KARPAGAM ACADEMY OF HIGHER EDUCATION
(Deemed to beUniversity Established Under Section 3 of UGC Act 1956)

DEPARTMENT OF CHEMISTRY
SYLLABUS

17CHU212 S-BLOCK AND P- BLOCK ELEMENTS - PRACTICAL 4H 2C

Instruction Hours/week:L: 0 T:0 P:4 Marks: Internal: 40 External: 60 Total:100

Scope
This is a laboratory course that provides an introduction to basic techniques for the estimation of metals by iodimetric and complexometric titrations. It also deals with the preparations of inorganic metal complexes

Objectives
1. To estimate the metal ions by iodimetric titrations
2. To estimate the metal ions by complexemetric titrations using EDTA
3. To carry out the preparations of inorganic metal complexes.

Methodology
Iodimetric titrations, Complexometric titrations, Inorganic preperations.

(A) Iodo / Iodimetric Titrations
- (i) Estimation of Cu(II) and $K_2Cr_2O_7$ using sodium thiosulphate solution (Iodometrically).
- (ii) Estimation of antimony in tartar-emetic iodimetrically

(B) Complexometric titrations using disodium salt of EDTA
- (i) Estimation of Mg^{2+}, Zn^{2+}
- (ii) Estimation of Ca^{2+} by substitution method

(C) Inorganic preparations
- (i) Cuprous Chloride, Cu_2Cl_2
- (ii) Manganese (III) phosphate, $MnPO_4.H_2O$
- (iii) Aluminium potassium sulphate $KAl(SO_4)_2.12H_2O$ (Potash alum) or Chrome alum.

Suggested Readings

Text Books

1. Vogel, A.I. (1978). *A Textbook of Quantitative Inorganic Analysis*, ELBS.

Reference Books

2. Marr, G. and Rockett, R.W. (1972). *Practical Inorganic Chemistry,* Van Nostrand Reinhold.
3. Deepak Pant.P. (2010). *Inorganic Chemistry Practical*, BookRix.

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KARPAGAM ACADEMY OF HIGHER EDUCATION

Coimbatore-21

DEPARTMENT OF CHEMISTRY

LECTURE PLAN

S-BLOCK AND P-BLOCK ELEMENTS PRACTICAL

Name of the Faculty	: Dr.M.R.Ezhilarasi & B. Prabha	Department	: CHEMISTRY
Semester	: II	Year	: I
Course Code	: 17CHU212	Section	: A & B

Total no. of hour's: 60 Hours

S.NO	EXPERIMENT TOPICS	SUPPORT MATERIALS
1	Writing the experimental Procedure	
2	Estimation of Cu(II) using sodium thiosulphate solution (Iodometrically).	**T1:135,T2:108**
3	Estimation of $K_2Cr_2O_7$ using sodium thiosulphate solution (Iodometrically).	**T1:132,T2:110**
4	Estimation of Mg^{2+}	**T1:152,T2:121**
5	Estimation of Zn^{2+}	**T1:150**
6	Estimation of Ca^{2+} by substitution method	**T1:152,T2:120**
7	Viva-voce	
8	Estimation of available Chlorine in bleaching powder	**T1:139**
9	Preparation of Cuprous Chloride, Cu_2Cl_2	**T1:78,T2:138**
10	Preparation of Manganese (III) phosphate, $MnPO_4.H_2O$	**W1**
11	Preparation of Aluminium potassium sulphate $K_2\ SO_4\ Al(SO_4)_2.12H_2O$ (Potash alum)	**T2:140**
12	Preparation of Chrome alum.	**T2:138**
13	Viva-voce	
14	Revision	
15	Model Exam	

Support Materials:

Text books :

T1: V.Venkateswaran, R.Veerasamy and A.R.Kulandaivelu. Basic Principles of Practical Chemistry S.Chand and Sons, New Delhi

T2:O.P.Pandey, D.N.Bajpai and S.Giri.Practical Chemistry.2001, S.Chand and Company, New Delhi

KARPAGAM ACADEMY OF HIGHER EDUCATION

COIMBATORE – 21

DEPARTMENT OF CHEMISTRY

INORGANIC CHEMISTRY PRACTICAL-II

(S-BLOCK AND P-BLOCK ELEMENTS)

PRACTICAL MANUAL

CONTENT

ESTIMATION OF COPPER

CALCULATION:

Weight of the $K_2Cr_2O_7$ present in the whole of the given solution = W g

Normality of standard $K_2Cr_2O_7$ $= \frac{W \times 4}{Eq.mass\ of\ K_2Cr_2O_7}$

$= \frac{W \times 4}{49}$

N_1 = ------- N

TITRATION-I: Standardization of Sodium Thiosulphate

(Std. $K_2Cr_2O_7$ solution Vs Sodium thio solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**x**
2.					
3.					

