

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

**DEPARTMENT OF CHEMISTRY**  
**SYLLABUS**

<b>17CHU212</b>	<b>S-BLOCK AND P- BLOCK ELEMENTS - PRACTICAL</b>	<b>4H 2C</b>
<b>Instruction Hours/week: L: 0 T:0 P:4</b>		<b>Marks: Internal: 40 External: 60 Total:100</b>

**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 complexometric titrations using EDTA
3. To carry out the preparations of inorganic metal complexes.

**Methodology**

Iodimetric titrations, Complexometric titrations, Inorganic preparations.

**(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,  $MnPO_4 \cdot H_2O$
- (iii) Aluminium potassium sulphate  $KAl(SO_4)_2 \cdot 12H_2O$  (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.



## KARPAGAM ACADEMY OF HIGHER EDUCATION

Coimbatore-21

## DEPARTMENT OF CHEMISTRY

LECTURE PLANS-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).	<b>T1:135,T2:108</b>
3	Estimation of K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> using sodium thiosulphate solution (Iodometrically).	<b>T1:132,T2:110</b>
4	Estimation of Mg <sup>2+</sup>	<b>T1:152,T2:121</b>
5	Estimation of Zn <sup>2+</sup>	<b>T1:150</b>
6	Estimation of Ca <sup>2+</sup> by substitution method	<b>T1:152,T2:120</b>
7	Viva-voce	
8	Estimation of available Chlorine in bleaching powder	<b>T1:139</b>
9	Preparation of Cuprous Chloride, Cu <sub>2</sub> Cl <sub>2</sub>	<b>T1:78,T2:138</b>
10	Preparation of Manganese (III) phosphate, MnPO <sub>4</sub> .H <sub>2</sub> O	<b>W1</b>
11	Preparation of Aluminium potassium sulphate K <sub>2</sub> SO <sub>4</sub> Al(SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O (Potash alum)	<b>T2:140</b>
12	Preparation of Chrome alum.	<b>T2:138</b>
13	Viva-voce	
14	Revision	
15	Model Exam	

**Support Materials:****Text books :**

**T1:** V.Venkateswaran, R.Veerasingam 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

S.No	Title of the Experiment	Page No.
1.	<u>IODOMETRY</u> Estimation of Copper	3
2.	Estimation of Potassium Di Chromate ( $K_2Cr_2O_7$ )	7
3.	Estimation of available chlorine in Bleaching Powder	11
4.	<u>COMPLEXOMETRY</u> Estimation of Zn	15
5.	Estimation of Mg	19
6.	Estimation of Ca	23
7.	<u>PREPARATION</u> Preparation of Cuprous Chloride ( $CuCl_2$ )	27
8.	Preparation of Manganese Sulphate ( $MnSO_4$ )	28
9.	Preparation of Potash Alum	29
10.	Preparation of Chrome Alum	30

KAHE

## ESTIMATION OF COPPER

### **CALCULATION:**

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

$$\begin{aligned}\text{Normality of standard } K_2Cr_2O_7 &= \frac{W \times 4}{\text{Eq.mass of } K_2Cr_2O_7} \\ &= \frac{W \times 4}{49}\end{aligned}$$

$$N_1 = \text{-----} N$$

### **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  $V_1 = 20\text{ml}$

Normality of Std.  $K_2Cr_2O_7$  solution  $N_1 = 0.1\text{ N}$

Volume of Sodium thio solution  $V_2 = x\text{ V}$

Normality of Sodium thio solution  $N_2 = ?\text{ M}$

$$V_1N_1 = V_2N_2$$

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

Normality of Sodium thio solution  $N_2 = y\text{ N}$

### **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.					

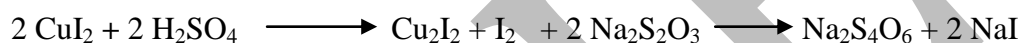
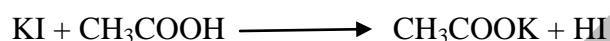
## **ESTIMATION OF COPPER**

### **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



Equivalent mass of Copper = Molecular mass of copper = 63.54

Equivalent mass of Copper Sulphate ( $CuSO_4 \cdot 5H_2O$ ) = Molecular mass of Copper Sulphate ( $CuSO_4 \cdot 5 H_2O$ ) = 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	$N_2$	=	?	

