Group: A (2) Gravimetric Estimation for Single Constituents

[1] Gravimetric estimation Ni as $[Ni(DMG)_2]$.

Aim: To determine the amount of Nickel from given sample by Gravimetrically.

Requirement: Stock solution of Nickel ammonium sulphate; 1% DMG; 1:1 NH4OH solution; Methyl red, Distilled water.

Reaction: Ni2++2DMG →NH4OH Ni(DMG)2↓

Procedure: Prepare 250 mL of the given solution using a 250 mL volumetric flask. Take the 50ml solution from 250ml solution in 500ml beaker. Add dilute HCl to make the solution slightly acidic. To this solution add 25ml distilled water and 1-2 drops of Methyl red. Then add 25-30ml of 1% DMG with constant stirring. Now go on adding 1:1 NH4OH solution drop wise till the solution become alkaline (Yellow colour). Digest the ppt's on water bath for about 20 minutes. Then test for complete precipitation by adding few drops of DMG. Filtrate the ppts in filter paper, which are equal weighed. Wash the ppts with hot water. Collect the filtrate and washing in beaker. Now dry the ppts at 110 C in an electric oven for about an hour. Cool the ppts and weight.

Observation:

1. Weight of precipitate with filter paper: _____ gm
2. Weight of empty filter paper: _____ gm
3. Weight of $[Ni(DMG)_2]$:_____ gm

Calculation:

$Ni[C_8H_{14}N_4O_4]$ = Ni

288.71 gm of complex = 58.7 gm Ni

$$\_\_\_\_\_ \text{ gm of ppts} = \frac{\_\_\_\_\_ \times 58.7}{288.71} = \_\_\_\_\_ \; gm\ Ni$$

Amount of Ni in given (250ml) solution:

$$= \frac{250 \times \_\_\_\_}{50} = \_\_\_\_\_ gm\ Ni.$$

[2] Gravimetric Estimation of Zn as Zinc Ammonium Phosphate

Aim: To estimate the amount of Zinc as $(NH_4)ZnPO_4$ in the given solution of zinc sulphate containing.

Chemicals: Zinc salt (zinc sulphate), diammonium hydrogen phosphate, H2S gas, HCl, H2SO4.
Apparatus: Beaker, conical flask, funnel, Whatman 41, glass rod with policemen, oven.
Reaction:

Zn2+ (aq) + (NH4)2HPO4(aq) = NH4ZnPO4(s)↓+ NH4 + (aq) + H+ (aq)

Procedure:

Prepare 250 mL of the given solution using a 250 mL volumetric flask Pipette out 50 mL of the aqueous solution of Zn ions in a 500 mL beaker. Heat the solution. To this hot solution add 2 g of NH4Cl and few drops of methyl red. Add 1:1 NH3 solution dropwise till the solution becomes yellow and heat it nearly to boiling. Add 25% diammonium hydrogen phosphate till the precipitation of Zn is complete. (Check for complete precipitation). Filter the precipitate through a Whatman filter paper No. 41, wash with cold water. Cool the ppts, dry it in oven and weigh the ppt's.

Observation:

1. Weight of precipitate with filter paper: _____ gm
2. Weight of empty filter paper: _____ gm
3. Weight of $(NH_4)ZnPO_4$:_____ gm

Calculation:

$(NH_4)ZnPO_4$ = Zn

178.4 gm of $(NH_4)ZnPO_4$ = 65.38 gm Zn

$$\_\_\_\_\_ \text{ gm of ppts} = \frac{\_\_\_\_\_ \times 65.38}{178.4} = \_\_\_\_\_ \; gm\ Zn$$

Amount of Zn in given (250ml) solution:

$$= \frac{250 \times \_\_\_\_}{50} = \_\_\_\_\_ gm\ Zn.$$

[3] Gravimetric Estimation of Al as Al2O3

Aim: To estimate the amount of Al as Al_2O_3 in the given solution of Aluminium sulphate.

