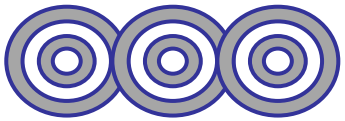
Instrumental Methods of Analysis



Introduction to Analytical Chemistry

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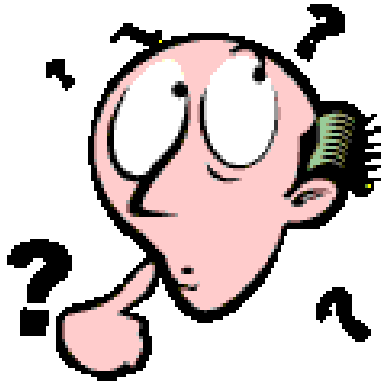
What is Chemistry ???

Chemistry is the study of the **properties** and **behavior** of **matter**.

Realms

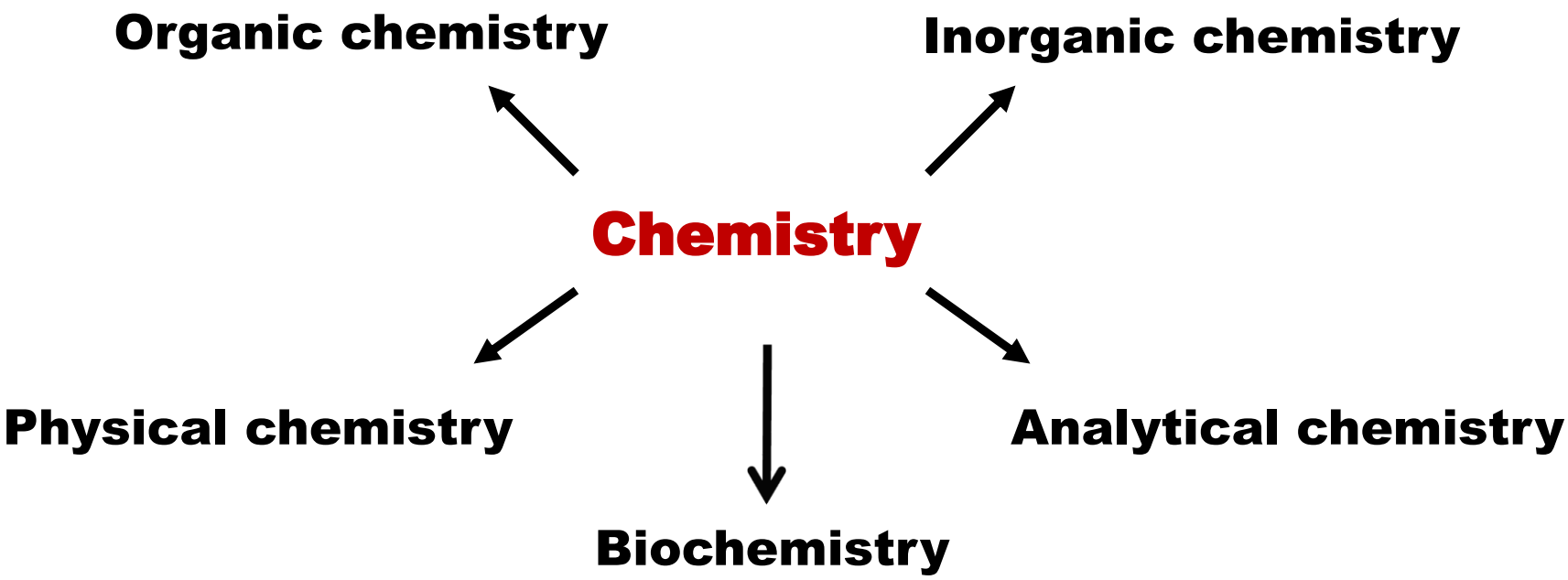
Macroscopic
Ordinary sized objects

Sub-microscopic
Atoms and molecules



Chemistry is the science that seeks to understand the properties and behavior of matter by studying the properties of atoms and molecules. Chemistry provides important understanding of our world and how it works.

