Introduction to Analytical Chemistry

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ANANLYTICAL CHEMISTRY

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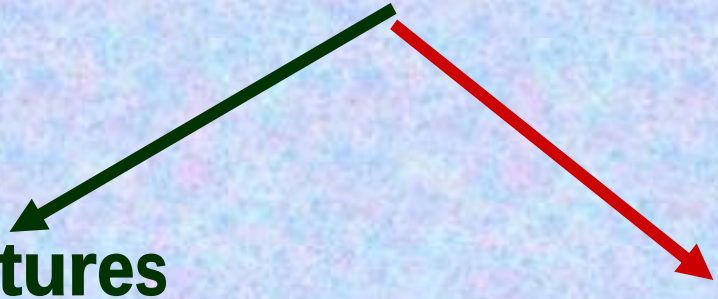
The study of the chemical composition of natural and artificial materials.....

Chemical Composition



Geometric features

e.g. **molecular morphologies** and **distributions of species** within a sample

Single dimensional features

e.g. **percent composition and species identity.**

Practical Applications

1. **Biomedical applications**
   (bioanlytical…) (Cancer !!! Chemicals present in blood !)
2. **Environmental monitoring.**
   (e.g. how much lead in drinking water?)
3. **Control of industrial manufacturing**
   - **Industrial Quality Assurance (QA)**
4. **Forensic science**

Analytical chemistry is concerned with questions such as

"what chemicals are present ?”

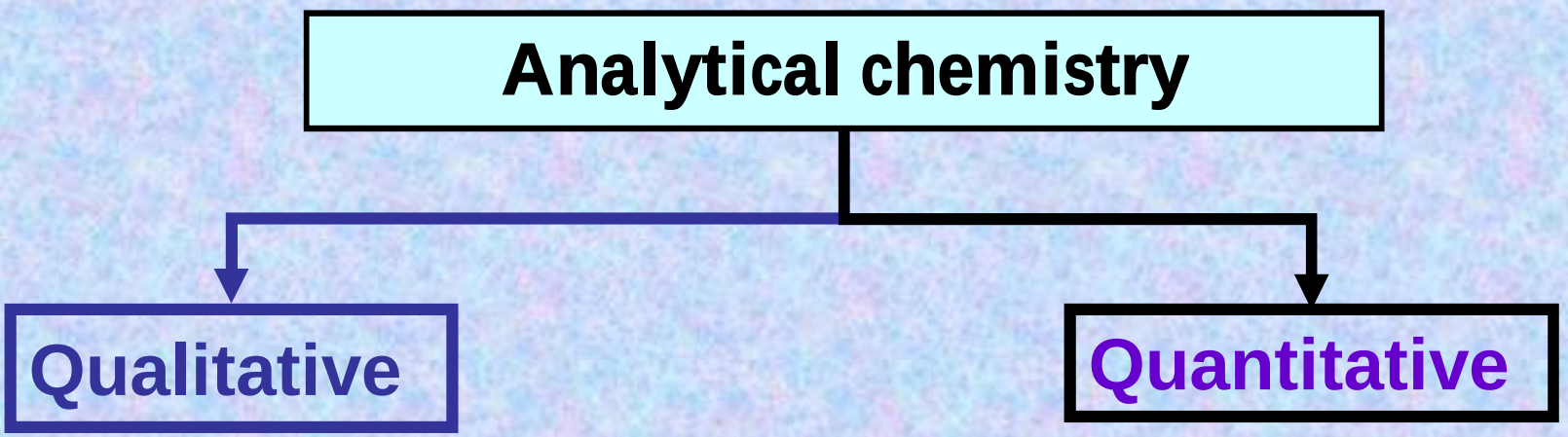
what are their characteristics?

what quantities are they present?

It stops after they are answered

Modern analytical chemistry

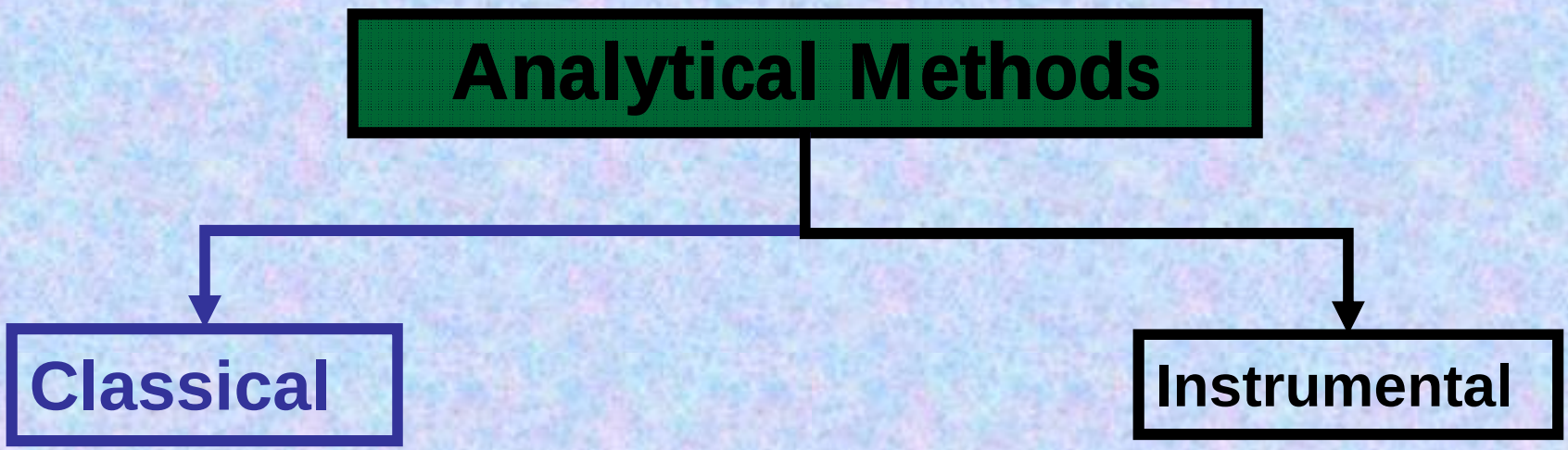
- Dominated by **instrumental analysis**
- Academics tend to either **focus on new applications and discoveries** or on new methods of analysis.
- Analytical chemistry plays an important role in the **pharmaceutical industry** e.g. **discovery of new drug ,**
- **understanding the interactions between the drug and the patient**



Description of chemical composition in terms of

- elements, compounds,
- or structural units

The measurement of the amount of a given element or compound in a sample



The use of simple apparatus e.g. burettes, pipettes and weighing balances

The use of electronic equipments that generate electrical signals related to some property of the substance being analysed

Classical Analytical Methods

Titrimetry/ Volumetric

Involves the addition of a reactant to a solution being analyzed until some equivalence point is reached Acid-base, Redox, precipitation & complexometric titrations.

Gravimetry

Determination of the amount of material present by weighing the sample before and/or after some transformation . Amount of water in a hydrate- difference in weight -remove the water by heating.

Inorganic qualitative analysis

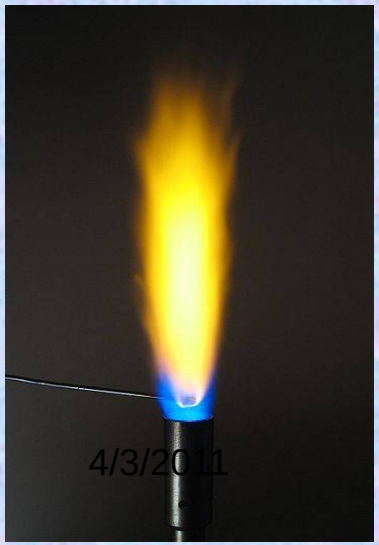
Classical Analytical Methods

Titrimetry/ Volumetric

Involves the addition of a reactant to a solution being analyzed until some equivalence point is reached

Gravimetry

Determination of the amount of material present by weighing the sample before and/or after some transformation



Inorganic qualitative analysis

Systematic scheme to confirm the presence of ions or elements (usually aqueous) by a series of reactions.

Instrumental Methods

1. Spectroscopy

Measures the interaction of the atoms or molecules with electromagnetic radiation e.g. AAS, AES, UV, IR, RS, NMR…

2. Mass Spectrometry

Measures mass-to-charge ratio of molecules using electric and magnetic fields.

3. Crystallography

Characterizes the chemical structure of materials at the atomic level usually by using x-rays

Instrumental Methods

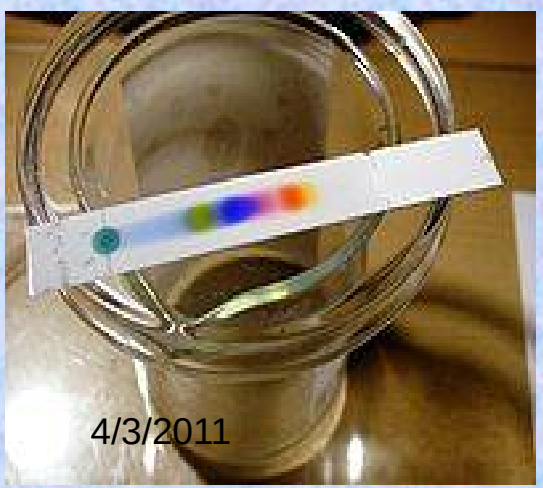
4. Electrochemical Analysis

Measures the interaction of the material with an electric field e.g. potentiometry, amperometery, coulometry

5. Thermal analysis

Measures the interaction of a material with heat. e.g. Calorimetry and thermogravimetric analysis

6. Separation



Decreases the complexity of material mixtures

e.g. Chromatography and electrophoresis

Instrumental Methods

7. Hybrid Techniques

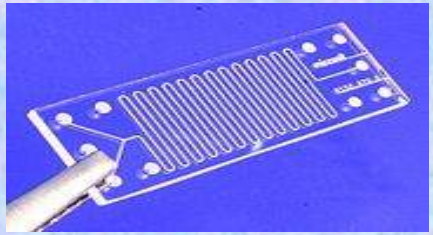
Hybrid or hyphenated techniques- Combinations of the above techniques

e.g. Gas Chromatography-Mass Spectrometry, GC-IR, LC-NMR, Capillary Electrophoresis-Mass Spectrometry (CEMS)

8. Microscopy

The visualization of single molecules, single cells, biological tissues and nano-micro materials- optical microscopy, electron microscopy, scanning probe microscopy,

9. Lab-on-a-chip



Micro total analysis System (μTAS)

A whole device can be visualized under a microscope.

Instrumental Methods

7. Hybrid Techniques

Combinations of the above techniques e.g. GC-MS, GC-IR, LC-NMR, CE-MS

8. Microscopy

The visualization of single molecules, single cells, biological tissues and nano- micro materials

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Micro total analysis System (μTAS)

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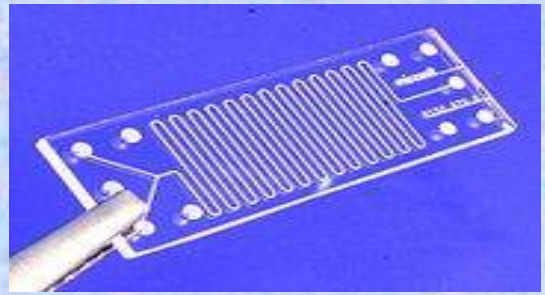
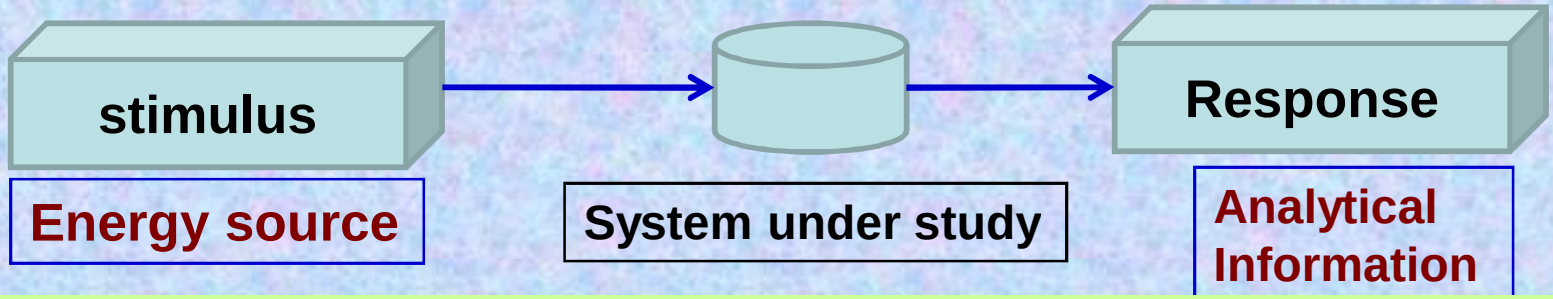


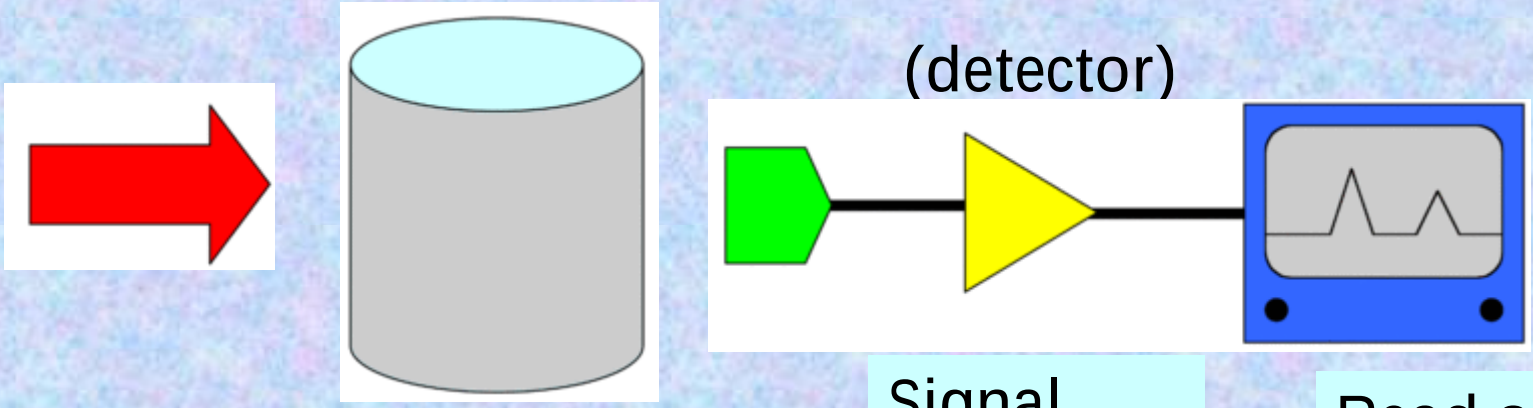


4 major components – 1) a sensor covert a solution property into weak signal 2) a signal processor –amplify the signal 3) a readout device 4) a power supply

Instrumental Analysis

Input Transducer

(detector)



Stimulus

- Light
- Heat
- Current
- Voltage

Sample

Signal Processor

- Amplifier
- digitizer

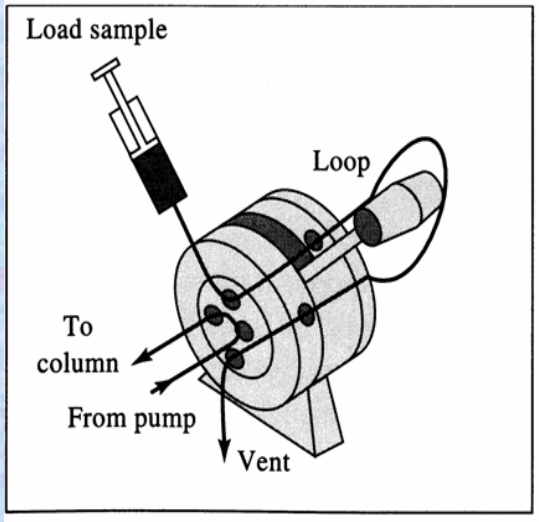
Read out

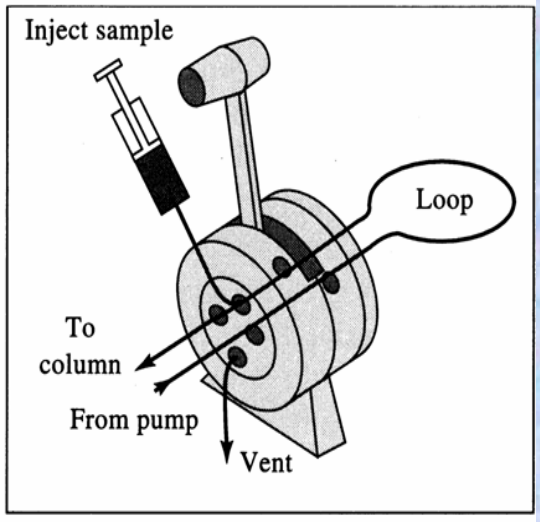
- Meter
- Plotter
- Computer

Introducing Samples & standards into an instrument

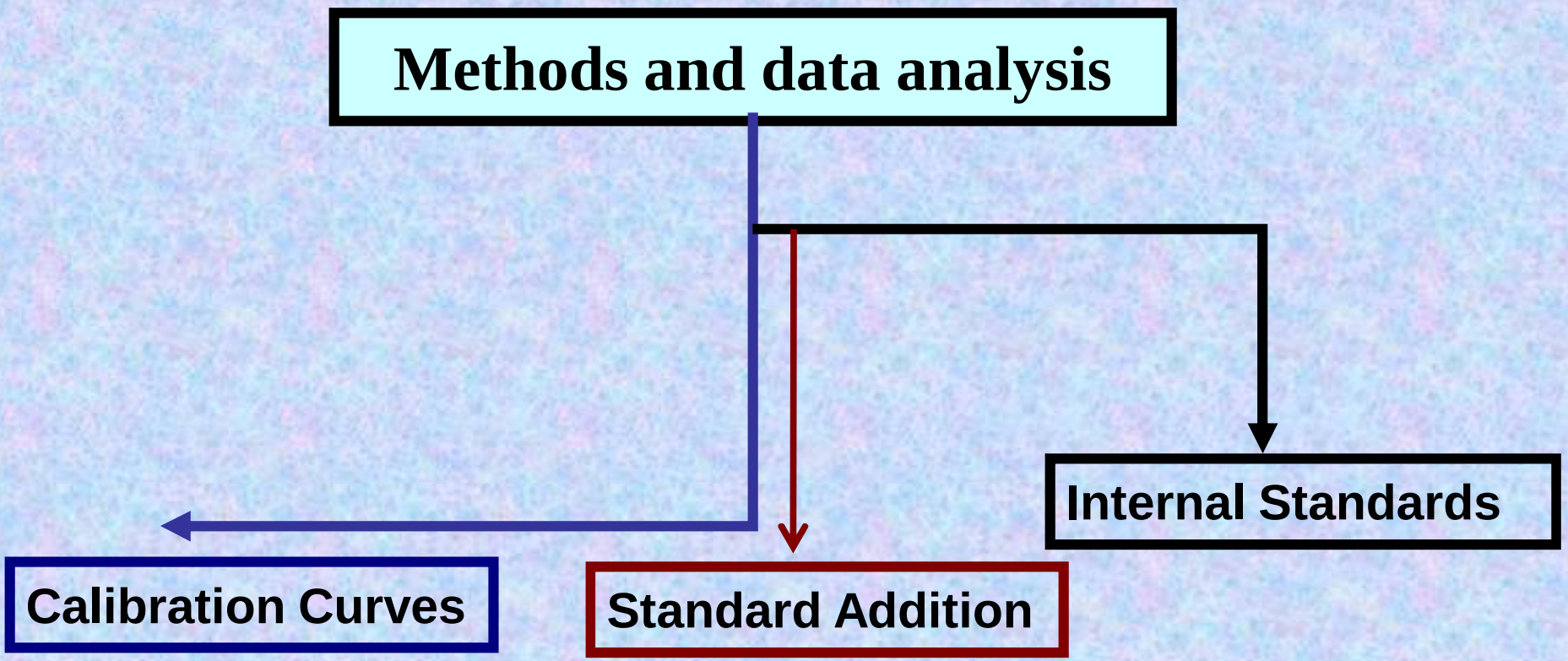
- **They can be sucked into the instrument**

- **Be placed inside the instrument**





- **Be injected with a syringe**



- **Standard Solutions;** solutions with known concentration of analyte
- **Blank solutions ;** Solutions containing all reagents except the analyte
- **Matrix**; the **components of a sample other than the analyte**

Methods and data analysis

Linear Calibration Curve

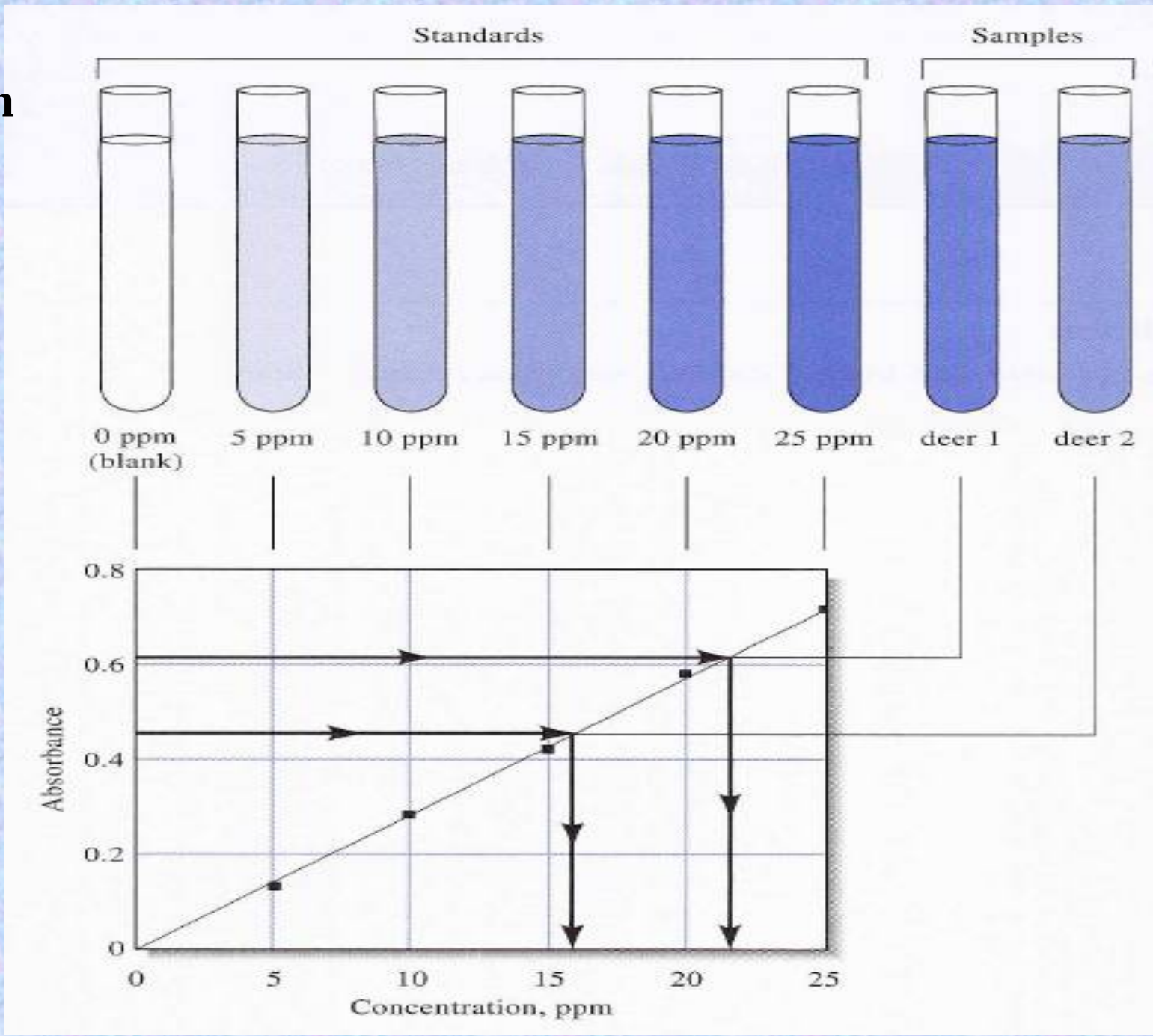
a graph of instrument signal versus analyte concentration

It can also be defined as:

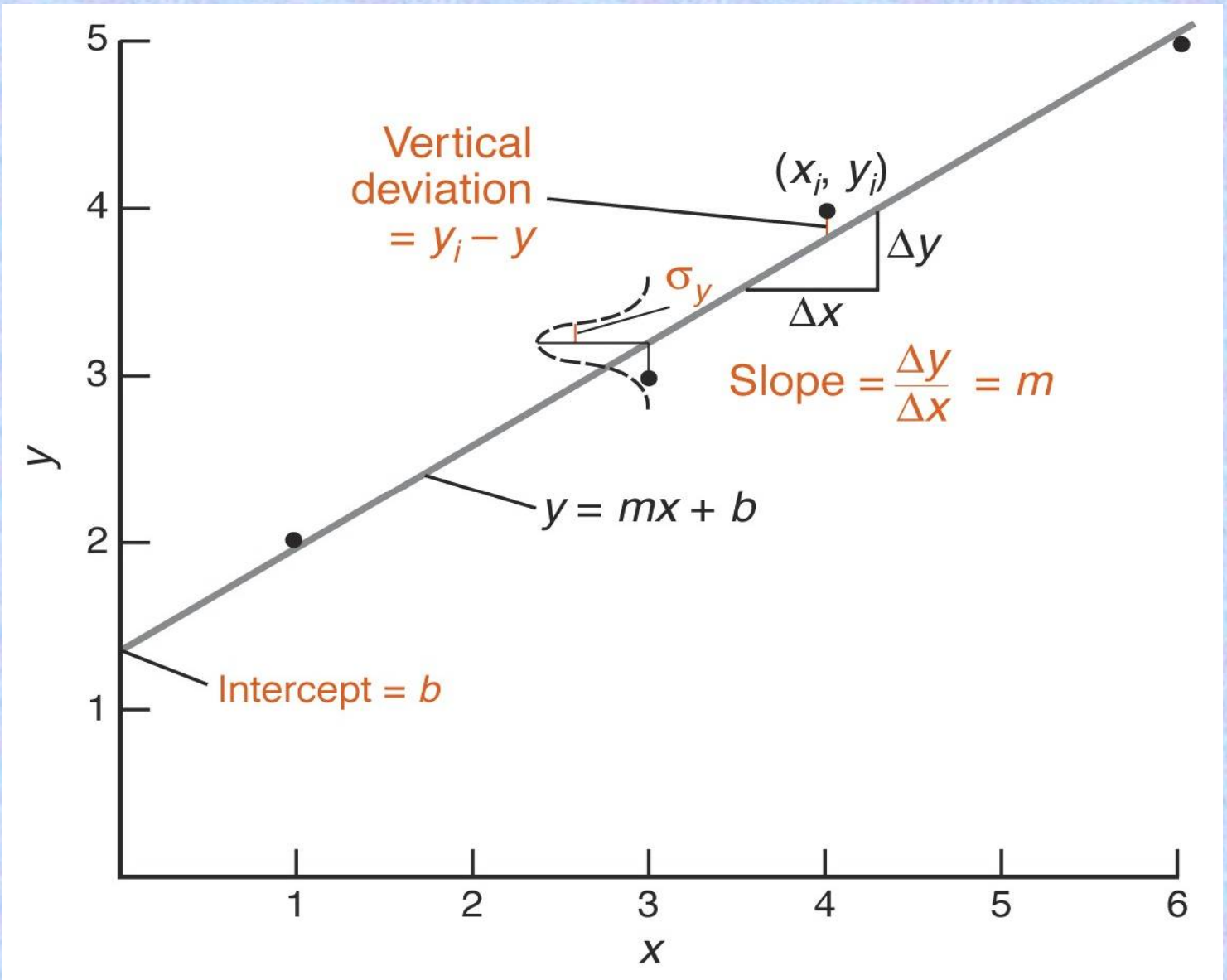
the response of a method to known quantities of analyte.

Several standards (with different concentration) containing exactly known concentrations of the analyte are measured and the responses recorded

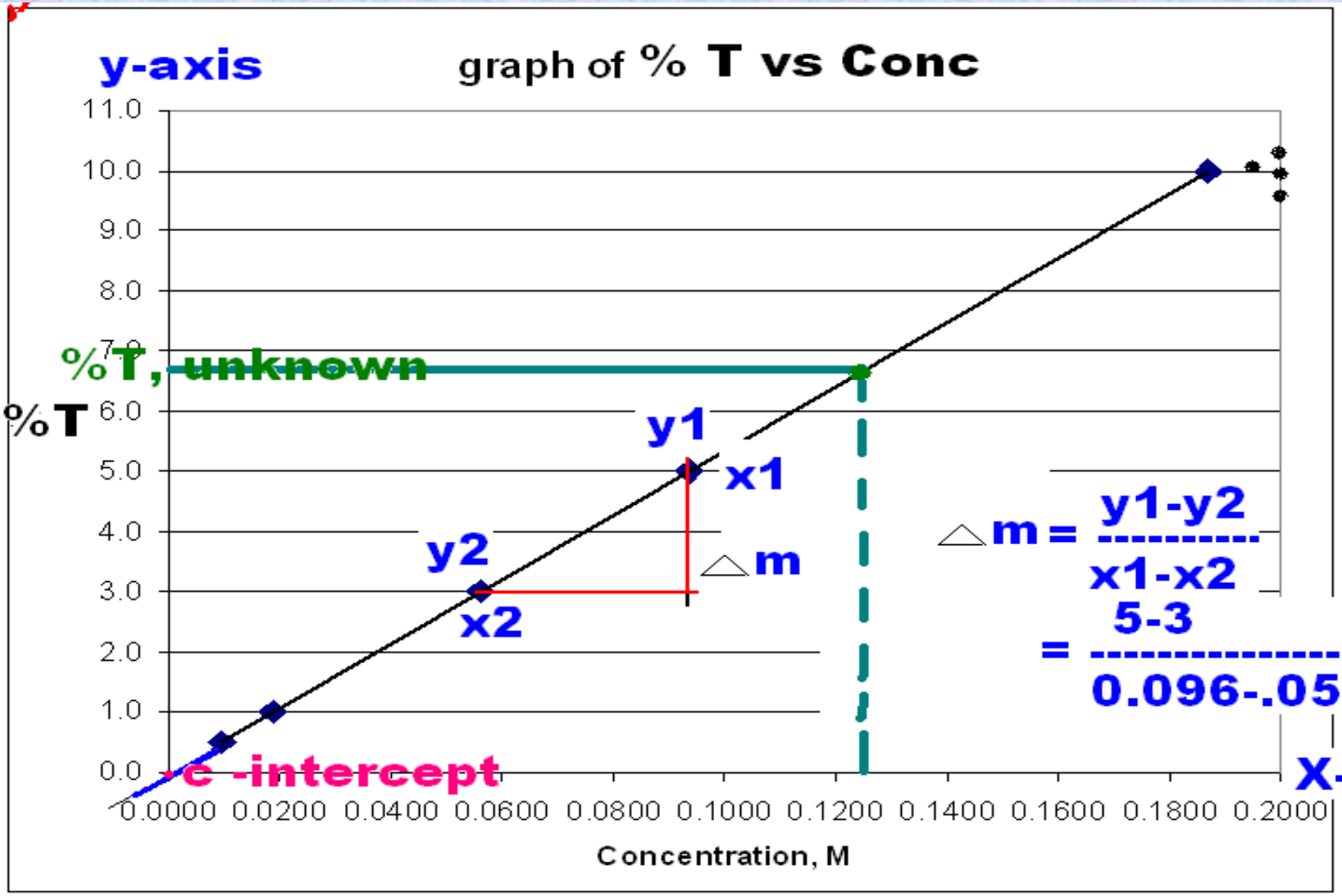
Linear calibration curve



Least Square Method – to get a linear calibration curve from scattered data



Least Square Method - Linear calibration curve

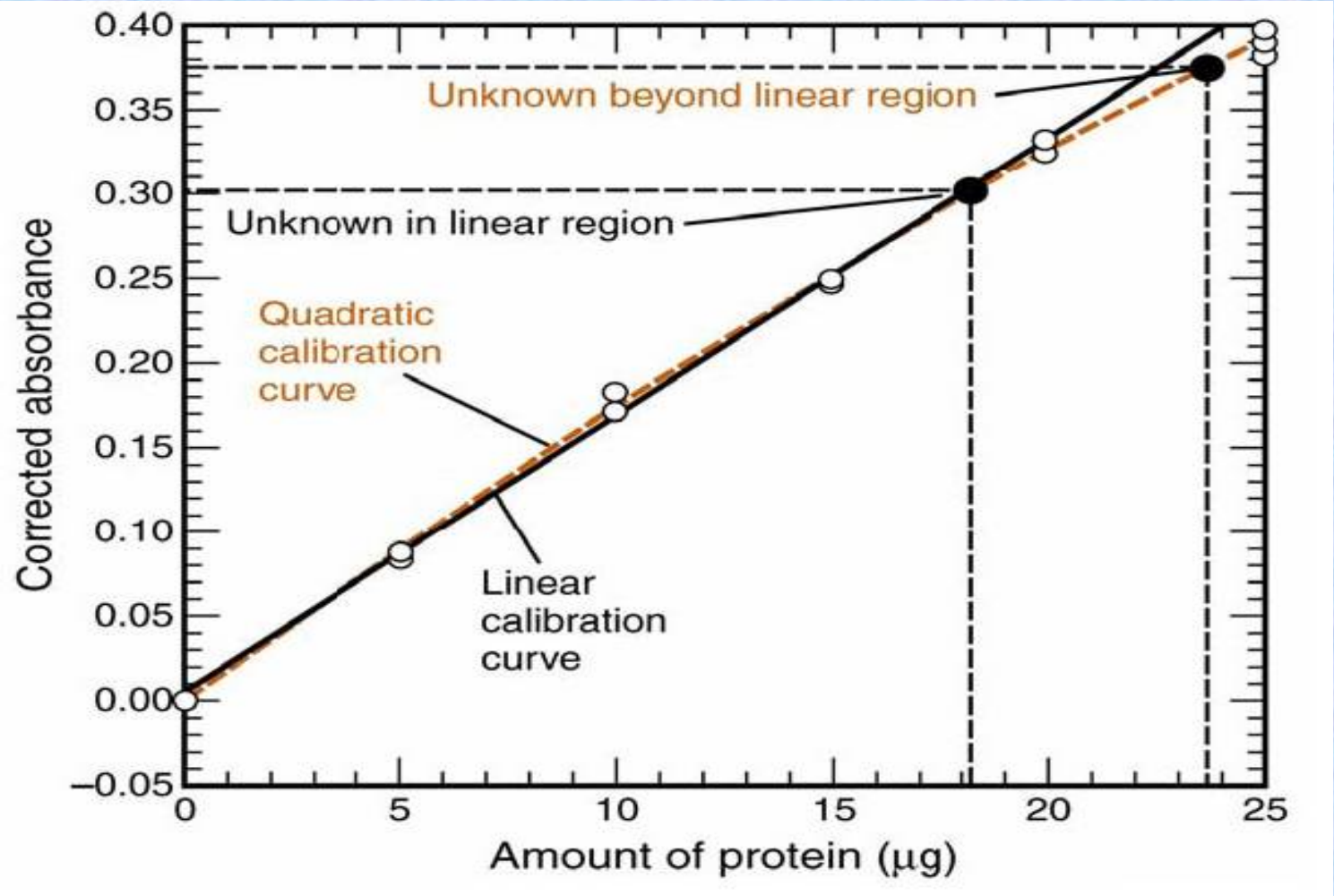


to solve for conc of unknown, x:

$$y = mx + c$$

$$x = \frac{y-c}{m}$$

Calibration Curve for Absorbance of a Protein



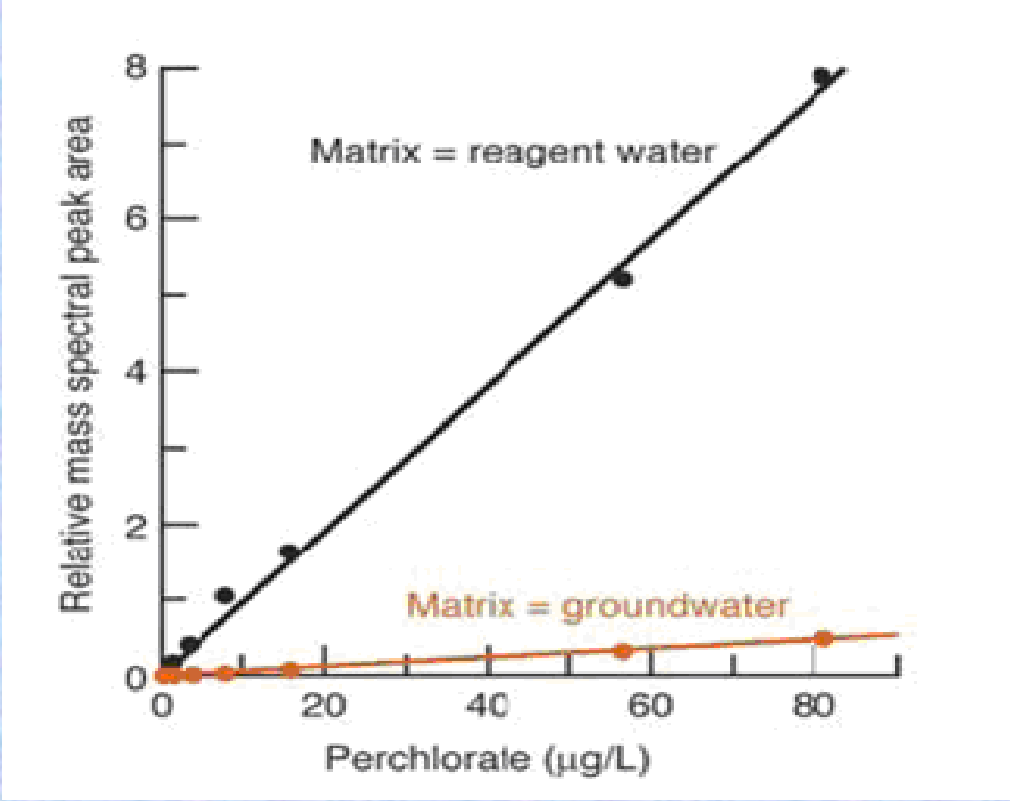
Methods and data analysis

2- Using Standard addition;

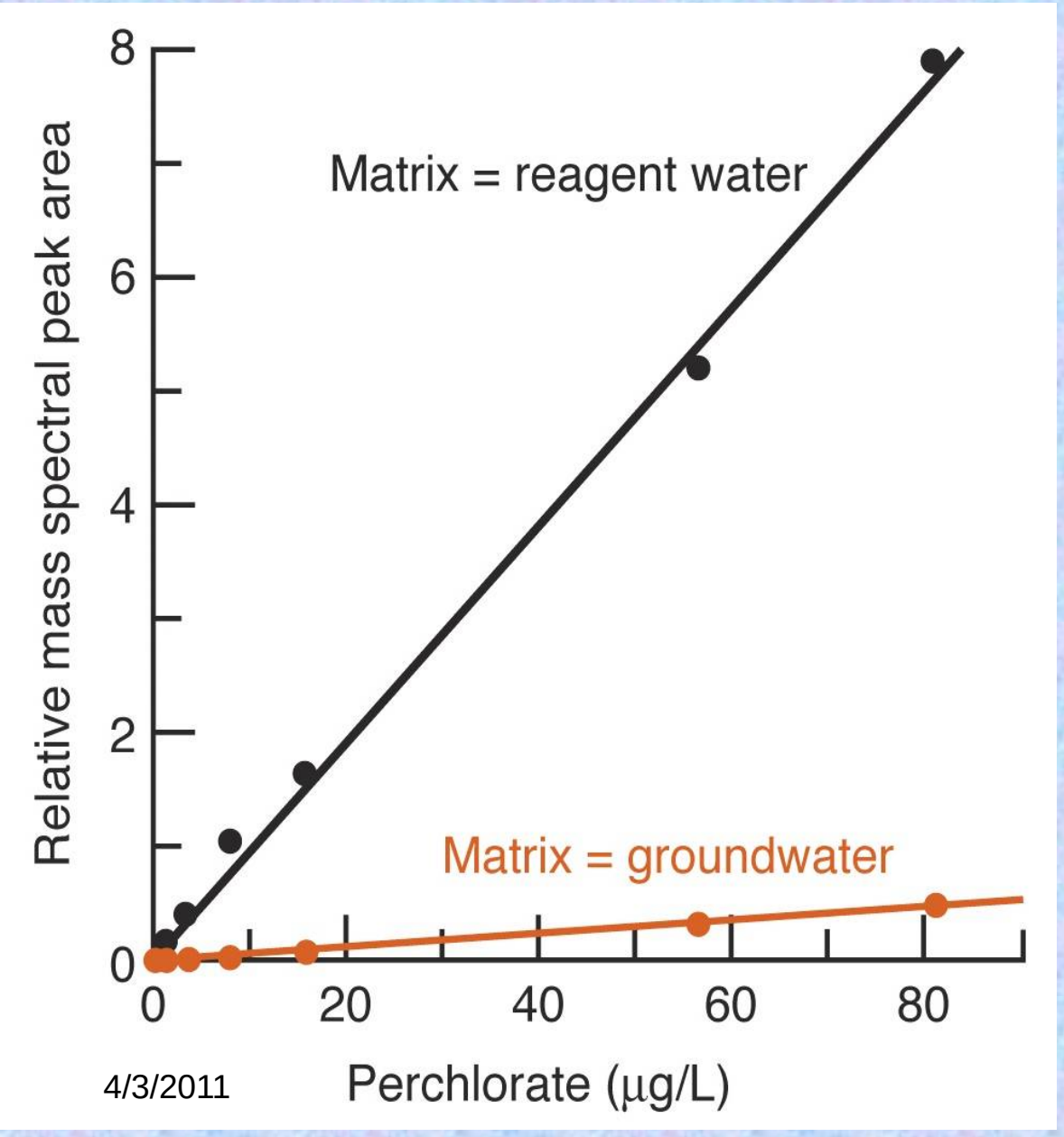
Known quantities of analyte are added to the unknown.

- When matrix effects can be substantial
- Standards are added directly to aliquots of the sample, therefore matrix components are the same.

- **Matrix;** anything in the sample other than the analyte.
- **Matrix effect;** a change in the analytical signal caused by anything in the sample other than analyte.

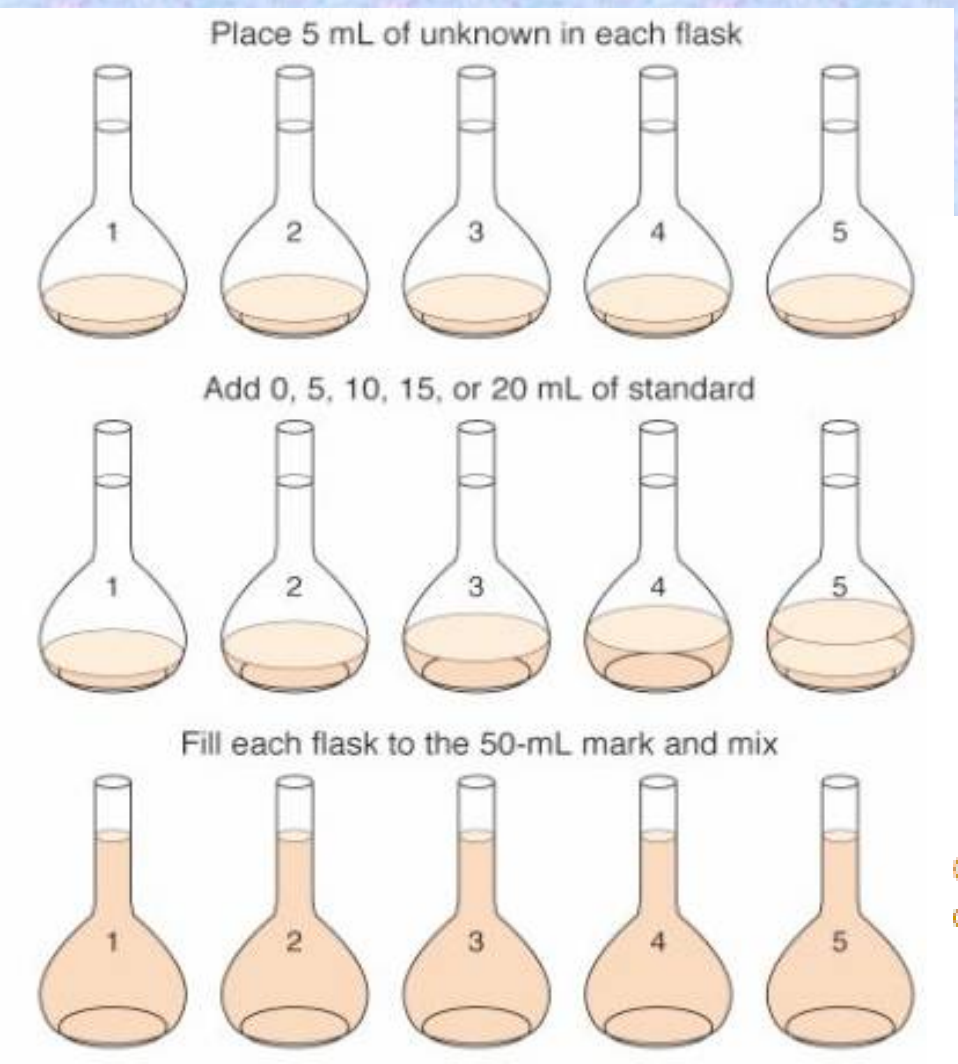


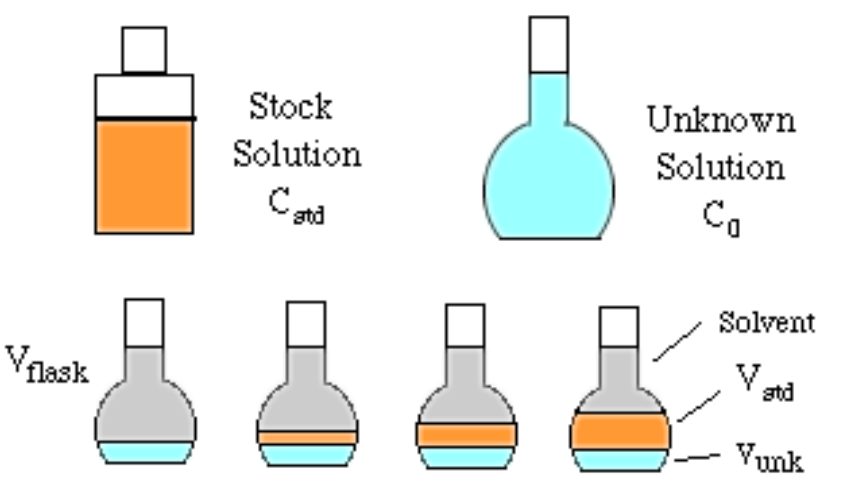
Calibration Curve for Perchlorate with Different Matrices

