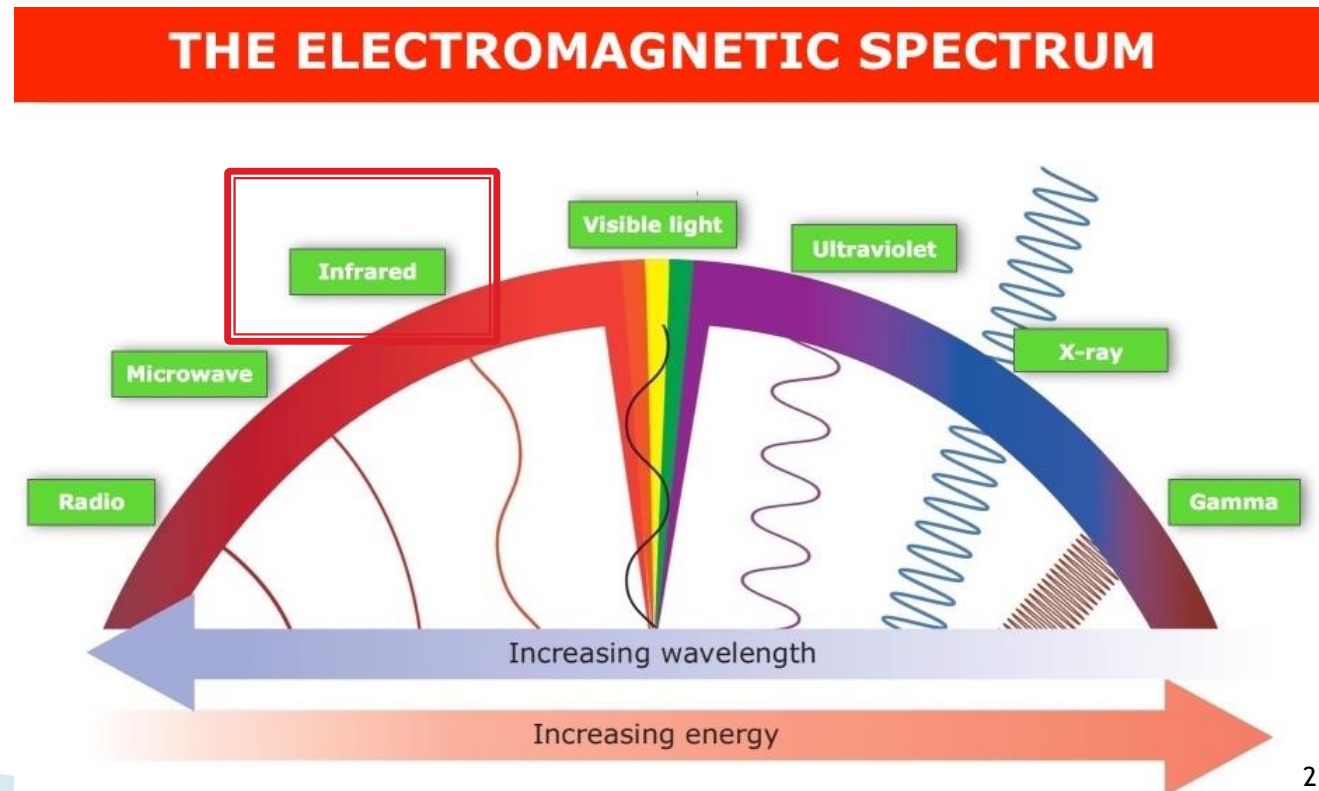


INFRARED SPECTROSCOPY

Introduction

- ▶ **IR spectroscopy** is the study of the interaction of IR radiation with matter as a function of photon frequency.
- ▶ **IR radiation** is electromagnetic radiation (light) with a longer wavelength than visible light.
- ▶ **IR radiations** refer broadly to that part of electromagnetic spectrum between visible and microwave region.



