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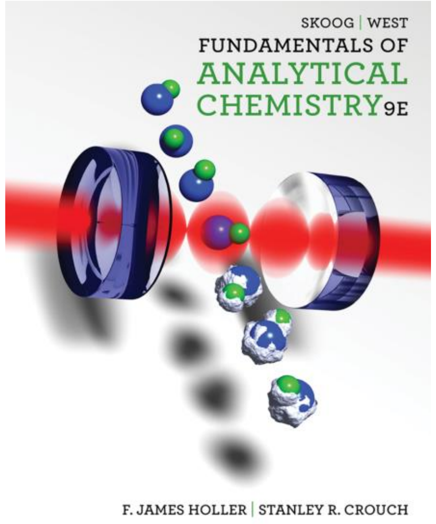

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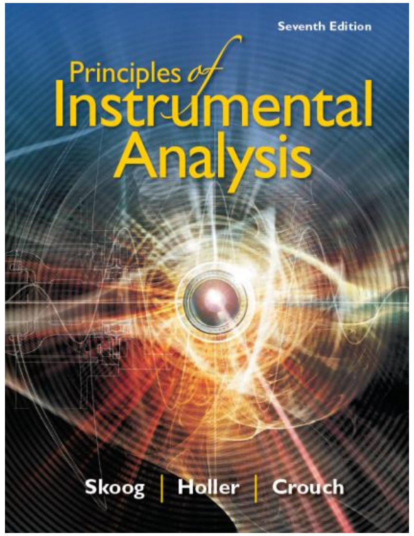


3.

ATAKOL's lecture notes

1. Skoog, D.A., West, D.M., Holler, J.F., Crouch, S.R. 2013. Fundamentals of Analytical Chemistry (9E). Cengage Learning, Belmont, USA.
2. Skoog, D.A., Holler, J.F., Crouch, S.R. 2016. Principles of Instrumental Analysis (7E). Cengage Learning, Boston, USA.
3. Prof. Dr. Orhan ATAKOL's lecture notes.

Introduction to Instrumental Analysis

In all instrumental analysis methods:

- The analyte is stimulated with a source.
- The analyte interacts with the signals from the source. During this process, some changes occur on the analyte or the analyte begins to emit unique signals.
- Qualitative or quantitative measurements made using these changes or signals, are called instrumental analysis.

Instrumental analyzes are divided into 5 basic classes:

1- Spectroscopic methods

2- Radiochemical methods

3- Electrochemical methods

4- Thermal methods

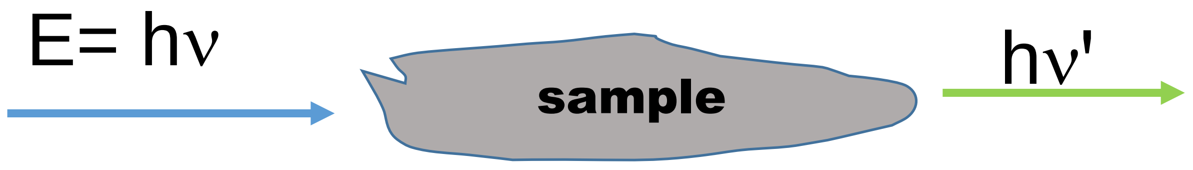
5- Chromatographic methods

With the rapid development of technology in the 1980s, computers began to take part in instrumental analysis. So that,

- The reproducibility of the instrumental analyzes and the signal / noise ratio levels in the devices have improved.
- The results from the analytical instruments have reached more reliable levels.
- Some instrumental analysis methods have become combined with each other such as GC-MS (Gas Chromatography- Mass Spectrometry), LC-MS (Liquid Chromatography- Mass Spectrometry), ICP-MS (Inductively Coupled Plasma- Mass Spectrometry).

Spectroscopic Methods

Spectroscopic methods are the instrumental analysis methods resulting from the interaction of matter (analyte) and radiation. In other words, the analyte (electrons) is stimulated by radiation with a certain energy. As a result of this stimulation, the analyte gives its own unique signals, often as a radiation. The groups of analytical methods based on the measurement of the intensity or wavelength of these signals are called spectroscopic methods. Spectroscopic methods constitute more than half of the instrumental analysis methods.



Radiochemical Methods

Radiochemical methods are similar to those used in spectroscopic methods, which are based on the stimulation of atomic nuclei in the analyte by using radiation and detecting signals from the analyte (radiation). In fact, they are similar to spectroscopic methods. However, since the magnitude of the energy used to stimulate the analyte is much higher than spectroscopy, they are included in a separate analysis group. However, there is no clear limit between these methods.

Electrochemical Methods

Electrochemical methods are based on the measurements electrical properties of a solution of the analyte, when it is made part of an electrochemical cell, by analog or digital methods.

Electroanalytical techniques are capable of producing low detection limits and a wealth of characterization information describing electrochemically accessible systems. Such information includes the stoichiometry and rate of interfacial charge transfer, the rate of mass transfer, the extent of adsorption or chemisorption, and the rates and equilibrium constants for chemical reactions.

Thermal Methods

Thermal analysis methods are based on the measurement of physical and chemical changes caused by temperature on the analyte and give more macro results than spectroscopic methods.

Usually, the substance is subjected to a controlled temperature program during the analysis.

Although there are more than a dozen thermal analysis techniques, it can be confined to four methods that provide primarily chemical information rather than physical information about samples of matter. These methods include thermogravimetric analysis (TGA), differential thermal analysis (DTA), differential scanning calorimetry (DSC), and microthermal analysis.

