Department Of Medical Instrumentation Techniques Engineering

Lecture six : Gravimetric Analysis

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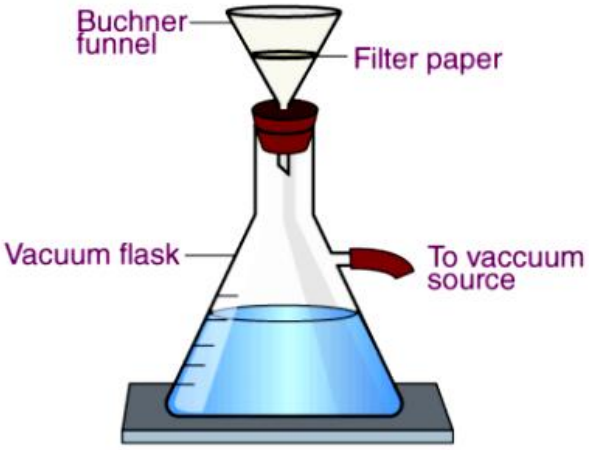
Gravimetric analysis

is one of the most accurate and precise methods of macroquan-titative analysis. In this process the analyte is selectively converted to an insoluble form.

The separated precipitate is dried or ignited, possibly to another form, and is accurately weighed. From the weight of the precipitate and a knowledge of its chemical composition, we can calculate the weight of analyte in the desired form.

The principle of Gravimetric Analysis:

The principle behind the gravimetric analysis is that the mass of an ion in a pure compound and can be determined. Later, used to find the mass percent of the same ion in a known quantity of an impure compound.



Gravimetric Analysis Apparatus

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-How to Perform a Successful Gravimetric Analysis

A successful gravimetric analysis consists of a number of important operations designed to obtain a pure and filterable precipitate suitable for weighing.

-WHAT STEPS ARE NEEDED?

The steps required in a gravimetric analysis, after the sample has been dissolved, can be summarized as follows:

1. Preparation of the solution
2. Precipitation
3. Digestion
4. Filtration
5. Washing
6. Drying
7. Weighing
8. Calculation

Types of Gravimetric Analysis:

There are 4 fundamental types of gravimetric analysis. Of which, there are 2 common types involving changes in the phase of the analyte to separate it from the rest of a mixture, resulting in a change in mass.

1-Volatilization gravimetry

Volatilization Gravimetry involves separating components of our mixture by heating or chemically decomposing the sample.

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2-Precipitation gravimetry

Precipitation Gravimetry uses a precipitation reaction to separate one or more parts of a solution by incorporating it into a solid.

3-Electrogravimetry

Electrogravimetry is a method used to separate and quantify ions of a substance, usually a metal.

4-Thermogravimetric

Thermogravimetric is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature or as a function of time.

-Advantages of Gravimetric Analysis:

If the methods are followed carefully, it provides exceedingly precise analysis. It is used to determine the atomic masses of many elements to six-figure accuracy. It provides a little room for instrumental error and does not require a series of standards for calculation of an unknown.

-Disadvantages of Gravimetric Analysis:

It usually provides only for the analysis of a single element, or a limited group of elements, at a time. Comparing modern dynamic flash combustion coupled with gas chromatography with traditional combustion analysis.

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Example:

Precipitation method for determining calcium in water.
In this technique.

1. An excess of oxalic acid, H2C2O4, is added to an aqueous solution of the sample.
2. Ammonia is then added, which neutralizes the acid and causes essentially all of the calcium in the sample to precipitate as calcium oxalate.
   The reactions are :

$$2NH_3 + H_2C_2O_4 \rightarrow 2NH_4^+ + C_2O_4^{2-}$$
$$Ca^{2+}(aq) + C_2O_4^{2-}(aq) \rightarrow CaC_2O_4(s)$$

3. The CaC2O4 precipitate is filtered using a weighed filtering crucible, then dried and ignited. This process converts the precipitate entirely to calcium oxide.

The reaction is

$$CaC_2O_4(s) \xrightarrow{\Delta} CaO(s) + CO(g) + CO_2(g)$$

4. After cooling, the crucible and precipitate are weighed, and the mass of calcium oxide is determined by subtracting the known mass of the crucible.

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Gravimetric Calculations—How Much Analyte Is There?

The **gravimetric factor** (GF) which represents the weight of analyte per unit weight of precipitate. It is obtained from the ratio of the formula weight of the analyte to that of the precipitate, multiplied by the moles of analyte per mole of precipitate obtained from each mole of analyte,

$$\text{GF} = \frac{\text{fw analyte (g/mol)}}{\text{fw precipitate (g/mol)}} \times \frac{a}{b}(\text{mol analyte/mol precipitate})$$

$$= \text{g analyte/g precipitate}$$

that is,

Example \\ Calculate the grams of analyte per gram of precipitate for the following conversions:

Analyte	Precipitate
P	Ag_3PO_4
K_2HPO_4	Ag_3PO_4
Bi_2S_3	$BaSO_4$

Solution

$$\text{g P/g } Ag_3PO_4 = \frac{\text{at wt P (g/mol)}}{\text{fw } Ag_3PO_4 \text{ (g/mol)}} = \frac{1}{1}(\text{mol P/mol } Ag_3PO_4)$$

$$\text{GF} = \frac{30.97 \text{ (g P/mol)}}{418.58 \text{ (g } Ag_3PO_4\text{/mol)}} \times \frac{1}{1} = 0.07399 \text{ g P/g } Ag_3PO_4$$

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$$\text{g } K_2HPO_4/\text{g } Ag_3PO_4 = \frac{\text{fw } K_2HPO_4 \text{ (g/mol)}}{\text{fw } Ag_3PO_4 \text{ (g/mol)}} \times \frac{1}{1} \text{ (mol } K_2HPO_4/\text{mol } Ag_3PO_4)$$

$$\text{GF} = \frac{174.18 \text{ (g } K_2HPO_4/\text{mol)}}{418.58 \text{ (g } Ag_3PO_4/\text{mol)}} \times \frac{1}{1} = 0.41612 \text{ g } K_2HPO_4/\text{g } Ag_3PO_4$$

$$\text{g } Bi_2S_3/\text{g } BaSO_4 = \frac{\text{fw } Bi_2S_3 \text{ (g/mol)}}{\text{fw } BaSO_4 \text{ (g/mol)}} \times \frac{1}{3} \text{ (mol } Bi_2S_3/\text{mol } BaSO_4)$$

$$\text{GF} = \frac{514.15 \text{ (g } Bi_2S_3/\text{mol)}}{233.40 \text{ (g } BaSO_4/\text{mol)}} \times \frac{1}{3} = 0.73429 \text{ g } Bi_2S_3/\text{g } BaSO_4$$ ■

In gravimetric analysis, we are generally interested in the percent composition by weight of the analyte in the sample, that is,

$$\% \text{ substance sought} = \frac{\text{weight of substance sought (g)}}{\text{weight of sample (g)}} \times 100\%$$

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Home Work :

1-What are the applications of gravimetric analysis?

2-What is the difference between gravimetric and volumetric analysis?