FDE 301 INSTRUMENTAL ANALYSIS

Instructors:

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Learning Objectives

- **The aim of the course is to introduce you to the modern methods of instrumental food analysis.**

- **You will gain ability to understand the fundamental principles behind the instrumental techniques, a working knowledge of instrument operation, and cognizance of the applications of instrumental analysis.**

- **This course should provide you with the fundamental background on the workings of many important types of instruments that you will likely encounter in the future, including absorption and emission spectroscopy, electrochemical techniques, and chromatographic separation.**

What is the course about?

- The instrumental analysis course is about the measurement of chemical systems using instruments.

- Each type of instrument has its own set of strengths and weaknesses that makes it suitable for some measurements, but not for others.

- Some methods are best for qualitative determinations and others are best for quantitative determinations.

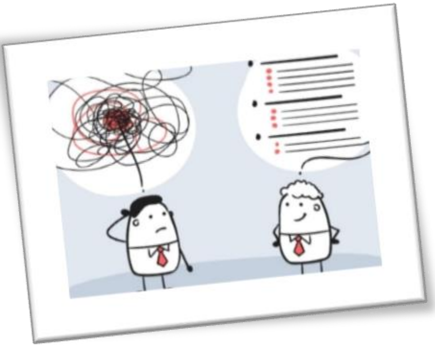
- During this course, you should develop an understanding of these advantages and disadvantages, and ultimately be able to suggest suitable instrumental methods for particular problems.

Instrumental analysis is the study of how best to use instruments to analyze the molecular composition of stuff.

Definition of Instrumental Analysis

The science and art of determining the composition of materials with the instrumental methods based on a physical property characteristic of a particular element or compound.

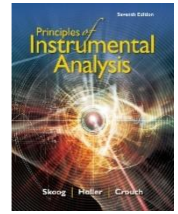
Instrumental analysis is a field of analytical chemistry that investigates analytes using scientific instruments.

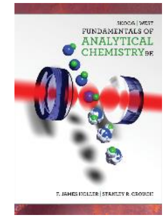


Course Outline

1	Classification of Analytical Methods, Introduction to Instrumental Analysis, Instrumental Methods in Food Analysis
2	Calibration of Instrumental Methods, pH meter
3	Basic principles of spectroscopic methods, UV-Visible Absorption Spectroscopy
4	Flourescence Spectroscopy
5	Infrared Spectroscopy
6	Atomic Spectroscopy: Atomic-Absorption Spectroscopy (AAS), Atomic-Emission Spectroscopy (AAS), Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)
7	Nuclear Magnetic Resonance Spectroscopy (NMR)
8	Mid-term exam
9	Basic principles of chromatographic Methods
10	Paper chromatography, Thin-Layer Chromatography (TLC)
11	High -Performance Liquid Chromatography (HPLC)
12	Gas Chromatography (GC)
13	Hyphenated Methods: Liquid Chromatography-Mass Spectrometry (LC-MS), Gas Chromatography-Mass Spectrometry (GC-MS)
14	Thermal Methods of Analysis: Thermogravimetric Analysis, Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC)

Reference Books





- Skoog, D.A., West, D.M., Holler, F.J., Crouch, S.R. (2013). **Fundamentals of Analytical Chemistry** (Ninth edition). Brooks/Cole, Cengage Learning, USA.
- Skoog, D.A., Holler, F.J., Crouch, S.R. (2018).**Principles of Instrumental Analysis** (Seventh edition). Cengage Learning, USA.
- Nielsen, S.S. (2010). **Food Analysis** (Fourth edition). Springer Science+Business Media, New York, USA.
- Harvey, D. (2000). **Modern Analytical Chemistry**. McGraw-Hill Companies.
- Harris, D.C., Lucy, C.A. (2016). **Quantitative Chemical Analysis** (Ninth edition). W. H. Freeman and Company, New York, USA.
- Cheung, P.C.K., Mehta, B.M. (2015). **Handbook of Food Chemistry (Chapter 8-Instrumental Food Analysis)**. Springer-Verlag GmbH Berlin Heidelberg.

Analytical chemistry

- **Analytical chemistry** deals with methods for determining the chemical composition of samples of matter.
- The main purpose of the analytical chemistry is to identify or quantify analytes in the sample.
- **Analytes** are the components of a sample that are determined.

"TODAY EVERYONE HAS TO KNOW 'WHAT'S IN THE FOOD?', 'WHAT'S IN THE WATER?', 'WHAT'S IN THE AIR?' THIS IS TRULY THE 'GOLDEN AGE OF ANALYTICAL CHEMISTRY.'"

