CHEMISTRY LABORATORY MANUAL

UNDER DBT - STAR COLLEGE SCHEME

सत्यमेव जयते

Department of Biotechnology
Ministry of Science & Technology
Government of India

Prepared by

MARIAN STAR CENTRE
DEPARTMENT OF CHEMISTRY
ST. MARY'S COLLEGE (AUTONOMOUS)
Re-accredited with 'A+' Grade by NAAC
Thoothukudi

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Direct inquiries: E.R. Arumugam
A-1, Thenmozhi Nagar, Third Street
Keelkattalai, Chennai - 600 117.
Ph : 044-2247 0770, 94440 40272
E-mail: cbapublisher@gmail.com

Preface

Chemistry is an experimental science. The experiments included in this manual have been selected to introduce the students to the basic techniques in chemistry and explore the basic concepts and principles of Introductory chemistry. The purpose of this manual is to make the young chemist to understand the lab skills which was an essential need for their career. This manual also includes experiments and procedures that illuminate the central principles of chemistry.

The Department of Chemistry, St. Mary's College (Autonomous) has great pleasure in publishing the laboratory manual for the undergraduate Chemistry students as per the syllabus 2018-2021. The principle, chemicals required, procedure, tables and calculations are given for each experiment.

The safety precautions to be followed are also mentioned. After successfully completing the lab exercises, students will be able to estimate quantitatively and qualitatively the chemical compounds and identify chemistry at work in everyday situations and do experiments in microscale level and take an active role in their learning through practical work. They will have a basic idea of preparation of simple organic compounds, prepare inorganic complexes and carry out physical experiments too. This manual will bridge the gap between the text book learning and the real science. We are publishing this manual with an aim to achieve experimental skills of our students.

Through the Star College Scheme sponsored by the Department of Biotechnology, New Delhi, the Department of chemistry is publishing this manual to help the students to carry out the practicals in a fruitful manner.

Acknowledgement

The Department of Chemistry express our gratitude to "THE ALMIGHTY". We kept our spirits up together to bring this book. We express our sincere and heartful thanks to Rev. Sr. Flora Mary, Secretary, Rev. Dr. Sr. A.S.J. Lucia Rose, Principal, for their continuous support and encouragement. We thank Dr. Sr. Arockia Jenecius Alphonse, Assistant Professor of Botany, Overall Coordinator and Member Secretary of DBT STAR College Scheme for her encouragement and continuous motivation to publish the Chemistry Laboratory Manual. We voice our sincere thanks to all the teachers who helped us to get a clear idea to bring this manual. We are very thankful to the Department of Biotechnology, Ministry of Science and Technology, Government of India, New Delhi for providing the financial aid for publishing this manual. We thank the Marian Star Centre for guiding us to publish this manual.

Department of Chemistry

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Thoothukudi

B.Sc. Chemistry

Contents

SYLLABUS

Major Practical I

Code : 18UCHCR1

Quantitative Analysis (Volumetric Methods)

A double titration involving the preparation of a primary standard, standardization of the link solution, making up of the given solution and its estimation. Concepts of acids, bases, oxidants, complex formation — Theory of Indicators. (Use of digital balance is permitted).

Titrimetric Quantitative Analysis:

	Substance to be estimated	**Primary Standard**
I. Acidimetry and alkalimetry	1. $NaOH / Na_2CO_3$	Na_2CO_3
	2. $HC1 / H_2SO_4$/ oxalic acid	Oxalic acid
II. Permanganometry	3. Oxalic acid	Oxalic acid
	4. Mohr'ssalt	Mohr's Salt
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III. Dichrometry - External indicator method		
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	10. Pb^{2+}	$Pb(NO_3)_2$
	11. Mn^{2+}	$MnSO_4.H_2O$
	12. Ni^{2+}	$ZnSO_4.7H_2O$

Estimation of Phenol /Aniline:

I. Course work (Not for external examination)

1. Estimation of acetic acid in vinegar samples.
2. Estimation of oxalate content in vegetables and fruits such as tomato, guava, grapes, etc.
3. Estimation of sodium carbonate and sodium Bicarbonate in a mixture.
4. Estimation of Total Hardness of water.

Core Practical II

Semimicro Inorganic Qualitative Analysis

Code : 18UCHCR2

Systematic qualitative analysis of a mixture containing two anions and two cations. One of the anions should be an interfering radical which should be eliminated. The two cations should be of different groups.

Principles of flame testing – concept of solubility and solubility product – concept of pH and Buffer action – common ion effect - theory of testing anions (Simple and interfering) – Principle of grouping of cations –Theory of testing cations.

The combination of mixture containing two halides,(sulphates along with lead, barium, strontium and calcium), (oxalate and carbonate) & (one oxidizing and one reducing group), should be avoided.

Anions:

(i)	Carbonate	(v)	Bromide	(ix)	Oxalate
(ii)	Sulphide	(vi)	Iodide	(x)	Fluoride
(iii)	Sulphate	(vii)	Nitrate	(xi)	Chromate
(iv)	Chloride	(viii)	Borate	(xii)	Phosphate

Cations:

(i)	Lead	(vi)	Nickel	(x)	Strontium
(ii)	Copper	(vii)	Manganese	(xi)	Calcium
(iii)	Bismuth	(viii)	Zinc	(xii)	Magnesium
(iv)	Cadmium	(ix)	Barium	(xiii)	Ammonium
(v)	Antimony				

Core Practical III

Physical Chemistry Experiments

Code : 18UCHCR3

1. Critical solution temperature of phenol water system and effect of impurities on CST.
2. Transition Temperature of a salt hydrate determination of molecular weight
3. Kinetics of Ester Hydrolysis
4. Conductometric Acid base Titration
5. Conductometric precipitation Titration
6. Potentiometric Redox Titration
7. Molecular weight determination by Rast Method
8. Phase Diagram – Simple eutectic
9. Phase Diagram – Compound formation
10. Heat of solution by solubility method ($K_2Cr_2O_7$/ oxalicacid)
11. Adsorption kinetics of oxalic acids/acetic acid on charcoal. Determination of concentration of the given acid.

Course Work

1. Verification of Beer's Law using spectrophotometer.

Core Practical IV

Organic Analysis and Organic Preparations

Code : 18UCHCR4

1. Organic Analysis:

Analysis of simple organic compounds

a) Nature of the compound- Aromatic / Aliphatic

b) Test for saturation/unsaturation.

c) Detection of element present/absent

d) Characterization of functional groups (Acids, amide, amines, phenol, aldehyde, ketone, anilide, ester, carbohydrates, nitro compounds), Confirmation by preparation of a solid derivative.

2. Preparation of Organic compounds involving the following chemical conversions

1. Oxidation
2. Hydrolysis
3. Nitration
4. Bromination
5. Diazotization
6. Benzoylation
7. Osazone formation

3. Determination of physical constant (melting point/boiling point)

4. Course work

i) Extraction of various phytochemicals using soxhelet apparatus and to analyse plant pigments using flame photometer

ii) Extraction of oil from plants using Clevenger apparatus.

Core Practical III

Gravimetry And Inorganic Preparation

Code : 18UCHCR5

a) Gravimetric Analysis

1. Estimation of Lead as Lead Chromate.
2. Estimation of Barium as Barium Chromate
3. Estimation of Zinc as Zinc Oxinate
4. Estimation of copper as copper (I) thiocyanate
5. Estimation of calcium as calcium oxalate

b) Inorganic Preparations

1. Preparation of Potash alum
2. Preparation of Hexammine nickel (II) chloride
3. Preparation of Tetrammine copper (II) sulphate
4. Preparation of Prussian blue.
5. Preparation of Potassium trioxalato chromate (III) trihydrate
6. Preparation of Potassium trisoxalato ferrate(III)
7. Preparation of Tristhiourea copper (I) sulphate

Course work

1. Estimation of Nickel as Nickel – DMG complex
2. Estimation of Iron/ Nickel by spectrophotometer.

Allied Biochemistry Practical

Gravimetry And Inorganic Preparation

Code : 18UBCAR1

Qualitative and Quantitative Analysis

Analysis of Biomolecule

I. Qualitative analysis of carbohydrates.
II. Qualitative analysis of aminoacids.
III. Colour reactions of Proteins.

Volumetric Analysis:

I. Estimation of Glycine by formal titration.
II. Estimation of Ascorbic acid.
III. Estimation of Protein by Biuret method.
IV. Determination of Saponification number of oil.
V. Determination of Iodine number of oil.
VI. Preparation of Buffer and Determination of its pH using pH meter.

Allied Chemistry Practicals

Code : 18UCHAR1/18UCHAR2

Organic Analysis

Analysis of simple organic compounds

a) Nature of the compound- Aromatic/Aliphatic
b) Test for Saturation/unsaturation.
c) Element present/absent
d) Characterization of functional groups (Acid, phenol (solid), aldehyde, ester,amide, primary amine, carbohydrates).

Volumetric Analysis

I. Acidimetry — Alkalimetry

1. Estimation of H_2SO_4 /HCl using standard oxalic acid .
2. Estimation of sodium hydroxide using standard sodium hydroxide.
3. Estimation of sodium carbonate using standard sodium carbonate.
4. Estimation of oxalic acid using standard oxalic acid

II. Permanganometry

5. Estimation of ferrous ion using standard ferrous ammonium sulphate.
6. Estimation of sodium oxalate/oxalic acid using standard oxalic acid.

III. Complexometry

7. Estimation of Zinc using standard Zinc sulphate.

Lab Etiquettes

- Student must use her own lab coat and lab manual in the lab.
- Mobile phones are strictly forbidden.
- Long hair and loose clothing must be confined.
- Students should come on time to lab with slippers.
- Students are advised to handle glasswares and instruments with proper care.
- While preparing solutions avoid communicating with others.
- Do your analysis with proper attention.
- Students should use own calculator.
- Wash your apparatus before and after properly.
- Before leaving the lab, wash your hands thoroughly.
- Keep all the reagent bottles in their respective places.
- Learn the procedure thoroughly before doing the experiment.
- Report any accidents or breakages immediately to the instructor.
- In case of a burn from acid or alkali, wash the affected area immediately with plenty of cool running water

QUANTITATIVE ANALYSIS

QUANTITATIVE ANALYSIS

Subject Code: 18UCHCR1

CONTENTS

Preparation of standard sodium carbonate

Weight of sodium carbonate + beaker = g

Weight of empty beaker = g

Weight of sodium carbonate in 200mL = g

Strength of sodium carbonate = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

= N

Titration I :

Standardisation of HCl **Std Na_2CO_3 Vs HCl**

S.No.	Volume of Na_2CO_3 mL	Burette Reading		Volume of HCl mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Methyl orange
3.	20.0	0			

Concordant value =

Calculation of strength of Hydrochloric acid

Volume of standard sodium carbonate (V_1) = mL

Strength of standard sodium carbonate (N_1) = N

Volume of hydrochloric acid (V_2) = mL

Strength of hydrochloric acid (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

= N

Ex.No:1

Date:

ESTIMATION OF SODIUM CARBONATE

Aim:

To estimate the amount of sodium carbonate present in the whole of the given solution being provided with analar sodium carbonate crystals and approximately decinormal solution of hydrochloric acid.

Principle:

Sodium carbonate reacts with hydrochloric acid as follows

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$$

Since one mole of sodium carbonate reacts with two equivalents of hydrochloric acid, equivalent weight of sodium carbonate is half of its molecular weight (106/2=53).

$$\underset{\text{(Std)}}{Na_2CO_3} \xleftrightarrow{\text{Titration I}} \underset{\text{(Link)}}{HCl} \xleftrightarrow{\text{Titration II}} \underset{\text{(Estimating Solution)}}{Na_2CO_3}$$

Procedure:

(a) Preparation of standard sodium carbonate solution:

About 1.06 g of analar sodium carbonate crystals are weighed out accurately, dissolved in distilled water and made up to 200 mL in a standard measuring flask. The solution is shaken well for uniform concentration.

(b) Standardisation of hydrochloric acid:

A clean burette is washed with water and rinsed with HCl and filled with the same. 20ml of the standard sodium carbonate solution is pipetted out into a clean conical flask and titrated against the hydrochloric acid using methyl orange indicator. The end point is the colour change from golden yellow to pale pink. The titration is repeated for concordant value. From the titre value the strength of HCl is calculated.

Titration II :

Estimation of Na_2CO_3 **HCl Vs Na_2CO_3**

S.No.	Volume of Na_2CO_3 mL	Burette Reading		Volume of HCl mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Methyl orange
3.	20.0	0			

Concordant value = _____

Calculation of strength of Hydrochloric acid

Volume of hydrochloric acid (V_1) = mL

Strength of hydrochloric acid (N_1) = N

Volume of given sodium carbonate (V_2) = mL

Strength of given sodium carbonate (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

=

= N

Weight of Na_2CO_3 present in 100ml of the given solution = $\frac{\text{Normality} \times \text{Equivalent weight}}{10}$

= g

(c) Estimation of sodium carbonate

The given sodium carbonate solution is made upto 100mL, in a standard measuring flask. 20ml of this solution is pipetted out into a clean conical flask and titrated against the hydrochloric acid using methyl orange indicator. The end point is the colour change from golden yellow to pale pink. The titration is repeated for concordant value. From the titre value, the weight of sodium carbonate present in the whole of the given solution is calculated.

Result:

Weight of sodium carbonate present

in the whole of the given solution = g

Preparation of standard oxalic acid solution

Weight of oxalic acid + beaker = g

Weight of empty beaker = g

Weight of oxalic acid in 100mL = g

Strength of oxalic acid = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

Strength of oxalic acid = N

Titration I :

Standardisation of NaOH **Std $H_2C_2O_4$ Vs NaOH**

S.No.	Volume of NaOH mL	Burette Reading		Volume of Oxalic acid mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Phenolphthalein
3.	20.0	0			

Concordant value = _____

Calculation of strength of NaOH

Volume of standard oxalic acid (V_1) = mL

Strength of standard oxalic acid (N_1) = N

Volume of sodium hydroxide (V_2) = mL

Strength of sodium hydroxide (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of sodium hydroxide = N

Ex.No: 2

Date:

ESTIMATION OF OXALIC ACID

Aim:

To estimate the amount of oxalic acid present in the whole of the given solution being provided with analar oxalic acid crystals and approximately decinormal sodium hydroxide solution.

Principle:

The estimation depends on the reaction between

Oxalic acid and sodium hydroxide

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

Oxalic acid requires two equivalents of sodium hydroxide and hence

Equivalent weight of oxalic acid = Molecular weight / 2 = 126/2 = 63

Here sodium hydroxide is used as the link solution and phenolphthalein is used as the indicator.

Oxalic acid	Titration I	NaOH	Titration II	Oxalic acid
(Std)		(Link)		(Estimating Solution)

Procedure:

(a) Preparation of standard oxalic acid solution:

About 0.63g of analar oxalic acid crystals are weighed out accurately, dissolved in distilled water and made up to 100mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of Oxalic acid **NaOH Vs Given oxalic acid**

S.No.	Volume of NaOH mL	Burette Reading		Volume of Oxalic acid mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Phenolphthalein
3.	20.0	0			

Concordant value = _____

Calculation of strength of Hydrochloric acid

Volume of sodium hydroxide (V_1) = mL

Strength of sodium hydroxide (N_1) = N

Volume of oxalic acid (V_2) = mL

Strength of oxalic acid (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

= N

Weight of the oxalic acid present in 100mL of the given solution = $\frac{\text{Normality} \times \text{Equivalent weight}}{10}$

= g

b) Standardisation of sodium hydroxide:

A clean burette is washed with water, rinsed with the standard oxalic acid and filled with the same. 20ml of sodium hydroxide solution is pipetted out into a conical flask and a drop of phenolphthalein indicator is added and then it is titrated against the oxalic acid taken in the burette. The end point is the just disappearance of pink colour. The titration is repeated for concordant value. From the titre value the strength of NaOH is calculated.

(c) Estimation of oxalic acid:

The given oxalic acid is made up to 100 mL in a standard measuring flask. A clean burette is rinsed with this made up solution and filled with the same. 20mL of sodium hydroxide solution is pipetted out into a clean conical flask and a drop of phenolphthalein is added. It is titrated against oxalic acid taken in the burette. The end point is the just disappearance of pink colour. The titration is repeated for concordant value. From the titre value, the weight of oxalic acid present in the whole of the given solution can be calculated.

Result:

The amount of oxalic acid present
in the whole of the given solution = g

Preparation of standard oxalic acid solution

Weight of oxalic acid+ beaker = g

Weight of empty beaker = g

Weight of oxalic acid in 100ml = g

Strength of oxalic acid solution = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

Strength of oxalic acid = N

Titration I :

Standardisation of $KMnO_4$ **Std oxalic acid Vs $KMnO_4$**

S.No.	Volume of Oxalic acid mL	Burette Reading		Volume of $KMnO_4$ mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Self Indicator
3.	20.0	0			

Concordant value = _____

Calculation of strength of $KMnO_4$

Volume of standard Oxalic acid (V_1) = mL

Strength of standard Oxalic acid (N_1) = N

Volume of $KMnO_4$ (V_2) = mL

Strength of KMnO4 solution, (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of $KMnO_4$ = N

Ex.No: 3

Date:

ESTIMATION OF OXALIC ACID (Permanganometry)

Aim:

To estimate the amount of oxalic acid present in the whole of the given solution. You are provided with approximately decinormal solution of potassium permanganate and analar oxalic acid crystals.

Principle:

Potassium permanganate in acid medium is an oxidising agent, two molecules of the substance giving 5 atoms of oxygen for oxidation. This oxygen oxidises reducing agents such as oxalic acid. The reaction between oxalic acid and potassium permanganate may be represented by the following equations.

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

$H_2C_2O_4 + (O) \rightarrow H_2O + 2CO_2$

Since $2KMnO_4$ gives 10 equivalents of oxygen,

equivalent weight of $KMnO_4$ is $\frac{316}{10} = 31.6$

Since a molecular mass of oxalic acid reacts with 2 equivalents of oxygen its equivalent weight is half its molecular weight. The equivalent weight of oxalic acid crystals is $\frac{126}{2} = 63$.

Potassium permanganate solution is pink colour and when reduced changes into potassium and manganous salts which are colourless in solution Therefore reducing agents decolorize the pink solution. So no other indicator is required to denote the end point in the titrations with $KMnO_4$

Oxalic acid	Titration I	$KMnO_4$	Titration II	Oxalic acid
(Std)		(Link)		(Estimating Solution)

Titration II :

Estimation of oxalic acid **$KMnO_4$ Vs. oxalic acid**

S.No.	Volume of Oxalic acid mL	Burette Reading		Volume of $KMnO_4$ mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Self Indicator
3.	20.0	0			

Concordant value = _____

Calculation of strength of Oxalic acid

Volume of $KMnO_4$ (V_1) = mL

Strength of $KMnO_4$ (N_1) = N

Volume of oxalic acid (V_2) = mL

Strength of oxalic acid solution, (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of oxalic acid (N_2) = N

Weight of oxalic acid present in 100mL of the given solution = $\frac{\text{Normality} \times \text{Equivalent weight}}{10}$

= g

Procedure:

a) Preparation of standard Oxalic acid Solution

About 0.63g of analar oxalic acid crystals are weighed out accurately, dissolved in distilled water and made up to 100 mL in a standard measuring flask. The solution is shaken well for uniform concentration.

b) Standardisation of Potassium Permanganate solution

A clean burette is rinsed with potassium permanganate solution and filled with the same. The initial reading is noted. A clean pipette is rinsed with the standard oxalic acid solution. 20mL of this solution is pipetted out into a clean conical flask and 20 mL of dilute sulphuric acid is added, the mixture is heated to 60°C (bearable warmth). The solution is then titrated with the potassium permanganate taken in the burette. The end point is the appearance of pale pink colour. The titration is repeated to get concordant value.

c) Estimation of oxalic acid

The given oxalic acid is made upto 100 mL in a standard measuring flask. 20 mL of the solution is pipetted out into clean conical flask and acidified with 20 mL dilute sulphuric acid: The mixture is heated to bearable warmth and then titrated with the potassium permanganate taken in the burette. The end point is the appearance of pale pink colour. The titration is repeated to get concordant value.

Result:

The amount of oxalic acid present in the whole of the given solution = g

Preparation of standard Mohr's salt solution

Weight of Mohr's salt + beaker = g

Weight of empty beaker = g

Weight of Mohr's salt in 100ml = g

Strength of Mohr's salt = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

Strength of Mohr's salt = N

Titration I :

Standardisation of $KMNnO_4$ **Std Mohr's salt Vs. $KMnO_4$**

S.No.	Volume of Mohr's salt mL	Burette Reading		Volume of $KMnO_4$ mL	Indicator
		Initial	Final		
1.	20.0	0			Self indicator
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of strength of $KMnO_4$

Volume of standard Mohr's salt (V_1) = 20 mL

Strength of standard Mohr's salt (N_1) = N

Volume of $KMnO_4$ (V_2) = mL

Strength of $KMnO_4$ solution (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of $KMnO_4$ = N

Ex.No: 4

Date:

ESTIMATION OF MOHR'S SALT

Aim:

To estimate the amount of Mohr's salt present in the whole of the given solution. Analar crystals of Mohr's salt and an approximately decinormal solution of potassium permanganate are provided.

Principle:

The estimation is based on the reaction between ferrous ion and potassium permanganate in acid medium. The permanganate oxidises ferrous sulphate to ferric sulphate itself being reduced to manganous sulphate.

$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5\,[O]$

$2FeSO_4 + H_2SO_4 + [O] \rightarrow Fe_2(SO_4)_3 + H_4O$

Molecular weight of Mohr's Salt = 392

Mohr's salt (Std) $\xleftrightarrow{\text{Titration I}}$ $KMnO_4$ (Link) $\xleftrightarrow{\text{Titration II}}$ Mohr's salt (Estimating Solution)

Procedure:

(a) Preparation of standard Mohr's salt

About 3.92g of analar Mohr Salt crystals are weighed out accurately and dissolved in distilled water. 3mL of dil. H_2SO_4 is added then made up to 100 mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of Mohr's salt **$KMnO_4$ Vs Mohr's salt**

S.No.	Volume of Mohr's salt mL	Burette Reading		Volume of $KMnO_4$ mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Self indicator
3.	20.0	0			

Concordant value = _____

Calculation of strength of Mohr's Salt

Volume of $KMnO_4$ solution (V_1) = mL

Strength of $KMnO_4$ solution (N_1) = N

Volume of Mohr's salt solution (V_2) = mL

Strength of Mohr's salt solution (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

= N

Weight of Mohr's salt present in 100mL of the given solution = $\frac{\text{Normality} \times \text{Equivalent weight}}{10}$

= g

(b) Standardisation of potassium permanganate

A clean burette is washed with water and rinsed with $KMnO_4$ and filled with the same. Exactly 20 mL of the standard Mohr Salt solution is pipetted out into a clean conical flask, 20 mL of dil. H_2SO_4 is added. Shaken the solution well and titrated against $KMnO_4$ taken in the burette. The end point is the appearance of permanent pale pink colour.The titration is repeated to get concordant value. From the titre value the strength of potassium permanganate is calculated.

(c) Estimation of Mohr's salt :

The given Mohr's salt solution is made up to 100 mL in a standard measuring flask. Exactly 20 mL of the made up solution is pipetted out into a clean conical flask and 20mL of dil H_2SO_4 is added. The contents of the flask is titrated against potassium permanganate, the end point is the appearance of permanent pale pink colour. The titration is repeated to get concordant value. From the titre value the strength and hence the amount of iron(II) present in the whole of the given solution is calculated.

Result :

The amount of Mohr's salt solution present in the whole of the given solution is
= _____ g.

Preparation of standard Mohr's salt

Weight of Mohr's salt + beaker = g

Weight of empty beaker = g

Weight of Mohr's salt in 100mL = g

Strength of Mohr's salt solution = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

Strength of Mohr's salt solution = N

Titration I :

Standardisation of $KMnO_4$ **Std Mohr's salt Vs. $KMnO_4$**

S.No.	Volume of Mohr's salt mL	Burette Reading		Volume of $KMnO_4$ mL	Indicator
		Initial	Final		
1.	20.0	0			Self indicator
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of strength of $KMnO_4$

Volume of Mohr's salt solution (V_1) = mL

Strength of Mohr's salt solution (N_1) = N

Volume of $KMnO_4$ solution (V_2) = mL

Strength of $KMnO_4$ solution (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of $KMnO_4$ = N

Ex.No: 5

Date:

ESTIMATION OF IRON (II)

Aim:

To estimate the amount of Iron (II) present in the whole of the given solution. Analar crystals of Mohr's salt and an approximately decinormal solution of potassium permanganate are provided.

Principle:

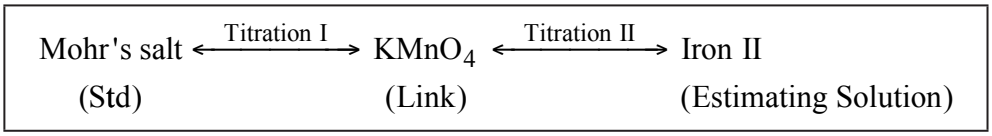
The estimation is based on the reaction between ferrous ion and potassium permanganate in acid medium. The permanganate oxidises ferrous sulphate to ferric sulphate itself being reduced to manganous sulphate.

$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$

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

Molecular weight of Mohr's Salt = 392

Atomic weight of Fe II = 55.85



Procedure:

(a) Preparation of standard Mohr's salt solution

About 3.92g of analar Mohr Salt crystals are weighed out accurately and dissolved in distilled water. 3mL of dil. H_2SO_4 is added then made up to 100 mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of Fe(II) ion **$KMnO_4$ Vs Fe (II)**

S.No.	Volume of	Burette Reading		Volume of	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Self indicator
3.	20.0	0			

Concordant value = _____

Calculation of strength of Fe(II)

Volume of $KMnO_4$ solution (V_1) = mL

Strength of $KMnO_4$ (N_1) = N

Volume of Fe (II) (V_2) = 20 mL

Strength of Fe (II) (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

= N

Weight of the Fe (II) present in 100mL of the given solution = $\frac{\times 55.85}{10}$

= g

(b) Standardisation of potassium permanganate salt

A clean burette is washed with water and rinsed with $KMnO_4$ and filled with the same. Exactly 20 mL of the standard Mohr's Salt solution is pipetted out into a clean conical flask, 20 mL of dil. H_2SO_4 is added. Shaken the solution well and titrated against $KMnO_4$ taken in the burette. The end point is the appearance of permanent pale pink colour. The titration is repeated to get concordant value. From the titre value the strength of potassium permanganate is calculated.

(c) Estimation of iron(II):

The given Mohr's salt solution is made up to 100 mL in a standard measuring flask. Exactly 20 mL of the made up solution is pipetted out into a clean conical flask and 20mL of dil H_2SO_4 is added. The contents of the flask is titrated against potassium permanganate, the end point is the appearance of permanent pale pink colour. The titration is repeated to get concordant value. From the titre value the strength and hence the amount of iron (II) present in the whole of the given solution is calculated.

Result :

The amount of Iron(II) present in the whole of the given solution is = g

Preparation of standard ferrous ammonium sulphate solution

Weight of Mohr's salt + beaker = g

Weight of beaker = g

Weight of Mohr's salt in 100 mL = g

Strength of Mohr's salt $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

Strength of Mohr's salt = N

Titration I :

Standardisation of $K_2Cr_2O_7$ **Mohr's salt Vs. $K_2Cr_2O_7$**

S.No.	Volume of Mohr's salt (mL)	Burette Reading (mL)		Volume of $K_2Cr_2O_7$ (mL)	Indicator
		Initial	Final		
1.	20.0	0			External (Potassium Ferri cyanide)
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of strength of $K_2Cr_2O_7$

Volume of Mohr's salt solution (V_1) = mL

Strength of Mohr's salt solution (N_1) = N

Volume of $K_2Cr_2O_7$ solution (V_2) = mL

Strength of $K_2Cr_2O_7$ solution (N_2) $= \frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of $K_2Cr_2O_7$ = N

Ex.No: 6

Date:

ESTIMATION OF IRON (II) (Dichrometry)

Aim:

To estimate the amount of iron (II) present in the whole of the given solution, pure Mohr's salt crystals and approximately decinormal solution of potassium dichromate are supplied.

Principle:

A standard solution of Mohr's salt is prepared and titrated against $K_2Cr_2O_7$ in the presence of dil.sulphuric acid. An external indicator viz. potassium ferricyanide is used to fix the end point.

This estimation is based on the reaction between $K_2Cr_2O_7$ in acid medium and ferrous sulphate. The potassium dichromate oxidizes Ferrous sulphate to Ferric sulphate, itself being reduced to green chromicsulphate.

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3(O)$$

$$6FeSO_4 + 3H_2SO_4 + 3(O) \rightarrow 3Fe_2(SO_4)_3 + 3H_2O$$

Mohr's salt $\xleftrightarrow{\text{Titration I}}$ $K_2Cr_2O_7$ $\xleftrightarrow{\text{Titration II}}$ Iron II

(Std) (Link) (Estimating Solution)

Procedure:

(i) Preparation of std. Mohr's salt solution:

About 3.92 g of analar Mohr Salt crystals are weighed out accurately and dissolved in distilled water. 3mL of dil. H_2SO_4 is added then made up to 100 mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of Iron (II) **$K_2Cr_2O_7$ Vs. Fe (II)**

S.No.	Volume of Mohr's salt (mL)	Burette Reading (mL)		Volume of $K_2Cr_2O_7$ (mL)	Indicator
		Initial	Final		
1.	20.0	0			External (Potassium Ferri cyanide)
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of strength of Fe(II)

Volume of $K_2Cr_2O_7$ (V_1) = mL

Strength of $K_2Cr_2O_7$ (N_1) = N

Volume of Fe (II) (V_2) = mL

Strength of Fe (II) (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

= N

Weight of Iron (II) present in the whole of the given solution = $\frac{\text{Strength of Fe(II)} \times 55.85}{10}$

= g

(ii) Standardisation of dichromate:

The burette is washed with water and filled with potassium dichromate after rinsing with the same. 20mL of the std. Mohr's salt solution is pipetted out into a conical flask, an equal volume of dil.sulphuric acid is added. It is then diluted to 150mL. A drop of the reaction mixture is withdrawn time to time from the burette. About 10mL of potassium dichromate solution is run down from the burette.

The solution is stirred well and a small drop is taken with a pointed glass rod and mixed with a very dilute solution of potassium ferricyanide indicator on the spot plate. The appearance of greenish blue colour indicates the presence of ferrous iron. The titration is repeated by adding 0.5mL of potassium dichromate solution at a time until no blue colour develops, which indicates the end point. The titrations are repeated to get concordant value. From the titre value, the strength of potassium dichromate can be calculated.

iii) Estimation of iron(II):

The given iron(II) solution is made upto 100mL in a standard measuring flask. 20mL of this solution is pipetted out into a conical flask, an equal volume of dil.sulphuric acid is added. It is then diluted to 150mL. It is then titrated against potassium dichromate using potassium ferricyanide indicator following the conditions as before. The titrations are repeated to get concordant value. From the titre value, the strength and hence the amount of iron(II) present in the whole of the given solution can be calculated.

Equivalent weight of Mohr's salt - 392

Equivalent weight of Iron (II) - 55.85

Result

Amount of iron (II) present in the whole of the given solution = _____ g

Preparation of standard potassium dichromate solution

Weight of potassium dichromate + beaker = g

Weight of empty beaker = g

Weight of potassium dichromate = g

Amount of potassium dichromate in 100ml of the given solution = g

Strength of potassium dichromate = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

Strength of potassium dichromate = N

Titration I :

Standardisation of sodium thiosulphate **Std $K_2Cr_2O_7$ Vs. $Na_2S_2O_3$**

S.No.	Volume of $K_2Cr_2O_7$ mL	Burette Reading		Volume of $Na_2S_2O_3$ mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			
3.	20.0	0			Starch

Concordant value = _____

Calculation of strength of sodium thiosulphate

Volume of potassium dichromate solution (V_1) = mL

Strength of potassium dichromate solution (N_1) = N

Volume of Sodium thiosulphate (V_2) = mL

Strength of Sodium thiosulphate (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of Sodium thiosulphate = N

Ex.No: 7

Date:

ESTIMATION OF POTASSIUM DICHROMATE

Aim:

To estimate the amount of potassium dichromate present in the whole of the given potassium dichromate solution being provided with approximately decinormal sodium thiosulphate solution and analar potassium dichromate.

Principle:

It is based on the reaction between potassium dichromate and potassium iodide.

$$6I^- + Cr_2O_7^{2-} + 14H^+ \rightarrow 3I_2 + 2Cr^{3+} + 7H_2O$$

The liberated iodine reacts with sodium thiosulphate solution.

$$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

The equivalent weight of link Thio 248.

The equivalent Weight of potassium dichromate is 49.

$$K_2Cr_2O_7 \xleftrightarrow{\text{Titration I}} \text{Thio} \xleftrightarrow{\text{Titration II}} K_2Cr_2O_7$$

(Std) (Link) (Estimating Solution)

Procedure:

(a) Preparation of standard potassium dichromate solution:

About 0.49 g of analar potassium dichromate crystals are weighed out accurately, dissolved in water and made up to 100ml in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of potassium dichromate **$K_2Cr_2O_7$ Vs. Std $Na_2S_2O_3$**

S.No.	Volume of $K_2Cr_2O_7$ mL	Burette Reading		Volume of $Na_2S_2O_3$ mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			
3.	20.0	0			Starch

Concordant value = _____

Calculation of the strength of $K_2Cr_2O_7$

Volume of $Na_2S_2O_3$ (V_1) = mL

Normality of $Na_2S_2O_3$ (N_1) = N

Volume of $K_2Cr_2O_7$ (V_2) = 20 mL

Strength of $K_2Cr_2O_7$ (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

= N

Amount of potassium dichromate present in 100 ml of the given solution = $\frac{\text{Normality} \times \text{Equivalent weight}}{10}$

= $\frac{\times 49}{10}$

= g

(b) Standardisation of sodium thiosulphate solution:

A clean burette is washed with water and rinsed with sodium thiosulphate and filled with the same.20ml of the standard potassium dichromate solution is pipetted out into a clean conical flask. About 5ml of pure concentrated hydrochloric acid is added. About 5ml of 10% potassium iodide solution is added and it is titrated against the sodium thiosulphate solution taken in the burette. The addition of sodium thiosulphate is continued until the solution is pale yellow in colour. About 2ml of starch solution is added and the titration is continued till the blue colour changes to green. The titration is repeated for concordant value. From the titre value, the strength of sodium thiosulphate is calculated.

