M.Sc. PROGRAMME

INORGANIC CHEMISTRY PRACTICALS

Laboratory Manual

Complex Preparations,
Quantitative (Volumetric, Gravimetric & Colorimetric)
and Qualitative Analyses

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CONTENT

PREPARATION

Ex. No. 1 Preparation of Chrome alum

Aim

To prepare chrome alum from potassium dichromate

Chemicals Required

Potassium dichromate – 5g; Con.H_2SO_4 – 4ml
Ethanol - 2.5ml.

Procedure

Chrome alum can be prepared by the reduction of potassium dichromate in acid medium.

$K_2Cr_2O_7 + 4H_2SO_4 + 3C_2H_5OH \rightarrow K_2SO_4+4H_2O+Cr_2(SO_4)_3+3CH_3COOH+ 3H_2$

5g of potassium dichromate was dissolved in 25ml of water. Added 4ml of con.H_2SO_4 to the beaker carefully, stirring the mixture by maintaining the temperature below 50°C. Warmed the mixture on water bath to dissolve the crystals maintaining the temperature below 50°C. Cooled and added 2.5 ml of ethanol (Temperature should not exceed 50°C during the addition of ethanol). Covered with a watch glass and set aside for recrystallisation. Crystallisation could be speeded up by cooling in ice and stirring. Filtered off at the pump. Dried the crystals and noted the yield.

Result

Yield of chrome alum is _______g.

Ex. No. 2 Preparation of Tris(thiourea) copper (I) sulphate

Aim

To prepare tris(thiourea) copper (I) sulphate from thiourea and copper(II) sulphate.

Principle

When a solution of copper sulphate and thiourea are shaken together they readily yield the complex. In the first step, thiourea reduces Cu^{2+} to Cu^{+} and then forms the complex as follows

$$\begin{array}{lcl} \mathrm{2CuSO_4 + H_2N - CS - NH_2} & \rightarrow & \mathrm{Cu_2SO_4 + H_2N - CS - NH - HSO_4} \\ \mathrm{Cu_2SO_4 + 6H_2N - CS - NH_2} & \rightarrow & \mathrm{[Cu(H_2N - CS - NH_2)_3]_2\,SO_4} \\ \hline \mathrm{2CuSO_4 + 7H_2N - CS - NH_2} & \rightarrow & \mathrm{[Cu(H_2N \bullet CS \bullet NH_2)_3]_2\,SO_4 +} \\ & & \qquad \mathrm{H_2N{-}CS{-}NH{-}HSO_4} \end{array}$$

Chemicals Required

Thiourea – 4g; Copper sulphate – 4g;

Procedure

Dissolved 2g of thiourea in 12.5ml of hot water and then cooled the solution to room temperature. Shaken the solution well while adding to it in portions a cold solution of 4g of copper sulphate in 20ml of water. Finally cooled thoroughly in running water while the separated yellowish oil adheres to the walls of the flask. Decanted and rejected the mother liquor. Shaken the oil vigorously with the solution of 2g of thiourea in 12.5ml of water, untill crystallization is complete. Filtered and washed the crystals with small amount of water. Recrystallised with 5% aqueous thiourea containing 2ml of 1N sulphuric acid. The recrystallisation temperature should not be allowed to rise above 80°C. Noted the yield.

Result

Yield of tris(thiourea) copper(I) sulphate is ______g.

Ex. No. 3 Preparation of Tris(thiourea) copper (I) chloride

Aim

To prepare tris(thiourea)copper(I) chloride from thiourea, copper turnings and hydrochloric acid.

Principle

$$2Cu + 2HCl + 6NH_2 - CS - NH_2 \rightarrow [Cu(H_2N - CS - NH_2)_3]_2\,Cl_2 + H_2\uparrow$$

Chemicals Required

Thiourea – 5g; Copper turnings – 1g; Con.HCl – 5ml.

Procedure

Dissolved 5g of thiourea in 25ml of hot water in a conical flask with a funnel. To the solution added 1g of copper turnings and then 5ml of con.HCl. Heated on the wire gauze while copper is dissolved with the liberation of hydrogen. After 5 minutes, the solution is filtered while hot and then allowed the solution to cool slowly. The crystals separated are filtered, washed with acetone and noted the yield.

Result

Yield of tris(thiourea)copper(I) chloride is ______g.

Ex. No. 4 Preparation of Potassium trioxalatochromate (III)

Aim

To prepare the complex potassium trioxalatochromate(III).

Principle

When potassium dichromate is treated with oxalic acid and potassium oxalate, green complex of potassium trioxalatochromate (III) is formed.

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

Chemicals Required

Oxalic acid – 3g; Potassium dichromate – 1g; Ethyl alcohol – 2ml; Potassium oxalate – 1.2g.

Procedure

To the solution of 3g of oxalic acid in 8ml of warm water, added in portions 1g of potassium dichromate. When the vigorous reaction has subsided, the resulting solution was heated to boiling and in it 1.2g of potassium oxalate was dissolved. The whole solution was cooled to room temperature. Added 1.2ml of ethanol and cooled. The green crystals separated out from an almost black solution. Collected the precipitate from Buchner funnel, washed with 1% ethanol and finally with pure ethanol. The yield was noted.

Result

Yield of potassium trioxalatochromate (III) is ______g.

Ex. No. 5 Preparation of Tetramminecopper(II) sulphate

Aim

To prepare tetramminecopper(II) sulphate from copper sulphate.

Principle

When a solution of copper sulphate is mixed with an excess of ammonia solution, initially a white precipitate of cuprous hydroxide formed, dissolved again to give a deep blue solution of cuprammonium sulphate complex. The complex is reprecipitated from the solution by the addition of alcohol in which it is insoluble.

$$CuSO_4 + 4NH_4OH \rightarrow \underset{\text{Tetramminecopper(II) sulphate tetrahydrate}}{[Cu(NH_3)_4]SO_4 . 4H_2O}$$

Chemicals Required

Copper sulphate – 2g; Ammonia – 3ml; Alcohol – 3ml.

Procedure

2.5g of copper sulphate was dissolved in 10ml of distilled water shaken well. To the clear solution 8ml of liquor ammonia (1:1) was added and shaken well. 8ml of ethanol was added to the resulting blue solution. Blue crystalline ppt obtained was heated on a water bath, filtered of not clear. Kept aside for 45 minutes Blue crystals were formed.

Result

Yield of tetramminecopper(II) sulphate is ______g.

Ex. No. 6 Preparation of Potassium trioxalatoferrate(III)

Aim

To prepare the complex potassium trioxalatoferrate(III).

Principle

Ferrous oxalate is obtained by mixing ferrous sulphate and oxalic acid to which potassium hydroxide and hydrogen peroxide are added to get the complex potassium trioxalatoferrate(III).

