

Infrared Spectroscopy

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WHAT IS INFRARED SPECTROSCOPY?

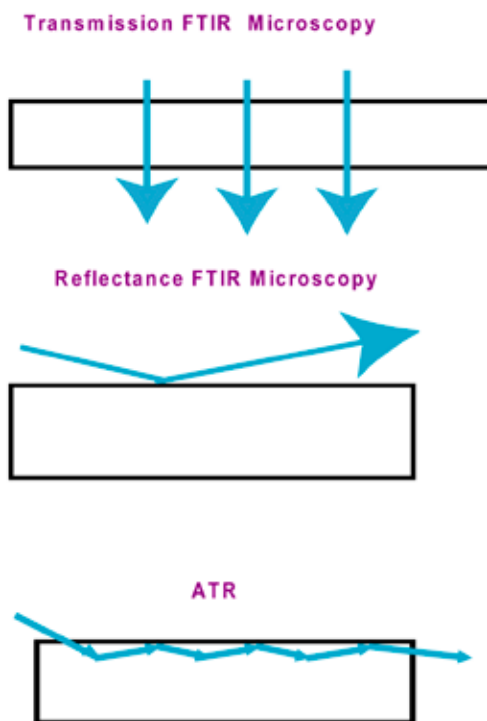
Infrared Spectroscopy (IR) is an analytical technique for chemical compound identification. It is based on the fact that different chemical functional groups absorb infrared light at different wavelengths dependent upon the nature of the particular chemical functional group. A Fourier Transform is a mathematical conversion that allows the split of the entire infrared light spectrum simultaneously, then converting the scanning results mathematically into a wavelength versus absorbance spectra. Combined together these two functions provide Fourier Transform Infrared Spectroscopy (FTIR) as an instrument that can be used in the identification and characterization of organic compounds. The relative simplicity of the resulting FTIR analytical methods allows it to be widely used for the analysis of a wide range of different materials. It is often used in the packaging industry to analyze monomeric materials for purity, and to identify polymers (polyethylene, polyester, nylon, and etc) and their compositions.

HOW IS IR USED?

It can be used to investigate solids as powders, films or blocks, liquids either pure or as solutions, and gases. It is particularly useful for the identification of pills and the component makeup of multi-layer coatings. FTIR is routinely used for forensic analysis, for example in the identification of

foreign materials in food or beverage products by matching the spectra of the material in question with the spectra of known compounds.

IR is typically operated in the Mid-IR range between 4000-400 cm^{-1} (2.5-25 μm) when it is used for compound identification purposes. IR in either the Far - IR range between 400-10 cm^{-1} (25-1000 μm) and the Near-IR 14,000-4,000 cm^{-1} (0.71-2.5 μm) is typically carried out for special purposes. Near IR applies to methods such as the routine rapid quantitation of macro components of complex mixtures such as protein and/or moisture in grains and flour. IR instruments today can be operated in either the transmission or reflectance mode. In the past, analyses were often limited by the thickness of the samples, the sample shape, and the sample's bulk quantity. These limitations have been largely overcome by the use of adapters that change the presentation of the samples to the IR beam. Attenuated Total Reflectance (ATR) and Diffuse Reflectance accessories allow the analysis of the surface of the coatings and allow comparison of the bulk properties with the surface properties of a material.



How does IR analysis work?

Modes of IR sampling

Currently, there are basically three procedures (modes) used for obtaining infrared spectra of samples. Each has its own unique advantages for optimizing the quality of the

spectrum obtained.

TRANSMISSION INFRARED The transmission sampling technique involves passing the infrared energy through the sample and detecting that portion of the beam that is transmitted, i.e. not absorbed. The infrared beam passes through the sample and the energy that comes through the sample is measured versus the respective wavelength to generate a spectrum.

