

ANALYTICAL METHODOLOGIES

Qualitative Analysis – provides presence/absence information only (i.e., microbial screening)

Quantitative Analysis – provides a measure of concentration (a number plus units)

1. Classical methods – ‘wet chemistry’

- a) Gravimetric analysis (based on mass of analyte containing precipitate)
- b) Volumetric analysis (based on the volume of titrant required to reach end-point)

Classical methods are generally accurate and precise, but may be time consuming and are usually limited to analysis of reasonably high concentrations of analyte (i.e., > 1 ppm)

2. Instrumental methods measure some form of a sensor/detector signal (usually a voltage or current) that is related either directly or indirectly to the analyte concentration via a calibration process. Instrumental methods are generally accurate, precise and readily automated, however require careful calibration procedures.

- a) electrochemical devices convert chemical concentrations directly into a measured voltage potential or electric current.
examples: dissolved oxygen (DO) meter
 ion selective electrode (ISE)
- b) spectrophotometric instruments involve the interaction of the analyte with electromagnetic radiation (light)
examples: UV/vis (colourimetric)
 atomic absorption spectrophotometry (AAS)
 atomic emission spectrophotometry (AES)
 atomic fluorescence spectrophotometry (AFS)
- c) chromatographic instruments serve to separate and quantify members of a closely related series of analytes
examples: gas chromatography (GC)
 high performance liquid chromatography (HPLC)
 ion chromatography (IC)
 capillary electrophoresis (CE)

3. Tandem Instrumental methods combine instruments in sequence, often coupling chromatographic separation with sensitive and selective detectors, such as mass spectrometry (MS) example: GC-MS/MS

Gravimetric Analysis

A precipitating reagent is added to convert a soluble analyte species into an insoluble solid that contains the analyte in some known combination.

The solid precipitate is collected, dried and the mass recorded.

Mass of precipitate is directly related to the mass of the analyte present in a known volume of sample.

Volumetric Analysis

A measured volume of a reagent of known concentration (the titrant) is added to a sample until a specified change occurs – the ‘end-point’.

The volume and concentration of titrant are related the concentration of the analyte in sample titrated.

Types of titrations:

acid/base

oxidation/reduction

precipitation

complex ion formation

Electrochemical Devices

An electrical signal (voltage or current) is dependent on the concentration of the analyte.

Meters must be calibrated using solutions of known concentration (standard solutions) and measurements must be temperature compensated.

Spectrophotometric (Molecular and Atomic)

Molecular spectrophotometry (UV/vis): A chemical reagent is added to the sample, which selectively combines with the analyte resulting in some measureable colour change.

The intensity the colour (as measured by the absorbance of light) is related to the concentration of analyte by Beer's Law.

Atomic Spectrophotometry: A sample is 'atomized' in a flame or high temperature plasma. The atoms of each element absorb or emit discrete quanta of electromagnetic energy (light) characteristic of their individual identities. The intensity of light absorbed or emitted is related to the concentration of the element.

Chromatographic Techniques

An analyte or mixture of analytes are separated on the basis of selective partitioning between a mobile and stationary phase.

Analytes are then quantified with an internal non-selective detector.

Specialty and Tandem Instruments

Eg.

Turbidity meter (nephelometer)

Total Organic Carbon (TOC) Analyzer

Mass Spectrometer Detector for GC, HPLC and IC

Field Portable Equipment/Instruments

Eg.

Digital titrators, pH and DO meters

Portable spectrometers (Hach DR 2000)

Field GC-MS instruments