Chemicals Required

Ferrous oxalate – 3.3g; Hydrogen peroxide – 12.5ml; Oxalic acid – 5g and potassium hydroxide – 5g; Ethanol – 50 ml.

Procedure

Suspended about 3.3g of ferrous oxalate in a warm solution of 5g of KOH in 15ml of water. Added 12.5ml of 20 volume of H_2O_2 from a burette stirring the liquid continuously during the addition and keeping its temperature to 40°C. Heated the mixture containing some ferrous hydroxide to boiling and dissolved the precipitate by adding first in one portion a 10ml solution of oxalic acid containing 5g of crystals per 50ml. Then a further small amount of about 2.5ml carefully drop by drop from a burette. During the addition of oxalic acid the solution should be nearly boiling. Excess of acid is to be added. Filtered the hot solution, added to the filtrate 50ml of ethanol, redissolved any precipitated crystals by gentle heating and then set aside the solution in cupboard for crystallization. Washed the precipitate at the pump using an equimolar mixture of ethanol, water and finally with acetone. The yield was noted.

Result

Yield of potassium trioxalatoferrate(III) is ______g.

Ex. No. 7 Preparation of Sodium (thiosulphato)cuprate(I)

Aim

To prepare the complex Sodium (thiosulphato)cuprate(I) complex.

Principle

$2CuSO_4 + 2Na_2S_2O_3 \rightarrow Cu_2SO_4 + Na_2S_4O_6 + Na_2SO_4$

$Cu_2SO_4 + Na_2S_2O_3 \rightarrow Cu_2S_2O_3 + Na_2SO_4$

$3Cu_2S_2O_3 + 2Na_2S_2O_3 \rightarrow Na_4[Cu_6(S_2O_3)_5]$

Chemicals Required

Cupric sulphate – 5g; Sodium thiosulphate – 9g;

Procedure

Dissolved 5g of cupric sulphate in 25ml of warm water and 9g of sodium thiosulphate crystals in 15ml of warm water. Adjusted the temperature of each solution to about 40°C. Then poured the solution of thiosulphate into well stirred cupric sulphate solution. After a short time the crystalline bright yellow complex begins to separate and was finally deposited in about an hour. It was then filtered off by the use of filter pump and the solid was thoroughly washed with cold water which was finally with acetone to facilitate drying of the product at a temperature not exceeding 50°C.

Result

The yield of sodium thiosulphatocuprate(I) is ______g.

Ex. No. 8 Preparation of Hexamminecobalt(III) chloride

Aim

To prepare the complex hexamminecobalt(III) chloride..

Principle

Preparation of hexammine cobalt(III) chloride involves the mixing of ammonium chloride and cobaltous chloride followed by the addition of concentrated ammonia and hydrogen peroxide.

$$2CoCl_2 + 2NH_4Cl + 10NH_3 + H_2O_2 \rightarrow 2[CO(NH_3)_6]Cl_3 + 2H_2O$$

Chemicals Required

Ammonium chloride – 3g; Cobaltous chloride – 4.5g; Charcoal – 0.25g; Hydrogen peroxide – 9ml.

Procedure

In a boiling solution of 3g of ammonium chloride in 6.2ml of water dissolved 4.5g of cobaltous chloride hexahydrate. Placed 0.25g of charcoal in a dry flask, introduced the hot solution and then cooled the whole in running water. Washed out the vessel in which the solution was first made with 10ml of concentrated ammonia and added liquor ammonia to the solution. Cooled the whole preparation in an ice bath to 10°C. Added slowly in portions 9ml of 20 volume hydrogen peroxide while briskly shaking the flask during the addition of the oxidizing agent. Heated the mixture gradually to 60°C and then maintained the temperature in the range 50 - 60°C with frequent temperature checking by shaking until a pinkish tint in the liquid was removed. Crystals began to separate at the close of the heating and were deposited.

Filtered off the crude solid without washing. Transferred it to a beaker containing a boiling mixture of 37.5ml of water and 5ml of con.HCl. When all the solid except the charcoal has dissolved, filtered the liquid while it was still hot. Added 5ml of con.HCl to the filtrate and then cooled the liquid in an ice bath. Golden yellow crystals of hexamminecobalt(III)chloride separated in almost quantitative yield.

Result

Yield of hexammine cobalt (III) chloride is _____g.

Standard magnesium Sulphate Vs EDTA

S. No.	Volume of Standard $MgSO_4$ in ml	Burette reading (ml)		Volume of EDTA in ml	Indicator
		Initial	Final		
					Eriochrome Black – T

Calculation

Weight of magnesium Sulphate in 250ml =

Strength of magnesium Sulphate =

=

Volume of magnesium sulphate solution =

Volume of EDTA =

Strength of EDTA =

=

Made up zinc sulphate Vs Standardised EDTA solution

S. No.	Volume of zinc sulphate in ml	Burette reading (ml)		Volume of EDTA in ml	Indicator
		Initial	Final		
					Eriochrome Black – T

Volume of EDTA solution =

Strength of EDTA solution =

Volume of $ZnSO_4$ =

Strength of $ZnSO_4$ =

=

Weight of zinc present in the whole
of the given solution =

=

EDTA TITRATIONS

Ex. No. 1

ESTIMATION OF ZINC

AIM

To estimate the strength of the given EDTA solution using standard magnesium sulphate solution and to estimate the amount of zinc present in the whole of the given solution.

PROCEDURE

STANDARDISATION OF EDTA SOLUTION

Prepared a standard solution of 0.01M $MgSO_4$ in a 250ml standard flask by weighing approximately 0.62 g of it. Pipetted out 20ml of it into a clean conical flask. Added 5ml of buffer solution of pH=10. Heated to bearable warmth. Added a pinch of Erio-T indicator and titrated against EDTA solution. The end point is the disappearance of pink colour and appearance of light blue colour. Repeated the titration for concordant values.

Made up the given solution to 100ml in a standard flask. Pipetted out 20ml of it into a clean conical flask. Added 5ml of buffer solution of pH 10, a pinch of Erio-T and titrated against standardized EDTA. The end point is the disappearance of pink colour and appearance of blue colour. Repeated the titration for concordant values.

RESULT

Amount of zinc present in the whole of the given solution = g.

Standard magnesium Sulphate Vs EDTA

S. No.	Volume of Standard $MgSO_4$ in ml	Burette reading (ml)		Volume of EDTA in ml	Indicator
		Initial	Final		
					Eriochrome Black – T

Calculation

Weight of magnesium Sulphate in 250ml =

Strength of magnesium Sulphate =

=

Volume of magnesium sulphate solution =

Volume of EDTA =

Strength of EDTA =

=

Hard water Vs Standardized EDTA

S. No.	Volume of Hard Water in ml	Burette reading (ml)		Volume of EDTA in ml	Indicator
		Initial	Final		
					Eriochrome Black – T

Volume of EDTA =

Strength of EDTA =

Volume of Hard water =

Strength of Hard water = = x

Ex. No. 2

DETERMINATION OF HARDNESS OF WATER

AIM

To determine the hardness of the given sample of water.