Volume of Std. $K_2Cr_2O_7$ solution V_1 = 20ml
Normality of Std. $K_2Cr_2O_7$ solution N_1 = 0.1 N
Volume of Sodium thio solution V_2 = x V
Normality of Sodium thio solution N_2 = ? M

$$V_1N_1 = V_2N_2$$

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

Normality of Sodium thio solution N_2 = y N

TITRATION-II: Estimation of Copper

(Std. Sodium thio sulphate solution Vs Unknown solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**z**
2.					
3.					

ESTIMATION OF COPPER

AIM

To estimate the amount of Copper present in the whole of the given solution being supplied with pure $K_2Cr_2O_7$ crystals.

PRINCIPLE

Copper is precipitated as cuprous iodide in acetic acid medium and an equivalent amount of iodine is liberated. The liberated iodine is titrated against sodium thiosulphate using starch as an indicator. In a similar manner the given copper sulphate solution is titrated against standard thio solution

$$KI + CH_3COOH \longrightarrow CH_3COOK + HI$$

$$2\ CuSO_4 + 4HI \longrightarrow 2CuI_2 + 2\ H_2SO_4$$

$$2\ CuI_2 + 2\ H_2SO_4 \longrightarrow Cu_2I_2 + I_2 + 2\ Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2\ NaI$$

Equivalent mass of Copper = Molecular mass of copper = 63.54

Equivalent mass of Copper Sulphate ($CuSO_4.5H_2O$) = Molecular mass of Copper Sulphate ($CuSO_4.5\ H_2O$) = 249.6

PROCEDURE

(a) Preparation of standard N/10 $K_2Cr_2O_7$ Solution

Accurately about 0.49 g of $K_2Cr_2O_7$ of AR quality is weighed in a chemical balance. It is transferred into 100 ml flask and the solution is made up to the mark. The solution is shaken well to get a homogenous solution. Solution of this known strength is taken in the burette after thoroughly rinsing with it.

Titration-I

Standardization of Sodium Thio Sulphate solution

(Std. $K_2Cr_2O_7$ solution Vs Sodium thiosulphate solution)

The burette is filled with the given sodium thiosulphate solution. Exactly 20 ml of standard $K_2Cr_2O_7$ solution is pipetted out into a clean conical flask. 5 ml of Con. HCl is added (20 ml of dil. HCl) followed by 10 ml of 10% KI (2g of solid KI) solution. The brown coloured solution liberates iodine which is titrated against thiosulphate taken in the burette. When the colour of the solution becomes pale yellow, about 2 ml of freshly prepared starch solution is added.

Volume of Sodium thio solution V_1 = z V
Normality of Sodium thio solution N_1 = y N
Volume of Given Unknown solution V_2 = 20ml
Normality of Given Unknown solution N_2 = ?

$$V_1N_1 = V_2N_2$$
$$N_2 = \frac{V_1N_1}{V_2}$$

Normality of Given Unknown Solution N_2 = a N

The amount of Copper present in the whole of the given solution

$$= \frac{Normality\ (a) \times Equivalent\ mass\ of\ Cu}{10}$$

$$= \frac{a \times 63.54}{10}$$

= ---------- g

A blue colour is obtained. The titration is continued till the end point is reached. The end point is the disappearance of blue colour leaving behind a green colour. The titration is repeated for concordant values. From the titre values the strength of thiosulphate is calculated.

Titration – II

Estimation of Copper

(Std. Sodium thio sulphate Vs Unknown Solution)

The given Unknown solution is made up to 100 ml in a standard flask. Exactly 20 ml of the made up solution is pipette out into a clean conical flask. Ammonium Hydroxide solution is added drop by drop to neutralize the excess of acid till a faint precipitate is obtained. About 2 ml of dil. Acetic acid is added to get a clear solution. Nearly 2g of KI is added and mixed well, diluted with water and then the brown coloured solution liberates iodine which is titrated against thiosulphate taken in the burette. When colour of the solution becomes pale yellow, about 2 ml of freshly prepared starch solution is added and the titration is continued till the end point is reached. The end point is the appearance of creamy white precipitate.

A spoon full of ammonium thiocyanate is added and mixed well. If the blue colour is developed, the titration is continued till the blue colour is discharge. If there is no blue colour, the burette reading is noted. The titration is repeated for concordant values. From the titre values the strength and amount of given unknown solution is calculated.

Result:

The amount of Copper present in the whole of the given solution =…….g

ESTIMATION OF POTASSIUM DICHROMATE

CALCULATION:

Weight of the $K_2Cr_2O_7$ present in the whole of the given solution = W g

Normality of standard $K_2Cr_2O_7$ $= \frac{W \times 4}{Eq.mass\ of\ K_2Cr_2O_7}$

$= \frac{W \times 4}{49}$

N_1 = ------- N

TITRATION-I: Standardization of Sodium Thiosulphate

(Std. $K_2Cr_2O_7$ solution Vs Sodium thio solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**x**
2.					
3.					

