CHM 213

(Analytical Chemistry)

Topic: Gravimetric Analysis

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Practical procedure for gravimetric analysis

1. Sample preparation
2. Precipitation
3. Digestion of precipitate
4. Filtration
5.Drying and ignition
6. Weighing
7. Calculation

Calculations involving Gravimetric factor

- The weight of the element or ion to be determined is calculated from the weight of the precipitate with the aid of a gravimetric factor (GF).

$$GF = \frac{\text{Formula wt. (Subs. sought)}}{\text{Formula wt. (Subs. Weighed)}}$$

The expression for a gravimetric factor may be derived from simple proportion.

For, sulphur to barium sulphate,

$$\frac{S\ (grams)}{BaSO_4\ (grams)} = \frac{S\ (form\ wt)}{BaSO_4\ (form\ wt)}$$

If BaSO4 (grams) is known and wish to calculate S (grams), solving this equation gives

$$S\ (grams) = BaSO_4\ (grams) \times \frac{S\ (form\ wt.)}{BaSO_4\ (form\ wt.)}$$

Example 1: Derive an expression for the gravimetric factor required to calculate the weight of magnesium in a precipitate of magnesium pyrophosphate $Mg_2P_2O_7$.

Solution: $\frac{\text{Mg } (grams)}{Mg_2P_2O_7\ (grams)} = \frac{\text{Mg } (form\ wt)}{Mg_2P_2O_7\ (form\ wt)}$

Solving for the weight of Mg

$$\text{Mg (grams)} = Mg_2P_2O_7\ (\text{grams}) \times \frac{2\text{Mg (form wt)}}{Mg_2P_2O_7\ (form\ wt)}$$

$$GF = \frac{\text{Formula weight of sub. Sought for}}{\text{Formula weight of the substance weighed}} \times \frac{\text{no of moles of sub. Sought for}}{\text{no of moles of sub. Weighed}}$$

OR

$$GF = \frac{\text{Formula weight of Analyte}}{\text{Formula weight of precipitate}} \times \frac{\text{no of moles of Analyte}}{\text{no of moles of precipitate}}$$

$$\% \text{ of Analyte} = \frac{G.F \times \text{Mass of ppt}}{\text{Mass of Sample}}$$

Example 2: A 0.5656g sample contains a bromide salts is treated with excess $AgNO_3$, the AgBr formed is filtered and weighed, yielding 0.7624g of precipitate. Calculate the % bromide in the sample.

Solution:

Applying the formula

$$\frac{\text{Br } (grams)}{\text{AgBr } (grams)} = \frac{\text{Br } (form\ wt)}{\text{AgBr } (form\ wt)}$$

$$\text{Br } (grams) = \text{AgBr } (grams) \times \frac{\text{Br } (form\ wt)}{\text{AgBr } (form\ wt)}$$

Substituting the values

$$\text{Br } (grams) = 0.7624 \times \frac{79.9}{187.7} = 0.3245\text{g of Br}$$

$$\%\text{ of Br in the Sample} = \frac{0.3245}{0.5656} \times 100 = 57\%$$

Classwork

1. Attempt the question on page 36 of CHM 213 material

2. A 0.703g sample of a commercial detergent was ignited at red heat to destroy the organic matter. The residue was taken up in hot HCl which converted the phosphorus to phosphoric acid H_3PO_4. The phosphate was precipitated as $MgNH_4PO_4.6H_2O$ by addition of Mg^{2+} followed by aqueous ammonium (NH_4). After being filtered and washed the precipitate was converted to $Mg_2P_2O_7$ by ignition at 100°C. The residue weighed 0,274g. Calculate the % phosphorus in the sample.

Contamination of precipitates

It involves simultaneous precipitation of materials other than the desired analyte species constitute impurities.

The impurities sets into precipitate in two general ways

1. Co-precipitation
2. Post- precipitation

Types of Co-precipitation

1. Isomorphous Inclusion:

- Compounds with the same type of formula crystallizing in similar geometric forms are said to be isomorphous.
- Lattice dimensions of two isomorphous compounds are about the same, one compound can replace part of the other in the crystal.
- This result in the formation of mixed crystals.
- E.g. $MgNH_4PO_4$ $MgKPO_4$ are isomorphous, the ionic radii of K^+ and $NH4^+$ are the same.

2Occlusion:

- Mechanical entrapment of impurities within the growing crystals.
- The impurities are distributed merely through the ppt, mostly occupying places where the crystal structure of the ppt is imperfect.

Occlusion can be removed by

i. Aging
ii. Recrystallization
iii. Precipitation

3.Surface Adsorption

- Surface phenomenon and is important for ppt with large surface area.

Correction or minimization of surface adsorption

i. Washing of ppt
ii. Reprecipitation or recrystallization
iii. Precipitation from homogenous solution
iv. Digestion of the ppt.