INFRARED SPECTROSCOPY

Applications

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What is Infrared?

- Infrared radiation lies between the visible and microwave portions of the electromagnetic spectrum.
- Infrared waves have wavelengths longer than visible and shorter than microwaves, and have frequencies which are lower than visible and higher than microwaves.
- The Infrared region is divided into: near, mid and far-infrared.
  - Near-infrared refers to the part of the infrared spectrum that is closest to visible light and far-infrared refers to the part that is closer to the microwave region.
  - Mid-infrared is the region between these two.
The primary source of infrared radiation is thermal radiation. (heat)

It is the radiation produced by the motion of atoms and molecules in an object. The higher the temperature, the more the atoms and molecules move and the more infrared radiation they produce.

Any object radiates in the infrared. Even an ice cube, emits infrared.
Humans, at normal body temperature, radiate most strongly in the infrared, at a wavelength of about 10 microns (A micron is the term commonly used in astronomy for a micrometer or one millionth of a meter). In the image to the left, the red areas are the warmest, followed by yellow, green and blue (coolest).

The image to the right shows a cat in the infrared. The yellow-white areas are the warmest and the purple areas are the coldest. This image gives us a different view of a familiar animal as well as information that we could not get from a visible light picture. Notice the cold nose and the heat from the cat's eyes, mouth and ears.
Infrared Spectroscopy

The bonds between atoms in the molecule stretch and bend, absorbing infrared energy and creating the infrared spectrum.

A molecule such as $\text{H}_2\text{O}$ will absorb infrared light when the vibration (stretch or bend) results in a molecular dipole moment change.
Infrared Spectroscopy

A molecule can be characterized (identified) by its molecular vibrations, based on the absorption and intensity of specific infrared wavelengths.
Infrared Spectroscopy

For isopropyl alcohol, CH(CH$_3$)$_2$OH, the infrared absorption bands identify the various functional groups of the molecule.
Capabilities of Infrared Analysis

- Identification and quantitation of organic solid, liquid or gas samples.
- Analysis of powders, solids, gels, emulsions, pastes, pure liquids and solutions, polymers, pure and mixed gases.
- Infrared used for research, methods development, quality control and quality assurance applications.
- Samples range in size from single fibers only 20 microns in length to atmospheric pollution studies involving large areas.
Applications of Infrared Analysis

- Pharmaceutical research
- Forensic investigations
- Polymer analysis
- Study of a chemical reaction
- Geometrical isomerism
- Quality assurance and control
- Study of keto-enol tautomerism
- Detection of impurity in a compound
- Etc.
Geometrical Isomerism

Dipole moment = 1.85 D
Cis isomer of 1,2-dichloroethene

Dipole moment = 0
Trans isomer of 1,2-dichloroethene
Study of Keto-enol tautomerism

\[
\begin{align*}
\text{H}_3\text{C} & \text{C} \text{CH}_2\text{COOC}_2\text{H}_5 \quad & \text{CH}_3 & \text{C} \text{CHCOOC}_2\text{H}_5 \\
keto \text{ form (92.3 \%)} & \quad & \text{enol} \text{ form (7.7 \%)}
\end{align*}
\]

C=O str 1733 cm\(^{-1}\)

O-H str 3300 cm\(^{-1}\)

C=O str 1645 cm\(^{-1}\)
INTRODUCTION

Infrared spectrometry is applied to the qualitative & quantitative determination of molecular species of all types. The most widely used region is the mid-infrared that extends from about 670 to 4000 cm⁻¹ (2.5 to 14.9 μm). The near-infrared region from 4000 to 14,000 cm⁻¹ (0.75 to 2.5 μm) also finds considerable use for the routine quantitative determination. The far-infrared region has been for the determination of the structures of inorganic and metal-organic species.
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<td>Mid-IR</td>
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<td>Far-IR</td>
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Qualitative Analysis

