V.V.VANNIAPERUMAL COLLEGE FOR WOMEN

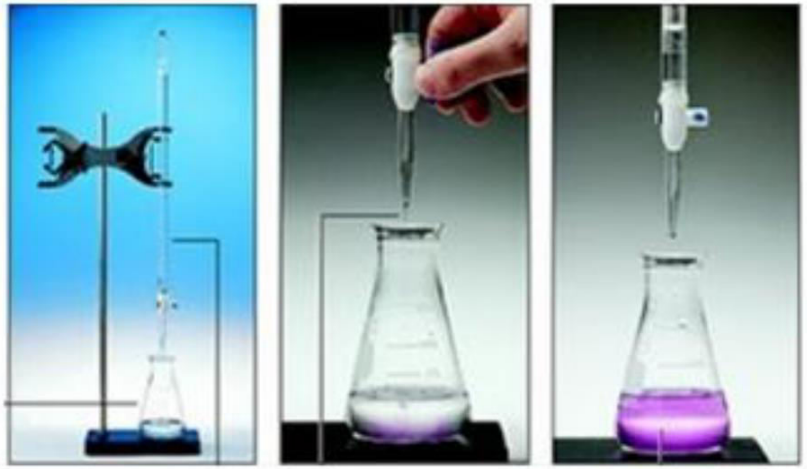
VIRUDHUNAGAR

LAB MANUAL

VOLUMETRIC ANALYSIS

(UNDER DBT STAR COLLEGE SCHEME)

Department of Biotechnology, Ministry of Science and Technology, MHRD, New Delhi



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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

V.V.VANNIAPERUMAL COLLEGE FOR WOMEN

(Belonging to Virudhunagar Hindu Nadars)
An Autonomous Institution Affiliated to Madurai Kamaraj University
Re-accredited with 'A' Grade (3rd cycle) by NAAC
VIRUDHUNAGAR – 626 001 (TAMIL NADU)

DBT STAR COLLEGE SCHEME

Department of Biotechnology, Ministry of Science and Technology
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FOREWORD

The Lab Manual for **"VOLUMETRIC ANALYSIS"** is designed to acquaint the student with essential skills and techniques in accordancewith the updated syllabus under DBT Star College Scheme sponsored by the Department of Biotechnology, Ministry of Science and Technology, MHRD, New Delhi. The skill of analysis and performing experiments will reinforce the theoretical knowledge of learnt concepts.

We thank the **Department of Biotechnology, The Ministry of Science and Technology, MHRD, New Delhi** for providing a good opportunity under Star College Scheme (No HRD11011/163/2020-HRD-DBT Dt. 24.08.2020). Under this scheme, we have purchased Flame Photometer, Chemicals and Glassware. This provision enables the students for better understanding of basic concepts in Chemistry and to develop curiosity for further progress.

We hope this manual surely fulfil the student's need to enhance their attitude towards research and empower them as a better chemist.

MEMBER SECRETARY/COORDINATOR **CHAIRMAN/PRINCIPAL**

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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

LIST OF PRACTICALS

S.No.	TITLE	Page No.
	Acidimetry and Alkalimetry	
1.	**Estimation of sodium carbonate**	**1**
2.	**Estimation of sodium hydroxide**	**5**
3.	**Estimation of oxalic acid**	**9**
	Permanganometry	
4.	**Estimation of oxalic acid using $KMnO_4$**	**13**
5.	**Estimation of oxalic acid using ferrous ion**	**17**
6.	**Estimation of ferrous ions**	**21**
	Dichrometry	
7.	**Estimation of $K_2Cr_2O_7$**	**25**
	Iodometry and Iodimetry	
8.	**Estimation of potassium dichromate**	**31**
9.	**Estimation of copper**	**37**
10.	**Estimation of iodine**	**41**
11.	**Estimation of arsenious oxide**	**45**
	Argentometry	
12.	**Estimation of KCl**	**49**
13.	**Analysis of Sodium Level in Various Junk Foods by Flame Photometer**	**53**

VOLUMETRIC ANALYSIS

Volumetric analysis is a branch of quantitative estimation. This estimation is done by measuring accurately the volumes of the reacting liquids accurately.

I) TERMS INVOLVED IN VOLUMETRIC ANALYSIS:

TIRATION:

The process of finding out the exact volumes of the reacting liquids is referred as **titration**.

END POINT:

The end point of a reaction is the stage at which the reaction just completes. The end point is usually determined by an indicator which indicates the completion of the reaction by a marked colour change.

STANDARD SOLUTION:

A standard solution is a solution of known strength or concentration. The standard solution of primary standard substances can be directly prepared by dissolving an accurately weighed amount of the substances. The primary standard substances are substances which are

a) Available in a high degree of purity
b) Stable and unaffected by the atmosphere
c) Readily soluble and stable in water

Examples of primary standard substances are anhydrous sodium carbonate, potassium carbonate, Mohr's Salt, crystals of oxalic acid, potassium dichromate and copper sulphate.

Standard solutions for other substances can be obtained by standardizing their solutions using the standard solutions of primary standard substances.

NORMALITY:

One way of expressing the concentration of a solution is normality. It is mere number. The number gives the number of gram equivalents of the substances in one litre of the solution. It can be given by the following equation;

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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

$$N = \frac{\text{Weight in one litre of the solution}}{\text{Gram equivalent weight of the substance}}$$

EQUIVALENT WEIGHT:

Equivalent weight of a substance is the parts by weight of the substance which combine with or displace 1.008 parts by weight of hydrogen or equivalent weight of any other substance. If the equivalent weight is expressed in grams it is known as gram equivalent weight or gram equivalent.

II) PRINCIPLES OF VOLUMETRIC ANALYSIS:

When two solutions react with each other, the product of volume and normality of one solution will be equal to the product of volume and normality of the other solution.

$$V_1N_1 = V_2N_2$$

V_1 = Volume of the Solution 1
N_1 = Normality of the Solution 1
V_2 = Volume of the Solution 2
N_2 = Normality of the Solution 2

V_1 and V_2 are obtained from the titration. Knowing the normality of one solution, normality of the other solution can be calculated using the above equation. Knowing the normality, the weight of the substance in one litre of the solution can be calculated using the following expression:

Weight of the substance in 1 litre of the solution = Normality X Gram equivalent weight.

ACIDIMETRY AND ALKALIMETRY – INTRODUCTION

Acidimetry is the estimation of alkali solution using standard acid solution.

Alkalimetry is the estimation of acid solution using standard alkali solution.

EQUIVALENT WEIGHT:

Equivalent weight of an acid may be defined as the number of parts by weight of an acid which contains 1.008 parts by weight of replaceable hydrogen.

BASICITY:

Basicity of an acid is the number of replaceable hydrogen atom present in one molecule of the acid.

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

Equivalent weight of the base may be defined as the number of parts by weight of the base which combines with one equivalent of the acid,

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

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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

EQUIVALENT WEIGHT OF ACIDS AND BASES:

Name of acid	Mol.wt.	Basicity	Equivalent wt.= $\frac{\text{Mol. wt.}}{\text{Basicity}}$
Hydrochloric acid (HCl)	36.5	1	36.5
Nitric acid (HNO_3)	63	1	63
Sulphuric acid (H_2SO_4)	98	2	49
Oxalic acid ($C_2H_2O_4$)	126	2	63

Name of acid	Mol.wt.	Acidity	Equivalent wt.= $\frac{\text{Mol. wt.}}{\text{Acidity}}$
Sodium hydroxide (NaOH)	40	1	40
Potassium hydroxide (KOH)	56	1	56
Anhy. Sodium carbonate (Na_2CO_3)	106	2	53
Anhy. Potassium carbonate (K_2CO_3)	138.2	2	69.1

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

One molecule of Na_2CO_3 combines with two molecules or two equivalents of HCl.

Acidity of Na_2CO_3 = 2

$$\text{Equivalent wt. of } Na_2CO_3 = \frac{\text{Mol. Wt of } Na_2CO_3}{\text{Acidity}}$$

`

$$= \frac{106}{2}$$

$$= 53$$

CHOICE OF INDICATORS:

For acidimetry – alkalimetry titrations phenolphthalein and methyl orange are commonly used as indicatorsrespectively. They indicate the end point by distinct colour changes as shown in the table.

Indicator	Colour in acid medium	Colour in alkali medium	Titrants
Phenolphthalein	Colourless	Pink	Strong acid x Strong base Weak acid x Strong base
Methyl Orange	Pink	Yellow	Strong acid x Strong base Strong acid x Weak base

For titration involving strong acid and strong base any one of the above two indicators may be used. For titration of strong acid against weak base, only methyl orange can be used. For titration of weak acid against strong base, only phenolphthalein can be used. Weak acid and weak base are not titrated. For an experiment involving double titration same indicator is to be used for both titrations.

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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

1. ESTIMATION OF SODIUM CARBONATE

CALCULATION

$$\text{Normality of standard } Na_2CO_3 = \frac{\text{Weight/ Litre}}{\text{Equivalent weight}}$$

TITRATION - I

STANDARDISATION OF HCl SOLUTION USING STANDARD Na2Co3 SOLUTION

S.NO	VOLUME OF Na_2CO_3 (ml)	BURETTE READING (ml) INITIAL	BURETTE READING (ml) FINAL	VOLUME OF HCl (ml)	CONCORDANT VALUE (ml)	INDICATOR
1	20					Methyl orange
2	20					

Volume of std. Na_2CO_3 (V_1) =

Normality of std. Na_2CO_3 (N_1) =

Volume of HCl(V_2) =

Normality of HCl N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

1. ESTIMATION OF SODIUM CARBONATE

Estimate the amount of sodium carbonate (Na_2CO_3)present in the whole of the given solution. You are provided with pure anhydrous crystals of Na_2CO_3 and an approximately decinormal solution of hydrochloric acid (HCl).

AIM:

To estimate the amount of Na_2CO_3 in the whole of the given solution. Pure crystals of analar Na_2CO_3 and an approximately decinormalHCl solution are given.