Branches of Chemistry



Definition of Analytical Chemistry

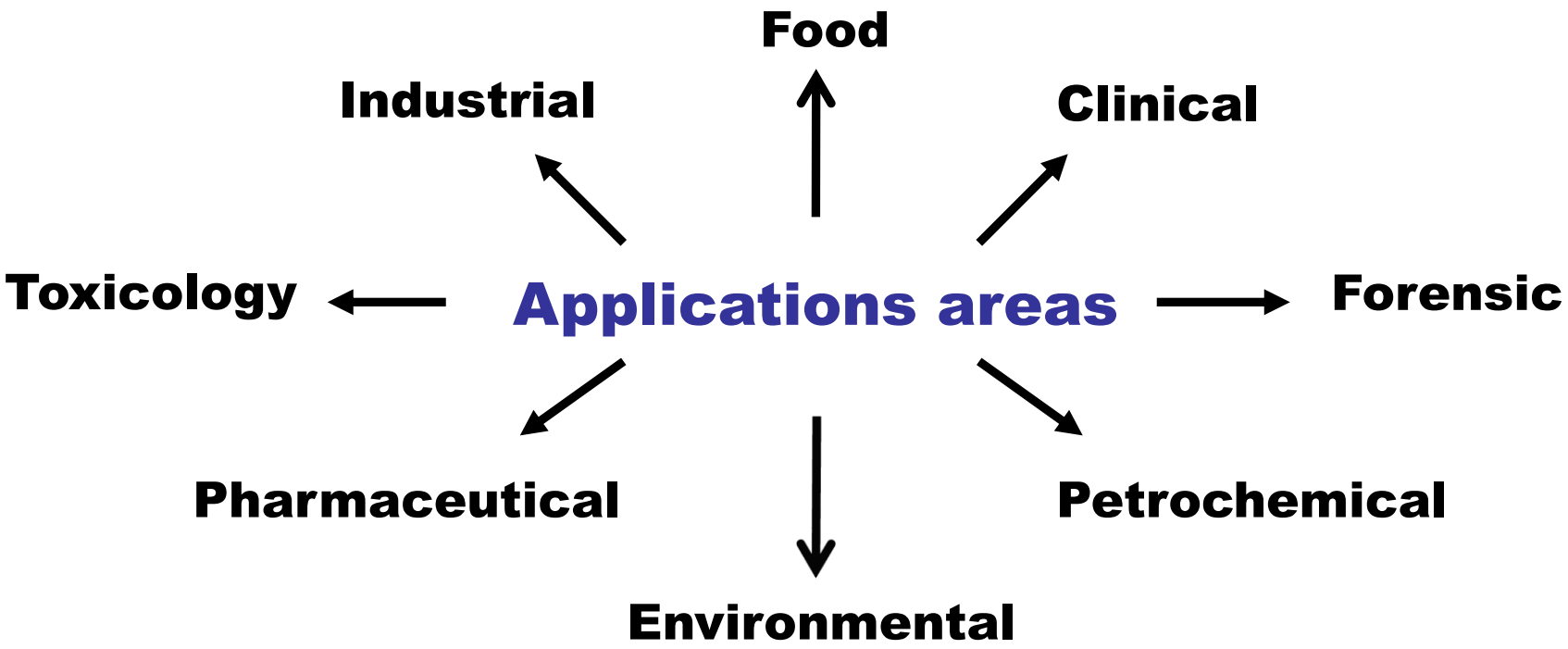
Subdivision of chemistry dealing with the **qualitative** and **quantitative** determination of chemical components of materials.

Analytical Chemistry is the science of chemical measurement. Its object is the generation, treatment and evaluation of **signals** from which information is obtained on the composition and structure of matter.

