Introduction to Analytical chemistry ,definition, scope ,classification and Gravimetric analysis

Analytical chemistry is the branch of chemistry that deals with the analysis of different substances, and it involves the separation, identification, and the quantification of matter. by using of classical methods along with modern scientific instruments to achieve all these purposes.

Analytical chemistry is often described as the area of chemistry responsible for:

1. Characterizing the composition of matter, both qualitatively and quantitatively.
2. Improving established analytical methods.
3. Extending existing analytical methods to new types of samples.
4. Developing new analytical methods for measuring chemical phenomena...

The scope of analytical chemistry:

- The science seeks ever-improved means of measuring the chemical composition of natural and artificial materials by using techniques to identify the substances that may be present in a material and to determine the exact amounts of the identified substance.
- Analytical chemistry involves the analysis of matter to determine its composition and the quantity of each kind of matter that is present.
- Analytical chemists detect traces of toxic chemicals in water and air.
- A detection of the component in qualitative analysis can be the basis of the method or the procedure of its quantitative analysis.
- The reaction may be incomplete in qualitative analysis, while in quantitative analysis the reaction should be complete and give clear and known products.

◆ Analytical chemistry consists of:

(A) Qualitative analysis: which deals with the identification of elements, ions, or compounds present in a sample (tells us what chemicals are present in a sample).

(B) Quantitative analysis: which is dealing with the determination of how much of one or more constituents is present (tells how much amounts of chemicals are present in a sample). This analysis can be divided into three types:

(1) Volumetric analysis (Titrimetric analysis): is measured the volume of a solution containing sufficient reagent to react completely with the analyte.

(2) Gravimetric analysis: Gravimetric methods, determine the mass of the analyte or some compound chemically related to it.

(3) Instrumental analysis: These methods are based on the measurement of physical or chemical properties using special instruments. These properties are related to the concentrations or amounts of the components in the sample. These methods are compared directly or indirectly with typical standard methods. These methods consist of:

a) Spectroscopic methods: are based on measurement of the interaction between electromagnetic radiation and analyte atoms or molecules or on the production of such radiation by analytes (ultraviolet, visible, or infrared), fluorimetry, atomic spectroscopy (absorption, emission), mass spectrometry, nuclear magnetic resonance spectrometry (NMR), X-ray spectroscopy (absorption, fluorescence).

b) Electroanalytical methods: involve the measurement of such electrical properties that wanted to be determined, such as pH measurements, electrodeposition, voltametry, thermal analysis, potential, current, resistance, and quantity of electrical charge.

c) Separation methods: They mean the isolation of one component or more from a mixture of components in solid, liquid and gas cases. These methods are included with instrumental methods since the instruments and equipment's are used in separation processes. These methods involve precipitation, volatilization, ion exchange, extraction with solvent and various chromatographic methods.

◆ Measurements in Analytical Chemistry:

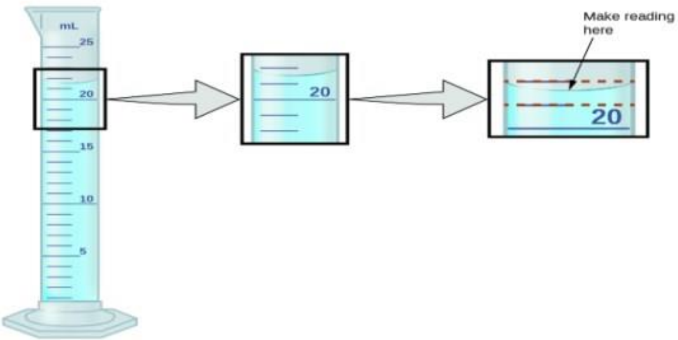
Units of Measurement: A measurement usually consists of a unit and a number expressing the quantity of that unit. These units are called SI units after the Système International ’Unités. Sometimes it is preferable to express measurements without the exponential term, replacing it with a prefix (Table 1).

Table 1. Common Prefix for Exponential Notation

Prefix	Symbol	Factor	Prefix	Symbol	Factor	Prefix	Symbol	Factor
yotta	Y	10^{24}	kilo	k	10^{3}	micro	μ	10^{-6}
zetta	Z	10^{21}	hecto	h	10^{2}	nano	n	10^{-9}
eta	E	10^{18}	deka	da	10^{1}	pico	p	10^{-12}
peta	P	10^{15}	-	-	10^{0}	femto	f	10^{-15}
tera	T	10^{12}	deci	d	10^{-1}	atto	a	10^{-18}
giga	G	10^{9}	centi	c	10^{-2}	zepto	z	10^{-21}
mega	M	10^{6}	milli	m	10^{-3}	yocto	y	10^{-24}

Uncertainty in Measurements:

Uncertainty in Measurements A measurement provides information about its magnitude and its uncertainty.