Transmission using IR Sampling Cards. These accessories are very often used to conduct transmission analyses. A microporous sampling substrate that is chemically inert or resistant and usually non-hygroscopic is utilized. The microporous sample substrate permits rapid solvent evaporation when the sample is applied as a solution. The two most popular types of cards are polytetrafluoroethylene cards which provide an absorption free background from 4000-1400 cm^{-1} and polyethylene substrates which provide an absorption free background for the entire spectrum except in the region of 3000-2800 cm^{-1} . Using these two cards in combination allows full transmission scanning of the spectral range from 4000-400 cm^{-1} . Because they are disposable, use of the IR cards typically shortens analysis time, eliminates cross contamination concerns, and reduces solvent clean up waste. IR cards allow the analysis of a wide range of samples, including but not limited to inorganic and organic liquids of low volatility, solids that can be solubilized in volatile solvents, insoluble solids, semi-solids, pastes, lubricants, paints, etc.

Compressed Pellets. Another widely used technique for the investigation of solid samples in the transmission mode is the pressing of an alkali halide pellet (most often a potassium bromide pellet). The method consists of grinding the sample together with pure, dry spectroscopic grade KBr to a fine powder, then transferring the mixture to a compression die. The mixture is then placed under high pressure until the mixture forms a pellet that is transparent to infrared light.

DIFFUSE and SPECULAR REFLECTANCE.

Reflectance IR analysis is an optically simpler technique (particularly for solids) than transmission IR and involves reflecting the infrared light off of the sample. When infrared radiation is directed onto the surface of solid sample two types of reflected energy can result: specular and diffuse reflection. The specular component is the radiation that reflects directly off the sample surface (it is the energy, which is not absorbed by the sample). Diffuse reflectance is the radiation that penetrates into the surface of sample (usually to a short depth) and then reemerges after reflecting off internal portions of the sample. External reflection techniques provide a non-destructive

method for measurement surfaces and coatings without sample preparation.

Si-Carb paper. Si-Carb paper (a small disk of silicon carbide) is used for diffuse reflectance analyses. This technique allows abrading the surface and transferring a small amount of sample (hard polymer, paints, and coatings) to the disk. An infrared spectrum is obtained of the material that clings to the surface of Si-Carb disk after abrading. For abrading extremely hard samples diamond paper can be used.

ATTENUATED TOTAL REFLECTANCE (ATR).

ATR spectroscopy functions to measure the changes that occur when a totally, internally reflected, infrared beam comes into contact with the sample. Of the available reflection methods, ATR finds increasing application due to its ease of use and the limited sample preparation it requires. In the ATR mode of analysis, the sample is pressed into place in intimate physical contact with a special ATR crystal. The sample and crystal are then placed in the IR beam in such a position that the IR beam enters the crystal at an angle and is multiply reflected along the length of the sample. IR energy exiting the opposite end of the crystal is measured versus the respective wavelength. This technique is ideal for rapid quantitative and/or qualitative analyses, as no sample preparation is required for most samples. Single and multiple internal reflection ATR accessories are well suited for highly infrared absorbant materials such as rubbers and polymers. In addition, ATR is well suited for providing information about the surface makeup or surface conditions of a material. Single reflection ATR technology is also used in the ATR Microscopy objectives.

FTIR MICROSCOPY

FTIR microscopy is a technique that uses a modified light microscope in combination with and FTIR spectroscope. The design goal of a quality microscope modified for infrared use is to collect infrared spectra free of spectral contributions of the surrounding matrix, and to focus on the fine detail of the sample. An example would be looking at an individual layer of a multilayered film structure. Application of IR microscopy capabilities is for solving typical problems such as:

- Micro-contaminants in plastics, paper, pharmaceutical products and packaging materials
- Surface analysis of rubbers, paints and coatings
- Forensic Science - paint chips, crystals, powders and fibers.

Special attention should be paid to FTIR microscopy as a method of the analysis of multi-layer coatings.

FTIR LIBRARY MATCHING

The powerful computing capabilities of the FTIR spectrometer can also be used for comparing spectra of unknowns against library spectra of known compounds. Typically, FTIR equipment suppliers provide the instrument user with a library of spectra including spectra of different polymers and organic and inorganic materials. The computing software allows direct comparisons of the library and recorded spectra using special software.

References

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A publication of Medallion Labs
Dr. Jonathan DeVries, Editor

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