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$

Normality of Given Unknown Solution  $N_2$  = a N

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

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

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

$$= \text{----- g}$$

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

$$\begin{aligned}\text{Normality of standard } K_2Cr_2O_7 &= \frac{W \times 4}{\text{Eq.mass of } K_2Cr_2O_7} \\ &= \frac{W \times 4}{49} \\ N_1 &= \text{-----} N\end{aligned}$$

### **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.					

$$\begin{aligned}\text{Volume of Std. } K_2Cr_2O_7 \text{ solution } V_1 &= 20\text{ml} \\ \text{Normality of Std. } K_2Cr_2O_7 \text{ solution } N_1 &= 0.1 \text{ N} \\ \text{Volume of Sodium thio solution } V_2 &= x \text{ V} \\ \text{Normality of Sodium thio solution } N_2 &= ? \text{ M} \\ V_1 N_1 &= V_2 N_2 \\ N_2 &= \frac{V_1 N_1}{V_2}\end{aligned}$$

$$\text{Normality of Sodium thio solution } N_2 = y \text{ N}$$

### **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.					

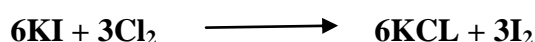
## ESTIMATION OF POTASSIUM DICHROMATE

### 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.

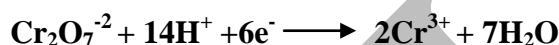
### PRINCIPLE

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



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

Ionically,



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

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

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



### 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	$N_2$	=	?	

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$

Normality of Given  $K_2Cr_2O_7$   $N_2$  = a N

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

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

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

$$= \text{----- g}$$

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 Potassium 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

$$\begin{aligned}\text{Normality of standard } K_2Cr_2O_7 &= \frac{W \times 4}{\text{Eq.mass of } K_2Cr_2O_7} \\ &= \frac{W \times 4}{49} \\ N_1 &= \text{-----} N\end{aligned}$$

### **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.					

$$\begin{aligned}\text{Volume of Std. } K_2Cr_2O_7 \text{ solution } V_1 &= 20\text{ml} \\ \text{Normality of Std. } K_2Cr_2O_7 \text{ solution } N_1 &= 0.1 \text{ N} \\ \text{Volume of Sodium thio solution } V_2 &= x \text{ V} \\ \text{Normality of Sodium thio solution } N_2 &= ? \text{ M} \\ V_1 N_1 &= V_2 N_2 \\ N_2 &= \frac{V_1 N_1}{V_2}\end{aligned}$$

$$\text{Normality of Sodium thio solution } N_2 = y \text{ N}$$

### **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**

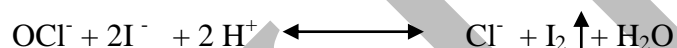
### **AIM**

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,



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.

$$\begin{array}{llll}
 \text{Volume of Sodium thio solution} & V_1 & = & z \quad V \\
 \text{Normality of Sodium thio solution} & N_1 & = & y \quad N \\
 \text{Volume of Bleaching powder solution} & V_2 & = & 20\text{ml} \\
 \text{Normality of Bleaching powder solution} & N_2 & = & ?
 \end{array}$$

$$\begin{aligned}
 V_1 N_1 &= V_2 N_2 \\
 N_2 &= \frac{V_1 N_1}{V_2}
 \end{aligned}$$

$$\text{Normality of Bleaching powder Solution } N_2 = a \quad N$$

The amount of Cl present in the whole of the given sample of bleaching powder solution

$$\begin{aligned}
 &= \frac{\text{Normality (a)} \times \text{Equivalent mass of Cl}}{10} \\
 &= \frac{a \times 35.46}{10} \\
 &= \text{----- g}
 \end{aligned}$$

% of Available Chlorine in the given sample of bleaching

$$\begin{aligned}
 \text{powder} &= \frac{\text{Amount of Liberated Cl}}{\text{Weight of the Sample}} \times 100 \\
 &= \frac{\quad}{5} \times 100 \\
 &= \text{----- \%}
 \end{aligned}$$

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.