PROCEDURE

STANDARDISATION OF EDTA SOLUTION

Prepared a standard solution of 0.01M. $MgSO_4$ in a 250ml standard flask by weighing about 0.62g of it. Pipetted out 20ml of it into a clean conical flask. Added 5ml of buffer solution of pH = 10. Heated to bearable warmth, added a pinch of Erio-T indicator and titrated against EDTA solution. The end point is the disappearance of pink colour and appearance of light blue colour. Repeated the titration for concordant values.

DETERMINATION OF TOTAL HARDNESS

Buretted out 50ml of the sample water into a clean conical flask. Added 1ml of buffer solution of pH 10 and then a pinch of Erio-T indicator. 0.1ml of 0.1M magnesium-EDTA complex solution was added. (1ml of 0.01M EDTA $\equiv$ 1mg of $CaCO_3$). Titrated the solution against standardized EDTA solution until the colour changed from wine red to pale blue. From the titre values, the total hardness of water was calculated in parts per million (ppm).

Weight/litre of Ca^{2+} = ‘x’ x 40.08

= ‘y’ g of Ca

40.08g of Ca^{2+} is present in 100.08g of $CaCO_3$

$\therefore$ ‘y’ g of Ca^{2+} is present in = $\frac{y \times 100.08}{40.08}$ = ‘z’ g of $CaCO_3$

10^6CC hard water will contain = $\frac{z}{1000} \times 10^6$ = ppm.

Permanent hard water Vs Standardized EDTA

S. No.	Volume of Permanent Hard Water in ml	Burette reading (ml)		Volume of EDTA in ml	Indicator
		Initial	Final		
					Eriochrome Black – T

Volume of EDTA =

Strength of EDTA =

Volume of Hard water =

Strength of Hard water = = x

=

Weight per litre of calcium = 40.08 × x = y

40.08g of Ca^{2+} is present in 100.08g of $CaCO_3$

$\therefore$ ‘y’ g of Ca^{2+} is present in = $\frac{y \times 100.08}{40.08}$

10^6CC of hard water will contain = $\frac{z}{1000} \times 10^6$ = ppm

Permanent hardness of water =

Temporary hardness of water = Total hardness – permanent hardness

=

=

DETERMINATION OF PERMANENT HARDNESS

250ml of the sample of water was buretted out into a 400ml beaker. Boiled for about 30 minutes. Cooled the solution and filtered through Whatmann No.42 filter paper directly into a 250ml standard flask. Diluted the filtrate and made upto 250ml. Pipetted out 50ml of this solution and titrated against standardized EDTA solution using the same procedure as for the total hardness. The temporary hardness could be found by subtracting the permanent hardness from the total hardness.

Result

Total hardness of water =

Permanent hardness =

Temporary hardness =

Standard Magnesium Sulphate Vs EDTA

S. No.	Volume of Standard $MgSO_4$ in ml	Burette reading (ml)		Volume of EDTA in ml	Indicator
		Initial	Final		
					Eriochrome Black – T

Calculation

Weight of magnesium Sulphate in 250ml =

Strength of magnesium Sulphate =

=

Volume of magnesium sulphate solution =

Volume of EDTA =

Strength of EDTA =

=

Nickel ammonium Sulphate Vs Standardised EDTA

S. No.	Volume of Nickel ammonium sulphate in ml	Burette reading (ml)		Volume of EDTA in ml	Indicator
		Initial	Final		
					Murexide

Ex. No. 3

DETERMINATION OF PERCENTAGE PURITY OF NICKEL AMMONIUM SULPHATE

AIM

To determine the percentage purity of the given sample of nickel ammonium sulphate.

PROCEDURE

STANDARDISATION OF EDTA SOLUTION

Prepared a standard solution of 0.01M $MgSO_4$ by weighing about 0.62g of it in a 250ml standard flask. Pipetted out 20ml of it into a clean conical flask. Added 5ml of buffer solution of pH10. Heated to bearable warmth, added a pinch of Erio-T indicator and titrated against EDTA solution. The end point is the disappearance of pink colour and appearance of light blue colour. Repeated the titration for concordant values.

Prepared approximately 0.01M solution of nickel ammonium sulphate in a 100ml standard flask by weighing around 0..4g of it. Pipetted out 20ml of the solution into a clean conical flask, added 5ml of buffer solution of pH=10, warmed the solution to about 60°C. Added murexide indicator and titrated against standardized EDTA solution till the colour changes from yellow to violet. Repeated the titration for concordant values. Calculated the amount of nickel present and determined the percentage purity of the given sample of nickel ammonium sulphate.

Volume of EDTA =

Strength of EDTA =

Volume of nickel Ammonium Sulphate =

Strength of nickel Ammonium Sulphate =

= x

Weight per litre of nickel ammonium sulphate = x × 394.97

= y

394.97g of nickel ammonium sulphate contains 58.7g of nickel

y g of nickel ammonium sulphate contains = $\frac{58.7}{394.97} \times y$

= z

Weight of nickel ammonium sulphate in 100ml = weight taken

Weight of nickel ammonium sulphate in 1000ml= weight taken x 10 = ‘a’

Weight of nickel present in ‘a’ g of nickel ammonium sulphate = $a \times \frac{58.7}{394.97}$ = ‘b’

= ‘b’ g of nickel

Percentage purity of the given sample of nickel ammonium sulphate = $\frac{Experimental\ value}{Theoretical\ value} \times 100$

= $\frac{z}{b} \times 100$

= %

RESULT

Percentage purity of the given sample of nickel ammonium sulphate =

Standard Magnesium Sulphate Vs EDTA

S. No.	**Volume of Standard $MgSO_4$ in ml**	**Burette reading (ml)**		**Volume of EDTA in ml**	**Indicator**
		Initial	**Final**		
					Eriochrome Black – T

Calculation

Weight of magnesium sulphate in 250ml =

Strength of magnesium sulphate =

=

Volume of magnesium sulphate solution = 20 ml

Volume of EDTA = V_1 ml

Strength of EDTA =

= N_1

Excess EDTA solution Vs Standard $MgSO_4$

S. No.	**Volume of Nickel solution in ml**	**Burette reading (ml)**		**Volume of $MgSO_4$ in ml**	**Indicator**
		Initial	**Final**		
					Eriochrome Black – T

Ex. No. 4

ESTIMATION OF NICKEL (BACK TITRATION)

AIM

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

PRINCIPLE

Nickel is very slow in complex formation with EDTA solution. So instead of doing the direct titration, nickel can be estimated by back titration using Erio-T as indicator.