REQUIREMENTS:

EQUIPMENT	: Digital balance
CHEMICALS:	: Na_2CO_3, HCl, methyl orange indicator.
APPARATUS:	:Burette, pipette, conical flask, standard measuring flask, funnel

PRINCIPLE:

This is an acid-base titration. Neutralisation takes place according to the equation

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

PROCEDURE:

WEIGHT OF Na_2CO_3FOR PREPARING 0.1N SOLUTION:

Weight of Na_2CO_3 in 1litre of the solution = Eq.wt. x 0.1N
= 53 x 0.1
= 5.3g

Weight of Na_2CO_3 in 200ml of the solution = $\frac{5.3 \times 200}{1000}$
= 1.06 g

PREPARATION OF STANDARD Na_2CO_3 SOLUTION:

About 1.06g of Na_2CO_3 is weighed accurately, transferred into a 200ml standard measuring flask, dissolved in distilled water and made upto the mark.

TITRATION – I

STANDARDISATION OF HCL SOLUTION USING STANDARD Na_2CO_3 SOLUTION:

The burette is filled with HCl. Pipetted out 20ml of standard Na_2CO_3 into a clean conical flask. 2 drops of methyl orange is added as an indicator into the conical flask. The solution turns yellow in colour. It is titrated against HCl solution in the burette. The end point is the change of colour from yellow to pale pink. The experiment is repeated for concordant value. The readings are noted in the tabular column.

Making up of the given solution:

The solution given in the small bottle is transferred into a 200ml standard measuring flask; diluted with distilled water and is made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN Na_2CO_3 USING STANDARDISED HCl:

S.NO	VOLUME OF Na_2CO_3 (ml)	BURETTE READING (ml)		VOLUME OF HCl (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
1	20					Methyl orange
2	20					

Volume of std. Na_2CO_3 (V1) =

Normality of std. Na_2CO_3(N1) =

Volume of HCl (V_1) =

Normality of HCl(N_2) = ?

$$V1N_1 = V_2N_2$$

$$N_2 = \frac{V_1N_1}{V_2}$$

Weight of Na_2CO_3 present in 1 litre = Normality X Equivalent weight

Weight of Na_2CO_3 present in 100 ml of the solution = $\frac{\text{Normality x 53x100}}{1000}$

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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

TITRATION – II

STANDARDISATION OF GIVEN Na_2CO_3USING STANDARDIZED HCL:

The burette is filled with HCl. Pipetted out 20ml of the given Na_2CO_3 into a clean conical flask. 2 drops of methyl orange is added as an indicator into the conical flask. The solution turns yellow in colour. It is titrated against HCl solution in the burette. The end point is the change of colour from yellow to pale pink. The experiment is repeated for concordant value.The readings are noted in the tabular column. Finally the calculation is done.

Equivalent weight of Na_2CO_3 = 53

RESULT:

Amount of Na_2CO_3 present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis , the students are able to

- take the accurate weight of the substances.
- handle the apparatus
- calculate the strength of the solution.
- calculate the amount of Na_2CO_3 present in the whole of the given solution.

2.ESTIMATION OF SODIUM HYDROXIDE

CALCULATION

TITRATION – I

STANDARDISATION OF HCl SOLUTION USING STANDARD Na_2CO_3 SOLUTION

S.NO	VOLUME OF Na_2CO_3 (ml)	BURETTE READING (ml) INITIAL	FINAL	VOLUME OF HCl (ml)	CONCORDANT VALUE (ml)	INDICATOR
						Methyl orange

Volume of std. Na_2CO_3 (V_1) =

Normality of std. Na_2CO_3 (N_1) =

Volume of HCl (V_2) =

Normality of HCl (N_2) = ?

$$V1N_1 = V_2N_2$$

$$N_2 = \frac{V_1N_1}{V_2}$$

2.ESTIMATION OF SODIUM HYDROXIDE

Estimate the amount of sodium hydroxide(NaOH) present in the whole of the given solution. You are provided with pure anhydrous crystals of sodium carbonate (Na_2CO_3) and an approximately decinormal solution of Hydrochloric acid (HCl).

AIM:

To estimate the amount of NaOH in the whole of the given solution. Pure crystals of analar Na_2CO_3 and an approximately decinormalHCl solution are given.

REQUIREMENTS:

EQUIPMENT :	Digital balance
CHEMICALS :	Na_2CO_3, HCl,methyl orange
GLASSWARES :	Burette , pipette, conical flask, standard measuring flask, funnel, .

PRINCIPLE:

This is an acid-base titration.

Neutralisation takes place according to the following equations

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

$NaOH + HCl \longrightarrow NaCl + H_2O$

PROCEDURE:

WEIGHT OF Na_2CO_3 FOR PREPARING 0.1N SOLUTION:

Weight of Na_2CO_3 in 1litre of the solution = Eq.wt. x 0.1N

= 53 x 0.1

= 5.3g

Weight of Na_2CO_3 in 200ml of the solution = $\frac{5.3 \times 200}{1000}$

= 1.06 g

PREPARATION OF STANDARD Na_2CO_3 SOLUTION:

About 1.06g of Na_2CO_3 is weighed accurately, transferred into a 200ml standard measuring flask, dissolved in distilled water and made upto the mark.

TITRATION – I

STANDARDISATION OF HCL SOLUTION USING STANDARD Na_2CO_3 SOLUTION:

The burette is filled with HCl. Pipetted out 20ml of standard Na_2CO_3 into a clean conical flask, 2 drops of methyl orange is added as an indicator into the conical flask. The solution turns yellow in colour. It is titrated against HCl solution in the burette. The end point is the change of colour from yellow to pale pink. The experiment is repeated for concordant value. The readings are noted in the tabular column.

MAKING UP OF THE GIVEN SOLUTION: The solution given in the small bottle is transferred into a 200ml standard measuring flask; diluted with distilled water and is made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN NaOH USING STANDARDISED HCl

S.NO	VOLUME OF NaOH (ml)	BURETTE READING (ml) INITIAL	FINAL	VOLUME OF HCl (ml)	CONCORDANT VALUE (ml)	INDICATOR
1	20					Methyl orange
2	20					

Volume of std. HCl (V_1) =

Normality of std. HCl (N_1) =

Volume of NaOH (V_2) =

Normality of NaOH (N_2) = ?

$V1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

Weight of NaOH present in 1 litre = Normality X Equivalent weight

Weight of NaOH present in 100 ml of the solution in $= \frac{\text{Normality x } 40\text{x}100}{1000}$

TITRATION – II

STANDARDISATION OF GIVEN NaOH USING STANDARDIZED HCl:

The burette is filled with HCl. Pipetted out 20ml of given NaOH into a clean conical flask. 2 drops of methyl orange is added as an indicator into the conical flask. The solution turns yellow in colour. It is titrated against HCl solution in the burette. The end point is the change of colour from yellow to pale pink. The experiment is repeated for getting concordant value. The readings are noted in the tabular column. Finally the calculation is done.

Equivalent weight of Na_2CO_3 = 53

Equivalent weight of NaOH = 40

RESULT:

Amount of NaOH present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis , the students are able to ;

- Take the accurate weight of the substances.
- Handle the apparatus
- Calculate the strength of the solution.
- Calculate the amount of NaOH present in the whole of the given solution.

3. ESTIMATION OF OXALIC ACID

CALCULATION

Normality of standard oxalic acid = $\frac{\text{weight /litre}}{\text{Equivalent weight}}$

TITRATION – I
STANDARDISATION OF NaOH SOLUTION USING STANDARD OXALIC ACID

S.NO	VOLUME OF NaOH SOLUTION (ml)	BURETTE READING (ml) INITIAL	BURETTE READING (ml) FINAL	VOLUME OF OXALIC ACID (ml)	CONCORDANT VALUE (ml)	INDICATOR
						PHENOLPHTHALEIN

Volume of standardOxalic acid (V_1) =

Normality of standard Oxalic acid (N_1) =

Volume of NaOH (V_2) =

Normality of NaOH (N_2) = ?

$$V1N_1 = V_2N_2$$

$$N_2 = \frac{V_1N_1}{V_2}$$

3.ESTIMATION OF OXALIC ACID

Estimate the amount of oxalic acid present in the whole of the given solution. You are provided with pure crystals of analar oxalic acid and an approximately decinormal solution of sodium hydroxide(NaOH).

AIM:

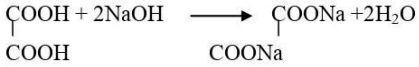
To estimate the amount of oxalic acid in the whole of the given solution. Pure crystals of analar oxalic acid and an approximately decinormalNaOH solution are given.

REQUIREMENTS:

EQUIPMENT	:	Digital balance
CHEMICALS	:	Oxalic acid,NaOH, phenolphthalein.
GLASSWARES	:	Burette, pipette,conicalflask,standard measuring flask, funnel,

PRINCIPLE:

This is an acid-base titration, Neutralization takes place according to the equation.



PROCEDURE:

WEIGHT OF OXALIC ACID FOR PREPARING 0.1N SOLUTION:

Weight of oxalic acid in 1litre of the solution = Eq.wt. x 0.1N
= 63 x 0.1
= 6.3g

Weight of oxalic acid in 200ml of the solution = $\frac{6.3 \times 200}{1000}$
= 1.26 g

PREPARATION OF STANDARD OXALIC ACID SOLUTION:

About 1.26g of oxalic acid is weighed accurately, dissolved in distilled water and made upto the mark in a 200ml standard measuring flask.

TITRATION – I

STANDARDISATION OF NaOH USING STANDARD OXALIC ACID:

The burette is filled with oxalic acid. 20ml of NaOH is pipetted out into a clean conical flask. A drop of phenolphthalein is added as an indicator into the conical flask. The solution turns pink in colour and is titrated against oxalic acid in the burette. The end point is the disappearance of pink color. The titration is repeated for getting concordant value. The readings are noted in the tabular column.