Classification of Analytical Methods

Analytical chemistry is the study of the separation, identification and quantification of the chemical components of natural and artificial materials. The separation of components is often performed prior to analysis.

- **Qualitative Analysis** (What is it?)

Qualitative analysis reveals the *identity* of the elements and compounds (atomic or molecular species or the functional groups) in a sample.

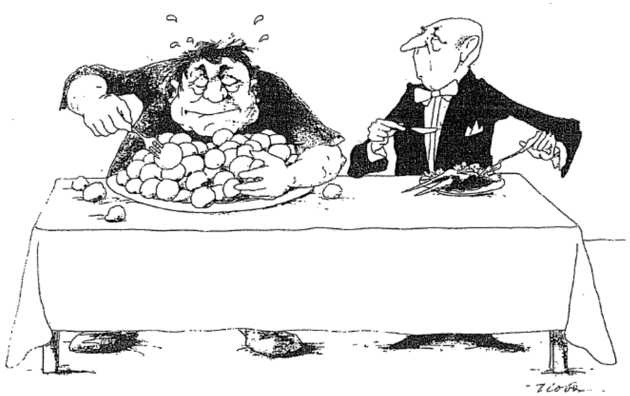
- **Quantitative Analysis** (How much is it?)

Quantitative analysis indicates the *amount* of each substance in a sample.

Analytical chemistry gives answer to two important questions.

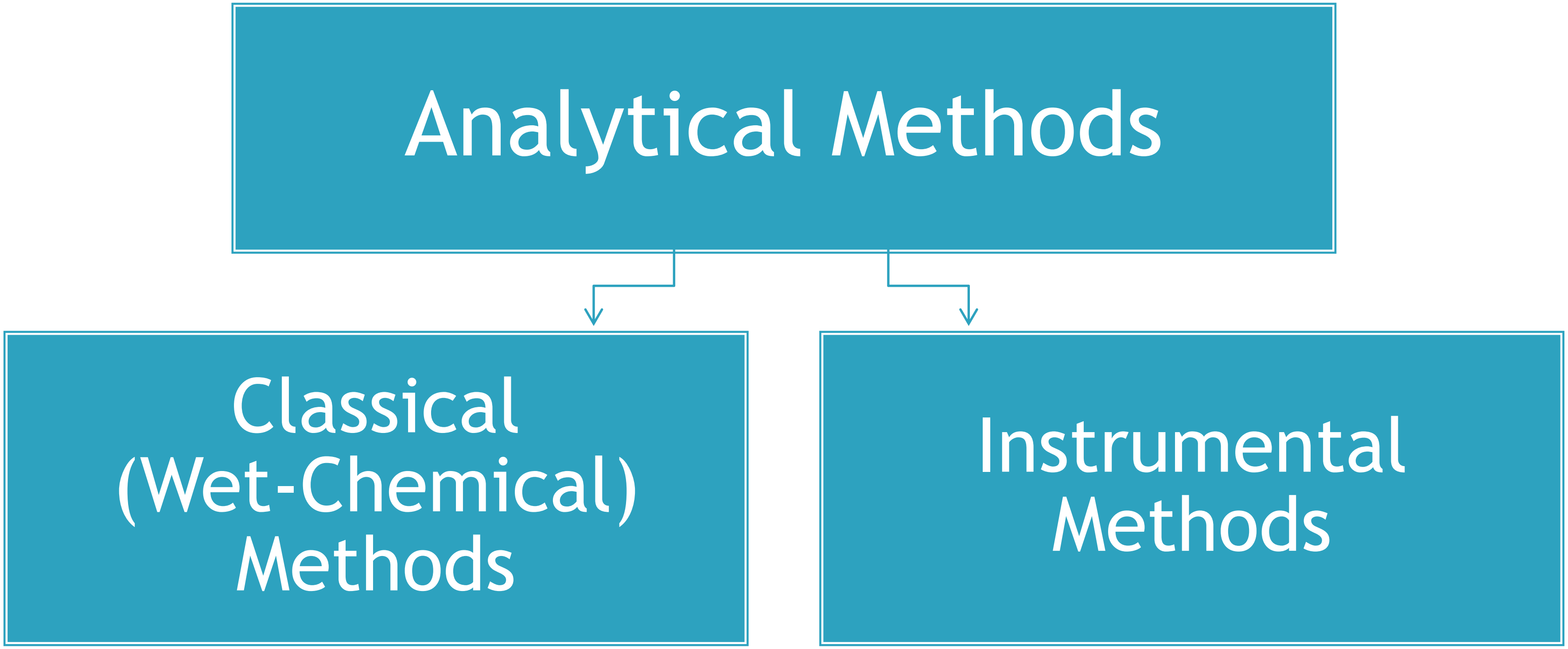
✓ What is it? (Qualitative analysis)