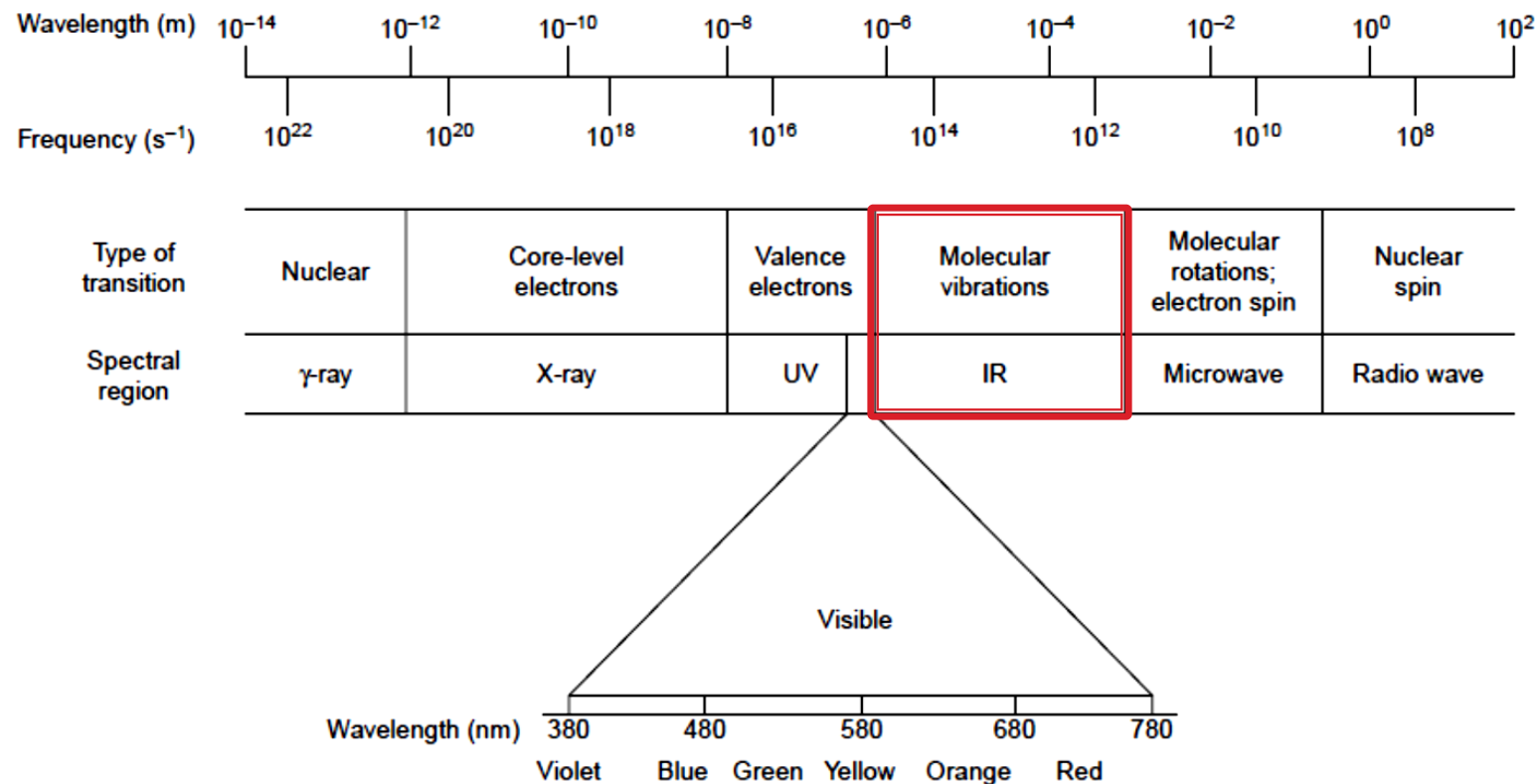
Introduction

- ▶ **Infrared (IR) spectroscopy** refers to measurement of the absorption of different frequencies of IR radiation by foods or other solids, liquids, gases.
- ▶ It is an important tool used to identify functional groups in organic compounds.
- ▶ **IR spectroscopy** is used widely in the food industry for both qualitative and quantitative analysis of ingredients and finished foods.
- ▶ **IR spectroscopy** measures the absorption of radiation in the near- ($\lambda = 0.8\text{-}2.5\ \mu\text{m}$) or mid- ($\lambda = 2.5\text{-}15\ \mu\text{m}$) IR regions by molecules in food or other substances.
- ▶ **IR radiation** is absorbed as molecules change their vibrational energy levels.