Accuracy : The closeness of an experimental measurement or result to the true or accepted value.

Precision: The random or indeterminate error associated with a measurement or result. Sometimes called the variability, it can be represented statistically by the standard deviation or relative standard deviation (coefficient of variation)

Concentration: It is a general measurement unit stating the amount of solute present in a known amount of solution or (The ratio of the amount of solute to the amount of solution.) Although we associate the terms "solute" and "solution" with liquid samples, we can extend their use to gas-phase and solid-phase samples as well.

Solution : Homogeneous mixture of two or more substance produce from dissolved (disappeared) solute particle (ions, atoms, molecules) (lesser amount) between solvent particle (larger amount).

Solvent (larger amount) + Solute (lesser amount) = Solution

Solvent : The medium in which the molecules or ions are dissolved.

Solute: Any substance dissolved in a solvent.

Analyte: Constituent of the sample which is to be studied by quantitative measurements or identified qualitatively.

Molarity and Formality : Both molarity and formality express concentration as moles of solute per liter of solution; however, there is a subtle difference between them. Molarity is the concentration of a particular chemical species. Formality, on the other hand, is a substance's total concentration without regard to its specific chemical form. There is no difference between a compound's molarity and formality if it dissolves without dissociating into ions. The formal concentration of a solution of glucose, for example, is the same as its molarity.

Normality: Normality defines concentration in terms of an equivalent, which is the amount of one chemical species that reacts stoichiometrically with another chemical species.

Molality :It is used in thermodynamic calculations where a temperature independent unit of concentration is needed. Molarity is based on the volume of solution that contains the solute. Since density is a temperature dependent property, a solution's volume, and thus its molar concentration, changes with temperature. By using the solvent's mass in place of the solution's volume, the resulting concentration becomes independent of temperature.

Weight, Volume, and Weight-to-Volume:

Percents Weight percent (% w/w), volume percent (% v/v) and weight-to volume percent (% w/v) express concentration as the units of solute present in 100 units of solution.

Parts Per Million and Parts Per Billion:

Parts per million (ppm) and parts per billion (ppb) are ratios that give the grams of solute in, respectively, one million or one billion grams of sample. If we approximate the density of an aqueous solution as 1.00 g/mL, then we can express solution concentrations in ppm or ppb using the following relationships.

$$\text{ppm} = \frac{\mu g}{g} = \frac{mg}{L} = \frac{\mu g}{mL} \qquad \text{ppb} = \frac{ng}{g} = \frac{\mu g}{L} = \frac{ng}{mL}$$

For gases a part per million usually is expressed as a volume ratio

Constituent:

A component of a sample; it may be further classified as:

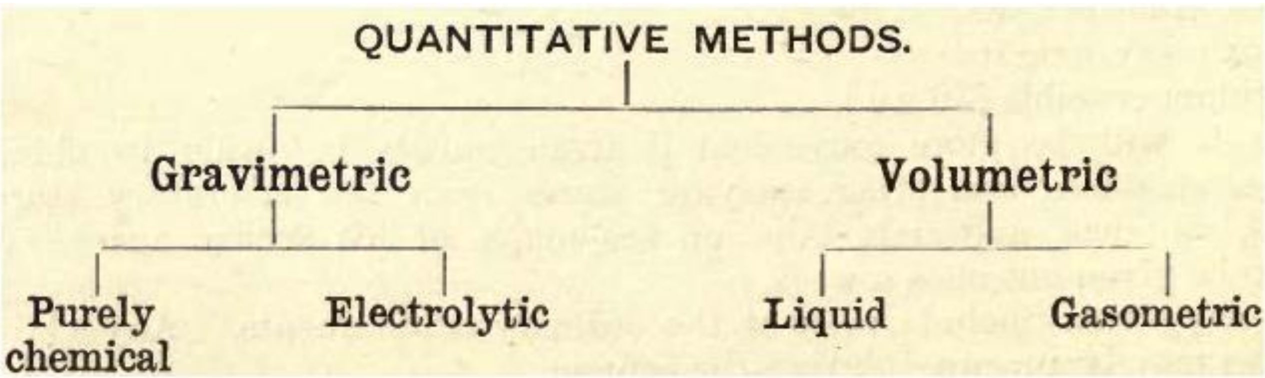
- Major > 10%
- Minor 0.01–10%
- Trace 1–100 ppm (0.0001–0.01%) ✦ Ultratrace < 1 ppm 2.15

Detection Limit:

The smallest amount or concentration of an analyte that can be detected by a given procedure and with a given degree of confidence.