Volume of Std. $K_2Cr_2O_7$ solution V_1 = 20ml
Normality of Std. $K_2Cr_2O_7$ solution N_1 = 0.1 N
Volume of Sodium thio solution V_2 = x V
Normality of Sodium thio solution N_2 = ? M

$$V_1N_1 = V_2N_2$$

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

Normality of Sodium thio solution N_2 = y N

TITRATION-II: Estimation of $K_2Cr_2O_7$

(Std. Sodium thio sulphate solution Vs Unknown solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**z**
2.					
3.					

ESTIMATION OF POTASSIUM DICHROMATE

AIM

To estimate the amount of $K_2Cr_2O_7$ present in the whole of the given solution being supplied with $K_2Cr_2O_7$ crystals of AR quantity and an approximately decinormal solution of sodium thiosulphate.

PRINCIPLE

In the presence of HCL acid, $K_2Cr_2O_7$ liberates iodine from a solution of KI as per equation given below.

$$\mathbf{K_2Cr_2O_7 + 14\ HCL \longrightarrow 2HCL + 2CrCI_3 + 7H_2O + 3Cl_2}$$

$$\mathbf{6KI + 3Cl_2 \longrightarrow 6KCL + 3I_2}$$

One molecular mass of $K_2Cr_2O_7$ liberates six equivalent of iodine from KI solution.

Ionically,

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

$$\text{Equivalent mass of } K_2Cr_2O_7 = \frac{Molecular\ mass}{6}$$

$$= \frac{294}{6} = 49$$

The liberated I_2 is titrated against sodium thio sulphate using starch as indicator. The reaction may be written as

$$I_2 + 2\,Na_2S_2O_3 \longrightarrow \underset{\text{Sodium tetrathionate}}{Na_2S_4O_6} + 2\,NaI$$

PROCEDURE

Preparation of standard N/10 $K_2Cr_2O_7$ Solution

Accurately about 0.49 g of $K_2Cr_2O_7$ of AR quality is weighed in a chemical balance. It is transferred into 100 ml flask and the solution is made up to the mark. The solution is shaken well to get a homogenous solution. Solution of this known strength is taken in the burette after thoroughly rinsing with it.

Titration-I

Standardization of Sodium Thio Sulphate solution

(Std. $K_2Cr_2O_7$ solution Vs Sodium thiosulphate solution)

The burette is filled with the given sodium thiosulphate solution. Exactly 20 ml of standard $K_2Cr_2O_7$ solution is pipetted out into a clean conical flask. 5 ml of Con. HCl is added (20 ml

Volume of Sodium thio solution V_1 = z V
Normality of Sodium thio solution N_1 = y N
Volume of Given $K_2Cr_2O_7$ solution V_2 = 20ml
Normality of Given $K_2Cr_2O_7$ solution N_2 = ?

$$V_1N_1 = V_2N_2$$
$$N_2 = \frac{V_1N_1}{V_2}$$

Normality of Given $K_2Cr_2O_7$ N_2 = a N

The amount of Zn present in the whole of the given solution

$$= \frac{Normality\ (a) \times Equivalent\ mass\ of\ K_2Cr_2O_7}{10}$$

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

= ---------- g

of dil. HCl) followed by 10 ml of 10% KI solution. The brown coloured solution liberates iodine which is titrated against thiosulphate taken in the burette. When the colour of the solution becomes pale yellow, about 2 ml of freshly prepared starch solution is added and the titration is continued till the end point is reached. The end point is the disappearance of blue colour and appearance of green colour. The titration is repeated for concordant values. From the titre values the strength of thiosulphate is calculated.

Titration – II

Estimation of Potssium Dichromate

(Std. Sodium thio sulphate Vs Unknown Solution)

The given $K_2Cr_2O_7$ solution is made up to 100 ml in a standard flask. Exactly 20 ml of the made up solution is pipette out into a clean conical flask. 5 ml of 5 ml of Con. HCl is added (20 ml of dil. HCl) followed by 10 ml of 10% KI solution. The brown coloured solution liberates iodine which is titrated against thiosulphate taken in the burette. When colour of the solution becomes pale yellow, about 2 ml of freshly prepared starch solution is added and the titration is continued till the end point is reached. The end point is the disappearance of blue colour and appearance of green colour. The titration is repeated for concordant values. From the titre values the strength and amount of given $K_2Cr_2O_7$ solution is calculated.

Result:

The amount of $K_2Cr_2O_7$ present in the whole of the given solution =…….g

ESTIMATION OF AVAILABLE CHLORINE IN THE BLEACHING POWDER

CALCULATION:

Weight of the $K_2Cr_2O_7$ present in the whole of the given solution = W g

Normality of standard $K_2Cr_2O_7$ $= \frac{W \times 4}{Eq.mass\ of\ K_2Cr_2O_7}$

$= \frac{W \times 4}{49}$

N_1 = ------- N

TITRATION-I: Standardization of Sodium Thiosulphate

(Std. $K_2Cr_2O_7$ solution Vs Sodium thio solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**x**
2.					
3.					