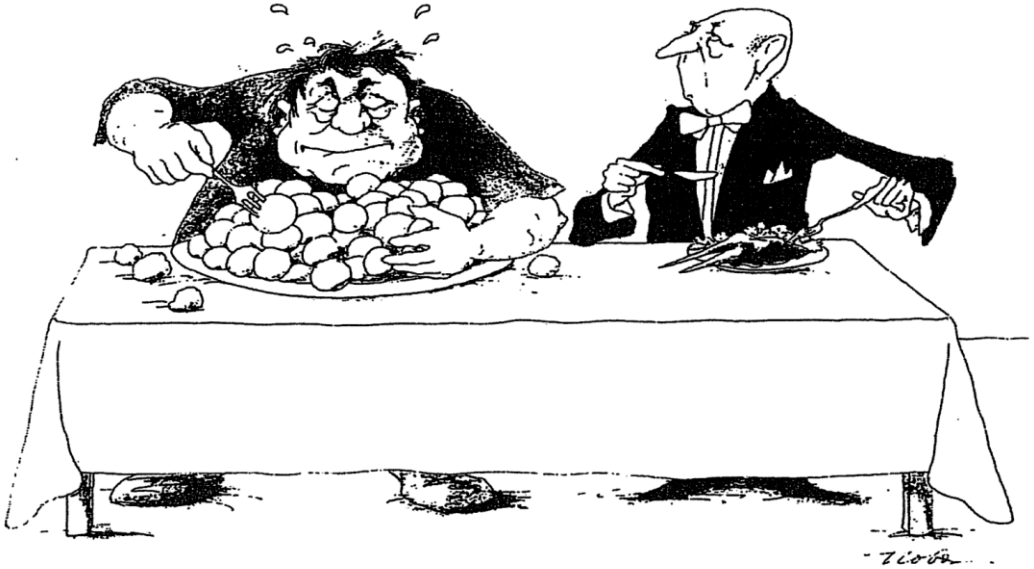
Analytical chemistry also is concerned with developing the tools used to examine chemical **compositions**.

Interactions with all other fields

In recent times, analytical chemistry has stimulated not only chemistry but many fields of science, technology and society. Conversely, analytical chemistry itself has always been heavily influenced by fields like engineering, materials science, environmental protection, biology and medicine.



Analytical chemistry study the **identification** and **quantification** of the chemical components of natural and artificial materials.



Quantitative analysis

Determination of the amount of analyte in the sample.

Examples:

-Measuring the concentration of glucose in blood.

-Determining the concentration of metals and ions in water (drinking water standard).

Qualitative analysis

Identify what is present in the sample.

Examples:

-Identifying the products of chemical reaction.