✓ How much is it? (Quantitative analysis)



Quantitative analysis   Qualitative analysis

Classification of Analytical Methods



Classical Methods

- Separation of the analytes
  - Precipitation
  - Extraction
  - Distillation

- Qualitative analyses
  - Recognation of the products arising from treatment of the seperated components with reagents by their colors, their boiling or melting points, their solubilities in a series of solvents, their odors, their optical activities, or their refractive indexes.
- Quantitative analyses
  - Gravimetric measurements
  - Volumetric measurements

<u>In gravimetric measurements</u>, the mass of the analyte or some compound produced from the analyte was determined.

<u>In volumetric, also called *titrimetric*, procedures</u>, the volume or mass of a standard reagent required to react completely with the analyte was measured.

Instrumental Methods

- ***Instrumental methods of analysis*** are based on measurement of physical properties of analytes such as conductivity, electrode potential, light absorption or emission, mass-to-charge ratio, and fluorescence and use of electronic, thermal or optical principles for determination of presence or concentration of analytes.

- Furthermore, highly efficient chromatographic and electrophoretic techniques began to replace distillation, extraction, and precipitation for the separation of components of complex mixtures prior to their qualitative or quantitative determination.

- These methods are able to analyze **low concentrations of a substance** and are able to analyze **more than one substance simultaneously** using either a single instrument or a combination of instrument.

- These newer methods for separating and determining chemical species are known collectively as ***instrumental methods of analysis***.

Comparison of Classical and Instrumental Methods

- In classical methods, no mechanical or electrical instruments are used except simple apparatus. Chemical reactions are used to quantify the analyte of interest.
- Instrumental methods of analysis depend on instruments to carry measurements or to perform the entire analysis.

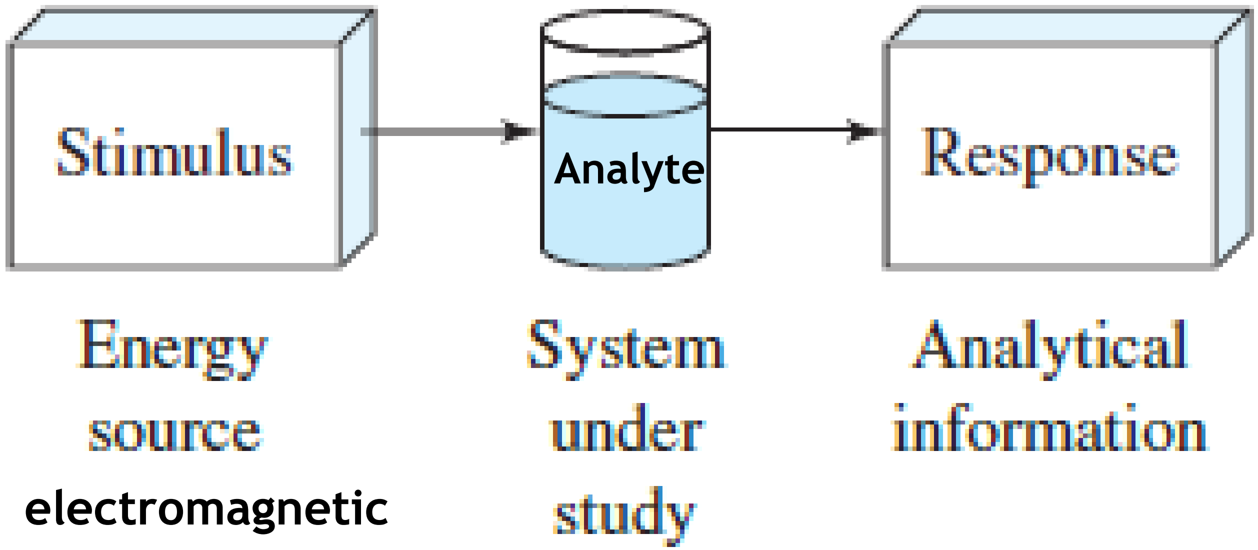
- In classical analysis, the signal depends on the chemical properties of the sample: a reagent reacts completely with the analyte, and the relationship between the measured signal and the analyte concentration is determined by chemical stoichioimetry.
- In instrumental analysis, some physical property of the sample is measured, such as the electrical potential difference between two electrodes immersed in a solution of the sample, or the ability of the sample to absorb light.