(c) Estimation of potassium dichromate solution:

The given potassium dichromate solution is made upto 100mL in a standard measuring flask. 20ml of the given potassium dichromate solution is pipetted out into a clean conical flask. About 5ml of pure concentrated hydrochloric acid is added. About 5ml of 10% potassium iodide solution is added and it is titrated against the sodium thiosulphate solution taken in the burette. The addition of sodium thiosulphate is continued until the solution is pale yellow in colour. About 2ml of starch solution is added and the titration is continued till the blue colour changes to green. The titrationis repeated for concordant value. From the titre value, the strength of potassium dichromate and hence the amount of potassium dichromate present in the whole of the given solution is calculated.

Result:

The amount of potassium dichromate in 100ml of the given solution = g.

Preparation of standard copper sulphate solution

Weight of copper sulphate + beaker = g

Weight of empty beaker = g

Weight of copper sulphate = g

Amount of copper sulphate in 100ml of the given solution = g

Strength of copper sulphate solution = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

Strength of copper sulphate solution = N

Titration I :

Standardisation of sodium thiosulphte **Std $CuSO_4$ Vs. $Na_2S_2O_3$**

S.No.	Volume of $CuSO_4$ mL	Burette Reading		Volume of $Na_2S_2O_3$ mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			
3.	20.0	0			Starch

Concordant value = _____

Calculation of strength of sodium thiosulphate

Volume of copper sulphate solution (V_1) = 20.0 mL

Strength of copper sulphate solution (N_1) = N

Volume of Sodium thiosulphate (V_2) = mL

Strength of Sodium thiosulphate (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of Sodium thiosulphate = N

Ex.No: 8

Date:

ESTIMATION OF POTASSIUM PERMANGANATE

Aim:

To estimate the amount of potassium permanganate present in the whole of the given solution, being provided with approximately decinormal sodium thiosulphate solution and analar copper sulphate.

Principle:

It is based on the reaction between copper sulphate and potassium iodide.

$$CuSO_4 + 2KI \rightarrow CuI_4 + K_2SO_4$$

$$2CuI_2 \rightarrow Cu_2I_2 + I_2$$

The liberated iodine reacts with sodium thiosulphate solution.

$$2Na_2S_2O_3 + I_2 \rightarrow Na_2\ S_4O_6 + 2NaI$$

The equivalent Weight of potassium permanganate is 31.6.

Procedure:

(a) Preparation of standard copper sulphate solution:

About 0.80 g of analar copper sulphate crystals are weighed out accurately, dissolved in distilled water and made up to 100ml in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of potassium permanganate solution **$KMnO_4$ Vs. Std $Na_2S_2O_3$**

S.No.	Volume of $KMnO_4$ mL	Burette Reading		Volume of $Na_2S_2O_3$ mL	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			Starch
3.	20.0	0			

Concordant value = _____

Calculation of strength of $KMnO_4$

Volume of $Na_2S_2O_3$ (V_1) = mL

Strength of $Na_2S_2O_3$ (N_1) = N

Volume of $KMnO_4$ (V_2) = mL

Strength of $KMnO_4$ (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

= N

Amount of $KMnO_4$ present in in 100mL of the given solution = $\frac{\text{Normality} \times \text{Equivalent weight}}{10}$

= $\frac{\times 31.6}{10}$

= g

(b) Standardisation of sodium thiosulphate solution:

A clean burette is washed with water and rinsed with sodium thiosulphate and filled with the same. Exactly 20ml of standard copper sulphate solution is pipetted out into a clean conical flask. The mineral acid present is carefully neutralised with a few drops of dilute ammonia till a pale blue precipitate of $Cu(OH)_2$ is formed. The precipitate is just dissolved in one or two drops of 4N acetic acid. About 10ml of 10% potassium iodide solution is added and the liberated iodine is immediately titrated against the given sodium thiosulphate solution taken in a burette. When the colour becomes straw yellow, about 3-4 drops of freshly prepared 1% aqueous starch solution is added as indicator, which gives blue colour to the solution. The titration is continued until the blue colour commences to fade. Very close to the end point about 1-2g of ammonium thiocyanate crystals is added and the titration is continued till the blue colour just disappears, this is the end point. The titration is repeated to get concordant value. From the titre value the strength of sodium thiosulphate is calculated.

(c) Estimation of potassium permanganate solution:

The given Potassium permanganate solution is made up to 100mL in a SMF. Exactly 20ml of the made up solution is pipetted out into a clean conical flask. About 5ml of pure concentrated hydrochloric acid is added. About 5ml of 10% potassium iodide solution is added and it is titrated against the sodium thiosulphate solution taken in the burette. The addition of sodium thiosulphate is continued until the solution is pale yellow in colour. About 2ml of starch solution is added and the titration is continued till the blue colour changes to green. The titration is repeated for concordant value. From the titre value, the strength of potassium permanganate and hence the amount of potassium permanganate present in the whole of the given solution is calculated.

Result:

The amount of potassium permanganate in 100ml of the given solution = g.

Preparation of standard $ZnSO_4$ solution

Weight of $ZnSO_4.7H_2O$ + beaker = g

Weight of empty beaker = g

Weight of $ZnSO_4.7H_2O$ = g

$$\text{Strength of } ZnSO_4 = \frac{\text{Weight / Litre}}{\text{M. W}} = \frac{\times 10}{287.54}$$

Strength of $ZnSO_4$ = M

Titration I:

Standardisation of EDTA **Std $ZnSO_4$ Vs. Link EDTA**

S.No.	Volume of $ZnSO_4$ mL	Burette Reading		Volume of EDTA mL	Indicator
		Initial	Final		
1.	20.0	0			Solochrome black
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of strength of EDTA

Volume of standard $ZnSO_4$ (V_1) = mL

Strength of standard $ZnSO_4$ (N_1) = M

Volume of link EDTA (V_2) = mL

Strength of link EDTA (M_2) = $\frac{V_1 M_1}{V_2}$ [since $V_1M_1 = V_2M_2$]

= M

Strength of EDTA solution = M

Ex.No: 9

Date:

ESTIMATION OF ZINC (II) (Direct Method)

Aim:

To estimate the amount of zinc (II) present in the whole of the given solution by complexometric titration being provided with approximately 0.01M EDTA solution and analar zinc sulphate crystals.

Principle:

Zinc (II) forms a stable water soluble complex with EDTA in the pH range of 7-11 and hence it can be quantitatively estimated using EDTA at a pH of ~10 by solochrome black (SB) indicator. The zinc-indicator complex is wine red in colour and the zinc -EDTA complex is colourless. At the end point, colour of the solution changes from wine red to blue due to the release of the indicator in the free state.

$$[ZnIn]^- + H_2Y_2^- \rightarrow [ZnY]^{2-} + HIn^{2-} + H^+$$

Wine red Colourless Blue

Molecular weight of $ZnSO_4.7H_4O$ = 287.54

Atomic weight of Zinc = 65.39

$$ZnSO_4 \xleftrightarrow{\text{Titration I}} EDTA \xleftrightarrow{\text{Titration II}} Zn\,II$$

(Std) (Link) (Estimating Solution)

Procedure:

(a) Preparation of 0.01MStandard $ZnSO_4$ Solution

About 0.29g of analar $ZnSO_4.7H_2O$ crystals are accurately weighed out accurately, dissolved in distilled water and made up to 100mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of Zinc (II) **Std EDTA Vs Zn^{2+} solution**

S.No.	Volume of Zn^{2+} solution mL	Burette Reading		Volume of EDTA mL	Indicator
		Initial	Final		
1.	20.0	0			Solochrome black
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of strength of Zn^{2+} solution

Volume of EDTA (V_1) = mL

Strength of EDTA (M_1) = M

Volume of given Zn^{2+} solution (V_2) = 20.0 mL

Strength of given Zn^{2+} solution (M_2) = $\frac{V_1 M_1}{V_2}$ [since $V_1M_1 = V_2M_2$]

= M

Weight of Zinc (II) present in 100mL of the given solution = $\frac{\times 63.59}{10}$

= g

(b) Standardisation of EDTA Solution

A clean burette is washed with water and rinsed with EDTA and filled with the same. Exactly 20mL of the standard zinc sulphate solution is pipetted out into a clean conical flask, 50mL of de-ionised water and 1mL of $NH_3(aq) - NH_4Cl$ buffer (pH ~ 10) are added. A drop of solochrome black indicator is added and the wine red solution is titrated against EDTA solution taken in a burette until the colour is changed from wine red to blue, which is the end point.The titration is repeated to get concordant value. From the titre value the strength of EDTA is calculated.

(c) Estimation of Zinc(II)

The given zinc(Il) solution is made up to 100mL in a standard measuring flask. Exactly 20mL of the made up solution is pipetted out into a clean conical flask, 50mL of de-ionised water and 1mL of $NH_3(aq) - NH_4Cl$ buffer (pH ~ 10) are added. A drop of solochrome black indicator is added and the wine red solution is titrated against the standardized EDTA solution taken in the burette until the colour is changed from wine red to blue, which is the end point. The titration is repeated to get concordant value. From the titre value the strength and hence the amount of zinc (II) present in the whole of the given solution is calculated.

Result:

The amount of zinc(II) present in the whole of the given solution = g

Preparation of standard $Pb(NO_3)_2$ solution

Weight of $Pb(NO_3)_2$ + beaker = g

Weight of empty beaker = g

Weight of $Pb(NO_3)_2$ = g

Strength of $Pb(NO_3)_2 = \frac{\text{Weight / Litre}}{\text{M. W}} = \frac{\times 5}{331.20}$

Strength of $Pb(NO_3)_2$ = M

Titration I :

Standardisation of EDTA **(Std $Pb(NO_3)_2$ Vs. Link EDTA)**

S.No.	Volume of $Pb(NO_3)_2$ mL	Burette Reading		Volume of EDTA mL	Indicator
		Initial	Final		
1.					Xylenol orange
2.					
3.					

Concordant value = _____

Calculation of strength of EDTA

Volume of standard $Pb(NO_3)_2$ solution (V_1) = 20.0 mL

Strength of standard $Pb(NO_3)_2$ solution (M_1) = M

Volume of link EDTA solution (V_2) = mL

Strength of EDTA (N_2) = $\frac{V_1 M_1}{V_2}$ [since $V_1M_1 = V_2M_2$]

= M

Strength of EDTA solution = M

Ex.No: 10

Date:

ESTIMATION OF LEAD (II) (Direct Method)

Aim:

To estimate the amount of lead (II) present in the whole of the given solution by complexometric titration being provided with approximately 0.01M EDTA solution and analar lead nitrate crystals.

Principle

Lead (II) can be quantitatively estimated using EDTA at a pH of ~6 using xylenol orange indicator. The lead-indicator complex is red (reddish purple) in colour and the lead - EDTA complex is colourless. At the end point colour of the solution changes from red to lemon yellow due to the release of the indicator in the free state.

$$[Pb\text{-}In] + H_2Y^{2-} \rightarrow [ZnY]^{2-} + In + 2H^+$$

Red Colourless lemon yellow

Molecular weight of $Pb(NO_3)_2$ = 331.20

Atomic weight of Lead = 207.20

$$Pb(NO)_3 \xleftrightarrow{\text{Titration I}} EDTA \xleftrightarrow{\text{Titration II}} Pb\,II$$

(Std) (Link) (Estimating Solution)

Procedure

(a) Preparation of 0.01M Standard $Pb(NO_3)_2$ Solution

About 0.66g of analar $Pb(NO_3)_2$ crystals are accurately weighed out accurately, dissolved in distilled water and made up to 200mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of Pb(II) **Std EDTA Vs. Pb(II) solution**

S.No.	Volume of Pb (II) solution mL	Burette Reading		Volume of EDTA mL	Indicator
		Initial	Final		
1.					Xylenol orange
2.					
3.					

Concordant value = _____

Calculation of strength of

Volume of EDTA solution (V_1) = mL

Strength of EDTA solution (M_1) = M

Volume of Pb(II) solution (V_2) = 20.0 mL

Strength of Pb(II) solution (M_2) = $\frac{V_1 M_1}{V_2}$ [since $V_1N_1 = V_2M_2$]

= M

Weight of the lead(II) present in 100mL of the given solution = $\frac{\quad \times 207.20}{10}$

= g

(b) Standardisation of EDTA Solution

A clean burette is washed with water and rinsed with EDTA and filled with the same. Exactly 20mL of the standard $Pb(NO_3)_2$ solution is pipetted out into a clean conical flask, diluted to 100mL with de-ionised water. 2mL of 5% aqueous solution of hexamine (or ~0.25g of solid hexamine) is added to maintain the pH to ~6. Two drops of xylenol orange indictor are added and the red colour solution is titrated against the EDTA solution taken in a burette, the end point is the colour change from red to lemon yellow. The titration is repeated to get concordant values. From the titre value the strength of EDTA is calculated.

(c) Estimation of Pb(II)

The given Pb(II) solution is made up to 100mL in a standard measuring flask. Exactly 20mL of the made up solution is pipetted out into a clean conical flask, diluted to 100mL with de-ionised water and 2mL of 5% aqueous solution of hexamine (or ~0.25g of solid hexamine) is added (pH ~6) Two drops of xylenol orange indicator are added and the red colour solution is titrated against EDTA solution taken in the burette ,the end point is colour change from red to lemon yellow.The titration is repeated to get concordant values. From the titre value the strength and hence the amount of Pb (II) present in the whole of the given solution is calculated.

Result:

The amount of Pb(II) present in the whole of the given solution = g.

Note:

1. Only two to three drops of indicator should be added otherwise identification of the end point would be difficult.

Preparation of standard $MnSO_4$ solution

Weight of $MnSO_4 . H_2O$ + beaker = g

Weight of empty beaker = g

Weight of $MnSO_4 . H_2O$ = g

$$\text{Strength of } MnSO_4 . H_2O = \frac{\text{Weight / Litre}}{\text{Molecular Weight}} = \frac{\quad \times 10}{169.02}$$

Strength of $MnSO_4 . H_2O$ = M

Titration I :

Standardisation of EDTA **Std $MnSO_4$ Vs. Link EDTA**

S.No.	Volume of $MnSO_4$ mL	Burette Reading		Volume of EDTA mL	Indicator
		Initial	Final		
1.	20.0	0			Solochrome black
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of the strength of EDTA solution

Volume of standard $MnSO_4$ solution, (V_1) = 20.0 mL

Strength of standard $MnSO_4$ solution, (M_1) = M

Volume of link EDTA solution, (V_2) = mL

Strength of EDTA (M_2) = $\frac{V_1 M_1}{V_2}$ [since $V_1M_1 = V_2M_2$]

Strength of EDTA = M

Ex.No: 11

Date:

ESTIMATION OF MANGANESE (II)

Aim:

To estimate the amount of manganese (II) present in the whole of the given solution by complexometric titration being provided with approximately 0.01M EDTA solution and analar manganese sulphate crystals.

Principle:

Manganese (II) forms a stable water soluble complex with EDTA at a pH of ~10 and hence it can be quantitatively estimated using EDTA at a pH of ~10 by solochrome black indicator. The manganese-indicator complex is wine red in colour and the Mn - EDTA complex is colourless. At the equivalent point EDTA quantitatively replaces the indicator from the metal-indicator complex and is set free which gives blue colour to the solution. Hence at the end point the colour changes from wine red to blue.

$[Mn\ ln]^- + H_2Y^{2-} \rightarrow [MnY]^{2-} + HIn^{2-} + H^+$

Red Colourless Blue

Molecular weight of $MnSO_4 . H_2O$ = 169.02

Atomic weight of Manganese = 54.94

$$MnSO_4 \xleftrightarrow{\text{Titration I}} EDTA \xleftrightarrow{\text{Titration II}} Mn(II)$$

(Std) (Link) (Estimating Solution)

Procedure:

(a) Preparation of 0.01M Standard $MnSO_4$ Solution

About 0.17g of analar $MnSO_4 . H_2O$ crystals are accurately weighed out accurately, dissolved in distilled water and made up to 100mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of Mn (II) **Std EDTA Vs Mn (II) solution**

S.No.	Volume of Mn (II) Solution mL	Burette Reading		Volume of EDTA mL	Indicator
		Initial	Final		
1.					Solochrome black
2.					
3.					

Concordant value = _____

Calculation of strength of EDTA solution

Volume of standardised EDTA solution, (V_1) = mL

Strength of standardised EDTA solution, (N_1) = M

Volume of given Mn (II) solution, (V_2) = mL

Strength of given Mn (II) solution $(M_2) = \frac{V_1 M_1}{V_2}$ [since $V_1M_1 = V_2M_2$]

= M

Weight of the Mn (II) present in 100mL of the given solution $= \frac{\text{Normality} \times \text{Atomic weight}}{10}$

= g

(b) Standardisation of EDTA Solution

A clean burette is washed with water and rinsed with EDTA and filled with the same. Exactly 20mL of the standard $MnSO_4$ solution is pipetted out into a clean conical flask, 50mL of de-ionised water 2mL of 5% potassium sodium tartarate solution are added. Then 2mL of 10% hydroxylamine hydrochloride and 2mL of concentrated aqueous ammonia solution are added. The solution is warmed to 60°C. A drop of solochrome black indicator is added and the wine red solution is titrated against EDTA solution taken in a burette until the colour is changed from wine red to blue, which is the end point. The titration is repeated to get concordant value. From the titre value, the strength of EDTA is calculated.

(c) Estimation of Manganese (II)

The given manganese (II) solution is made up to 100mL in a standard measuring flask. Exactly 20 mL of the made up solution is pipetted out into a clean conical flask, 50mL of de-ionised water and 2mL of 5% potassium sodium tartarate solution are added. Then 2mL of 10% hydroxylamine hydrochloride and 2mL of concentrated aqueous ammonia solution are added. The solution is warmed to 60°C, 2 drops of solochrome black indicator are added and the wine red solution is titrated against the standardised EDTA solution taken in the burette until the colour is changed from wine red to blue, which is the end point. Titration is repeated to get concordant value. From the titre value ,the strength and hence the amount of manganese (II) present in the whole of the given solution is calculated.

Result:

The amount of Mn (II) present in the whole of the given solution = g

Preparation of standard $NiSO_4$ solution

Weight of $NiSO_4.6H_2O$ + beaker = g

Weight of empty beaker = g

Weight of $NiSO_4.6H_2O$ = g

Strength of $NiSO_4$ $= \dfrac{\text{Weight / Litre}}{\text{Molecular Weight}} = \dfrac{\times 10}{262.86}$

Strength of $NiSO_4$ = M

Titration I :

Standardisation of EDTA **Std $NiSO_4$ Vs. Link EDTA**

S.No.	Volume of $NiSO_4$ mL	Burette Reading		Volume of EDTA mL	Indicator
		Initial	Final		
1.					Murexide
2.					
3.					

Concordant value = _____

Calculation of strength of EDTA solution

Volume of standard $NiSO_4$ (V_1) = mL

Strength of standard $NiSO_4$ (M_1) = M

Volume of EDTA (V_2) = mL

Strength of EDTA (M_2) = $\dfrac{V_1 M_1}{V_2}$ [since $V_1M_1 = V_2M_2$]

Strength of EDTA = M

Ex.No: 12

Date:

ESTIMATION OF NICKEL (II)

Aim:

To estimate the amount of Nickel (II) present in the whole of the given solution by complexometric titration being provided with approximately 0.01M EDTA solution and analar nickel sulphate crystals.

Principle:

Nickel (II) can be quantitatively estimated using EDTA at a pH of 10-11 by murexide indicator. The nickel murexide complex is yellow in colour. At the end point the metal ion indicator is released by EDTA resulting in a sharp colour change from yellow to violet.

$$[\text{Ni-In}] + H_2Y^{2-} \rightarrow [NiY]^{2-} + In + 2H^+$$

Yellow — Colourless — Violet

Molecular weight of $NiSO_4 . 6H_2O$ = 262.86

Atomic weight of Nickel = 58.69

$$MiSO_4 \xleftrightarrow{\text{Titration I}} EDTA \xleftrightarrow{\text{Titration II}} Ni\,II$$

(Std) — (Link) — (Estimating Solution)

Procedure:

(a) Preparation of 0.01M Nickel sulphate solution

About 0.26g of analar Nickel Sulphate crystals are accurately weighed out accurately, dissolved in distilled water and made up to 100mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration II :

Estimation of Ni (II) **Std EDTA Vs $NiSO_4$**

S.No.	Volume of $NiSO_4$ mL	Burette Reading		Volume of EDTA mL	Indicator
		Initial	Final		
1.					Murexide
2.					
3.					

Concordant value = _____

Calculation of strength of Ni (II) solution

Volume of EDTA (V_1) = mL

Strength of EDTA (M_1) = M

Volume of Ni (II) solution (V_2) = mL

Strength of Ni (II) solution (M_2) = $\frac{V_1 M_1}{V_2}$ [since $V_1M_1 = V_2M_2$]

= M

Weight of the Ni (II) present in 100mL of the given solution = $\frac{\text{Molarity} \times \text{Atomic weight}}{10}$

= g

(b) Standardisation of EDTA Solution

A clean burette is washed with water and rinsed with EDTA and filled with the same. Exactly 20mL of standard Ni^{2+} solution is pipetted out into a clean conical flask diluted to 100mL with de-ionised water .5mL of 1 M NH4Cl and 3-4 mL of concentrated ammonia solution are added. Around 2-5mg of murexide potassium nitrate mixture is added and the solution is titrated against EDTA solution taken in the burette. The end point is the change of colour from yellow to violet. The titration is repeated to get concordant value. From the titre value, the strength of EDTA is calculated.

(c) Estimation of Nickel(II)

The given nickel (II) solution is made up to 100mL in a standard measuring flask. Exactly 20mL of the made up solution is pipetted out into a conical flask. The solution is diluted to 100mL with de-ionised water, 5mL of 1M NH_4Cl and 3-4 mL of concentrated ammonia solution are added. Around 2-5mg of murexide- potassium nitrate mixture is added and the solution is titrated against EDTA taken in the burette. The end point is the change of colour from yellow to violet. The titration is repeated to get concordant value. From the titre value, the strength and hence the amount of nickel (II) present in the whole of the given solution is calculated.

Result:

The amount of nickel (II) present in
the whole of the given solution = g

Calculations

I) Preparation of $K_2Cr_2O_7$ solution

Weight of weighing bottle + $K_2Cr_2O_7$ =

Weight of empty weighing bottle =

Weight of $K_2Cr_2O_7$ crystals = _____ g

Strength of $K_2Cr_2O_7$ solution = $\frac{\text{Weight} \times 10}{49}$

= _____ N

II) Standardisation of Thio solution

S.No.	Volume of $K_2Cr_2O_7$ Solution (mL)	Burette Reading		Volume of Thio Solution (mL)
		Initial	Final	
1.	20			
2.	20			

Concordant value = _____

Strength of Thio solution = $\frac{V_{Dichromate} \times N_{Dichromate}}{V_{Thio}}$

= _____ N

COURSE WORK

Ex.No: 13

Date:

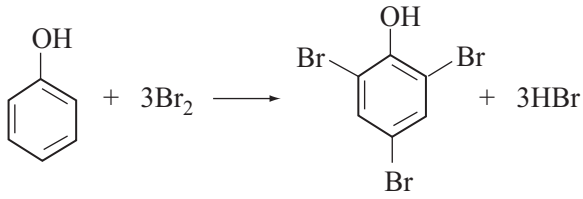
ESTIMATION OF PHENOL

Aim:

To estimate the amount of phenol present in the whole of the given solution. Provided with the standard $K_2Cr_2O_7$ containing 5g/ litre.

Principle

Phenol reacts with bromine to give sym-tribromophenol.



$C_6H_5OH \equiv 3Br2 \equiv 6$ Equivalents

Eq. wt of phenol = Mol.wt / 6 = 94 / 6 = 15.67

Phenol is treated with a known excess of acidified bromide – bromated mixture (winkler's solution). The unreacted bromine liberates I_2 from KI and the liberated I_2 is titrated against standard thio solution. From the titre value, the quantity of bromine reacted and hence the amount of phenol present in the given solution can be calculated.

Procedure

i) Preparation of Std $K_2Cr_2O_7$ solution

About 0.49gm AR potassium dichromate crystals are weighed accurately, dissolved in distilled water and made up to 100ml in a standard measuring flask. The solution is shaken well for uniform concentration.

III) Standardisation of Brominating solution

S.No.	Volume of Winkler Solution (ml)	Burette Reading		Volume of Thio Solution (mL)
		Initial	Final	
1.	20			V_2
2.	20			V_2

Concordant value = _____

IV) Estimation of Phenol

S.No.	Volume of Phenol + Winkler Solution (ml)	Burette Reading		Volume of Thio Solution (mL)
		Initial	Final	
1.	20 + 40			V_3
2.	20 + 40			V_3

Concordant value = _____

Strength of phenol $= \dfrac{(2V_2 - V_3) \times N_{Thio}}{20}$

$=$ _____ N

Weight of phenol in 200 ml $= \dfrac{N_{phenol} \times 15.67}{5}$

$=$ _____ g

ii) Standardisation of Thio solution

20 ml of the std potassium dichromate solution is pipetted out into a conical flask. About 5 ml of conc. HCl and 5 ml of 10% KI solution are added and the liberated iodine is titrated against the thio solution taken in the burette. When the solution becomes straw yellow in colour, 1 ml of freshly prepared starch solution is added and the titration is continued by adding thio in drops. The end point is the colour change from blue to green. The titration is repeated for concordant value. From the titre value, the strength of thio solution is calculated.

iii) Standardisation of Brominating solution

20 ml of Winkler solution is pipetted out into a conical flask and diluted with about 40 ml of water. Now, 5 ml of conc. HCl and 15 ml of 10% KI solutions are added and the liberated iodine is titrated against the thio solution taken in the burette. Starch is the indicator. Disappearance of blue colour is the endpoint. The titration is repeated for concordant value.

iv) Estimation of Phenol

The given phenol solution is made up to 200 ml in a standard measuring flask. 20 ml of the made up solution is pipetted out into a stoppered conical flask (Iodine flask). Exactly 40 ml of Winkler solution is added. The solution is diluted with 40 ml of water and 5 ml of conc. HCl is added. The flask is closed immediately and shaken well. The contents of the flask are allowed to stand for 15 minutes with occasional shaking. Now, about 15 ml of 10% KI solution is added and the liberated iodine is titrated against the thio solution taken in the burette. Disappearance of blue colour is the endpoint. A duplicate is also done.

Result

Weight of phenol present in the whole of the given solution = _____

Calculations

I) Preparation of $K_2Cr_2O_7$ solution

Weight of weighing bottle + $K_2Cr_2O_7$ =

Weight of empty weighing bottle =

Weight of $K_2Cr_2O_7$ crystals = _____ g

Strength of $K_2Cr_2O_7$ solution = $\frac{\text{Weight} \times 10}{49}$

= _____ N

II) Standardisation of Thio solution

S.No.	Volume of $K_2Cr_2O_7$ Solution (mL)	Burette Reading		Volume of Thio Solution (mL)
		Initial	Final	
1.	20			
2.	20			

Concordant value = _____

Strength of Thio solution = $\frac{V_{Dichromate} \times N_{Dichromate}}{V_{Thio}}$

= _____ N

Ex.No: 14

Date:

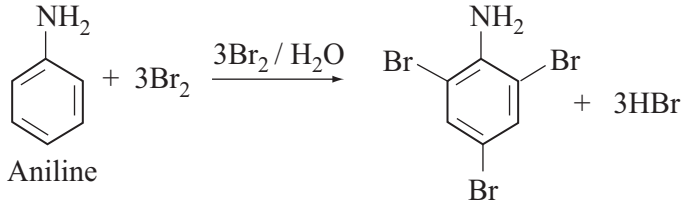
ESTIMATION OF ANILINE

Aim:

To estimate the amount of aniline present in the whole of the given solution. Provided with the standard $K_2Cr_2O_7$ containing 5g / litre.

Principle

Like phenol, aniline is readily brominated with a known excess of acidified bromide-bromate mixture to give sym- tribromo aniline.



Aniline

2,4,6 - Tribromoaniline

$$KBrO_3 + 5KBr + 6HCl \rightarrow 6\,KCl + 3\,Br_2 + 3H_2O$$

$$C_6H_5NH_2 \equiv 3\,Br_2 \equiv 6 \text{ Equivalents}$$

Eq.wt of Aniline = Mol. wt / 6 = 15.5

The unreacted bromine liberates I_2 from KI and the liberated iodine is titrated against standard thio solution. From the titre value, the quantity of bromine reacted is calculated. This, in turn, is used to calculate the amount of aniline present in the whole of the given solution.

Procedure

I) Preparation of std $K_2Cr_2O_7$ solution

About 0.49gm of AR potassium dichromate crystals are weighed accurately, dissolved

III) Standardisation of Brominating solution

S.No.	Volume of Winkler Solution (ml)	Burette Reading		Volume of Thio Solution (mL)
		Initial	Final	
1.	20			V_2
2.	20			V_2

Concordant value = _____

IV) Estimation of Aniline

S.No.	Volume of Phenol + Winkler Solution (ml)	Burette Reading		Volume of Thio Solution (mL)
		Initial	Final	
1.	20 + 40			V_3
2.	20 + 40			V_3

Concordant value = _____

Strength of Aniline $= \dfrac{(2V_2 - V_3) \times N_{Thio}}{20}$

$=$ _____ N

Weight of Aniline in 200 ml $= \dfrac{N_{Aniline} \times 15.5}{5}$

$=$ _____ g

in water and made up to 100ml in a standard measuring flask. The solution is shaken well for uniform concentration.

II) Standardisation of Thio solution

20ml of the std potassium dichromate solution is pipetted out into a conical flask. About 5ml of conc.HCl and 5ml of 10% KI solution are added. The liberated iodine is titrated against the thio solution taken in the burette. When the solution becomes straw yellow in colour, 1ml of a freshly prepared solution of starch is added and the titration is continued by adding thio in drops. The endpoint is the change of colour from blue to green. The titration is repeated to get concordant value. From the titre value, the strength of thio solution is calculated.

III) Standardisation of Brominating solution

20ml of Winkler solution is pipetted out into a conical flask and diluted with about 40ml of water. Now, 5ml of conc. HCl and 15ml 10% KI solution are added and the liberated iodine is titrated against the thio solution taken in a burette. Starch is the indicator and the disappearance of blue colour is the endpoint. The titration is repeated to get concordant value.

IV) Estimation of Aniline

The given aniline solution is made up to 200ml in a SMF. 20ml of the made up solution is pipetted out into a stoppered conical flask (Iodine flask). Exactly 40ml of Winkler solution is added and the solution is diluted with 40ml of water. Now, 5ml of conc. HCl is added and the flask is immediately closed and shaken well. The contents of the flask are allowed to stand for 15 minutes with occasional shaking. Now, about 15ml of KI solution is added and the liberated iodine is titrated against the thio solution. Starch is the indicator and the disappearance of blue colour is the endpoint. A duplicate titration is also done.

Result

Weight of aniline present in the whole of the given solution = _____ g

I) Preparation of standard acetic acid solution

Weight of acetic acid + beaker = g

Weight of empty beaker = g

Weight of acetic acid = g

Strength of acetic acid solution = $\frac{\text{Weight} \times 10}{49}$

= N

Titration I:

II) Standardisation of Thio solution

S.No.	Volume of NaOH mL	Burette Reading		Volume of Acetic acid mL	Volume of Thio Solution (mL)
		Initial	Final		
1.	20.0	0			Phenolphthalein
2.	20.0	0			
2.	20.0	0			

Concordant value = _____

Calculation of the strength of sodium hydroxide

Volume of standard acetic acid (V_1) = mL

Strength of standard acetic acid (N_1) = N

Volume of link sodium hydroxide (V_2) = mL

Strength of sodium hydroxide (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of sodium hydroxide = N

Ex.No: 15

Date:

ESTIMATION OF ACETIC ACID IN COMMERCIAL VINEGAR

Aim:

To estimate the amount of acetic acid in the vinegar sample.

Principle:

A known weight of glacial acetic acid is taken and diluted to a definite volume in a standard measuring flask. It is titrated against sodium hydroxide solution. From the titre value the normality of sodium hydroxide is calculated.

The given sample of commercial vinegar is made upto 100mL in a standard measuring flask. It is titrated against sodium hydroxide. From the titre value the normality of acetic acid and the weight of acetic acid is calculated.

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$

Equivalent weight of acetic acid = 60

Procedure

Preparation of standard acetic acid

Weigh accurately about 1.2 g of acetic acid in a beaker and make it up to 200mL in a standard measuring flask.

Standardisation of sodium hydroxide

Wash the burette with distilled water, rinse with the standard acetic acid and fill the burette upto the mark. Pipette out 20mL of sodium hydroxide into a clean conical flask and add 1 or 2 drops of phenolphthalein.

Titration II :

Estimation of Vinegar **NaOH Vs. Vinegar**

S.No.	Volume of NaOH mL	Burette Reading		Volume of Vinegar mL	Indicator
		Initial	Final		
1.	20.0	0			Phenolphthalein
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of strength of vinegar

Volume of sodium hydroxide (V_1) = mL

Strength of sodium hydroxide (N_1) = N

Volume of vinegar (V_2) = mL

Strength of vinegar = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

= N

The Amount of acetic acid present in 200mL of the given solution = $\frac{\quad \times 60.05}{5}$

= g

Titrate this solution against standard acetic acid , the end point is the disappearance of pink colour. The titration is repeated for concordant value. From the titre value , the strength of sodium hydroxide is calculated.

Estimation of acetic acid

Make up the given sample of vinegar to 100mL . Wash the burette with water and rinse with vinegar and fill it upto the mark. Pipette out 20mL of sodium hydroxide into a conical flask. Add one drop of phenolphthalein and titrate this solution against acetic acid. The end point is the disappearance of pink colour. The titration is repeated for concordant value. From the titre value, the strength of acetic acid is calculated.

Result

The amount of acetic acid present in the whole of the given solution = _____ g.