Volume of Std. $K_2Cr_2O_7$ solution V_1 = 20ml
Normality of Std. $K_2Cr_2O_7$ solution N_1 = 0.1 N
Volume of Sodium thio solution V_2 = x V
Normality of Sodium thio solution N_2 = ? M

$$V_1N_1 = V_2N_2$$

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

Normality of Sodium thio solution N_2 = y N

TITRATION-II: Estimation of Available Chlorine

(Std. Sodium thio sulphate solution Vs Unknown Bleaching Powder solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**z**
2.					
3.					

ESTIMATION OF AVAILABLE CHLORINE IN THE BLEACHING POWDER

AIM

To estimate the amount of available chlorine present in the given sample of bleaching powder being supplied with $K_2Cr_2O_7$ crystals of AR quantity and an approximately decinormal solution of sodium thiosulphate are provided.

PRINCIPLE

By available chlorine means that a sample of bleaching powder liberates chlorine by the action of dilute acids on it. Its amount is expressed as percentage of chlorine available in the sample. Commercially, obtainable bleaching powder contains 36% - 38% of available chlorine.

Bleaching powder essentially consists of Calcium hypochloride $Ca(OCl_2)$. When a solution of bleaching powder is treated with an excess of a solution of potassium iodide and strongly acidified with acetic acid, the following reaction takes place,

$$OCl^- + 2I^- + 2H^+ \longleftrightarrow Cl^- + I_2\uparrow + H_2O$$

The liberated iodine is treated with standard sodium thiosulphate solution using starch solution as the indicator. From the amount of liberated I_2, the equivalent of Cl_2 and hence the available chlorine in the sample of bleaching powder is calculated.

PROCEDURE

Preparation of standard N/10 $K_2Cr_2O_7$ Solution

Accurately about 0.49 g of $K_2Cr_2O_7$ of AR quality is weighed in a chemical balance. It is transferred into 100 ml flask and the solution is made up to the mark. The solution is shaken well to get a homogenous solution. Solution of this known strength is taken in the burette after thoroughly rinsing with it.

Titration-I

Standardization of Sodium Thio Sulphate solution

(Std. $K_2Cr_2O_7$ solution Vs Sodium thiosulphate solution)

The burette is filled with the given sodium thiosulphate solution. Exactly 20 ml of standard $K_2Cr_2O_7$ solution is pipetted out into a clean conical flask. About 20 ml of 2N dil. HCl (5 ml of Con. HCl) is added followed by 10 ml of 10% KI solution. The dark brown coloured solution liberates iodine which is titrated against thiosulphate taken in the burette. When the colour of the solution becomes pale yellow, about 1 ml of freshly prepared starch solution is added and the titration is continued till the end point is reached.

Volume of Sodium thio solution V_1 = z V
Normality of Sodium thio solution N_1 = y N
Volume of Bleaching powder solution V_2 = 20ml
Normality of Bleaching powder solution N_2 = ?

$$V_1N_1 = V_2N_2$$

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

Normality of Bleaching powder Solution N_2 = a N

The amount of Cl present in the whole of the given sample of bleaching powder solution $= \frac{Normality\ (a) \times Equivalent\ mass\ of\ Cl}{10}$

$$= \frac{a \times 35.46}{10}$$

= ---------- g

% of Available Chlorine in the given sample of bleaching powder = $\frac{\mathbf{Amount\ of\ Liberated\ Cl}}{\mathbf{Weight\ of\ the\ Sample}} \times \mathbf{100}$

$$= \frac{}{\mathbf{5}} \times \mathbf{100}$$

= ----------- %

The end point is the disappearance of starch blue colour leaving behind bright green colour. The titration is repeated for concordant values. From the titre values the strength of thiosulphate is calculated.

<u>Titration – II</u>

Estimation of Available Chlorine
(Std. Sodium thio sulphate Vs Unknown Solution)

The given $K_2Cr_2O_7$ solution is made up to 100 ml in a standard flask. Exactly 20 ml of the made up solution is pipette out into a clean conical flask. 5 ml of 5 ml of Con. HCl is added (20 ml of dil. HCl) followed by 10 ml of 10% KI solution. The brown coloured solution liberates iodine which is titrated against thiosulphate taken in the burette. When colour of the solution becomes pale yellow, about 2 ml of freshly prepared starch solution is added and the titration is continued till the end point is reached. The end point is the disappearance of blue colour and appearance of green colour. The titration is repeated for concordant values. From the titre values the strength and amount of available Chlorine in the given sample solution is calculated. From that, the percentage of available chlorine in the sample bleaching powder solution is calculated.