MAKING UP OF THE GIVEN SOLUTION:

The solution given in the small bottle is transferred into a 200ml standard measuring flask, diluted with distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN OXALIC ACID USING STANDARDISED NaOH

S.NO	VOLUME OF NaOH SOLUTION (ml)	BURETTE READING (ml)		VOLUME OF OXALIC ACID (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						PHENOLPHTHALEIN

Volume of std. NaOH (V_1) =

Normality of std. NaOH (N_1) =

Volume of given oxalic acid (V_2) =

Normality of given oxalic acid (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

Weight of oxalic acid present in 1 litre = Normality X Equivalent weight

$$\text{Weight of given oxalic acid present in 100 ml of the solution} = \frac{\text{Normality x 40x100}}{1000}$$

TITRATION – II

STANDARDISATION OF GIVEN OXALIC ACID USING STANDARDIZED NaOH:

The burette is filled with given oxalic acid. 20ml of NaOH is pipetted out into a clean conical flask. A drop of phenolphthalein is added as an indicator into the conical flask. The solution turns pink in colour and is titrated against oxalic acid in the burette. The end point is the disappearance of pink color. The titration is repeated for getting concordant value. The readings are noted in the tabular column. Finally the calculation is done.

Equivalent weight of oxalic acid = 63

RESULT:

Amount of oxalic acid present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES : 50

OUTCOME: After the completion of the volumetric analysis , the students are able to

- take the accurate weight of the substances.
- handle the apparatus
- calculate the strength of the solution.

calculate the amount of oxalic acid present in the whole of the given solution.

V.V.Vanniaperumal College for Women, Virudhunagar Tamilnadu.
No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

PERMANGANOMETRY –INTRODUCTION

Permanganometry involves the titrations between the oxidizing agent- postassium permanganate ($KMnO_4$)and a reducing agent.

EQUIVALENT WEIGHT:

Equivalent Weight of an oxidizing agent can be defined as the number of parts by weight of it which furnishes eight parts by weight of oxygen (one Equivalent Weight of oxygen) for oxidation.

$$\text{Equivalent Weight of Oxidizing agent} = \frac{\text{Molecular weight of oxidizing agent}}{\text{No. of equivalent weights of oxygen given}}$$

by one molecule of oxidizing agent.

$KMnO_4$ behaves as an oxidizing agent in the medium of dil. H_2SO_4 as per the following equation.

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

According to the equation,

$$\text{Equivalent Weight of } KMnO_4 = \frac{\text{Molecular weight of } KMnO_4}{5}$$

$$= 158/5$$

$$= 31.6$$

$KMnO_4$ oxidises the reducing agents -ferrous sulphate ($FeSO_4$), Ferrous ammonium Sulphate(FAS) oxalic acid etc.

Equivalent Weight of reducing agents is the parts by weight of the reducing agent which react with eight parts by weight of oxygen.

$$\text{Equivalent Weight of reducing agent} = \frac{\text{Molecular weight of reducing agent}}{\text{No. of equivalents of oxygen with which one molecule reacts.}}$$

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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

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

Equivalent Weight of $FeSO_4$ = Molecular weight of $FeSO_4$/1

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

Equivalent Weight of crystalline $FeSO_4$ ($FeSO_4.7\ H_2O$) = 278

Equivalent Weight of Mohr's salt (ie) Ferrous ammonium Sulphate = 392
($FeSO_4.(NH_4)_2SO_4.6\ H_2O$)

Equivalent Weight of oxalic acid = 63

ESTIMATION OF OXALIC ACID USING $KMnO_4$

CALCULATION

TITRATION – I

STANDARDISATION OF NaOH SOLUTION USING STANDARD OXALIC ACID

S.NO	VOLUME OF NaOH SOLUTION (ml)	BURETTE READING (ml)		VOLUME OF OXALIC ACID (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						PHENOLPHTHALEIN

Volume of standard. Oxalic acid (V_1) =

Normality of standard.oxalic acid (N_1) =

Volume of NaOH (V_2) =

Normality of NaOH (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

4. ESTIMATION OF OXALIC ACID USING $KMnO_4$

Estimate the amount of oxalic acid present in the whole of the given solution. You are provided with pure crystals of analar oxalic acid and an approximately decinormal solution of $KMnO_4$.

AIM:

To estimate the amount of oxalic acid present in the whole of the given solution. Pure crystals of analar oxalic acid and an approximately decinormal $KMnO_4$ solution are given.

REQUIREMENTS:

EQUIPMENT:	Digital balance
CHEMICALS:	Oxalic acid,NaOH, phenolphthalein.
GLASSWARES:	Burette, pipette,conicalflask,standard measuring flask, funnel,

PRINCIPLE:

This is a redox titration. In the presence of dil. H_2SO_4, $KMnO_4$ oxidizes oxalic acid to CO_2 and H_2O.

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

$COOH.2H_2O + [O] \longrightarrow 3H_2O + 2CO_2$

$COOH$

PROCEDURE:

WEIGHT OF OXALIC ACID FOR PREPARING 0.1N SOLUTION:

Weight of oxalic acid in 1litre of the solution =Eq.wt. x 0.1N

= 63 x 0.1

= 6.3g

Weight of oxalic acid in 200ml of the solution = $\frac{6.3 \times 200}{1000}$ = 1.26 g

PREPARATION OF STANDARD OXALIC ACID SOLUTION:

About 1.26g of oxalic acid is weighed accurately, dissolved in distilled water and made upto the mark in a 200ml standard measuring flask.

TITRATION – I

STANDARDISATION OF $KMnO_4$ USING STANDARD OXALIC ACID:

The burette is filled with $KMnO_4$. 20ml of Oxalic acid is pipetted out into a clean conical flask. Equal volume of 2N sulphuric acid is added in to the conical flask. The mixture in the conical flask is heated to 70^0C and is titrated against $KMnO_4$ into the burette. $KMnO_4$ itself acts as an indicator. The end point is the appearance of permanent pale pink color. The titration is repeated for concordant value. The readings are noted in the tabular column.

MAKING UP OF THE GIVEN SOLUTION:

The solution given in the small bottle is transferred to a 200ml standard measuring flask; diluted with distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN OXALIC ACID USING STANDARDISED $KMnO_4$

S.NO	VOLUME OF OXALIC ACID (ml)	BURETTE READING (ml)		VOLUME OF $KMnO_4$ (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						Self indicator $KMnO_4$

Volume of standard. KMnO4 (V_1) =

Normality of standard. KMnO4 (N_1) =

Volume of given oxalic acid (V_2) =

Normality of given oxalic acid (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

Weight of oxalic acid present in 1 litre = Normality X Equivalent weight

Weight of given oxalic acid present in 100 ml of the solution = $\frac{\text{Normality x } 40\text{x}100}{1000}$

TITRATION – II

STANDARDISATION OF GIVEN OXALIC ACID USING STANDARDIZED $KMnO_4$:

The burette is filled with $KMnO_4$. 20ml of Oxalic acid is pipetted out into a clean conical flask. Equal volume of 2Nsulphuric acid is added to the conical flask. The mixture in the conical flask is heated to 70^0C and is titrated against $KMnO_4$ in the burette. $KMnO_4$ itself acts as an indicator. The end point is the appearance of permanent pale pink color. The titration is repeated for concordant value. The readings are noted in the tabular column. Finally the calculation is done.

Equivalent weight of oxalic acid = 63

RESULT:

Amount of Oxalic acid present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis, the students are able to;

- take the accurate weight of the substances.
- handle the apparatus
- calculate the strength of the solution.
- calculate the amount of oxalic acid present in the whole of the given solution.

5. ESTIMATION OF OXALIC ACID USING FERROUS ION

CALCULATION

Normality of standard ferrous sulphate =weight /litre

Equivalent weight

TITRATION – I

STANDARDISATION OF KMnO4 USING STANDARD FERROUS SULPHATE

S.NO	VOLUME OF FERROUS SULPHATE (ml)	BURETTE READING (ml)		VOLUME OF $KMnO_4$ (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						Self indicator ($KMnO_4$)

Volume of Ferrous sulphate (V_1) =

Normality of Ferrous sulphate (N_1) =

Volume of $KMnO_4$ (V_2) =

Normality of $KMnO_4$ (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

5. ESTIMATION OF OXALIC ACID USING FERROUS ION

Estimate the amount of oxalic acid present in the whole of the given solution. You are provided with pure analar crystals of ferrous sulphate / ferrous ammonium sulphate and an approximately decinormal solution of $KMnO_4$.

AIM:

To estimate the amount of oxalic acid present in the whole of the given solution. Pure analar crystals of ferrous sulphate(FS) / Ferrous ammonium Sulphate(FAS) and an approximately decinormal $KMnO_4$ solution are given.

REQUIREMENTS:

CHEMICALS: Ferrous sulphate,$KMnO_4$, Oxalic acid , dil.H_2SO_4,

GLASSWARES: Burette, pipette,conical flask,standard measuring flask, funnel.

PRINCIPLE:

This is a redox titration. In the presence of dil. H_2SO_4, $KMnO_4$ oxidizes ferrous sulphate(FS) to ferric sulphate and oxalic acid to CO_2 and H_2O.

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

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

$2FeSO_4.(NH_4)_2SO_4.6H_2O + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3 + 2(NH_4)_2SO_4 + 13H_2O$

$\begin{matrix}COOH \\ | \\ COOH\end{matrix} .2H_2O + [O] \longrightarrow 2CO_2 + 3H_2O$

PROCEDURE:

WEIGHT OF FS/FAS FOR PREPARING 0.1N SOLUTION:

Weight of FS in 1litre of the solution = Eq.wt. x 0.1N

= 278 x 0.1

= 27.8g

Weight of FS in 100ml of the solution = $\frac{27.8 \times 100}{1000}$

= 2.78 g

Weight of FAS in 1litre of the solution = Eq.wt. x 0.1N

= 392 x 0.1

= 39.2g

Weight of FAS in 100ml of the solution = $\frac{39.2 \times 100}{1000}$

= 3.92 g

TITRATION – II

STANDARDISATION OF GIVEN OXALIC ACID USING STANDARDISED KMnO4

S.NO	VOLUME OF OXALIC ACID (ml)	BURETTE READING (ml)		VOLUME OF KMnO4 (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						Self indicator KMnO4

Volume of std. KMnO4 (V_1) =

Normality of std. KMnO4 (N_1) =

Volume of given oxalic acid (V_2) =

Normality of given oxalic acid (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

Weight of oxalic acid present in 1 litre = Normality X Equivalent weight

Weight of given oxalic acid present in 100 ml of the solution = $\frac{\text{Normality} \times 63 \times 100}{1000}$

PREPARATION OF STANDARD FS/FAS SOLUTION:

About 2.78g of FS / 3.92g of FAS is weighed accurately, dissolved in distilled water; added a little amount of dil. H_2SO_4 (toprevent hydrolysis) and made upto the mark in a 100ml standard measuring flask using distilled water.