- Classical methods are most useful for accurate and precise measurements of analyte concentrations at the **0.1% level or higher**.
- On the other hand, some specialized instrumental techniques are capable of detecting individual atoms or molecules in a sample. Analysis at the **ppm (μg/mL)** and even **ppb (ng/mL) level** is routine.

Classical vs. Instrumental

	Classical Methods	Instrumental Methods
	Accounts for about 10% of all the current analytical work.	Accounts for about 90% of all the current analytical work.
Reliability and Confidence	Low accuracy, low specifity (complex matrix is a challenge)	More confident in trace analysis (high accuracy, precision, selectivity, sensitivity)
Sample size	Large	Low
Skill required for operator	Easy to use. No need for advance training	Need specific training but easily automated
Cost and availability of equipment	Low cost equipments but each analyte need specific reagents and analytical method	High cost, occupy large space, always need instrument calibration and reference standard
Number of samples/day and per sample cost	Low number of samples with moderate cost	Large number with low cost

Block diagram of an instrumental measurement

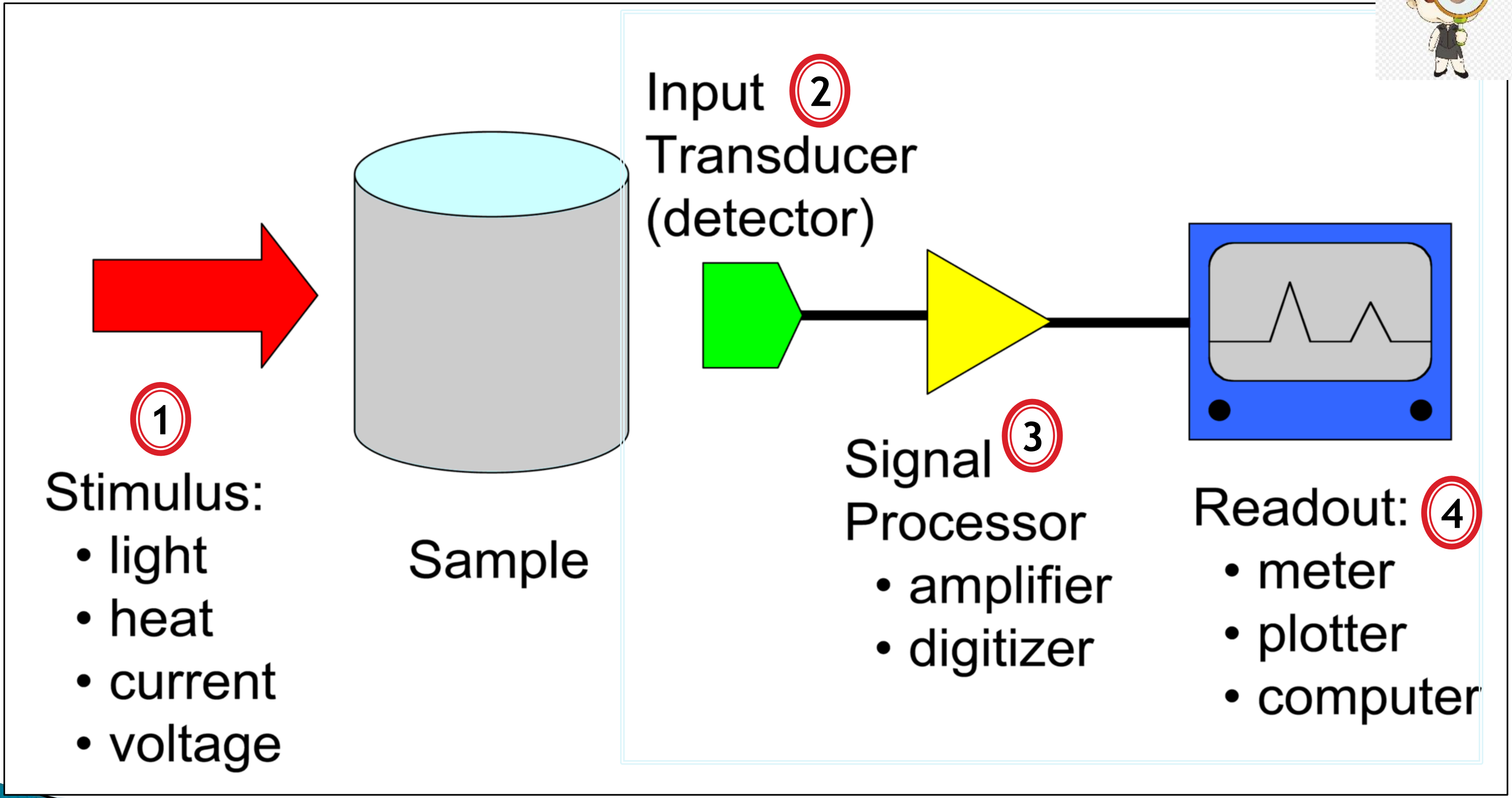


- electromagnetic
- electrical
- mechanical
- nuclear energy

An example:
Passing a narrow band of wavelengths of visible light through a sample to measure the extent of its absorption by the analyte.