Estimation of Oxalate Content

Volume of the fruit extract = V_1 ml

Volume of $KMnO_4$ = V_2 ml

Strength of $KMnO_4$ = N_2

Strength of the fruit extract, N1 =

$$= \frac{V_1 N_1}{V_2}$$

Weight of oxalate content (oxalic acid)
in 100 ml of the fruit extract = g

Ex.No: 16

Date:

ESTIMATION OF OXALATE CONTENT IN VEGETABLES AND FRUITS

Aim:

To estimate the amount of oxalate present in fruits and vegetables.

Procedure:

5 g of the fruit is weighed and crushed into fine pulp using pestle and mortar. The crushed pulp is transferred into a beaker and about 5 ml of dil.sulphuric acid is added. The contents are boiled for about 10 minutes, cooled and then filtered. It is then transferred into a standard measuring flask and made up to 100 ml with distilled water. 20 ml of this fruit extract is pipetted out from the standard measuring flask into a conical flask and acidified with 20 ml of dil.sulphuric acid. The mixture is heated to about 60ºC and then it is titrated against 0.1N potassium permanganate solution taken in the burette which is standardized using standard oxalic acid. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant value. From the titre value, the amount of oxalate content is calculated.

Result:

Amount of oxalate content present 100 ml of the fruit extract = ---------- g.

Preparation of 0.1 N standard sodium carbonate Solution

Preparation of std Na_2CO_3 solution:

Weight of Na_2CO_3 + Weighing bottle = g

Weight of empty weighing beaker = g

Weight of Na_2CO_3 = g

Strength of Na_2CO_3 solution = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

Strength of Na_2CO_3 = N

Titration I :

Standardisation of Hydrochloric acid **Std Na_2CO_3 Vs. HCl**

S.No.	Volume of Na_2CO_3 mL	Burette Reading		Volume of HCI mL	Indicator
		Initial	Final		
1.	20.0	0			Methyl orange
2.	20.0	0			
3.	20.0	0			

Concordant value = _____

Calculation of strength of hydrochloric acid solution

Volume of standard sodium carbonate (V_1) = 20.0 mL

Strength of standard sodium carbonate (N_1) = N

Volume of link hydrochloric acid (V_2) = mL

Strength of hydrochloric acid solution (N_2) = $\frac{V_1 N_1}{V_2}$ [since $V_1N_1 = V_2N_2$]

Strength of hydrochloric acid solution = N

Ex.No: 17

Date:

ESTIMATION OF SODIUM CARBONATE AND SODIUM BICARBONATE IN A MIXTURE

Aim:

To estimate the amount of sodium carbonate and sodium bicarbonate present in the whole of the given solution by Warder's double indicator method. Pure sodium carbonate and an approximately 0.1 N solution of hydrochloric acid are provided.

Principle:

The sodium carbonate and sodium bicarbonate present in a mixture can be estimated by titrating with hydrochloric acid using phenolphthalein and methylorange indicators. The reaction between sodium carbonate and hydrochloric acid takes place in two steps.

$$Na_2CO_3 + HCl \rightarrow NaHCO_3 + NaCl$$

$$\underline{NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2}$$

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$$

The equivalent point for the first step of the reaction is at pH 8.3, hence the end point of this reaction can be determined using phenolphthalein indicator.

As soon as the first step is over, the carbonic acid produced from the second step decreases the pH, and hence the colour changes from pink to colourless. The volume of hydrochloric acid, (V1) is equivalent to half of the amount of sodium carbonate present in the mixture.

At this stage, if methyl orange is added and the titration is continued, the sodium bicarbonate reacts with hydrochloric acid and at the equivalent point the colour changes from golden yellow to red orange. This titre value (V_3) gives the acid equivalent of sodium bicarbonate originally present in the mixture and sodium bicarbonate formed from the sodium carbonate in the first step of the reaction. From the titre values V_1 and V_3 the amount of sodium carbonate and sodium bicarbonate respectively present in the whole of the given solution can be calculated.

Titration II :

Estimation of Na_2CO_3 and $NaHCO_3$ in the mixture **Std HCl Vs. Given mixture**

S.No.	Phenolphthalein				Methyl Orange		
	Volume mixture mL (V_2)	Burette Reading		Volume of HCl = 1/2 of Na_2CO_3 mL (V_1)	Burette Reading		Volume of HCl = 1/2 of $NaHCO_3$ mL (V_3)
		Initial	Final		Initial	Final	
1.							
2.							
3.							

Concordant value = _____

Calculation of the weight of Na_2CO_3:

Volume of HCl required to neutralise half of Na_2CO_3, (V_1) = mL

Volume of HCl required to neutralise whole half of Na_2CO_3, ($2V_1$) = mL

Strength of standardised HCl, (N_1) = N

Volume of given mixture (V_2) = 20.0 mL

Strength of Na_2CO_3 in the mixture (N_2) = $\frac{2V_1 N_1}{V_2}$ =

Strength of Na_2CO_3 in the mixture = N

The amount of Na_2CO_3 present in 100mL of the given solution = $\frac{\quad \times 53}{10}$

= g

Procedure

(a) Preparation of 0.1 N standard sodium carbonate Solution:

About 0.53g of sodium carbonate is weighed accurately and dissolved in distilled water and made up to 100mL in a standard measuring flask.

(b) Standardisation of hydrochloric acid:

Exactly 20mL of the made up solution is pipetted out in to a clean conical flask and two drops of methyl orange indicator are added. The golden yellow colour solution is titrated against the link hydrochloric acid solution taken in the burette. The end point is the colour change from golden yellow to red orange. The titration is repeated to get concordant values. From the titre value the strength of hydrochloric acid is calculated.

(c) Estimation of sodium carbonate and sodium bicarbonate in the mixture:

The given sodium carbonate - bicarbonate mixture is made upto l00mL in a standard measuring flask. Exactly 20mL of the made up solution is pipetted out into a clean conical flask. Two drops of phenolphthalein indicator are added and the pink colour solution is titrated against the standardised hydrochloric acid taken in the burette. The end point is the disappearance of pink colour. This volume, V_1 is eqiuvalent to half of the sodium carbonate present in the mixture.

Then two drops of methyl orange indicator is added to the same solution titrated against the same hydrochloric acid until the colour changes from golden yellow to pale red orange which is the end point. This volume V_3 is equivalent to the total sodium bicarbonate present in the mixture. The titration is repeated to get concordant values. From the titre value the amount of sodium carbonate and sodium bicarbonate present in the whole of the given mixture is calculated.

Calculation of the weight of $NaHCO_3$:

Volume of HCl required to neutralise the whole of $NaHCO_3$, V_3-V_1 = mL

Strength of HCl, (N_1) = N

Volume of given mixture (V_2) = 20.0 mL

Strength of $NaHCO_3$ in the mixture (N_2) = $\frac{(V_3 - V_1)N_1}{V_2}$ =

The amount of $NaHCO_3$ present in 100mL of the given solution = $\frac{\quad}{\quad} \times 84$

= g

Result:

The amount of sodium carbonate present
in the whole of the given mixture = g

The amount of sodium bicarbonate present
in the whole of the given mixture = g

Calculation

i) 50 ml of std hard water $= V_1$ ml of EDTA

(or) 50×1 mg of $CaCO_3 = V_1$ ml of EDTA

$\therefore$ 1 ml of EDTA $= \frac{50}{V_1}$ gm of $CaCO_3$

ii) 50 ml of hard water $= V_2$ ml of EDTA

$= V_2 \times \frac{50}{V_1}$ mg of $CaCO_3$

$\therefore$ 1000 ml of hard water $= \frac{V_2}{V_1} \times 1000$ mg of $CaCO_3$

(or) Total hardness of water (H_T) $= \frac{V_2}{V_1} \times 1000$ mg / litre

$= \frac{V_2}{V_1} \times 1000$ ppm

Ex.No: 18

Date:

ESTIMATION OF TOTAL HARDNESS OF WATER

Aim:

To determine the hardness of the given sample of water by EDTA method. You are provided with a 0.01M EDTA solution.

Principle:

The convenient method of estimating hardness of water is by titrating hard water against the disodium salt of ethylene diamine tetracetic acid (EDTA) using Eriochrome black T indicator. The alcoholic solution of the indicator is *blue* in colour.

When 2-3 drops of the indicator are added to hard water, a weak indicator – Ca^{2+} or Mg^{2+} complex is formed. This is *wine red* in colour. When this coloured solution is treated with EDTA solution, the Ca^{2+} or Mg^{2+} ions form a stable complex with EDTA and the indicator is set free. Thus, the change of colour from wine red to blue indicates the end point.

Procedure

1) **Preparation of Standard Hard Water:** Dissolve 1 gm of pure calcium carbonate in minimum amount of dil. HCl and make up the solution to 1 litre with distilled water. The hardness of this solution is 1 mg / ml.

2) **Standardisation of EDTA Solution:** Fill the burette with the EDTA solution. Take 50 ml of standard hard water in a conical flask and add 10 ml of a buffer solution containing NH_4Cl and NH_4OH (to adjust the pH to 10). Now, add 4-5 drops of Eriochrome black T indicator. The solution turns wine red. Titrate this solution against the EDTA solution taken in the burette till the colour changes to steel blue. Let the volume of EDTA solution consumed by V_1 ml.

3) **Estimation of Hardness of Water:** Titrate 50 ml of the water sample against the EDTA solution as in step (ii). Let the titre value by V_2 ml.

Result

Total hardness of water = _____ ppm

List of Equivalent weights, Atomic weights and Molecular weights

S. No.	Substance	Equivalent Weight
1.	Oxalic acid	63
2.	Sodium carbonate	53
3.	Hydrochloric acid	36.5
4.	Sulphuric acid	49
5.	Ferrous ammonium sulphate (FAS)	392
6.	Potassium permanganate	31.6
7.	Potassium dichromate	49
8.	Iodine	127
9.	Sodium bicarbonate	84
10.	Sodium hydroxide	40

S. No.	Substance	Atomic weight
1.	Iron / Fe^{2+}	55.85
2.	Manganese	54.94
3.	Magnesium	24.31
4.	Nickel	58.69
5.	Zinc	65.39
6.	Lead	207.20

S. No.	Substance	Molecular weight
1.	$CuSO_4 . 5H_2O$	249.68
2.	$MnSO_4 . 7H_2O$	169.02
3.	$MgSO_4 . 7H_2O$	246.48
4.	$NiSO_4 . 7H_2O$	280.69
5.	$ZnSO_4 . 7H_2O$	287.54
6.	$Pb(NO_3)_2$	331.20

SHORT PROCEDURE

1. Estimation of Hydrochloric Acid

Standard	Link	**Estimating solution**
Oxalic acid	NaOH	Oxalic acid

Preparation of std. oxalic acid solution:

About 0.63 g of analar oxalic acid crystals is weighed out accurately, dissolved in distilled water and made upto 100 mL in a standard measuring flask. The solution is shaken well for uniform concentration.

Titration 1

Burette Solution	:	Oxalic acid
Pipette Solution	:	20 mL of Sodium hydroxide
Condition and medium	:	—
Temperature	:	Room Temperature
Indicator	:	Phenolphthalein
End point	:	Disappearance of pink colour.

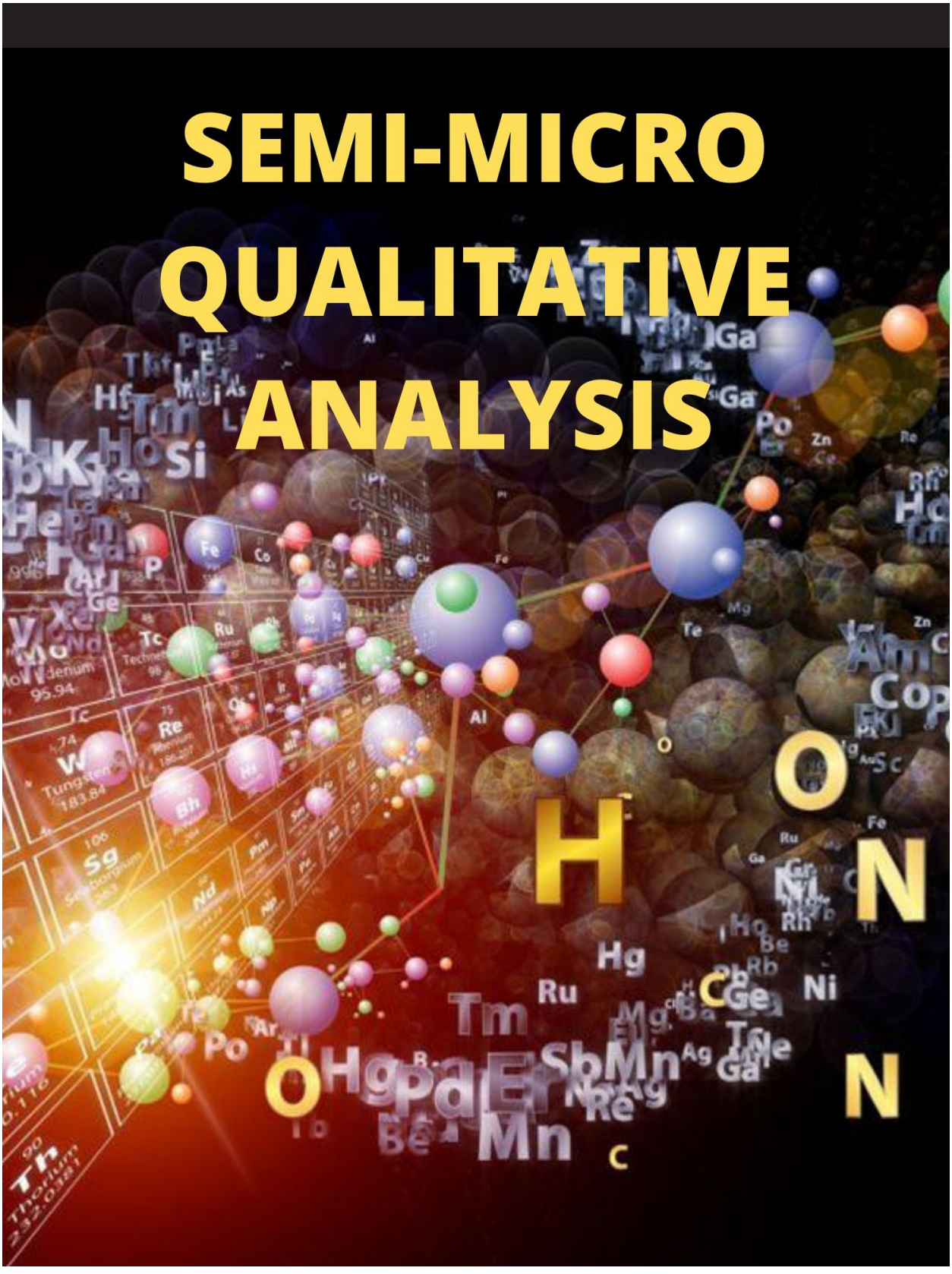
Titration 2

The given Oxalic acid is made up to 100 mL in a 100 mL standard measuring flask.

Burette Solution	:	Given Oxalic acid
Pipette Solution	:	20 mL of NaOH
Condition and medium	:	—
Temperature	:	Room Temperature
Indicator	:	Phenolphthalein
End point	:	Disappearance of pink colour.

Equivalent weight of Oxalic acid (standard) =

SEMI-MICRO QUALITATIVE ANALYSIS



SEMI MICRO INORGANIC QUALITATIVE ANALYSIS

Subject Code: 18UCHCR2

CONTENTS

SEMI MICRO INORGANIC QUALITATIVE ANALYSIS

Analysis of Acid Radicals

EXPERIMENT	OBSERVATION	INFERENCE
1. Colour of the substance is noted	Blue	May be copper
	Bright Green	May be copper or nickel
	Pale green	May be ferrous
	Yellow	May be chromate
	Pink or flesh	May be cobalt or manganese
	White	Absence of copper, iron, cobalt, chromate, manganese and nickel
2. Action of heat A little of the substance is heated strongly in a dry test tube.	Colourless gas turning lime water milky	May be carbonate or oxalate
	Reddish brown gas	May be nitrate or bromide
	Violet vapours	Presence of iodide
	Yellow when hot and white when cold.	Presence of zinc
	Colourless pungent smelling gas giving white dense fumes with a glass rod dipped in con.HCl.	Presence of ammonium
	Black residue	May be copper, cobalt, nickel or manganese
	No characteristic change	Absence of carbonate, oxalate, nitrate, bromide, iodide ammonium, zinc, copper, cobalt, manganese and nickel
3. Solubility test i) In water	Soluble	Presence of ammonium
	Insoluble	Absence of ammonium

ii) In dil. HCl	Insoluble	Presence of I group
	Soluble	Absence of I group
4. Flame test a) With conc. HCl. The substance is made into a paste with conc. HCl in a watch glass. A little of this is shown in the flame.	Green or bluish green	Presence of copper or borate
	Pale Green	Presence of barium
	Crimson red	Presence of strontium
	Brick red	Presence of calcium
	No characteristic coloured flame	Absence of copper, borate, barium, strontium and calcium
b) With CaF_2 and conc. H_2SO_4. The substance is mixed with a little CaF_2 and conc. H_2SO_4 and made into a paste in a watch glass. A little of the paste is shown in the flame.	Deep green colour	Presence of borate
	No green colour	Absence of borate
5. Action of NaOH A little of the substance is heated with NaOH solution	Colourless gas with pungent smell which gives white dense fumes with a glass rod dipped in conc. HCl and turning red litmus paper blue	Presence of ammonium
	No pungent smelling gas is evolved	Absence of ammonium
Wet tests		
6. Action of dil.HCl a) A little of the substance is treated with dil. HCl	Colourless gas with brisk effervescence turning lime water milky	Presence of carbonate is confirmed
	No brisk effervescence	Absence of carbonate
b) The above solution is warmed.	Colourless gas with rotten egg smell turning lead acetate paper black.	Presence of sulphide is confirmed.
	No rotten egg smell	Absence of sulphide.

7. Action of MnO_2 and dil. H_2SO_4 A little of the substance is warmed with dil. H_2SO_4. To the hot solution a pinch of MnO_2 is added.	Effervescence and colourless gas turning lime water milky.	Presence of oxalate.
	No effervescence	Absence of oxalate.
8. Action of conc. HCl A little of the substance is heated with con. HCl.	Greenish yellow gas is evolved and the solution turned green.	Presence of chromate.
	No greenish yellow gas	Absence of chromate.
9. Action of conc. H_2SO_4 A little of the substance is warmed with con. H_2SO_4.	Reddish brown vapours	Presence of nitrate or bromide
	Colourless gas giving white dense fumes with a glass rod dipped in NH_4OH	Presence of chloride
	Violet vapours	Presence of iodide
	Colourless gas giving a white deposit on a moist glass rod and oily drops at the bottom of the tube	Presence of fluoride
	No characteristic change	Absence of halides and nitrate
10.Action of conc. H_2SO_4 and MnO_2 A pinch of the substance is warmed with con. H_2SO_4 and MnO_2	Greenish yellow gas turning starch-iodide paper blue.	Presence of chloride.
	Reddish brown gas turning starch paper yellow.	Presence of bromide.
	Violet vapours turning starch paper blue.	Presence of iodide.
	No characteristic change.	Absence of chloride, bromide and iodide.

11. Test for nitrate A little of the substance is heated with a few drops of con. H_2SO_4 and cellulose (small filter paper rolled into a ball) is heated.	Reddish brown gas	Presence of nitrate is confirmed
	No reddish brown gas	Absence of nitrate
12. Ethyl Borate Test A little of the substance is warmed with a few drops of con. H_2SO_4 and ethyl alcohol. The outcoming vapours are burnt.	Green edged flame	Presence of borate is confirmed
	No green edged flame.	Absence of borate
13. Ammonium molybdate test A pinch of the substance is heated with few drops of con. HNO_3 and cooled. This solution is added to ammonium molybdate taken in another test tube.	Yellow precipitate in cold condition	Presence of phosphate
	No yellow precipitate	Absence of phosphate
14 a) Chromyl Chloride Test A pinch of the substance is heated with solid NaCl and con. H_2SO_4.	Red orange vapours.	Presence of chromate is confirmed
Red orange vapours are passed into water taken in another test tube.	Yellow solution	
To the yellow solution, acetic acid and lead acetate solution is added.	Yellow precipitate	
	No red orange vapours	Absence of chromate
b) Chromyl Chloride Test A pinch of the substance is heated with solid $K_2Cr_2O_7$ and con. H_2SO_4.	Red orange vapours.	

Red orange vapours are passed into water taken in another test tube.	Yellow solution.	Presence of chloride is confirmed.
To the yellow solution, acetic acid and lead acetate solution is added.	Yellow precipitate.	
	No red orange vapours	Absence of chloride.

Preparation of sodium carbonate extract

A pinch of the substance is mixed with thrice its weight of anhydrous sodium carbonate and 5 ml. of distilled water, and boiled for about 10 minutes. It is then cooled and centrifuged. With the centrifugate (sodium carbonate extract) the following tests are conducted.

EXPERIMENT	OBSERVATION	INFERENCE
1. Colour Colour of the extract is noted.	a) Yellow colouration	Presence of chromate.
	b) colourless	Absence of chromate.
2. Silver nitrate test a) A little of the extract is acidified with dil. HNO_3, boiled off CO_2, and $AgNO_3$ solution is added.	a) White curdy precipitate soluble in NH_4OH.	Presence of chloride
	b) Pale yellow precipitate partly soluble in NH_4OH.	Presence of bromide
	c) Yellow precipitate insoluble in NH_4OH.	Presence of iodide
	d) Black precipitate	Presence of sulphide
	e) No characteristic precipitate.	Absence of halides and sulphide.
b) To the above centrifugate from the above experiment, excess of $AgNO_3$ is added followed by NH_4OH in drops.	a) Yellow ring	Presence of phosphate.
	b) Red ring	Presence of chromate is confirmed.
	c) No characteristic coloured ring	Absence of phosphate and chromate.

3.Barium chloride test (Test for Sulphate) A little of the extract is acidified with dil. HCl, boiled off CO_2 and $BaCl_2$ solution is added.	White precipitate insoluble in con. HCl.	Presence of sulphate is confirmed
	No white precipitate.	Absence of sulphate
4. Test for Oxalate or Fluoride Calcium chloride test a) A little of the extract is acidified with acetic acid, boiled off CO_2 and $CaCl_2$ solution is added.	White precipitate	Presence of fluoride or oxalate.
	No white precipitate.	Absence of fluoride and oxalate
b) The above precipitate is dissolved in warm dil. H_2SO_4 and a drop of dil. $KMnO_4$ is added.	Pink colour is decolourised.	Presence of oxalate is confirmed
	Pink colour is not decolourised.	Presence of fluoride is confirmed
5. Test for nitrate Brown Ring Test The extract is acidified with dil. H_2SO_4 and heated with a few drops of freshly prepared $FeSO_4$ solution. To this mixture con. H_2SO_4 is added carefully along the sides without shaking the test tube.	Brown ring is formed at the junction of the liquids.	Presence of nitrate is confirmed
	No brown ring	Absence of nitrate

Elimination of Interfering Radicals

Elimination of Oxalate

The substance is roasted in a china dish for about ten minutes. The roasted residue is then dissolved in water or dil. HCl. This is the original solution.

Elimination of Borate/ Fluoride / Chromate

Borate / Fluoride / Chromate is eliminated by repeated evaporation of the substance with con. HCl in a china dish. The residue is dissolved in water/dil.HCl. This is the original solution.

Preparation of Original Solution

The original solution is prepared by dissolving the substance in water/ dil. HCl /dil. HNO_3/Con. HNO_3.(for phosphate).

Elimination of Phosphate

Phosphate has to be eliminated only after the II group separation.

* Phosphate is eliminated by the repeated addition of zirconyl chloride (or) zirconyl nitrate solution. The white precipitate of zirconyl phosphate is removed by centrifugation. The clear solution is used for III group and so on.

Analysis of Basic Radicals

Intergroup separation:

<table>
<tr><td colspan="6">To the eliminated solution dilute hydrochloric acid is added and centrifuged.</td></tr>
<tr><td rowspan="6">Residue:
Presence of I group</td><td colspan="5">Centrifugate:
H_2S is passed through the centrifugate and centrifuged.</td></tr>
<tr><td rowspan="5">Residue:
Presence of II group</td><td colspan="4">Centrifugate:
H_2S is boiled off. A few drops of con. HNO_3 are added and boiled*. NH_4Cl and excess of NH_4OH are then added and centrifuged.</td></tr>
<tr><td rowspan="4">Residue:
Presence of III group</td><td colspan="3">Centrifugate:
H_2S is passed through the centrifugate and centrifuged.</td></tr>
<tr><td rowspan="3">Residue:
Presence of IV group</td><td colspan="2">Centrifugate:
H_2S is boiled off. NH_4Cl, NH_4OH and $(NH_4)_2CO_3$ are added and centrifuged.</td></tr>
<tr><td>Residue:
Presence of V group</td><td>Centrifugate:
Concentrated the centrifugate and tested for Mg and found to be present / absent.
The original solution is tested for ammonium and found to be present / absent.</td></tr>
</table>

Analysis of Group I

The precipitate is boiled with water and centrifuged while hot.	
Residue:	**Centrifugate:**
Nil	Divided into two parts.
Absence of Mercury and Silver.	i) Acetic acid and potassium chromate solution are added – Yellow precipitate – Presence of Lead.
	ii) To the second part KI solution is added – Yellow precipitate is got. It is heated with water and cooled- Appearance of Golden spangles – Presence of Lead is confirmed.

Analysis of group II

Separation of II A and II B

The precipitate obtained in Group II is boiled with little NaOH solution.	
Residue:	**Centrifugate:**
Presence of II A metals (Hg, Pb, Bi, Cu or Cd).	Acidified with dil. HCl and passed H_2S – No precipitate – Absence of Group II B.

Analysis of group II A

<table>
<tr><td colspan="4">The Group II A precipitate is washed with water and boiled with dil. HNO_3 and centrifuged.</td></tr>
<tr><td rowspan="3">Residue:
Nil.
Absence of Hg.</td><td colspan="3">Centrifugate:
Dil. H_2SO_4 is added in excess and centrifuged.</td></tr>
<tr><td rowspan="2">Residue:
Nil-Absence of Lead</td><td colspan="2">Centrifugate:
NH_4OH is added in excess and centrifuged.</td></tr>
<tr><td>Residue:
White residue is dissolved in dil. HCl and divided into two parts.
i) To one part water is added – White turbidity – Presence of Bismuth.
ii) To another part Sodium stannite solution (NaOH + $SnCl_2$) is added – Black precipitate – Presence of Bismuth.</td><td>Centrifugate:
The centrifugate is divided into two parts.
i) To one part acetic acid and potassium ferrocyanide solution are added – Brown precipitate – Presence of Copper.
ii) To the second part, H_2S is passed – Yellow precipitate – Presence of Cadmium.</td></tr>
</table>

Analysis of group II B

<table>
<tr><td colspan="2">The group II B precipitate is boiled with con. HCl, diluted with water and centrifuged.</td></tr>
<tr><td>Residue:
Nil.
Absence of Arsenic.</td><td>Centrifugate: The centrifugate is divided into two parts.
i) To one part water is added. It is then warmed with iron filings and centrifuged.
Mercuric chloride solution is added – Silky white precipitate – Presence of Tin.
ii) The second portion is diluted with water and H_2S is passed – Red Orange precipitate – Presence of Antimony.</td></tr>
</table>

Analysis of group III

The third group precipitate is boiled with water and hydrogen peroxide. It is diluted with water and centrifuged.

Residue:

It is divided into two parts.

1. One portion is dissolved in HCl and the following tests are done.

i) To one part Potassium ferrocyanide solution is added – Blue precipitate – Presence of Iron.

ii) To another part Ammonium sulphocyanide solution is added – Blood red colouration – Presence of Iron is confirmed.

iii) Potassium ferrocyanide solution is added to the original solution – Blue precipitate – Presence of Ferric.

iv) Potassium ferricyanide solution is added to the original solution – Blue precipitate – Presence of Ferrous.

2. The other part of the residue is boiled with con. HNO_3 and lead dioxide diluted with water and allowed to stand – Pink colour – Presence of Manganese

Centrifugate:

The centrifugate is divided into two parts.

i) To one part acetic acid and lead acetate solutions are added – Yellow precipitate – Presence of Chromium.

ii) The second portion is acidified with dil.HCl and NH_4OH is added in excess and allowed to stand for a few minutes – White gelatinous precipitate – Presence of Aluminium.

Analysis of group IV

<table>
<tr><td colspan="3">The group IV precipitate is washed with water and shaken with very dil. HCl and centrifuged.</td></tr>
<tr><td>Residue:
Black – Presence of Co or Ni.
It is dissolved in aqua regia and evaporated to dryness in a china dish. The residue is dissolved in water and divided into 2 parts.
i) To one part, NH_4Cl, NH_4OH and potassium ferricyanide solutions are added – Reddish brown precipitate – Presence of Cobalt.
ii) To the otherpart, dimethyl glyoxime and NH_4OH are added – Rose red precipitate – Presence of Nickel is confirmed.</td><td colspan="2">Centrifugate:
H_2S is boiled off. NaOH is added in excess and centrifuged.</td></tr>
<tr><td></td><td>Residue:
White turning brown.
The residue is boiled with con. HNO_3 and PbO_2 diluted with water and kept for sometime – Pink colour – Presence of Manganese is confirmed.</td><td>Centrifugate:
It is divided into two parts.
i) H_2S is passed through one portion – White precipitate – Presence of Zinc.
ii) The second part is acidified with acetic acid and potassium ferrocyanide is added – White precipitate – Presence of Zinc is confirmed</td></tr>
</table>

Analysis of group V

<table>
<tr><td colspan="3">The V group precipitate is dissolved in warm acetic acid and K_2CrO_4 solution is added.</td></tr>
<tr><td>Residue:
Yellow – Presence of Barium.
Flame test is conducted – Apple green colour – Presence of Barium</td><td colspan="2">Centrifugate:
To the centrifugate dil. H_2SO_4 is added</td></tr>
<tr><td></td><td>Residue:
White precipitate – Presence of Strontium.</td><td>Centrifugate:
NH_4OH and ammonium oxalate solution are added – White precipitate – Presence of Calcium</td></tr>
</table>

Analysis of group VI

1.	The centrifugate from group V is concentrated and added 2 drops of NH_4OH and an excess of disodium hydrogen phosphate (Na_2HPO_4) solution, shaken and the sides of the tube are scratched with a glass rod – White crystalline precipitate – Presence of Magnesium is confirmed.
2.	To the aqueous solution, NaOH is added and warmed - Pungent smelling gas turning red litmus blue – Presence of Ammonium.
3.	To the aqueous solution, 2 drops of NaOH solution and an excess of Nessler's Reagent are added - Reddish brown precipitate - Presence of Ammonium is confirmed.

Report:

The given mixture contains

i) ---------- and ---------- as anions.

ii) ---------- and ---------- as cations.

Spot Test for Cations

1. **Bismuth:** To the salt solution added few drops of dil.HNO_3 and a pinch of thiourea- yellow colouration.

2. **Copper:** To the salt solution NH_4OH is added drop by drop in excess- pale blue precipitate dissolves in excess giving blue solution.

3. **Manganese:** A little of the substance is dissolved in dil.HNO_3 and a pinch of sodium bismuthate is added- pink colour.

4. **Nickel:** To the salt solution NH_4Cl , NH_4OH and dimethyl glyoxime are added – Rose red precipitate.

Table 1 List of interfering and simple acid radicals

Simple acid Radicals	Interfering acid radicals
Carbonate, Chloride, Bromide, Iodide, Nitrate, Sulphide, Sulphate	Fluoride, Oxalate, Borate, Chromate, Phosphate

Table 2 List of basic radicals

Group	Basic Radicals	Group Reagent	Intergroup separation Residue	Colour of the residue
I	Pb, Ag, Hg	HCl	$PbCl_2$	White
IIA	Cd, Cu, Bi	HCl +H_2S	CdS	Yellow
			CuS	Black
			Bi_2S_3	Brown
IIB	Sb	H_2S	Sb_2S_3	Red orange
III	Fe, Al, Cr	NH_4Cl and NH_4OH	Hydroxides	–
IV	Co, Ni, Mn, Zn	NH_4Cl, NH_4OH + H_2S	NiS	Black
			MnS	Pink or flesh
			ZnS	White
V	Ba, Sr, Ca	NH_4Cl, NH_4OH and $(NH_4)_2CO_3$	$BaCO_3$ $SrCO_3$ $CaCO_3$	White White White

Report:

Mix. No	Date	Acid Radicals		Basic radicals		Mark	Sign
		Simple	Interfering				
1.							
2.							
3.							
4.							
5.							
6.							

Mix. No	Date	Acid Radicals		Basic radicals		Mark	Sign
		Simple	Interfering				
7.							
8.							
9.							
10.							
11.							
12.							

PHYSICAL CHEMISTRY

PHYSICAL CHEMISTRY EXPERIMENTS

Subject Code: 18UCHCR3

CONTENTS

EXPERIMENTS

Table 3.1.1 Determination of C.S.T

S.No	Volume of phenol (mL)	Volume of Water (mL)	% of phenol	Miscibility Temperature (°C)

Ex.No:1

Date:

CRITICAL SOLUTION TEMPERATURE

Aim:

To determine (i) The critical solution temperature (C.S.T) of phenol-water system (ii) The strength of the given NaCl solution.

Principle:

Phenol is partially miscible with water at ordinary temperature. When mixed, they form two different layers. However the mutual solubility of the two liquids increases on heating and become completely miscible at a particular temperature called miscibility temperature. Above this temperature, the two liquids of a definite composition exist in a single layer. On cooling, the liquids separate into two layers below the miscibility temperature. Mixture of phenol and water of varying composition are taken and their miscibility temperatures are determined. When the miscibility temperatures are plotted against the composition of the liquid pair, a parabola shaped curve is got. The maximum temperature on the curve obtained is the critical solution temperature. Above this temperature, phenol and water are completely miscible at all proportions.

Whenever an electrolyte like NaCl is added to phenol- water system, the mutual solubility decreases and hence the miscibility temperature increases. The miscibility temperature varies linearly with the concentration of NaCl solution. The given NaCl solution is mixed with phenol and the miscibility temperature is determined. The strength of the given NaCl solution is then found out using the graph.