### **Titration – II**

#### **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  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  = 287.36

Molarity of magnesium sulphate solution = 0.1 M

### **TITRATION-I: Standardization of EDTA**

(Std.  $\text{ZnSO}_4$  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 ml

Molarity of EDTA  $M_2$  = ? M

$$V_1 M_1 = V_2 M_2$$

$$M_2 = \frac{V_1 M_1}{V_2}$$

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

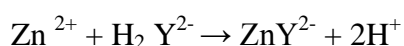
## **ESTIMATION OF ZINC**

### **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



The end point is detected by using the metal sensitive indicator namely eriochrome black T which has a blue colour in the pH range between 7 to 11. This indicator reacts with Zn ions to produce Zn-eriochrome 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 T indicator is added, which gives the solution a wine red colour. It is then titrated against EDTA solution. The free zinc ion reacts with EDTA to give Zn-EDTA complex, the addition of next drop of the EDTA causes the decomposition of Zinc-indicator complex and the free indicator liberated gives a blue colour at the end point. Using the standardised EDTA, the strength of the given 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  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  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 ( $\text{NH}_4\text{Cl}-\text{NH}_4\text{OH}$ ) is added from a burette. 3-4 drops of Eriochrome black T indicator is added and titrated.

Molarity of EDTA  $M_1 = y \text{ M}$   
 Volume of given Zinc sulphate solution  $V_2 = 20 \text{ ml}$   
 Molarity of the given Zinc sulphate solution  $M_2 = ? \text{ M}$

$$V_1 M_1 = V_2 M_2$$

$$M_2 = \frac{V_1 M_1}{V_2}$$

Molarity of Given Zinc Sulphate Solution  $M_2 = a \text{ M}$

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

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

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

$$= \text{----- g}$$

against EDTA until the colour changes 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 Eriochrome 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  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  = 246.312

Molarity of magnesium sulphate solution = 0.1 M

### **TITRATION-I: Standardization of EDTA**

(Std.  $\text{MgSO}_4$  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.					<b>x</b>
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

$$V_1 M_1 = V_2 M_2$$

$$M_2 = \frac{V_1 M_1}{V_2}$$

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.					<b>z</b>
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  $\text{Mg}^{2+} + \text{H}_2\text{Y}^{2-} + 2\text{H}^+ \rightarrow \text{MgY}^{2-} + 2\text{H}^+$ . 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 gives a wine red colour in the  $\text{pH}$  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  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  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 ( $\text{pH}=10$ ) are added from a burette. 4 drops of Eriochrome black T indicator are added. The solution is warmed to  $40^\circ\text{C}$  and titrated against EDTA until the colour changes 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  $40^\circ\text{C}$  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  $M_1 = y \text{ M}$   
 Volume of given magnesium sulphate solution  $V_2 = 20 \text{ ml}$   
 Molarity of the given magnesium sulphate solution  $M_2 = ? \text{ M}$

$$V_1 M_1 = V_2 M_2$$

$$M_2 = \frac{V_1 M_1}{V_2}$$

Molarity of Given Magnesium Sulphate Solution  $M_2 = a \text{ M}$

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

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

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

$$= \text{----- g}$$

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

KAHE

**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 (  $N_1$  ) =  $W \times 4 / 63$

=.....

#### Titration I: Standardization of $KMnO_4$ 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  $KMnO_4$  solution (  $V_2$  ) = X

Normality of Link  $KMnO_4$  solution (  $N_2$  ) = ?

$$V_1 \times N_1 = V_2 \times N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

$$= 20 \times N_{H_2C_2O_4} / x_1 \text{ ml}$$

$$N_{KMnO_4} = \text{-----}$$

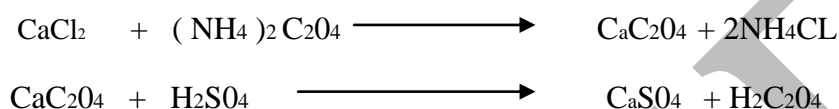
## ESTIMATION OF CALCIUM – DIRECT METHOD

### 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.



(0) From potassium  
Permanganate

Equivalent mass of Calcium = Atomic mass  
= 20.04

### PROCEDURE:

#### Titration I : Standardisation of $\text{KMnO}_4$ 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 carefully washed down into the flask by a jet 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 of dilute  $\text{H}_2\text{SO}_4$  is added and the mixture heated to  $60^\circ\text{C}$  to  $80^\circ\text{C}$  on wire gauze. The hot solution is then titrated with  $\text{KMnO}_4$  solution taken in the burette. The first few drops of  $\text{KMnO}_4$  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  $\text{Ca}^{+2}$**