Chemicals: aluminium sulphate, ammonium chloride, ammonium hydroxide. Apparatus: Beaker, funnel, Whatman 41, glass rod with policemen, water bath, silica crucible.

Reactions:

Al3+ (aq) + 3OH- (aq) =Al(OH)3 (s) ↓

2Al(OH)3(s)) + Heat = Al_2O_3 + 3H2O

Procedure:

Prepare 250 mL of the given solution using a 250 mL volumetric flask. Pipette out 50 mL of the solution in a 250 mL beaker and add 2 drops of methyl red indicator. Add 2 g of NH4Cl and warm the solution. Add 6M NH4OH dropwise till solution turns yellow (white gelatinous precipitate) and digest the precipitate on water bath for 30 min. After 30 min, cool the solution and add 2-3 drops of of NH4OH solution along the sides of the beaker to check complete precipitation of Al. Collect the precipitate on a Whatman 41 filter paper and wash with 1% NH4NO3 distilled water until the filtrate is free from SO_4^{2-} ions. Carefully transfer the precipitate with the filter paper in a silica crucible and heat it on a flame. Once the paper is charged, ignite the precipitate to get Al_2O_3. Cool and weigh the crucible containing the precipitate.

Observation:

1. Weight of precipitate with crucible: _____ gm
2. Weight of empty crucible: _____ gm
3. Weight of Al_2O_3 (ppts):_____ gm

Calculation:

Al_2O_3 = 2Al

101.92 gm of Al_2O_3 = 53.96 gm Al

$$\_\_\_\_\_ \text{ gm of ppts} = \frac{\_\_\_\_\_ \times 53.96}{101.92} = \_\_\_\_\_ \; gm\ Al$$

Amount of Al in given (250ml) solution:

$$= \frac{250 \times \_\_\_\_}{50} = \_\_\_\_\_ gm\ Al.$$

[4] Gravimetric Estimation of Fe as Fe2O3

Aim: To estimate the amount of Fe as Fe_2O_3 in the given solution of Ferrous Ammonium sulphate.

Reaction:

$Fe2+ (aq) + 3OH- (aq) = Fe(OH)3 (s) \downarrow$

$2Fe(OH)3 \Delta Fe2O3+3H2O$

Procedure:

Prepare 250 mL of the given solution using a 250 mL volumetric flask. Take 50ml solution of Fe in 500ml beaker. To this solution add 25ml distilled water and then add 2-3gm of NH4Cl and 1-2 drops of Methyl red. Add 3 ml of Con. HNO3 and heat the solution to boiling. Now discontinue heating. To this solution add 50%. NH4OH solution drop wise with stirring till the colour of solution is becomes yellow. Boil the solution again 1-2 minutes and allow the ppts to settle down. Test the Surprenant liquid for complete precipitation. Filter the ppts on What-man filter paper No-1. Wash the ppt. using 1% NH_4NO_3 or D.W. until the washing free from Cl and SO_4 ions. Allow the filter paper to drain thoroughly. Dry the ppt on a hot air cone. After drying, remove the filter paper from the funnel and fold the edges over ppts to packet and put into a weighed crucible. Heat the ppts for half an hour. Cool the ppts and weight the ppts of **Fe_2O_3**.

Observation:

1. Weight of precipitate with crucible: _____ gm
2. Weight of empty crucible: _____ gm
3. Weight of Fe_2O_3 (ppts):_____ gm

Calculation:

Fe_2O_3 = 2Fe

159.70 gm of Fe_2O_3 = 111.70 gm Fe

$$\_\_\_\_\_ \text{ gm of ppts} = \frac{\_\_\_\_\_ \times 111.7}{189.7} = \_\_\_\_\_ gm\ Fe$$

Amount of Fe in given (250ml) solution:

$$= \frac{250 \times \_\_\_\_}{50} = \_\_\_\_\_ gm\ Fe.$$

[5] Gravimetric Estimation of Barium as Barium Sulphate ($BaSO_4$)

Aim: To estimate the amount of barium (Ba^{2+}) present in a given solution by precipitating it as barium sulphate ($BaSO_4$).