Procedure

I) Determination of C.S.T

Exactly 5mL of freshly distilled phenol is added in a boiling tube provided with a sensitive thermometer and a stirrer. 3mL of distilled water is added to phenol in the boiling tube from a burette. The tube is heated in a water bath with constant stirring of

Table 3.1.2 Determination of strength of NaCl

S.No	Strength of NaCl (M)	Volume of NaCl (mL)	Volume of phenol (mL)	Miscibility Temperature (°C)

the mixture. When the two liquids become completely miscible to form a clear solution, the tube is taken out of the water bath and allowed to cool slowly by placing in a conical flask (air jacket). The liquid mixture is stirred smoothly and cooling is continued till turbidity appears. The temperature at which the turbidity just appears is noted. This is the miscibility temperature of phenol-water mixture of a given composition.

The experiment is repeated by adding water in part of 1mL at a time (a total of 12 additions) and the miscibility temperature is recorded after each addition as described before. The miscibility temperatures are plotted against the volume percentage of phenol. The maximum temperature on the curve is taken as the critical solution temperature.

ii) Determination of the Strength of NaCl Solution

250mL of 0.1M stock solution of NaCl is prepared and diluted to various concentrations such as 0.08M, 0.06M, 0.04M, 0.02M in different 100mL standard flasks. 3mL of each solution is mixed separately with 3mL of phenol and the miscibility temperatures are determined. A plot of miscibility temperatures Vs strength of NaCl solution gives a straight line. Now, 3mL of the given NaCl solution is mixed with 3mL of phenol and the miscibility temperature is determined. The strength of the given NaCl solution is found out from the graph.

Result

i) CST of phenol-water system =

ii) Strength of given NaCl solution =

Table 3.2.1 Determination of molecular weight

Salt hydrate (x)		Salt hydrate (x) + Solute (y)		Salt hydrate (x) + Solute (z)	
Time sec	Temperature °C	Temperature, °C for 0.2 g	Temperature, °C for 0.4 g	Temperature, °C for 0.2 g	Temperature, °C for 0.4 g

Ex.No: 2

Date:

TRANSITION TEMPERATURE METHOD DETERMINATION OF MOLECULAR WEIGHT

Aim

To determine (i) the transition temperature of the given solvent(ii) the molal depression constant of the solvent and (iii) the molecular weight of the given substance.

Principle

Salt hydrates on heating lose their water of crystallization at a particular temperature and change into their less hydrated form. The reverse change takes place on cooling and the lower hydrate is transformed into a higher hydrate. The temperature at which a salt hydrate changes from one form to another form is called the transition temperature or transition point. At this point, the temperature remains constant for some time.

When a hot solution of the salt hydrate is cooled in air in an undisturbed manner, super cooling occurs below the transition point. Addition of a small crystal of the hydrate (seeding) to the super cooled solution causes a sudden rise in temperature. It remains constant for sometime and then decreases. This constant temperature is taken as the transition point of the salt hydrate.

When a non-volatile solute is added to a salt hydrate (solvent), a decrease in transition temperature is observed .The depression in transition temperature of the salt hydrate is related to the molecular weight of the solute by the formula,

$$\Delta T_{tr} = \frac{K_{tr} \times W_2 \times 1000}{W_1 \times M_2}$$

where, ΔT_{tr} = Depression of transition point

K_{tr} = Molal depression constant of the solvent (salt hydrate)

W_1 = Weight of the solvent

M_2 = Molecular weight of the solute

K_{tr} is first determined by using a solute of known molecular weight.

The experiment is then repeated with the given substance and its molecular weight is determined using the calculated value of K_{tr}

Procedure

1) Determination of K_{tr}

About 4g of the salt hydrate (x) is weighed accurately and taken in a dry boiling tube fitted with a sensitive thermometer and a stirrer. The tube is heated in a water bath until the solid is melted and the temperature of the melt is raised by about 5°C. The tube is then taken out of the water bath and cooled slowly by placing in a conical flask (air jacket). The temperature of the melt is recorded for every 30 seconds. At one point, the temperature remains constant for some time. Cooling is continued further for about 5-6°C. Now, a small crystal of the salt hydrate is added and the liquid is stirred well. The temperature raises suddenly, remains constant for some time and then decreases. This constant temperature recorded from a plot of time vs temperature, is the transition temperature (T_0).

Now, about 0.2g of the solute (y) of known molecular weight is accurately weighed and added to the salt hydrate in the boiling tube. The transition temperature (T) of this mixture is determined as described before. Again 0.2g of the solute is added and transition temperature is determined as before. The difference between T_0 and T gives ΔT_{tr}. From this, K_{tr} is calculated.

2) Determination of M_2

A fresh quantity of about 4g of the salt hydrate and 0.2g of the given substance (z) are weighed accurately and taken in a dry boiling tube fitted with a sensitive thermometer and a stirrer. The transition temperature (T) of this mixture is determined as before. Again 0.2g of the solute is added and transition temperature is determined as before. $T_0 - T_1$ gives ΔT_{tr} . Using the values of ΔT_{tr} and K_{tr} the molecular weight of the given substance is calculated.

Result

1) Molal depression constant of the solvent (K_{tr}) =
2) The molecular weight of the given substance =

Table 3.3.1 Determination of K_a

S.No	Time min	Volume of NaOH mL	$(V_\infty - V_0)$	log $(V_\infty - V_t)$	K_a min^{-1}

$(V_\infty - V_0)$ = = mL

$$K_a = 2.303/t \times \log(V_\infty - V_0) / (V_\infty - V_t)$$

Model Calculation

K_a =

K_a Average =

Ex.No: 3

Date:

KINETICS OF HYDROLYSIS OF METHYL ACETATE

Aim

To study the kinetics of acid catalyzed hydrolysis of methyl acetate and to compare the strengths of the given acids A and B.

Principle

Methyl acetate is hydrolysed by water in presence of mineral acids

$$CH_3COOCH_3 + H_2O \xrightarrow{H^+} CH_3COOH + CH_3OH$$

Since water is present in large excess, the rate of the reaction depends only on the concentration of the ester and the first order kinetics is followed.

$$K = \frac{2.303}{t} \log \frac{a}{a-x}$$

where,

a = initial concentration of the ester.

$(a-x)$ = concentration of ester at any time t minutes.

The reaction can be followed from the rate of disappearance of the ester or the rate of formation of acetic acid at various time intervals by titrating the reaction mixture against standard NaOH solution. Let V_0 ,V_t and V_∞ be the volume of NaOH consumed at the start, after a time interval of t minutes and at the completion of reaction respectively. Now,

a = Initial concentration of ester

= Total amount of acetic acid formed

= $V_\infty - V_0$

$(a-x)$ = Concentration of ester at any time t.

= Amount of acetic acid formed at any time t.

= $V_\infty - V_t$

Table 3.3.2 Determination of K_b

S.No	Time (min)	Volume of NaOH mL	$(V_\infty - V_0)$	log $(V_\infty - V_t)$	K_b min^{-1}

$(V_\infty - V_0)$ = = mL

$K_b = 2.303/t \times \log(V_\infty - V_0) / (V_\infty - V_t)$

K_b Average =

Inserting these values into the first order rate equation

$$K = \frac{2.303}{t} \log [V_\infty - V_0 / V_\infty - V_t]$$

The *k* values are calculated at different time intervals are nearly a constant.

A plot of log $(V_\infty - V_t)$ against time is a straight line of slope equal to k/2.303.

$\therefore$ k = slope $\times$ 2.303

If K_a and K_b are the rate constants for the reaction catalysed by acid A and B respectively, the relative strength of the acid A to B = k_a / k_b

Procedure:

The bottles containing the given acid (A) and Methyl acetate are kept in a trough of water for 15 minutes to attain thermal equilibrium

Exactly 100mL of the acid A is taken in a 250mL reaction bottle and exactly 5mL of the ester is added to it using a pipette. A stop watch is started when the pipette is half-emptied. The bottle is stopped, shaken well and immediately 10mL of the solution is pipetted into a clean conical flask containing ice cold water. It is then titrated against NaOH solution using phenolphthalein indictor. The end point is the appearance of a pale permanent pink colour. The titre value gives V_0.

Similarly, 10mL of the reaction mixture is withdrawn at regular time intervals of 5, 15,25,35,50 and 65 minutes and is titrated against NaOH solution. The titre value gives V_t.

The reaction is brought to completion by keeping the reaction mixture in a hot water bath at 60°C for about 30 minutes and then cooled. Now, 10mL of the reaction mixture is titrated against NaOH solution as before. The titre value of V$\propto$

The experiment is repeated with the given acid (B) and the rate constants K_a and K_b are calculated. The ratio K_a / K_b gives the relative strength of acid A and B.

Result:

(i) Relative strength of the two acids from calculation k_a / k_b =

(ii) Relative strength of the two acids from graph k_a / k_b =

Calculation of strength of oxalic acid

Weight of Oxalic acid in 200mL =

Strength of Oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}} = \frac{\quad \times 5}{63} = \_\_\_\_\_ N$

Table 3.4.1 Standard $H_2C_2O_4$ Vs. Link NaOH

Volume of Oxalic acid = 40 mL

Volume of NaOH, mL	Conductivity, mho

To find the strength of NaOH

Volume of oxalic acid (V_1) = 40mL

Strength of oxalic acid (N_1) =

Volume of NaOH (V_2) =

Strength of NaOH (N_2) =

Ex.No: 4

Date:

CONDUCTOMETRIC ACID BASE TITRATION

Aim

To determine the strength of given hydrochloric acid using approximately decinormal solution of NaOH solution conductometrically. You are provided with analar crystals of oxalic acid.

Principle

Since oxalic acid is a dibasic acid, it gives two neutralization points. One will correspond to the half neutralization point. The second neutralization point will correspond to the replacement of the second acidic hydrogen atom.

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

When HCl is titrated against NaOH, the fast moving H^+ ions are replaced by slow moving Na^+ ions. As a result the conductance of the solution decreases.

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

This decrease in conductance will take place until the end point is reached. Further addition of NaOH beyond the end point raises the conductance sharply owing to the introduction of fast moving OH^- ions. When the values of conductance measured are plotted against the volume of NaOH added, a 'V' shaped graph is obtained. The point of intersection of the two lines gives the end point.

Preparation of standard solution

About 0.126g of oxalic acid is weighed accurately and dissolved in distilled water and made upto 200mL in a SMF. The solution is shaken well for uniform concentration.

Table 3.4.2 Link NaOH Vs given HCl

Volume of HCl = 40 mL

Volume of NaOH, mL	Conductivity, mho

To find the strength of HCl

Volume of NaOH (V_1) =

Strength of NaOH (N_1) =

Volume of HCl (V_2) =

Strength of HCl (N_2) =

Procedure

Standardisation of NaOH (Link)

Exactly 40mL of the std. $H_2C_2O_4$ solution is taken in a 100mL beaker. A glass rod and the conductivity cell are introduced into the solution. The conductivity cell is connected to the conductivity bridge. 0.4mL of NaOH is added in small portions and the conductance is measured in each addition. The process of adding NaOH and measuring the conductance is obtained until atleast five readings are taken beyond the end point.

Estimation of HCl

The given HCl solution is made upto 100mL in a 100mL SMF. 40mL of this made up solution is taken in a beaker. The experiment is repeated as above.

Two sets of graphs are drawn (std. $H_2C_2O_4$ Vs. link NaOH and std NaOH Vs given HCl) by plotting conductance against volume of NaOH. The point of intersection gives the end point. From the volume of NaOH, the strength of HCl is calculated.

Result:

Strength of given HCl solution =

Calculation of strength of $MgSO_4$

Weight of $MgSO_4$ in 200mL =

Strength of $MgSO_4$ $= \dfrac{\text{Weight / Litre}}{\text{Equivalent weight}} = \dfrac{\quad \times 5}{123.24} =$ _____N

Table 3.5.1 Standard $MgSO_4$ Vs. Link $BaCl_2$

Volume of $MgSO_4$ = 40 mL

Volume of $BaCl_2$, mL	Conductivity, mho

To find the strength of $BaCl_2$

Volume of $MgSO_4$ (V_1) = 40mL

Strength of $MgSO_4$ (N_1) =

Volume of $BaCl_2$ (V_2) =

Strength of $BaCl_2$ (N_2) =

Ex.No: 5

Date:

CONDUCTOMETRIC PRECIPITATION TITRATION

Aim

To evaluate the strength of given magnesium sulphate by conductometric precipitation titration using approximately decinormal solution of barium chloride solution. You are provided with analar crystals of magnesium sulphate.

Principle

When $BaCl_2$ is added to $MgSO_4$ solution, $BaSO_4$ get precipitated.

$$BaCl_2 + MgSO_4 \rightarrow BaSO_4 + MgCl_2$$

The net effect of the reaction begins the replacement of Ba^{2+} ions by Mg^{2+} ions and the conductivity remains more or less constant or decrease very slightly and after the end point it increases sharply.

Preparation of standard solution

About 0.246 g of $MgSO_4$ is weighed accurately and dissolved in distilled water and made upto 200mL in a SMF.

Procedure

Standardisation of $BaCl_2$

Exactly 40mL of the std. $MgSO_4$ solution is taken in a 100mL beaker. A glass rod and the conductivity cell are introduced into the solution. The conductivity cell is connected to the conductivity bridge. 0.4mL of $BaCl_2$ is added in small portions and the conductance is measured in each addition. The process of adding $BaCl_2$ and measuring the conductance is obtained until atleast five readings are taken beyond the end point.

Table 3.5.2 Standard $BaCl_2$ Vs. given $MgSO_4$

Volume of $MgSO_4$ = 40 mL

Volume of $BaCl_2$, mL	Conductivity, mho

To find the strength of $MgSO_4$

Volume of $BaCl_2$ (V_1) =

Strength of $BaCl_2$ (N_1) =

Volume of $MgSO_4$ (V_2) = 40mL

Strength of $MgSO_4$ (N_2) =

Estimation of MgSO4

The given $MgSO_4$ is made upto 100mL in a SMF. 40mL of the made up solution is taken in a beaker. The experiment is repeated as above.

Two sets of graphs are drawn (std. $MgSO_4$ Vs. link $BaCl_2$ and std $BaCl_2$ Vs. given $MgSO_4$) by plotting conductance against volume of $BaCl_2$. The point of intersection gives the end point. From the volume of $BaCl_2$ the strength of $MgSO_4$ is calculated.

Result

The strength of given $MgSO_4$ =

Calculation of strength of FAS

Weight of FAS in 100mL =

$$\text{Strength of FAS} = \frac{\text{Weight / Litre}}{\text{Equivalent weight}} = \frac{\times 10}{392} = \_\_\_\_\_ \text{N}$$

Table 3.6.1 Std. FAS Vs. Link $KMnO_4$

Trial titration

S. No	Volume of $KMnO_4$ (mL)	emf (Volt)

Fair titration

S. No	Volume of $KMnO_4$ (mL)	emf (Volt)	$\frac{V_1 + V_2}{2}$	ΔE	ΔV	$\Delta E / \Delta V$

To find the strength of $BaCl_2$

Volume of FAS (V_1) = 20mL

Strength of FAS (N_1) =

Volume of $KMnO_4$ (V_2) =

Strength of $KMnO_4$ (N_2) =

Ex.No: 6

Date:

POTENTIOMETRIC TITRATION I

Aim

To determine the strength of the given Fe^{2+} solution by titrating it against link KMnO4 potentiometrically. Analar crystals of FAS are provided.

Principle

When a FAS solution is titrated with link $KMnO_4$ in the presence of mineral acid, Fe^{2+} is oxidized to Fe^{3+}.If a platinum wire is dipped into the titration mixture a redox electrode Fe^{2+}/Fe^{3+}, Pt is setup. The potential of this electrode which varies inversely with Fe^{2+}/Fe^{3+} ratio can be measured by coupling it with a saturated calomel electrode.

$$SCE // KCl // Fe^{2+}, Fe^{3+} / Pt$$

When a ferrous salt is titrated with $KMnO_4$ in the presence of mineral acid, Fe^{2+} is oxidized to Fe^{3+}. Addition of $KMnO_4$ converts Fe^{2+} to Fe^{3+} and the ratio of Fe^{2+}/Fe^{3+} decreases. Consequently the observed emf of the cell gradually increases. At the end point there will be sharp jump in emf due to sudden removal of all Fe^{2+} ions. A plot of the $\Delta E/\Delta V$ against volume of $KMnO_4$ added is drawn and the end point is determined graphically.

Preparation of standard FAS solution

About 3.92g of FAS is weighed accurately and 2 drops of conc. H_2SO_4 are added and made upto 100mL in a SMF.

Procedure

Standardisation of $KMnO_4$ (link)

Exactly 20mL of the std. FAS solution is pipetted out into 100mL beaker. About 20mL of dil. H_2SO_4 acid is added and platinum electrode and calomel electrodes are dipped

Table 3.6.2 Std. $KMnO_4$ Vs. given FAS

Trial titration

S. No	Volume of $KMnO_4$ (mL)	emf (Volt)

Fair titration

S. No	Volume of $KMnO_4$ (mL)	emf (Volt)	$\frac{V_1 + V_2}{2}$	ΔE	ΔV	$\Delta E / \Delta V$

To find the strength of FAS

Volume of $KMnO_4$ (V_1) =

Strength of $KMnO_4$ (N_1) =

Volume of FAS (V_2) = 20mL

Strength of FAS (N_2) =

into the solution. The platinum and calomel electrodes are connected respectively to the positive and negative terminals of the potentiometer. To start with 10mL of $KMnO_4$ is added from the burette. The solution is stirred and the emf is measured after each addition. The addition is continued in portions of 2mL at a time and emf is measured after each addition. The volume of $KMnO_4$ added is noted when there is a sharp jump in emf. This trial titration helps to fix the volume range at which the end point lies.

Now a second titration is carried out with a fresh mixture of exactly 20mL of FAS and about an equal volume of dil. H_2SO_4 acid. $KMnO_4$ is added from the burette in a bulk upto a volume 1mL short of that required for a jump in emf in the previous rough titration. The titration is continued by adding $KMnO_4$ in portions of 0.2mL at a time and the emf is measured after each addition. This process is carried out until atleast 5 readings are taken beyond the end point.

Estimation of FAS

The given solution is made upto 100mL in a SMF. 20mL of the made up solution is taken in a beaker and the experiment is repeated as above.

Two sets of graphs are drawn (Std FAS vs link $KMnO_4$ and std $KMnO_4$ vs Given Fe^{2+}) by plotting $\Delta E / \Delta V$ against $(V_1 + V_2) / 2$. The peak of the curve gives the end point. From the volume of $KMnO_4$ the strength of FAS is calculated.

Result

The strength of given FAS solution =

Calculation of strength of $K_2Cr_2O_7$

Weight of $K_2Cr_2O_7$ in 200mL=

$$\text{Strength of } K_2Cr_2O_7 = \frac{\text{Weight / Litre}}{\text{Equivalent weight}} = \frac{\quad \times 5}{49} = \_\_\_\_\_ N$$

Table 3.7.1 Std. $K_2Cr_2O_7$ Vs. FAS

Trial titration

S. No	Volume of $K_2Cr_2O_7$ (mL)	emf (Volt)

Fair titration

S. No	Volume of $K_2Cr_2O_7$ (mL)	emf (Volt)	$\frac{V_1 + V_2}{2}$	ΔE	ΔV	$\Delta E / \Delta V$

To find the strength of FAS

Volume of $K_2Cr_2O_7$ (V_1) =

Strength of $K_2Cr_2O_7$ (N_1) =

Volume of FAS (V_2) = 20mL

Strength of FAS (N_2) =

Ex.No: 7

Date:

POTENTIOMETRIC TITRATION II

Aim

To determine the strength of the given $KMnO_4$ solution by titrating it against link FAS potentiometrically. Analar crystals of $K_2Cr_2O_7$ are provided.

Principle

When a FAS solution in the presence of mineral acid is titrated with $K_2Cr_2O_7$ or $KMnO_4$ solution, Fe^{2+} is oxidized to Fe^{3+}. If a platinum wire is dipped into the titration mixture a redox electrode Fe^{2+}/Fe^{3+}, Pt is setup. The potential of this electrode which varies inversely with Fe^{2+}/Fe^{3+} ratio can be measured by coupling it with a saturated calomel electrode.

$$SCE /\!/ KCl /\!/ Fe^{2+}, Fe^{3+} / Pt$$

When a ferrous salt is titrated with $K_2Cr_2O_7$ or $KMnO_4$ in the presence of mineral acid, Fe^{2+} is oxidized to Fe^{3+}. Addition of $K_2Cr_2O_7$ or $KMnO_4$ converts Fe^{2+} to Fe^{3+} and the ratio of Fe^{2+}/Fe^{3+} decreases. Consequently the observed emf of the cell gradually increases. At the end point there will be sharp jump in emf due to sudden removal of all Fe^{2+} ions. A plot of the $\Delta E/\Delta V$ against volume of titrant is drawn and the end point is determined graphically.

Preparation of standard $K_2Cr_2O_7$ solution

About 0.98g of $K_2Cr_2O_7$ is weighed accurately, dissolved in distilled water and made upto 200mL in a SMF.

Table 3.7.2 Std. FAS Vs. given $KMnO_4$

Trial titration

S. No	Volume of $KMnO_4$ (mL)	emf (Volt)

Fair titration

S. No	Volume of $KMnO_4$ (mL)	emf (Volt)	$\frac{V_1 + V_2}{2}$	ΔE	ΔV	$\Delta E / \Delta V$

To find the strength of FAS

Volume of FAS (V_1) = 20mL

Strength of FAS (N_1) =

Volume of $KMnO_4$ (V_2) =

Strength of $KMnO_4$ (N_2) =

Procedure

Standardisation of link FAS

Exactly 20mL of the link FAS solution is pipetted out into 100mL beaker. About 20mL of dil. H_2SO_4 acid is added and platinum electrode and calomel electrodes are dipped into the solution. The platinum and calomel electrodes are connected respectively to the positive and negative terminals of the potentiometer. To start with 10mL of $K_2Cr_2O_7$ is added from the burette. The solution is stirred and the emf is measured after each addition. The addition is continued in portions of 2mL at a time and emf is measured after each addition.

The volume of $K_2Cr_2O_7$ added is noted when there is a sharp jump in emf. This trial titration helps to fix the volume range at which the end point lies.

Now a second titration is carried out with a fresh mixture of exactly 20mL of FAS and about an equal volume of dil.H_2SO_4. $K_2Cr_2O_7$ is added from the burette in a bulk upto a volume 1mL short of that required for a jump in emf in the previous rough titration. The titration is continued by adding $K_2Cr_2O_7$ in portions of 0.2mL at a time and the emf is measured after each addition. This process is carried out until atleast 5 readings are taken beyond the end point.

Estimation of $KMnO_4$

The given $KMnO_4$ is made upto 100mL in a SMF. 20mL of the made up solution is taken in a beaker and the experiment is repeated as above.

Two sets of graphs are drawn(Std $K_2Cr_2O_7$ vs link FAS and std FAS vs Given $KMnO_4$) by plotting $\Delta E/\Delta V$ against $(V_1+ V_2) / 2$. The peak of the curve gives the end point. From the volume of $KMnO_4$ the strength of $KMnO_4$ is calculated.

Result

The strength of given $KMnO_4$ solution =

Table 3.8.1 Determination of molecular weight

Solvent (x)		Solvent (x) + Solute (y)		Solvent (x) + Solute (z)	
Time sec	Temperature °C	Temperature, °C for 0.2 g	Temperature, °C for 0.4 g	Temperature, °C for 0.2 g	Temperature, °C for 0.4 g

Ex.No: 8

Date:

DETERMINATION OF MOLECULAR WEIGHT - RAST'S METHOD

Aim

To determine (i) the molal depression constant of the given solvent and (ii) the molecular weight of the given substance by Rast's macro method.

Principle

Whena non-volatile solute is added to a solvent, the freezing point of the solvent is lowered. The depression in the freezing point of the solvent is related to the molecular weight of the solute by the expression

$$\Delta T_f = \frac{k_f \times w_2 \times 1000}{w_1 \times M_2}$$

where, ΔT_f = Depression of freezing point

K_f = molal depression constant of the solvent

W_1 = weight of the solvent

W_2 = weight of the solute

M_2 = molecular weight of the solute

In the first part of the experiment, k_f is determined by using a solute of known molecular weight. Knowing the value of k_f of the solvent, the experiment is repeated with the given substance and its molecular weight is determined.

Procedure

1) Determination of k_f

About 4g of the solvent (x) is weighed accurately and taken in a dry boiling tube provided with a sensitive thermometer and a stirrer. The tube is heated in a water bath until the solid is melted and the temperature of melt is raised by about 5°C. The tube is then taken

out of the water bath and cooled slowly by introducing into a conical flask (air jacket). The temperature at which turbidity appears is noted. It gives the freezing point (T_0) of the solvent. The experiment is repeated to get concordant value.

Now, about 0.2g of the solute (y) of known molecular weight is accurately weighed and added to the solvent in the boiling tube. The freezing point (T) of the solution is determined as before. Again 0.2g of the solute is added and freezing point is determined as before. The difference between T_0 and T gives ΔT_f. From this, K_f is calculated.

2) Determination of M_2

A fresh portion of about 4g of the solvent and 0.2g of the given substance (z) are weighed accurately and taken in a dry boiling tube provided with a sensitive thermometer and a stirrer. The freezing point (T) of this mixture is determined as before. Again 0.2g of the solute is added and freezing point is determined as before. $T_0 - T$ gives ΔT_f. Using the values of ΔT_f, the molecular weight of the given substance is calculated.

Result

(i) Molal depression constant of the solvent K_f =

(ii) The molecular weight of the given substance =

Table 3.9.1 Determination of Freezing Point

S.No	Weight of the substance A (g)	Weight of the substance B (g)	Composition of B%	Freezing Point (°C)

Ex.No: 9

Date:

PHASE DIAGRAM (SIMPLE EUTECTIC/COMPOUND FORMATION)

Aim

To construct the phase diagram for the given two components system and to determine the eutectic temperature and composition.

Principle

If the components of a binary mixture are chemically dissimilar, each lowers the freezing point of the other suppose addition of B lowers the freezing point of A along AC and A lowers the freezing point of B along BC. At C, the binary mixture gets solidified. The temperature at which the solid mixture crystallizes out is called eutectic temperature, T_e. The composition of the mixture at Te is referred to as eutectic composition, X_e.

Procedure

Exactly 5g of the substance A is taken in a boiling tube provided with a sensitive thermometer and a stirrer. The tube is heated in a water bath until the solid is completely melted and the temperature of the melt is raised by 5°C. The boiling tube is then taken out of the bath, introduced into a conical flask (air jacket) and allowed to cool slowly with constant stirring.

The temperature at which turbidity appears is noted. This is taken as the freezing point of A. Now, substance B is added in portions of 0.5g at a time to A in the boiling tube and after each addition, the freezing point of the mixture is determined as described before.

The experiment is repeated with 5g of substance B and then adding portions of 0.5g of A at a time.

Table 3.9.2 Determination of Freezing Point

S.No	Weight of the substance B (g)	Weight of the substance A (g)	Composition of B%	Freezing Point (°C)

The freezing points are plotted against percentage composition of the mixture. From the graph, the eutectic temperature and composition are recorded.

Result

1) Freezing point of A =
2) Freezing point of B =
3) Eutectic temperature T_e =
4) Eutectic composition X_e =

Table 3.10.1 Determination of saturation point

Molecular Weight of A =

Volume of the Solvent	No. of moles of the Solvent	Temperature		Mole fraction of the Solute N_2	$2 + \log N_2$	$1/T \times 10^{-3}/K$
		°C	K			

Ex.No: 10

Date:

DETERMINATION OF HEAT OF SOLUTION

Aim

To find out the heat of solution of a substance in water by thermometric method.

Principle

In a binary solution the component present in large amount is called the solvent and that present in small amount is called solute. It is known that at saturation point, the solute is in equilibrium with the solution. By finding out the saturation point for different given compositions of the solution, the solution enthalpy can be easily estimated. This method is simpler than the conventional method of saturation solubility by analysis.

Procedure

The solubility of the solute A in water is first quantitatively found out. If the solute is readily soluble then 2g of the solute is taken. Now the amount of water VmL that will dissolve completely 5g of solute at 60°C - 65°C is found out roughly.

After the trial experiment, 5g of the solute is accurately weighed into the freezing tube, and the known amount of VmL of water is pipetted out. The tube is then placed in a water bath, whose temperature is gradually increased till the solid state is completely dissolved in the solvent. The solvent is then removed from the water bath and placed in a conical flask (250mL) air jacket and cooled slowly with uniform stirring.The temperature at which the first crystals just appear is noted. This is the saturation temperature for that composition. Now 0.5mL of the solvent is pipetted out into the solution in the freezing tube and the saturation point is found out as before. This procedure is repeated several times with the same solvent addition become the variation of saturation solution with the temperature is given by

$$\text{Log } N_2 = \frac{-\Delta H}{2.303RT} + C$$

Table 3.10.2 Determination of saturation point

Molecular Weight of B =

Volume of the Solvent	No. of moles of the Solvent	Temperature		Mole fraction of the Solute N_2	2 +log N_2	1/T × 10^{-3}/K
		°C	K			

A plot of log N_2 Vs. 1/T is made and the heat of solution in $KJmol^{-1}$ can be calculated from the slope. The above experiment is done for solute B and the saturation point is calculated.

Result

i) The heat of solution of the solute A in water, ΔH =

ii) The heat of solution of the solute B in water, ΔH =

Table 3.11.1 Determination of C_i

Bottle No.	Volume of oxalic acid (mL)	Volume of water (mL)	Burette reading	C_i (N)

$$C_i = \frac{\text{Volume of oxalic acid} \times \text{Normality of oxalic acid}}{100}$$

Ex.No: 11

Date:

FREUNDLICH ADSORPTION ISOTHERM

Aim

To study the adsorption isotherm of the given acid on activated charcoal.

Principle

Solids especially in porous and finely divided state have the property of holding foreign molecules on their surface. This phenomenon is known as adsorption. The relationship between the mass of the equilibrium concentration of the absorbate at the constant temperature is called the adsorption isotherm. Freundlich isotherm relates the ratio of the mass of the absorbate adsorbed x to that of the adsorbent m (i.e) x/m to the equilibrium concentration of the absorbate in solution,

$$x / m = KC^{1/n}$$

where 'k' and 'n' are empirical constants.

Procedure

A standard solution of oxalic acid (0.2N) and a solution of sodium hydroxide (0.04N) are prepared. The sodium hydroxide solution is standardized using standard oxalic acid solution.

Five corning bottles (250 mL) are cleaned with chromic acid washed and dried. They are labeled from 1 to 5. Exactly 2g of activated charcoal is weighed out into each of the five corning bottles. 20, 25, 30, 40 and 50 mL of the stock oxalic acid solutions are run down from the burette into bottles 1 to 5 and the total volume is adjusted to 100 mL in all the bottles with water using burette. The bottles are kept in the mechanical shaker for about 20 minutes. After the adsorption equilibrium is estimated, the solutions in the bottles are filtered through filter paper. The first few mL of the filterate is rejected. The

Table 3.11.2

Bottle No.	C_i (N)	C_{eq} (N)	C_{ad} (N)	x	x/m	Log x/m	log C_{eq} (N)

concentration of the acid in different bottles are estimated by pipetting out 10 mL from bottles 1 to 3 and 20 mL from bottles 4 and 5 into conical flask and titrating against the standard sodium hydroxide solution.

From the titre values the equilibrium concentration of oxalic acid [ox_{eq}] can be calculated.

$$\log x/m = \log k + 1/n \log [ox_{eq}]$$

Thus a plot of $\log x/m$ against $\log [ox]_{eq}$ is linear. From this plot, k and n can be obtained.

Result

(i) Value of k =

(ii) Value of n =

Table 3.12.1 Preparation of Various Concentration of Fe^{3+} solution

S. No	Volume of Iron solution (mL)	Volume of HNO_3 (mL)	Volume of NH_4SCN (mL)	Volume of distilled water (mL)	Iron Concentration (ppm)
1.	1	1	1	7	1
2.	2	1	1	6	2
3.	3	1	1	5	3
4.	4	1	1	4	4
5.	5	1	1	3	5
6.	6	1	1	2	6

Measurement of absorbance λ = 480 nmc

Course work

Ex.No: 12

Date:

VERIFICATION OF BEER'S LAW USING SPECTROPHOTOMETER-DETERMINATION OF THE IRON CONTENT BY SPECTROPHOTOMETRY

Aim:

To determine the amount of iron content of an unknown solution by Spectrophotometry.

Principle

Spectrophotometer is an instrument used to measure the intensity of light absorbed by a substance. The intensity of a transmitted light beam of monochromatic light decreases exponentially as the concentration of the adsorbing substances and the path length of the light through solution increases. Absorbance and concentration are related by Beer-Lambert's law,

$$\mathbf{Log}\frac{\mathbf{I_0}}{\mathbf{I}} = \mathbf{A} = \mathbf{\epsilon cx}$$

where, I_0 - Intensity of incident light

I - Intensity of transmitted light

€ - Molar extinction coefficient

X - Cell thickness

C - Concentration

A - Absorbance

$$Fe^{3+} + 6KSCN \longrightarrow [Fe\,(SCN)_6]^{3-} + 6K^+$$

Red colour complex

Table 3.12.2 Preparation of Various Concentration of Fe^{3+} solution

S. No	Concentration (ppm)		Absorbance
	ppm	Normality (N)	
1.	1	0.01	
2.	2	0.02	
3.	3	0.03	
4.	4	0.04	
5.	5	0.05	
6.	6	0.06	
7	Unknown		

$$Fe^{3+} + 6NH_4(SCN) \longrightarrow [Fe(SCN)_6]^{3-} + 6NH^{4+}$$

Ferrous iron solution is acidified with HNO_3 to convert Fe^{2+} to Fe^{3+}. Fe^{3+} reacts with potassium thiocyanate or ammonium thiocyanate to give red colour. The complex formed gives absorption in the region $\lambda = 480$nm. The absorbances of various concentrations are measured.