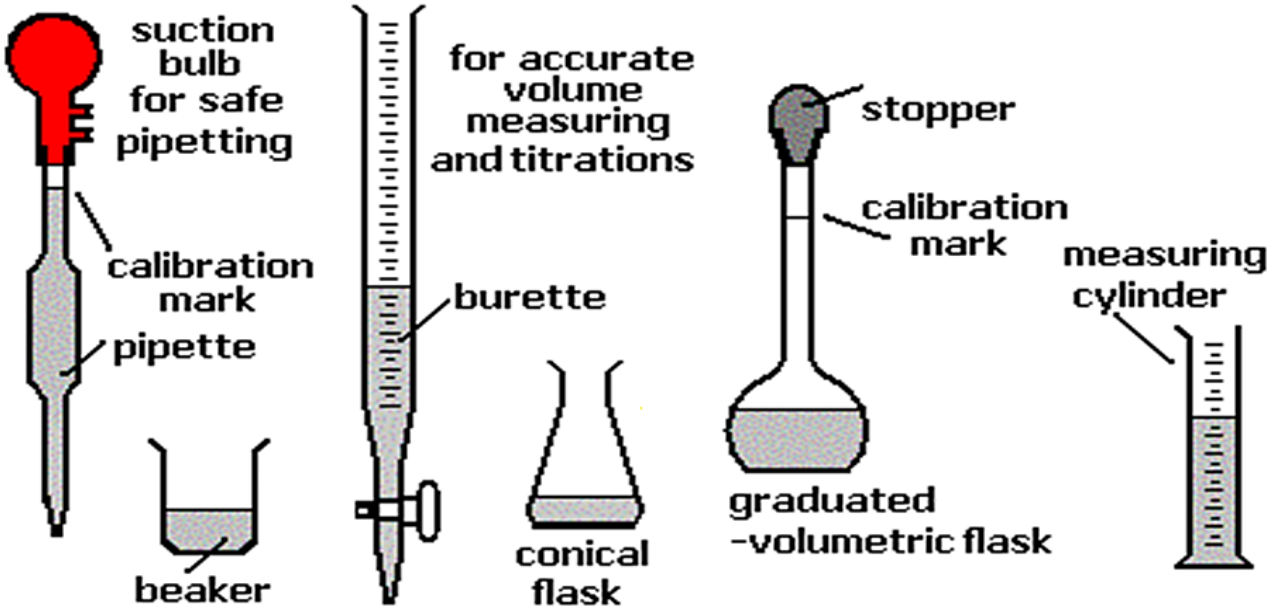
-Screening athlete's urine (Stimulants).

Methods and Equipment

Analytical methods can be separated into **classical** and **instrumental**.

Classical methods (also known as wet chemistry or bench chemistry)
Classical methods involve the use of laboratory glass and plastic wares, such as beakers and flasks, and excludes chemical analysis using instrumentation.

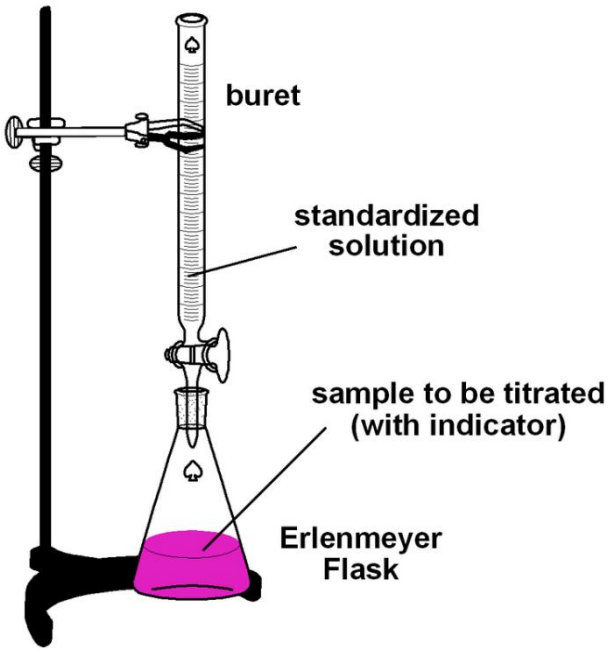
Examples:
Precipitation, extraction, recrystallization, distillation and qualitative analysis by color, odor or melting point.

