Analytical Chemistry

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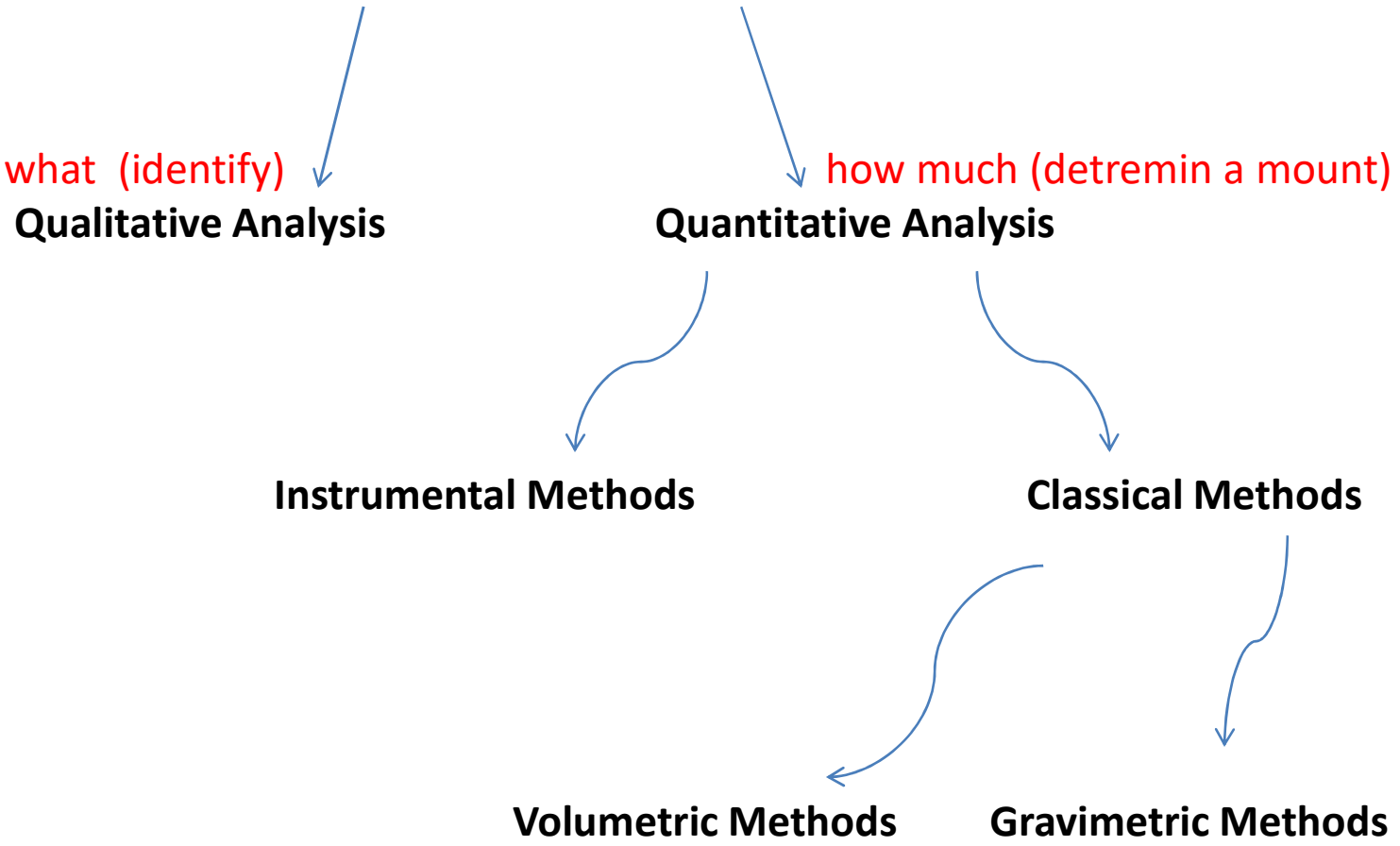
Lecture 2

Analytical Chemistry

Introduction

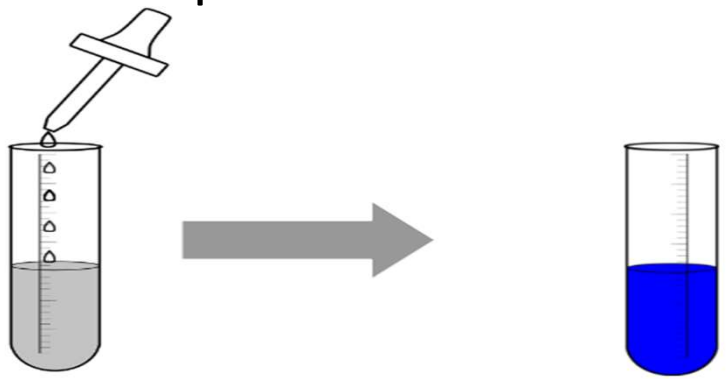
- Analytical chemistry is the branch of chemistry deals with the analysis of substance (**analytes**) present in the sample **qualitatively** and **quantitatively** .
- In order to accomplish this analysis we must know the **physical** and **chemical properties** of these substances (analyte).
- Analytical chemistry deal with the **separation** ,**identification** and **determination** of substances (analyte) in a sample .
- Sample is any thing that comes to mind in air, water ,soil , food and living organism such as piece of rock or apiece of meat or some water from the tank of house or from a river or a lack or a sea or some tissue or blood from huamans or animals or some vegetables.
- The sample is taken to the laboratory and analyzed for its substance (analytes) after pretreatment and the final step is calculation of the percentage of each analyte in the sample.

