BGS SCIENCE ACADEMY & RESEARCH CENTER
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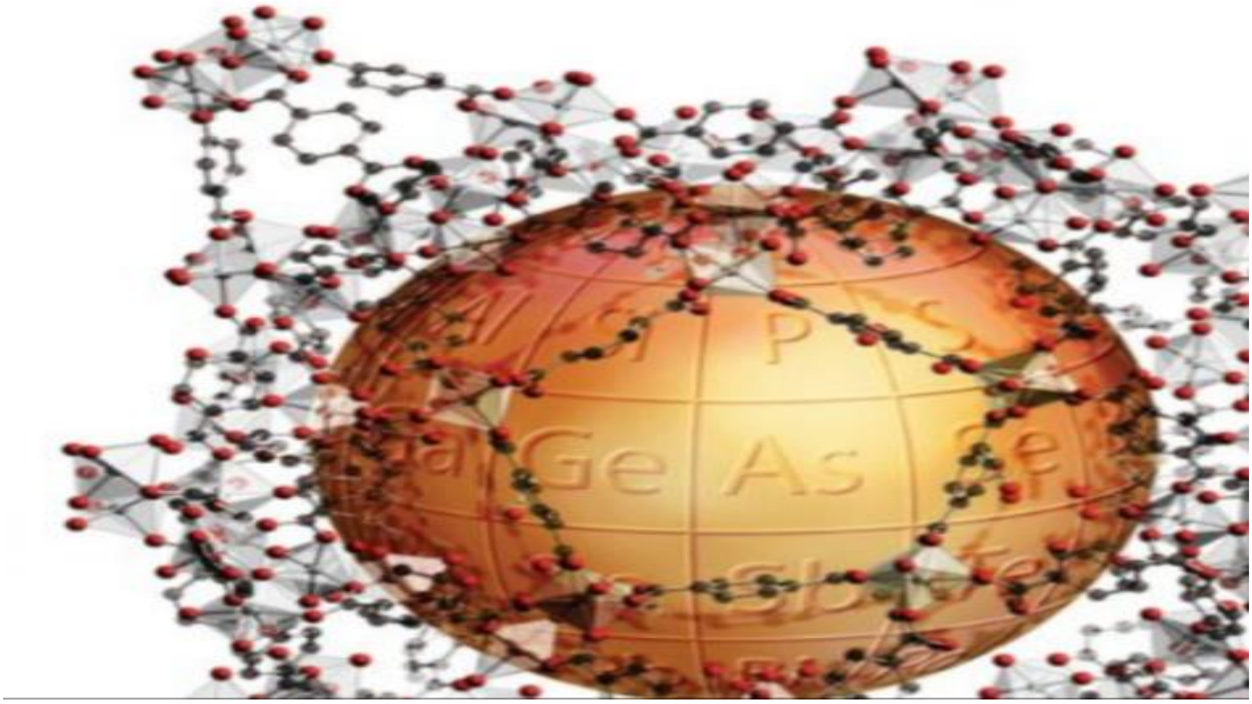
VI Semester B.Sc.,
Paper-VII
Inorganic Chemistry Laboratory Manual



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*** Procedure Writing Experiments**

EXPERIMENT NO.1

AIM: Preparation of Cuprammonium sulphate complex, the determination of λ_{max} and Crystal field splitting energy of the complex

Principle: A solution of copper sulphate reacts with excess of ammonia to form a deep blue complex Cuprammonium sulphate. The absorbance of light of this blue colored complex is measured at different wavelengths and a plot of absorbance vs wavelength, λmax is determined. From the Planck's equation $\Delta E = hc/\lambda$ crystal field stabilization energy can be calculated.

Procedure: Standard solution of copper sulphate containing 1mg/cm^3 Cu^{2+} is prepared by dissolving 400mg of $CuSO_4 . 5H_2O$ crystals in a 100ml standard flask, 5cm^3 liquor ammonia is added to it and made up to the mark by adding distilled water. An intense blue colored solution of cuprammonium sulphate results. The absorbance of this solution is measured using different filters(wavelengths) in a spectrophotometer.

A plot of Absorbance Vs Wavelength is drawn, the wavelength of the corresponding the maximum absorb-ance is recorded from the graph which gives the value of λmax.

From the value λmax the Crystal field stabilization energy is calculated using the equation: CFSE):=hv=hc/ λmax, later CFSE for one mole can be calculated by multiplying the value using Avagadro number.

OBSERVATIONS: Reaction: $Cu + 4NH_3 \longrightarrow [Cu(NH_3)_4]SO_4$

1. Mass of empty beaker …………… ……. ……… …… …… …… = m_1 g.
2. Mass of empty beaker + $CuSO_4$. $5H_2O$ crystals…………… = m_2 g.
3. Mass of empty $CuSO_4$. $5H_2O$ crystals transferred ……….. = $(m_1 - m_2)$ g.

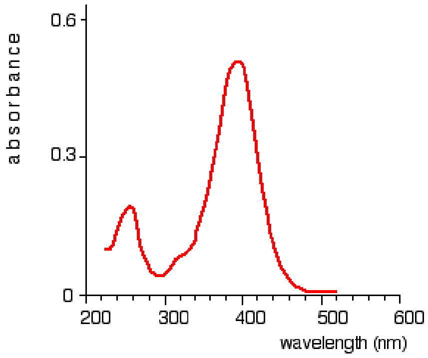
249.5g of $CuSO_4$. $5H_2O$ contains------------------------ 63.5g of Copper
$(m_1 - m_2)$ g. of $CuSO_4$. $5H_2O$ contains -------63.5/249.5 x $(m_1 - m_2)$ g of Copper= 'X'g.

100 cm^3 of copper sulphate solution contains------------- 'X'g. of copper
Hence 1 cm^3 of copper sulphate solution contains------- 0.01X g. of copper.

Calculation of λmax:

Trial No.	λ (nm)	Absorbance or O.D.
1		
2		
3		
.		
.		
.		