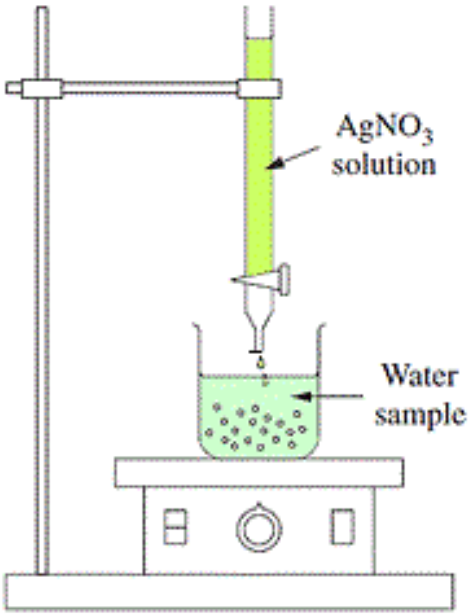


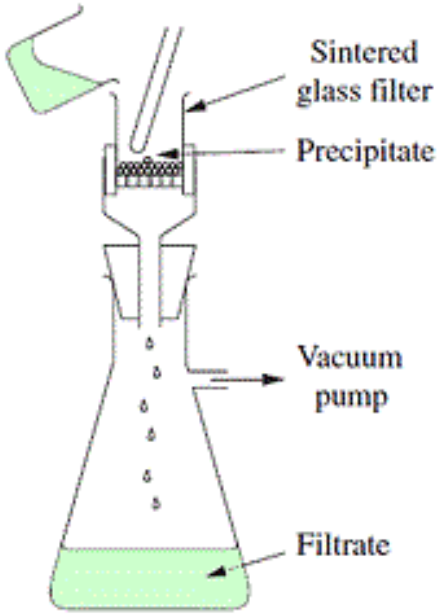
Classical quantitative analysis is achieved by measurement of **weight** or **volume**.

Gravimetric analysis: determining the amount of material present by weighing the sample before and/or after some transformation.

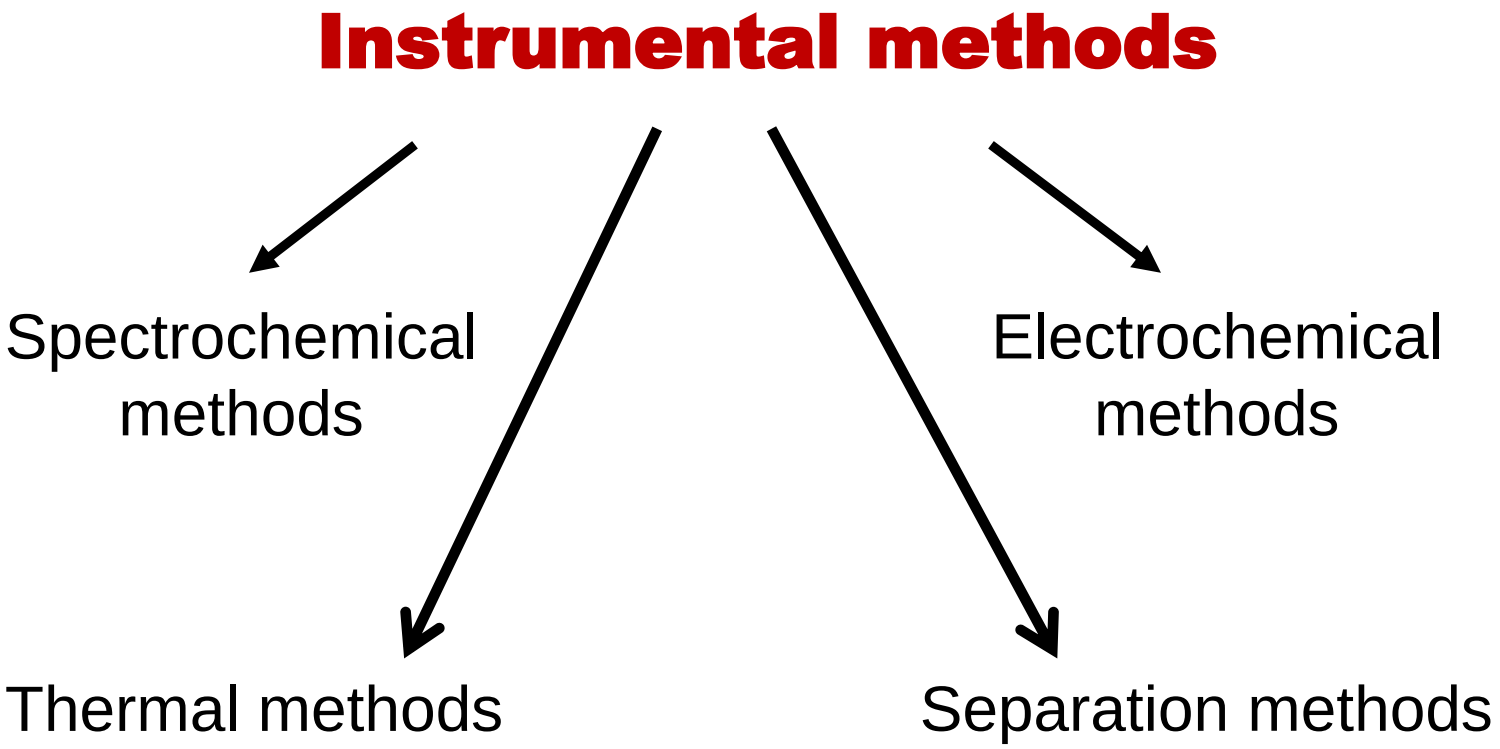
Volumetric analysis: titration involves the addition of a reactant to a solution being analyzed until some equivalence point is reached.