TITRATION – I

STANDARDISATION OF $KMnO_4$ USING STANDARD FS/FAS SOLUTION:

The burette is filled with $KMnO_4$. Accurately 20ml of FS/FAS is pipetted out into a clean conical flask. Equal volume of 2N sulphuric acid is added to the conical flask. The mixture in the conical flask is titrated against $KMnO_4$ in the burette. $KMnO_4$ itself acts as an indicator. The end point is the appearance of permanent pale pink color. The titration is repeated for obtaining concordant value. The readings are noted in the tabular column.

MAKING UP OF THE GIVEN SOLUTION:

The solution given in the small bottle is transferred to a 200ml standard measuring flask; diluted with distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN OXALIC ACID USING STANDARDIZED $KMnO_4$:

The burette is filled with $KMnO_4$. A known volume of 20ml of oxalic acid is pipetted out into a clean conical flask. Equal volume of 2N sulphuric acid is added to the conical flask. The mixture in the conical flask is heated to 70^0C and is titrated against $KMnO_4$ in the burette. $KMnO_4$ itself acts as an indicator. The end point is the appearance of permanent pale pink color. The titration is repeated till the concordant value. The readings are noted in the tabular column. Finally the calculation is done.

Equivalent weight of ferrous sulphate = 278

Equivalent weight of ferrous ammonium sulphate= 392

Equivalent weight of oxalic acid = 63

RESULT:

Amount of oxalic acid present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis , the students are able to;

- take the accurate weight of the substances.
- handle the apparatus
- calculate the strength of the solution.
- calculate the amount of oxalic acid present in the whole of the given solution.

6. ESTIMATION OF FERROUS IONS

CALCULATION

Normality of standard oxalic acid = $\frac{\text{weight /litre}}{\text{Equivalent weight}}$

TITRATION – I

STANDARDISATION OF $KMnO_4$ USING STANDARD FERROUS AMMONIUM SULPHATE

S.NO	VOLUME OF FAS (ml)	BURETTE READING (ml) INITIAL	FINAL	VOLUME OF $KMnO_4$ (ml)	CONCORDANT VALUE (ml)	INDICATOR
						Self indicator ($KMnO_4$)

Volume of ferrous ammonium sulphate(V_1) =

Normality of ferrous ammonium sulphate (N_1) =

Volume of $KMnO_4$ (V_2) =

Normality of $KMnO_4$(N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

6. ESTIMATION OF FERROUS IONS

Estimate the amount of ferrous ions present in the whole of the given ferrous sulphate solution. You are given an approximately decinormal solution of $KMnO_4$ and pure analar crystals of ferrous ammonium sulphate (Mohr's salt).

AIM:

To estimate the amount of ferrous ions present in the whole of the given solution. Pure analar crystals of ferrous ammonium sulphate (Mohr's salt) and an approximately decinormal $KMnO_4$ solution are given.

REQUIREMENTS:

EQUIPMENT :	Digital balance
CHEMICALS :	Ferrous ammonium sulphate,$KMnO_4$,dil.H_2SO_4,
GLASSWARES :	Burette, pipette,conicalflask,standard measuring flask, funnel,

PRINCIPLE:

This is a redox titration. In the presence of dil. H_2SO_4, $KMnO_4$ oxidizes ferrous ammoniumsulphate and ferrous sulphate to ferric sulphate.

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

$2FeSO_4.(NH_4)_2SO_4.6H_2O + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3 + 2(NH_4)_2SO_4 + 13H_2O$

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

PROCEDURE:

WEIGHT OF FAS FOR PREPARING 0.1N SOLUTION:

Weight of FAS in 1litre of the solution = Eq.wt. x 0.1N

= 392 x 0.1

= 39.2g

Weight of FAS in 100ml of the solution = $\frac{39.2}{1000} \times 100$ = 3.92 g

PREPARATION OF STANDARD FAS (MOHR'S SALT) SOLUTION:

About 3.92g of FAS is accurately weighed; dissolved in distilled water; added a little amount of dil. H_2SO_4 and made upto the mark in a 100ml standard measuring flask using distilled water.

TITRATION – I

STANDARDISATION OF $KMnO_4$ USING STANDARD FAS SOLUTION:

The burette is filled with $KMnO_4$. Accurately 20ml of FAS is pipetted out into a clean conical flask. Equal volume of 2N sulphuric acid is added to the conical flask. The mixture in the conical flask is titrated against $KMnO_4$ in the burette. $KMnO_4$ itself acts as an indicator. The end point is the appearance of permanent pale pink color. The titration is repeated till the concordant value is obtained. The readings are noted in the tabular column.

MAKING UP OF THE GIVEN SOLUTION:

The solution given in the small bottle is transferred to a 200ml standard measuring flask; diluted with distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN FERROUS SULPHATE AGAINST STANDARDISED $KMnO_4$

S.NO	VOLUME OF FERROUS SULPHATE (ml)	BURETTE READING (ml)		VOLUME OF $KMnO_4$ (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						Self indicator KMnO4

Volume of std. $KMnO_4$ (V_1) =

Normality of std. $KMnO_4$ (N_1) =

Volume of ferrous sulphate (V_2) =

Normality of ferrous sulphate (N_2) = ?

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V_1N_1}{V_2}$$

Weight of ferrous ions present in 1 litre = Normality of ferrous sulphate X equivalent weight of Fe^{2+}

Weight of given ferrous ions present in whole of the solution=Normality x55.85x200/1000

TITRATION – II

STANDARDISATION OF GIVEN FERROUS SULPHATE USING STANDARDIZED $KMNO_4$:

The burette is filled with $KMnO_4$. Accurately 20ml of ferrous sulphate is pipetted out into a clean conical flask. Equal volume of 2N sulphuric acid is added to the conical flask. The mixture in the conical flask is titrated against $KMnO_4$ in the burette. $KMnO_4$ itself acts as an indicator. The end point is the appearance of permanent pale pink color. The titration is repeated to get the concordant value.The readings are noted in the tabular column. Finally the calculation is done.

Equivalent weight of FAS (Mohr's salt) = 392

Equivalent weight of Ferrous ions = 55.85

RESULT:

Amount of Ferrous ions present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis , the students are able to

- take the accurate weight of the substances.
- handle the apparatus
- calculate the strength of the solution.
- calculate the amount of ferrous ions present in the whole of the given solution.

V.V.Vanniaperumal College for Women, Virudhunagar Tamilnadu.
No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

DICHROMETRY – INTRODUCTION

Dichrometry involves the titrations between the oxidizing agent- potassium dichromate and a reducing agent.

EQUIVALENT WEIGHT:

$K_2Cr_2O_7$ behaves as an oxidizing agent in the medium of dil. H_2SO_4 as per the following equations:

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

$$\text{Equivalent Weight of } K_2Cr_2O_7 = \frac{\text{Molecular weight of } K_2Cr_2O_7}{\text{No. of equivalent weights of oxygen given by one molecule of } K_2Cr_2O_7}$$

$$= \frac{\text{Molecular weight of } K_2Cr_2O_7}{6}$$

$$= 294/6$$

$$= 49$$

Equivalent Weight of $FeSO_4.7\,H_2O$ = 278

Equivalent Weight of $FeSO_4(NH_4)_2SO_4.6H_2O$ = 392

Equivalent Weight of Iron = 55.85

7. ESTIMATION OF $K_2Cr_2O_7$

CALCULATION

TITRATION – I

STANDARDISATION OF FERROUS SULPHATE USING STANDARD K_2Cr2O_7

S.NO	VOLUME OF FERROUS SULPHATE (ml)	BURETTE READING (ml)		VOLUME OF K_2Cr2O_7 (ml)	CONCORDANT VALUE	INDICATOR
		INITIAL	FINAL			
						$K_3Fe(CN)_6$

Volume of std. $K_2Cr2O_7(V_1)$ =

Normality of std. $K_2Cr2O_7(N_1)$ =

Volume of Ferrous sulphate (V_2 =

Normality of Ferrous sulphate(N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V2}$

7. ESTIMATION OF $K_2Cr_2O_7$

Estimate the amount of $K_2Cr_2O_7$ present in the whole of the given solution. You are provided with pure analor crystals of $K_2Cr_2O_7$ and an approximately decinormal solution of ferrous sulphate.

AIM:

To estimate the amount of $K_2Cr_2O_7$ present in the whole of the given solution. Pure analar crystals of $K_2Cr_2O_7$ and an approximately decinormal solution of ferrous sulphate are given.

REQUIREMENTS:

EQUIPMENT:	Digital balance
CHEMICALS:	Potassium dichromate,ferroussulphate,potassiumferricyanide,
GLASSWARES:	Burette, pipette,conicalflask,standard measuring flask, funnel.

PRINCIPLE:

The estimation is based on the reaction between $K_2Cr_2O_7$ and ferrous sulphate in acid medium (redox reaction). The dichromate oxidises ferrous sulphate to ferric sulphate, itself being reduced to green chromic salt.

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow Cr_2(SO_4)_3 + K_2SO_4 + 4H_2O + 3[O]$$

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

The use of potassium ferricyanide as external indicator because of the reaction between $K_3[Fe(CN)_6]$ and ferrous ions which gives intense blue colour.