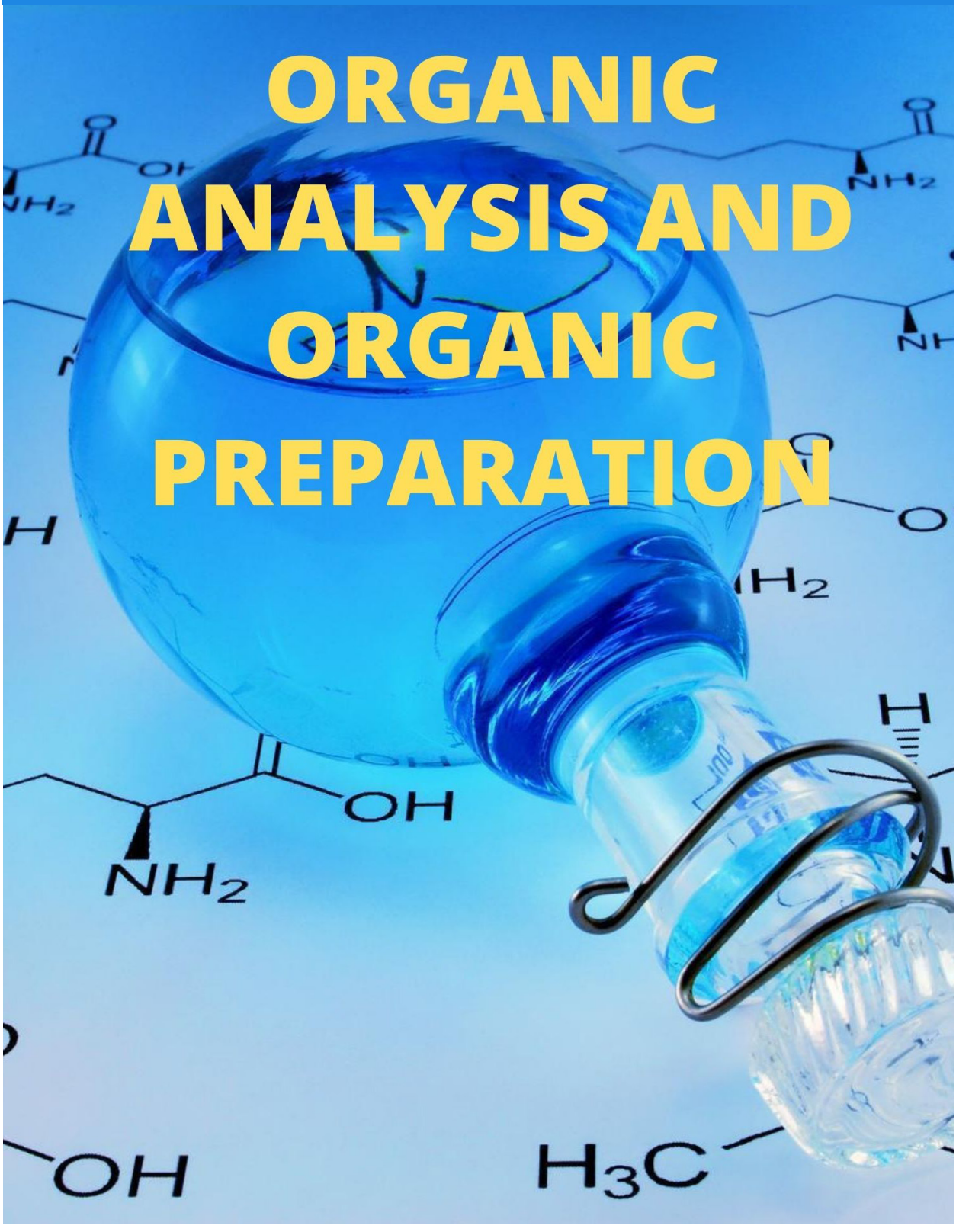
A calibration curve is drawn by measuring the absorbance of solutions of known concentration. The absorbance of the unknown solution is measured and the concentration of unknown solution is determined from the calibration curve.

Procedure

Various concentration of Fe^{3+} is prepared. The Spectrophotometer is warmed up for 10 minutes. The monochromator is adjusted to $\lambda = 480$nm. The absorbance of blank solution is noted as zero. The absorbance of all standard solutions is measured. The absorbance of unknown solution is measured. The calibration graph is drawn between the concentration and absorbance. From the absorbance, the concentration of unknown solution is measured.

Result

ORGANIC ANALYSIS AND ORGANIC PREPARATION



ORGANIC ANALYSIS AND ORGANIC PREPARATIONS

Subject Code: 18UCHCR4

CONTENTS

ORGANIC ANALYSIS

SYSTEMATIC ANALYSIS OF ORGANIC SUBSTANCE

EXPERIMENT	OBSERVATION	INFERENCE
I. PRELIMINARY TESTS **1. Colour and appearance**	Yellow solid or liquid	Presence of aromatic nitro compound.
	Dark coloured solid or liquid	Presence of phenol or aromatic amine.
	Colourless liquid turning brown on exposure to air	Presence of aromatic amine
	White solid	Presence of carbohydrate, aromatic acid or amide.
	Colourless liquid	Presence of aldehyde or ester.
2. Odour	Fruity smell	Presence of ester.
	Smell of bitter almonds	Presence of aromatic aldehyde.
	Carbolic smell	Presence of phenol.
	Fishy smell	Presence of aromatic amine.
	Odourless	Absence of esters, amines, acid, aldehydes and phenols.
3. Solubility in water Solubility of the substance in the following solvents are tested. i) water	a)Soluble in cold water	Presence of carbohydrates or diamide
	b) Soluble in hot water and crystallizes on cooling	Presence of aromatic acid or phenolic acid.
	c) insoluble	Presence of aromatic amine, aldehyde or ester.
ii) 5% sodium bicarbonate	Soluble	Presence of acid

EXPERIMENT	OBSERVATION	INFERENCE
iii) 5% sodium hydroxide	Soluble	Presence of acid or phenol
iv) 5% hydrochloric acid	Soluble	Presence of amines
4. Tests for Aromaticity ***a) Ignition Test*** A little of the substance is ignited on a nickel spatula	Burns with a smoky flame Burns with a non-smoky flame	Presence of aromatic compound. Presence of aliphatic compound.
b) Nitration Test: A little of the substance is mixed with 3 mL of conc. HNO_3 and 1 mL of con. H_2SO_4 in a semi micro tube. It is heated in a boiling water bath and poured into 50 mL of water in a beaker	Yellow solution	Presence of aromatic compound.
	Colourless solution	Presence of Aliphatic compound.
5. Test for Saturation / unsaturation a) A little of the substance is mixed with bromine water in a semi micro test tube	Decolourisation	Presence of unsaturated compound
	Decolourisation with the formation of a white precipitate	Presence of saturated, but easily brominated compounds like phenol, amine or aldehyde.
	No decolourisation	Presence of saturated compound.
b) A little of the substance in water is treated with a few drops of dilute $KMnO_4$ solution	Decolourisation	Presence of unsaturated compound.
	Decolourisation with the formation of brown precipitate	Presence of saturated but easily oxidisable compounds like amines, phenols or aldehydes.
	No decolourisation	Presence of saturated compound

EXPERIMENT	OBSERVATION	INFERENCE
DETECTION OF ELEMENTS **Lassaigne's Test (Sodium fusion Test)** A small piece of metallic sodium is taken in a semi micro test tube and melted. A small amount of the substance is then added and again heated strongly. Water is then added, heated to boiling and cooled. This is called as the sodium fusion extract which is used for the detection of elements.		
1. Test for Nitrogen About 3 drops of the extract is fused with $FeSO_4$ crystals. A drop of the solution is mixed with one drop of NaOH and one drop of Con. HCl.	Green (or) blue colouration	Presence of nitrogen
	No Green (or) blue colouration	Absence of nitrogen
2. Test for Halogens A drop of the extract is mixed with a drop of dil.nitric acid and is heated, cooled and a drop $AgNO_3$ solution is added.	White precipitate soluble in NH_4OH.	Presence of chlorine
	Pale yellow precipitate sparingly soluble in NH_4OH	Presence of bromine
	Yellow precipitate insoluble in NH_4OH	Presence of iodine
	No characteristic precipitate	Absence of halogens
3. Test for Sulphur A drop of the extract is mixed with freshly prepared sodium nitroprusside solution	Violet colouration	Presence of Sulphur
	No violet colouration	Absence of Sulphur
II. DETECTION OF FUNCTIONAL GROUPS		
1. Action of $NaHCO_3$ A little of the substance is added to a saturated solution of $NaHCO_3$ without shaking.	Brisk effervescence	Presence of carboxylic acid
	No brisk effervescence	Absence of acid

EXPERIMENT	OBSERVATION	INFERENCE
2. Action of con. H_2SO_4: A small amount of the substance is heated with 2 mL of conc. H_2SO_4	Charring with effervescence	Presence of carbohydrate
	No characteristic change	Absence of carbohydrates
3. Action of neutral $FeCl_3$ To a solution of the substance in water added 2 drops of neutral $FeCl_3$ solution.	Violet colouration	Presence of phenol
	No violet colouration	Absence of phenol
	Blue or green colour changing into a white precipitate	Presence of 2-naphthol
	A white precipitate	Presence of 1-naphthol
	No characteristic change	Absence of phenol, 1 –naphthol and 2-naphthol
4. Borsche's reagent test: A small amount of the substance is added to 3 mL of Borsche's reagent. A few drops of conc. HCl is added and boiled for 2 minutes and cooled. Then a little water is added.	Yellow or red orange precipitate	Presence of aldehyde
	No yellow or red orange precipitate	Absence of aldehyde
5. Action of NaOH a) A small amount of the substance is added to a strong solution of NaOH and then heated to boiling.	Ammonia gas is evolved on continued boiling	Presence of amide
	Substance dissolved gradually on warming	Presence of ester
	No characteristic change	Absence of amide and ester
b) The above solution is acidified with conc. HCl.	White precipitate	Presence of monamide or ester
	No white precipitate	Absence of monamide and ester

EXPERIMENT	OBSERVATION	INFERENCE
6. Action of HCl A small amount of the substance is shaken with 1:1 HCl.	Substance readily dissolved and regenerated on adding NaOH solution	Presence of aromatic amine
	No characteristic change	Absence of aromatic amine
7. Test for anilide About 1g of the substance is boiled under reflux with 2mL of conc. HCl. The solution is cooled in ice, diazotized with sodium nitrite and mixed with β-naphthol dissolved in NaOH	Scarlet red dye	Presence of anilide
1) TEST FOR CARBOXYLIC ACID ***i) Reaction with $NaHCO_3$:*** A little of the substance is added to a saturated solution of $NaHCO_3$ without shaking.	Brisk effervescence	Presence of acid
ii) Ester formation A little of the substance is heated with 2 mL of alcohol and a few drops of conc. H_2SO_4 and poured into about 30 mL of a dilute solution of Na_2CO_3.	Fruity smell	Presence of carboxylic acid
iii) Fluorescein Test A little of the substance is heated with twice its weight of resorcinol and 2 – 3 drops of conc. H_2SO_4, cooled and poured into water containing NaOH.	Greenish yellow fluorescence	Presence of dicarboxylic acid
	No greenish yellow fluorescence	Absence of dicarboxylic acid

EXPERIMENT	OBSERVATION	INFERENCE
2) TEST FOR CARBOHYDRATE: ***i) Action of con.*** **H_2SO_4*:*** A small amount of the substance is heated with 2 mL of con. H_2SO_4	Charring with effervescence	Presence of carbohydrate
ii) Tollen's reagent test: A pinch of the substance is heated with Tollen's reagent	Bright silver mirror	Presence of monosaccharide
iii) Barfoed's test: A little of the substance dissolved in 1 mL of water is mixed with 1 mL of Barfoed's reagent and heated in a boiling water bath	Red precipitate	Presence of monosaccharide
iv) Fehling's solution test: a) A little of the substance is heated with Fehling's Solution A and Fehling's solution B in a boiling water bath	Reddish brown precipitate	Presence of monosaccharide
v) Osazone test a) A little of the substance is dissolved in water. And added Phenylhydrazine and sodium acetate. The mixture is heated in a boiling water bath	Yellow crystalline precipitate	Presence of monosaccharide
vi) Molisch's test i) A little of the substance is dissolved in 2 mL of water, a few drops of an alcoholic solution of α-naphthol is added followed by 1 mL of con. H_2SO_4 along the sides of the test tube.	Violet ring at the junction	Presence of monosaccharide

EXPERIMENT	OBSERVATION	INFERENCE
3) TEST FOR PHENOL ***i) Action of neutral*** **$FeCl_3$** To a solution of the substance in water added 2 drops of neutral $FeCl_3$ solution	Violet colouration	Presence of phenol
	White precipitate	Presence of 1-naphthol
	Blue (or) green colour changing into a white precipitate	Presence of 2-naphthol
ii) Libermann's test A little of the substance is heated with $NaNO_2$ and conc. H_2SO_4 and it is poured into water containing alkali	Blue (or) green colouration	Presence of phenol
iii) Phthalein fusion test A little of the substance is heated with phthalic anhydride and conc. H_2SO_4 and it is poured into water containing alkali.	Pink (or) red colouration	Presence of phenol or phenolic acid (monohydric)
	Greenish yellow fluorescence	Presence of dihydric phenol
iv) Tollen's reagent test: A little of the substance is heated with Tollen's reagent.	Bright silver mirror	Presence of dihydric phenol.
v) Test for 1-naphthol: A pinch of the substance is dissolved in 1 mL of alcohol. 3 drops of sugar solution are added to it followed by the addition of con. H_2SO_4 in drops along the sides of the test tube.	Violet ring at the junction of two liquids.	Presence of 1-naphthol

EXPERIMENT	OBSERVATION	INFERENCE
vi) Test for 2-naphthol: Three drops of aniline is dissolved in slight excess of dil. HCl, cooled in ice added a strong solution of sodium nitrite solution to it drop by drop. About 0.5 mL of strong solution of sodium acetate is added and the mixture is poured in to a solution of the substance in strong NaOH solution.	A scarlet red dye	Presence of 2-naphthol
4) TEST FOR ALDEHYDE/ KETONES **I *a) Borsche's reagent test:*** A drop of the alcoholic solution of the substance is mixed with Borsche'sreagent followed by 2 drops of conc. HCl. The mixture is heated for 15 minutes and diluted the solution.	Red orange or yellow precipitate	Presence of aldehyde or aliphatic ketone
b) Action of Semicarbazide hydrochloride: 0.2 gm of semicarbazide hydrochloride and 0.2 gm of sodium acetate are dissolved in water and warmed with a small amount of organic substance.	White crystalline precipitate	Presence of aldehyde/ ketone
II Test for aldehydes only ***a) Sodium bisulphite test*** A few drops of the substance in shaken with 3 mL of saturated $NaHSO_3$ solution	White crystalline precipitate	Presence of aldehyde

EXPERIMENT	OBSERVATION	INFERENCE
b) Schiff's reagent test About 3 drops of the substance is treated with 3 mL of Schiff's reagent and shaken well	Immediate pink or red colouration	Presence of aldehyde
	Pink colouration is formed very slowly	Presence of aliphatic ketones like acetone
c) Tollen's reagent test About three drops of the substance is treated with 2mL of Tollen's reagent A and B and heated under water bath.	Black precipitate or Bright silver mirror	Presence of aldehyde
d) Action of hydroxylamine hydrochloride Dissolved little of the substance in 1 mL of alcohol. This is mixed with hydroxylamine hydrochloride and 0.1 g of sodium acetate in a semi micro tube and placed in a boiling water bath.	White crystalline precipitate	Presence of aldehyde
III Test for Ketone ***a) Legal's Test*** To a little of the substance 2mL of water , 5 drops of sodium nitroprusside solution, 5 drops of NaOH solution followed by 5 drops of glacial acetic acid are added.	Orange colour changes to purple	Presence of Ketone
5) TEST FOR ESTER ***i) Acid formation*** A little of the substance is boiled with 10% NaOH solution for 10 minutes and cooled. It is then acidified with conc. HCl	White precipitate	Presence of ester

EXPERIMENT	OBSERVATION	INFERENCE
ii) Hydroxamic acid test A few drops of the substance is heated with ethanolic solution of hydroxylamine hydrochloride and 10% NaOH solution and cooled. The solution is acidified with dil. HCl and a few drops of $FeCl_3$ is added and diluted with little water.	Violet colouration	Presence of ester
TEST FOR COMPOUNDS CONTAINING NITROGEN		
1) Test for amide ***i) Acid formation*** a) About 1 gm of the substance is boiled with 15 mL of a 10% solution of NaOH	Evolution of ammonia gas	Presence of amide
b) The above solution is cooled and then acidified with conc. HCl	White precipitate	Presence of monoamide
ii) Biuret test A little of the substance is heated in a dry test-tube until it melts. The resulting white residue is cooled and dissolved in 2 mL of water. Then 2 drops of a very dilute $CuSO_4$ solution is added followed by NaOH solution in drops	Violet colouration	Presence of diamide
	No violet colouration	Absence of diamide
iii) Action of conc. **HNO_3** A little of the substance is dissolved in water, and added con. HNO_3	White crystalline precipitate	Presence of diamide

EXPERIMENT	OBSERVATION	INFERENCE
iv) Action of oxalic acid To a concentrated aqueous solution of the substance, a few drops of saturated solution of oxalic acid is added.	White crystalline precipitate	Presence of diamide
2) Test for amine ***i) Action of HCl*** A small amount of the substance is shaken with 1:1 HCl.	Substance readily dissolves and regenerated on adding NaOH solution	Presence of aromatic amine
ii) Benzoylation To a few drops of the substance 5mL of 10% sodium hydroxide and 1mL of benzoyl chloride is added and shaken well	White precipitate	Presence of aromatic primary amine
iii) Dye test A little of the substance is dissolved in 3 mL of dil. HCl and cooled in ice water. To this, 1 mL of a strong solution of $NaNO_2$ is added in drops with constant stirring. Then, 2 mL of sodium acetate solution is added and the mixture is poured into a solution of β-naphthol in NaOH solution	Scarlet red dye	Presence of aromatic primary amine
iv) A little of the substance is dissolved in dilute HCl, cooled in ice and a solution of $NaNO_2$ is added slowly	A yellowish sweet smelling oil is formed	Presence of secondary amine
v) A little of the substance is dissolved in dilute HCl and a solution of Potassium ferro cyanide is added	White Precipitate	Presence of tertiary amine

EXPERIMENT	OBSERVATION	INFERENCE
vi) Carbylamine Test A little of the substance is warmed with chloroform and alcoholic potassium hydroxide solution.	Offensive odour	Presence of primary amine
3. Test for anilide ***i) Bromination*** To a little of the substance 2mL of bromine in glacial actic acid is added and shaken well and poured into 10mL of water.	Pale yellow precipitate	Presence of anilide
ii) Dye test About 1g of the substance is boiled under reflux with 2mL of conc. HCl. The solution is cooled, diazotized with sodium nitrite and mixed with β-naphthol dissolved in NaOH.	Scarlet red dye	Presence of anilide
4. Test for nitrocompound ***i) Reduction in acid solution*** To 0.5mL of the substance 2mL of Conc. HCl and small piece of tin is added and boiled for 5 minutes. The solution is cooled and added with 3 mL of dil. HCl and cooled in ice water. To this, 1 mL of a strong solution of $NaNO_2$ is added in drops with constant stirring. Then, 2 mL of sodium acetate solution is added and the mixture is poured into a solution of β-naphthol in NaOH solution	A scarlet red dye	Presence of nitro compound

EXPERIMENT	OBSERVATION	INFERENCE
ii) Mulliken and Barker test A small amount of the substance is dissolved in 3mL alcohol, 2mL of calcium chloride solution and a pinch of zinc dust are added. Heated to boiling, it is then cooled and filtered. The filtrate is divided into two parts		
a) To one part 2mL of Tollen's reagent is added and heated in a water bath.	Black precipitate or Silver mirror	Presence of nitro compound
b) To the other part 1mL each of Fehling solution A or B are added and heated.	Red precipitate	Presence of nitro compound

PREPARATION OF SOLID DERIVATIVE

1) For carboxylic acid

i) Amide derivative (for monocarboxylic acid)

1g of the organic substance is dissolved in minimum amount of liq. NH_3 in a test tube. It is warmed carefully and poured in a chinadish. White precipitate of amide derivative is obtained.

ii) Anhydride derivative (for dicarboxylic acid)

A little of the substance is taken in a china dish, covered with an inverted funnel and heated. The anhydride derivative formed on the walls of the funnel are collected.

2) For carbohydrate

Osazone derivative

A solution of phenyl hydrazine is prepared by dissolving 1 gm of phenyl hydrazine hydrochloride and 1.5 gm of sodium acetate in 10 mL of warm water. To this, 1 drop of glacial acetic acid is added followed by 1 mL of saturated solution of the substance. The mixture is heated in a boiling water bath for 15 minutes and cooled. Yellow crystals of osazone are obtained.

3) For Phenol and Amine

i) Benzoyl derivative

About 1 gm of the substance is mixed with 20 mL of 10% NaOH and 5 mL of benzoyl chloride in a conical flask. The flask is corked and shaken vigourously till the smell of benzoyl chloride no longer persists. The contents are then poured into 100 mL of water in a beaker. White precipitate of phenyl benzoate is obtained.

ii) Bromo derivative

About 0.5 gm the substance is treated with saturated NaOH added bromine water till a yellow colour persists in the solution. The mixture is shaken well to get yellow precipitate of 2, 4, 6 – tribromophenol.

4) For aldehyde / Ketone

i) Sodium bisulphite derivative

About 0.5 gm of the substance is shaken with 2 mL of saturated solution of sodium bisulphite. White crystalline precipitate of sodium bisulphite compound is formed.

ii) 2, 4 dinitrophenylhydrazone derivative

The substance is dissolved in ethanol, then added Borsche's reagent. The mixture is heated in a water bath for 15 minutes cooled and poured into water. Yellow orange crystals separate on cooling.

5) For Amide and Ester

Acid derivative

About 1 mL of the substance is mixed with 10 mL of 20% NaOH solution and boiled under reflux for about half an hour (until the oily layer disappears). The mixture is poured into a beaker containing 100 mL of water and stirred well. The solution on acidification with conc. HCl gives a white precipitate of acid.

6) For Diamide

i) Oxalate derivative

About 5 mL of a concentrated solution of diamide is added drop by drop to a saturated solution of oxalic acid in water with shaking. Urea oxalate separates as a white crystalline precipitate.

ii) Nitrate derivative

To about 5 mL of a concentrated solution of diamide a few drops of conc. HNO_3 are added with shaking. A white crystalline precipitate of urea nitrate separates.

7) For anilide

Bromo derivative

About 0.5 gm the substance is treated with saturated NaOH added bromine water till a yellow colour persists in the solution. The mixture is shaken well to get yellow precipitate.

Report :

The given organic substance is _____

PREPARATION OF ORGANIC COMPOUNDS

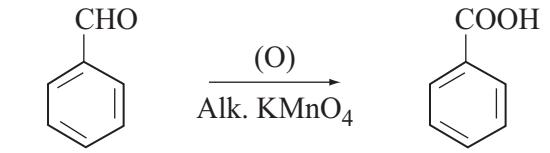
Ex.No: 1

Date:

PREPARATION OF BENZOIC ACID FROM BENZALDEHYDE (OXIDATION)

Principle

Benzaldehyde is readily oxidised to benzoic acid by alkaline $KMnO_4$ solution.



Benzaldehyde Benzoic acid

Chemicals Required

Benzaldehyde	- 5 mL
Anhydrous Na_2CO_3	- 5 g
Potassium permanganate (6%)	- 20mL
Sodium bisulphite	- 8 g

Procedure

About 5 g of anhydrous sodium carbonate is dissolved in 50 mL of water in a RB flask. 5 mL of benzaldehdye and a few porcelain pieces are added. The flask is fitted with an air condenser and heated gently on a wire gauze. When the mixture begins to boil, a saturated solution of about 20mL of $KMnO_4$ is added little by little through the condenser until the solution in the flask coloured pink. The contents of the flask are boiled for about 45 minutes until the pink colour is disappeared. The flask is then cooled and the supernatant liquid is transferred to a beaker. About 8 g of sodium bisulphite is added followed by conc. HCl in small quantities at a time with constant stirring till the solution becomes distinctly acidic. The contents of the beaker are cooled very well and the precipitated benzoic acid is filtered off at a suction pump, washed with water and dried.

Recrystallisation

A portion of the sample is recrystallised from boiling water.

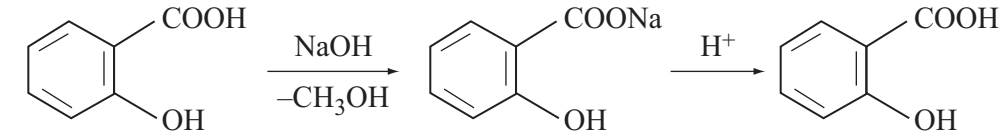
Ex.No: 2

Date:

PREPARATION OF SALICYLIC ACID FROM METHYL SALICYLATE (HYDROLYSIS)

Principle

Methyl salicylate is readily hydrolysed by sodium hydroxide solution to form sodium salicylate which on acidification gives salicylic acid.



Methyl salicylate — Sodium salicylate — Salicylic acid

Chemicals required

Methyl salicylate	-	5 mL
10% NaOH solution	-	40 mL

Procedure

About 5 mL of methyl salicylate and 40 mL of 10% NaOH solution are taken in a RB flask. A few porcelain pieces are added and the contents of the flask are heated on a wire gauze using a water condenser for about 45 minutes. The hydrolysis is complete when no oily drops are seen in the flask. The heating is then stopped. The flask is cooled. The contents are poured into a beaker containing 200 mL of water and stirred well. Conc. HCl is added in small quantities at a time with constant stirring till the solution becomes distinctly acidic. (The solution is tested with blue litmus which turns red). The precipitated salicylic acid is filtered off using a Buchner funnel, washed with water and then dried.

Recrystallisation

A portion of the substance is recrystallised from hot water.

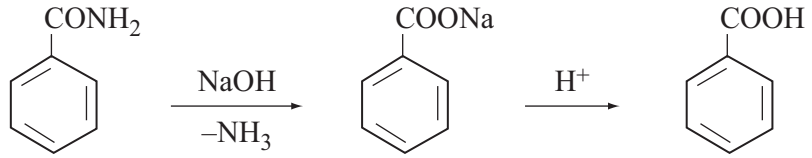
Ex.No: 3

Date:

PREPARATION OF BENZOIC ACID FROM BENZAMIDE (HYDROLYSIS)

Principle

Benzamide is hydrolysed to sodium benzoate by NaOH solution. The sodium salt on acidification gives benzoic acid.



Benzamide Sodium benzoate Benzoic acid

Chemicals Required

Benzamide	-	4 g
10% NaOH solution	-	40 mL

Procedure

4 g of benzamide mixed with 40 mL of 10% NaOH solution is taken in a RB flask. A few porcelain pieces are added and the flask is heated on a wire gauze using a water condenser for about 45 minutes. Sodium benzoate is formed in solution with the evolution of ammonia. The heating is stopped when the evolution of ammonia ceases. The flask is then cooled and the contents are poured into 200 mL of water taken in a beaker. Conc. HCl is added in a thin stream with constant stirring till the solution becomes distinctly acidic (tested with a blue litmus which turns red). The precipitated benzoic acid is filtered at a suction pump using a Buchner funnel, washed with cold water and dried.

Recrystallisation

A portion of the sample is recrystallised from boiling water

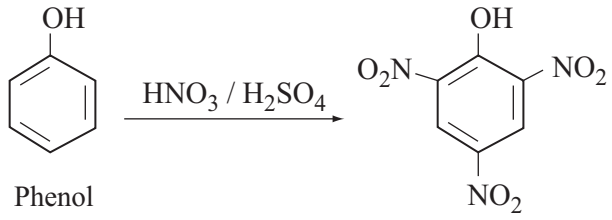
Ex.No: 4

Date:

PREPARATION OF PICRIC ACID FROM PHENOL (NITRATION)

Principle

Phenol on direct nitration with a mixture of conc. HNO_3 and conc. H_2SO_4 gives sym-trinitrophenol (picric acid). However, the yield is poor due to oxidation of phenol by nitric acid. To avoid this, phenol is first sulphonated and then nitrated to get picric acid.



Phenol

Picric acid

Chemicals Required

Phenol - 4 mL

Fuming HNO_3 - 15 mL

Conc. H_2SO_4 - 5 mL

Procedure

About 4 mL of phenol and 5 mL of conc. H_2SO_4 are taken in a dry flask fitted with cork. The contents are shaken and heated on a water bath for 30 minutes. The flask is cooled in an ice bath (10°C) and added 15mL of concentrated nitric acid in drops with shaking. Immediate evolution of brown fumes takes place. When the evolution of brown fumes stop, the contents are heated on a water bath for 1 hour with occasional shaking. It is then cooled and poured into 100mL of water containing ice. A yellow solid separates out which are filtered.

Recrystallisation

A portion of the sample is recrystallised from dilute alcohol.

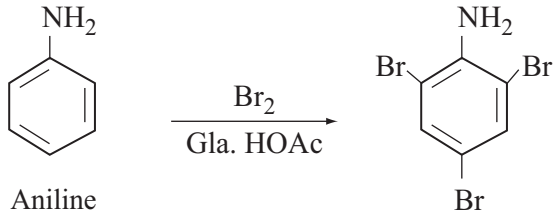
Ex.No: 5

Date:

PREPARATION OF 2, 4, 6 - TRIBROMOANILINE FROM ANILINE (BROMINATION)

Principle

In aromatic electrophilic disubstitution the monovalent functional groups, generally direct the incoming electrophile towards the ortho– and para– positions. Depending upon the size of the group already present and that of the incoming electrophile the percentage ratio of the ortho– and para– products may vary. Because of the small size of the amino group, the bromine atoms enter into the two ortho– positions as well as the para– position and leads to the formation of the tribromo derivative.



Aniline

2,4,6 - Tribromoaniline

Chemicals Required

Aniline	-	2.5 mL
Glacial acetic acid	-	5 mL
Bromine in glacial acetic acid	-	7 mL (2.5 ml of bromine in 4.5 ml of glacial acetic acid)

Procedure

2.5 mL of aniline is dissolved in 10 mL of glacial acetic acid in a conical flask kept in ice – water bath (since the reaction is exothermic, the container has to be placed in an ice – water bath). Into this bromine in glacial acetic acid (14 mL) is slowly run down

from a tap funnel with continuous stirring. When the reaction mixture becomes yellow in colour, the addition of bromine may be stopped. After about 10 minutes, the reaction mixture is poured into excess of water. The crude tribromoaniline is filtered at the pump, washed thoroughly with water and dried.

Recrystallisation

A portion of the sample is recrystallised from methylated spirit.

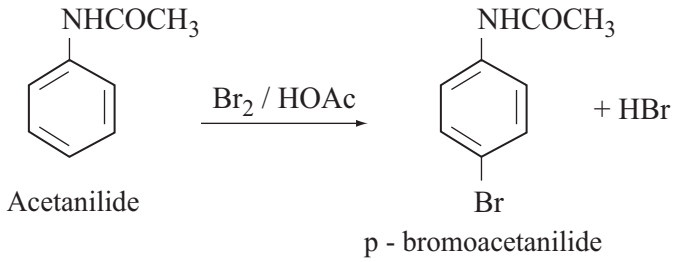
Ex.No: 6

Date:

PREPARATION OF PARABROMOACETANILIDE FROM ACETANILIDE (BROMINATION)

Principle

Acetanilide is readily brominated in acetic acid medium to give p-bromoacetanilide as the main product.



Acetanilide

p - bromoacetanilide

Chemicals Required

Acetanilide	-	3.5 g
Glacial acetic acid	-	12.5 mL
Bromine in glacial acetic acid	-	8 mL (1.5mL of bromine in 6.5 mL of glacial acetic acid)

Procedure

About 3.5 g of finely powdered acetanilide is dissolved in 12.5 mL of glacial acetic acid in a conical flask. About 1.5 mL of bromine liquid is dissolved in 6.5 mL of glacial acetic acid in a boiling tube and transferred into a burette. The bromine solution is then added in small portions into the conical flask kept in ice cold water. After each addition, the flask is closed with a cork and shaken well for a minute to ensure thorough mixing. When all the bromine has been added, the solution will have an orange colour. The contents of the flask are kept aside for 15 minutes with occasional shaking and then poured into a

beaker containing 200 mL of ice cold water. Colourless crystals of p-bromo acetanilide are precipitated. The precipitate is filtered off with suction on a Buchner funnel, washed thoroughly with cold water and dried.

Recrystallisation

A portion of the sample is recrystallised from 60% alcohol.

Ex.No: 7

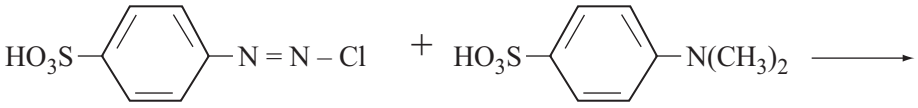
Date:

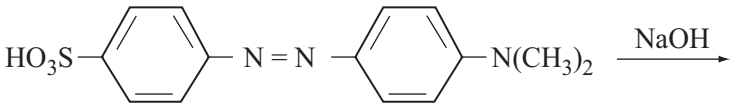
PREPARATION OF METHYL ORANGE (DIAZOTIZATION)

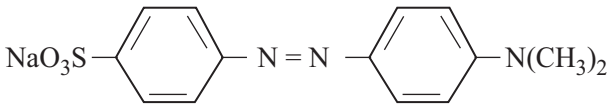
Principle

Methyl orange is prepared by diazotisingsulphanilic acid and coupling it with N, N-dimethyl aniline in presence of excess of hydrochloric acid. Methyl orange formed is made alkaline with sodium hydroxide and then it is salted out by adding sodium chloride.

Sulphanilic acid







Methyl orange

Chemicals Required

Sulphanilic acid	- 3g
Dimethyl aniline	- 2 mL

Anhydrous sodium carbonate	- 1 g
Sodium nitrite	- 1.2 g
Sodium chloride	- 7.5 g

Procedure

One gram of anhydrous sodium carbonate is dissolved in about 35 mL of water in a beaker. 3 g of sulphanilic acid is added to the beaker and brought into solution by warming. The solution is then cooled in ice and 1.2 g of sodium nitrite dissolved in 5 mL of water is added. A mixture of concentrated hydrochloric acid (2 mL) and water (2.5 mL) is then added slowly, so that the temperature does not rise above 5ºC. To the solution of thediazonium salt thus prepared, an ice cold solution of 2 g of dimethyl aniline in dilute hydrochloric acid (2 mL of strong hydrochloric acid diluted with 10 mL of water) is added. The mixed solution is made alkaline with 2N sodium hydroxide solution and the dye salted out by adding about 7.5 g of sodium chloride. After keeping for an hour the sodium salt is filtered, washed with a little ice water, dried and the yield noted.