PROCEDURE

STANDARDISATION OF EDTA

Prepared a standard solution of 0.01M $MgSO_4$ by weighing about 0.62g of it in a 250ml standard flask. Pipetted out 20ml of it into a clean conical flask. Added 5ml of buffer solution of pH10. Heated to bearable warmth, added a pinch of Erio-T indicator and titrated against EDTA solution. The end point is the disappearance of pink colour and appearance of light blue colour. Repeated the titration for concordant values.

V_1ml of EDTA requires 20 ml of $MgSO_4$ solution

$\therefore V_2$ ml of $MgSO_4$ required = $\frac{V_1 \times V_2}{20}$ ml of EDTA

= x ml

Total volume of EDTA taken = 40 ml

Volume of EDTA required for 20ml of Nickel ammonium sulphate = $40 - x = y$ ml

Strength of EDTA solution = N_1

Volume of nickel solution = 20 ml

Strength of nickel solution = $\frac{y \times N_1}{20}$

= N_2

Amount of nickel present in the whole of the given solution = $\frac{N_2 \times 58.7}{10}$

=

Made up the given solution to 100ml in a standard flask. Pipetted out 20ml of this solution into a clean conical flask. Added 40ml of approximately 0.01 M standardized EDTA solution, 5ml of buffer solution of pH 10, added a pinch of Erio-T and titrated with standard magnesium sulphate solution until the colour changes from blue to red. The indicator should be added after the addition of EDTA and buffer solution. Repeated the titration for concordant values.

RESULT

Amount of nickel present in the whole of the given solution =

ESTIMATEION OF COPPER

Standardisation of thio

Standard Copper Sulphate Vs Thiosulphate solution

S. No.	Volume of Copper sulphate (ml)	Burette reading (ml)		Volume of Thiosulphate (ml)	Indicator
		Initial	Final		
					Starch

Weight of $CuSO_4 . 5H_2O$ in 100 ml =

Strength of $CuSO_4 . 5H_2O$ =

=

Volume of $CuSO_4$ =

Volume of $Na_2S_2O_3$ =

Strength of $Na_2S_2O_3$ =

=

VOLUMETRIC AND GRAVIMETRIC ESTIMATIONS

Ex. No. 1

ESTIMATION OF COPPER AND NICKEL

AIM

To estimate the amount of copper and nickel present in the whole of the given solution.

PRINCIPLE

Copper is estimated iodometrically and nickel gravimetrically after the separation of copper as sulphide.

PROCEDURE

ESTIMATION OF NICKEL

Made up the given solution to 250ml. in a standard flask. Pipetted out 40ml of the made up solution into a 250ml beaker, added about 10ml of concentrated hydrochloric acid. Diluted to about 70ml and heated to boiling. To the hot solution passed H_2S gas till the precipitate settles down readily. Filtered through Whatmann number 41 filter paper. Collected the filtrate in a 400ml beaker covering the precipitate always with solution or water. Washed the precipitate with water containing H_2S gas and then several times with distilled water. Concentrated the filtrate to about 150ml. Tested for complete removal of H_2S gas. Made the solution slightly acidic. Heated to about 70°C and added slight excess of dimethyl glyoxime about 15-20ml followed by the addition of 1:1 ammonia dropwise directly to the solution with constant stirring. Added a slight excess of ammonia. Digested the precipitate in a water bath for about 20-30 minutes. Tested for complete precipitation.

Given Copper-nickel mixture solution Vs Standardised Thio

S. No.	Volume of given CuNi (ml)	Burette reading (ml)		Volume of Thiosulphate (ml)	Indicator
		Initial	Final		
					Starch

Volume of $Na_2S_2O_3$ =

Strength of $Na_2S_2O_3$ =

Volume of given solution =

Strength of given solution =

=

Weight / litre of copper = 63.55 x

Amount of copper present in 250ml =

=

Amount of copper present in the whole of the given solution =

Filtered the cold solution through a previously weighed G_3 crucible. Washed the precipitate with water containing few drops of NH_3 till free from chloride solution. Dried at 110° to 120°C and weighed as nickel dimethyl glyoxime complex. Repeated drying and weighing till a constant weight is got. Duplicated the experiment.

ESTIMATION OF COPPER

Pipetted out 40ml of the solution into a conical flask. Added ammonia dropwise until a precipitate is obtained. Added dilute acetic acid in drops to dissolve the precipitate and added two drops in excess. Added about 10ml of 15% solution of potassium iodide and titrated the liberated iodine against sodium thiosulphate solution. When the solution becomes pale yellow, added starch and continued the titration until the blue colour fades. Added 1g of analar ammonium thiocyanate and titrated until the blue colour is discharged. Repeated the procedure for concordant values. Standardised the given thiosulphate solution using standard copper sulphate solution prepared by weighing approximately 2.5g and dissolving it in 100ml.

RESULT

1. Amount of copper present in the whole of the given solution =
2. Amount of nickel present in the whole of the given solution =

ESTIMATION OF NICKEL

CRUCIBLE A

Weight of crucible + $Ni(DMG)_2$ =

Weight of empty curcible =

Weight of $Ni(DMG)_2$ =

288.93g of $Ni(DMG)_2$ contains 58.71g of Ni.

$\therefore$ g of $Ni(DMG)_2$ contains =

=

Weight of Nickel present in the whole of the given solution

=

CRUCIBLE B

Weight of crucible + $Ni(DMG)_2$ =

Weight of empty crucible =

Weight of $Ni(DMG)_2$ =

288.93g of $Ni(DMG)_2$ contains 58.71g of Ni.

$\therefore$ g of $Ni(DMG)_2$ contains =

=

Weight of Nickel present in the whole of the given solution

=

ESTIMATEION OF IRON

Made up Iron-zinc mixture solution Vs standard potassium dichromate

S. No.	**Volume of Iron-zinc mixture (ml)**	**Burette reading (ml)**		**Volume of $K_2Cr_2O_7$(ml)**	**Indicator**
		Initial	**Final**		
					Diphenylamine

Weight of $K_2Cr_2O_7$ in 250 ml =

Strength of $K_2Cr_2O_7$ =

=

a) Volume of $K_2Cr_2O_7$ =

Volume of Iron and zinc solution =

Strength of Iron and zinc solution =

=

Amount of Iron present in the whole of the given solution =

=

b) Volume of $K_2Cr_2O_7$ =

Strength of $K_2Cr_2O_7$ =

Volume of Iron and zinc solution =

Strength of Iron and zinc solution =

=

Ex. No. 6

ESTIMATION OF IRON AND ZINC

AIM

To estimate the amount of iron and zinc present in the whole of the given solution.

PRINCIPLE

Iron is estimated volumetrically and zinc gravimetrically after the separation of iron.