Principles of IR Spectroscopy

The IR Region of the Electromagnetic Spectrum

- ▶ **Infrared radiation** is electromagnetic energy with wavelengths (λ) longer than visible light , but shorter than microwaves.



Principles of IR Spectroscopy

The IR Region of the Electromagnetic Spectrum

The wavelengths **from 0.8 to 100 μm** can be used for IR spectroscopy.

The infrared (IR) region of the electromagnetic spectrum lies between ~ 10 and $12\,800\text{ cm}^{-1}$.

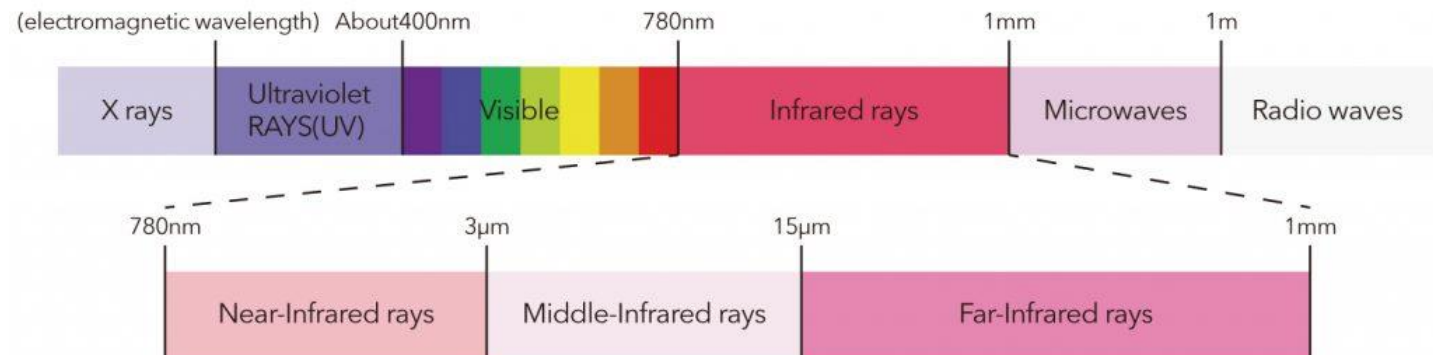
Various Regions of the Infrared Range of Spectrum

Region	Wavelength range (μm)	Wave number range (cm^{-1})
Near-IR	0.8-2.5	12800-4000
Mid-IR	2.5-15	4000-650
Far-IR	15-100	650-100



Most useful for quantitative and qualitative analysis of foods.

* $1\mu\text{m} = 10^{-6}\text{ m}$



Principles of IR Spectroscopy

The IR Region of the Electromagnetic Spectrum

- ▶ **IR radiation** also can be measured in terms of its frequency.
- ▶ (It is useful, because frequency is directly related to the energy of the radiation by the following relationship:

$$E = h\nu$$

where:

E = energy of the system

h = Planck's constant

ν = frequency, in hertz

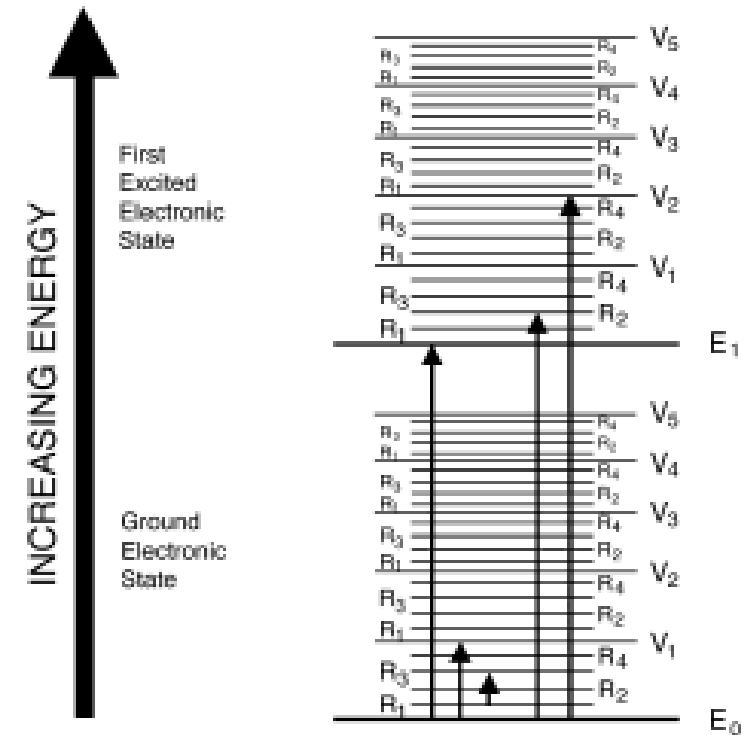
➤ Frequencies are commonly expressed as wavenumbers ($\bar{\nu}$, in reciprocal centimeters, cm^{-1}).

➤ Wavenumbers are calculated as follows:

$$\bar{\nu} = 1 / (\lambda \text{ in cm}) = 10^4 / (\lambda \text{ in } \mu\text{m})$$

Principles of IR Spectroscopy

- ▶ The principle of **IR spectroscopy** is related to the **vibrational and rotational energy** of a molecule.
- ▶ When the frequency of the IR radiation is equal to the natural frequency of vibration, the molecule absorb IR radiation.
- ▶ Absorption of IR radiation causes an excitation of molecule from a lower to the higher vibrational level.
- ▶ Each vibrational level is associated with a number of closely placed rotational level.
- ▶ Therefore, the **IR spectroscopy** is also called as «**vibrational-rotational spectroscopy**».



Some General Trends:

- 1) Stretching frequencies are higher than corresponding bending frequencies.
(It is easier to bend a bond than to stretch or compress it.)
- 2) Bonds to hydrogen have higher stretching frequencies than those to heavier atoms.
- 3) Triple bonds have higher stretching frequencies than corresponding double bonds, which in turn have higher frequencies than single bonds.
(Except for bonds to hydrogen).

Principles of IR Spectroscopy

Molecular Vibrations

Conditions required for a molecule to absorb IR radiation

When IR radiation is shone on a molecule, it can only absorb it if:

- 1) The energy of the radiation corresponds to the energy required to vibrate the molecule, and
- 2) The vibration leads to the dipole moment of the molecule changing.

How does infrared light interact with matter?

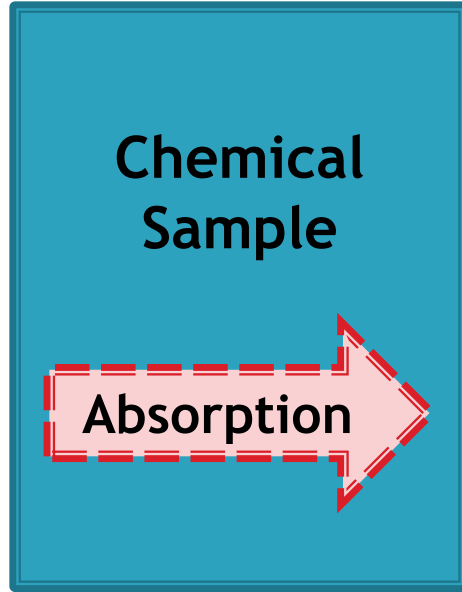
IR Source



Incident IR Light



Chemical Sample



Absorption



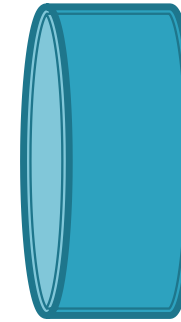
Reflection



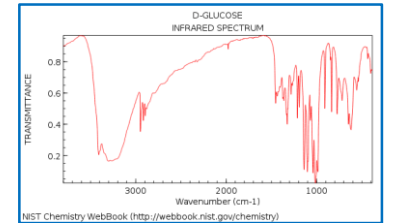
Transmission



Detector



IR Spectrum



Visual representation
or
Spectrum

- When a chemical sample is exposed to the action of IR Light, it can absorb some frequencies and transmit the rest. Some of the light can also be reflected back to the source.
- From all the frequencies it receives, the chemical sample can absorb specific frequencies and allow the rest to pass through it (transmitted light). Specific bonds respond to (absorb) specific frequencies.
- The detector detects the transmitted frequencies, and by doing so also reveals the values of the absorbed frequencies.
- In brief, upon irradiation with infrared light, certain bonds respond by vibrating faster. This response can be detected and translated into a visual representation called a spectrum.