Recrystallisation

A portion of the sample is recrystallised from boiling water.

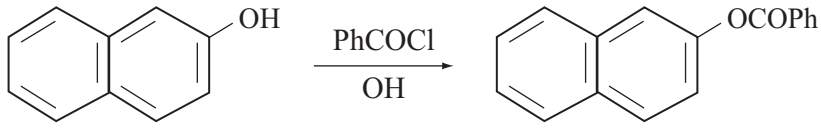
Ex.No: 8

Date:

PREPARATION OF β – NAPHTHYLBENZOATE FROM β – NAPHTHOL (BENZOYLATION)

Principle

Benzoylation of phenols and aromatic amines in slightly alkaline medium is called as Schotten – Baumann Reaction. Under alkaline conditions the acidic hydrogens in these compounds can be readily replaced, thereby facilitating the process of benzoylation. Thus with β –naphthol, we get β – naphthylbenzoate and with aromatic primary amines like aniline, we get benzamide.



β- Naphthol β- Naphthylbenzoate

Chemical Required

β – Naphthol	-	2 gm
NaOH (10%)	-	30mL
Benzoyl Chloride	-	3mL

Procedure

Powdered β –naphthol (2 grams) and 10% sodium hydroxide (30 mL) are taken in a conical flask fitted with a cork and shaken well. 3 mL of benzoyl chloride are added and shaken well for about 10 minutes. The reaction mixture is cooled under running tap water, as the reaction is exothermic. The solid lumps are broken and the benzoyl derivative is filtered at the pump. It is then washed thoroughly with water to remove the alkali.

Recrystallisation

A portion of the sample is recrystallised from alcohol.

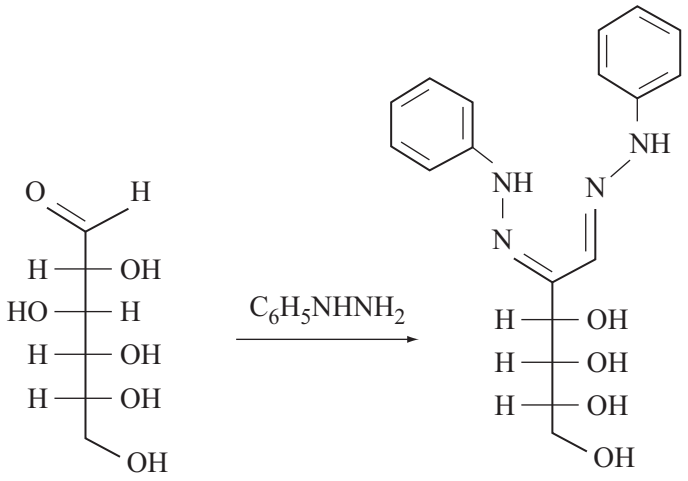
Ex.No: 9

Date:

PREPARATION OF GLUCOSAZONE FROM GLUCOSE (OSAZONE FORMATION)

Principle

Glucosazone is formed by the reaction of glucose with phenyl hydrazine



Chemicals Required

Glucose	-	1 g
Phenyl hydrazine	-	1 g
Sodium acetate	-	1.5 g
Glacial acetic acid	-	1 mL

Procedure

A solution of phenyl hydrazine is prepared by dissolving 1 gm of phenyl hydrazine hydrochloride and 1.5 gm of sodium acetate in 10 mL of warm water. To this, 1 drop of

glacial acetic acid is added followed by 1 mL of saturated solution of the substance. The mixture is heated in a boiling water bath for 15 minutes and cooled. Yellow crystals of glucosazone are obtained. The precipitate is filtered at a suction pump using a Buchner funnel, washed with cold water and dried.

Recrystallisation

A portion of the sample is recrystallised from ethanol.

Ex.No: 10

Date:

DETERMINATION OF PHYSICAL CONSTANT MELTING POINT

Aim

To determine the melting point of the given substance

Procedure

A 100mL beaker containing liquid paraffin upto a convenient is taken and placed on wire gauze kept over a tripod stand. A glass stirrer is placed in the paraffin. A thermometer is inserted into a one holed cork and clamped in position with the bulb immersed in the paraffin, about 1cm above the bottom of the beaker.

A small capillary tube is fused at one end by gentle fusion in the bunsen flame. The substance whose melting point is to be determined is powdered and pressed on a porcelain tile by means of a capillary tube pressed down into the heap. On inverting the capillary and gently tapping the loosened powder falls down. The substance should occupy a column of about 2mm in the capillary tube. It is then attached by moistening it with paraffin to the lower end of the thermometer bulb. The bath is heated steadily by a small flame with stirring. The temperature at which the substance begins to melt giving clear liquid is noted. The determination can be repeated using a fresh capillary tube filled with the substance.

Result

Melting point of the given substance = _____°C

Ex.No: 11

Date:

DETERMINATION OF PHYSICAL CONSTANT BOILING POINT

Aim

To determine the boiling point of the given liquid

Procedure

The boiling point of the liquid is usually determined in a 50mL distilling flask or in a pyrex test tube with the side tube. The liquid whose boiling point is to be determined is taken in the distilling flask. A few bits of porous porcelain are added for uniform boiling. The flask is fitted with a thermometer in such a way that the bulb of the thermometer must be kept close to the exit of the side tube. The side tube is connected to an adaptor which in turn is introduced into a receiver.

The flask is slowly heated. The liquid gets boiled and vapours escape through the exit. When the liquid begins to distill, the temperature remains steady and it is noted. This gives the boiling point of the liquid. The distillate is collected in the receiver.

Result

The boiling point of the given liquid = _____°C

Course Work

Ex.No: 12

Date:

Extraction of various phytochemicals using soxhlet apparatus and to analyse plant pigments using flame photometer

Aim

To extract the phytochemicals using soxhlet apparatus and to analyse plant pigments using flame photometer

Procedure

The solid substance is placed in the porous thimble A (made of tough filter paper) and the latter is placed in the inner tube of the Soxhlet apparatus The apparatus is then fitted to a round-bottomed flask C of appropriate size containing the solvent and boiling chips, and to a reflux condenser D (preferably of the double surface type). The solvent is boiled gently; the vapour passes up through the tube E, is condensed by the condenser D and the condensed solvent falls into the thimble A and slowly fills the body of the Soxhlet. When the solvent reaches the top of the tube F, it siphons in to the flask C, and thus removes that portion of the substance which it has extracted in A. The process is repeated automatically for complete extraction.

Analysis

When the extract is aspirated in to the flame, the Na, K and Mg ion present in solution is atomized in flame and the atoms are excited and the radiation of specific wavelength is emitted. The amount of radiation is proportional to the concentration of the solution and it is measured in a flame photometer by using a suitable filter (Na -580n.m, K-768n.m and Mg-285nm)

Table 1 Determination of unknown concentration

S. No	Concentration (ppm)			Absorbance		
1.	**Na**	**K**	**Mg**	**Na**	**K**	**Mg**
2.						
3.						
4.						
5.						
6.						
7.						
8.	Unknown					

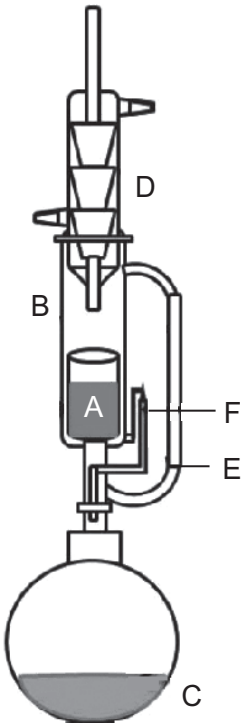


Figure 4.12.1 Soxhlet Apparatus

Preparation of solution

a. Preparation of Blank Solution

- 25 mL 1 N HCl (2.5mL dissolved in 25 mL of deionized-distilled water)

b. Preparation of Standard Solution

- Standard NaCl solution: 2.542 g AR grade NaCl is dissolve in 1 liter of deionized-distilled water (1000ppm)
- Standard KCl solution: 1.907 g of dried AR grade KCl is dissolved in 1 liter of deionized-distilled water.
- Standard Magnesium solution: 1g of unoxidized Mg metal (reagent grade) is taken in a 100-mL beaker. 50 mL of 1:1 HCl is added and heated gently until the magnesium dissolves. Cooled and made up to 1000 mL in SMF.

c. Sample Preparation

1 g of dried, ground plant sample is rinsed 1 N HCl and 25 mL of 1 N HCl is added from a burette and the it's allowed to stand for 24 hours. The contents are shaken well and filtered through Whatman No. 1 filter paper.

Preparation of Standard Graph

Various concentrations of sodium, potassium and magnesium solution is made from standard solutions. The flame photometer is calibrated by feeding the standards. The sample solution containing the ions is aspirated in to the flame and corresponding absorbance value is noted. A calibration curve is drawn by measuring the absorbance of solutions of known concentration. The absorbance of the unknown solution is measured and the concentration of unknown solution is determined from the calibration curve.

Result

Amount of Sodium, Potassium and Magnesium present in the solution = _____ ppm.

Ex.No: 13

Date:

Extraction of oil from plants using Clevenger apparatus

Aim

To extract oil from plants using Clevenger apparatus

Procedure

Clevenger apparatus is used for the extraction and determination of percentage of volatile oils present in the oil-bearing material such as clove, lemon grass, peppermint, eucalyptus etc. Weighed quantities of plant material are taken in a RB flask and water is added. Generally 1 part of plant material and 8 parts of water is taken in the RB flask. The flask is connected with Clevenger apparatus and the tap water is allowed to flow through the condenser. The mantle is heated gently so that oil with water vapours comes into the graduated distillate through receiver tube and the excess of water goes back into the flask. The volatile components have much lower boiling point than that of water and hence they volatilize and get distilled over and with water vapour. Heating is continued for 8 hours and the assembly is cooled and the oil is extracted from the assembly. The traces of water can be removed by rotary evaporator and the flask is cooled in a desiccator.

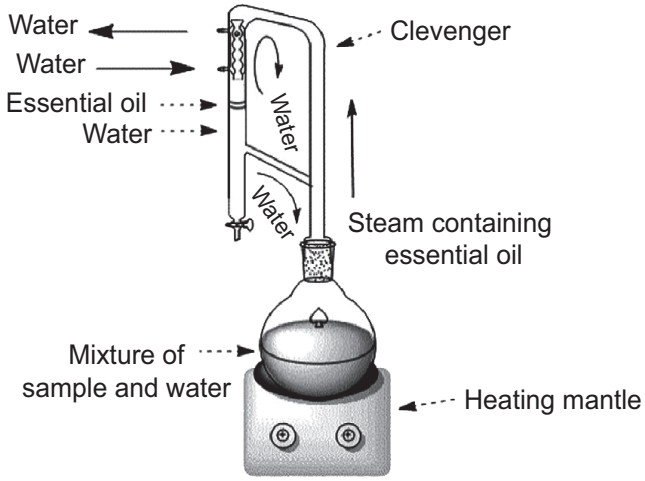


Figure 4.13.1 Clevenger apparatus

Organic Analysis

S.No	Date	Organic Substance	Derivative	Sign

S.No	Date	Organic Substance	Derivative	Sign

PREPARATION OF REAGENTS

1. **Borsche's Reagent:** It is 1% methyl alcoholic solution of 2, 4- dinitro phenylhydrazine. 1g of 2, 4-dinitrophenylhydrazine is refluxed with 100 mL of methyl alcohol till dissolved or alternatively 12 g of 2, 4-dinitrophenylhydrazine is dissolved in 60mL of conc. sulphuric acid. This solution is added with stirring to 80mL of water and 280mL of 95% ethyl alcohol. The solution is mixed thoroughly and filtered.

2. **Bromine water:** 1mL of liquid bromine per litre of the solution. It should be kept in coloured bottles.

3. **Fehling's A:** 6.9g of copper sulphate crystals are dissolved in 40mL of water and diluted to 100mL.

4. **Fehling's B:** 15g of sodium hydroxide and 36g of sodium potassium tartrate (Rochellesalt) are dissolved separately in 30mL of water each, mixed and diluted to 100mL after cooling. Equal volumes of A and B are mixed before use.

5. **Neutral Ferric Chloride:** About 1g of ferric chloride is dissolved in 100mL water. Sodium carbonate solution is added little by little to the above solution till the slight turbidity persists even after shaking. The precipitate is filtered off and the filtrate is used as neutral ferric chloride solution.

6. **Schiff's Reagent:** 0.2 g of para-rosaniline (Fuchsin) as hydrochloride or acetate is dissolved in 20mL of water and saturated with sulphurdioxide. After the solution has become colourless, it is filtered, diluted to 200mL with water and kept in dark bottles.

7. **Tollen's reagent:** It is ammonical silver nitrate solution. 100mL of commercial ammonia is diluted with an equal volume of water. 20g of silver nitrate is dissolved in the dilute ammonia prepared. A 10% aqueous solution of caustic soda is also prepared and kept separate.

GRAVIMETRIC AND INORGANIC PREPARATION

GRAVIMETRY AND INORGANIC PREPARATION

Subject Code: 18UCHCR5

CONTENTS

GRAVIMETRIC ANALYSIS

Ex.No:1

Date:

ESTIMATION OF LEAD AS LEAD CHROMATE

Aim

To estimate the amount of lead present in the whole of the given solution

Principle

The lead in a soluble lead salt solution is precipitated as Lead chromate using Potassium chromate in the presence of acetic acid. The precipitate is collected in a sintered glass crucible, dried at 120°C and weighed as Lead chromate.

$$Pb^{2+} + K_2CrO_4 \longrightarrow PbCrO_4 + 2K^+$$

Procedure

The given solution of lead salt is made upto 100mL in a standard flask using distilled water. 20mL of the made up solution is pipetted out into a clean beaker. 6mL of 2N acetic acid is added. The solution is diluted to about 100mL and heated to boiling. To the hot solution about 20mL of 4% Potassium chromate solution is added. The precipitate is digested on a steam bath for about 30minutes. The supernatant liquid must be coloured slightly yellow. The solution is tested for complete precipitation. The precipitate is then filtered using a previously cleaned, dried and previously weighed sintered glass crucible. It is washed thoroughly with hot water, dried at 120°C and weighed as lead chromate. The process of heating, cooling and weighing is repeated till a constant weight is obtained. A duplicate is conducted. From the weight of lead chromate, the weight of lead present in the whole of the given solution is calculated.

323.2g of lead chromate contains 207.2g of Lead

Result

Weight of lead present in the whole of the given solution = _____ g.

Ex.No: 2

Date:

ESTIMATION OF BARIUM AS BARIUM CHROMATE

Aim

To estimate the amount of Barium present in the whole of the given solution

Principle

The Barium in a soluble barium salt solution is precipitated as barium chromate using potassium chromate in the presence of acetic acid. The precipitate is collected in a sintered glass crucible, dried at 120°C and weighed as Barium chromate.

$$Ba^{2+} + K_2CrO_4 \longrightarrow BaCrO_4 + 2K^+$$

Procedure

The given solution of barium chloride is made upto 100mL in a standard flask using distilled water. 20mL of the made up solution is pipetted out into a clean beaker. 6mL of 2N acetic acid is added. The solution is diluted to about 100mL and heated to boiling. To the hot solution about 20mL of 4% Potassium chromate solution is added. The precipitate is digested on a steam bath for about 30minutes. The supernatant liquid must be coloured slightly yellow. The solution is tested for complete precipitation. The precipitate is then filtered using a previously cleaned, dried and weighed sintered glass crucible. It is washed thoroughly with hot water, dried at 120°C and weighed as barium chromate. The process of heating, cooling and weighing is repeated till a constant weight is obtained. A duplicate is conducted. From the weight of barium chromate, the weight of barium present in the whole of the given solution is calculated.

253.4g of barium chromate contains 137.4g of barium

Result

Weight of barium present in the whole of the given solution = _____ g.

Ex.No: 3

Date:

ESTIMATION OF ZINC AS ZINC OXINATE

Aim

To estimate gravimetrically the amount of zinc present in the whole of the given solution of Zinc sulphate.

Principle

Zinc is precipitated as zinc oxinate in alkaline medium using 8 - hydroxyquinoline (oxine) as the precipitant.

$$ZnSO_4.7H_2O + 2\ C_9H_7ON \longrightarrow Zn\ (C_9H_6ON)_2 + 7\ H_2O + H_2SO_4$$

The precipitate is filtered, washed and dried at 130 – 140 ºC and weighed.

Procedure

The given solution of Zinc sulphate is made upto 100 mL in a standard flask. 20mL of the made up solution is pipetted out into a 250 mL beaker and diluted to 100 mL with distilled water. About 5 g of sodium tartrate and 25 mL of 2% alcoholic solution of oxine is added in the whole. The solution is then warmed at 60º C until the yellow precipitate becomes crystalline (if the supernatant liquid is not coloured more of the reagent is added till it becomes yellow). The precipitate is digested at 60-80ºC for 15 minutes and allow to stand for 10-20 minutes. The supernatant liquid is tested for completion of precipitation. The precipitate is then filtered through a previously weighed sintered crucible and washed several times with cold water containing a little NH_4OH. The precipitate is finally dried at 130 – 140ºC for one hour, cooled in a dessicator and weighed. A duplicate is conducted. From the weight of zinc oxinate, the weight of zinc is determined.

353.38 g of zinc oxinate contains 65.38 g of zinc

Result

Weight of zinc present in the whole of the given solution = _____ g.

Ex.No: 4

Date:

ESTIMATION OF COPPER AS COPPER THIOCYANATE

Aim

To estimate the amount of copper present in the whole of the given solution

Principle

The copper in a soluble copper salt solution is precipitated as cuprous thiocyanate by the addition of ammonium thiocyanate in the presence of sulphurous acid. The cuprous thiocyanate is collected in a sintered glass crucible, dried at 120°C and weighed. From the weight of cuprous thiocyanate, the amount of copper is estimated.

$$2CuSO_4 + SO_2 + 2H_2O + 2NH_4CNS \longrightarrow Cu_2(CNS)_2 + (NH_4)_2SO_4 + 2H_2SO_4$$

Procedure

The given Copper sulphate solution is made upto 100mL. 20mL of the made up solution is pipetted out into a clean beaker. About 20mL of dil.Hydrochloric acid (or sulphuric acid) and 10 to 15mL saturated solution of Sodium sulphite are added. The solution is diluted to about 100mL and boiled. To the hot solution, a freshly prepared 10% solution of ammonium thiocyanate (20mL) is added with constant stirring, when white cuprous thiocyanate is precipitated. The solution should be colourless and must smell strongly of sulphur dioxide if the precipitation has been correctly done. The precipitate is digested on a steam bath for about 30minutes. It is filtered through a previously weighed sintered crucible. It is washed with cold water containing ammonium thiocyanate and a little sulphurous acid. Finally the precipitate is washed with 20% alcohol to remove the thiocyanate. It is then dried at 120°C and weighed. Heating, cooling and weighing are repeated till a constant weight is obtained. A duplicate is conducted. From the weight of

cuprous thiocyanate, the weight of copper present in the whole of the given solution is calculated.

243.1g of cuprous thiocyanate contains 127.1g of copper

Result:

The weight of copper present in the whole of the given solution = _____ g.

Ex.No: 5

Date:

ESTIMATION OF CALCIUM AS CALCIUM OXALATE

Aim

To estimate the amount of calcium present in the whole of the given solution

Principle

The calcium from the calcium salt solution is precipated as calcium oxalate monohydrate by treating the hot solution of the calcium salt slightly acidified with hydrochloric acid, with a hot solution of ammonium oxalate and neutralising with ammonium hydroxide. The precipitate is collected in a sintered glass crucible, dried at 100°C and weighed as calcium oxalate monohydrate.

$$Ca^{2+} + (NH_4)_2\, C_2O_4 \longrightarrow CaC_2O_4 + 2NH_4^+$$

Procedure

The given calcium chloride solution is made upto 100mL. 20mL of the made up solution is pipetted out into a clean beaker. It is diluted to about 100mL. A drop of methyl orange is added when a pink colour is seen. The solution is carefully neutralised with ammonia (colour changes to yellow) and then 1mL of dil.hydrochloric acid is added. The solution is now heated to boiling to the hot solution. About 20mL of hot ammonium oxalate solution (5%) is added with vigorous stirring. Now dil.ammonium hydroxide is added till the mixture is distinctly alkaline. The colour of the solution should change to yellow. The precipitate is digested for about 30 minutes. The solution is tested for complete precipitation. Then the precipitate is filtered through a previously weighed sintered crucible. The precipitate is washed with water containing ammonium hydroxide, dried at about 100-110°C and then weighed as calcium oxalate monohydrate. A duplicate is performed. From the weight of calcium oxalate monohydrate, the weight of calcium in the whole of the given solution is calculated.

146.1g of calcium oxalate monohydrate contains 40.1g of calcium

Result

Weight of calcium present in the whole of the given solution = _____ g.

INORGANIC PREPARATIONS

Ex.No: 1

Date:

PREPARATION OF POTASH ALUM

$K_2SO_4 . Al_2 (SO_4)_3 . 24H_2O$

Aim

To prepare the crystals of Potash alum from K_2SO_4 and $Al_2 (SO_4)_3$.

Principle

Potash alum is a double salt which can be obtained by mixing equimolar aqueous solution of K_2SO_4 and $Al_2 (SO_4)_3$ and evaporating the resultant solution.

$$K_2SO_4 + Al_2 (SO_4)_3 + 24H_2O \longrightarrow K_2SO_4 . Al_2 (SO_4)_3 . 24H_2O$$

Chemicals required

Potassium sulphate - 2 g
Aluminium sulphate - 8 g
Dilute Sulphuric acid - 1 mL

Procedure

8g of Aluminium sulphate is dissolved in 20mL of water in a china dish and 1mL of dilute Sulphuric acid is added to it. It is then warmed to make the solution clear. To this solution 2g of Potassium sulphate is added and heated in a water bath for 30 minutes. The solution is cooled in ice. White crystals of potash alum are separated. It is filtered at pump and dried between filter papers.

Ex.No: 2

Date:

PREPARATION OF HEXAMMINE NICKEL (II) CHLORIDE

$[Ni\ (NH_3)_6]\ Cl_2$

Aim

To prepare pure crystals of Hexammine Nickel (II) chloride.

Principle

When ammonia is added to a solution of nickel (II) chloride, the ammonia molecules compete with the water in bonding Ni^{2+}. In this case ammonia forms a stronger bond than water and hence ammonia replaces the water according to

$$NiCl_2 . 6H_2O + 6NH_3 \longrightarrow [Ni\ (NH_3)_6]\ Cl_2 + 6\ H_2O$$

Chemical Required

Nickel chloride - 4g

Conc.NH_3 - 10mL

Solid NH_4Cl - 1g

Procedure

A concentrated solution of 4 g of Nickel choride in about 10 mL of con.NH_3 is taken in a test tube and cooled in running water. The precipitation of the crystal of the complex is completed by the addition of 1g of solid NH_4Cl. The precipitate is filtered off by air suction and dried in air.

Ex.No: 3

Date:

PREPARATION OF CUPRAMMONIUM SULPHATE (TETRAMMINE COPPER (II) SULPHATE)

$Cu(NH_3)_4\,SO_4\,.\,H_2O$

Aim

To prepare pure crystals of Cuprammonium Sulphate.

Principle

Cuprammonium Sulphate is a complex salt which is obtained by adding excess of ammonia to a solution of copper sulphate.

$$CuSO_4 + 4NH_3 + H_2O \longrightarrow [Cu(NH_3)_4\,SO_4\,H_2O]$$

Chemicals required

Copper Sulphate - 2 g
Conc. NH_3 - 5 mL
Alcohol - 7.5 mL

Procedure

To a solution of copper sulphate pentahydrate in water (about 7-10mL), added a concentrated ammonia solution (1:1) till a clear blue solution is obtained. In case the solution is turbid, added more ammonia solution. To the clear blue solution alcohol is added in drops with constant stirring. A dark blue precipitate of cuprammonium sulphate is formed. The solution is heated in a water bath at 50º for about 10 minutes to dissolve the precipitate. The solution is allowed to cool slowly. The crystals of cuprammonium sulphate are separated which was washed with alcohol and dried.

Ex.No: 4

Date:

PREPARATION OF PRUSSIAN BLUE

$Fe_4 [Fe(CN)_6]_3$

Aim

To prepare pure crystals of Prussian blue.

Principle

Prussian blue can be prepared by mixing Ferric chloride solution with Potassium ferrocyanide solution.

$$4\,FeCl_3 + 3K_4[Fe(CN)_6] \longrightarrow Fe_4[Fe(CN)_6]_3 + 12\,KCl$$

Chemicals required

Ferric chloride - 2 g

Potassium ferrocyanide - 5 g

Procedure

Ferric chloride and Potassium ferrocyanide are dissolved separately in water (3mL for each solution). The two solutions are then mixed gently. A blue coloured paste of Prussian blue is formed. It is filtered by decantation and dried between folds of filter paper.

Ex.No: 5

Date:

PREPARATION OF POTASIUM TRIOXALATOCHROMATE (III) TRIHYDRATE

$K_3[CR(C_2O_4)_3]$

Aim

To prepare pure crystals of Potasium trioxalatochromate(III)trihydrate

Procedure

It is prepared by treating potassium dichromate with oxalic acid and potassium oxalate.

$$2\ K_2C_2O_4 + 7\ H_2C_2O_4 + K_2Cr_2O_7 \longrightarrow 2\ K_3[Cr(C_2O_4)_3] + 6\ CO_2 + 7\ H_2O$$

Chemicals required

Potassium oxalate (monohydrate)	-	1g
Oxalic acid	-	2.3 g
Potassium dichromate	-	0.9 g
Acetone	-	5 mL

Procedure

About 1g of Potassium oxalate and 2.3 g of oxalic acid are dissolved in 35 mL of water in a 100 mL conical flask. To the solution 0.9 g of potassium dichromate is added in small portions with vigorous stirring. The solution is evaporated to approximately one-third of its original volume and allowed to stand. The crystals are then filtered under suction and washed with 5 mL of acetone and dried.

Ex.No: 6

Date:

PREPARATION OF POTASSIUM TRISOXALATOFERRATE(III)

$2K_3[Fe(C_2O_4)_3]$

Aim

To prepare pure crystals of Potassium trisoxalatoferrate (III).

Procedure

The photosensitive bright green crystals of Potassium trisoxalatoferrate(III) is obtained by the action of ferric chloride with potassium oxalate.

$$2FeCl_3 + 3H_2C_2O_4 + 3K_2C_2O_4 \longrightarrow 2\ K_3[Fe(C_2O_4)_3] + 6HCl$$

Chemicals required

Potassium oxalate solution (1.5 M) - 45 mL
Ferric chloride solution (1.5 M) - 15 mL

Procedure

About 45 mL of 1.5 M solution of Potassium oxalate is mixed with 15 mL of 1.5 M Ferric chloride solution in a beaker. The sides of the container are scratched well. The mixture is cooled in an ice bath when green crystals of Potassium trisoxalatoferrate(III) are separated. Then it is filtered through Buchner funnel, washed with an equivolume mixture of water and ethanol and finally with acetone. It is dried by continuous suction and pressing between filter paper.

Ex.No: 7

Date:

PREPARATION OF TRISTHIOUREA COPPER (I) SULPHATE

$[Cu(H_2NCSNH_2)_3]\ SO_4$

Aim

To prepare pure crystals of tristhioureacopper (I) sulphate.

Principle

The octahedral shaped crystals of tristhioureacopper (I) sulphate is obtained by the action of thiourea with coppersulphate.

$$H_2N{-}\underset{\underset{\|}{S}}{C}{-}NH_2 + CuSO_4 \cdot 5H_2O \longrightarrow [Cu(H_2NCSHN_2)_3]\ SO_4$$

Chemicals Required

Thiourea - 4g

$CuSO_4$ - 4g

Procedure

About 4 g of thiourea is dissolved in 25 mL of hot water and the solution is cooled to room temperature. The solution is shaken well by adding to it portions of cold solution containing 4 g of $CuSO_4$ in 20 mL of water. It is finally cooled thoroughly in running water until separate yellowish oil adheres to the side of the flask. It is decanted and the mother liquor is rejected. The oil is shaken vigorously with a solution containing 2 g of thiourea in 20 mL of water until crystallisation is complete. The crystals are filtered and washed with a small volume of water.

COURSE WORK

Ex.No: 1

Date:

ESTIMATION OF NICKEL AS NICKEL DIMETHYLGLYOXIME

Aim

To estimate gravimetrically the amount of nickel present in the whole of the given solution of nickel sulphate.

Principle

Nickel is precipitated as nickel dimethylglyoxime (Ni-DMG) in alkaline medium using an alcoholic solution of dimethylglyoxime as the precipitant.

$$NiSO_4 + 2\ DMG \longrightarrow Ni\ (DMG)_2 + H_2SO_4$$

The precipitate is filtered, washed and dried at 110ºC and weighed.

Procedure

The given solution of nickel sulphate is made upto 100 mL in a SMF. 20 mL of the made up solution is pipetted out into a 400 mL beaker. About 5 mL of 1:1 HCl is added and the solution is diluted to 200 mL with distilled water. The solution is heated to about 80ºC and to the hot solution about 20 mL of 1% alcoholic solution of DMG is added in drops with constant stirring. Immediately 15 mL of dil. NH_4OH solution is added drop by drop directly into the solution and not along the sides of the beaker till the solution is slightly alkaline (indicated by the smell of NH_3 coming out). The rosy red precipitate of Ni-DMG is digested over a hot water bath for half an hour (care is taken to avoid bumping). The completion of precipitation is tested by adding a drop or two drops of DMG to the supernatant liquid. The precipitate is allowed to settle by heating the beaker in a basin of cold water. The precipitate is filtered through a previously weighed sintered crucible.

The precipitate is washed several times with hot water till the washing is free from chloride. The precipitate is then dried in an air oven at 100ºC for an hour, cooled in

a dessicator and weighed. Heating, cooling and weighing are repeated till a constant weight is obtained. A duplicate experiment is conducted simultaneously.

288.69 g of Ni-DMG contains 58.69 g of nickel

Result

Weight of nickel present in the whole of the given solution = _____ g.

Ex.No: 2

Date:

DETERMINATION OF IRON IN VITAMIN TABLETS BY SPECTROPHOTOMETRY

Aim

To determine iron content in Vitamin tablets by spectrophotometry

Principle

Iron from a vitamin supplement tablet is dissolved in acid, reduces to Fe^{2+} with hydroquinone. The iron gets complexed with o-phenanthroline to form an intensely colored complex. The intensity of o-phenanthroline complex is determined spectrophotometrically at a λ_{max} of 508nm against iron standard.

Procedure

Place one tablet of the iron containing vitamin in a 125mL flask or 100mL beaker and boil gently (in a fume hood)with 25mL of 6 M HCl for 15minutes. Transfer directly into a 100mL volumetric flask. Wash the beaker several times with small portions of water to complete a quantitative transfer and make to the mark. Allow the solution to cool and filter. Dilute 5 mL of this solution to100mL in a fresh volumetric flask. If the label indicates that the tablet contains <15mg of Fe, use 10mL instead of 5mL. Pipette out 10mL of diluted Fe solution into a beaker and measure the pH (with pH paper or a glass electrode). Add sodium citrate solution dropwise until a pH of 3.5 is reached. Transfer to a 100mL volumetric flask and add 2mL of hydroquinone solution and 3mL of o-phenanthroline solution, dilute to the mark with water, and mix well. Prepare three more solutions from 5,2 and 1mL of Fe standard (FAS solution) and prepare a blank containing no Fe. Use sodium citrate solution in proportion to the volume of Fe solution for pH adjustment to pH 3.5. Allow the solution to stand for atleast 10 minutes. Then measure the absorbance of each solution at 508nm. (The color is stable, so all solutions

may be prepared and all the absorbances measured at once). Use distilled water in the reference cuvette and subtract the absorbance of the blank from the absorbance of the Fe standards.

Result

Content of iron in vitamin tablet = _____ mg.

Ex.No: 3

Date:

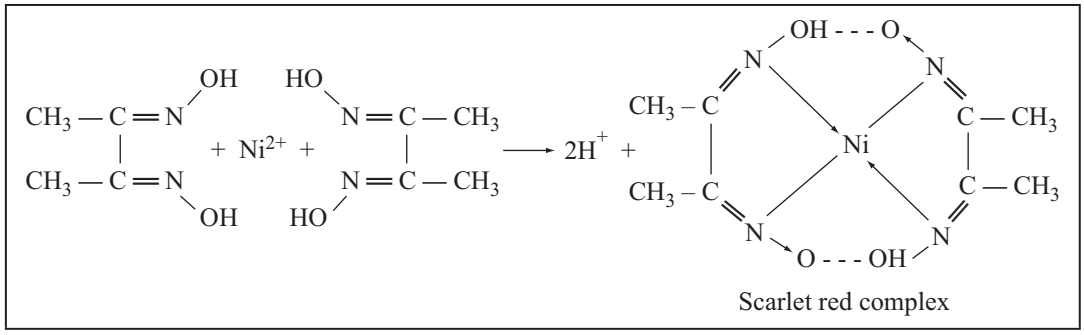
ESTIMATION OF NICKEL BY SPECTROPHOTOMETER

Aim

To estimate spectrophotometrically the weight of ferric ion in milligrams present in the whole of the given solution.

Principle

Ni is known to produce a red colour with the DMG in alkaline medium.



Scarlet red complex

Procedure

Weigh accurately about 0.1682gm of Nickel ammonium sulphate $NiSO_4$ $(NH_4)_2$ $SO_4.6H_2O$ and dissolve it in distilled water and make the volume to 250mL. Take 10mL of the made up solution and dilute to 100mL in a standard flask. Take 1,2,3 mL of the madeup solution in different cuvettes. Add 1mL of Br_2/H_2O and 2mL of 7N NH_4OH and shake the solution followed by the addition of 1mL of 1%DMG and dilute it to 10mL. The produced red colour is measured using a filter (470nm). Record the absorbance. A graph is plotted by taking the absorbance against the concentration. Make up the given solution to 100mL. Take different volumes in separate cuvettes and repeat the procedure as above. Read the absorbance correspondingly.