Chromatographic Methods

Chromatography encompasses a diverse and important group of methods that allow the separation, identification, and determination of closely related components of complex mixtures; many of these separations are impossible by other means.

In all chromatographic separations the sample is dissolved in a *mobile phase*, which may be a gas, a liquid, or a supercritical fluid.

According to the mobile phases used, the chromatographic methods are divided into three general categories: gas chromatography (GC), liquid chromatography (LC) and supercritical fluid chromatography (SFC).

Introduction to Spectrochemical Methods

Spectroscopic analytical methods are based on measuring the amount of radiation produced or absorbed by molecular or atomic species of interest. Spectroscopic methods can be classified according to the region of the electromagnetic spectrum used or produced in the measurement. Spectrochemical methods have provided perhaps the most widely used tools for the elucidation of molecular structure as well as the quantitative and qualitative determination of both inorganic and organic compounds.

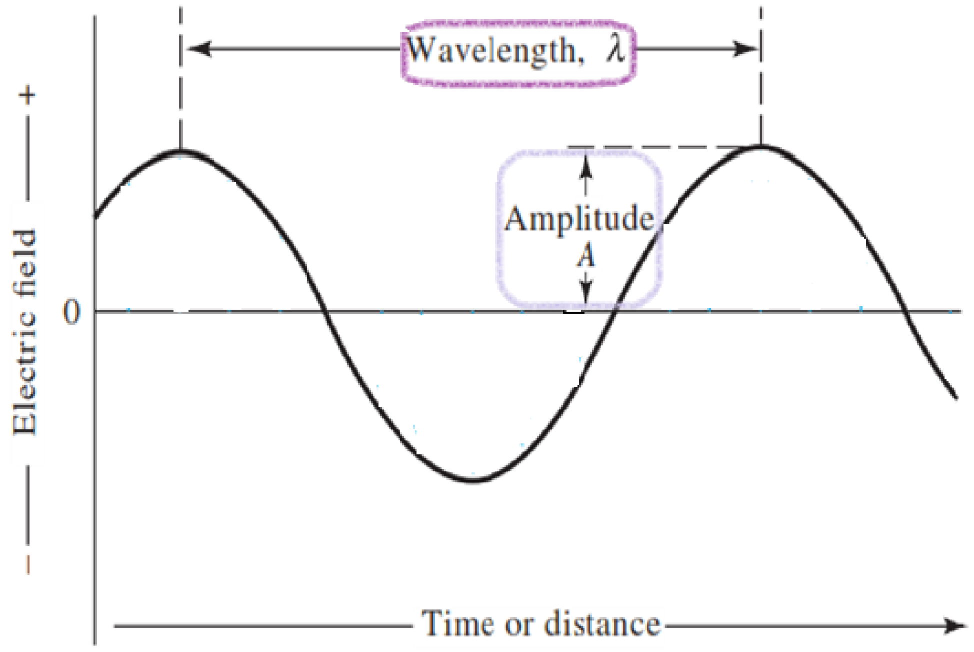
Properties of electromagnetic radiation

Electromagnetic radiation is a form of energy that is transmitted through space at enormous velocities. We can call electromagnetic radiation in the UV/visible and sometimes in the IR region, **light**, although strictly speaking the term refers only to visible radiation.

Electromagnetic radiation can be described as **a wave** with properties of wavelength, frequency, velocity, and amplitude.

The wave model fails to account for phenomena associated with the absorption and emission of radiant energy. Electromagnetic radiation also can be treated as discrete packets of energy or particles called **photons or quanta**.

Wave Characteristics of Light



- **Amplitude**: a vector quantity that provides a measure of the electric or magnetic field strength at a maximum in the wave.
- **Period**: the time in seconds for successive maxima or minima to pass a point in space.
- **Frequency**: the number of oscillations that occur in one second. The unit of frequency is the hertz (Hz), which corresponds to one cycle per second, that is, 1 Hz=1 s^{-1}.
- **Velocity**, υ: Of the wave front through a medium depends on both the medium and the frequency.
- **Wavelength**, λ: is the linear distance between successive maxima or minima of a wave.

$$\upsilon = \nu\lambda$$

The Speed of Light

* In a vacuum, light travels at its maximum velocity. This velocity, which is given the special symbol ***c.***

$$c = \nu\lambda = 3.00 \times 10^8 \text{ ms}^{-1} = 3.00 \times 10^{10} \text{ cms}^{-1}$$

In a medium containing matter, light travels with a velocity less than c because of interaction between the electromagnetic field and electrons in the atoms or molecules of the medium. Since the frequency of the radiation is constant, the wavelength must decrease as the light passes from a vacuum to a medium containing matter.

* The **wavenumber**, , is another way to describe electromagnetic radiation. It is defined as the number of waves per centimeter and is equal to 1/λ.

* The **radiant power**, P, in watts (W) is the energy of a beam that reaches a given area per unit time.

* The **intensity** is the radiant power-per-unit solid angle.

Particle Characteristics of Light : Photons

- In many radiation/matter interactions, it is useful to emphasize the particle nature of light as a stream of photons or quanta
- A photonis a particle of electromagnetic radiation having zero mass and an energy of *hv*
- We relate the energy of a single photon to its wavelength, frequency, and wavenumber by

$$E = h\nu = \frac{h\,c}{\lambda} = h\,c\,\bar{\nu}$$