A graph of O.D or Absorbance Vs λ is plotted, the peak value of wave length gives λmax



From the graph: λmax = maximum absorbance corresponding wavelength.

Cystal field splitting energy(CFSE): = hν = hc/ λmax; where h= 6.634 x 10^{-34J}.S,
c = $3x10^8$m/s.

CFSE = 6.634 x 10^{-34} x $3x10^8$/λmax = 'Y' Joules.

CFSE for one mole of copper ions in the complex: $[Cu(NH_3)_4]_{2+}$
= 6.023 x 10^{23} x 'Y' = 'Z' J/mol.

Result: λ_{max} = maximum absorbance corresponding wavelength = ………..nm,
Cystal field splitting energy (CFSE) of the complex = 'Z' J/mol.

EXPERIMENT NO.2

AIM: TO ESTIMATE THE AMOUNT OF BARIUM PRESENT IN 250 Cm^3 OF THE GIVEN SOLUTION GRAVIMETRICALLY AS BARIUM SULPHATE

PRINCIPLE: Barium is precipitated as Barium sulphate using barium chloride solution.A hot solution containing sulphate in faintly acidic medium is treated with warm 5% Barium chloride solution till the precipitation is complete. The precipitate is filtered using Whatmann No.42 filter paper, dried ,ignited and weighed as Barium sulphate.

PROCEDURE: The given sulphate solution in the 250 cm^3 standard flask is made up to the mark using distilled water and shaken well. 25 cm^3 of the made up solution is pipetted out into a clean beaker. 1 cm^3 of Con.HCl is added to it and diluted to 30 cm^{3}by adding 2 test tubes of distilled water. The contents are heated to boiling ,10 cm^{3}of hot 0.5M $BaCl_2$ solution is slowly added with constant stirring. The white solid is digested in hot water bath till the precipitate settles down. The completion of the precipitate is tested by adding a few drops of barium chloride solution. The precipitate is digested for half an hour. The precipitate is filtered in whatmann No.41/42 ashless filter paper. The precipitate is washed with hot water till the solid is freed from chloride ions that is the filtrate fails to given white precipitate with silver nitrate solution.

The completely dried filter paper along the sulphate precipitate is placed in a previously weighed porcelain crucible. The crucible is gently heated at first so that the paper chars and volatile matter is expelled. After charring, the temperature is raised to dull redness and burn off any cabon present. The crucible is heated to red hot for 10 to 15 minutes. The crucible is cooled in air and transferred in the desiccator. The crucible along with the precipitate is weighed accurately after 15 minutes till constant weight is obtained, its mass is recorded.

OBSERVATIONS:

Reaction: $SO_4^{2-} + BaCl_2 \rightarrow BaSO_4 + 2\,Cl^-$

1. Mass of empty crucible……W_1 g = ……………….g
2. Mass of empty crucible + $BaSO_4$ precipitate = W_2g =………g
3. Mass of $BaSO_4$ precipitate = (W_2-W_1) g = (………………………………) = ……………….g

233g of Barium sulphate contains ………. 137 g of Barium

W g of Barium sulphate contains ………. $\frac{137 \times W}{233}$ g of Barium

= ……………….. = x g of Barium

25 cm^3 of the solution contains x g of Barium =……………….g

250 cm^3 of the solution contains x X 10 g of Barium = …………………….g

Result: The mass of Barium present in the given solution= ………………g

EXPERIMENT NO.3:

PREPARATION AND ANALYSIS OF FERROUS OXALATE AND ITS ANALYSIS (BOTH IRON AND OXALATE)

AIM: TO PREPARE FERROUS OXALATE AND TO ESTIMATE THE AMOUNT OF OXALATE ($C_2O_4^{2-}$) & Fe^{2+} IN FERROUS OXALATE.

Principle: Ferrous oxalate is prepared by heating oxalic acid solution with ferrous amm-onium sulphate solution acidified with dilute H_2SO_4 as:

$$Fe^{2+}aq + [C_2O_4]^{2-}aq \xrightarrow{H^+} FeC_2O_4.\,2H_2O$$

Ferrous oxalate reacts with potassium permanganate in acidic medium during titration, ferrous is reduced to ferric ions and the oxalic acid formed is titrated against potassium permanganate solution, where oxalate ions can be estimated. Ferric ions in the solution is reduced to ferrous ions using zinc bits, the ferrous ions is titrated against the same per-manganate solution, the titre values are used to estimate the amount of ferrous ions.

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 8H_2O + 10CO_2 \uparrow$$

$$MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 4H_2O + 5Fe^{3+}.$$

Procedure: **(A) Preparation of ferrous oxalate**: About 7.5g of ferrous ammonium sulphate were weighed accurately and transferred into a 250cm^3 beaker. About 60cm^3 of distilled water and 10cm^3 of H_2SO_4(2N) were added and warmed. About 37.5cm^3 of 10% oxalic acid solution was added with constant stirring. The mixture was heated gently for 5 minutes. Yellow precipitate of ferrous oxalate was obtained. The precipitate is filtered and dried.

B) Analysis of ferrous oxalate:

Estimation of oxalate ion and Fe^{2+} in ferrous oxalate using standardized Potasssium per-manganate solution

About 0.05g of ferrous oxalate crystals prepared were weighed accurately. It was transferred quantitatively in a clean conical flask and 25cm^3 of dil. H_2SO_4(2N) was added to dissolve the same. It was heated till bubbles just appeared (55°C - 60°C). The hot solution was titrated against the std. potassium permanganate solution in the burette till the solution turned pale pink, the titre value is recorded (t_1).

About 2g of Zn dust was added to the above solution and boiled for 25 minutes. The residue obtained was dissolved in dil. H_2SO_4(2N) and filtered. It was washed with dil. H_2SO_4(2N) and the washings were added to the filtrate. The filtrate was titrated against std. $KMnO_4$ solution in the burette till the solution turned pale pink. the titre value is recorded (t_2).