Instrumental methods use an apparatus for chemical analysis of the analyte.

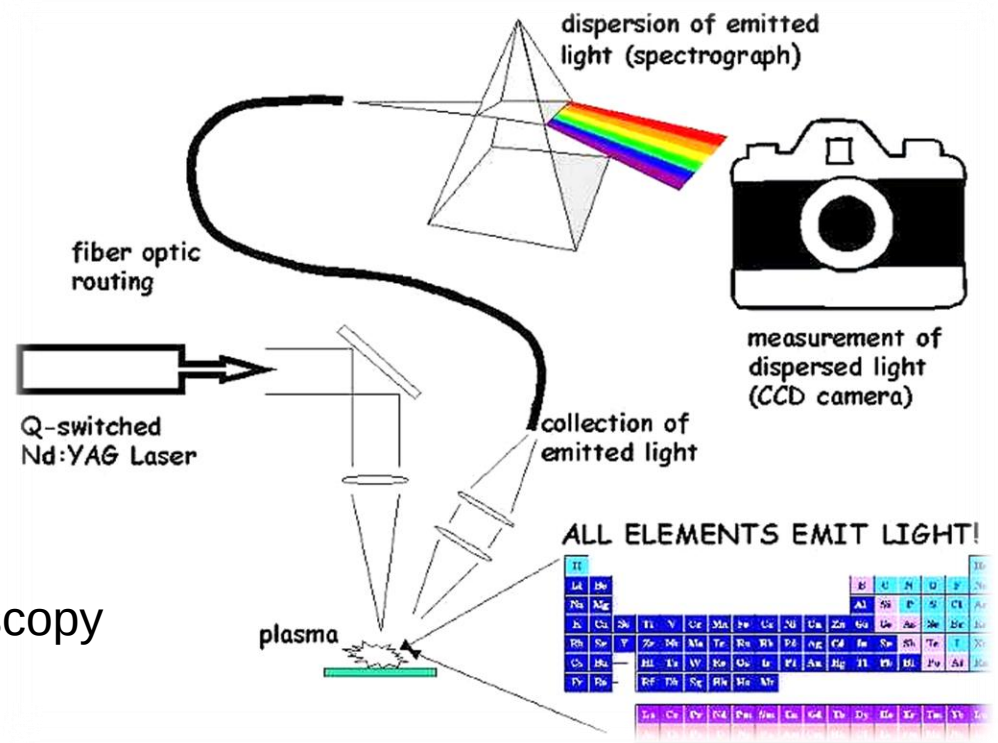


Spectrochemical Methods

Is the study of the interaction between matter and radiated energy. Spectroscopy concept is now expanded to comprise any interaction with radiative energy as a function of its wavelength or frequency. Spectroscopic data is often represented by a spectrum, a plot of the response of interest as a function of wavelength or frequency.

