College of science for women
Department of Chemistry

Second Stage

Gravimetric Analysis

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Gravimetric Analysis

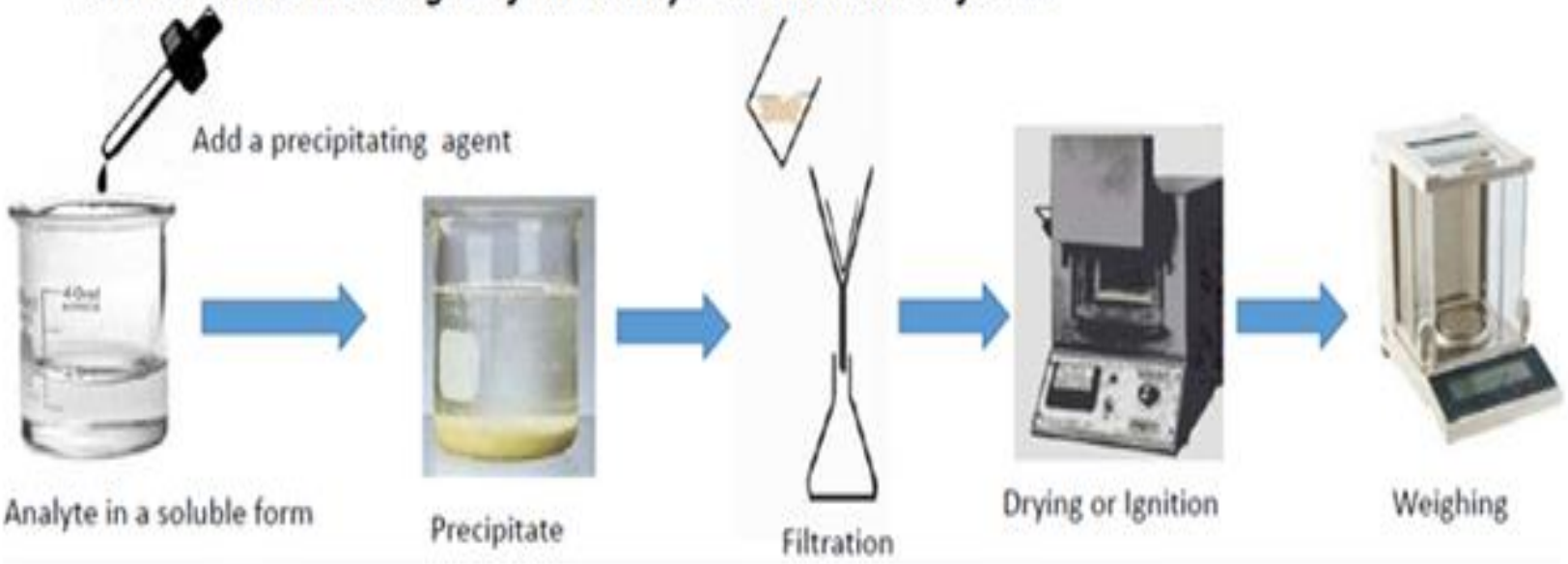
Gravimetric methods – are quantitative methods in which the mass of the analyte or some compound that is chemically related to the analyte is determined.

PRINCIPLE :

Gravimetric analysis is the process of isolating and weighing an element or a definite compound of the element in as pure a form as possible. The element or compound of the element is separated from a weighed portion of the substance being examined. A large portion of the determinations in gravimetric analysis is concerned with the transformation of the element or radical to be determined into a pure stable compound which can be readily converted into a form suitable for weighing. The weight of the element or radical may then be readily calculated from a knowledge of the formula of the compound and the relative atomic masses of the constituent elements

Gravimetric analysis

- Gravimetric analysis is one of the most accurate and precise methods of macro-quantitative analysis.
- The analyte is selectively converted into an insoluble form (precipitate).
- The separated precipitate is then dried or ignited, possibly to another form and is accurately weighed.
- From the weight of the precipitate and knowledge of its chemical composition, *we can calculate the weight of the analyte in the desired form.*



Properties of gravimetric analysis:

1- Tradition method

2- Cheap, easily available apparatus, simple to carry out

3- Slow, especially when accurate results are required

4- Wide range of concentration (ng-kg)

5- No calibration required except for the balance

6- Accurate

- *Gravimetric methods:* The **quantitative methods** that are based on determining the **mass** of a **pure compound** to which the **analyte** is **chemically related**.
- **1- Precipitation gravimetry**: The **analyte** is separated from a solution of the sample as a **precipitate** and is converted to a compound of known composition that can be weighed.
- **2-Volatilization gravimetry**: The analyte is separated from other constituents of a sample by converting it to a gas of known chemical composition that can be weighed.
- **3-Electrogravimetry**: The analyte is separated by deposition on an electrode by an electrical current.

Precipitation gravimetry

The analyte is converted to a **sparingly soluble precipitate** that is then **filtered**, **washed free of impurities** and converted to a product of **known composition** by suitable **heat** treatment and **weighed**.