WEIGHT OF $K_2Cr_2O_7$ FOR PREPARING 0.1N SOLUTION:

Weight of $K_2Cr_2O_7$ in 1litre of the solution = Eq.wt x 0.1N

= 49 x 0.1

= 4.9g

Weight of $K_2Cr_2O_7$ in 100ml of the solution = $\frac{4.9 \times 100}{1000}$

= 0.49 g

PROCEDURE:

PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION:

About 0.49g of pure, analar $K_2Cr_2O_7$ is weighed; transferred to a 100ml Standard measuring flask dissolved in distilled water and made upto the mark using distilled water.

TITRATION – II

STANDARDISATION OF GIVEN K_2Cr2O_7 USING STANDARDISED FERROUS SULPHATE

S.NO	VOLUME OF $FeSO_4$ (ml)	BURETTE READING (ml)		VOLUME OF K_2Cr2O_7 (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						$K_3Fe(CN)_6$

Volume of $FeSO_4(V_1)$ =

Normality of $FeSO_4$ (N_1) =

Volume of given $K_2Cr_2O_7$ (V_2) =

Normality of given $K_2Cr_2O_7(N_2)$ = ?

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V_1N_1}{V_2}$$

Weight of $K_2Cr_2O_7$ present in 1 litre = Normality X Equivalent weight

Weight of given $K_2Cr_2O_7$ Present in the solution 100 ml of = $\frac{\text{Normality x 49x100}}{1000}$

TITRATION – I

STANDARDISATION OF FERROUS SULPHATE USING STANDARD $K_2Cr_2O_7$ SOLUTION:

The burette is filled with standard $K_2Cr_2O_7$ solution. 20ml of ferrous sulphateis pipetted out into a clean 400ml beaker with a glass rod. Equal volume of 2N H_2SO_4 is added to the beaker and dilutedto 150ml. The mixture in the conical flask is titrated against standard $K_2Cr_2O_7$ using potassium ferricyanide as an external indicator. Drops of $K_3[Fe(CN)_6]$ are placed on a clean and dry porcelain tile by means of a glass rod. A drop of the reaction mixture in the beaker is withdrawn from time to time using the glass rod in it; placed nearer to a drop of the indicator and mixed by a glass rod. At first a blue colour is developed indicating the presence of ferrous salt. The addition of standard $K_2Cr_2O_7$ solution from the burette is continued till the reaction mixture does not produce any blue colouration with the indicator, which is the end point of the titration. The titration is repeated for concordant value. The readings are noted in the tabular column.

MAKING UP OF THE GIVEN SOLUTION:

The solution given in the small bottle is transferred to a 100ml Standard measuring flask diluted with distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN $K_2Cr_2O_7$ USING STANDARDIZED $FESO_4$ SOLUTION:

The burette is filled with $K_2Cr_2O_7$ solution. 20ml of ferrous sulphate is pipetted out into a clean 400ml beaker stirred well using a glass rod. Equal volume of 2N H_2SO_4 is added to the beaker and is then dilutedto 150ml. The mixture in the conical flask is titrated against $K_2Cr_2O_7$ using potassium ferricyanide as an external indicator. Drops of $K_3[Fe(CN)_6]$ are placed on a clean and dry porcelain tile by means of a glass rod. A drop of the reaction mixture in the beaker is withdrawn from time to time using the glass rod in it; placed nearer to a drop of the indicator and mixed by a glass rod. At first a blue colour is developed indicating the presence of ferrous salt. The addition of $K_2Cr_2O_7$ solution from the burette is continued till the reaction mixture does not produce any blue colouration with the indicator, which is the end point of the titration. The titration is repeated for concordant value. The readings are noted in the tabular column. Finally the calculation is done using the formula given below.

Equivalent weight of $K_2Cr_2O_7$ = 49

Normality of standard $K_2Cr_2O_7$ = $\frac{\text{weight /litre}}{\text{Equivalent weight}}$

RESULT:

Amount of $K_2Cr_2O_7$ present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis , the students are able to

- take the accurate weight of the substances.
- handle the apparatus
- calculate the strength of the solution.
- calculate the amount of Potassium dichromate (K_2Cr2O_7) present in the whole of the given solution.

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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

IODIMETRY AND IODOMETRY – INTRODUCTION

Iodimetry involves titrations with a solution of Iodine.

Eg. Iodine can be titrated with sodium thiosulphate solution

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

Sodium tetrathionate

Iodometry deals with the titrations of Iodine liberated in chemical reactions.

Eg. (i) Oxidising agents such as $KMnO_4$, $K_2Cr_2O_7$, $CuSO_4$ are treated with a large excess of KI in acid or neutral medium, equivalent amount of iodine is liberated and

(ii) The liberated iodine is titrated against the reducing agent $Na_2S_2O_3$.

(i) $2KMnO_4 + 16HCl + 10KI \longrightarrow 12KCl + 2MnCl_2 + 8H_2O + 5I_2$

$K_2Cr_2O_7 + 14HCl + 6KI \longrightarrow 8KCl + 2CrCl_3 + 7H_2O + 3I_2$

$2CuSO_4 + 4KI \longrightarrow 2K_2SO_4 + Cu_2I_2 + I_2$

(ii) $I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$

Equivalent weight of an oxidizing agent can be defined as the number of parts by weight of it which displaced 126.9 parts by weight (one equivalent weight) of iodine from the solution of potassium iodide

It can be given by the following equation,

$$\textbf{Equivalent weight of an oxidizing agent} = \frac{\text{Molecular weight of the oxidizing agent}}{\text{No. of equivalents of Iodine displaced by a molecule}}$$

According to the chemical equations,

Equivalent weight of $KMnO_4$ $= \frac{\text{Mol.wt}}{5}$

$= \frac{158.0}{5}$

$= 31.6$

Equivalent weight of $K_2Cr_2O_7$ $= \frac{\text{Mol.wt}}{6}$

$= \frac{294.0}{6} = 49.0$

Titration in Iodimetry and Iodometry:

Burette Solution	Solution taken in the conical flask	Indicator	Colour change at the end point
$Na_2S_2O_3$	Iodine	Starch	Blue to colourless
$Na_2S_2O_3$	$KMnO_4$ + dil.HCl + KI	Starch	Blue to colourless
$Na_2S_2O_3$	$K_2Cr_2O_7$ +KI + dil. HCl	Starch	Blue to green
$Na_2S_2O_3$	$CuSO_4$+ NH_4OH +CH_3COOH +KI	Starch	Blue to colourless (with the formation of dirty white precipitate)

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No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

Equivalent weight of $CuSO_4.5H_2O$ $= \frac{Mol.wt}{1} = \frac{249.7}{1} = 249.7$

Equivalent weight of copper = Atomic weight of Cu = 63.57

Equivalent weight of a reducing agent can be defined as the number of parts by weight of it which combines with 126.9 parts by weight (one equivalent weight) of iodine. This can be expressed by the following equation:

$$\text{Equivalent weight of a reducing agent} = \frac{\text{Mol. wt. of the reducing agent}}{\text{No. of equivalents of Iodine with which one molecule combines}}$$

According to the chemical equation,

Equivalent weight of $Na_2S_2O_3$ $= \frac{Mol.wt}{1}$

$= \frac{248.2}{1}$

$= 248.2$

♣♣♣♣♣

8. ESTIMATION OF POTTASIUM DICHROMATE

CALCULATION

Normality of standard $K_2Cr_2O_7$= weight /litre

Equivalent weight

TITRATION – I

STANDARDISATION OF THIO SOLUTION USING STANDARD $K_2Cr_2O_7$ SOLUTION

S.NO	VOLUME OF $K_2Cr_2O_7$ (ml)	BURETTE	READING (ml)	VOLUME OF THIO SOLUTION (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						$K_3Fe(CN)_6$

Volume of std. $K_2Cr_2O_7(V_1)$ =

Normality of std. $K_2Cr_2O_7(N_1)$ =

Volume of Thio solution (V_2) =

Normality of Thiosolution (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

8. ESTIMATION OF POTTASIUM DICHROMATE

Estimate the amount of $KMnO_4$ present in the whole of the given solution. You are provided with pure, analar crystals of $K_2Cr_2O_7$ and an approximately decinormal solution of sodium thiosulphate.

AIM:

To estimate the amount of $KMnO_4$ present in the whole of the given solution. Pure, analar crystals of $K_2Cr_2O_7$ and an approximately decinormal solution of sodium thiosulphate are given.

REQUIREMENTS:

EQUIPMENT:	Digital balance
CHEMICALS:	Potassium dichromate,sodiumthio sulphate,KMno4,starch,2N HCl, 10% KI,
GLASSWARES:	Burette, pipette,conicalflask,standard measuring flask, funnel

.

PRINCIPLE:

This is an iodometric titration. In the presence of HCl, both $K_2Cr_2O_7$ and $KMnO_4$ liberate iodine from KI as per the following equations.

$K_2Cr_2O_7 + 14HCl \longrightarrow 2KCl+2CrCl_3+ 7H_2O + 3Cl_2$

$3Cl_2 + 6KI \longrightarrow 6KCl + 5I_2$

$KMnO_4 + 16HCl \longrightarrow 2KCl + 2MnCl_2 + 8H_2O + 5Cl_2$

$5Cl_2 + 10KI \longrightarrow 10KCl + 5I_2$

The liberated iodine is titrated against sodium thoisulphate solution.

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

WEIGHT OF $K_2Cr_2O_7$ FOR PREPARING 0.1N SOLUTION:

Weight of $K_2Cr_2O_7$ in 1litre of the solution = Eq.wt. x 0.1N

= 49 x 0.1

= 4.9g

Weight of $K_2Cr_2O_7$ in 100ml of the solution = $\frac{4.9 \times 100}{1000}$

= 0.49 g

TITRATION – II

STANDARDISATION OF GIVEN KMnO4 USING STANDARDISED THIO

S.NO	VOLUME OF GIVEN KMnO4 (ml)	BURETTE READING (ml)		VOLUME OF THIO (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
						starch

Volume of Thio solution(V_1) =

Normality of Thiosolution (N_1) =

Volume of $KMnO_4$ (V_2) =

Normality of $KMnO_4$ (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

Weight of $KMnO_4$ present in 1 litre = Normality X Equivalent weight

Weight of KMnO4 present in 100ml of the solution = $\frac{\text{Normality x } 31.6\text{x}100}{1000}$

PROCEDURE:

PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION:

About 0.49g of pure, analar $K_2Cr_2O_7$ is accurately weighed; transferred to a 100ml SMF; dissolved in distilled waterand made upto the mark.

