

Signature of the faculty

EXP. NO:

DATE:

Determination the Adsorption Parameters of Acetic acid on activated carbon

Theory: Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (the adsorbate). It is different from absorption, in which a substance diffuses into a liquid or solid to form a solution.

Types of adsorption:

1) Physical adsorption is a type of adsorption in which the adsorbate associated with surface only through Van der Waals (weak intermolecular) interactions

2) Chemical absorption: is a type of adsorption whereby a molecule associated with surface through the formation of a chemical bond

Procedure:

1) Prepare 0.1M of NaOH and 0.5M acetic acid.

2) Prepare different solution in concentration 0.0625, 0.125, and 0.25M from acetic acid.

3) Take 15mL from solution number (1) and add to it 0.5 g of activated carbon after 10min add 0.5g of activated carbon to solution number (2) with continuous shacking until to solution number (4).

4) Filter all the solution after 30min from adding activated carbon, neglect the first 3mL of filtered.

5) Titrate 10mL from filtered solution with NaOH in presence of ph.ph indicator.

Calculations:

1) Calculate the molarity of acetic acid after adsorptionby=
(M×10) acetic acid = (0.1×V)sodium hydroxide

2) Calculate the acetic acid weight before and after adsorption=

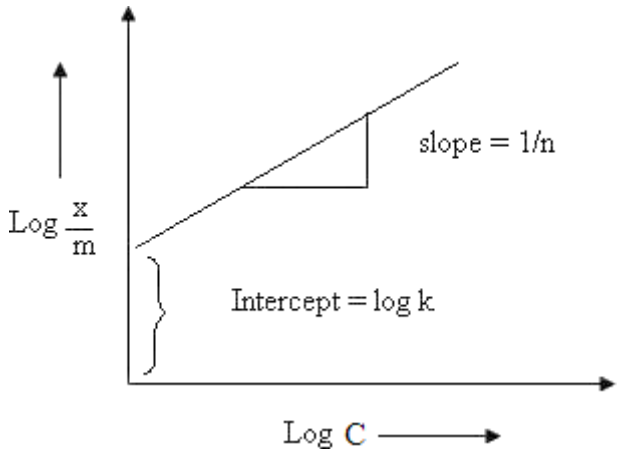
$$Wt.= \frac{MX\ Mol.wt.Xv\ (ml)}{1000}$$

w_{t0}= acetic acid weight before adsorption
w_t= acetic acid after adsorption

Observations:

No.	C	Log C	$M=W_{T0}-W_T$	x	m/x	Log m/x
1						
2						
3						
4						

4.Plot log m/x against log C: logm/x=log K+1/n log C



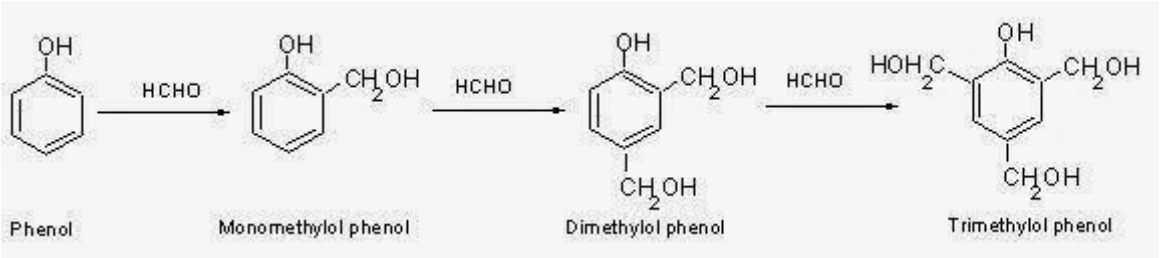
Determine **n** from the slope (***n* = number of adsorbed layer**)