The appearance of I.R. instrument revolutionized the way chemists went about identifying organic, inorganic, and biological species. The time required to perform a structural determination was reduced by a factor of ten, one hundred, or even one thousand. Identification of an organic compound is a two-step process. The first step involves determining what functional groups are most likely present by examining the group frequency region.
The **second step** then involves a detailed comparison of the spectrum of the unknown with the spectra of pure compounds that contain all of the functional groups found in the first step. The fingerprint region, from 1200 to 600 cm\(^{-1}\) is particularly useful because small differences in the structure and constitution of a molecule result in significant changes in the appearance and distribution of absorption peaks in this region.
Frequencies of various group vibrations in the group frequency region and in fingerprint region
Qualitative Analysis

**Computer Search Systems:** Virtually all infrared instrument manufacturers now offer computer search systems to assist chemist in identifying compounds from stored infrared spectral data. The position and relative magnitudes of peaks in the spectrum of the analyte are determined and stored in memory to give a peak profile, which can then be compared with profiles of pure compounds stored. The computer then matches profiles and prints a list of compounds having spectra similar to that of the analyte. Usually the spectrum of the analyte and that of each potential match can then be shown simultaneously on the computer display for comparison.
Quantitative Applications

Quantitative infrared absorption methods differ somewhat from ultraviolet/visible molecular spectroscopic methods because of the greater complexity of the spectra, the narrowness of the absorption bands, and the instrumental limitations of infrared instruments. Quantitative data obtained with infrared instruments are generally significantly inferior in quality to data obtained with ultraviolet/visible spectrophotometers.
Near-infrared Spectroscopy

The near-infrared (NIR) region of the spectrum extends from the upper wavelength end of the visible region at about 770 nm to 2500 nm (13,000 to 4000 cm$^{-1}$). Absorption bands in this region are overtones or combinations of fundamental stretching vibrational bands that occur in the region of 3000 to 1700 cm$^{-1}$ . The bonds involved are usually C—H, N—H, and O—H. Because the bands are overtones or combination, their molar absorptivities are low.
NIR agricultural applications

The NIRS technology can be applied to the sorting of fruit at commercial packline speeds. Indeed, it is in commercial use in Japan, primarily for melons and citrus fruit. The Japanese firms are currently marketing **NIR based sorting and grading systems** for use with **citrus, pome fruits and stone fruits** in Japan. It is perhaps not surprising that the technology has been applied quickly in Japan, where fruit are with huge price (single melons selling for routinely at $30). However, with modification and reduction of price, the technology is applicable to markets with less reward for premium quality.
The development of a high speed system incorporated into a packing line would enable the sorting of fruit based on quality parameters such as dry matter content.

The technology may be used to exclude fruit with internal defects.

A portable unit would enable random checks of fruit in the field, or in the wholesale or retail store.

On-harvester estimation of grain protein content

for precision agriculture and estimation of the sugar content of fibrated sugar cane (beet) in sugar mills.

In the future, we should expect to see the application of NIR technology to assessing a range of food products for various aspects of quality and safety.
Commodity areas and possible NIR applications that have been investigated are:

- Sweet Corn - insect damage and insect detection
- Coal - moisture determination
- Macadamia kernel - quality
- Citrus - quality attributes
- Pineapples, mango's, strawberries - quality attributes
- Stone fruit - quality attributes
- Melons - quality attributes
Far-infrared Spectroscopy

The far-infrared region is particularly useful for inorganic studies because absorption due to stretching and bending vibrations of bonds between metal atoms and both inorganic and organic ligands generally occur at frequencies lower than 650 cm\(^{-1}\) (>15\(\mu\)m). For example, heavy-metal iodides generally absorb in the region below 100 cm\(^{-1}\). Far-infrared studies of inorganic solids have also provided useful information about lattice energies of crystals and transition energies of semiconducting materials.
References

1. Elementary organic spectroscopy by Y.R. SHARMA; Pg.No: 69-150
2. Instrumental methods of chemical analysis by GURDEEP R. CHATWAL; Pg.No: 2.29-2.82
3. WWW.PHARMAINFO.NET
4. en.WIKIPEDIA.ORG
5. Principles of instrumental analysis by SKOOG & HOLLER; Pg. No: 404-426
Thank You