PROCEDURE

ESTIMATION OF IRON

Made up the given solution to 250ml with distilled water. Pipetted out 40ml into a clean conical flask and added 10ml of analar hydrochloric acid. Heated it to boiling and to the hot solution added freshly prepared stannous chloride in drops till a pale green colour is obtained. Cooled the solution well and then added saturated solution of mercuric chloride in drops, till a silky white precipitate is obtained. If a black precipitate is formed or a precipitate is not seen, the reduction is incomplete and so the solution is discarded. Added 20ml of mixture of sulphuric acid and phosphoric acid and 3 drops of diphenylamine indicator. Titrated the solution versus standard potassium dichromate prepared by weighing approximately 0.6g and dissolving it in 250ml taken in the burette. The end point is the appearance of bluish violet colour. Repeated the titration for concordant values.

ESTIMATION OF ZINC

Pipetted out 40ml of the made up solution into a 250ml beaker. Diluted to 50ml added about 5g of ammonium chloride, heated to boiling and added 1:1

Amount of iron present in the whole of

the given solution =

=

ESTIMATION OF ZINC

CRUCIBLE A

Weight of crucible + zinc ammonium phosphate =

Weight of empty crucible =

Weight of zinc ammonium phosphate =

178.32g of zinc ammonium phosphate contains 65.37g of zinc

∴ of zinc ammonium phosphate contains =

=

Amount of zinc present in the whole of the given solution =

CRUCIBLE B

Weight of crucible + zinc ammonium phosphate =

Weight of empty crucible =

Weight of zinc ammonium phosphate =

178.32g of zinc ammonium phosphate contains 65.37g of zinc

∴ of zinc ammonium phosphate contains =

=

Amount of zinc present in the whole of the given solution =

ammonia till a precipitate is obtained and allowed the precipitate to settle. Filtered the precipitate through Whatmann No.41 filter paper and collected the filtrate in a 400ml beaker.

Washed the precipitate with 1:100 ammonia solution. Dissolved the precipitate in minimum amount of warm dilute hydrochloric acid and collected the filtrate in the beaker in which the precipitation was already done. Precipitated the iron in the solution as before by adding ammonium chloride and ammonium hydroxide and filtered through the same filter paper. Collected the filtrate along with first filtrate in the same 400ml beaker. Washed the precipitate with hot water containing few ml of 1% ammonium nitrate solution. Evaporated the combined filtrate and reduced the volume.

Neutralised the solution, heated to boiling and then added 20-25ml of 20% diammonium phosphate from a burette with constant stirring. Digested the flocculant precipitate till it is crystalline and settles down readily ($\simeq$ 60min). Filtered the precipitate through a previously weighed sintered glass crucible. Washed the precipitate with 1% diammonium hydrogen phosphate solution and then distilled water. Dried the precipitate at 125°C for about one hour and weighed as zinc ammonium phosphate.

RESULT

1) Amount of iron present in the whole of the given solution

    i)

    ii)

2) Amount of zinc present in the whole of the given solution

    i)

    ii)

ESTIMATION OF ZINC

Standard zinc sulphate Vs $K_4[Fe(CN)_6]$

S. No.	Volume of $ZnSO_4$ (ml)	Burette reading (ml)		Volume of $K_4[Fe(CN)_6]$ (ml)	Indicator
		Initial	Final		
					Diphenylamine

Weight of $ZnSO_4$ in 100ml =

Strength of $ZnSO_4$ =

=

Volume of $ZnSO_4$ =

Volume of $K_4[Fe(CN)_6]$ =

Strength of $K_4[Fe(CN)_6]$ =

=

Ex. No. 7

ESTIMATION OF COPPER AND ZINC

AIM

To estimate the amount of copper gravimetrically and the amount of zinc volumetrically.

PRINCIPLE

This is an excellent method for estimating copper since the thiocyanates of other metals are soluble. Separation may thus be effected from Bi, Cd, As, Sb, Sn, Ni, Fe, Mn and Zn.

ESSENTIAL CONDITIONS OF PRECIPITATION

1) Slight excess of acidity with hydrochloric acid since solubilities increase appreciably with decrease in pH.
2) Presence of reducing agents such as ammonium bisulphate (or) sulphurous acid.
3) Slight excess of ammonium thiocyanate-large excess increases the solubility of cuprous thiocyanate due to the formation of the thiocyanate complex.

In the absence of oxidizing agents, the reaction may be represented as

$$2Cu^{2+} + HSO_3^- + H_2O \rightarrow 2Cu^+ + HSO_4^- + 2H^+$$

$$Cu^+ + CNS^- \rightarrow Cu(CNS)\downarrow$$

Given Copper-zinc mixture solution Vs standardized $K_4[Fe(CN)_6]$

S. No.	Volume of given solution (ml)	Burette reading (ml)		Volume of $K_4[Fe(CN)_6]$ (ml)	Indicator
		Initial	Final		
					Diphenylamine

Volume of $K_4[Fe(CN)_6]$ =

Volume of given solution =

Strength of given solution =

=

Weight of Zn present in 100ml = 65.37 x

=

Weight of Zinc present in the whole
Of the given solution =

=

PROCEDURE

The given solution was made upto 250ml. Pipetted out 40ml of it into a 400ml beaker, added a few drops of dilute hydrochloric acid and then about 20ml of saturated solution of H_2SO_3. Diluted the solution to 150ml and heated to boiling and added freshly prepared 10% solution of ammonium thiocyanate slowly with constant stirring from a burette until a slight excess was present. (The precipitate of cuprous thiocyanate must be white and mother liquor colourless with the smell of SO_2). Allowed the precipitate to settle for two hours preferably over night. Filtered through a previously weighed G_4 sintered glass crucible and washed the precipitate 10-15 times with a cold wash solution. The wash solution was prepared by adding 1ml of 10% NH_4SCN and 6 drops of saturated sulphurous acid solution to 100ml of water. Finally washed several times with 20% alcohol to remove NH_4SCN. Dried the precipitate to constant weight at 110-120°C and weighed as cuprous thiocyanate.

ESTIMATION OF ZINC

PRINCIPLE

Zinc ions in neutral or acid medium react with potassium ferrocyanide solution to form a slightly soluble precipitate $K_2Zn_3[Fe(CN)_6]_2$

$$3Zn^{2+} + 2K_4[Fe(CN)_6] \rightarrow K_2Zn_3\,[Fe(CN)_6]_2 + 6K^+$$

For this titration internal indicators like sodium diphenyl amine sulphonate, diphenyl benzidine, 3,3′-diphenyl naphthalidine or 3,3′-diphenyl naphthalidine disulphonic acid are used. The substances are oxidation reduction