Observations and Calculations: A: Preparation of ferrous oxalate:

Mass of ferrous oxalate prepared = ……..g

Mass of ferrous oxalate crystals taken for analysis = 0.05g

Volume of $KMnO_4$ consumed for $C_2O_4^{2-}$ and Fe^{2+} = t_1 cm^3=……………… cm^3

Volume of $KMnO_4$ consumed for Fe^{2+} only = t_2 cm^3 =…………. cm^3

-

B: (i) Estimation of oxalate ion in ferrous oxalate using standardized $KMnO_4$solution

Volume of $KMnO_4$ consumed for $C_2O_4^{2-}$only = $t_1 – t_2$ = _____________ cm^3 (y)

Since, 1cm^3 0.1N $KMnO_4$ ≡ 0.0044g $C_2O_4^{2-}$

ycm^3 0.1N $KMnO_4$ ≡ 0.0044 x y g $C_2O_4^{2-}$

= _______________ g (a g)

0.05g of ferrous oxalate contains __________ (a) g of $C_2O_4^{2-}$

Hence 100g of ferrous oxalate contains: $\frac{100\ x\ (a)}{0.05}$ g of $C_2O_4^{2-}$

= ___________ g of $C_2O_4^{2-}$

Hence % of oxalate ions in ferrous oxalate is = _______________ %

(iii) Estimation of Fe^{2+} in ferrous oxalate using standardized KMnO4 solution

Since, 1cm3 0.1N $KMnO_4$≡ 0.005585g Fe^{2+}

t_2 cm3 0.1N $KMnO_4$ ≡ 0.005585 x t_2 g Fe^{2+}= _______________ g (b g)

0.05g of ferrous oxalate contains __________ (b) g of Fe^{2+}

100g of ferrous oxalate contains …..$\frac{100\ x\ (b)}{0.05}$ g of Fe^{2+}

= ___________g

Hence % of Ferrous ions in ferrous oxalate is = ___________ %

Result: (a) Mass of ferrous oxalate prepared = __________ g

(b) Percentage of $C_2O_4^{2-}$in Fe $C_2O_4.2H_2O$ = _____________

(c) Percentage of Fe^{2+} in Fe $C_2O_4.2H_2O$ = _____________

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OBSERVATIONS: **Reactions**: $Na_2H_2Y \rightarrow 2Na^+ + H_2Y^{2-}$

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

Na_2H_2Y = Disodium salt of EDTA – $C_{10}H_{14}N_2Na_2.2H_2O$

PART-I: a) Preparation of standard Zinc sulphate solution

1. Mass of weighing bottle with $ZnSO_4.7H_2O$ crystals(W_1) =……………….g
2. Mass of Empty weighing bottle with the remainings (W_2) =……………….g
3. Mass of $ZnSO_4.7H_2O$ crystals transferred(W_1-W_2) =……………….g

Molarity of $ZnSO_4$ solution(M_1) = $\frac{(W_1-W_2)x4}{287.5}$ = =………………..M

b) Standarization of EDTA solution:

Burette: EDTA solution

Conical flask: 25 cm^3 of $ZnSO_4$ solution + 5 $cm^3$of pH-10 buffer solution

Indicator: A pinch of Eriochrome black-T

End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(cm^3)			

Concordant Value = ……………. cm^3

$V_2M_2 = V_1M_1$, , Molarity of EDTA solution(M_2) = 25x M_1/V_2 = 25 x………/

EDTA ZnSO4 = ……………..M

PART-II: Estimation of Zinc:

Burette: Standardised EDTA solution

Conical flask: 25 cm^3 of $ZnSO_4$ solution(given) + 5 cm^3 of pH-10 buffer solution

Indicator: A pinch of Eriochrome black-T

End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(cm^3)			

Concordant Value = ……………. cm^3

$V_3M_3 = V_2M_2$ Molarity of Zn^{2+} solution(M_3) = V_2 x M_2/V_3 = …….. x………../25

Zn^{2+} EDTA = …………….M

Mass of Zn^{2+} ions present in 250 cm^3 of the given Zinc sulphate solution = Molar mass x Molarity/4 = 65.37 x Molarity/4

= ……………………..g

EXPERIMENT NO. 4

AIM: TO ESTIMATE THE AMOUNT/ MASS OF ZINC PRESENT IN 250 Cm^3 OF THE GIVEN SOLUTION USING 0.05M EDTA SOLUTION AND ZINC SULPHATE CRYSTALS

PRINCIPLE: Zinc ions in solution form coloured complex with the indicator as well as colourles complex with EDTA. Zinc-indicator complex is less stable than Zn-EDTA complex. At the end point all the free Zinc ions are complexed with EDTA. Now EDTA removes the metal forms the less stable metal-indicator complex releasing the free indicator which has a blue colour at pH-10.

PROCEDURE:

PART I : PREPARATION OF STANDARD ZINC SULPHATE SOLUTION AND STANDARDIZATION OF EDTA SOLUTION

About 3.6 g of pure zinc sulphate crystals are weighed accurately dissolved in about one test tube of dil.sulphuric acid and transferred into 250 cm^3 standard flask and made up to the mark using distilled water and shaken well for uniform concentration.

25cm3 of the made up solution is pipetted out into a clean conical flask, 5 cm^3 of ammonia-ammonium chloride buffer of pH-10 are added and about 2 crystals of Erio-chrome black-T indicator are added. The wine red solution is titrated against the given EDTA solution till the colour changes from wine red to blue colour. The titration is repeated for concordant values.

PART-II: ESTIMATION OF ZINC

The given Zinc sulphate solution in the standard flask is made up to the mark using distilled water and shaken well. 25cm3 of the made up solution is pipetted out into a clean conical flask, 5 cm3 of ammonia-ammonium chloride buffer of pH-10 are added and about 2 crystals of Eriochrome black-T indicator are added. The wine red solution is titrated against the given EDTA solution till the colour changes from wine red to blue colour. The titration is repeated for concordant values.