Result:

The % of Chlorine present in the whole of the given solution =…….g

ESTIMATION OF ZINC

CALCULATION:

Mass of analar zinc sulphate crystals taken = W g

Molecular mass of $ZnSO_4.7HO$ = 287.36

Molarity of magnesium sulphate solution = 0.1 M

TITRATION-I: Standardization of EDTA

(Std. $ZnSO_4$ solution Vs EDTA solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**x**
2.					
3.					

Volume of Zinc sulphate solution V_1 = 20ml
Molarity of Zinc sulphate solution M_1 = 0.1 M
Volume of EDTA V_2 = x V
Molarity of EDTA M_2 = ? M

$$V_1M_1 = V_2M_2$$

$$M_2 = \frac{V_1M_1}{V_2}$$

Molarity of EDTA Solution M_2 = y M

TITRATION-II: Estimation of Zinc

(Std. EDTA solution Vs Unknown solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**z**
2.					
3.					

Volume of EDTA V_1 = z ml

ESTIMATION OF ZINC

AIM

Estimation the mass Of Zinc In The Whole Given Solution Of Zinc Sulphate, Being Supplied With Approximately 0.025 Molar EDTA And Analar Zinc Sulphate Crystals.

PRINCIPLE AND OUTLINE:

Zinc ions form complexes with EDTA which can be represented the equation

$$Zn^{2+} + H_2Y^{2-} \rightarrow ZnY^{2-} + 2H^+$$

The end point is detected by using the metal sensitive indicator namely eriochrome black Twhich has a blue colour in the p range between 7 to 11. This indicator reacts with Zn ions to produce Zn-erichrome T indicator complex which has a wine red colour.

A standard solution of zinc sulphate is prepared and 20ml of the solution is pipetted out into a conical flask .about 3-4 drops of the eriochrome black Tindicator is added, which gives the solution a with red colour .it is then titrated against EDTA solution. The free zinc ion react with EDTA to give Zn-EDTA complex ,the addition of next drop of the EDTA causes the de composition of Zinc-indicator complex and the free indicator liberated gives a blue cololur at the end point. Using the standardised EDTA , the strength of the give zinc salt solution and hence the mass of zinc ions in the whole of the solution can be calculated

PROCEDURE

(a) ***Preparation of 0.025 molar standard zinc sulphate solution***

Molecular mass of $Zn\,SO_4 . 7H_2O$ being 287.36 about 0.72g of A.R zinc sulphate is accurately weighed out into 100 ml standard measuring flask , dissolved in water and made up to the mark.

(b) ***Titration – I: Standardization of EDTA***

20 ml of the standard magnesium sulphate solution is pipetted out into a conical flask and diluted to 100 ml with distilled water. 2ml of buffer solution (NH_4Cl-NH_4OH) is added from a burette. 3-4 drops of Eriocrome black T indicator is added and titrated.

Molarity of EDTA M_1 = y M

Volume of given Zinc sulphate solution V_2 = 20 ml

Molarity of the given Zinc sulphate solution M_2 = ? M

$$V_1M_1 = V_2M_2$$

$$M_2 = \frac{V_1M_1}{V_2}$$

Molarity of Given Zinc Sulphate Solution M_2 = a M

The amount of Zn present in the whole of the given solution

$$= \frac{Strength\ of\ Zinc\ sulphate\ solution \times Equivalent\ mass\ of\ Zn}{10}$$

$$= \frac{a \times 63.57}{10}$$

= ---------- g

against EDTA until the colour changer from red to pure blue. Titration are repeated till concordant value are obtained.

(c) ***Titration – II: Estimation of Zinc.***

The given zinc sulphate solution is pipetted out a conical flask and diluted to 100 ml in a standard measuring flask. 20 ml of the made up solution is pipetted out into a conical flask. It is diluted to 100 ml with distilled water. 2 ml of the buffer solution are added from a burette. 4 drops of Eriocrome black T indicator are added and titrated against the standardized EDTA until the colour changes from red to pure blue. Titration are repeated till concordant results are obtained are obtained.

From the titre values, strength of zinc sulphate solution and the amount of magnesium in the whole of the given solution is calculated.

Result:

Mass of Zinc in the whole of the given solution =…….g

ESTIMATION OF MEGNESIUM

CALCULATION:

Mass of analar magnesium sulphate crystals taken = W g

Molecular mass of MgSO.7HO =246.312

Molarity of magnesium sulphate solution = 0.1 M

TITRATION-I: Standardization of EDTA

(Std. $MgSO_4$ solution Vs EDTA solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**x**
2.					
3.					

Volume of magnesium sulphate solution V_1 = 20ml
Molarity of magnesium sulphate solution M_1 = 0.1 M
Volume of EDTA V_2 = x V
Molarity of EDTA M_2 = ? M

$$V_1M_1 = V_2M_2$$

$$M_2 = \frac{V_1M_1}{V_2}$$

Molarity of EDTA Solution M_2 = y M

TITRATION-II: Estimation of Magnesium

(Std. EDTA solution Vs Unknown solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**z**
2.					
3.					

Volume of EDTA V_1 = z ml

ESTIMATION OF MEGNESIUM

AIM

Estimation The Amount Of Magnesium In The Whole Of The Given Solution Of Magnesium Sulphate , Supplied With Approximately 0.025 Mole EDTA And Analar Magnesium Sulphate Crystals.

PRINCIPLE AND OUTLINE

The estimation is based on the reaction Mg^{2+} +H_2Y^{2-} + $2H^+$ · A standard solution of magnesium sulphate is prepared and the given EDTA is standardised using erio-chrome black T as the indicator. in presence of metal ions the indicator given a wine red colour in the p^H range of 7-11. At the end point, all the metal ions are converted to metal ions metal – EDTA complex and the free indicator is liberated which has blue colour. using the standardised EDTA, the given magnesium sulphate solution is estimation . Eriochrome black T is used as the indicator.

PROCEDURE

(a) Preparation of 0.025 molar standard magnesium sulphate solution

Molecular mass of $MgSO_4.7H_2O$ being 246.31 about 0.615g of A.R magnesium sulphate is accurately weighed out into 100 ml standard measuring flask , dissolved in water and made up to the mark.

(b) Titration – I: Standardization of EDTA

20 ml of the standard magnesium sulphate solution is pipetted out into a conical flask and diluted to 100 ml with distilled water . 2ml of buffer solution (pH=10) are added from a burette. 4 drops of Eriochrome black T indicator are added . the solution is warmed to 400C and titrated against EDTA until the colour changer from red to pure blue . Titration is repeated till concordant value is obtained.

(c) Titration – II: Estimation of magnesium.

The given magnesium sulphate solution is pipetted out a conical flask and diluted to 100 ml in a standard measuring flask. 20 ml of the made up solution is pipetted out into a conical flask. It is diluted to 100 ml with distilled water. 2 ml of the buffer solution is added from a burette. 4 drops of Eriochrome black T indicator is added. The solution is warmed to 400ºC and titrated against the standardized EDTA until the colour changes from red to pure blue. Titration are repeated till concordant results are obtained are obtained.

Molarity of EDTA M_1 = y M
Volume of given magnesium sulphate solution V_2 = 20 ml
Molarity of the given magnesium sulphate solution M_2 = ? M

$$V_1M_1 = V_2M_2$$
$$M_2 = \frac{V_1M_1}{V_2}$$

Molarity of Given Magnesium SulphateSolution M_2 = a M

The amount of Mg present in the whole of the given solution

$$= \frac{Strength\ of\ magnesium\ sulphate\ solution \times Equivalent\ mass\ of\ Mg}{10}$$

$$= \frac{a \times 24.01}{10}$$

= ---------- g

From the titre values, strength of magnesium sulphate solution and the amount of magnesium in the whole of the given solution is calculated.

Result:

Mass of magnesium in the whole of the given solution =…….g

ESTIMATION OF CALCIUM – DIRECT METHOD

Calculation:

Weight of Oxalic acid crystal in 250 ml = W g

Normality of Oxalic acid solution (N_1) = W x 4 / 63

=………

Titration I: Standardization of $KMn0_4$ solution

(Std. Oxalic acid solution Vs $KMnO_4$ Solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**X**
2.					
3.					

Volume of Oxalic acid solution (V_1) = 20 ml

Volume of Link $KMn0_4$ solution (V_2) = X

Normality of Link $KMn0_4$ solution (N_2) = ?

$$V_1 \text{ x } N_1 = V_2 \text{ x } N_2$$

$$N_2 = V_1 \text{ x } N_1 / V_2$$

$$= 20 \text{ x } N_{H2C2O4} / x_1 \text{ ml}$$

$$N_{KMnO4} = \text{-----------}$$

ESTIMATION OF CALCIUM – DIRECT METHOD

AIM:

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

PRINCIPLE:

First, potassium permanganate is standardized using a standard solution of oxalic acid. In the next step, calcium is precipitated as calcium oxalate in ammoniacal medium, using a 3% solution of Ammonium oxalate. The precipitate is filtered and dissolved in dilute sulphuric acid and the liberated oxalic acid is titrated against standard potassium permanganate solution.

$$CaCl_2 + (NH_4)_2C_20_4 \longrightarrow C_aC_20_4 + 2NH_4CL$$

$$CaC_20_4 + H_2S0_4 \longrightarrow C_aS0_4 + H_2C_20_4$$

(0) From potassium
Permanganate

Equivalent mass of Calcium = Atomic mass

= 20.04

PROCEDURE:

Titration I : Standardisation of KMn04 Solution:

About 1.5 g of oxalic acid crystal are accurately weighed and transferred into a funnel placed over a 250 ml standard flask. The crystals are then carefilly washed down into the flask by a jct of distilled water. The crystals are dissolved and the solution made up to the mark and well shaken.