ESTIMATION OF COPPER

CRUCIBLE A

Weight of crucible + cuprous thiocyanate=

Weight of empty crucible =

Weight of cuprous thiocyanate =

121.63g of cuprous thiocyanate contains 63.55g of copper

∴ of cuprous thiocyanate contains =

=

Weight of copper present in the whole of
the given solution =

=

CRUCIBLE B

Weight of crucible + cuprous thiocyanate=

Weight of empty crucible =

Weight of cuprous thiocyanate =

121.63g of cuprous thiocyanate contains 63.55g of copper

∴ of cuprous thiocyanate contains =

=

Weight of copper present in the whole of
the given solution =

=

indicators. Therefore they are dependent on the ratio of concentration of ferrocyanide to ferricyanide ions in solution. Usually 0.08 molar solution of $K_4[Fe(CN)_6]$ to which 0.3g of $K_3[Fe(CN)_6]$ per litre is added and employed. The solution is accordingly approximately 0.001M with respect to ferricyanide. The reduction potential of $[Fe(CN)_6]^{3-}$ to $[Fe(CN)_6]^{4-}$ electrode is given by the equation $E = E^o - 0.05195 \log \frac{[Fe(CN)_6]^{3-}}{[Fe(CN)_6]^{4-}}$

As long as excess of zinc ion remain in solution concentration of $[Fe(CN)_6]^{4-}$ is very small and the potential is very large. As soon as Zn^{2+} are precipitated the next drop of $[Fe(CN)_6]^{4-}$ solution makes a sudden increase in concentration of $[Fe(CN)_6]^{4-}$ ions. Hence there is a sudden decrease in potential. The end point may be detected by any one of the redox indicators. In this determination it is essential to work under uniform condition to get concordant values.

The titration must not be carried out too rapidly and the solution is shaken through out the experiment. Reducing substances and oxidizing agents should not be present.

PREPARATION OF 0.05M $K_4[Fe(CN)_6]$ SOLUTION

Weighed about 2.112g of AR $K_4[Fe(CN)_6]$ and 0.3g of AR $K_3[Fe(CN)_6]$. Dissolved in distilled water and made upto 100ml.

STANDARDISATION OF $K_4[Fe(CN)_6]$

Prepared a standard solution of 0.1M $ZnSO_4$ by weighing approximately 2.6g and dissolving it in 100ml. Pipetted out 20ml of it into a clean conical flask. Added 20ml of 4N sulphuric acid, 1g of AR$(NH_4)_2SO_4$ and diluted to 50ml. Added 3 drops of diphenylamine indicator. Titrated against $K_4[Fe(CN)_6]$ until the colour changes from blue to pale green. Repeated the titration for concordant values.

PROCEDURE

Pipetted out 40ml of made up solution into a 250ml beaker and added 5ml of concentrated hydrochloric acid, diluted to 100ml, heated to boiling and passed H_2S gas through the hot solution till the precipitate settles readily. Filtered through Whatmann 41 filter paper always covering the precipitate with the solution. Collected the filtrate and expelled all H_2S. Reduced the volume to about 50ml. Titrated this solution against $K_4[Fe(CN)_6]$ as in the case of standardization of zinc. Perfomed a duplicate.

RESULT

1. Amount of copper present in the whole of the given solution =
2. Amount of zinc present in the whole of the given solution =

ESTIMATION OF IRON

Given iron-nickel mixture solution Vs standard Potassium Dichromate

S. No.	Volume of given solution (ml)	Burette reading (ml)		Volume of $K_2Cr_2O_7$ (ml)	Indicator
		Initial	Final		
					Diphenylamine

Weight of $K_2Cr_2O_7$ in 250ml =

Strength of $K_2Cr_2O_7$ =

=

Volume of $K_2Cr_2O_7$ =

Volume of the given solution =

Strength of the given solution =

=

Weight / litre of iron = 55.85 x

=

Amount of iron present in the whole of the given solution =

=

Volume of $K_2Cr_2O_7$ =

Volume of the given solution =

Strength of the given solution =

=

Weight / litre of iron =

=

Amount of iron present in the whole of the given solution =

=

Ex. No. 8

ESTIMATION OF IRON AND NICKEL

AIM

To estimate the amount of iron and nickel present in the whole of the given solution.

PRINCIPLE

Iron is estimated volumetrically and nickel is estimated gravimetrically after the separation of iron as hydroxide.

ESTIMATION OF IRON

Prepared a standard solution of potassium dichromate in 250ml standard flask by weighing about 0.6g of potassium dichromate. Made up the given solution to 250ml. Pipetted out 40ml of this into a conical flask. Added 10ml of concentrated hydrochloric acid and heated to boiling and to the hot solution added stannous chloride drop by drop till the colour becomes pale, added two drops in excess. Cooled the solution well and added 3ml of mercuric chloride, till a silky white precipitate is obtained. If a black precipitate is formed or a precipitate is not seen, the reduction is incomplete and so the solution is discarded. Added 20ml of mixture of sulphuric acid and phosphoric acid and 3 drops of diphenylamine indicator. Titrated it against standard potassium dichromate. The end point is the appearance of bluish violet colour. Repeated the titration for concordant values.

ESTIMATION OF NICKEL

Crucible : A

Weight of crucible and $Ni(DMG)_2$ =

Weight of empty crucible =

Weight of $Ni(DMG)_2$ =

288.93g $Ni(DMG)_2$ contains 58.71g of Ni

g of $Ni(DMG)_2$ contains =

=

Weight of nickel present in the whole
of the given solution =

=

Crucible : B

Weight of crucible and $Ni(DMG)_2$ =

Weight of empty crucible =

Weight of $Ni(DMG)_2$ =

288.93g $Ni(DMG)_2$ contains 58.71g of Ni

g of $Ni(DMG)_2$ contains =

=

Weight of nickel present in the whole
of the given solution =

=

ESTIMATION OF NICKEL

Pipetted out 40ml of the made up solution into a 250ml beaker, diluted to 50ml, added about 5g of ammonium chloride solid and heated to boiling. Added 1:1 ammonia until a precipitate was obtained and then added a slight excess. Heated for few minutes and allowed the precipitate to settle. Filtered through Whatmann 41 filterpaper. Then collected the filtrate in a 400ml beaker. Washed the precipitate with 1:1 ammonia solution. Replaced the beaker by beaker in which the precipitation was done. Dissolved the precipitate in a minimum amount of warm dilute hydrochloric acid. Precipitated iron by adding ammonium chloride and ammonia. Filtered through the same filter paper and collected the filtrate along with the first filtrate in the same 400ml beaker. Washed the precipitate thoroughly with hot water containing a small amount of NH_4NO_3 and few drops of ammonia. Evaporated the combined filtrate and reduced the volume to about 100ml. Neutralised the solution and then made the solution slightly acidic by adding few drops of 1:1 dilute HCl, heated to about 70°C. Added a slight excess of 1% DMG and immediately added dilute NH_3 solution dropwise directly into the solution and not along the walls of the beaker with constant stirring until precipitation takes place and there is a slight excess. (Allowed the precipitate to stand over the steam bath to settle for half an hour). Filtered it through previously weighed G_3 sintered crucible and washed the precipitate with cold water until free from chloride. A duplicate is also performed.