Result

Weight of Nickel present in the whole of the given solution = _____ mg.


ALLIED
BIOCHEMISTRY
200 ml
APPROX.
150
100
50

ALLIED BIOCHEMISTRY PRACTICAL

Subject Code: 18UBCAR1

CONTENTS

Analysis of Biomolecule

Volumetric Analysis

ANALYSIS OF BIOMOLECULE

Qualitative Analysis of Carbohydrate, Amino acids and Proteins

Experiment	Observation	Inference
Preliminary Test		
1. Colour and appearance	Colourless liquid	Presence of aminoacids or proteins or carbohydrate.
2. Action of conc. H_2SO_4 A small amount of the substance is heated with conc. H_2SO_4.	Charring with effervescence	Presence of carbohydrates.
	No Charring takes place	Absence of carbohydrates.
3. Molisch's Test To about few mL of the substance, two drops of Molisch's reagent is added and shaken well. 2mL of conc. H_2SO_4 is added carefully along the sides of the test tube.	Violet ring	Presence of carbohydrates.
	No violet ring	Absence of carbohydrates.
4. Ninhydrin Test To 1mL of the solution, 1mL of 2% ninhydrin is added. It is then heated in a water bath for 5 mins.	Purple colour (Rheumann's purple)	Presence of amino acids or proteins.
	No characteristic colour change	Absence of amino acids and proteins.
5. Biuret Test To 1mL of the solution, 5 drops of biuret reagent is added and mixed well.	Violet or purple colour	Presence of proteins.
	No characteristic colour change	Absence of proteins.
Tests For compounds Containing carbohydrate		
1. Fehling's Test To few mL of the solution, 2mL of Fehling's solution (1mL of Fehling's A and 1mL of Fehling's B) is added. It is then boiled in a water bath for 5 mins.	Reddish brown precipitate	Presence of monosaccharide.

Experiment	Observation	Inference
	No reddish brown precipitate	Absence of monosaccharide.
2. Benedict's test To 1mL of the solution, 2 drops of Benedict's solution is added. It is then boiled in a water bath for 5 mins.	Colour change from blue to green, yellow, orange or red	Presence of monosaccharide.
	Solution remains blue	Absence of monosaccharide.
3. Tollen's test To 1mL of the solution, 2mL of Tollen's reagent (1mL of Tollen's A and 1mL of Tollen's B) is added to the test tube It is then boiled in a water bath for 5 mins.	Bright silver mirror or black precipitate	Presence of monosaccharide.
	No silver mirror	Absence of monosaccharide.
4. Iodine test To about 1mL of the solution , 2 drops of 0.1N HCl and 2 drops of iodine solution are added.	Blue colouration	Presence of polysaccharide.
	No characteristic colour change	Absence of polysaccharide.
Test for monosaccharide		
1. Barfoed's Test To 1mL of the solution, 2mL of Barfoed's solution is added.It is then boiled in a water bath for 5mins.	Brick red precipitate (within 5 mins.)	Presence of monosaccharide.
	No brick red precipitate	Absence of monosaccharide.
2. Anthrone test To 1mL of the solution, 2 drops of anthrone reagent is added along the sides of the test tube and shaken well. (If there is no colour change keep it in a water bath)	Green colouration	Presence of monosaccharide (glucose).
	No characteristic colour change	Presence of monosaccharide.

Experiment	Observation	Inference
3. Seliwanoff's test To 1mL of the solution, 3 drops of Seliwanoff's reagent is added and boiled in a water bath for 5 mins.	Cherry red colour	Presence of Ketose (Fructose).
	No characteristic colour	Absence of Ketose (Fructose).
4. Fougler's test To one drop of a solution in a test tube, 3 drops of Foulger's reagent is added and boiled for 2mins.	Deep blue colour	Presence of Fructose.
	No characteristic colour change	Absence of Fructose.
5. Bial's test To 2mL of the solution in a test tube, 5 drops of Bial's reagent is added. It is then boiled in a water bath for 5 mins.	Green colour will appear within 10 minutes.	Presence of Pentose.
	No green colour	Absence of Pentose.
6. Osazone Test To 2mL of the solution, 2 drops of phenylhydrazine reagent is added. It is then boiled for 5 minutes.	Yellow crystalline precipitate is formed within 4 minutes	Presence of Fructose.
	Within 10 minutes, Yellow crystalline precipitate is formed	Presence of Glucose.
	After 30 minutes, Yellow crystalline precipitate will appear.	Presence of Galactose.
	After boiling the solution for 20 minutes, Yellow crystalline precipitate will appear. (crystals appear after cooling the hot solution)	Presence of Maltose.
	Yellow crystalline precipitate is formed only after 20 minutes of boiling (ball like structure appear after cooling the hot solution)	Presence of Lactose.

Experiment	Observation	Inference
Tests For Compounds Containing Amino acids/ Proteins		
1. Xanthoproteic test To about 1mL of the solution in a test tube, 0.5mL of conc. HNO_3 is added, boiled, cooled. To this excess of 40% NaOH is added.	Yellow colour solution	Presence of aromatic amino acids or proteins with aromatic acids (Tyrosine or Tryptophan).
	No characteristic colour change	Presence of aliphatic amino acids.
2. Pauly's test To 1mL of the solution in a test tube, 1 drop of sulphanilic acid is added and cooled in ice. To this, add 1 drop of sodium nitrite solution, heat, cool and add 2 drops of 1% Na_2CO_3 solution.	Red colour	Presence of Tyrosine and Tryptophan.
	No red colour	Absence of Tyrosine and Tryptophan.
3.Millon's test (Modified Millon's Test) To 1mL of the solution in a test tube,0.5mL of Millon's reagent is added and boiled in a water bath for 10 mins.Cool the mixture and add 5 drops of 1% sodium nitrite solution.	Red colour	Presence of Tyrosine or protein with Tyrosine.
	No red colour	Absence of Tyrosine or protein with tyrosine.
4. Ehrlich's Test To 1mL of the solution, add 1 drop of Ehrlich's reagent.	Deep red colour	Presence of Tryptophan.
	No deep red colour	Absence of Tryptophan.
5. Hopkins-Cole Test To 1mL of the solution added 2mL of glacial acetic acid, mixed well and then carefully added 2 drops of conc. H_2SO_4 along the sides of the test tube.	Violet ring at the junction of the two liquids.	Presence of tryptophan or protein with tryptophan.
	No characteristic colour change	Absence of protein.

Experiment	Observation	Inference
6. Sodium Nitroprusside Test To 1mL of the solution 5 drops of freshly prepared 2% solution of sodium nitroprusside and 5 drops of 10% NaOH is added.	Red colour	Presence of Cysteine and Cystine.
	No red colour	Absence of Cysteine and Cystine.
7. Sulphur Test To 1mL of the solution in a test tube, add 2 drops of 40% NaOH and 1 drop lead acetate solution. The test tube is boiled for a minute and cooled.	Brown colour changes to black	Presence of Cysteine or protein with Cysteine.
	No characteristic colour change	Absence of protein.
8. Sakaguchi Reaction To 3mL of the solution in a test tube, add 1drop of 10% NaOH and 2 drops of 1% α-napthol in alcohol. After a few minutes add 1 drop of sodium hypobromite solution (Br_2 in NaOH).	Intense red colour	Presence of Arginine or protein with Arginine.
	No red colour	Absence of protein.

VOLUMETRIC ANALYSIS

Titration I

Standardisation of NaOH **Std Oxalic acid Vs. NaOH**

S.No.	Volume of NaOH (mL)	Burette Reading (mL)		Volume of Oxalic acid (mL)	Indicator
		Initial	Final		
1.	20.0	0			
2.	20.0	0			

Volume of Oxalic acid (V_1) = mL

Strength of Oxalic acid (N_1) = N

Volume of Sodium hydroxide (V_2) = mL

Strength of Sodium hydroxide (N_2) = N

Strength of Sodium hydroxide (N_2) = $\frac{V_1 N_1}{V_2}$

= _____ N

Titration II

Formaldehyde Vs. NaOH

S.No.	Volume of formaldehyde (mL)	Burette Reading (mL)		Volume of NaOH (mL)	Indicator
		Initial	Final		
1.	20.0				
2.	20.0				

Ex.No: 1

Date:

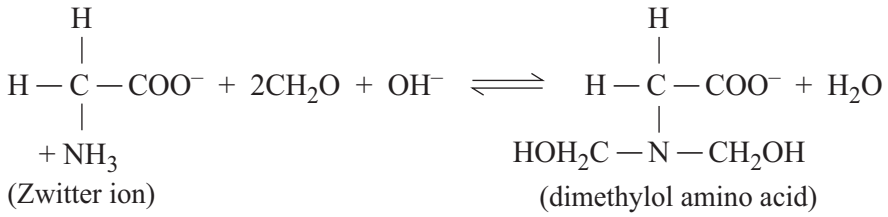
ESTIMATION OF GLYCINE BY FORMAL TITRATION

Aim

To estimate the amount of glycine present in the whole of the given solution by formal titration. You are provided with exactly 0.1N oxalic acid solution and approximately decinormal solution of sodium hydroxide solution.

Principle

Amino acids contain amino group and carboxyl group. The carboxyl group of α-amino acids react with the basic amino groups to form zwitter ions. Zwitter ions are held together by electrostatic attraction. The zwitter ions are not completely decomposed at the end point of alkaline indicators such as phenolphthalein. When amino acid solutions are treated with large excess of neutralised formaldehyde the amino group combines with formaldehyde to form dimethylol amino acid. This reacts with alkali in the presence of phenolphthalein indicator to give a sharp end point.



$$\text{Equivalent weight of glycine} = \frac{\text{Molecular Weight}}{1} = \frac{75}{1} = 75$$

Procedure

Titration I: Standardization of NaOH

Pipette out 20mL solution hydroxide solution into a clean conical flask. Add a drop of phenolphthalein indicator. Titrate this solution against the standard oxalic acid taken in

Titration III

Estimation of Glycine

NaOH Vs. Given glycine

S. No.	Volume of glycine (mL) + 10mL of formalin (mL)	Burette Reading (mL)		Volume of NaOH (V_y) (mL)	Indicator
		Initial	Final		
	20.0				
	20.0				

Volume of Sodium hydroxide (V_1) = $(V_y - V_x)$ mL

Strength of Sodium hydroxide (N_1) = N

Volume of glycine (V_2) = 20 mL

Strength of glycine (N_2) = $\frac{V_1 N_1}{V_2}$

= _____ N

Weight glycine present in the whole of the given solution = $\frac{N \times 75}{10}$ g

= _____ g

the burette. The end point is the disappearance of pink colour. Repeat the titration for concordant values. From the titre value, the strength of sodium hydroxide solution can be determined.

Titration II: Formaldehyde Versus Sodium hydroxide

Pipette out 10mL of formalin and 20mL of water into a conical flask. Keep the mixture as such with occasional shaking. After 10 minutes, add a drop of phenolphthalein indicator. Titrate this mixture against the sodium hydroxide taken in the burette. The end point is the appearanceof pale permanent pink colour. Repeat the titration for concordant value.

Titration III: Estimation of Glycine

Make up the given solution of glycine in a 100mL standard flask. Pipette out 20mL of this made up glycine into a conical flask. Add 10mL of formalin shake the contents well and allow the reaction to take place for 10 minutes. Now add a drop of phenolphthalein indicator. Titrate the contents against the sodium hydroxide taken in the burette. The end point is the appearance of pale, permanent pink colour. Repeat the titration for concordant value. From the titre value, the strength and hence the weight of glycine in the whole of the given solution can be calculated.

Result

Weight of glycine present in the whole of the given solution = _____ g.

Titration I

Standardisation of dye **Standard ascorbic acid Vs. Dye solution**

S.No.	Volume of standard ascorbic acid (mL)	Burette Reading (mL)		Volume of dye (mL) (V_1)
		Initial	Final	
1.	20.0	0		
2.	20.0	0		

1 mL of standard ascorbic acid contains 0.05 mg of ascorbic acid

20 mL of standard ascorbic acid contain = $20 \times 0.05 = 1.0$ mg

(V_1) _____ mL of dye = 1.0 mg of ascorbic acid

1 mL of dye = $\frac{1.0}{(V_1)}$ mg of ascorbic acid

= _____ mg.

Ex.No: 2

Date:

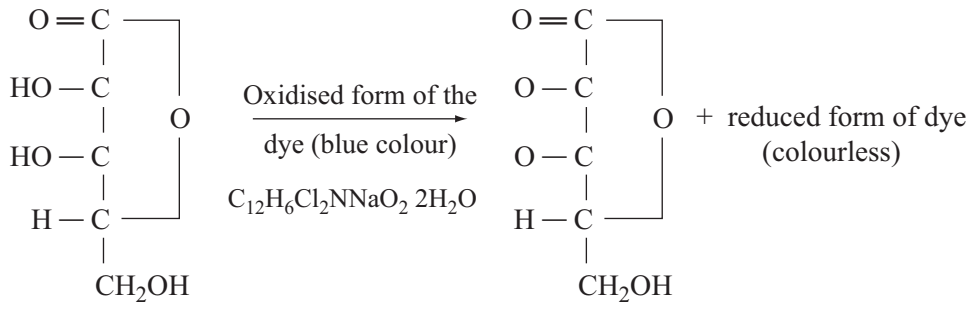
ESTIMATION OF ASCORBIC ACID

Aim

To estimate the amount of ascorbic acid (Vitamin C) present in the given food sample. You are provided with a standard solution of ascorbic acid containing 0.050 mg/mL and approximately 0.025% solution of sodium 2,6-dichlorophenol indophenol dye as link solution.

Principle

The oxidizing agent 2, 6-dichlorophenol indophenol oxidises ascorbic acid into dehydro ascorbic acid. During this oxidation, the oxidized form of the dye (blue colour) gets reduced to colourless substance. The dye is pink colour in acidic medium.



L - ascorbic acid

L - dehydroascorbic acid

Procedure:

Titration I: Standardisation of 2,4-dichlorophenol indophenol (dye)

Wash and rinse the burette with the dye and fill up the burette with the dye. Wash and rinse the pipette with the standard ascorbic acid. Pipette out 20 mL of ascorbic acid into a clean conical flask and titrate against the dye. The end point is the appearance of pink colour which persists for 10 seconds. Repeat the titration for concordant value.

Titration II

Estimation of Unknown ascorbic acid

Unknown ascorbic acid Vs. Dye solution

S.No.	Volume of Unknown ascorbic acid (mL)	Burette Reading (mL)		Volume of dye (mL) (V_2)
		Initial	Final	
1.	20.0			
2.	20.0			

1 mL of dye = $\frac{1.0}{(V_1)}$ mg of ascorbic acid

(V_2) …….. mL dye = $\frac{1.0}{(V_2)}$ mg of ascorbic acid

20 mL of unknown solution of ascorbic acid contain = _____ mg of ascorbic acid

100mL of unknown solution of ascorbic acid contain = $\frac{1.0 \times V_2}{V_1} \times 5mg$ of ascorbic acid

= _____ mg of ascorbic acid

Titration II: Estimation of Unknown ascorbic acid

Make up the given unknown ascorbic acid solution to 100 mL in a standard flask. Pipette out 20 mL of this made up solution into a clean conical flask. Titrate it against the dye taken in the burette as before. The end point is the appearance of pink colour which persists for 10 seconds. Repeat the titration for concordant value.

Result

The weight of ascorbic acid present in the whole of the given solution = _____ mg.

Table 6.3.1 Estimation of Protein by Biuret method

Volume of Protein (mL)	Volume of Water (mL)	Volume of Biuret reagent (mL)	Optical Density (% T)	Concentration (mg)
1	4	6		
1.5	3.5	6		
2	3	6		
2.5	2.5	6		
3	2	6		
3.5	1.5	6		
4	1	6		
4.5	0.5	6		
5	0	6		
Unknown I		6		
Unknown II		6		

Ex.No: 3

Date:

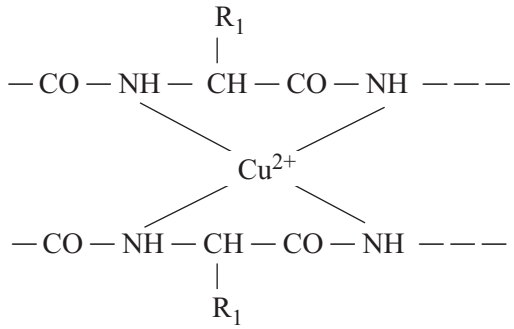
ESTIMATION OF PROTEIN BY BIURET METHOD

Aim

To estimate the amount of protein present in the whole of the given sample of serum by biuret method.

Principle

Cu^{2+} in alkaline solution complexes with nitrogen atoms of the peptide bonds in proteins. This complex gives a purple colour. The colour is measured at 520 n.m (green filter).



Procedure

Prepare a standard solution of protein (stock solution) by dissolving 15 g of Bovine serum albumin in 250 mL of distilled water. Prepare a working standard by diluting 10 mL of stock solution into 100 mL in a standard flask using distilled water. This working standard contains 6 mg of protein / mL. Pipette out into a series of tubes (S_1 to S_{10}) 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 mL of the protein solution and make up the total volume to 5.0 mL with addition of distilled water. A blank tube (B) will contain only 5.0 mL of water. Add 6.0 mL of biuret reagent to each tube and mix well. Keep the test tubes at room temperature for 10 minutes. Measure the optical density of each tube at 520 n.m (green filter) using the reagent blank. Draw a standard graph using concentration

along x-axis and optical density along y-axis. Make up the given serum to 100 mL in a standard flask. Pipette out 2 mL and 4 mL from this made up solution into different test tubes (T_1 and T_2) and make up the volume to 5 mL with water and repeat the above same procedure with these test solutions also. Then plot the standard graph using the Optical density obtained for the test solutions. This gives the concentration of protein in the test solutions. From this, calculate the amount of protein present in the sample of serum given.

Result

Concentration of unknown protein I = _____ mg

Concentration of unknown protein II = _____ mg.

Titration I

Standardisation of HCl **Standard Na_2CO_3 Vs. HCl**

S.No.	Volume of Na_2CO_3 (mL)	Burette Reading (mL)		Volume of HCl (mL)	Indicator
		Initial	Final		

Concordant value = _____

Volume of Na_2CO_3 (V_1) = mL

Strength of Na_2CO_3 (N_1) = N

Volume of HCl (V_2) = mL

Strength of HCl (N_2) = $\frac{V_1 N_1}{V_2}$

= N

Titration II

Test Vs. 0.5N HCl

S.No.	Volume of test solution	Burette Reading (mL)		Volume of HCl (mL)
		Initial	Final	
	1g of oil + 100mL of ethanol/ether Mixture + 25mL of 0.5N alco. KOH			

Ex.No: 4

Date:

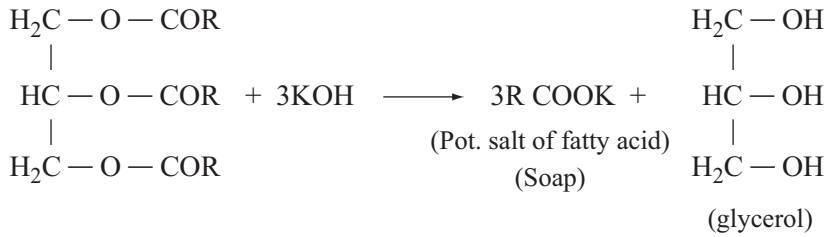
DETERMINATION OF SAPONIFICATION NUMBER OF OIL

Aim

To estimate the saponification number of the given oil. You are provided with 0.5N HCl.

Principle

When oil (triacyl glycerol) is heated with KOH, it is saponified (hydrolysed) and releases fatty acid and glycerol.



Fatty acids neutralise the potassium hydroxide and the titration with HCl detects the amount of alkali used for the saponification. The milligrams of KOH required to saponify 1 g of oil is defined as the saponification number of the oil.

Procedure

Titration I

Standardisation of HCl

A clean burette is washed with water and rinsed with the HCl and filled with the same. 20mL of the standard sodium carbonate solution is pipetted out into a clean conical flask and titrated against the hydrochloric acid using methyl orange indicator. The end point is the colour change from golden yellow to pale pink. The titration is repeated for concordant value.

Titration III

Blank Vs. 0.5N HCl

S.No.	Volume of test solution	Burette Reading (mL)		Volume of HCl (mL)
		Initial	Final	
	100mL of ethanol/ether mixture + 25mL of 0.5N alco.KOH			

Volume of _____ N HCl required for the blank = mL (V_1)

Volume of _____ N HCl required for the Test = mL (V_2)

The difference in the volume of _____ N HCl required by the test and blank ($V_2 - V_1$) = Volume of 0.5N KOH required for saponification

Saponification number of the given sample oil = $\frac{56 \times (V_2 - V_1) \times \quad N}{1}$

= _____ .

From the titre value the strength of HCl is calculated.

Titration II

Test Vs 0.5N HCl

Weigh out exactly 1 g of oil into a 250 mL conical flask and about 10 mL of ethanol / ether mixture (2:1 V/V). Add exactly 25 mL of 0.5N alcoholic KOH and fit it with an air-condenser. Keep the flask for 30 minutes in a boiling water bath (Test). Cool the contents. Titrate the test against exactly 0.5N HCl using phenolphthalein as indicator. The endpoint is just disappearance of pink colour.

Titration III

Blank Vs 0.5N HCl

About 10 mL of ethanol / ether mixture (2:1 V/V) is taken in a 250 mL conical flask. Add exactly 25 mL of 0.5N alcoholic KOH and fit it with an air-condenser. Keep the flask for 30 minutes in a boiling water bath (Blank). Cool the contents. Titrate the blank against exactly 0.5N HCl using phenolphthalein as indicator. The endpoint is the just disappearance of pink colour.

From the difference between the titre values of the blank and the test solutions, calculate the amount of KOH in mg that has been used by 1 g of oil for the saponification process. This is known as the saponification value of the given sample of oil.

Result

The saponification number of the given sample of oil is _____

Titration I

Standardisation of $Na_2S_2O_3$ **Std. $K_2Cr_2O_7$ Vs. $Na_2S_2O_3$**

S.No.	Volume of $K_2Cr_2O_7$ (mL)	Burette Reading (mL)		Volume of HCl (mL)	Indicator
		Initial	Final		
1.					
2.					

Concordant value = _____

Volume of $K_2Cr_2O_7$ (V_1) = 20 mL

Strength of $K_2Cr_2O_7$ (N_1) = 0.1 N

Volume of $Na_2S_2O_3$ (V_2) =

Strength of $Na_2S_2O_3$ (N_2) = $\frac{V_1 N_1}{V_2} = 20 \times 0.1 =$ _____N

= _____ N

Titration II

Estimation of Iodine number **Test Vsthio**

Volume of test solution	Burette Reading (mL)		Volume of HCl (mL)	Indicator
	Initial	Final		
1g of oil + 10mL of chloroform + 80mL of Iodine solution + 10mL of 15% KI solution				

Ex.No: 5

Date:

DETERMINATION OF IODINE NUMBER OF OIL

Aim

To estimate the iodine number of the given sample of oil. You are provided with approximately decinormal thio sulphate solution.

Principle

The fatty acids in triacylglycerols may be saturated or unsaturated. The amount of unsaturation can be determined by measuring the amount of iodine taken up by the unsaturated fatty acids. Thus iodine value is a measure of the degree of unsaturation of an oil. It is a constant for a particular oil or fat. Higher the iodine value (higher unsaturation) greater the possibility of the oil to go rancid.

Procedure:

TitrationI: Standardization of thiosulphate:

Pipette out 20 mL of standard (0.1N) potassium dichromate solution into a conical flask. Add 5 mL of conc.HCl and 1 test tube of 10% KI. Titrate this mixture against thiosulphate till the solution turns straw yellow in colour. Now add 1 mL starch and continue the titration. The endpoint is the colour change from blue to green. Repeat the titration for concordant value. From the titre value find out the strength of thiosulphate.

Titration II: Estimation of iodine number:

Weigh out 1g of oil (1.2 mL of coconut oil weigh 1g) into an iodine flask and dissolve in 10 mL of chloroform. Add 80 mL of iodine solution and allow to stand with occasional shaking for 30 minutes. Add 5 mL of water between the mouth and the lid of the iodine flask. Add 10mL of 15% KI solution, shake well and then add 100 mL of water. Titrate against thiosulphate till the yellow colour just disappears. Add starch (1 mL) and continue

Titration III

Blank Vs. 0.5N thio

Volume of test solution	Burette Reading (mL)		Volume of HCl (mL)	Indicator
	Initial	Final		
10mL of chloroform + 80mL of Iodine solution + 10mL of 15% KI solution				Starch

Volume of thiosulphate required for blank = _____ mL (V_1)

Volume of thiosulphate required for the test = _____ mL (V_2)

The difference in the volume of thiosulphate required ($V_1 - V_2$) mL = amount of Iodine absorbed

The thiosulphate equivalents of iodine absorbed by 1g of oil $= \frac{(V_1 - V_2) \times \text{St. of thio} \times 127}{1}$

Iodine number of the given sample of oil $= \frac{(V_1 - V_2) \times \text{St. of thio} \times 127 \times 100}{1}$

= _____.

the titration. The end point is the colour change fron blue to colourless. Repeat with a blank with no oil.

Result

Iodine number of the given sample of oil is _____.

Table 6.5.1 Determination of pH using pH meter

$P^H = P^{Ka} + \log_{10} \frac{[Salt]}{[acid]}$ (P^{Ka} for acetic acid = 4.76)

Sl.No	Volume of 0.2M acetic acid (mL)	Volume of 0.2M sodium acetate (mL)	[acid]	[Salt]	$\log_{10} \frac{[Salt]}{[acid]}$	Calculated pH	Measured pH
A	40	10	0.08	0.02	$\log_{10}$ 0.02/0.08		
B	30	20	0.06	0.04	$\log_{10}$ 0.04/0.06		
C	20	30	0.04	0.06	$\log_{10}$ 0.06/0.04		
D	10	40	0.02	0.08	$\log_{10}$ 0.08/0.02		

Ex.No: 6

Date:

PREPARATION OF A BUFFER AND DETERMINATION OF ITS pH USING pH METER

Aim

To determine the pH value of different solutions using pH meter.

Principle

The substance which resists any change in its pH even after the addition of either small amount of acid (or) a base is known as buffer. Example: Mixture of weak acid and its salt with a strong base (or) mixture of weak base and its salt with a strong acid. Acetate buffer consists of acetic acid and sodium acetate. The pH of a buffer solution depends up on the pK_a/pK_b value of the acid (or) base and the logarithmic ratio of [salt] / [acid] (or) [salt] / [base]

(i.e) $pH = pK_a + \log_{10}$ [salt] / [acid] (or) $pH = pK_b + \log_{10}$ [salt] / [base]

This equation is known as Henderson – Hasselbalch equation.

Procedure

Preparation of a buffer

Prepare a stock solution of 0.2M acetic acid by taking 11.5mL of glacial acetic acid and making it up to one litre. Similarly prepare a stock solution of 0.2M sodium acetate by dissolving 27.2g of sodium acetate trihydrate (or) 16.4g of anhydrous sodium acetate in one litre of water.

Take different volume of acid and salts such as 40 mL / 10 mL, 30mL / 20mL, 20mL / 30mL and 10mL / 40mL and dilute to a total of 100mL, calculate the pH of buffer solution using Henderson – Hasselbalch equation.

Determination of pH using pH meter

The calomel and glass electrodes which are available in the pH meter are dipped into standard buffer solution of known pH. The dial should read this value. Otherwise adjustment is made with the suitable knob. The electrodes are now removed, washed well with distilled water and dipped into the buffer solution whose pH is to be measured. The dial reads the pH value. The electrodes are now removed, washed well with distilled water and dipped into the second unknown buffer solution to find out its pH. Similarly the P^H values of other two buffer solutions can also be measured.

Result

The pH values of different buffer solutions are as follows:

Solution	Calculated pH	Measured pH
A		
B		
C		
D		

BASICS OF EXPERIMENTS

Qualitative Analysis:

Qualitative analysis is the determination of chemical composition of a sample.

Quantitative Analysis:

Quantitative analysis is the determination of amount of substance present in a sample.

Solution:

A homogeneous mixture of two or more substances which may be solids, liquids, gases, or a combination of these.

Solvent:

A substance in which another substance is dissolved, forming a solution.

Solute:

The substance dissolved in a solvent in forming a solution.

Strength:

The amount of solute in gram present in one litre of the solution.

Normality:

Normality is the number of gram equivalents of the substance dissolved per litre of the solution. It is denoted by N.

Molarity:

Molarity is the number of moles of solute per litre of the solution. It is denoted by M.

Molality:

Molality is the number of moles of the substance dissolved in 1000gms of the solvent.

Standard solution:

A solution whose concentration is known.

Equivalent weight:

Equivalent weight is the mass of an element or compound that could combine with or displace one gram of hydrogen.

Molecular weight:

Molecular weight is the relative average weight of a molecule of a substance, expressed by a number equal to the sum of the atomic weights of all atoms in the molecule.

Mole:

The quantity of a chemical substance having a weight in grams numerically equal to its molecular weight.

Saponification Number:

The number of milligrams of potassium hydroxide (KOH) required to saponify one gram of fat

Iodine Number:

The mass of iodine in grams that is consumed by 100 grams of a chemical substance is called Iodine number. With the help of Iodine number, unsaturation in fats, oils and waxes can be determined.

pH:

The scale used to denote the acidity or basicity of a sample.

Biomolecule:

It is a chemical found in living organisms which are the building blocks of life.

Carbohydrate:

Carbohydrates are the sugars, starches and fibers found in fruits, grains, vegetables and milk products. A carbohydrate is a biomolecule consisting of carbon, hydrogen and oxygen atoms.

Aminoacids:

Amino acids are organic compounds that contain amine and carboxyl functional groups. They are the building blocks of proteins.

Proteins:

An essential nutrients for the human body which are the building blocks of body tissue.

Water bath:

An equipment which maintain samples in water at a constant temperature over a long period of time.

Monosaccharide:

Simplest form of sugar. Colourless water soluble solids. They are soluble in water due to the presence of OH groups.

Example: Glucose, Fructose, Galactose.

Disaccharide:

Two monosaccharides combine to form a disaccharide. They are soluble in water.

Example: Sucrose, Lactose and Maltose.

Polysaccharide:

A large molecule made up of many Polysaccharide.