RESULT: The Amount or mass of Zinc present in 250 cm^3 of the given Zinc sulphate solution is…………………….g.

OBSERVATIONS: **Reactions**: $Na_2H_2Y \rightarrow 2Na^+ + H_2Y^{2-}$

$Ni^{2+} + H_2Y^{2-} \rightarrow NiY^{2-} + 2H^+$

Na_2H_2Y = Disodium salt of EDTA – $C_{10}H_{14}N_2Na_2.2H_2O$

PART-I: a) Preparation of standard Zinc sulphate solution

1. Mass of weighing bottle with $ZnSO_4.7H_2O$ crystals(W_1) =……………….g
2. Mass of Empty weighing bottle with the remainings (W_2) =……………….g
3. Mass of $ZnSO_4.7H_2O$ crystals transferred(W_1-W_2) =……………….g

Molarity of $ZnSO_4$ solution(M_1) = $\frac{(W_1-W_2)x4}{287.5}$ = =………………..M

b) Standarization of EDTA solution:

Burette: EDTA solution

Conical flask: 25 cm^3 of $ZnSO_4$ solution + 5 $cm^3$of pH-10 buffer solution

Indicator: A pinch of Eriochrome black-T

End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(cm^3)			

Concordant value = ……………. cm^3

$V_2M_2 = V_1M_1$, , Molarity of EDTA solution(M_2) = 25x M_1/V_2 = 25 x……../

EDTA ZnSO4 = ……………..M

PartB: Estimation of Ni^{2+} by back titration

Burette: Standard $ZnSO_4$ solution

Conical flask: 35ml. EDTA solution 25 cm^3 of Ni^{2+} soln + 2 cm^3 of NH_3-NH_4Cl buffer

Indicator: Eiochrome Black-T

End-point: Blue to wine red

Trial No.	1	2	3
Final burette reading			
Initial burette reading			
Volume of $ZnSO_4$ run down in cm^3			

Concordant titre value = _________ cm^3

Volume of EDTA= $V_{EDTA} = (MV)_{ZnSO4}/M_{EDTA}$

Volume of EDTA consumed;

V_{EDTA}(con) = 35 - V_{EDTA} = (35 -). $M_{Ni2+} \times V_{Ni2+} = M_{EDTA}\ V_{EDTA}$(cons)

$M_{Ni2+} = \frac{M_{EDTA} \text{ x } V_{EDTA}(\text{cons})}{VNi2+} = \frac{M_{EDTA} \text{ x } V_{EDTA}(\text{cons})}{25}$ =_______________ = __________M

Mass of Ni^{2+} in 250 cm^3 = $\frac{M_{Ni2+} \text{ x } 58.69}{4}$ = ____________ = _________g.

EXPERIMENT NO. 5 Volumetric Estimation of Ni^{2+} using EDTA

Aim: **Estimation of Ni^{2+} present in 250 cm^3 of the given solution by back titration.**

Principle: Ethylene diamine tetracetic acid (EDTA) forms complexes with a large number of cations such as Zn^{2+}, $Mg^{2+,}$ Ni^{2+} etc. The disodium salt of EDTA has two replaceable hydrogen ions and may be represented as H2Y. It reacts with M2+as follows:

$$M^{2+} + H_2Y^{2-} \rightarrow MY^{2-} + 2H^+$$

The above titration can be performed at pH 10 using Erichrome Black-T indicator. Since the above reaction involves liberation of H^+, a buffer mixture has to be used to maintain the pH-10. The buffer mixture used is $NH_3 – NH_4Cl$. Ni^{2+}can be estimated by back titration.

Procedure: **Part A: Preparation of standard $ZnSO_4$solution and standardization of EDTA**

About 3.59g of $ZnSO_47H_2O$ is weighed accurately and transfered into a 250 cm^3standard flask. The weighed solid is dissolved in distilled water and a drop of conc. H_2SO_4 is added, the contents of the flask are mixed well and diluted upto the mark using distilled water. The molarity of $ZnSO_4$ solution is calculated.

25 cm^3 of this solution is pipetted into a clean conical flask. 2 cm^3 of $NH_3 – NH_4Cl$ buffer and 3 drops of Erichrome Black-T indicator are added to it, then diluted with 75 cm^3 (3t.t.) of water. The resulting mixture is titrated against the EDTA solution taken in the burette till the colour of the solution changes from wine-red to blue. The titration is repeated to get concordant values.

Part B: **Estimation of Ni^{2+} by back titration**

The solution given in the standard flask is made upto the mark using distilled water and mixed well. 25 cm^3 of this solution is pipetted into a clean conical flask, the standardised EDTA solution is added from burette in excess (about 30-35 cm^3). 2 cm^3 of $NH_3 – NH_4Cl$ buffer and 3 drops of Erichrome Black-T indicator are added to it, then diluted with 75 cm^3 (3t.t.) of water. The resulting mixture is titrated against the EDTA solution taken in the burette till the colour of the solution changes from blue to wine-red. The titration is Repeated to get concordant values.

Result: Mass of Ni^{2+} estimated in the 250 cm^3 of the given solution is ______g.