MID-IR Spectroscopy

- ▶ **Mid-IR spectroscopy** measure a sample's ability to absorb light in the **2.5-15 μm (4000-650 cm^{-1})** region.
- ▶ **Fundamental absorptions** are primarily observed in this spectral region.

MID-IR Spectroscopy

Instrumentation

Two types of spectrometers are used for mid-IR spectroscopy:

- 1) Dispersive instruments
- 2) Fourier transform (FT) instruments

→ Almost all newer instruments are of the FT type.

MID-IR Spectroscopy

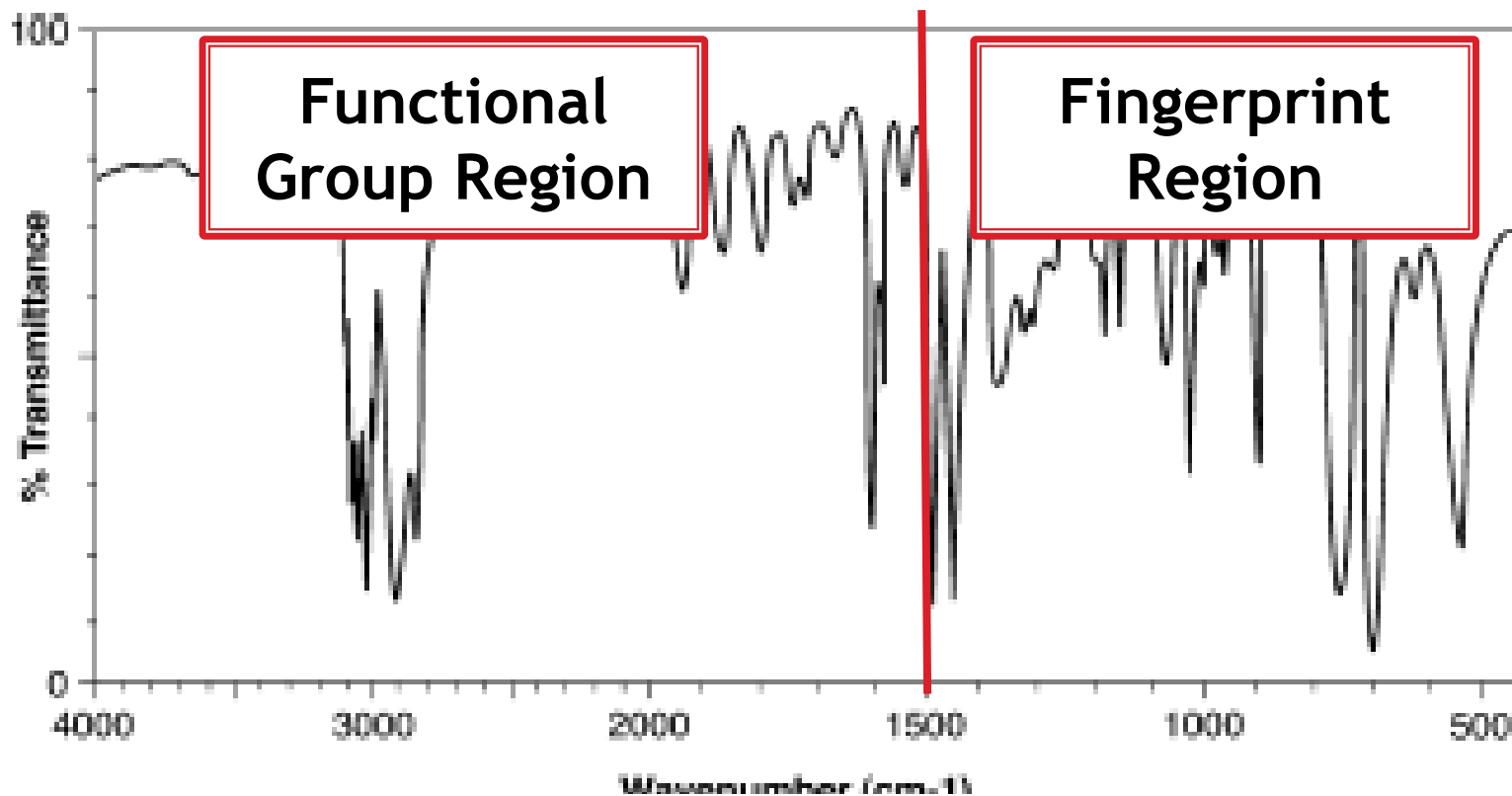
Applications of Mid-IR Spectroscopy

Absorption Bands of Organic Functional Groups

<i>Group</i>	<i>Absorbing Feature</i>	<i>Frequency (cm⁻¹)</i>
Alkanes	-CH stretch and bend -CH ₂ and -CH ₃ bend	3000-2800 1470-1420 and 1380-1340
Alkenes	Olefinic -CH stretch	3100-3000
Alkynes	Acetylenic -CH stretch	3300
Aromatics	Aromatic -CH stretch -C=C- stretch	3100-3000 1600
Alcohols	-OH stretch -OH bend C-O stretch	3600-3200 1500-1300 1220-1000
Ethers	C-O asymmetric stretch	1220-1000
Amines	Primary and secondary -NH stretch	3500-3300
Aldehydes and ketones	-C=O stretch -CH (doublet)	1735-1700 2850-2700
Carboxylic acids	-C=O stretch	1740-1720
Amides	-C=O stretch -NH stretch -NH bend	1670-1640 3500-3100 1640-1550



Mid-IR Absorption Frequencies of Various Organic Functional Groups



Regions

An IR Spectrum is often interpreted as having two regions.

- **functional group region** ($\geq 1500 \text{ cm}^{-1}$)

In the functional region, there are one to a few troughs per functional group.

- **fingerprint region** ($< 1500 \text{ cm}^{-1}$)

In the fingerprint region, there are many troughs which form an intricate pattern which can be used like a fingerprint to determine the compound.

Near-IR Spectroscopy

- ▶ Measurements in the **near-IR** (NIR) spectral region (**0.7-2.5 μm** , equal to **700-2500 nm**) are more widely used for quantitative analysis of foods than are mid-IR measurements.
- ▶ Several commercial instruments are available for compositional analysis of foods using **NIR spectroscopy**.
- ▶ A major advantage of **NIR spectroscopy** is its ability to measure directly the composition of solid food products by use of diffuse reflection techniques.

