

Lab8

Gravimetric Analysis

Gravimetry encompasses all techniques (method) in which the signal is a mass or change in mass, There are many type of gravimetric method :-

- 1- precipitation gravimetry method
- 2- electro gravimetry method
- 3- volatilization gravimetry method
- 4- particulate gravimetry method

The most methods is the first one(precipitation gravimetry) which is based on the formation of an insoluble compound following the addition of a precipitating reagent, or **precipitant**, to a solution of the analyte .

Experiment N0. (8)

Determination of Nickle by Di methyl glyoxime (DMG H₂)

Dimethylglyoxime is a chemical compound described by formula $\text{CH}_3(\text{NOH})\text{C}(\text{NOH})\text{CH}_3$. is a solid , colorless ,derivative of the diketone diacetyl (also known as 2,3- butane Dione) . DMG H₂ is used in the analysis of nickel . Its coordination complexes are of theoretical interest as models for enzymes and as catalysts . Many related ligand can be prepared from other diketones .

The Requirement Materials

- 1- $\text{NiSO}_4(\text{NH}_4)_2 \cdot 6\text{H}_2\text{O}$ (sample).
- 2-(DMG H₂) solution 1% .
- 3- NH_4OH (diluted solution)
- 4- HCl (diluted solution) .

Procedure :

- 1- Weigh about (0.3-0.4) gm from $\text{NiSO}_4(\text{NH}_4)_2\cdot 6\text{H}_2\text{O}$, dissolve with D.W in a beaker .
- 2- Add to the mixture (5ml) from diluted hydrochloric acid (1:1) then diluted the mixture with D.W to(200 ml)
- 3- Heat the mixture to 80°C on a water bath or a hot plat .
- 4- Add excess 20 ml of di methyl glyoxime , then add NH_4OH (1:1) with good stirring until red precipitate appear . The odor of NH_3 indicates that the medium is alkaline .
- 5- Leave the precipitate of Ni – dimethyl glyoximate for half an hour to complete the precipitate.
- 6- Filter the mixture by using Clean , dry and weigh filter paper .
- 7- Wash the precipitate with hot distilled water several times .
- 8- Dry the precipitate with crucible in the oven at $110 - 120^\circ\text{C}$ for 20 min, then weigh it .
- 9- Calculate the % Ni in the sample .

$$\begin{aligned} \text{Gf.}_{\text{Ni}^{+2}} &= \text{A. wt.}_{\text{Ni}^{+2}} / \text{M. wt.}_{\text{Ni}(\text{C}_4\text{H}_7\text{O}_2\text{N}_2)_2} \\ &= 0.20319 \end{aligned}$$

$$\% \text{ Ni} = (\text{wt.ppt} * \text{Gf}_{\text{Ni}^{+2}} / \text{wt. sample}) * 100$$