OBSERVATIONS:

Reactions: $Na_2H_2Y \rightarrow 2Na^+ + H_2Y^{2-}$

$M^{2+} + H_2Y^{2-} \rightarrow MY^{2-} + 2H^+$(M = Ca, Mg, Zn)

Na_2H_2Y = Disodium salt of EDTA – $C_{10}H_{14}N_2Na_2.2H_2O$

PART-I: a) Preparation of standard Zinc sulphate solution

1. Mass of weighing bottle with $ZnSO_4.7H_2O$ crystals(W_1) =……………….g
2. Mass of Empty weighing bottle with the remainings (W_2) =……………….g
3. Mass of $ZnSO_4.7H_2O$ crystals transferred(W_1-W_2) =……………….g

Molarity of $ZnSO_4$ solution(M_1) = $\frac{(W_1-W_2)x4}{287.5}$ = =…………………M

b) Standarization of EDTA solution:

Burette: EDTA solution

Conical flask: 25 cm^3 of $ZnSO_4$ solution + 5 $cm^3$of pH-10 buffer solution

Indicator: A pinch of Eriochrome black-T

End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(cm^3)			

Concordant value = ……………. cm^3

V_2M_2 = V_1M_1, , Molarity of EDTA solution(M_2) = 25x M_1/V_2 = 25 x………/

EDTA ZnSO4 = ………………M

PART-II: Determination of Total hardness of water

Burette: Standardised EDTA solution

Conical flask: 50 cm^3 of the given sample of water + 5 cm^3 of pH-10 buffer solution

Indicator: 3-4 crystals of Eriochrome black-T; End point: Wine red to blue.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of EDTA run down(cm3)			

Concordant Value = ……………. cm^3

V_3M_3 = V_2M_2, , Molarity of Ca^{2+} solution(M^3) = V_2 x M_2/V_3 = …….. x………../50

(Ca^{2+}) (EDTA)

= ……………..M

Hence Mass of $CaCO_3$ present in 250 cm^3 = M_3x 100/4 =…………………g

Amount of $CaCO_3$ in 10^6 cm^3 = …………x 10^6/250 =………………p

EXPERIMENT NO.6 DETERMINATION OF TOTAL HARDNESS OF OF WATER

AIM: TO ESTIMATE THE TOTAL HARDNESS OF THE GIVEN SAMPLE OF WATER USING 0.05M EDTA SOLUTION AND ZINC SULPHATE CRYSTALS

PRINCIPLE: Hardness of water is due to the presence of calcium and magnesium ions. These ions form characteristic coloured complexes with the indicator and colourless complex with the metal ions. The EDTA –metal ion complex is more stable than the metal-indicator complex.

The metal-ion indicator complex is wine red in colour while the free indicator is blue between the pH 7-11. During titration of a sample of hard water against EDTA, the colour of the solution changes from wine red to deep blue. Total hardness of water iscalculated from the titre value and expressed in parts per millions of CaCO3.

PROCEDURE:

PART I : PREPARATION OF STANDARD ZINC SULPHATE AND STANDARDIZATION OF EDTA SOLUTION

About 3.6 g of pure zinc sulphate crystals are weighed accurately dissolved in about one test tube of dil.sulphuric acid and transferred into 250 cm^3 standard flask and made up to the mark using distilled water and shaken well for uniform concentration.

25cm^3 of the made up solution is pipetted out into a clean conical flask, 5 cm^3 of ammonia-ammonium chloride buffer of pH-10 are added and about 2 crystals of Erio-chrome black-T indicator are added. The wine red solution is titrated against the given EDTA solution till the colour changes from wine red to blue colour. The titration is repeated for concordant values.

PART II: DETERMINATION OF TOTAL HARDNESS OF WATER

50 cm^3 of the given sample of hard water is pipette out into a clean conical flask, 5 cm^3 ofammonia-ammonium chloride buffer of pH-10 are added and about 2 crystals of Erio-chrome black-T indicator are added. The wine red solution is titrated against the given EDTA solution till the colour changes from wine red to blue colour. The titration is repeated for concordant values.

RESULT: The total hardness of the given sample of water is ……………ppm of $CaCO_3$

Reactions: $2Cu^{2+} + 4I^- \rightarrow Cu_2I_2 + I_2$

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

OBSERVATIONS: PART-I: a) Preparation of standard solution of Potassium dichromate

1. Mass of weighing bottle with crystals of Potassium dichromate (W_1)=……………….g
2. Mass of Empty weighing bottle with the remainings (W_2) =……………….g
3. Mass of Potassium dichromate crystals transferred (W_1-W_2) =……………….g

Normality of FAS = $\frac{(W_1-W_2) \times 4}{49}$ = =…………………N

b) Standarization of Sodium thiosulphate solution:

Burette: Sodium thiosulphate solution

Conical flask: 10 cm^3 of $K_2Cr_2O_7$ solution + 2 cm^3 of Con.HCl + 5 $cm^3$of 10% KI soln.

Indicator: Freshely prepared starch

End point: Disappearance of blue colour.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of $Na_2S_2O_3$ run down(cm^3)			

Concordant Value = ……………. cm^3

$V_2N_2 = V_1N_1$, , Normality of $Na_2S_2O_3$ solution(N_2) = 25x N_1/V_2

Hypo ($K_2Cr_2O_7$) = 25 x………/ = --------------N

PART-II: **ESTIMATION OF COPPER**

Burette: Standardized Sodium thiosulphate solution

Conical flask: 10 cm^3 of Cu^{2+} solution + NH_4OH + CH_3COOH + 5 $cm^3$of 10% KI soln.

Indicator: Freshely prepared starch

End point: Disappearance of blue colour.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of $Na_2S_2O_3$ run down(cm^3)			

Concordant Value = ……………. cm^3

$V_3N_3 = V_2N_2$, Normality Cu^{2+} solution(N_3) = V_2N_2/V_3 = …………x ………/10

Cu^{2+} Hypo = …………….N

Mass of copper in 100 cm^3 of the solution = N_3 x 63/10 =……….x 6.3

= ………. g.= w g

Percentage of Copper in Brass = w x 100/2 = ……….x 50 = …………

EXPERIMENT NO. 7 Analysis of Copper in Brass

AIM: TO ESTIMATE THE PERCENTAGE OF COPPER IN THE GIVEN SAMPLE OF BRASS ALLOY(2g OF WHICH IS DISSOLVED IN 100 Cm3 OF THE GIVEN SOLUTION) USING POTASSIUM DICHROMATE CRYSTALS AND SODIUM THIOSULPHATE SOLUTION BY IODOMETRIC METHOD

PRINCIPLE: Brass is an alloy of copper and zinc with small amounts of tin, lead and iron. Brass is dissolved in 1:1 Nitric acid to obtain a solution containing cupric ions. Cupric ions react with potassium iodide in the presence of acetic acid to liberate iodine quantitatively, the liberated iodine is titrated against the standard solution of sodium thiosulphate.