RESULT

1. Amount of iron present in the whole of the given solution

   = (i)

   (ii)

2. Amount of nickel present in the whole of the given solution

   = (i)

   (ii)

S. No.	Volume in ml	Optical Density
1.	5	
2.	10	
3.	15	
4.	20	
5.	25	
6.	Unknown 10ml	
7.	Unknown 20ml	

Weight of $CuSO_4 . 5H_2O$ in 100ml =

Weight of copper sulphate in 1ml =

=

Amount of $CuSO_4 . 5H_2O$ in 5 times diluted solutions =

=

249.65 g of $CuSO_4$ contains 63.55 of Cu.

∴ Weight of copper in 1ml =

=

=

a. 10ml of unknown solution ≡ ml of standard solution

Weight of copper in 10ml = =

Weight of copper in 100ml = =

b. 20ml of unknown solution ≡ ml of standard solution

Weight of copper in 20ml = =

Weight of copper in 100ml = =

COLORIMETRIC ESTIAMTIONS

Ex. No. 1 ESTIMATION OF COPPER

AIM

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

THEORY

The amount of copper present in dilute solution can be estimated from the intensity of the reddish brown colour produced by reaction with 1% potassium ferrocyanide. The method is rendered more sensitive in the presence of potassium nitrite, ammonium chloride and ammonium nitrate. In their presence this method will show one part of copper in 2.5 million parts of the solution.

PREPARATION OF STANDARD SOLUTION

0.4080g of copper sulphate pentahydrate was weighed, transferred into a standard flask and made upto 100ml.

PROCEDURE

Buretted out 20ml of standard solution into a 100ml standard flask and madeup to the mark.

From the 5 times diluted standard solution 5ml, 10ml, 15ml, 20ml and 25ml are taken, added 3cc of potassium ferrocyanide and made upto 100ml. The corresponding percentage transmittance are found out. The given unknown solution was made upto 100ml. From this 10 and 20ml are taken and added 3ml of ferrocyanide and made upto 100ml. The optical density values are noted. A plot of concentration Vs optical density was drawn. The concentration of the given solution was calculated from the graph and hence the amount of copper present in the whole of the given solution was calculated.

RESULT

The amount of copper present in the whole of the given solution is

(a) (b)

S. No.	**Volume in ml**	**Optical Density**
1.	1	
2.	2	
3.	3	
4.	4	
5.	5	
6.	Unknown 2ml	
7.	Unknown 4ml	

Weight of Ferric alum in 100ml =

Weight of Ferric alum in 1ml =

=

Weight of ferric alum solution in 10 times diluted solutions =

=

482.2 g of ferric alum contains 55.85 g of ferric iron.

g of ferric alum contains =

=

= mg of ferric iron

Ex. No. 2 ESTIMATION OF FERRIC IRON

AIM

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

PRINCIPLE

Ferric iron reacts with thiocyanate to given an intense red coloured compound which remains in solution whereas ferrous ion does not react. The colour is usually assumed to be due to ferric thiocyanate. Depending upon the thiocyanate concentration a series of complexes can be formulated as $[Fe(SCN)n]^{3-n}$ where n can be 1-6. At low thiocyanate concentration the predominant species is $[Fe(SCN)_6]^{3-}$. In the colorimetric estimation large excess of thiocyanate should be used because it increases the intensity and also the stability of the colour.

$$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H^+$$

Strong acids like sulphuric acid is not recommended because sulphate ion has certain tendency to form complex with ferric ion. Mercurous and stannous salts should be converted to mercuric and stannic form. Otherwise the colour is destroyed. The phosphate ion, arsenate ion, fluoride ion, oxalate ion, tartarate ion interfere because they form firmly stable isotopes with ferric ion. When large quantities of interfering substances are present it is usually best to proceed in ether of the following way. The ferric thiocyanate is extracted with pure ether or with a mixture of amyl alcohol and pure ether and the organic layer was employed for colour comparison.

a. 2ml of unknown solution ≡ ml of standard solution

Weight of iron in 2ml of unknown solution =

Weight of iron in 100ml = =

b. 4ml of unknown solution ≡ ml of standard solution

Weight of iron in 4ml of unknown solution = =

Weight of iron in 100ml = =

PREPARATION OF STANDARD SOLUTION

About 0.85g of ferric alum was weighed , added 5ml of freshly prepared 4N H_2SO_4 and transferred to a 100ml standard flask and made upto the mark. 10ml of the standard solution was buretted out into a 100ml standard flask and made upto the mark. From the diluted solution 1,2,3,4 and 5ml are taken and added 5ml of 20% of ammonium thiocyanate and 5ml of 4N HNO_3 and made upto 100ml. The percentage transmittance was found out. The given unknown solution was made upto 100ml. From this 2 ml and 4ml are taken and added the same amount of reagents as for the standard and made upto 100ml. The plot of concentration versus log T was drawn. From the graph the concentration of the unknown and hence the amount of iron present can be calculated.

RESULT

Amount of ferric ion present in the whole of the given solution is

(a) (b)

S. No.	Volume in ml	Optical Density
1.	5	
2.	8	
3.	11	
4.	17	
5.	21	
6.	Unknown 20ml	
7.	Unknown 30ml	

Weight of ferrous Ammonium sulphate in 250ml =

Weight of ferrous Ammonium sulphate in 1ml =

Weight of ferric Ammonium sulphate in
10 times diluted solution =

=

392.14 g of ferrous Ammounium Sulphate contains 55.85 g of ferrous iron.

g of ferrous Ammonium Sulphate contains =

=

Ex. No. 3 ESTIMATION OF FERROUS IRON

AIM

To estimate the amount of Iron(II) present in the whole of the given solution.

PRINCIPLE

Ferric ion reacts with thiocyanate to give an intense red compound which remains in solution where as ferrous ion does not react. The colour is usually assumed to be due to ferric thiocyanate. Depending upon the thiocyanate concentration a series of complexes can be got. The complexes are red and formulated as $[Fe(SCN)_n]^{3-n}$ where n can be 1-6. At low thiocyanate concentration the predominant species is $[Fe(SCN)_2]^+$. At 0.1N thiocyanate concentration predominant species is $[Fe(SCN)_2]^+$ and at very high thiocyanate concentration the species is $[Fe(SCN)_6]^{3-}$. In the colorimetric estimation large excess of thiocyanate should be used. Because it increases the intensity and also the stability of colour. Strong acids should be present to suppress the hydrolysis.

$$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H^+$$

Sulphuric acid is not recommended, because sulphate ion has the tendency to form complex with ferric ion. Mercurous and stannous salts if present should be converted to mercuric and stannic form. Otherwise the colour is destroyed. Phosphate ion, arsenate ion, fluoride ion, oxalate ion and tartarate ion interfere because they form firmly stable complexes with ferric iron. When large quantities of interfering substances are present, it is usually best to proceed either with a mixture of amyl alcohol and pure ether and organic layer was employed for colour comparison.