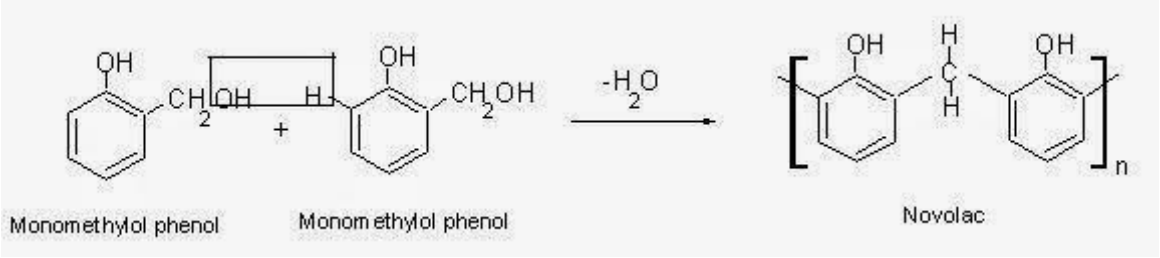
Signature of the faculty

Preparation of nylon-6, 6 and Bakelite

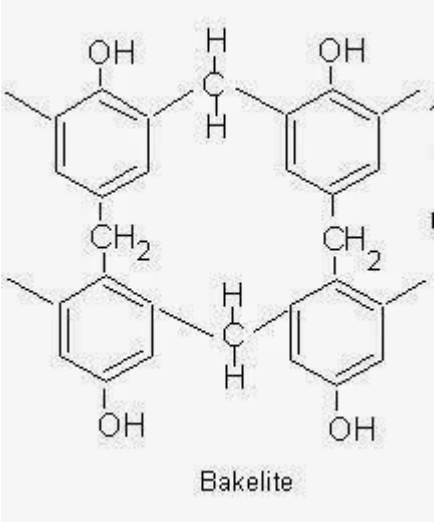
Preparation of bakelite:

Bakelite is the commercial name for the polymer obtained by the polymerization of phenol and formaldehyde.





When novalac resin is further heated in presence of HCHO producer i.e. hexa methelene diamine (curing agent) a cross linked polymer Bakelite can be obtained.



Properties of Bakelite

Bakelite is a non-conductive and heat resistant material makes it ideal for electrical insulators. It's rigid, hard,scratch resistant, infusible, water resistant, insoluble solid and good insulator.

UsesofBakelite

It was used for its non-conductive and heat-resistant properties in electrical insulators, and also in radios and telephones. It was also used in other diverse products such as jewellery, kitchenware, and toys. Bakelite was used for many things when it first came to light. In the 1920s, Bakelite production of jewellery came popular.

Nylon -6,6:

Synthesized by polycondensation of hexamethylenediamine and adipic acid. Equivalent amounts of hexamethylenediamine and adipic acid are combined with water in a reactor. This is crystallized to make nylon salt, an ammonium/carboxylate mixture. The nylon salt goes into a reaction vessel where polymerization process takes place either in batches or continuously.

$$n\ HOOC\text{-}(CH_2)_4\text{-}COOH + n\ H_2N\text{-}(CH_2)_6\text{-}NH_2 \rightarrow [\text{-}OC\text{-}(\ CH_2)_4\text{-}CO\text{-}NH\text{-}(CH_2)_6\text{-}NH\text{-}]_n + (2n\text{-}1)\ H_2O$$

Removing water drives the reaction toward polymerization through the formation of amide bonds from the acid and amine functions. Thus molten nylon 66 is formed. It can either be extruded and granulated at this point or directly spun into fibers by extrusion through a spinneret (a small metal plate with fine holes) and cooling to form filaments.

LABORATORY REAGENTS STRENGTH Concentrated acids

Name and formula of acid	Formula weight	Equivalent weight	% Weight	Specific Gravity	Aprox.strength
Acetic acid Glacial(CH_3COOH)	60.05	60.05	100	1.05	17.4 N Or 17.4 M
Hydrochloric acid (HCl)	36.46	36.46	36	1.18	11.6 M & N
Hydrobromic acid (HBr)	80.92	80.92	47.8	1.49	9 M &N
Hydrofluoric acid (HF)	20.01	20.01	46	1.5	26.5 N&M
Hydriodic acid (HI)	127.91	127.91	47.47	1.50	5.5 N&M
Nitric acid (HNO_3)	63.01	63.01	69.5 or 70	1.42	16 N & 5.5 M
Phosphoric acid (H_3PO_4)	98.00	---	85	1.69	40.5 N & 13.5 M
Perchloric acid ($HClO_4$)	100.46	---	70	1.66	9 N & M
Sulphuric acid (H_2SO_4)	98.08	49.0	96	1.84	36 N & 18.0 M