Near-IR Spectroscopy

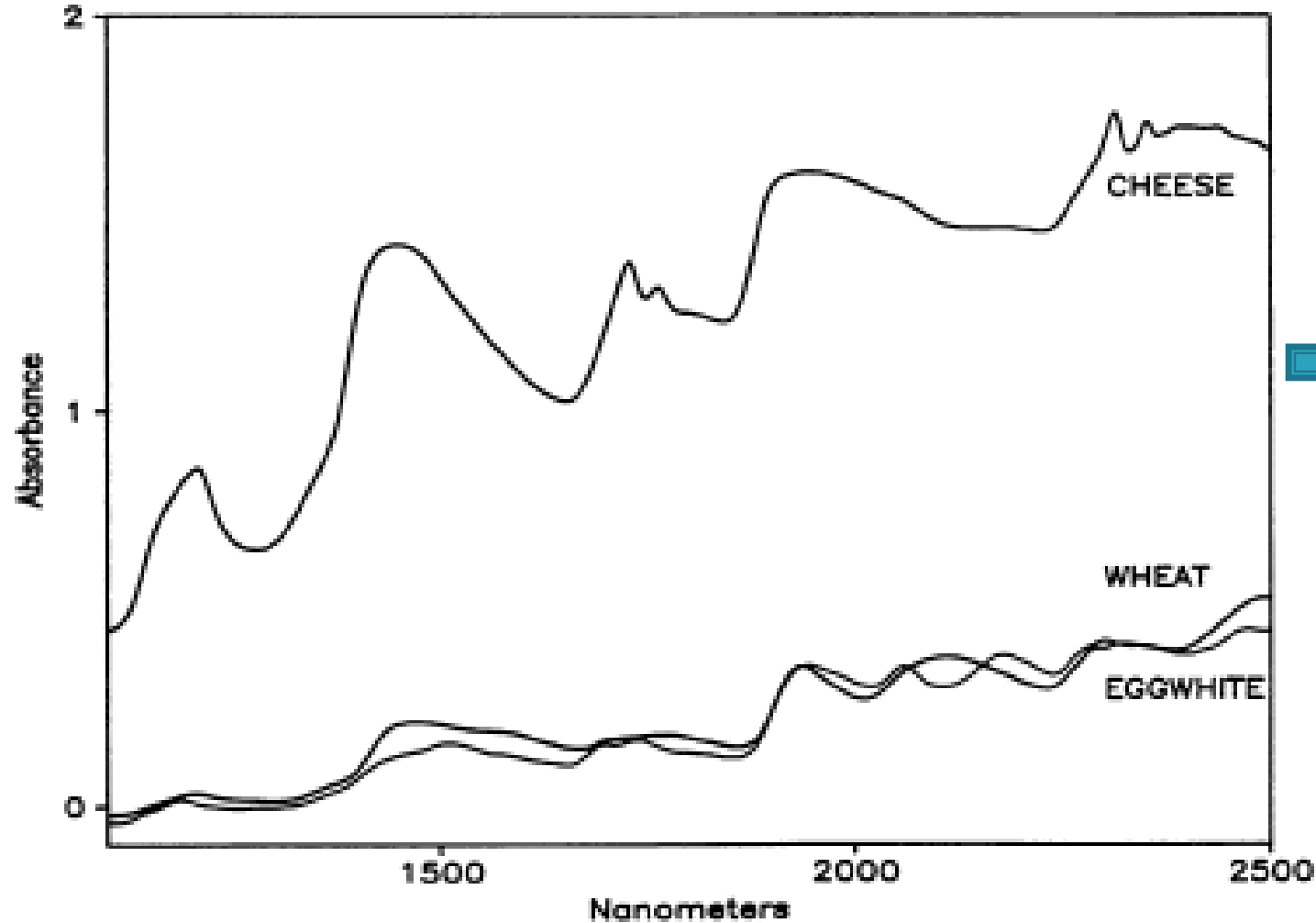
Absorption Bands in the NIR Region

Near-IR Absorption Bands of Various Food Constituents

<i>Constituent</i>	<i>Absorber</i>	<i>Wavelength (nm)</i>
Water	-OH stretch/deformation combination	1920–1950
	-OH stretch	1400–1450
Protein – peptides	-NH deformation	2080–2220 and 1560–1670
Lipid	Methylene -CH stretch	2300–2350
	-CH ₂ and -CH ₃ stretch	1680–1760
Carbohydrate	C–O, O–H stretching combination	2060–2150

Near-IR Spectroscopy

Absorption Bands in the NIR Region



Typical NIR spectra of cheese, wheat and dried egg white plotted as absorbance vs. wavelength in nm

- **Mid-IR spectroscopy** is especially used for [qualitative analysis](#), such as identifying specific functional groups present in a substance.
- Different functional groups absorb different frequencies of radiation, allowing the groups to be identified from the spectrum of a sample.
- Quantitative analysis can also be achieved by **Mid-IR spectroscopy**, such as milk analysis.
- **Near-IR spectroscopy** is used most extensively for quantitative applications, using either transmission or diffuse reflection measurements that can be taken directly from solid foods.
- By using multivariate statistical techniques, **NIR instruments** can be calibrated to measure the amounts of various constituents in a food sample based on the amount of radiation absorbed at specific wavelengths.
- **NIR spectroscopy** requires much less time to perform quantitative analysis than do many conventional wet chemical or chromatographic techniques.