Chemical analysis



❖Qualitative Analysis.(What element atom founded in substance)

- The analysis detects **(identify)** the type of all or some of substances (analytes)present in the sample (**element and compounds**)
- These substances can be detected either by a **chemical reaction**. For example , when we add silver nitrate solution to the sample solution a white precipitate indicates the presence of chloride ion in the sample.



Colour change observed.

❖Quantitative Analysis (how much)

- this analysis gives knowledge of the a **mount** of all or some of the substances present in the sample and uses two type of analysis depending on the concentration of the substance in the sample namely **classical chemical analysis** and **instrumental analysis**.

❖Instrumental Analysis

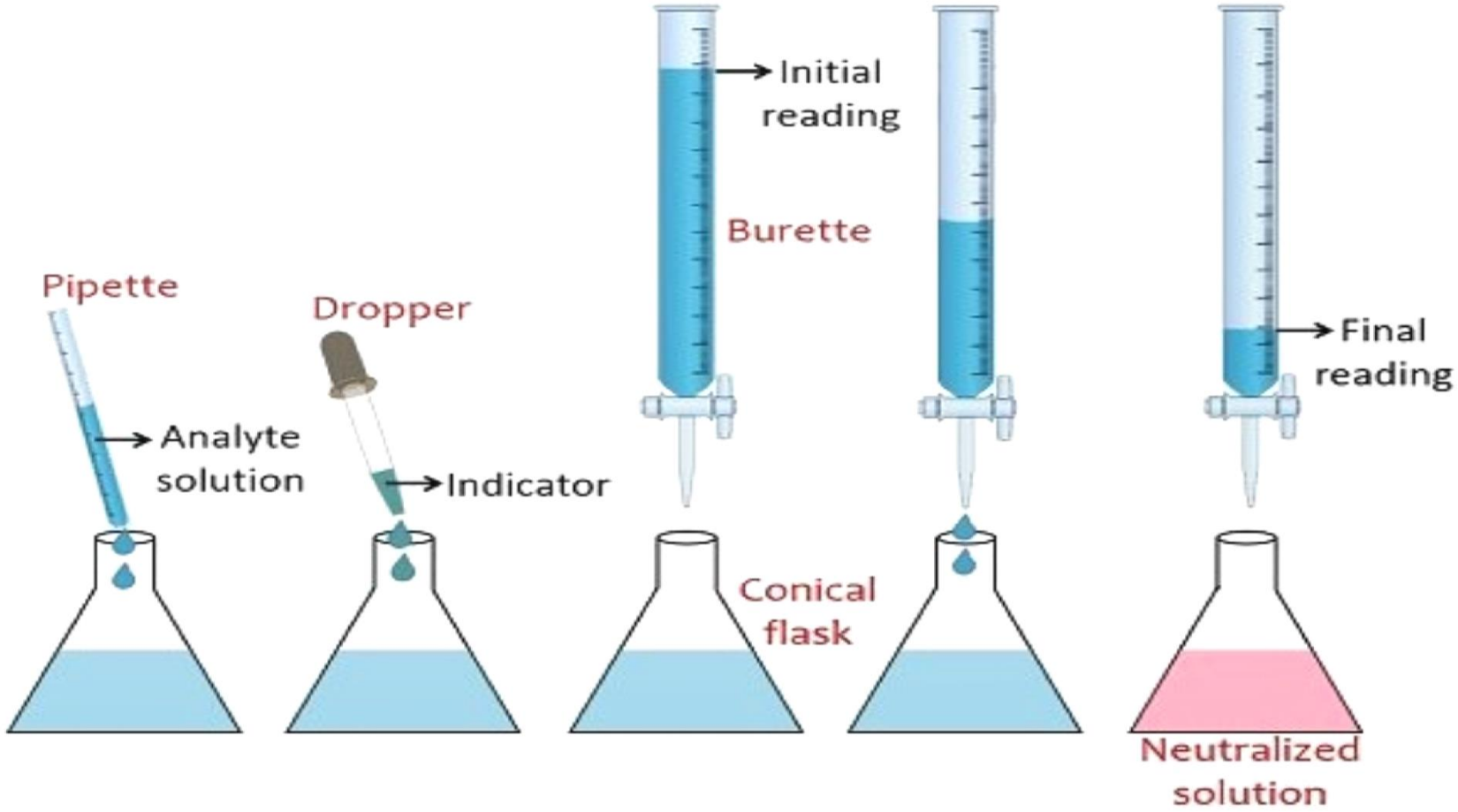
- Currently there are many instruments that are separated and distinguish substances (organic or inorganic) in the sample , such as gas chromatography – mass spectrometry (GS- MS) , High Performance liquid chromatography –mass spectrometry (HPLC – MS), Infrared Spectra (IR) and ect..