Chemical Reaction:

$BaCl_2(aq)+H_2SO_4(aq)\rightarrow BaSO_4(s)+2HCl(aq$

Reagents Required:

- Barium chloride solution ($BaCl_2$),Dilute sulfuric acid (H_2SO_4),Distilled water, Dilute HCl (for washing)

Procedure:

Prepare 250 mL of the given solution using a 250 mL volumetric flask. Take 50ml of solution in a beaker. Heat the solution to about 70–80°C. Add dilute H_2SO_4 slowly with constant stirring until no further precipitation occurs. Allow the mixture to stand for 1–for complete precipitation and settling. Filter the precipitate through pre-weighed filter paper. Wash the precipitate several times with hot water followed by a small amount of dilute HCl to remove chloride ions (test filtrate with $AgNO_3$ — no white ppt should form). Dry the filter paper with precipitate in an oven. Cool in a desiccator and weigh the crucible with $BaSO_4$.

Observation:

1. Weight of precipitate with crucible: _____ gm
2. Weight of empty crucible: _____ gm
3. Weight of $BaSO_4$. (ppts):_____ gm

Calculation:

$BaSO_4$. = Ba

233.39 gm of $BaSO_4$. = **137.33** gm Ba

$$\_\_\_\_\_ \text{ gm of ppts} = \frac{\_\_\_\_\_ \times 137.33}{233.39} = \_\_\_\_\_ \; gm\ Ba$$

Amount of Ba in given (250ml) solution:

$$= \frac{250 \times \_\_\_\_}{50} = \_\_\_\_\_ gm\ Ba.$$

Group: B (1) Inorganic Preparation

Preparation :[1]

Aim: To prepare Hexathiourea Lead(II) Nitrate.

Reaction:

$Pb(NO_3)_2$ (aq)+6 NH_2CSNH_2 (aq)→[Pb(tu)6](NO3)2(s)

Reagents Required:

Lead nitrate [$Pb(NO_3)_2$], Thiourea [NH_2CSNH_2], Distilled water

Procedure: Dissolve 6 g of $Pb(NO_3)_2$ in about 20 mL of hot distilled water in a beaker. In another beaker, dissolve 9 g of thiourea (NH_2CSNH_2) in 20 mL of hot distilled water. Slowly add the thiourea solution to the lead nitrate solution with constant stirring while warm (~60°C). A white crystalline precipitate of complex forms gradually. Continue stirring for 10–15 minutes and cool the mixture to room temperature, then further cool in an ice bath to enhance crystallization. Filter the precipitate. Wash the solid with a small amount of cold water followed by cold ethanol to remove unreacted thiourea and nitrate ions. Dry the product and weight.

Observation:

1. Weight of precipitate of Hexa thiourea lead nitrate: _____ gm

Calculation:

1. Theoretical yield of complex.

Pb(NO3)2 = Pb(tu)6](NO3)2 2

331.2gm = 787.94 gm

6 gm of Pb(NO3)2 gives $= \frac{6 \times 787.94}{331.2} = 14.274\ gm\ complex$

2. Practical Yield : _____ gm complex.

3. % of Yield

$$= \frac{100 \times \_\_\_\_}{14.274} = \_\_\_\_\_\%$$

Preparation :[2]

Aim: To prepare Tetraamminecupric Sulphate monohydrate, a deep blue coordination complex.