PREPARATION OF STANDARD SOLUTION

About 0.5g of ferrous ammonium sulphate was weighed and transferred into a 250ml standard flask and made upto the mark. Before making up added 5ml of sulphuric acid. Buretted out 25ml of standard solution into a clean beaker. Added 10ml of 1:1 nitric acid, heated for oxidation, cooled and made upto 250ml in a standard flask.

a. 20ml of unknown solution ≡ ml of 10 times diluted standard solution

Weight of iron in 20ml of unknown solution =

=

Weight of iron present in the whole of the given solution =

=

b. 30ml of unknown solution ≡ ml of 10 times diluted standard solution

Weight of iron in 30ml of unknown solution =

=

Weight of iron present in the whole of the given solution =

=

PROCEDURE

From the oxidized standard solution, 5,8,11,17, 21ml were taken and added 5ml of 20% ammonium thiocyanate and 5ml of 4N nitric acid and made up to the mark in a 100ml standard flask. The corresponding percentage transmittance were found out. The given unknown solution was made upto 100ml and 10ml is oxidized as earlier and made upto 100ml. From this 20ml and 30ml are taken and added the same amount of reagents and made upto 100ml. The 'T' values are noted. A plot of log T vs concentration was drawn. From the graph the concentration of unknown solution is noted at 470nm and hence the amount of iron present in the whole of the given solution was calculated.

RESULT

The amount of iron present in the whole of the given solution is

a)

b)

S. No.	Volume in ml	Optical Density
1.	10	
2.	15	
3.	20	
4.	25	
5.	30	
6.	Unknown 10ml	
7.	Unknown 20ml	

Weight of nickel ammonium sulphate in 100ml =

Weight of nickel ammonium sulphate in 1ml =

Weight of nickel ammonium sulphate in 1ml of 100 times diluted solution =

=

394.97 g of nickel ammonium sulphate contains 58.7 g of nickel.

g of nickel ammonium sulphate contains =

=

Ex. No. 4 ESTIMATION OF NICKEL

AIM

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

THEORY

When dimethylglyoxime is added to an alkaline solution of nickel ion which has been treated with an oxidizing agent, a red coloration is obtained. This reaction is very sensitive one. The red soluble complex contains nickel in a higher oxidation state probably +4. In the gravimetric estimation of nickel in ammoniacal solution, nickel forms a complex in a divalent state. Comparison of colours should be complete within 10 minutes. Since the colour slowly increases and finally decreases, the trace of iron present does not interfere but if present in large quantities it should be eliminated. Cobalt and copper also will give colour with this reagent but the interference due to them can be eliminated by the addition of KCN so that they are kept in solution as cyano compounds.

REAGENTS

Saturated solution of bromine water, 1:1 ammonium hydroxide, 1% dimethyl Glyoxime in rectified spirit.

PREPARATION OF STANDARD SOLUTION

About 0.673g of nickel ammonium sulphate was weighed, transferred into a 100ml standard flask and made upto the mark. The solution was then diluted 100 times.

a. 10ml of 10 times diluted unknown solution ≡ ml of 10 times diluted standard solution

Weight of nickel present in 10ml of unknown solution =

=

Weight of nickel present in the whole of the given solution =

=

b. 20ml of 10 times diluted unknown solution = ml of 10 times diluted standard solution

Weight of nickel present in 10ml of unknown solution =

=

Weight of nickel present in the whole of the given solution =

=

PROCEDURE

From the above solution 10, 15, 20, 25, 30ml were taken and added 2ml of bromine water, 1ml of dimethyl glyoxime, 2ml of ammonium hydroxide were added and made upto 100ml. The corresponding percentage transmittance values for these solutions were found out. The given solution was made upto 100ml. 10ml of this solution was pipetted out into a 100ml standard flask and made upto the mark. From this 10ml and 20ml are taken and added the same amount of reagents and made upto 100ml. The T-values for these solutions were found out at 470nm. A plot of concentration versus log O-D was drawn. From the graph the concentration of the given solution and hence the amount of nickel present in the whole of the given solution can be calculated.

RESULT

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

a)

b)

S. No.	Volume in ml	Optical Density
1.	5	
2.	10	
3.	15	
4.	20	
5.	25	
6.	Unknown 10ml	
7.	Unknown 20ml	

Weight of manganese sulphate present in 250ml =

Weight of manganese sulphate present in 1ml =

=

Weight of manganese sulphate in 10 times diluted solution =

=

169.01 g of manganese sulphate contains 54.95 g of manganese.

g of manganese sulphate contains =

=

=

Ex. No. 5 ESTIMATION OF MANGANESE

AIM

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

PRINCIPLE

Amount of manganese can be determined colorimetrically by oxidation to permanganic acid. The oxidizing agents commonly used are aluminium sulphate, concentrated phosphoric acid in nitric acid in the presence of little silver nitrate as catalyst and potassium periodate. The latter is widely used in non acid solutions. Periodate oxidizes Mn^{2+} to permanganic acid quantitatively.

$$2Mn^{2+} + 5IO_4^- + 3H_2O \rightarrow 2MnO_4^- + 5IO_3^- + 6H^+$$

The merits of periodate method are

i. The concentration of acid has little effect and may be varied within wide limits.

ii. The boiling may be prolonged beyond the time necessary to oxidize manganese without dentriment dentriment effect.

iii. Permanganic acid solution can be kept for several months unchanged even if excess of potassium periodate is present.

a. 10ml of 10 times diluted unknown solution = ml of 10 times diluted standard solution

Weight of manganese present in 10ml of unknown solution =

=

Weight of manganese present in the whole of the given solution =

=

b. 20ml of 10 times diluted unknown solution = ml of 10 times diluted standard solution

Weight of manganese present in 20ml of unknown solution =

=

Weight of manganese present in the whole of the given solution =

=

PREPARTION OF STANDARD SOLUTION

0.3755g of manganese sulphate was made upto 250ml in a standard flask. Pipetted out 10ml of the solution into a beaker and added 5ml of syrupy phosphoric acid and boiled the solution. To the hot solution added 0.5g of KIO_4 and boiled for a minute. Kept hot for 10 minutes cooled and made upto 100ml.

PROCEDURE

From the above solution 5ml, 10ml, 15ml, 20ml, 25ml of standard solution were taken and each one was made upto 100ml and corresponding percentage transmittance was determined. The unknown solution was diluted 10 times. From that 10ml and 20ml of the solution were taken oxidized as before made upto 100ml and corresponding percentage transmittance was determined at 520nm. A plot of T Vs concentration was drawn. From the graph the concentration of unknown solution and hence the amount of manganese present in the whole of the given solution was determined.

RESULT

The amount of manganese present in the whole of the given solution is

a)

b)