Examples:

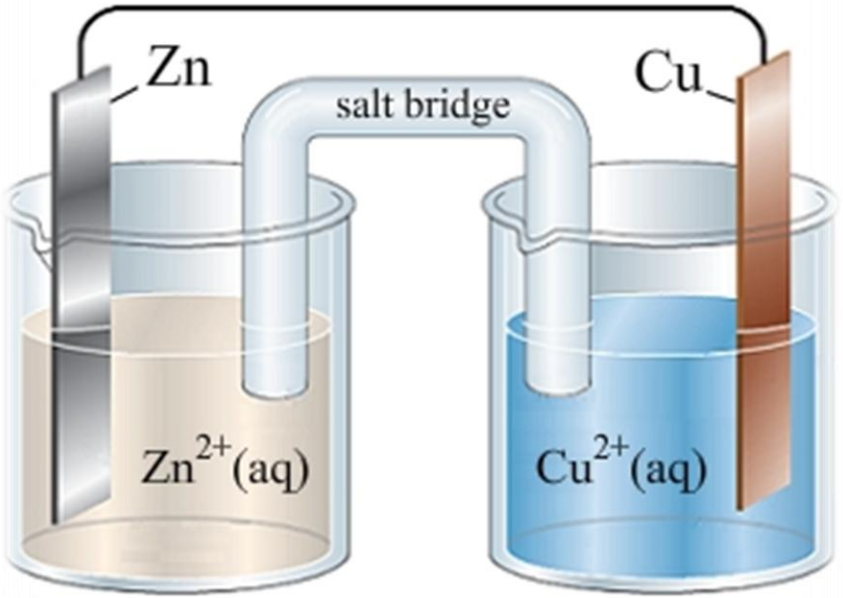
-Atomic absorption spectroscopy
-Flame emission spectroscopy
-Visible and ultraviolet spectroscopy
-Infrared spectroscopy
-Nuclear magnetic resonance spectroscopy



Electrochemical Methods

Is the study of chemical reactions which take place at the interface of an electrode, usually a solid metal or a semiconductor, and an ionic conductor, the electrolyte. These reactions involve electric charges moving between the electrodes and the electrolyte (or ionic species in a solution). Thus electrochemistry deals with the interaction between electrical energy and chemical change, and involve measurement of potential, current, or charge in an electrochemical cell that serves as the analytical signal.

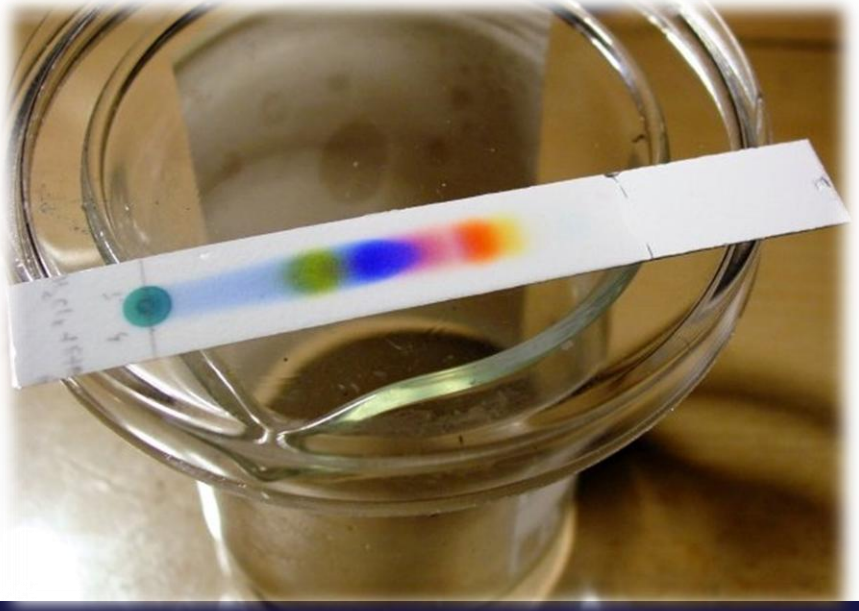
Examples:
-Potentiometric methods
-Coulometric methods
-Voltammetric methods