Reactions:

1. Formation of precipitate:
   $CuSO4+2NH3+2H2O \rightarrow Cu(OH)2\downarrow+(NH4)2SO4$
   $Cu(OH)2+4NH3 \rightarrow [Cu(NH3)4]2+H2O$

Materials Required:

Copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) – 2.5 g, Concentrated aqueous ammonia (NH_4OH),Distilled water

Procedure:

Dissolve 2.5 g of $CuSO_4 \cdot 5H_2O$ in minimum quantity of distilled water in a beaker. Add ammonia solution dropwise with constant stirring. A light blue precipitate of $Cu(OH)_2$ will form initially. Continue adding ammonia until the precipitate just dissolves and a deep royal blue solution form. Cool the solution to room temperature and then After crystals form, filter the blue crystals using filter paper. Wash the crystals with a small amount of cold ethanol. Dry the product ane weight.

Observation:

Weight of precipitate of Hexa thiourea lead nitrate: _____ gm

Calculation:

1. Theoretical yield of complex.

$$\mathbf{CuSO_4 \cdot 5H_2O = [Cu(NH3)4]2.H2O}$$

$$\mathbf{249.70\ gm = 245.77\ gm}$$

2.5 gm of $CuSO_4 \cdot 5H_2O$ gives $= \frac{2.5 \times 245.77}{249.70} = 2.460\ gm\ complex$

2. Practical Yield : _____ gm complex.
3. % of Yield

$$= \frac{100 \times \_\_\_\_}{2.460} = \_\_\_\_\_\%$$

Preparation :[3]

Aim: To prepare Mohr's Salt by crystallization from a solution of ferrous sulfate and ammonium sulphate.

Chemical Reaction:
FeSO4+(NH4)2SO4+6H2O→FeSO4·(NH4)2SO4·6H2O

Materials Required:
Ferrous sulfate ($FeSO_4 \cdot 7H_2O$) – ~10 g, Ammonium sulfate ($(NH_4)_2SO_4$) – ~5 g, Dilute sulfuric acid, Distilled water

Procedure:
Dissolve **10 g** of $FeSO_4 \cdot 7H_2O$ in 15 mL of warm distilled water in a beaker. Add a few drops of dilute sulfuric acid to prevent oxidation of Fe^{2+}. In a separate beaker, dissolve **5 g** of $(NH_4)_2SO_4$ in about 15 mL of water. Mix both solutions together with stirring. Add more water if needed to dissolve all solids. Filter the solution if any insoluble matter is present. Evaporate solution until crystallization by boiling. Allow the solution to cool slowly at room temperature, then place in an ice bath to crystallize. After crystallization, filter the light green Mohr's salt crystals, and wash with a small amount of cold water containing a few drops of dilute H_2SO_4. Dry the crystals by pressing between filter paper or keeping in a desiccator.

Observation:

Weight of precipitate of **Mohr's salt** : _____ gm

Calculation:

1. Theoretical yield of complex.

$$\mathbf{FeSO_4 \cdot 7H_2O} = \mathbf{Mohr's\ salt}$$

$$\mathbf{278\ gm} = \mathbf{392.2\ gm}$$

$$10 \text{ gm of } FeSO_4 \cdot 7H_2O \text{ gives} = \frac{10 \times 392.2}{278} = 14.001\ gm\ Mohr's\ Solt$$

2. Practical Yield : _____ gm complex.
3. % of Yield

$$= \frac{100 \times \_\_\_\_}{14} = \_\_\_\_\_\%$$

Preparation :[4]

Aim: To prepare Potash Alum crystals by crystallizing a mixture of potassium sulphate (K_2SO_4) and aluminium sulphate ($Al_2(SO_4)_3$) from hot aqueous solution.