PROCEDURE:

PART I: PREPARATION OF STANDARD SOLUTION OF POTASSIUM DICHROMATE AND STANDARDIZATION OF SODIUM THIOSULPHATE SOLUTION

About 0.49g of potassium dichromate crystals are weighed accurately and dissolved in distilled water, the solution is diluted to 100 cm^3 in a standard flask and shaken well. 10 cm^3 of this solution is pipetted out into a clean conical flask. 2cm3 of con.HCl and 5 cm^3 of 10% solution of potassium iodide are added. The flask is covered with a watch glass and allowed to stand for two minutes. The liberated iodine is titrated against the given sodium thiosulphate solution taken in the burette till the solution turns greenish yellow. 1 cm^3 of freshly prepared starch indicator is added and the titration is continued till the blue colour just disappears. The titration is repeated for concordant values.

PART-II: ESTIMATION OF COPPER

The given brass solution in 100 cm^3 is made up to the mark using distilled water, shaken well. 10 cm^3 of this solution is pipetted out into a clean conical flask. Ammonium hydroxide is added dropwise till a pale blue precipitate is obtained. Dilute acetic acid is added till the precipitate dissolves and then slight excess of acid is added. 5 cm^3 of 10% solution of potassium iodide is added, then the flask is covered with a watch glass and allowed to stand for two minutes. The liberated iodine is titrated against the given sodium thio-sulphate solution taken in the burette till the solution turns to yellow. 1 cm^3 of freshly prepared starch indicator is added and the titration is continued till the blue colour just disappears. The titration is repeated for concordant values.

RESULT: The percentage of Copper in the given sample of Brass is …………

OBSERVATIONS:

Reactions:

$$Fe_2O_3 + 6HCl \rightarrow FeCl_3 + 3H_2O$$

$$2Fe^{3+} + Sn^{2+} \rightarrow 2Fe^{2+} + Sn^{4+}$$

$$SnCl_2 + 2HgCl_2 \rightarrow SnCl_4 + Hg_2Cl_2$$

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

PART-I: **a) Preparation of standard ferrous ammonium sulphate solution**

1. Mass of weighing bottle with Ferrous Ammonium Sulphate crystals(W_1)=………………..g
2. Mass of Empty weighing bottle with the remainings (W_2) =………………..g
3. Mass of Ferrous Ammonium Sulphate crystals transferred(W_1-W_2) =………………..g

Normality of FAS = $\frac{(W_1-W_2)x4}{392}$ = =…………………N

b) Standarization of Potassium dichromate solution:

Burette: Potassium dichromate solution; **Conical flask**: 25 cm^3 of FAS solution + H_3PO_4(5 cm^3) + H_2SO_4(5 cm^3 **Indicator**: Barium Diphenyl amine sulphonate indicator, **End point**: Green to violet.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of $K_2Cr_2O_7$ (cm^3)			

Concordant Value = ……………. cm^3

V_2N_2 = V_1N_1, ,
($K_2Cr_2O_7$) FAS

Normality of $K_2Cr_2O_7$ solution(N_2) = 25x N_1/V_2 = 25 x………/
= ………………N

PART-II: **Estimation of Iron**:

Burette: Potassium dichromate solution; **Conical flask**: 25 cm^3 of reduced haematite ore solution + H_3PO_4(5 cm^3) + H_2SO_4(5 cm^3) **Indicator:** Barium Diphenyl amine sulphonate **End point**: Green to violet.

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of $K_2Cr_2O_7$(cm^3)			

Concordant Value = ……………. cm^3

Normality of Ore solution containing iron(N_3) = V_2x N_2/V_3 = …….x……….. /25 =
= ________ N

Mass of Iron present in 250 cm^3 of the given Haematite ore solution = $\frac{N_3 \text{ x Gm.Eq.Wt.}}{4}$

= $\frac{\_\_\_\_\_\_ \text{x55.85}}{4}$ = …………….g

Percentage of Iron in the given sample of Haematite ore = $\frac{\text{Mass of Iron x 100}}{2.5}$ = …………x40 =…………….

EXPERIMENT NO.8: ANALYSIS HAEMATITE ORE

AIM: DETERMINATION OF THE PERCENTAGE OF IRON IN THE GIVEN SAMPLE OF HAEMATITE ORE(2.5g of the ore is dissolved in in 250 cm^3 of the given solution) USING POTASSIUM DICHROMATE SOLUTION AND FERROUS AMMONIUM SULPHATE CRYSTALS

PRINCIPLE: Haematite ore mainly contains iron having the composition Fe_2O_3 dissolved in 1:1 HCl and filtered. Ferric ions in the solution is reduced to ferrous state by stannous chloride and con.HCl, the resulting ferrous ions is estimated using potassium dichromate solution in acid medium and diphenyl amine as internal indicator.

PROCEDURE:

PART-I: PREPARATION OF STANDARD FERROUS AMMONIUM SULPHATE SOLUTION AND STANDARDIZATION OF POTASSIUM DICHROMATE SOLUTION

About of 10 g of ferrous ammonium sulphate crystals are weighed in a weighing bottle and transferred into 250 cm^3 standard flask and transferred into a beaker, 1 cm^{3}of con. sulphuric acid is added, dissolved in water and transferred into 250 cm^3 standard flask and made up to the mark using distilled water.

25 cm^3 of the made up solution is pipetted out into a clean conical flask, one test tube of acidmixture(Half test tube of phosphoric acid and half test tube of con.sulphuric acid mixture) are added into it, 2 drops of diphenyl amine indicator is added and titrated against the given potassium dichromate solution taken in the burette till the green solution changes into violet. The titration is repeated for concordant values.