Separation Methods

Is a method to achieve any mass transfer phenomenon that converts a mixture of substances into two or more distinct product mixtures or its pure constituents. Separations are carried out based on differences in chemical properties, or physical properties such as size, shape, mass, density, boiling and melting points, solubility, or chemical affinity, between the constituents of a mixture, and are often classified according to the particular differences they use to achieve separation. Usually there is only physical movement, no substantial chemical modification.

Examples:
-Solvent extraction
-Chromatography
-Electrophoresis

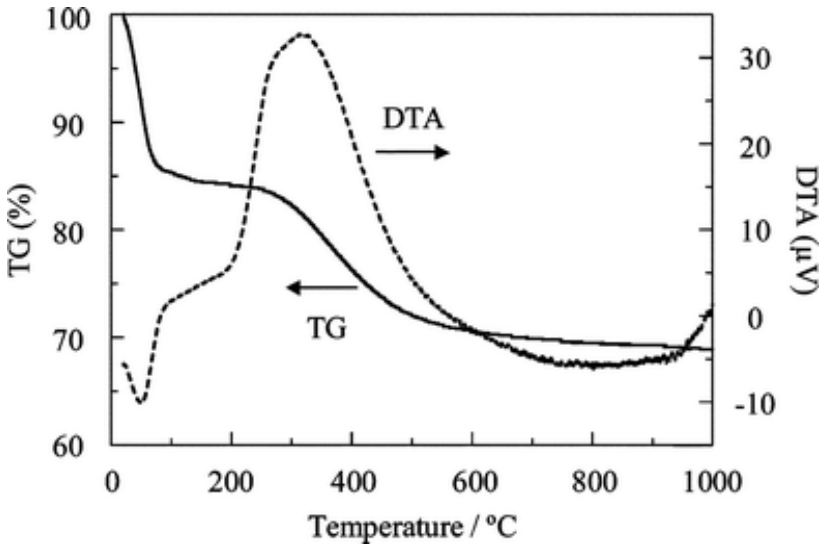


Thermal Methods

Is a branch of materials science where the properties of materials are studied as they change with temperature. Several methods are commonly used, these are distinguished from one another by the property which is measured.

Examples:

-Dielectric thermal analysis (DEA)
Measure dielectric permittivity and loss factor
-Differential thermal analysis (DTA)
Measure temperature difference
-Differential scanning calorimetry (DSC)
Measure heat difference
-Dilatometry (DIL)
Measure volume
-Thermogravimetric analysis (TGA)
Measure mass



Steps in an Analysis

1- Define the problem (target)

What is the problem, what we need to found, qualitative and/or quantitative, objectives, what will the information be used for and who will use it, how accurate and precise does it have to be, what is the budget, ….etc.

2- Select a method

Sample type, size, sample preparation needed, concentration and range, sensitivity needed, selectivity needed, interferences, accuracy and precision needed, tools and instruments available, expertise and experience, cost, speed, manual or automated, are literature methods and standard available, ….etc.

3- Sampling (prepare the sample for analysis)

Sample collection then sample preparation.

Sample type, homogeneity, size, statistics and errors, physical state, solid, liquid, gas, dissolve, ash or digest, separation and avoiding matrix effect needed, need to concentrate the analyte, preconcentration, need to change and derivatize the analyte, need to adjust solution conditions, pH, add reagents, ….etc.

4- Perform any necessary separations

Distillation, precipitation, extraction, chromatography, electrophoresis, ….etc.

5- Perform the measurement

Calibration, validation, controls, blanks, replicates, ….etc.

6- Analyzing data and interpreting results

Statistical analysis, reliability, validation, report results, ….etc.


Thank You!