Chemical Reaction:

K2SO4+Al2(SO4)3+24H2O→2KAl(SO4)2·12H2O

Materials Required:

Potassium sulfate (K_2SO_4) – ~5 g, Aluminium sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$) – ~12 g, Dilute sulfuric acid – few drops, Distilled water

Procedure:

Dissolve 5 g of K_2SO_4 in about 20 mL of hot distilled water in a beaker. Dissolve 12 g of $Al_2(SO_4)_3 \cdot 18H_2O$ in about 20 mL of hot water in a separate beaker. Add 1–2 mL of dilute H_2SO_4 to prevent hydrolysis of aluminium ions. Mix both hot solutions in a larger beaker with constant stirring. Heat gently if necessary to get a clear solution, then filter to remove any impurities. Evaporate solution until crystallization by boiling. Allow the solution to cool slowly to room temperature, then place in an ice bath to induce crystallization. After crystals form, filter the potash alum crystals, wash with a small amount of cold water, and dry between filter paper or in a desiccator.

Observation:

Weight of precipitate of **Potash Alum** : _____ gm

Calculation:

1. Theoretical yield of complex.

K_2SO_4 = Potash Alum 2KAl(SO4)2·12H2O

174.27 gm = 732.6 gm

$$\textbf{05 gm of } FeSO_4 \cdot 7H_2O \textbf{ gives} = \frac{05 \times 732.6}{174.27} = 21\ gm\ Ptash\ ALum$$

2. Practical Yield : _____ gm complex.
3. % of Yield

$$= \frac{100 \times \_\_\_\_}{21} = \_\_\_\_\_\%$$

Preparation :[5]

Aim: Preparation of CuCl from $CuSO_4$ using Vitamin C (L-ascorbic acid)

Requirement: CuSO4, L-ascorbic acid ($C_6H_8O_6$), NaCl, dil HCl

Equation:

$$CuSO_4 + 2NaCl \rightarrow CuCl_2 + Na_2SO_4$$

$$2CuCl_2 + C_6H_8O_6 \xrightarrow[\text{reaction}]{\text{Redox}} 2CuCl + C_6H_6O_6 + 2HCl$$

$C_6H_8O_6 + 2\ Cu^{2+} \rightarrow C_6H_6O_6$ (dehydroascorbic acid) + 2 Cu^{+} + 2 H^{+}
2 Cu^{+} + 2 $Cl^{-} \rightarrow$ 2 CuCl (s)

Procedure:

Dissolve 12.5 g $CuSO_4 \cdot 5H_2O$ with stirring. The solution should be blue. Add 5.84 g NaCl and stir until fully dissolved. The solution remains blue-green. Acidify slightly. If the solution is neutral/alkaline, add 2–5 mL of dil. HCl to make the medium mildly acidic. Do not over-acidify — only slight acidity is needed to avoid $Cu(OH)_2$ formation. Cool the solution Place the beaker in an ice bath. Prepare ascorbic solution. In a small beaker, dissolve 4.4 g L-ascorbic acid in ~25 mL cold water. Use fresh ascorbic acid (store in dark). Add ascorbic acid slowly with stirring and the solution cooled, add the ascorbic solution slowly (dropwise or in a thin stream) to the $CuSO_4$/NaCl solution over 5–10 minutes. Stir continuously. Blue color will fade as Cu^{2+} is reduced; a white/cream precipitate of CuCl should appear. After addition, stir the mixture for another 10–20 minutes while keeping it cold. If blue color persists, add a small additional portion of ascorbic acid solution until blue disappears. Allow the slurry to settle for 10–20 minutes. Filter and wash quickly. Wash the solid briefly with cold distilled water (small portions) to remove soluble sulphate and sodium salts. Transfer the wet CuCl in a desiccator. If oven-drying, keep temperature low (≤ 40 °C) and exclude air.

Observation:

Weight of precipitate of CuCl : _____ gm

Calculation:

1. Theoretical yield of complex.

CuSO4 5H2O = CuCl

249.69 gm = 98.99 gm

12.5 gm of $CuSO_4 \cdot 5H_2O$ gives $= \frac{12.5 \times 98.99}{249.69} =$ _____ ***gm CuCl***

2. Practical Yield : _____ gm complex.
3. % of Yield

$$= \frac{100 \times P.Y}{T.Y} =$$ _____%