Ex. for determining the $[Ca^{2+}]$ in water:

$$2NH_{3(aq)} + H_2C_2O_{4(aq)} \rightarrow 2NH^{+}_{4(aq)} + C_2O^{2-}_{4(aq)}$$

$$Ca^{2+} + C_2O^{2-}_{4(aq)} \rightarrow CaC_2O_{4(s)}$$

filtered, dried, ignited

$$CaC_2O_{4(s)} \xrightarrow{\Delta} CaO_{(s)} + CO_{(g)} + CO_{2(g)}$$

After cooling, the precipitate is weighed and the mass is determined.

Volatilization Gravimetry

The two most common: determining water and carbon dioxide.

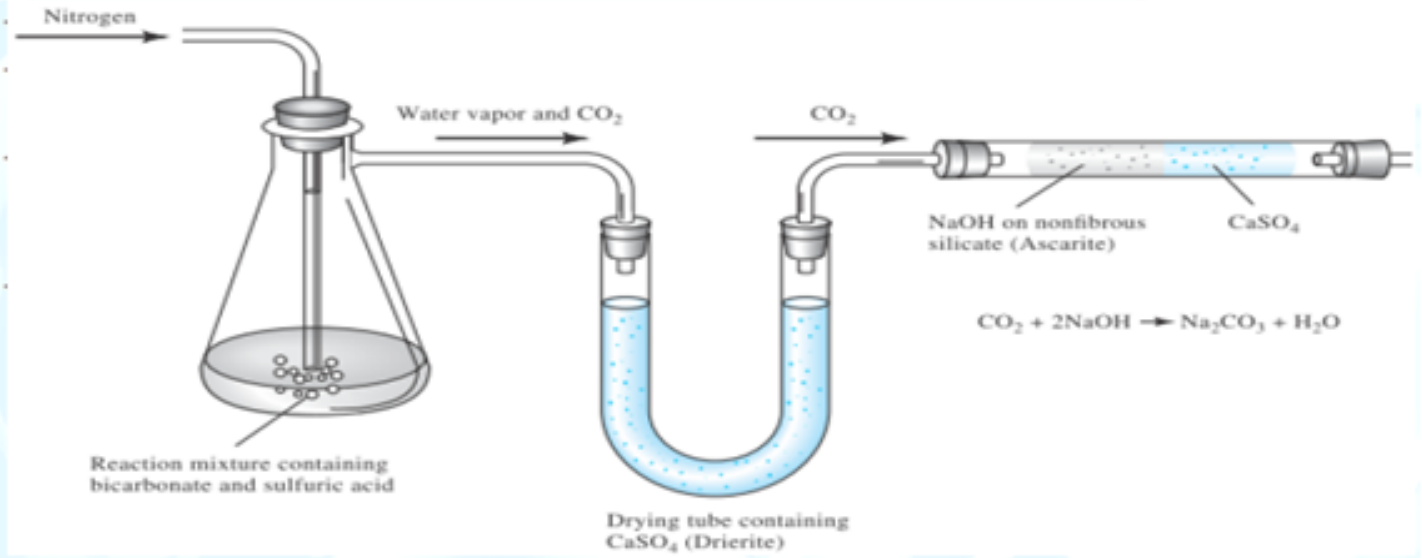


Figure 12-8 Apparatus for determining the sodium hydrogen carbonate content of antacid tablets by a gravimetric volatilization procedure.

$$NaHCO_3(aq) + H_2SO_4(aq) \rightarrow CO_2(g) + H_2O(l) + NaHSO_4(aq$$

- 1-Sulfides and sulfites can also be determined by volatilization. Hydrogen sulfide or sulfur dioxide evolved from the sample after treatment with acid is collected in a suitable absorbent.

- 2-Finally, the classical method for the determination of carbon and hydrogen in organic compounds is a gravimetric volatilization procedure in which the combustion products (H2O and CO2) are collected selectively on weighed absorbents.

3-The increase in mass serves as the analytical variable

The most important is the precipitation method in gravimetric analysis, which is explained in detail below.

A weighed sample of the substance to be analysed is brought into solution by a suitable method, and the element to be determined is then precipitated as an insoluble compound. The precipitate is filtered off,washed throughly, ignited (or dried) and weighed accurately. The content of the element is calculated from the weight of the precipitate and its formula and expressed as a percentage of the sample taken. The precipitate may be collected on a filter paper or in a Gooch crucible.

Properties of Precipitates and Precipitating Reagents

A gravimetric **precipitating agent** should react **specifically** or at least **selectively** with the **analyte** and give **precipitates** that is:

1. Enough **particle size** for retaining on filter paper
2. High **purity** (free of contaminants)
3. **Low solubility** that no significant loss of the analyte occurs during filtration and washing
4. Unreactive with air **(stable)**
5. **Known stoichimetric composition** after it is dried or, if necessary, ignited

* **Specific** reagents: react only with a single chemical species.

Selective reagents: react with a limited number of species