TITRATION – I

STANDARDISATION OF THIO SOLUTION USING STANDARD $K_2Cr_2O_7$ SOLUTION:

The burette is filled with sodium thiosulphatesolution. 10ml of $K_2Cr_2O_7$ is pipetted out into a clean conical flask. 5ml of 2N HCl and 10ml of 10% KI are added to the conical flask. Iodine is liberated. The liberated iodine is titrated against thio solution in the burette until the solution in the conical flask turned straw yellow in colour. Then add 1ml of starch as an indicator. The solution turns deep blue in colour. The deep blue solution is now titrated against thio solution in the burette till the end point is reached. The end point is the change of colour from blue to green. The titration is repeated for concordant value. The readings are tabulated.

MAKING UP OF THE GIVEN SOLUTION:

The given solution is transferred into a 100ml standard measuring flask. It is diluted with distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN $KMnO_4$ USING STANDARDIZED THIO SOLUTION:

The burette is filled with sodium thiosulphatesolution. 10ml of $KMnO_4$ is pipetted out into a clean conical flask. 5ml of 2N HCl and 10ml of 10% KI are added to the conical flask. Iodine is liberated. The liberated iodine is titrated against thio solution in the burette until the solution in the conical flask turned straw yellow in colour. Then added 1ml of starch as an indicator. The solution turns deep blue in colour. The deep blue solution is now titrated against thiosolution in the burette till the end point is reached. The end point is the change of colour from blue to colourless. The titration is repeated for concordant value. The readings are tabulated. Finally the calculation is done.

Equivalent weight of $K_2Cr_2O_7$ = 49

Equivalent weight of $KMnO_4$ = 31.6

V.V.Vanniaperumal College for Women, Virudhunagar Tamilnadu.
No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

RESULT

Amount of $KMnO_4$ present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis , the students are able to

- take the accurate weight of the substances.
- handle the apparatus
- calculate the strength of the solution.
- calculate the amount of $KMnO_4$ present in the whole of the given solution.

9. ESTIMATION OF COPPER

CALCULATION

Normality of standard $CuSO_4$= weight /litre
Equivalent weight

TITRATION – I

STANDARDISATION OF THIO SOLUTION USING STANDARD $CuSO_4$ SOLUTION

S.NO	VOLUME OF $CuSO_4$ SOLUTION (ml)	BURETTE READING (ml) INITIAL	FINAL	VOLUME OF THIO (ml)	CONCORDANT VALUE (ml)	INDICATOR
1	20					Starch
2	20					

Volume of std. $CuSO_4$ (V_1) =
Normality of std. $CuSO_4$ (N_1) =
Volume of Thio solution (V_2) =
Normality of Thio solution (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

9. ESTIMATION OF COPPER

Estimate the amount of copper present in the whole of the given solution. You are given pure, analar crystals of copper sulphate and an approximately decinormalthio solution.

AIM:

To estimate the amount of copper present in the whole of the given solution. Pure, analar crystals of copper sulphate and an approximately decinormal solution of thio solution are given.

REQUIREMENTS:

EQUIPMENT:	Digital Balance
CHEMICALS:	Copper Sulphate, sodiumthiosulphate,ammoniumhydroxide, acetic acid,10% Potassium iodide (KI),starch,
GLASSWARES:	Burette, pipette,conical flask,standard measuring flask, funnel.

PRINCIPLE:

This is an iodometric titration. Iodometry deals with the titration of iodine liberated in the chemical reaction.Copper sulphate reacts with large excess of KI to liberate an equivalent amount of iodine.

$CuSO_4 + 2KI \longrightarrow CuI_2 + K_2SO_4$

$2CuI_2 \longrightarrow Cu_2I_2 + I_2$

The liberated iodine is titrated against thio solution.

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

WEIGHT FOR PREPARING 0.1N $CuSO_4$ SOLUTION:

Weight of $CuSO_4$ in 1litre of the solution = Eq.wt .x 0.1N
= 249.7 x 0.1
= 24.97g

Weight of $CuSO_4$ in 100ml of the solution = $\frac{24.97 \times 100}{1000}$
= 2.497 g

PROCEDURE:

PREPARATION OF STANDARD $CuSO_4$ SOLUTION

About 2.5g of pure, analar $CuSO_4$ is accurately weighed; transferred to a 100ml SMF; dissolved in distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN $CuSO_4$ USING STANDARDISED THIO SOLUTION

S.NO	VOLUME OF $CuSO_4$ (ml)	BURETTE READING (ml)		VOLUME OF THIO SOLUTION (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
1	20					starch
2	20					

Volume of Thio solution (V_1) =

Normality of Thio solution (N_1) =

Volume of given $CuSO_4$solution (V_2) =

Normality of given $CuSO_4$solution (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

Weight of copper present in 1 litre = Normality X Equivalent weight

Weight of given of copper present in100 ml of the solution = $\frac{\text{Normality x 63.54x100}}{1000}$

TITRATION – I

STANDARDISATION OF THIOSOLUTION USING STANDARD COPPER SULPHATE SOLUTION:

The burette is filled with sodium thiosulphatesolution. 10ml of copper sulphate is pipetted out into a clean conical flask. Ammonium hydroxide is added drop by drop till a permanent blue colourprecipitate is formed. This precipitate is redissolved by adding little amount of acetic acid. Then about 10ml of 10% KI is added. Iodine is liberated. The liberated iodine is titrated against thio solution in the burette. The solution becomes straw yellow in colour. Now 1ml of starch is added as an indicator. The solution turns deep blue in colour. The titration is continued till the end point is reached.The end point is the disappearance of blue colour with the formation of dirty white precipitate. The titration is repeated for concordant value. The readings are tabulated.

MAKING UP OF THE GIVEN SOLUTION:

The solution given in the small bottle is transferred to a 100ml SMF; diluted with distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN $CuSO_4$ USING STANDARDIZED THIO SOLUTION:

The burette is filled with sodium thiosulphate solution. 10ml of copper sulphate is pipetted out into a clean conical flask. Ammonium hydroxide is added drop by drop till a permanent blue colourprecipitate is formed. This precipitate is redissolved by adding little amount of acetic acid. Then about 10ml of 10% KI is added. Iodine is liberated. The liberated iodine is titrated against thio solution in the burette. The solution becomes straw yellow in colour. Now 1ml of starch is added as an indicator. The solution turns deep blue in colour. The titraton is continued till the end point is reached. The end point is the disappearance of blue colour with the formation of dirty white precipitate. The titration is repeated for concordant value. The readings are tabulated. Finally the calculation is done.

Equivalent weight of $CuSO_4$ = 249.7

Equivalent weight of copper = 63.54

RESULT

Amount of copper present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis , the students are able to;

- take accurate weight of the substances.
- handle the apparatus.
- calculate the strength of the solution.
- calculate the amount of oxalic acid present in the whole of the given solution.

10. ESTIMATION OF IODINE

CALCULATION

Normality of standard $CuSO_4$ = weight /litre
Equivalent weight

TITRATION – I

STANDARDISATION OF THIO SOLUTION USING STANDARD $CuSO_4$

SOLUTION

S.NO	VOLUME OF $CuSO_4$ SOLUTION (ml)	BURETTE READING (ml) INITIAL	FINAL	VOLUME OF THIO (ml)	CONCORDANT VALUE (ml)	INDICATOR
1	20					starch
2	20					

Volume of std. $CuSO_4$ (V_1) =
Normality of std. $CuSO_4$ (N_1) =
Volume of thio solution (V_2) =
Normality of thio solution (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

10. ESTIMATION OF IODINE

Estimate the amount of iodine present in the whole of the given solution. You are given pure, analar crystals of copper sulphate ($CuSO_4$) and an approximately decinormal solution of thiosulphate.

AIM:

To estimate the amount of iodine in the whole of the given solution. Pure, analar crystals of $CuSO_4$ and an approximately decinormal solution of thiosulphate are given.

REQUIREMENTS:

EQUIPMENT:	Digital Balance
CHEMICALS :	Copper sulphate ($CuSO_4$), sodiumthiosulphate, iodine, ammonium hydroxide,acetic acid, 10% KI, starch,
GLASSWARES:	Burette, pipette,conicalflask,standard measuring flask, funnel.

PRINCIPLE:

This is a combination of iodometric and iodimetric titration. Iodometry deals with the titration of iodine liberated in chemical reaction.Coppersulphate reacts with excess of KI to liberate an equivalent amount of iodine.

$CuSO_4 + 2KI \longrightarrow CuI_2 + K_2SO_4$

$2CuI_2 \longrightarrow Cu_2I_2 + I_2$

The liberated iodine is titrated against thio.

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

Iodimetry involves titration with a solution of iodine.

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

WEIGHT OF $CuSO_4$ FOR PREPARING 0.1N SOLUTION:

Weight of $CuSO_4$ in 1litre of the solution = Eq.wt. x 0.1N
= 249.7 x 0.1
= 24.97g

Weight of $CuSO_4$ in 100ml of the solution = $\frac{24.97 \times 100}{1000}$
= 2.497 g

PROCEDURE:

PREPARATION OF STANDARD $CuSO_4$ SOLUTION:

About 2.5g of pure, analar $CuSO_4$ is accurately weighed; transferred to a 100ml SMF; dissolved in distilled water and made upto the mark using distilled water.