PART-II: ESTIMATION OF PERCENTAGE OF IRON IN THE HAEMATITE ORE

The given haematite ore solution in the standard flask is made up to the mark using distilled water and shaken well. 25 cm^3 of the made up solution is pipetted out into a clean conical flask, to this solution half test of Con.HCl is added. The resulting solution is boiled, stannous chloride solution is added in drops till the solution becomes colourless

(to ensure the complete reduction 2 drops are added in excess), 2 test tubes of water are added and cooled to the room temperature. Half test tube of saturated mercuric chloride is added at a stretch, silky white precipitate is obtained(if it turns grey, the solution is rejected and the reduction is repeated). To this solution, 1 test tube of acid mixture is added(Half test tube of phosphoric acid and half test tube of con.sulphuric acid mixture) are added into it, 2 drops of diphenyl amine indicator is added and titrated against the standardized potassium dichromate solution till the colour of the solution changes green to violet. The titration is repeated for concordant readings.

Result: 1. Amount/Mass of iron present in 250 cm^{3}of the given solution = …………..g
2. The percentage of iron in the given sample of Haematite ore = ……………..

OBSERVATIONS: **Reactions**: $CaCO_3 + 2HCl \rightarrow CaCl_2 + CO_2 + H_2O$

$Ca^{2+} + C_2O_4^{2-} \rightarrow Ca\,C_2O_4$

$Ca\,C_2O_4 + 2\,H+ \rightarrow Ca\,C_2O_4 + Ca^{2+}$

$5\,C_2O_4^{2-} + 2MnO_4^{-} + 16H^{+} \rightarrow 2Mn^{2+} + 8H_2O + 10CO_2$

PART I: STANDARDISATION OF POTASSIUM PERMANGANATE SOLUTION

1. Mass of weighing bottle with Sodium oxalate crystals(W_1) =………………..g
2. Mass of Empty weighing bottle with the remainings (W_2) =………………..g
3. Mass of Sodium oxalate crystals transferred(W_1-W_2) =………………..g

Normality of Sodium oxalate solution = $\frac{(W_1-W_2)x4}{67}$ = =……………..N

Burette: Potassium permanganate solution

Conical flask: 25 cm^3 of sodium oxalate solution +2 t.t of dil.H_2SO_4

Indicator: KMnO4 as self-indicator, End point: Permanent pale pink colour

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of KMnO4 (cm^3)			

Concordant Value = ……………. cm^3

$V_2N_2 = V_1N_1$,

($KMnO_4$) Sod.ox. Normality of $KMnO_4$ solution(N_2) = 25x N_1/V_2 = 25 x………/

= ……………..N

PART-II: ESTIMATION OF CALCIUM IN LIME STONE

Burette: Potassium permanganate solution

Conical flask: 25 cm^3 of lime stone solution with calcium oxalate ppt. + 2 tt. of dil.H_2SO_4

Indicator: $KMnO_4$ as self-indicator; End point: Permanent pale pink colour

Trial No.	1	2	3
Final Burette Reading			
Initial Burette Reading			
Vol. of $KMnO_4$ cm^3			

Concordant Value = ……………. cm^3

$V_3N_3 = V_2N_2$, ,

Ca^{2+} ($KMnO_4$) Normality of Ca^{2+} solution(N_3) = V_2 x N2/V_3 = ………. x………/25

= ……………..N

Mass/Amount of Calcium in 250 cm^3 of its solution = N_3x20/4 = ………..x5 =………….g

. .. Percentage of Calcium in the given sample of lime stone = ……….x 100/2 =…………

EXPERIMENT NO.9 ANALYSIS LIME STONE

AIM: TO ESTIMATE THE PERCENTAGE OF CALCIUM IN THE GIVEN SAMPLE OF LIME STONE, 2g OF WHICH IS DISSOLVED IN 250 cm^3 OF THE SOLUTION USING 0.1N POTASSIUM PERMANGANATE SOLUTION & SODIUM OXALATE CRYSTALS

PRINCIPLE: Lime stone is essentially calcium carbonate with varying amounts of magnesium carbonate. Lime stone is dissolved in 1:1 HCl, calcium is precipitated as calcium oxalate by adding a hot solution of ammonium oxalate. The precipitate is collected in quantitative filter paper which is dissolved in dil.sulphuric acid and heated, the hot solution is titrated against standardized solution of potassium permanganate.

PROCEDURE: PART I: STANDARDIZATION OF POTASSIUM PERMANGANATE SOLUTION

About 1.7g of sodium oxalate crystals is accurately weighed and transferred into 250 cm^3 standard flask. It is dissolved in distilled water and made up to the mark for uniform concentration. 25 cm^{3}of the made up solution is pipetted out into a clean conical flask, 2 test tubes of dil.sulphuric acid is added. The contents of the flask are heated nearly to boiling, the hot solution is titrated against the given potassium permanganate solution till the appearance of permanent pale pink colour.

PART II: ESTIMATION OF CALCIUM IN THE LIME STONE

The lime stone solution given in the 250 cm^3 standard flask is made up to the mark with distilled water and shaken well. 25 cm^{3}of this solution is pipetted out into a clean beaker, to this 2 drops of methyl orange indicator is added, it turns red. To this solution ammonium hydroxide solution is added in dropwise till it turns yellow. The about 3 cm^{3}of acetic acid is added till the solution becomes orange red. 4 test tubes of distilled water is added and heated nearly to boiling. To the hot solution 30 cm^3 of ammonium oxalate solution is added slowly with constant stirring. The solution is made slightly alkaline by adding little amount of ammonium hydroxide solution. The white precipitate of calcium oxalate formed is allowed settle down or digested for half an hour. Completion of the precipitation is tested using little amount of ammonium oxalate. The precipitate is filtered through a quantitative filter paper(whatman No.41/42), it is washed with cold distilled water till the washings are freed from chloride ions. The precipitate is then transferred along with the filter paper into the same beaker in which the precipitation was carried out. Two test tubes full of dil.sulphuric acid is added and titrated against standardized $KMnO_4$ solution till permanent pale pink colour is obtained. The titration is repeated for concordancy.