Example: Starch

PREPARATION OF REAGENTS

Qualitative Analysis

S.No	Reagent	Quantity	Preparation
1.	Molisch Reagent	100mL	10g 1-napthol in 100mL ethanol
2.	Iodine solution	100mL	4g potassium iodide + 2g Iodine (powdered and dissolved in 100 mL water)
3.	Fehling's A	100 mL	6.9g copper sulphate dissolved in water and made upto 100 mL
4.	Fehling's B	100 mL	25g sodium potassium tartarate + 10g Sodium hydroxide
5.	Benedict's Reagent	250	**Solution 1:** 43.25 g sodium citrate + 25g sodium carbonate (Dissolved in 200mL water and filtered)
			Solution 2: 4.33g Copper sulphate in 20mL water.
			Solution 1 and 2 are mixed and made upto 250mL
6.	Barfoed's reagent	100mL	6.6g copper acetate in 100mL water + 0.9 mL glacial acetic acid
7.	Tollen's reagent (A)	100mL	50mL ammonia + 50mL water + 20g silver nitrate
8.	Tollen's reagent (B)	100mL	10g NaOH in 100mL water
9.	Anthrone reagent	100mL	200mg anthrone in 100mL Con. H_2SO_4
10.	Seliwanoff's reagent	100mL	300mg resorcinol in 100 mL Con.HCl

S.No	Reagent	Quantity	Preparation
11.	Bial's Reagent	100mL	300mg orcinol in 100 mL Con.HCl
12.	Phenyl Hydrazine Reagent	16mL (amber bottle)	4g phenyl hydrazine hydrochloride + 16mL water + warmed + cooled + 6g sodium acetate + 1drop glacial acetic acid
13.	Fougler's reagent	100mL	40g urea + 80mL H_2SO_4 (40%) + 2g stannous chloride + Boiled + (till clear) + Cooled + Made upto 100mL with 40% H_2SO4
14.	Millon's reagent	100mL	15g mercuric sulphate in 100mL 15% H_2SO4
15.	Ehrlich's reagent	100mL	1g p-dimethyl amino benzaldehyde. In 50mL ethanol + 50mL Con. HCl
16.	Sodium hypobromite solution	100mL	20g NaOH in 75mL water + 5mL bromine Dilute to 100mL
17.	Biuret Reagent	100mL	0.15g copper sulphate + 0.6 sodium potassium tartarate + 50mL water + 30mL 10% NaOH (made upto 100mL)
18.	lead acetate solution	100mL	9.5g lead acetate in 100mL water

Quantitative Analysis

I. Estimation of Glycine by formal titration

S.No	Name of the reagent	Quantity	N	Preparation
1.	NaOH	1 Litre	0.05	2g in 1000mL water
2.	Oxalic acid	1 Litre	0.05	3.15g in 1000mL water
3.	Glycine	1 Litre	0.25	18.75g in 1000mL water

II. Estimation of Ascorbic acid

S.No	Name of the reagent	Quantity	N	Preparation
1.	Standard Ascorbic acid	1 Litre	1	66mg in 1000mL water

III. Estimation of Protein by Biuret method

S.No	Name of the reagent	Quantity	N	Preparation
1.	Biuret reagent	1 Litre	—	3g copper sulphate + 9g sodium potassium tartarate in 500mL of 0.2N NaOH + 5g KI (made upto 1L with 0.2N NaOH)
2.	Bovine	100 mL	—	0.6g in 100mL water

IV. Determination of Saponification number of oil

S.No	Name of the reagent	Quantity	N	Preparation
1.	HCl	1 Litre	0.5	50mL in 1000mL water
2.	Na_2CO_3	1Litre	0.5	26.5g in 500mL water
3.	Alcoholic KOH	100 mL	0.5	28g KOH in alcohol
4.	Ethanolic – ether mixture	80 mL	—	55mL ethanol + 25mL ether

V. Determination of Iodine number of oil

S.No	Name of the reagent	Quantity	N	Preparation
1.	Thio solution	1 Litre	0.1	24.84g thio in 1000mL water
2.	$K_2Cr_2O_7$	1Litre	0.5	4.9g in 1000mL water
3.	KI solution	100 mL	10%	10g KI in 100mL water
4.	Iodine solution	1L	—	13.2g iodine in 1L glacial acetic acid + 3mL bromine

Important Molecular weights

Molecular Formula	Molecular Weight
Na_2CO_3	106
KOH	56.1056
HCl	36.5
NaOH	40
$K_2Cr_2O_7$	294.18
H_2SO_4	98
HNO_3	63
Oxalic acid	90.03

Important Equivalent weights

Molecular Formula	Molecular Weight
$Na_2S_2O_3.5H_2O$	248.21
$K_2Cr_2O_7$	49
Oxalic acid	63
Ascorbic acid	176
Glycine	75

ALLIED CHEMISTRY PRACTICALS

ALLIED CHEMISTRY PRACTICAL

Subject Code: 18UCHAR1/18UCHAR2

CONTENTS

ANALYSIS OF SIMPLE ORGANIC COMPOUNDS

EXPERIMENT	OBSERVATION	INFERENCE
I. Preliminary tests		
1. Colour and appearance	Dark coloured liquid	Presence of aromatic amine.
	Flesh coloured solid	Presence of dihydric phenol
	White solid	Presence of carbohydrate or acid or amide
	Colourless liquid	Presence of aldehyde or ester
2. Odour	Fruity smell	Presence of ester
	Smell of bitter almonds	Presence of aldehyde
	Fishy smell	Presence of aromatic amine.
	Odourless	Absence of ester, aldehyde and aromatic amine
3. Solubility in water To a little of the substance water is added and shaken well. The above solution is heated	Soluble in cold water	Presence of carbohydrates, diamide or dihydric phenol
	Soluble in hot water and crystallizes on cooling	Presence of aromatic acid
	Insoluble in water	Presence of aldehyde or ester or amine
4. Tests for Aromaticity ***a) Ignition Test :*** A little of the substance is ignited on a nickel spatula.	Burns with a smoky flame	Presence of aromatic compound
	Burns with a non-smoky flame	Presence of aliphatic compound

EXPERIMENT	OBSERVATION	INFERENCE
b. Nitration Test: A little of the substance is mixed with 3 drops of conc. HNO_3 and 3 drops of con. H_2SO_4 in a semi micro tube. It is heated in a boiling water bath and poured into water taken in another test tube.	Yellow solution	Presence of aromatic compound
	Colourless solution	Presence of aliphatic compound
5. Test for Saturation / unsaturation 1. A little of the substance is mixed with bromine water in a semi micro tube.	Decolourisation	Presence of unsaturated compound
	No decolourisation	Presence of saturated compound
2. A little of the substance in water is treated with a few drops of dilute $KMnO_4$ solution.	Decolourisation	Presence of unsaturated compound.
	No decolourisation	Presence of saturated compound.
DETECTION OF ELEMENTS **Lassaigne's Test (Sodium fusion Test)** A small piece of metallic sodium is taken in a semi micro test tube and melted. A small amount of the substance is then added and again heated strongly. Water is then added, heated to boiling and cooled. This is called as the sodium fusion extract which is used for the detection of elements.		
1. Test for Nitrogen About 3 drops of the extract is boiled with $FeSO_4$ crystals. To this one drop of NaOH and one drop of Con. HCl is added.	Green (or) blue colouration	Presence of nitrogen
	No Green (or) blue colouration	Absence of nitrogen
2. A drop of the extract is mixed with freshly prepared sodium nitroprusside solution	Violet colouration	Presence of sulphur
	No violet colouration	Absence of sulphur

EXPERIMENT	OBSERVATION	INFERENCE
II. Detection of Functional groups		
1. Action of $NaHCO_3$ A little of the substance is treated with a drop of saturated solution of $NaHCO_3$ on a porcelain tile.	Brisk effervescence	Presence of carboxylic acid
	No brisk effervescence	Absence of acid
2. Action of conc. H_2SO_4 A small amount of the substance is heated with 2 drops of conc. H_2SO_4.	Charring with effervescence	Presence of carbohydrate
	No charring with effervescence	Absence of carbohydrate
3. Schiff's reagent test About 3 drops of the substance is treated with 2 drops of Schiff's reagent and shaken well.	Pink colouration	Presence of aldehyde
	No pink colouration	Absence of aldehyde
4. Acid formation a) A small amount of the substance is added to a strong solution of NaOH and then heated to boiling.	Ammonia gas is evolved on continued boiling	Presence of amide
	Substance dissolved gradually on warming	Presence of ester
	No characteristic change	Absence of amide and ester
b) The above solution is acidified with conc. HCl.	White precipitate	Presence of monamide or ester
	No White precipitate	Absence of monamide and ester
5. Action of neutral ferric chloride To a little of the substance is added 2 drops of neutral Ferric chloride.	Violet colouration	Presence of phenol
	No violet colouration	Absence of phenol

EXPERIMENT	OBSERVATION	INFERENCE
6. Action of HCl A small amount of the substance is shaken with 1:1 HCl.	Substance readily dissolved and regenerated on adding NaOH solution	Presence of aromatic amine
	No characteristic change	Absence of aromatic amine
1) Test for Carboxylic acid ***Fluorescein test*** A little of the substance is heated with twice its weight of resorcinol and 2 – 3 drops of conc. H_2SO_4. This mixture is taken on the tip of a glass rod and slowly immersed into a semi micro test tube containing 3mL of water and 3 drops of 50% NaOH.	Greenish yellow fluorescence	Presence of dicarboxylic acid
	No greenish yellow fluorescence	Presence of monocarboxylic acid
2) Test for Ester ***Hydroxamic acid test:*** About 2 drops of the substance is heated with ethanolic solution of hydroxylamine hydrochloride and 10% sodium hydroxide solution and cooled, The solution is acidified with dilute hydrochloric acid and 2 drops of neutral ferric chloride is added.	Violet colour	Presence of ester
3) Test for Aldehyde ***i) Tollen's reagent test*** A small amount of the substance is added to about 2 drops of Tollen's reagent (A&B) and heated in a boiling water bath for 10 minutes.	Bright silver mirror or black precipitate	Presence of aldehyde

EXPERIMENT	OBSERVATION	INFERENCE
ii) Borsche's Reagent Test A drop of the alcoholic solution of the substance is mixed with Borsche's reagent followed by 2 drops of conc. HCl . The mixture is heated for 15minutes and then diluted.	Red orange precipitate	Presence of aldehyde
4) Test for Carbohydrate ***i) Fehling's solution test*** A little of the substance is heated with 2 drops of Fehling's solution A and 2 drops of Fehling's solution B in a boiling water bath.	Reddish brown precipitate	Presence of carbohydrate
ii) Molisch's test A little of the substance is dissolved in 2 mL of water, a 2 drops of an alcoholic solution of α- naphthol (1- naphthol) is added followed by 2 drops of con. sulphuric acid along the sides of the test tube.	Violet ring at the junction	Presence of carbohydrate
5) Test for Dihydric phenol ***Phthalein fusion test*** A little of the substance is heated with an equal amount of phthalic anhydride and 2 drops of conc. H_2SO_4. This mixture is taken on the tip of a glass rod and slowly immersed into a semi micro test tube containing 3mL of water and 3 drops of 50% NaOH.	Greenish yellow fluorescence	Presence of dihydric phenol

EXPERIMENT	OBSERVATION	INFERENCE
Test for Compounds Containing Nitrogen		
1) Test for amide ***i) Acid formation*** a) About 1 g of the substance is boiled with 3 drops of a 10% solution of NaOH.	Evolution of ammonia gas	Presence of amide
b) The above solution is cooled and then acidified with 2 drops conc. HCl.	White precipitate	Presence of monoamide
ii) Biuret test A little of the substance is heated in a dry test-tube until it melts. The resulting white residue is cooled and dissolved in 2 mL of water. Then 2 drops of a very dilute $CuSO_4$ solution is added followed by NaOH solution in drops.	Violet colouration	Presence of diamide
	No violet colouration	Absence of diamide
iii) Action with conc. **HNO_3** A little of the substance is dissolved in water, and added con. HNO_3.	White crystalline precipitate	Presence of diamide
iv) Action of oxalic acid To a saturated solution of the substance saturated solution of oxalic acid is added.	White crystalline precipitate	Presence of diamide
2) Test for amine ***i) Action of HCl*** A small amount of the substance is shaken with 1:1 HCl.	Substance readily dissolved and regenerated on adding NaOH solution	Presence of aromatic amine
ii) A little of the substance is heated with acetic anhydride and the mixture is poured in to water.	White crystalline precipitate	Presence of aromatic primary amine

EXPERIMENT	OBSERVATION	INFERENCE
iii) Dye test A little of the substance is dissolved in 3 drops of dil. HCl and cooled in ice water. To this, 1 mL of a strong solution of $NaNO_2$ is added in drops with constant stirring. Then, 2 mL of sodium acetate solution is added and the mixture is poured into a solution of β-naphthol in NaOH solution.	Scarlet red dye	Presence of aromatic primary amine

Report

The given organic substance is = _____.

Organic Analysis

S.No	Date	Organic Substance	Sign	Marks

Preparation of Reagents

Organic Reagents

1. **Borsche's Reagent:** It is 1% methyl alcoholic solution of 2,4-dinitrophenylhydrazine. 1g of 2,4-dinitrophenylhydrazine is refluxed with 100mL of methyl alcohol till dissolved or alternatively 12 g of 2,4-dinitrophenylhydrazine is dissolved in 60mL of conc.sulphuric acid. This solution is added with stirring to 80mL of waterand 280mL of 95% ethyl alcohol. The solution is mixed thoroughly and filtered.

2. **Bromine water:** 1mL of liquid bromine per litre of the solution.It should be kept in coloured bottles.

3. **Fehling's A:** 6.9g of copper sulphate crystals are dissolved in 40mL of water and diluted to 100mL.

4. **Fehling's B:** 15g of sodium hydroxide and 36g of sodium potassium tartrate (Rochellesalt) are dissolved separately in 30mL of water each, mixed and diluted to 100mL after cooling. Equal volumes of A and B are mixed before use.

5. **Neutral Ferric Chloride:** About 1g of ferric chloride is dissolved in 100mL water. Sodium carbonate solution is added little by little to the above solution till the slight turbidity persists even after shaking. The precipitate is filtered off and the filtrate is used as neutral ferric chloride solution.

6. **Schiff's Reagent:** 0.2 g of para-rosaniline (fuchsine) as hydrochloride or acetate is dissolved in 20mL of water and saturated with sulphurdioxide.After the solution has become colourless , it is filtered, diluted to 200mL with water and kept in dark bottles.

7. **Tollen's reagent:** Tollen' s A reagent: 20g of AgNO3 is dissolved in 50ml water and 50ml of ammonia. Tollen's B reagent: 20g of NaOH is dissolved in 200ml water.

Titration I

Standardisation of NaOH **Std. $H_2C_2O_4$ Vs. NaOH**

S.No.	Volume of NaOH (mL)	Burette Reading (mL)		Volume of Oxalic acid (mL)	Indicator
		Initial	Final		
1.	20.0	0			Phenolphthalein
2.	20.0	0			

Concordant Value = _____

Strength of Oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}} = \frac{6.3}{63} = 0.1$ N

Volume of Oxalic acid (V_1) =

Strength of Oxalic acid (N_1) = 0.1N

Volume of Sodium hydroxide (V_2) = 20 mL

Strength of Sodium hydroxide (N_2) $= \frac{V_1 N_1}{V_2} =$ _____ N

Ex.No: 1

Date:

ESTIMATION OF HYDROCHLORIC ACID/SULPHURIC ACID USING STANDARD OXALIC ACID

Aim

To estimate the amount of hydrochloric acid / sulphuric acid present in the whole of the given solution, a standard solution of oxalic acid containing 6.3 g per litre is supplied.

Principle

Both the solutions supplied are acids. Therefore a solution of a base i.e. sodium hydroxide is used as the link solution.

$$2\,NaOH + H_2C_2O_4 \longrightarrow Na_2\,C_2O_4 + 2H_2O$$

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

$$2NaOH + H_2SO_4 \longrightarrow Na_2SO_4 + 2H_2O$$

$$\text{Oxalic acid (Std)} \xleftrightarrow{\text{Titration I}} \text{NaOH (Link)} \xleftrightarrow{\text{Titration II}} \text{HCl / } H_2SO_4 \text{ (Estimating Solution)}$$

Since both the acid and the base are strong, either methyl orange or phenolphthalein may be used as indicator.

Procedure

(i) Standardisation of sodium hydroxide (link) solution

A clean burette is rinsed and filled with standard oxalic acid. 20 mL of sodium hydroxide solution is pipetted out into a clean conical flask, a drop of phenolphthalein is added and titrated against the acid. The end point is the just disappearance of pink colour. The titration is repeated to get concordant value. From the titre value the strength of sodium hydroxide is calculated.

Titration II

Estimation of HCl/ H_2SO_4 **NaOH Vs. HCl / H_2SO_4**

S.No.	Volume of NaOH (mL)	Burette Reading (mL)		Volume of HCl / H_2SO_4 (mL)	Indicator
		Initial	Final		
1.	20.0				Phenolphthalein
2.	20.0				

Concordant Value = _____

Volume of sodium hydroxide (V_1) = 20 mL

Strength of sodium hydroxide (N_1) = N

Volume of hydrochloric acid (V_2) = mL

Strength of hydrochloric acid (N_2) = $\frac{V_1 N_1}{V_2}$ =

Amount of hydrochloric acid / sulphuric acid present in the whole of the given solution = $\frac{\text{Strength} \times \text{Eq. Wt.}}{10}$

(ii) Estimation of hydrochloric acid / sulphuric acid

The given solution of hydrochloric acid / sulphuric acid is made upto 100mL in a standard flask and taken in the clean burette after rinsing. 20 mL of sodium hydroxide solution is pipetted out into a conical flask, a drop of phenolphthalein is added and titrated against the acid as before. The titration is repeated to get concordant value. From the titre value, the strength and hence the amount of hydrochloric acid/sulphuric acid is the whole of the given solution is calculated.

Result

Amount of hydrochloric acid / sulphuric acid
present in thewhole of the given solution = ———————— g

Titration I

Standardisation of HCl **NaOH Vs. HCl**

S.No.	Volume of NaOH (mL)	Burette Reading (mL)		Volume of Oxalic acid (mL)	Indicator
		Initial	Final		
1.					Phenolphthalein
2.					

Concordant Value = _____

Strength of NaOH $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}} = 0.1$ N

Volume of NaOH (V_1) =

Strength of NaOH (N_1) = 0.1N

Volume of HCl (V_2) = 20 mL

$\therefore$ Strength of HCl $(N_2) = \frac{V_1 N_1}{V_2} =$ _____ N

Ex.No: 2

Date:

ESTIMATION OF SODIUM HYDROXIDE USING STANDARD SODIUM HYDROXIDE

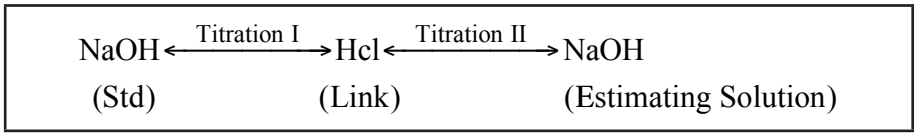
Aim

To estimate the amount of sodium hydroxide present in the whole of the given solution. A standard solution of sodium hydroxide containing 4 g/lit is supplied.

Principle

Both the solutions supplied are bases. Therefore a solution of an acid ie. hydrochloric acid is used as the link solution.

$$NaOH + HCl \rightarrow NaCl + H_2O$$



Procedure

(i) Standardisation of hydrochloric acid (link) solution

A clean burette is rinsed and filled with the HCI. 20mL of sodium hydroxide solution is pipetted out into a clean conical flask, a drop of phenolphthalein is added and titrated against the acid. The end point is the disappearance of pink colour. The titration is repeated to get concordant value. From the titre value, the strength of HCl is calculated.

(ii) Estimation of sodium hydroxide

The given solution of NaOH is made upto _____ mL, in a standard measuring flask and standardised HCl is taken in the clean burette after rinsing with the same. 20mL of sodium hydroxide solution is pipetted out into a conical flask, a drop of phenolphthalein is added and titrated against the acid as before. The end point is the disappearance of pink

Titration II

Estimation of NaOH **HCl Vs. NaOH**

S.No.	Volume of NaOH (mL)	Burette Reading (mL)		Volume of HCl (mL)	Indicator
		Initial	Final		
1.	20.0	0			Phenolphthalein
2.	20.0	0			

Concordant Value = _____

Volume of HCl (V_1) =

Strength of HCl (N_1) =

Volume of NaOH (V_2) =

Strength of NaOH (N_2) = $\frac{V_1\ N_1}{V_2}$ =

Amount of sodium hydroxide present in the whole of the given solution = $\frac{\text{Strength} \times \text{Eq. Wt.}}{10}$

colour. The titration is repeated to get concordant value. From the titre value, the strength and hence the amount of NaOH in the whole of the given solution is calculated.

Result

Amount of sodium hydroxide present
in the whole of the given solution = _____ g

Titration I

Standardisation of HCl **Std. Na_2CO_3 Vs. HCl**

S.No.	Volume of Na_2CO_3 (mL)	Burette Reading (mL)		Volume of HCl (mL)	Indicator
		Initial	Final		
1.					Methyl orange
2.					

Concordant Value = _____

Strength of Sodium Carbonate $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}} = \frac{5.2}{53} = 0.0981$ N

Volume of sodium carbonate (V_1) =

Strength of sodium carbonate (N_1) =

Volume of hydrochloric acid (V_2) =

$\therefore$ Strength of hydrochloric acid $(N_2) = \frac{V_1 N_1}{V_2} =$

Ex.No: 3

Date:

ESTIMATION OF SODIUM CARBONATE USING STANDARD SODIUM CARBONATE

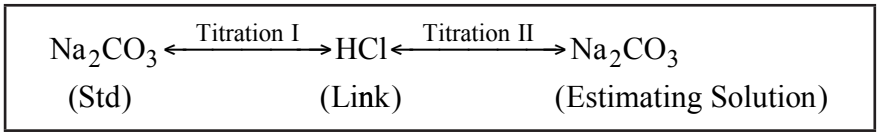
Aim

To estimate the amount of sodium carbonate present in the whole of the given solution. A standard solution of sodium carbonate containing 5.2 g/lit is supplied.

Principle

Both the solutions supplied are bases. Therefore a solution of an acid ie. hydrochloric acid is used as the link solution.

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$$



Procedure

(i) Standardisation of hydrochloric acid (link) solution

A clean burette is rinsed and filled with the HCl. 20mL of standard sodium carbonate solution is pipetted out into a clean conical flask, a drop of methyl orange is added and titrated against the acid. The end point is change of colour from golden yellow to pale pink. The titration is repeated to get concordant value. From the titre value, the strength of HCl is calculated.

(ii) Estimation of sodium carbonate

The given solution of sodium carbonate is made upto 100mL, in a standard measuring flask and standardised HCl is taken in the clean burette after rinsing with the same.

Titration II

Estimation of Na_2CO_3 **HCl Vs. Na_2CO_3**

S.No.	Volume of Na_2CO_3 (mL)	Burette Reading (mL)		Volume of HCl (mL)	Indicator
		Initial	Final		
1.	20.0	0			Methyl orange
2.	20.0	0			

Concordant Value = _____

Volume of hydrochloric acid (V_1) =

Strength of hydrochloric acid (N_1) =

Volume of sodium carbonate (V_2) =

Strength of sodium carbonate (N_2) = $\frac{V_1 N_1}{V_2}$ =

Amount of sodium carbonate present in the whole of the given solution = $\frac{\text{Strength} \times \text{Eq. Wt.}}{10}$

20mL of sodium carbonate solution is pipetted out into a conical flask, a drop of methyl orange is added and titrated against the acid as before. The end point is change of colour from golden yellow to pale pink. The titration is repeated to get concordant value. From the titre value, the strength and hence the amount of sodium carbonate in the whole of the given solution is calculated.

Result

Amount of sodium carbonate present in the whole of the given solution = _____ g

Titration I

Standardisation of NaOH **Oxalic acid Vs. NaOH**

S.No.	Volume of Sodium Hydroxide (mL)	Burette Reading		Volume of Oxalic acid (mL)	Indicator
		Initial	Final		
					Phenolphthalein

Concordant Value = _____

Strength of oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}} = \frac{6.1}{63} = 0.0968$ N

Volume of oxalic acid (V_1) =

Strength of oxalic acid (N_1) =

Volume of NaOH (V_2) =

$\therefore$ Strength of NaOH $(N_2) = \frac{V_1 N_1}{V_2} =$

Ex.No: 4

Date:

ESTIMATION OF OXALIC ACID USING STANDARD OXALIC ACID

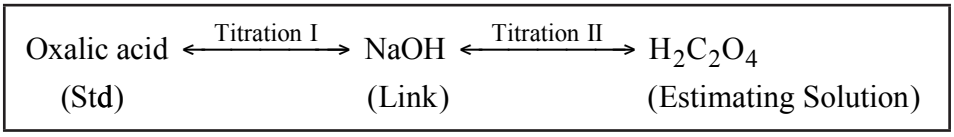
Aim

To estimate the amount of oxalic acid being provided with approximately decinormal solution of NaOH and solution of oxalic acid containing 6.1 g/lit .

Principle

Both the solutions supplied are acids. Therefore a solution of a base i.e. sodium hydroxide is used as the link solution.

$$2\ NaOH + H_2C_2O_4 \longrightarrow Na_2\ C_2O_4 + 2H_2O$$



Since both the acid and the base are strong, either methyl orange or phenolphthalein may be used as indicator.

Procedure

(i) Standardisation of sodium hydroxide (link) solution

A clean burette is rinsed and filled with standard oxalic acid. 20 mL of sodium hydroxide solution is pipetted out into a clean conical flask, a drop of phenolphthalein is added and titrated against the acid. The end point is the just disappearance of pink colour. The titration is repeated to get concordant value. From the titre value the strength of sodium hydroxide is calculated.

Titration II

Estimation of oxalic acid **NaOH Vs. Oxalic acid**

S.No.	Volume of Sodium Hydroxide (mL)	Burette Reading		Volume of oxalic acid (mL)	Indicator
		Initial	Final		
					Phenolphthalein

Concordant Value = _____

Volume of NaOH (V_1) =

Strength of NaOH (N_1) =

Volume of oxalic acid (V_2) =

Strength of oxalic acid (N_2) = $\frac{V_1 N_1}{V_2}$ =

Amount of oxalic acid present in the whole of the given solution = $\frac{\text{Strength} \times \text{Eq. Wt.}}{10}$

(ii) Estimation of Oxalic acid

The given solution of oxalic acid is made upto 100 mL in a standard flask and taken in the clean burette after rinsing. 20 mL of sodium hydroxide solution is pipetted out into a conical flask, a drop of phenolphthalein is added and titrated against the acid as before. The titration is repeated to get concordant value. From the titre value, the strength and hence the amount of oxalic acid in the whole of the given solution is calculated.

Result

Weight of oxalic acid present in the whole of the given solution = _____ g.

Titration I

Standardisation of $KMnO_4$ **Std FAS Vs. $KMnO_4$**

S.No.	Volume of FAS (mL)	Burette Reading (mL)		Volume of $KMnO_4$ (mL)	Indicator
		Initial	Final		
1.					Self
2.					

Concordant value = _____

Strength of Ferrous Ammonium Sulphate $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}} =$ =

Volume of FAS (V_1) =

Strength of FAS (N_1) =

Volume of $KMnO_4$ (V_2) =

Strength of $KMnO_4$ (N_2) $= \frac{V_1 N_1}{V_2} =$

Ex.No: 5

Date:

ESTIMATION OF FERROUS IRON USING STANDARD FERROUS AMMONIUM SULPHATE

Aim

To estimate the amount of Ferrous iron in the whole of the given solution. A standard solution of ferrous ammonium sulphate containing 40 g/lit is supplied.

Principle

Since both ferrous iron and ferrous ammonium sulphate are reducing agents , an oxidizing agent namely permanganate is used as the link solution.

The titrations are carried out in the presence of dil. sulphuric acid. In permanganometry, no indicator is necessary.

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$2FeSO_4 + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3 + H_2O$$

FAS	$\xleftrightarrow{\text{Titration I}}$	$KMnO_4$	$\xleftrightarrow{\text{Titration II}}$	$FeSO_4$
(Std)		(Link)		(Estimating Solution)

Procedure

(i) Standardisation of potassium permanganate solution

The permanganate solution is taken in the burette. 20mL of the standard ferrous ammonium sulphate solution is pipetted out into a conical flask, an equal volume of dil. sulphuric acid is added and the solution is titrated against the permanganate. The end point is the appearance of a pale permanent pink colour. The titration is repeated to get concordant value. From the titre value, the strength of potassium permanganate solution is calculated.

Titration II

Estimation of Fe^{2+} **$KMnO_4$ Vs. Fe^{2+}**

S.No.	Volume of Fe^{2+} (mL)	Burette Reading		Volume of $KMnO_4$ (mL)	Indicator
		Initial	Final		
1.					Self
2.					

Concordant Value = _____

Volume of $KMnO_4$ (V_1) =

Strength of $KMnO_4$ (N_1) =

Volume of Fe^{2+} (V_2) =

Strength of Fe^{2+} (N_2) = $\frac{V_1 N_1}{V_2}$ =

Amount of Fe^{2+} present in the whole of the given solution = $\frac{\text{Strength} \times \text{At. Wt. of iron}}{10}$

(ii) Estimation of Ferrous iron

The given ferrous iron is made upto 100mL, in a standard measuring flask. 20 mL of this solution is taken in a conical flask, equal volume of dil. sulphuric acid is added and the solution is titrated against the same permanganate as before. The titration is repeated to get concordant value. From the titre value, the strength and hence the amount of ferrous sulphate in the whole of the given solution is calculated.

Result

Amount of ferrous iron present in the whole of the given solution = _____ g.

Titration I

Standardisation of $KMnO_4$ **Oxalic acid Vs. $KMnO_4$**

S.No.	Volume of Oxalic acid (mL)	Burette Reading (mL)		Volume of $KMnO_4$ (mL)	Indicator
		Initial	Final		
					Self

Concordant value = _____

Strength of oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}} =$

Volume of oxalic acid $(V_1) =$

Strength of oxalic acid $(N_1) =$

Volume of $KMnO_4$ $(V_2) =$

Strength of $KMnO_4$ $(N_2) = \frac{V_1 N_1}{V_2} =$

Ex.No: 6

Date:

ESTIMATION OF SODIUM OXALATE / OXALIC ACID USING STANDARD OXALIC ACID

Aim

To estimate the amount of sodium oxalate / oxalic acid being provided with approximately decinormal solution of $KMnO_4$ and solution of oxalic acid containing 6.1 g/lit.

Principle

Potassium permanganate in acid medium is an oxidizing agent, two molecules of the substance gives five atoms of oxygen for oxidation. This oxygen oxidises reducing agents such as oxalic acid. The reaction between oxalic acid and potassium permanganate may be represented by the following equation.

$$Na_2C_2O_4 + H_2SO_4 \longrightarrow H_2C_2O_4 + Na_2SO_4$$

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$H_2C_2O_4 + [O] \longrightarrow H_2O + 2CO_2$$

Procedure

(i) Standardisation of $KMnO_4$

A clean burette is rinsed with $KMnO_4$ solution and filled with the same. 20mL of the standard oxalic acid solution is pipetted out into a clean conical flask and acidified with about 20mL of dilute sulphuric acid. The mixture is heated to 60°C and it is titrated against the potassium permanganate solution which is taken in the burette. The end point is the appearance of permanent pale pink colour. The titration is repeated to get the concordant value.

Titration II

Estimation of Sodium oxalate / Oxalic acid **$KMnO_4$ Vs. $Na_2C_2O_4$ / $H_2C_2O_4$**

S.No.	Volume of Sodium Oxalate / Oxalic acid (mL)	Burette Reading		Volume of $KMnO_4$ (mL)	Indicator
		Initial	Final		
					Self

Concordant Value = _____

Volume of $KMnO_4$ (V_1) =

Strength of $KMnO_4$ (N_1) =

Volume of Sodium Oxalate / Oxalic acid (V_2) =

Strength of Sodium Oxalate / Oxalic acid (N_2) = $\frac{V_1 N_1}{V_2}$ =

Amount of Sodium Oxalate / Oxalic acid present in the whole of the given solution = $\frac{\text{Strength} \times \text{Eq. Wt.}}{10}$

(ii) Estimation of Sodium oxalate/oxalic acid

The given Sodium oxalate / oxalic acid solution is made up to 100 mL in the standard measuring flask. 20mL of this solution is pipetted out into a clean conical flask and acidified with about 20mL of dil. H_2SO_4. The mixture is heated to 60°C and it is titrated against the permanganate solution until it turns pale permanent pink. The titration is repeated for concordant value.

Result

Weight of sodium oxalate/oxalic acid present in the

whole of the given solution = _____ g

Titration I

Standardisation of EDTA **Std. $ZnSO_4$ Vs. EDTA**

S.No.	Volume of $ZnSO_4$ (mL)	Burette Reading (mL)		Volume of EDTA (mL)	Indicator
		Initial	Final		
1.	20	0			Eriochrome black-T
2.	20	0			

Concordant value = _____

Volume of $ZnSO_4$ Solution (V_1) = mL

Strength of $ZnSO_4$ Solution (M_1) = M

Volume of EDTA (V_2) = mL

Strength of EDTA $(M_2) = \frac{V_1 M_1}{V_2}$

Ex.No: 7

Date:

ESTIMATION OF ZINC ION USING STANDARD ZINC SULPHATE

Aim

To estimate the amount of Zinc ion present in the whole of the given $ZnSO_4$ solution being provided with approximately 0.01M solution of EDTA and solution of $ZnSO_4$ containing 2.88g / litre.

Principle

The Disodium salt of EDTA is commonly represented as Na_2H_2Y and gives complex of ion H_2Y^{2-} in aqueous solution. The estimation is based on the reaction.

$$Zn^{2+} + H_2Y^{2-} \rightarrow ZnY^{2-} + 2H^+$$

$$ZnSO_4 \xleftrightarrow{\text{Titration I}} \underset{\text{(Link)}}{EDTA} \xleftrightarrow{\text{Titration II}} ZnSO_4$$

(Std) (Estimating Solution)

Procedure

(i) Standardisation of the EDTA Solution using $ZnSO_4$ solution

The EDTA solution is taken in the burette. 20mL of $ZnSO_4$ solution is pipetted out into a clean conical flask. 2 mL (pH ~ 10), of a buffer solution is added, a pinch of solid Eriochrome black–T indicator are added and titrated against the EDTA solution. The end point is the colour change from wine red to clear blue. Titrations are repeated to get concordant value. From the titre value, the molarity of EDTA is calculated.

(ii) Estimation of Zinc ion

The given $ZnSO_4$ solution is made upto 100 mL in the standard measuring flask. 20 mL of the solution is pipetted out into a clean conical flask. To this 2 mL of the buffer

Titration II

Estimation of $ZnSO_4$ **EDTA Vs. $ZnSO_4$**

S.No.	Volume of $ZnSO_4$ (mL)	Burette Reading (mL)		Volume of EDTA (mL)	Indicator
		Initial	Final		
1.	20	0			Eriochrome black-T
2.	20	0			

Concordant value = _____

Volume of EDTA (V_1) = mL

Strength of EDTA (M_1) = M

Volume of $ZnSO_4$ Solution (V_2) = mL

Strength of $ZnSO_4$ Solution (M_2) = $\frac{V_1 M_1}{V_2}$

Amount of Zinc ion present in the whole of the given solution = $\frac{\text{Molarity} \times \text{Atomic weight}}{10}$

solution (pH ~ 10) and a pinch of solid Eriochrome black-T indicator are added. Then the solution is titrated against EDTA solution in the burette. The end point is the change of colour from wine red to clear blue. The titrations are repeated for concordant value. From the titre value, the molarity of $ZnSO_4$ solution and hence the amount of Zinc ion in the whole of the given solution is calculated.

Result

Amount of Zinc ion present in the whole of the given solution = _____ g.

SHORT PROCEDURE

1. Estimation of Hydrochloric Acid /Sulphuric acid

Link solution: NaOH

S. No.	Titration I: Std. $H_2C_2O_4$ Vs. NaOH	
1.	Burette Solution	Oxalic acid
2.	Pipette Solution	20 mL of std. NaOH
3.	Medium	—
4.	Temperature	Room temperature
5.	Indicator	Phenolphthalein
6.	End point	Disappearance of pink colour

From the titre value the strength of the link solution (NaOH) is calculated.

S. No.	Titration I: Std. $H_2C_2O_4$ Vs. NaOH	
1.	Burette Solution	HCl / H_2SO_4
2.	Pipette Solution	20 mL of NaOH
3.	Medium	—
4.	Temperature	Room temperature
5.	Indicator	Phenolphthalein
6.	End point	Disappearance of pink colour

From the titre value the strength and hence the amount of the given Hydrochloric acid/Sulphuric acid is calculated.

Substance	Oxalic acid (std)	HCl /H_2SO_4 (to be estimated)
Equivalent mass	63	36.5/49

St. Mary's College founded in the year 1948, with just 21 students has grown multifold nurturing around 3000 students at present. The institution is committed to the noble cause of liberating young women, especially the ones belonging to coastal areas and preparing them to assume leading roles in making societal changes. Most of the students who have passed out from the institution and scaled greater heights are first generation learners. St. Mary's owns the proud privilege of educating such young women.

St. Mary's College (Autonomous) sent the proposal for Star College Scheme on 10th May 2018. The funds under Star College scheme was sanctioned on 16th March 2019. Four departments of the college namely Physics, Chemistry, Botany and Zoology have been recommended by the Department of Biotechnology (DBT), Ministry of Science & Technology, Government of India, New Delhi, to receive financial assistance under the Star College Scheme. For implementation of the Star College Scheme, the DBT, New Delhi has sanctioned 82 Lakhs.

The objective of the Star College Scheme is to strengthen Life Science and Biotechnology education and training at the undergraduate level to encourage and attract students to pursue a career in Life Sciences.

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