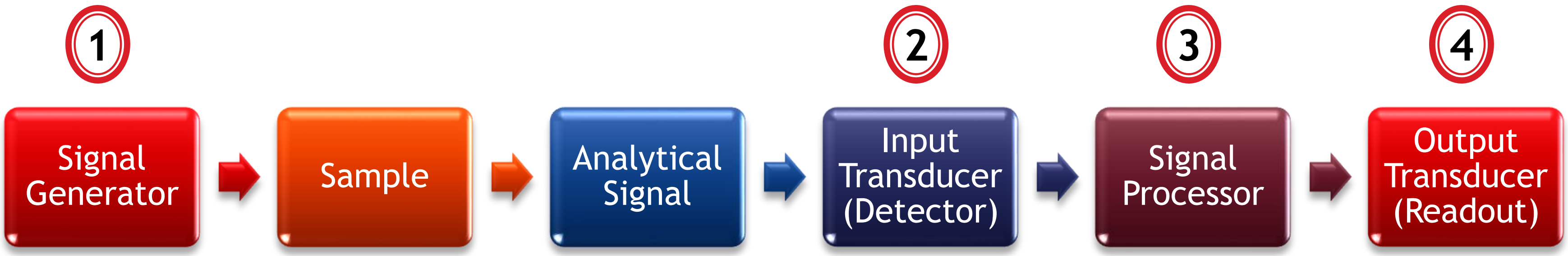
- The intensity of the light is determined before and after its interaction with the sample, and the ratio of these intensities provides a measure of the analyte concentration.

Block diagram of an analytical instrument showing the stimulus and measurement of response



https://commons.wikimedia.org/wiki/File:Analytical_instrument.png

Basic Instrument Components



Basic Instrument Components

1. Energy Source, Stimulus (Signal Generator): produces some forms of energy or mass that is relevant to the measurement at hand
   Sample holder or "cell": contains the sample with your analyte of interest
   Discriminator: selects the desired signal from the source or the sample
2. Input transducer (detector): detects the signal from the sample, source or discriminator
3. Processor: manipulates the signal electronically or mechanically to produce some useful data
4. Readout: displays the signal in some useful form

Need for Food Analysis?

- The food is analyzed for a variety of reasons;
  - complience with legal and labeling requirements
  - assessment of product quality
  - determination of nutritive value
  - detection of adulteration
  - research and development

Why do We Need Instrumentation in Food Analysis?

- Due to complex nature of food matrix, it often becomes impossible to accurately analyze one component in the presence of others using the classical method of analysis.

- More often than not, interferences are encountered during the measurement of minor components in the presence of the components present in bulk quantities.

- All this may lead to inaccurate and unreliable results and sometimes erroneous and false results because of lack of specificity and sensitivity of classical method. **Therefore, in order to achieve the reliability of results, today the instrumental analytical techniques have become mandatory in development, quality control and safety, exports of food products and meeting the regulatory norms of food products.**

Instrumental Methods in Food Analysis

- Spectroscopic Methods
- Chromatographic Methods
- Hyphenated Methods
- Thermal Methods of Analysis

Spectroscopic Methods

- **UV-Visible Absorption Spectroscopy**
- **Atomic Spectroscopy**
  - Atomic-Absorption Spectroscopy (AAS)
  - Atomic-Emission Spectroscopy (AAS)
  - Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)
- **Flourescence Spectroscopy**
- **Infrared Spectroscopy**
- **Nuclear Magnetic Resonance Spectroscopy (NMR)**
- **Mass Spectrometry**

Chromatographic Methods

- High -Performance Liquid Chromatography (HPLC)
- Gas Chromatography (GC)
- Paper Chromatography
- Thin-Layer Chromatography (TLC)

Hyphenated Methods

- Liquid Chromatography-Mass Spectrometry (LC-MS)
- Gas Chromatography-Mass Spectrometry (GC-MS)

Thermal Methods of Analysis

- Thermogravimetric Analysis
- Differential Thermal Analysis (DTA)
- Differential Scanning Calorimetry (DSC)