RESULT: The percentage of Calcium in the given sample of lime stone is …………

OBSERVATIONS

Reactions: $Ni^{2+} + 2DMG \rightarrow Ni(DMG)_2 + 2H_2O$

$NiSO_4 + 2C_4H_8O_2N_2 \rightarrow Ni(C_4H_7O_2N_2)_2 + H_2SO_4$

1. Mass of empty and dry sintered glass crucible……W_1 g = ……………….g
2. Mass of empty sintered glass crucible and NiDMG precipitate=W_2g =…………….g
3. Mass of NiDMG precipitate = (W_2-W_1) g = (………………………………) = ……………….g

288.7g of NiDMG complex contains ……….58.7 g of Nickel

W g of NiDMG complex contains ………..$\frac{58.7 \times W}{288.7}$ g of Nickel

= ……………….. = x g of Nickel

25 cm^3 of the solution contains x g of Nickel =……………….g

. . . 250 cm^3 of the solution contains x X 10 g of Nickel = …………………….g

EXPERIMENT NO.10

AIM: TO ESTIMATE THE AMOUNT OF NICKEL PRESENT IN 250 Cm3 OF THE GIVEN SOLUTION GRAVIMETRICALLY AS NICKEL DIMETHYL GLYOXIMATE

PRINCIPLE: Nickel is precipitated by the addition of 1% ethanolic solution of dimethyl glyoxime to hot faintly acidic solution of the nickel salt and then by adding a slight excess of ammonia solution(freed from carbonate). The precipitate is washed with cold water and then weighed as Nickel dimethyl glyoximate after drying at 110^oC - 120^oC.

PROCEDURE: The given nickel salt solution in the 250 cm^3 standard flask is made up to the mark using distilled water and shaken well. 25 cm^3 of the made up solution is pipetted out into a clean beaker. 5 cm^3 of dil.HCl is added to it and diluted to 150 cm^3. The contents of the beaker are heated to 80^oC and a slight excess of dimethyl glyoxime(10 cm^3) reagent is added with stirring. Immediately dilute ammonia solution is added dropwise till it smells. Digest the precipitate by heating on a steam bath for 30 minutes.The completion of the precipitation is tested by adding few more drops of the reagent.The precipitate is allowed to settle for half an hour. The precipitate is filtered througha previously weighed sintered glass crucible. The precipitate is washed repeatedly with cold water untill it is freed from chloride ions. Finally the precipitate is washed with few ml of rectified spirit to remove the excess reagent. The precipitate is dried at 110^oC for about an hour. It is cooled in a desiccator and weighed till concordant mass is obtained.

RESULT: The amount of Nickel present in 250 cm^3 of the given solution is …………………..g.

Experiment No. 11:

PREPARATION OF SODIUM TRI-OXALATO FERRATE (III) AND ESTIMATION OF IRON

AIM: **TO PREPARE SODIUM TRI-OXALATO FERRATE (III) AND ESTIMATE THE AMOUNT OF IRON IN 0.2G OF SODIUM TRI-OXALATO FERRATE (III).**

Principle: Ferrous oxalate reacts with sodium oxalate and oxalic acid in presence of hydrogen peroxide gives Sodium tri-oxalato ferrate (III) complex. The complex when heated with con.sulphuric acid decomposes and liberates carbon dioxide. Ferric ions formed can be reduced to ferrous ions by heating with xinc dust in pre-sence of a dilute acid. Ferrous ions can be estimated by titrating with standard potassium permanganate solution in acid medium. Amount of iron is calculated using the data 1dm^3 of 1N $KMnO_4$ is equivalent to 55.5 g of iron.

Procedure: **Part I**: **Preparation of sodium tri-oxalato ferrate (III):**

About 5g of sodium oxalate is dissolved in about 30 ml of water. To this about 3.25g of ferrous oxalate is added and gently heated. To the hot solution 12.5ml of 20 Volume hydrogen peroxide is added slowly with constant stirring. The mixture is boiled and 12.5ml of 10% oxalic acid solution is added slowly with constant stirring, The hot solution is filtered and 15ml of ethanol is added. The hot solution is filtered and 15ml of ethanol is added. The solution is cooled in a dark place when green crystals of trioxalatoferrate complex separate out. The complex is filtered and washed using 50% ethanol.

Part II : **Estimation of iron**:

About 1g of the complex prepared is weighed(W) and transferred into a chinadish, 2 to 3ml of con.sulphuric acid is added, it is covered with a watch glass and gently heated. When the effervescence stops, it is cooled and transferred into a conical flask. About 2g of zinc dust and 10ml of dil.sulphuric acid is added. It is boiled for 20 minutes, filtered and the filtrate is titrated against the standardized deci-normal potassium permanganate solution until a pale pink colour is obtained. The volume of potassium permanganate(V) is recorded and the process is repeated for concordant values. Mass of iron in 1g of the complex is calculated.

Reactions:

$$2FeC_2O_4 + H_2C_2O_4 + 3Na_2C_2O_4 + H_2O_2 \rightarrow 2Na_3[Fe(C_2O_4)_3] + 2H_2O$$

$$2KMnO_4 + 8H_2SO_4 + 10FeSO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$$

Observations and Calculations:

Mass of Iron in the complex = 55.85 x V x 0.1/ 1000w

Where w is the mass of the complex sodium trioxalato ferrate.

Result: (a) Mass of sodium tri-oxalato ferrate (III) = _________ g

(b) Amount of iron in $Na_3[Fe(C_2O_4)_3]$ = _________ g