Exactly 20 ml of the standard oxalic acid solution is pipetted out into a conical flask. About 20 ml 0f dilute H_2S0_4 is added and the mixture heated to 60ºC to 80ºC on wire gauze. The hot solution is then titrated with $KMn0_4$ solution taken in the burette. The first few drops of $KMn0_4$ take some time for decolorisation. But as the reaction proceeds the manganous sulphate formed acts as a catalyst and the reaction proceeded faster. The end point is indicated by the appearance of a pale pink colour in the solution. The titration is repeated to get concordant values.

Titration II: Estimation of Calcium

The whole of the given calcium solution is made uniform. By means of a rinsed and cleaned pipette, exactly 20 ml of the made up solution is pipette out into a cleansed pipette exactly 20 ml of made up solution is pipette out into a clean 250 ml beaker. A glass rod is placed. A drop of methyl orange and then ammonium hydroxide are added drop by drop, till

Titration II : Estimation of Ca^{+2}

(Std. $KMnO_4$ Solution Vs Given Solution)

S.No.	**Volume of Pipette Solution (ml)**	**Burette Reading (ml)**		**Volume of Burette Solution (ml)**	**Concordant Value (ml)**
		Initial	**Final**		
1.					**Y_1**
2.					
3.					

Volume of Ca^{+2} solution (V_1) = 20 ml

Normality of Ca^{+2} solution (N_1) = ?

Volume of KMn04 solution (V_2) = y_1 ml

Normality of KMn04 solution (N_2) = N_{KMnO4}

$$V_1 \text{ x } N_1 = V_2 \text{ x } N_2$$

$$N_1 = V_2 \text{ x } N_2 / V_1$$

$$= y_1 \text{ ml x } N_{KMnO4} / 20$$

$$NCa^{+2} = y_1 \text{ ml x } N_{KMnO4} / 20$$

= -----------

Amount of calcium present in the

whole of the given solution = N_{Ca}+2 solution x Eq. mass of Ca^{+2} / 10

= N_{Ca}+2 solution x 20.04 / 10

= -----------g.

the solution becomes yellow. 1 ml of concentrated hydrochloric acid is added. Diluted with distilled water and heated to boiling. About 20 ml of the 3%

Solution of ammonium oxalate solution is taken in a 100 ml breaker and heated to about 60ºC to 70ºC. The hot ammonium oxalate solution is added drop by drop to the calcium solution while stirring vigorously. Ammonium hydroxide is then added till the mixture is distinctly ammoniacal by smell. Heated over a low flame for about 15 minutes to allow the precipitate is settled down.

III Filtration

A whatmann No. 40 filter paper is placed in such a way that there is no air space. The contents of the beaker are tested for completion of precipitation by adding a drop of ammonium oxalate along the sides of the beaker. If there is no turbidity, the precipitation is complete. [If precipitation is incomplete some more ammonium oxalate and ammonium hydroxide are added.] Using a glass rod, the clear supernatant liquid is transferred into the filter paper and the filtrate is collected little ammonia and the clear solution is transferred into the filter paper. The process of washing is repeated until the filtrate gives no reaction for ammonium oxalate or chloride. The filtrate is rejected.

The precipitate on the filter paper is dissolved by adding hot dilute sulphuric acid and the filter paper is gently folded allowing the acid to come into contact with and the precipitate and the washing are collected in a beaker. There should be no precipitate left on the filter paper or funnel. If necessary, the filter paper is transferred into the beaker and some more dilute sulphuric acid is added. The contents are heated to about 70^0C and titrated against standard potassium are tabulated and the strength and hence the amount of calcium in the whole of the given solution is calculated.

Result:

Amount of calcium in the whole of the given solution = …… g.

PREPARATION OF CUPROUS CHLORIDE, Cu_2Cl_2

AIM:

To prepare a pure sample of crystals of cuprous chloride, Cu_2Cl_2.

PRINCIPLE:

While +2 oxidation state of copper is a stable state, +1 oxidation state is not that much stable.Cuprous chloride(copper in the +1 oxidation state), is prepared by reducing copper(II) with sulphur dioxide or sulphate ions in the presence of chloride ions. The copper(I) ions once formed, react with chloride ions to form cuprous chloride.

$CuCl_2$ SO_2 Cu_2Cl_2

Cupric chloride → Cuprous chloride

$2Cu^+ + 2Cl^- \rightarrow Cu_2Cl_2$

CHEMICALS REQUIRED:

(i) Sodium Sulphate = 2.5g

(ii)Cupric chloride = 3.25g

(iii)Sulphurous acid solution(H_2SO_3) =10g.Na_2SO_3 in one litre water

And add 10 ml dilute HCl

(iv)Glacial acetic acid =2.5 ml

(v) Ether = 7.5ml

PROCEDURE :

2.5 g of sodium sulphate is dissolved in 25 ml of water in a 100 ml beaker to get sodium sulphate solution. Sulphorous acid solution is prepared by dissolving 10g of sodium sulphate in one litre of water containing 10 ml of dilute HCl.