❖Classical chemical analysis

- Which depended on the **chemical reaction** , such as volumetric analysis and gravimetric analysis. And it used simple equipment such as burettes , balance , flame and furnace and is used to estimate high concentration (**more than 0,001M**)

❖Volumetric Analysis

- Volumetric analysis , a classical quantitative analysis method ,is a technique where a **solution a known concentration** is used to determine the concentration of an **unknown solution**.
- The reagent (the titrant) is added gradually or stepwise to the analyte from a burette.
- The key to performing a successful titrimetric analysis is to recognize the equivalence point of the titration (**the point at which the quantities of the two reaction species are equivalent**) ,typically observed as a color change.
- If no spontaneous color change occurs during the titration , a small amount of a chemical indicator is add to the analyte previous to the titration.
- Chemical indicators are available that change color at or near equivalence point of acid base ,oxidation reduction , complexation and precipitation titrations.


Pipette
Analyte solution
Dropper
Indicator
Initial reading
Burette
Conical flask
Final reading
Neutralized solution

- The volume of added titrant corresponding to the indicator color change is the **end point of the titration**.
- The end point is used as an approximation of the equivalence point and is employed ,with the known concentration of the titrant to calculate the amount or concentration of the analyte.

(**The end point in titration refers to the point at which the indicator changes color)**

- An indicator is often added to the reaction flask to signal when all of the analyte has reacted.
- The titrant volume where the signal is generated is called the end point volume or titer.
- The equivalence and end points are **rarely** the same (titration error)

❖Gravimetric Analysis

- Gravimetric analysis is a technique in which the a mount of an analyte in a sample is determined by **converting the analyte to some product and mass** of product can be easily measured.
- In gravimetric analysis ,a substance related to another substance with a know chemical structure is separated and its masse is measured correctly.
- Is one of the most accurate and precise methods of macro quantitative analysis
- In the process the analyte is selectively convert to an **insoluble form**. The separated precipitate is dried or ignited, possibly to another form , and is accurately weight from the weight of the precipitate and a knowledge of its chemical composition , we can calculate the weight of analyte in the desired form. In fact ,gravimetric analysis was used to determine the atomic masses of many elements.

❖ In gravimetric analysis, the target(analyte) substance is separated through different methods.

✓ **types of gravimetric analysis:**

1. **Precipitation gravimetry**: The analyte is separated from a solution of the sample as a precipitate and is converted to a compound of know composition that can be weighed.
2. **Volatilization gravimetry** : the analyte is separated from other components of a sample by conversion to a gas. The weight of this gas then serves as a measure of the analyte concentration.
3. **Electro gravimetry** : the analyte is separated by deposition on an electrode by an electrical current. The mass of this product then provides a measure of the analyte concentration.
4. **Isolation gravimetric methods**

   the analyte is separated by determined mass of particulate analyte following its separation from its matrix.

❖Precipitation gravimetric methods

- Gravimetric analysis method depends on precipitation reactions consist of some steps that need to be made **quantitatively** (that means in a way **without any increase or decrease in amount** of matter)
- Many metallic elements in their ionic forms react with negative counter ions to produce stable precipitates
- Precipitation reaction are employed in the method to separate the target (analyte) component from a sample, and they can convert a dissolved compound (liquid) in to a precipitate (solid) so that we can weight it.

- for example :
- silver ions form stable and highly insoluble (slightly soluble) salts with chloride, bromide and iodide.

$Ag^+ + Cl^- \rightarrow$ **AgCL (s)**

$Ag^+ + Br^- \rightarrow$ **AgBr (s)**

$Ag^+ + I^- \rightarrow$ **AgI (s)**

AgCl , AgBr and AgI can then be washed, dried and weighed in order to determine the concentration of the analyte in the original solution.

❖Step of Gravimetric analysis

1. Dissolve the analyte
2. First treatment of the analyte
3. Precipitation
4. Digestion
5. Filtration
6. Washing
7. Drying or burning
8. Weighing the precipitate
9. Calculation

❖Properties of precipitates and precipitating Reagents

- the ideal **gravimetric precipitating agent** should react specifically or at least selectively with the analyte.
- Specific reagents, which are rare, react only one with a single chemical species.
- Selective reagents, which are more common , react with a limited number of species.
- In addition to specificity and selectivity the ideal precipitating reagent would react with the analyte to give a product that is :

❑ Enough particle **size** for retaining on filter
❑ **High purity** (free of contamintal)
❑ **Low solubility** that no significant loss of the analyte occurs during filtration and washing
❑ Unreactive with air (stable)
❑ **Known chemical** structure after it is dried or if necessary ignited.