211 ANALYTICAL CHEMISTRY

Experiment No. 1

GRAVIMETRIC ANALYSIS

Gravimetric methods are based on the determining the mass of a pure compound to which the analyte is chemically related. Several quantitative methods are based on the mass measurements which are known as precipitation gravimetry, volatilization gravimetry and electrogravimetry.

- In precipitation gravimetry, the analyte is separated from other constituents of a sample as a precipitate. After filtration and other suitable treatment the solid residue of known chemical composition is weighed.

- In volatilization gravimetry, the analyte is separated from other constituents of a sample by conversion to a gas of known chemical composition. Here the analysis is based upon the mass of this volatilized substance or upon the mass of the nonvolatile residue.

- In electrogravimetry, the analyte is separated by deposition on an electrode by an electrical current. The mass of this product then provide a measure of the analyte.

Gravimetric methods have been developed for most inorganic anions and cations, as well as for such neutral species as water, sulfur dioxide, carbon dioxide, and iodine. A variety of organic substances can also be easily determined gravimetrically. Examples include lactose in milk, nicotine in pesticides, cholesterol in cereals and etc.

GRAVIMETRIC DETERMINATION OF SULFATE IN A SOLUBLE SAMPLE

The analysis of soluble sulfate is based upon its precipitation with barium ion.

$$Ba^{2+}_{(aq)} + SO_4^{2-}{}_{(aq)} \rightarrow BaSO_{4\,(s)}$$

The barium sulfate, $BaSO_4$, which forms as a crystalline precipitate is collected on a suitable filter, washed with water and strongly ignited.

Experimental:

A) Reagents:

1. Barium chloride, $BaCl_2$, solution containing 5 g $BaCl_2.2H_2O$ per liter of solution and allowed to stand at least 24 hours prior to use.
2. 6 M Hydrochloric acid, HCl.

B) Procedure:

1. Take 10 mL sample solutions (usually two solutions, containing sulfate) in 400 mL beakers. Treat each sample solutions individually.
2. Add 50 mL distilled water to each sample solution.
3. Add 4 mL 6 M HCl (Note 1) to each sample solutions and cover the beakers with watch glasses and heat gently till boiling.

4. For each sample heat 100 mL $BaCl_2.2H_2O$ solution nearly to boiling, and add quickly with vigorous stirring to the hot solution of the sample (Note 2).
5. Digest the precipitated $BaSO_4$ at just below the boiling point for 1 to 2 hours (Note 3).
6. Filter the solution while its hot through a fine ashless paper (Note 4).
7. Wash the precipitate remaining in the beaker into the filter with hot distilled water until the beaker is clean.
8. Wash the precipitate on the filter paper with hot water, decantating the washings through the filter.
9. Continue washings until a fresh portion of the filtrate yields no precipitate of silver chloride when acidified with nitric acid and treated with a few drops of the silver nitrate solution (Note 5).
10. Loosen the filter paper in the funnel and allow to drain for few minutes.
11. Place the filter paper in a porcelain crucible that has already been ignited to constant weight.
12. Place the crucible on a triangle on a tripod.
13. Heat the crucible gently with a small flame while continuously changing the point of heating until the moisture had been evaporated and the paper begins to smoke and char (Note 6).
14. Place the crucible into the oven and ignite at 800^oC until no black particles are left (Note 7).
15. Transfer it into a desiccator, and let it cool there for 30 minutes before weighing.
16. Continue ignition at 800^oC to a constant weight. Cool it in the desiccator and reweigh.
17. Report the result of analysis as weight of sulfate in the sample.

Notes:

1- Solubility of barium sulfate at room temperature is 0.3-0.4 mg per 100 g water. Solubility increases when excessive amount of mineral acid is present. For example, the solubilities per 100 g of water is 17.0 mg in 2 M HNO_3 and 10.1 mg in 2 M HCl respectively. On the other hand precipitation should be done in acidic medium. Because in neutral and basic solutions Ba^{2+} ions precipitate with PO_4^{2-} , CO_3^{2-} or OH^- ions present in the solution. Therefore, precipitation is carried on in weak acidic medium (such as 0.05 M acid.) and addition of excess acid is avoided. Precipitation in weak acidic medium provides precipitate which occurs to be in rather large particle size.

2- Use a separate stirring rod for each sample and leave it in the solution throughout the determination.

3- The digested precipitate can be allowed to stand several days without harm.

4- Use of Whatman no 42 paper is recommended.

5- Washing can be tested for Cl^- by collecting a few milliliters in a test tube, acidifying with HNO_3 and adding a few drops of 0.1 M $AgNO_3$. Washing is complete when little or no turbidity is observed.

6- At high temperature barium sulfate may be reduced to BaS by the reaction occurred with C of filter paper.

$$BaSO_{4\,(s)} + 4C_{(s)} \rightarrow BaS_{(s)} + 4CO_{(g)}$$

This reduction can be prevented by burning the filter paper at rather low temperature.

7- If ignition is done at very high temperature barium sulfate may decompose as follows:

$$BaSO_{4\,(s)} \text{ ==== } BaO_{(s)} + SO_{3\,(g)}$$

FE 211 ANALYTICAL CHEMISTRY DATA SHEET

EXPERIMENT NO : 1

GRAVIMETRIC DETERMINATION OF SULFATE IN SOLUBLE SAMPLE

Weight of empty crucible : …….

Weight of crucible + weight of $BaSO_4$: …….

Weight of $BaSO_4$: …….

Submitted by: Submitted to:

Date: