

Chapter 1



Analytical objectives

1.1 Qualitative and Quantitative Analysis

- Qualitative analysis:
- Quantitative analysis:

1.2 The Analytical Process

- 1) defining the problem
- 2) obtaining a representative sample
- 3) preparing the sample for analysis
- 4) performing necessary chemical separations
- 5) performing the measurement
- 6) calculating the results and presenting the data

1) .

- a) what information is needed
- b) what type of sample is to be analyzed
- c) how the sample is to be obtained
- d) how much is needed
- e) how sensitive the method must be
- f) how accurate and precise it must be
- g) what separations may be required to eliminate interferences

2) sample: solid, liquid, or gas

homogeneous or heterogeneous

gross sample:

consists of several portions of the material to be test

laboratory sample:

a small portion of gross sample, made homogeneous

analysis sample: that actually analyzed

The larger the particle size, the larger should be the gross sample

Certain precautions should be taken in handling and storing sample to prevent or minimize contamination, loss, decomposition, or matrix change.

We must contamination of the sample by

- ① the container
- ② the atmosphere
- ③ light

3) Solid:

dry in an oven at 110-120 for 1-2 hours.


Sample size:

depend on the concentration of the analyte.

usually replicate samples are taken for analysis.

To adjust pH.

To add “ masking agent ”.

The analyte  may have to be reacted with a reagent to convert it to a form suitable for measurement or separation.

To run blanks.

4) .

- a. eliminate interferences
- b. preconcentrate

methods: precipitation, extraction, chromatography, distillation...

5) .

- a. the amount of analyte present
- b. the accuracy and precision required

techniques: selectivity, sensitivity, accuracy, precision, cost, rapidity.

Gravimetric analysis:

Volumetric or titrimetric analysis:

accuracy and precision: 0.1% (parts per thousand) for major constituents

Instrumental analysis:

more sensitive and selective

less precision ($\approx 1\%$)

Table 1.1

Absolute methods:

Ex: gravimetric analysis

Relative methods:

Ex: titrimetric analysis

6) relative composition:

ppt, ppm (parts per million)

precision

(mean value + standard deviation)

1.3 Validation of A method

errors: 1) random errors
2) systematic errors

1) random errors: random distribution of results

Ex: Gaussian distribution

2) systematic errors: in one direction

Ex: sample matrix, balance, dried sample

To validate a method: To analyze a standard reference material of known composition

The National Institute of Standards and Technology (NIST)

Standard Reference Materials (SRMs)

1.4 Range

□ Classification of Analytical Methods According to size of Sample

Method	Sample weight, mg	Sample volume μL ()
meso	>100	>100
semimicro	10-100	50-100
micro	1-10	<50
ultramicro	<1	

□ The constituents in the sample

major: $>1\%$

trace: $<0.1\%$

minor: $0.1-1\%$

ultratrace: $<10^{-4}\%$