**(Std.  $\text{KMnO}_4$  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 <sub>1</sub>
2.					
3.					

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

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

Volume of  $\text{KMnO}_4$  solution ( $V_2$ ) =  $y_1$  ml

Normality of  $\text{KMnO}_4$  solution ( $N_2$ ) =  $N_{\text{KMnO}_4}$

$$V_1 \times N_1 = V_2 \times N_2$$

$$N_1 = V_2 \times N_2 / V_1$$

$$= y_1 \text{ ml} \times N_{\text{KMnO}_4} / 20$$

$$N_{\text{Ca}^{+2}} = y_1 \text{ ml} \times N_{\text{KMnO}_4} / 20$$

$$= \text{-----}$$

Amount of calcium present in the

whole of the given solution =  $N_{\text{Ca}^{+2}} \text{ solution} \times \text{Eq. mass of } \text{Ca}^{+2} / 10$

$$= N_{\text{Ca}^{+2}} \text{ solution} \times 20.04 / 10$$

$$= \text{-----g.}$$

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 60°C to 70°C. 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°C 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.

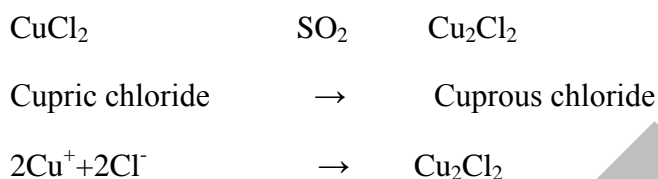
## PREPARATION OF CUPROUS CHLORIDE, $\text{Cu}_2\text{Cl}_2$

### AIM:

To prepare a pure sample of crystals of cuprous chloride,  $\text{Cu}_2\text{Cl}_2$ .

### 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.



### CHEMICALS REQUIRED:

- (i) Sodium Sulphate = 2.5g
- (ii) Cupric chloride = 3.25g
- (iii) Sulphurous acid solution ( $\text{H}_2\text{SO}_3$ ) = 10g.  $\text{Na}_2\text{SO}_3$  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 ( $\text{Cu}_2\text{Cl}_2$ ) obtained = ...g

## PREPARATION OF MANGANOUS SULPHATE

### AIM

To Prepare pure crystals of manganous sulphate ( $\text{MnSO}_4$ )

### PRINCIPLE

Pure crystals of manganous sulphate are prepared by heating the commercial black oxide of manganese ( $\text{MnO}_2$ ) with sulphuric acid



### CHEMICALS REQUIRED

Manganese dioxide ( $\text{MnO}_2$ ) = 4g

Conc.  $\text{H}_2\text{SO}_4$  = 5 ml

### PROCEDURE

About 4g of manganese dioxide is placed in a 100 ml clean beaker and 5 ml of conc.  $\text{H}_2\text{SO}_4$  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  $\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$  are obtained. The crystals are filtered at the pump and dried.

### RESULT:

The yield of  $\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$  = ----- g.

## PREPARATION OF POTASH ALUM, $K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$

### PRINCIPLE:

Potash alum is a double salt of potassium sulphate and aluminium sulphate. Hence it can be prepared by mixing  $K_2SO_4$  and  $Al_2(SO_4)_3 \cdot 18H_2O$  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.



### 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 concentrated 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.

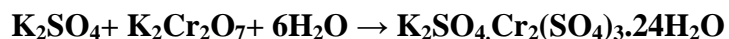
### RESULT:

Yield of potash alum = .....g

## PREPARATION OF CHROME ALUM, $K_2SO_4 \cdot Cr_2(SO_4)_3 \cdot 24H_2O$

### PRINCIPLE:

Potash alum is a double salt of potassium sulphate and aluminium sulphate. Hence it can be prepared by mixing  $K_2SO_4$  and  $Al_2(SO_4)_3 \cdot 18H_2O$  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.



### REQUIREMENTS:

1. Potassium dichromate = 5g
2. Con.  $H_2SO_4$  = 4 ml
3. Alcohol = 5 ml

Chrome alum [ $K_2SO_4 \cdot Cr_2(SO_4)_3 \cdot 24H_2O$ ]. 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  $50^\circ C$ . 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