TITRATION – II

STANDARDISATION OF GIVEN IODINE USING STANDARDISED THIO SOLUTION

S.NO	VOLUME OF GIVEN IODINE SOLUTION (ml)	BURETTE READING (ml)		VOLUME OF THIO SOLUTION (ml)	CONCORDANT VALUE (ml)	INDICATOR
		INITIAL	FINAL			
1	20					Starch
2	20					

Volume of thio solution (V_1) =
Normality of thio solution (N_1) =
Volume of given iodine solution (V_2) =
Normality of given iodine solution (N_2) = ?

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}$

Weight of iodine present in 1 litre = Normality X Equivalent weight

Weight of given iodine Present in 100 ml of the solution = $\frac{\text{Normality} \times 126.9 \times 100}{1000}$

TITRATION – I

STANDARDISATION OF THIO SOLUTION USING STANDARD COPPER SULPHATE SOLUTION:

The burette is filled with thiosolution. 10ml of copper sulphate is pipetted out into a clean conical flask. Ammonium hydroxide is added drop by drop till a permanent blue precipitate is formed. This precipitate is redissolved by adding little amount of acetic acid. Then about 10ml of 10% KI is added. Iodine is liberated. The liberated iodine is titrated against thio solution in the burette. The solution becomes straw yellow in colour. Now 1ml of starch is added as an indicator. The solution turns deep blue in colour. The titration is continued till the end point is reached. The end point is the disappearance of blue colour with the formation of dirty white precipitate. The titration is repeated for concordant value. The readings are noted in the tabular column.

MAKING UP OF THE GIVEN SOLUTION:

The solution given in the small bottle is transferred to a 100ml SMF; diluted with distilled water and made upto the mark.

TITRATION – II

STANDARDISATION OF GIVEN IODINE SOLUTION USING STANDARDIZED THIO SOLUTION:

The burette is filled with thio solution 10ml iodine solution is pipetted out into a clean conical flask. The solution is titrated against thio solution in the burette. The solution turns straw yellow in colour. Now 1ml of starch is added as an indicator. The solution turns deep blue in colour. The titration is continued till the end point is reached. The end point is the disappearance of blue colour. The titration is repeated for concordant value. The readings are tabulated. Finally the calculation is done.

Equivalent weight of $CuSO_4$ = 249.7

Equivalent weight of Iodine = 126.9

RESULT: Amount of Iodine present in the whole of the given solution is _____ g.

NUMBER OF BENEFICIARIES: 50

OUTCOME: After the completion of the volumetric analysis , the students are able to;

- Take accurate weight of the substances.
- Handle the apparatus.
- Calculate the strength of the solution.
- Calculate the amount of the oxalic acid present in the whole of the given solution.

11. ESTMATION OF ARSENIOUS OXIDE

CALCULATION

Normality of standard As_2O_3 = weight /litre

———————

Equivalent weight

TITRATION I

STANDARDISATION OF IODINE SOLUTION USING STANDARD ARSENIOUS OXIDE.

S.No	Volume of standard As_2O_3 solution (ml)	Burette reading (ml)		Vloume of Iodine solution (ml)	concordant value (ml)	Indicator
		Initial	Final			
1	10					1ml of starch
2	10					

Volume of standard As_2O_3 =(V_1)

Normality of standard As_2O_3 =(N_1)

Volume of Iodine solution =(V_2)

Normality of Iodine soulution N_2 = ?

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V2}$$

11. ESTMATION OF ARSENIOUS OXIDE

Estimate the amount of arsenious oxide present in the whole of the given solution. You are provided with pure analor crystals of As_2O_3 and an approximately decinormal solution of Iodine.

AIM:

To estimate the amount of arsenious oxide present in the whole of the given solution.

REQUIREMENTS:

CHEMICALS:

Arsenious oxide (As_2O_3), Iodine(I_2), Sodium hydroxide (NaOH), dilute Hydrocholoric acid (HCl), Phenolphthalein, sodium bicarbonate, starch.

GLASSWARES:

Burette, Pipette, conical flash, funnel, Standard measuring flask. (s.m.f)

PRINCIPLE:

Iodine oxidisesarsenious oxide, since the reaction is reversible, all the hydrogen iodide, formed must be removed as soon as sodium bicarbonate added in excess which removes hydrogen iodide and it has no action on iodine. Arsenious oxide is not freely soluble in water and it is transformed into sodium arsenite

$$As_2O_3 + 6NaoH \longrightarrow 2Na_3AsO_4 + 3H_2O$$

$$Na_2AsO_3 + I_2 + H_2O \longrightarrow Na_3AsO_4 + 3HI$$

PROCEDURE:

PREPARATION OF STANDARD ARSENIOUS OXIDE SOLUTION

About 0.5g of arsenious oxide is accurately weighed and transferred into beaker carefully. A few drops of distilled water and 2 on 3 pellets of sodium hydroxide is added and stirred well to dissolve arseniousoxide.The solution is transferred into a 100 ml standard measuring flask.

TITRATION I

STANDARDISATION OF IODINE SOLUTION USING STANDARD ARSENIOUS OXIDE:

The burette is filled with Iodine solution. 10ml of standard arsenious oxide is pipetted out into a clean conical flask. A drop of Phenolphthalein is added. Following dilute HClis added drop by drop till the pink colourdischarges, the spoon full of sodium bicarbonate is added. About 10ml of freshly prepared starch solution is added and titrated against Iodine solution taken in the burette. The end point is the appearance of blue colour. The titration is repeated to get concordant value. From the titre value, the strength of the Iodine is calculated.

TITRATION II

Standardation of given arsenious oxide using standardized iodine Solution

S.No	Volume of standard As_2O_3 solution (ml)	Burette reading (ml)		Vloume of Iodine solution (ml)	concordant value (ml)	Indicator
		Initial	Final			
1	10					1ml of starch
2	10					

Volume of Iodine solution (V_1) =

Normality of Iodine solution (N_1) =

Volume of given As_2O_3 N2 = $\frac{V_1 N_1}{V_2}$

Weight of given As_2O_3 in 100ml of the given solution

$$= \frac{\text{Normalilty of given } As_2O_3 \text{ x Equivalent weight of } As_2O_3}{1000} \text{ X } 100$$

$$= \frac{\text{Normality of given } As_2O_3 \text{ x49.46 x100}}{1000}$$

TITRATION II

STANDARDISATION OF GIVEN ARSENIOUS OXIDE USING STANDARDISED IODINE SOLUTION.

The given arsenious oxide solution is made upto 100ml in a standard measuring flask. The burette is filled with Iodine solution. 10ml of standard arsenious oxide is pipetted out into a clean conical flask. A drop of Phenolphthalein is added followed by adding dilute Hcl drop by drop till the pink colour discharges about the spoon full of sodium bicarbonate is added. About 10ml of freshly prepared starch solution is added and titrated against Iodine solution is taken in the burette. The end point is the appearance of blue colour. The titration is repeated to get concordant value. The strength of arsenious oxide is calculated and the amount of arsenious oxide in the whole of the given solution is calculated, using the following formula Equivalent weight of arsenious oxide = 49.46

RESULT:

The weight of the arseious oxide in the whole of the given solution is ----------g.

NUMBER OF BENEFICIARIES: 50

OUTCOME:

After the completion of this volumetric analysis students are able to.

- Take accurate weight of the substance
- Handle the apparatus.
- Calculate the strength of the solution.
- Calculate the amount of arsenious oxide present in the whole of the given solution.

12. ESTIMATION OF KCL

CALCULATION

Equivalent weight of KCl = 74.55

The amount of KCl in the whole of the given solution

$$\text{Normality of Standard NaCl} = \frac{\text{weight/litre}}{\text{Equivalent weight}}$$

$$= \frac{\text{weight perlitre}}{58.5}$$

TITRATION I:

STANDARDISATION OF $AgNO_3$ USING STANDARD NaCl

S.No	Volume of NaCl (ml)	Burette reading (ml)		Volume of $AgNO_3$ (ml)	concordant value (ml)	Indicator
		Initial	Final			
1	20					1ml of 1% K_2CrO_4
2	20					

Volume of Standard NaCl (V_1)

Normality of Standard NaCl (N_1)

Volume of $AgNO_3$ (V_2)

$$\text{Normality of } AgNO_3 \ (N_2) = \frac{V_1N_1}{V_2}$$

12. ESTIMATION OF KCl

AIM:

To estimate the amount of chloride present in the whole of the given solution of KCl pure crystals of NaCl and an approximately decinormal solution of $AgNO_3$.

REQUIREMENTS:

CHEMICALS:	Potassium chloride (KCl), Sodium chloride (NaCl), Silver nitrate ($AgNO_3$), potassium chromate.
GLASSWARES:	Burette, pipette, conicalflash, S.M.F, Funnel.

PRINCIPLE:

Silver nitrate reacts with NaCl and KCl. Potassium chromate is used as an indicator.

$AgNO_3 + NaCl \longrightarrow AgCl + NaNO_3$

$AgNO_3 + KCl \longrightarrow AgCl + KNO_3$

$K_2CrO_4 + 2AgNO_3 \longrightarrow Ag_2CrO_4 + 3KNO_3$

PROCEDURE:

PREPARATION OF STANDARD SOLUTION OF NACL

About 0.4 g of pure NaCl crystals are weighed accurately and transferred to a 100ml standard measuring flask. The crystals are dissolved in pure distilled water and the solution is made up to the mark.

Equivalent weight of NaCl = 58.5

TITRATION I

STANDARDISATION OF $AgNO_3$ USING STANDARD NaCl

Burette is filled with Silver nitrate solution. Accurately 20ml of NaCl solution is pipetted out into a clean conical flask. To this solution, 1 ml of 1% potassium chromate indicator is added. The solution is titrated against $AgNO_3$ taken in the burette. Near the end point, coagulation of silver chloride takes place. The end point is the first appearance of slight reddish brown colour. The titration is repeated for concordant value. From this value, the normality of $AgNO_3$ solution is calculated.

MAKING UP OF THE GIVEN SOLUTION:

The given solution of given KCl is made upto 100 ml in a standard measuring flask using distilled water and shake well for uniform concentration.