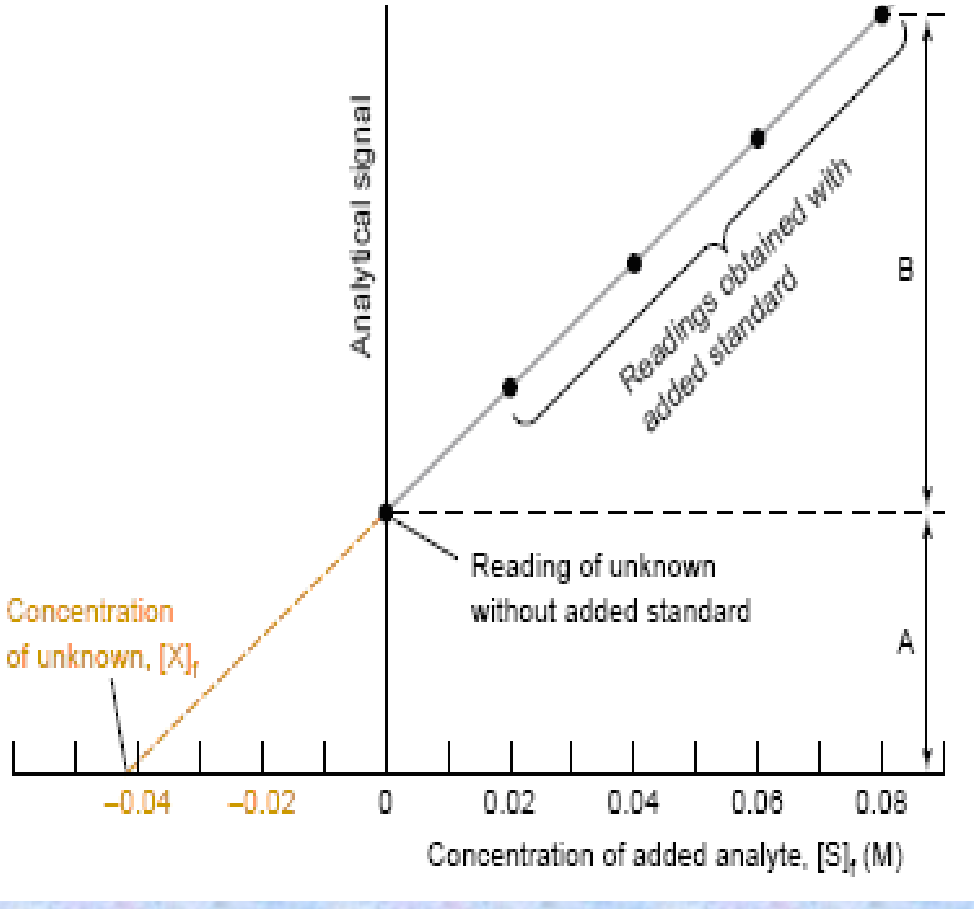


- Perchlorate (ClO_4^-) in drinking water affects production of thyroid hormone. ClO_4^- is usually detected by mass spectrometry (Ch. 22), but the response of the analyte is affected by other species, so you can see the response of calibration standards is very different from real samples.
- Application of standard addition method

Standard addition



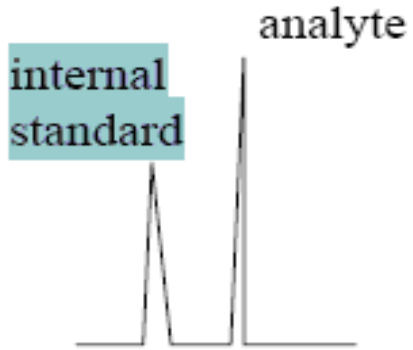




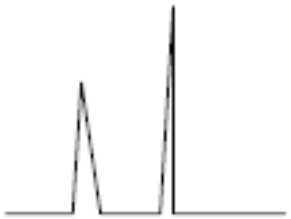
Methods and data analysis

3- Using Internal Standards is a substance different from analyte that is added in a constant amount to all samples, blanks and calibration standards in an analysis.

Run 1:



Run 2: You injected a slightly smaller volume.



$$\frac{\text{Area of analyte signal}}{\text{Concentration of analyte}} = F\left(\frac{\text{area of standard signal}}{\text{concentration of standard}}\right)$$

$$\frac{A_X}{[X]} = F\left(\frac{A_S}{[S]}\right)$$

Example

- In an experiment, a solution containing 0.0837 M analyte (X) and 0.0666 M standard (S) gave peak areas of A_x=423 and A_s=347 (areas in arbitrary units).

- To determine an unknown concentration of the analyte (X), 10.0 mL of 0.0146 M standard (S) was added to 10.0 mL of the unknown solution then the mixture was diluted to 25.0 mL in a volumetric flask. Analysis of this mixture resulted in peak areas of A_x=553 and A_s=582.

Answer

1

$$\frac{A_x}{[X]} = F\left(\frac{A_s}{[S]}\right)$$

$$\frac{423}{0.0837} = F\left(\frac{347}{0.0666}\right)$$

$$F = 0.970$$

2

$$[S] = (0.146\ M)\left(\frac{10.0}{25.0}\right) = 0.0584\ M$$

Initial Concentration

Dilution factor

3

$$\frac{A_x}{[X]} = F\left(\frac{A_s}{[S]}\right)$$

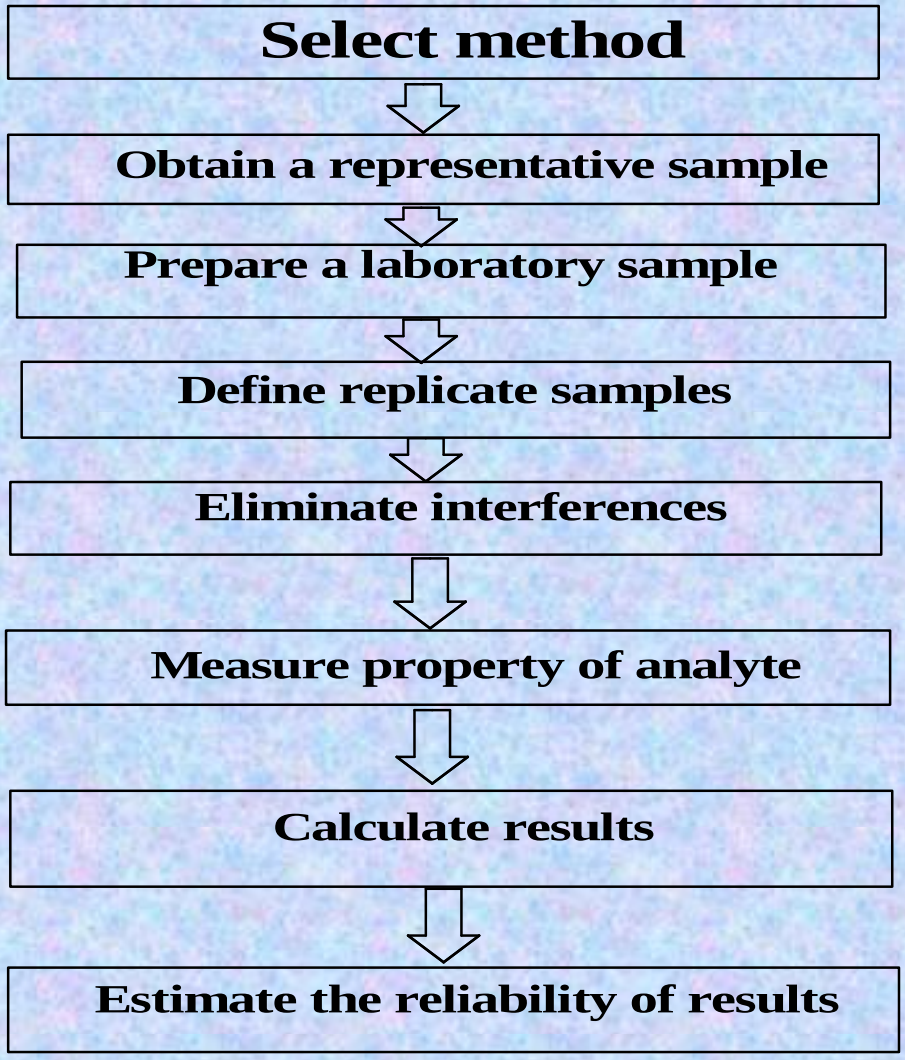
$$\frac{553}{[X]} = 0.970\left(\frac{582}{0.0584}\right)$$

$$[X] = 0.0572\ M$$

4

$$[X] = (0.0572\ M)\left(\frac{25.0}{10.0}\right) = 0.143\ M$$

General steps in any Chemical analysis



Instrumental Analysis

1- Obtain the sample.

2- Prepare the sample.

3- Prepare blanks and reference standards.

4- Obtain instrumental reading.

5-Use the data to calculate and/or plot the desired result.

6- Report and interpret the results.