EXPERIMENT: 11

AIM:

EXPLAIN QUANTITATIVE ESTIMATION (PRECIPITATION TITRATION) OF BARIUM SULPHATE

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REQUIREMENTS

A. Glassware & Instruments

- 1. Conical flasks
- 2. Beakers, pipettes
- 3. Filter papers
- 4. Balance with weight box
- 5. Hot air oven
- 6. Burette

B. Chemicals & Reagents

- 1. Methyl red indicator: Dissolve 100 mg methyl red sodium salt in dist. water to prepare 100 ml of solution.
- 2. Hydrochloric acid: 50% (V/V).
- 3. Barium chloride solution:Dissolve 100 gm. BaCl₂.2H₂O in distilled water to prepare 1 liter of solution. Filter the solution through a filter paper before use.
- 4. Silver nitrate: Nitric acid reagent Dissolve 8.5g of AgNO₃ and 0.5 ml conc. HNO₃ in distilled water to prepare 500 ml reagent.
- 5. Sulphate solution or sample water or soil sample etc.

PRINCIPLE

The addition of barium chloride solution precipitates sulphate as barium sulphate in a hydrochloric acid media. The reaction is conducted near the boiling point. The precipitate is filtered and washed to eliminate chlorides, after which it is dried or burned and weighed as BaSO₄.

PROCEDURE

- 1. A few drops of methyl red are added to 100 ml of sulphate solution in a conical flask after adjusting the pH to 4.5-5.0 with HCl solution (indicator colour changed to orange).
- 2. The solution is brought to a boil, and warm BaCl₂ solution is steadily added in excess until precipitation is complete.
- 3. The precipitate is heated between 80 and 90 degrees Celsius for at least two hours.
- 4. The precipitate is filtered through filter paper (What-man No. 42) and rinsed with warm deionized water until it is chloride-free. This is determined by testing the cleaned material with a solution of AgNO₃.
- 5. The precipitate is dried on filter paper and burned at 800°C for one hour in a crucible. Then, the substance was cooled and weighed.

CALCULATIONS

Observations

- 1. Sulphate solution turns orange when pH reaches 4.5-5.
- 2. On addition of BaCl₂, cloudiness is formed and later a white ppt of BaSO₄ is formed beneath the solution.
- 3. On filtration, a white precipitate of BaSO₄ is left on the filter paper and the filtrate obtained contains white gelatinous precipitate suspected to be BaCl₂ and lost BaSO₄ precipitate.

Mass of precipitate (BaSO₄) = mass of (dry filter paper + BaSO₄) - mass of filter paper Weight of SO₄²⁻ = weight of BaSO₄ * $\frac{a (gram formula weight of SO₄)}{b (gram formula weight of BaSO₄)}$

$$BaSO_4 \rightarrow Ba^{2+} + SO_4^{2-}$$

Thus a=1 and b=1

Weight of SO_4^{2-} = weight of ppt (BaSO4) * gravimetric factor

Percentage purity =
$$\frac{wt.of\ ppt*GF*100}{wt.of\ analyte}$$

RESULT

The anticipated aim of the experiment was explicitly achieved. Thus, the amount and concentration of the sulphate was determined through gravimetry.