TITRATION II

STANDARDISATION OF KCl USING STANDARDISED $AgNO_3$

S.No	Volume of KCl given (ml)	Burette reading (ml)		Volume of $AgNO_3$ (ml)	concordant value (ml)	Indicator
		Initial	Final			
1	20					1ml of 1% K_2CrO_4
2	20					

Volume of $AgNO_3(V_1)$ =

Normality of $AgNO_3(N_1)$ =

Volume of given $KCl(V_2)$

$$\text{Normality of given KCl } (N_2) = \frac{V_1N_1}{V_2}$$

Weight of given KCl in 100ml of the given solution

$$= \frac{\text{Normality of KCl X Equivalent weight of KCl X 100}}{1000}$$

$$= \frac{\text{Normality of KCl X 74.55 X 100}}{1000}$$

TITRATION II

STANDARDISATION OF KCl USING STANDARDISED $AgNO_3$

A known volume of 20 ml of the given solution is pipetted out into a clean conical flask. About 1 ml of 1% potassium chromate solution is added and this solution is titrated against silver nitrate solution taken in the burette. Near the end point coagulation of AgCl taking place. The end point is the first appearance of a reddish brown colour. The titration is repeated to get concordant titre value. From this value, the normality of given KCl and then the weight of KCl in the whole of the given solution is calculated using the formula

RESULT:

The weight of the chloride present in the whole of the given solution is ----------g.

NUMBER OF BENEFICIARIES: 50

OUTCOME:

After the completion of this volumetric analysis students are able to

- take accurate weight of the substance
- handle the apparatus.
- calculate the strength of the solution.
- calculate the amount of potassium chloride present in the whole of the given solution.

13.ANALYSIS OF SODIUM LEVEL IN VARIOUS JUNK FOODS BY FLAME PHOTOMETER

CALCULATIONS:

CALCULATION OF SODIUM ELEMENT:

1000ppm of standard solution contain 254 mg of NaCl which contains 100mg of Na.

Therefore, X ppm of sample solution contains,

$$\text{Amount of sodium} = \frac{\mathbf{X}\text{x } 100\text{mg}}{1000}$$

Therefore, amount of sodium = y mg/100mL

V.V.Vanniaperumal College for Women, Virudhunagar Tamilnadu.
No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 6

13.ANALYSIS OF SODIUM LEVEL IN VARIOUS JUNK FOODS BY FLAME PHOTOMETER

AIM: To analyze sodium level in various junk foods by using flame photometer.

PRINCIPLE OF FLAME PHOTOMETER

The compounds of the alkali and alkaline earth metals (Group II) dissociate into atoms when introduced into the flame. Some of these atoms further get excited to even higher levels. But these atoms are not stable at higher levels.

Hence, these atoms emit radiations when returning back to the ground state. These radiations generally lie in the visible region of the spectrum. Each of the alkali and alkaline earth metals has a specific wavelength.

Element	Emitted wavelength	Flame color
Sodium	589 nm	Yellow
Potassium	766 nm	Violet
Barium	554 nm	Lime green
Calcium	622 nm	Orange
Lithium	670 nm	Red

For certain concentration ranges,

The intensity of the emission is directly proportional to the number of atoms returning to the ground state. And the light emitted is in turn proportional to the concentration of the sample.

EQUIPMENT:	Flame Photometer, Electrical Balance
GLASSWARES:	Conical flasks, Beakers, Pipettes, Measuring cylinders, Standard measuring flasks
CHEMICALS:	Deionized water, Hydrochloric acid (HCl): 0.5% HCl(V/V) (to be used for standards), Sodium chloride (NaCl): For 1000ppm weigh 635mg of NaCl and dissolve it in 250mL of deionized water in a 250 mL standard flask, Potassium chloride (KCl): For 1000ppm weigh 477mg of KCl and dissolve it in 250mL of deionized water ina 250mL standard flask.

QUALITY OF SALTS REQUIRED:

1000ppm standard solution should have 1000mg of elements in 1000mL salt solution of the element. Thus 100mL standard solution should be made up of a quantity of salt of the element in mg., equivalent to (250 x Molecular Wt. salt / Atomic Wt. of element), to contain 250mg of element.

Element	Atomic Wt of element	Salt	Molecular Wt. of salt	Quantity of salt in mg. required for preparation of 100mL of 1000ppm standard solution
Na	22.99	NaCl	58.44	254
K	39.10	KCl	74.56	190.8
Ca	40.08	$CaCO_3$	100.09	249.6
Li	6.94	Li_2CO_3	73.89	532.4

PREPARATION OF STANDARD SOLUTIONS:

STOCK STANDARD SOLUTION

Weigh accurately 254 mg of analar quality of sodium chloride (NaCl) into a 100mL volumetric flask through a funnel. Weigh accurately 190.8 mg analar quality of potassium chloride (KCl) and transfer it into the same volumetric flask through the same funnel. Add double distilled water to the flask, dissolve the crystals and make up the solution to the mark with double distilled water. This stock standard solution contains 1000mEq/100mL of sodium(Na) and 1000mEq/100mL potassium(K).

WORKING STANDARD SOLUTIONS:

This Stock Standard solution is diluted 1:100 with double distilled water (10ML of Stock Standard solution make upto 1 Litre). This diluted Stock Standard solution is further diluted as below to obtain a series of Working Standard solutions of Na & K.

TABLE SHOWS THE AMOUNT OF SODIUM IN FOOD SAMPLES

SAMPLE NAME	ppm VALUES (1:10)	MILLIGRAM SODIUM PER 5g SAMPLE	MILLIGRAM SODIUM PER 100g SAMPLE
CHEESE			
NUTRILITE BUTTER			
PANI PURI MASALA			
PAV BHAJI MASALA			
MAGGIE MASALA			
BISCUIT			
LAYS CHIPS			
ACT II POPCORN			
KURKURE			
KISSAN TOMATO SAUCE			
SOYA SAUCE			
CADBURY CHOCOLATE			
APPY FIZZ			
COCA COLA			
AJINOMOTO			

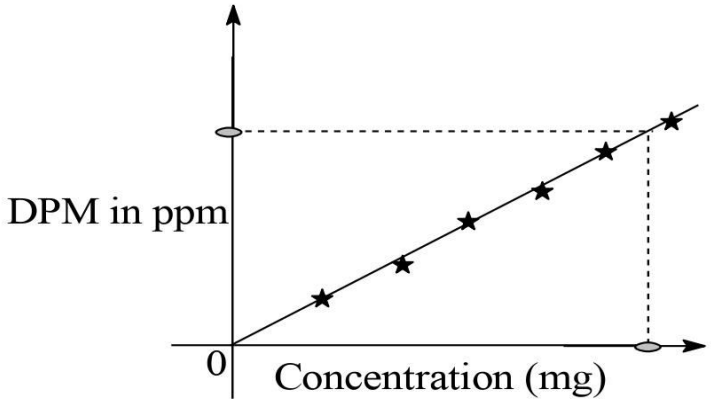
S. No	Diluted Stock Standard solution in mL	Double distilled water in mL	Concentration of mEq/L of Working Standard solution of Na & K (in ppm)
1	4	96	40
2	6	94	60
3	8	92	80
4	10	90	100
5	12	88	120

Samples: The samples used are foods that are rich in sodium levels. They are:

1.Amul Cheese
2. Nutrilite Butter
3. Panipuri masala
4. Pavbhaji masala
5. Maggie masala
6. Britannia Cashew Biscuits
7. Lays chips
8. Act II Popcorn
9. Kurkure
10. Kissan Tomato sauce
11. Weikfield Soya sauce
12. Cadbury chocolate
13. Appy fizz
14. Coca cola
15. Ajinomoto

SAMPLE PROCESSING:

The sample is prepared by weighing 5g of each (diced or ground) into 500mL Erlenmeyer flasks. Add 50 mL of the above working standard solution. Bring each to a boil on a hot plate, and then simmer for 5 min. Cool and filter each through Whatmann 1 filter paper into flasks. Now quantitatively transfer the supernatant for each to separate 100mL volumetric flasks. Dilute to the mark with distilled water and shake. This extract is used to prepare the appropriate dilutions required for the experiment.



WORKING PROCEDURE:

- Both the standard stock solution and sample solution are prepared in fresh distilled water.
- The flame of the photometer is calibrated by adjusting the air and gas. Then the flame is allowed to stabilize for about 5 min.
- Now the instrument id switched on and the lids of the filter chamber are opened to insert appropriate colour filters.
- The readings are adjusted to zero by sparaying distilled water into the flame.
- The sensitivity is adjusted by spraying the most concentrated standard working solution into the flame. Now the full scale deflection is recorded.
- Again distilled water is sprayed into the flame to attain constant readings of galvanometer. Then the galvanometer is readjusted to zero.
- Now each of the standard working solutions is sprayed into the flame for three times and the readings of galvanometer are recorded. After each spray, the apparatus must be thoroughly washed.
- Finally sample solution is sprayed into the flame for three times and the readings of galvanometer are recorded. After each spray, the apparatus must be thoroughly washed.
- Calculate the mean of the galvanometer reading.
- Plot the graph of concentration against the galovanometer reading to find out the concentration of the element in the sample.

The Flame Photometer has simplified the process. When calibrating, the Flame Photometer will prompt the operator to aspirate the Blank and Standards (with the option of one or several Standards) and enter their numerical values. The Flame Photometer electronically monitors for stable readings, stores all data points, develops the calibration curve, and interprets the sample readings.

RESULT:

1. Concentration of sodium level in unknown sample = ------- (from graph)
2. Table: Levels of Sodium in 1: 10 diluted sample

NO. OF BENEFICIARIES:45 students

OUTCOME: The students will be able to

- determine the levels of sodium in junk food and understand the benefits of sodium in diet and risks of high sodium in diet.
- analyze sodium levels in junk food, this can be used to create awareness among the younger generation to prevent problems like hypertension, hypernatremia and associated medical complications in their future.
- establish a procedure for analyzing sodium levels in various junk foods by Flame photometry and have a comparative study with processed foods, vegetables and fruits from the literature.