Bases

Base	Equivalent weight	Aprox.strength	Preparation of 1 lit solution
Conc.ammonium hydroxide (NH_4OH)	35.05	1.51 N Or 58.6% by weight of NH_4OH or 28.30% NH_3	Highest concentration with specific gravity 0.90
Dilute ammonium hydroxide (NH_4OH)	35.05	5 N (56.5 ml conc alkali per liter will give N solution and 166 ml /500 will give 5 N solution	Dilute 332 ml of conc.ammonia by making 1 litre.
Sodium hydroxide(NaOH)	40.00	5 N	Dilute 220 gms caustic soda pellets by making 1 litre.
Lime water($Ca(OH)_2$	37.05	0.04N	Shake 10 g of lime with 1 lit of water and allow it to stand for some time. Filter and keep it in a stoppered bottle

Strength of molecular compounds (Lab strength) = $\frac{10 \times \text{density \%} \times \text{specific gravity}}{\text{Molecular weight}}$

Standard solutions strength

Standard EDTA solution (M/100 or N/50): Weigh 3.723 g of AR disodium dihydrogen ethylene diammine tetra acetic acid having molecular weight 372.25(eq.wt.186.125) before weighing, it is dried in an air oven at 75^{o}C for 90 minutes and cooled. Dissolve it in distilled water in a measuring flask and make up the volume to 1 litre with distilled water.

Standard potassium dichromate solution (N/10): Dissolve 4.904 g of pure potassium dichromate in distilled water and dilute to 1000ml.

Sodium thiosulfate (N/10): Dissolve 24.82 g of A.R.$Na_2S_2O_3$. $5H_2O$(Mol. Wt.248.21) in 1 litre of boiled out distilled water.

Potassium permanganate(N/10): Dissolve 3.1607 g of AR $KMNO_4$having mol.wt.158.037 and eq.wt.31.607 in distilled water. Boil for 1 hour, cool filter and make up the volume to 1 litre with distilled water.

Potassium Iodate (N/10): Dissolve 3.567 g of A.R .KIO_3 (mol.wt.214and eq.wt.35.66) in 1 lit distilled water.

Potassium hydrogen phthalate solution(N/10): Dissolve 3.1607 g of A. R. Potassium hydrogen phthalate distilled in boiled water and make up the volume to 1 litre .

Copper sulfate(N/10): dissolve 3.567 g of a. R. $CuSO_4$. $5H_2O$ moleculer weight 249.6 in distilled water in 1 litre

Potassium iodide (0.1M): Dissolve 16.60 g of A.R. KI in distilled water and make up the volume to 1 litres

Oxalic acid (0.1M): Dissolve 6.303 g of A.R. oxalic acid (Mol.wt.126.068) in distilled water and make up the volume to 1 litre

Ferrous ammonium sulfate (N/4): Dissolve 49.0 g of A.R. $FeSO_4$ $(NH_4)_2$ SO_4. $6H_2O$ in boiled out distilled water containing 10 ml of conc. H_2SO_4 and dilute to 500 ml.

Potassium ferrocyanide: Dissolve 53 g of substance in one litre of distilled water (0.5N)

Sodium carbonate Na_2CO_3: Dissolve 5.30 g of substance in one litre of distilled water

Calcium chloride: Dissolve 27.5 g of CaCl substance in one litre of distilled water

Bromine solution (saturated): Shake 11 ml of liquid bromine in 1 litre of distilled water.

Iodine solution: Dissolve 20 g of KI and 13 g of iodine in 30 ml of distilled water and make up the solution to 1 litre.

Alcoholic potassium hydroxide (N/100): Dissolve 5.6 g of A.R. KOH (mol.wt> 56.0 eq. wt.56) pellets in 1 litre of 95 % alcohol. Mix thoroughly and let stand undisturbed, for any carbonate to settle down. Decant the clear supernatant solution

Calcon indicator: dissolve 0.2 g of dyestuff in 50 ml of methanol.

Murexide indicator: Dissolve 10 mg of Murexide with 490 mg of NaCl in distilled water.

Phenolphthalein: Dissolve 1 gm Phenolphthalein in 100 ml of distilled water and add to this paste 100 ml of boiled water with constant stirring. Boil for about 5 minutes and then cool.

Starch: Prepare a paste of 1 gm of powdered starch with distilled water and add to this paste 100 ml of boiled water with constant stirring. Boil for about 5 minutes and then cool.