3.25g of cupric chloride is dissolved in minimum quantity of water and then sulphate solution is slowly added with constant stirring. The cuprous chloride formed is in suspension and treated with 200ml of sulphorous acid solution so that cuprous chloride precipitate settles down. The supernatant is filtered off. The precipitated cuprous chloride is filtered at the pump. It is first washed with sulphorous acid followed by glacial acetic acid and ether. The cuprous chloride is dried at the pump and transferred into a test tube which is air tightly corked.

RESULT:

The weight of cuprous chloride (Cu_2Cl_2) obtained =…g

PREPARATION OF MANGANOUS SULPHATE

AIM

To Prepare pure crystals of manganous sulphate ($MnSO_4$)

PRINCIPLE

Pure crystals of manganous sulphate are prepared by heating the commercial black oxide of manganese (MnO_2) with sulphuric acid

$$\boldsymbol{MnO_2 + H_2SO_4 \rightarrow 2MnSO_4 + 2H_2O + O_2}$$

CHEMICALS REQUIRED

Manganese dioxide (MnO_2) = 4g

Conc. H_2SO_4 = 5 ml

PROCEDURE

About 4g of manganese dioxide is placed in a 100 ml clean beaker and 5 ml of conc. H_2SO_4 is added to it. The beaker is heated on a wiregauze until the black colour of the manganese dioxide fades away. Now the mixture is washed with 100 ml of water and the clear solution is filtered. The filtrate is allowed to cool slowly when rose pink crystals of the $MnSO_4 . 5H_2O$ are obtained. The crystals are filtered at the pump and dried.

RESULT:

The yield of $MnSO_4 . 5H_2O$ = --------- g.

PREPARATION OF POTASH ALUM,$K_2SO_4.Al_2(SO_4)_3.24H_2O$

PRINCIPLE:

Potash alum is a double salt of potassium sulphate and aluminium sulphate. Hence it can be prepared by mixing K_2SO_4 and $Al_2(SO_4)_3.18H_2O$ in the ratio of their molecular masses. Their concentrated solutions are prepared, mixed together and the resulting solution is heated to the crystallisation point when white crystal of potash alum are obtained.

$$K_2SO_4+Al_2(SO_4)_3.18H_2O+6H_2O\rightarrow K_2SO_4.Al_2(SO_4)_3.24H_2O$$

REQUIREMENTS:

1. Potassium sulphate = 3g
2. Aluminium sulphate = 12g
3. Dilute H_2SO_4 = 1ml

PROCEDURE:

3g of potassium sulphate is dissolved in 25ml of distilled water taken in 250ml beaker. 12g of aluminium sulphate is taken in another beaker and is dissolved in 25ml of distilled water and a few drops of dil. H_2SO_4 to prevent to the hydrolysis of aluminium sulphate phate. The two solutions are mixed and transferred into a china dish. The china disk is gently heated on a water bath and concentred to the crystallisation point the solution is allowed to cool, when white octahedral crystals of potash alum are obtained. The crystals are separated by decantation and washed with small amount of ice cold water. The crystals are dried between folds of filter paper and yield noted.

RESULT:

Yield of potash alum =………g

PREPARATION OF CHROME ALUM,$K_2SO_4.Cr_2(SO_4)_3.24H_2O$

PRINCIPLE:

Potash alum is a double salt of potassium sulphate and aluminium sulphate. Hence it can be prepared by mixing K_2SO_4 and $Al_2(SO_4)_3.18H_2O$ in the ratio of their molecular masses. Their concentrated solutions are prepared, mixed together and the resulting solution is heated to the crystallisation point when white crystal of potash alum are obtained.

$$K_2SO_4 + K_2Cr_2O_7 + 6H_2O \rightarrow K_2SO_4.Cr_2(SO_4)_3.24H_2O$$

REQUIREMENTS:

1. Potassium dichromate = 5g

2. Con. H_2SO_4 = 4 ml

3. Alcohol = 5 ml

Chrome alum [$K_2SO_4.Cr_2(SO_4)_3.24H_2O$]. Take about 5 gm of potassium dichromate and add to it 25-30 ml of water. Add slowly and carefully about 4 ml of conc. H_2SO_4 addition) on a water bath maintaining the temperature to near about 50°c. Add 5ml of alcohol and reduce the temperature by adding ice to water bath. Allow the content to stand for 5-6 hours. The large violet crystals of chrome alum are obtained. Yield is about 10gm.

RESULT:

Yield of Chrome alum =………g