Table 2. Common Units for Reporting Concentration

Name	Units	Symbol
molarity	$\frac{\text{moles solute}}{\text{liters solution}}$	M
formality	$\frac{\text{moles solute}}{\text{liters solution}}$	F
normality	$\frac{\text{equivalents solute}}{\text{liters solution}}$	N
molality	$\frac{\text{moles solute}}{\text{kilograms solvent}}$	m
weight percent	$\frac{\text{grams solute}}{\text{100 grams solution}}$	% w/w
volume percent	$\frac{\text{mL solute}}{\text{100 mL solution}}$	% v/v
weight-to-volume percent	$\frac{\text{grams solute}}{\text{100 mL solution}}$	% w/v
parts per million	$\frac{\text{grams solute}}{10^6\ \text{grams solution}}$	ppm
parts per billion	$\frac{\text{grams solute}}{10^9\ \text{grams solution}}$	ppb



Gravimetric Methods of Analysis:

- Gravimetric methods: The quantitative methods that are based on determining the mass of a pure compound to which the analyte is chemically related.
- Gravimetric analysis is one of the most accurate and precise methods of macro-quantitative 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 four main types of this method of analysis are precipitation, volatilization, electro-analytical and miscellaneous physical method.

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.
- 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 [Ca2+] 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.

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.
- The two most common: determining water and carbon dioxide.

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

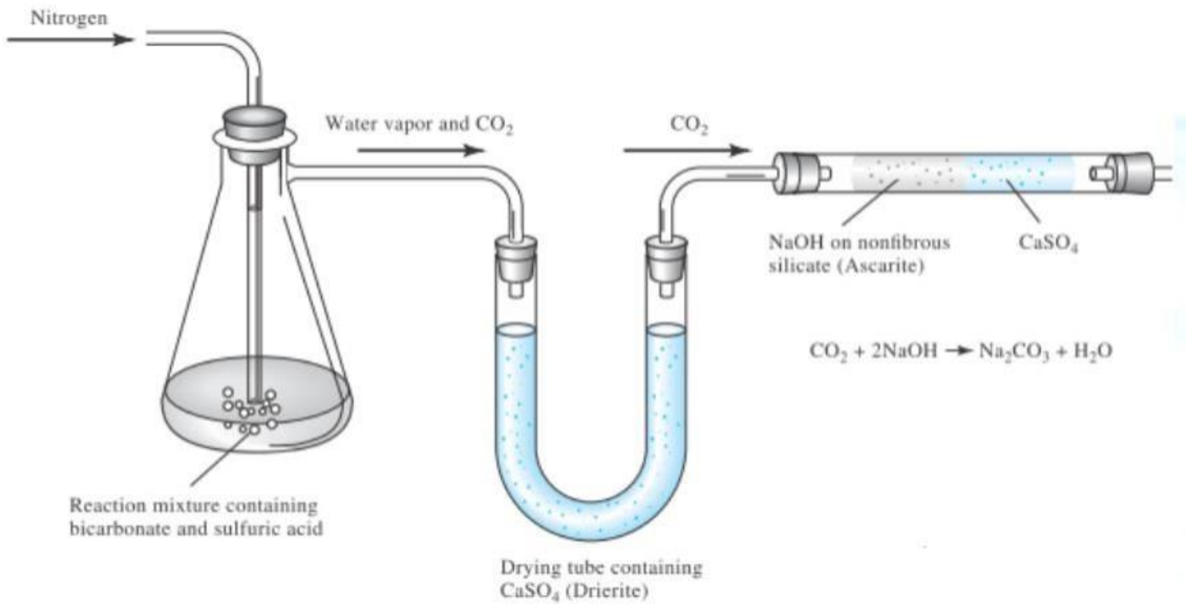


Figure : Apparatus for determining the sodium hydrogen carbonate content of antacid tablets by a gravimetric volatilization procedure.

3-Electrogravimetry:

- The analyte is separated by deposition on an electrode by an electrical current.
- Apply potential to cause a soluble species to reduce or deposit on a solid electrode e.g., reduce Cu2+ onto Pt cathode .

$$Cu^{2+}_{(aq)} + 2e^{-} \leftrightarrow Cu \text{ (metal on Pt)}$$

- Change in weight of dried cathode before & after deposition = amount of Cu in sample

4- Miscellaneous Methods:

The analytical methods fall into two categories; basically, their use involves either radioactivation of the elements of interest or radioactive tracing. In both cases the results are obtained by quantitative determinations of radioactivity.