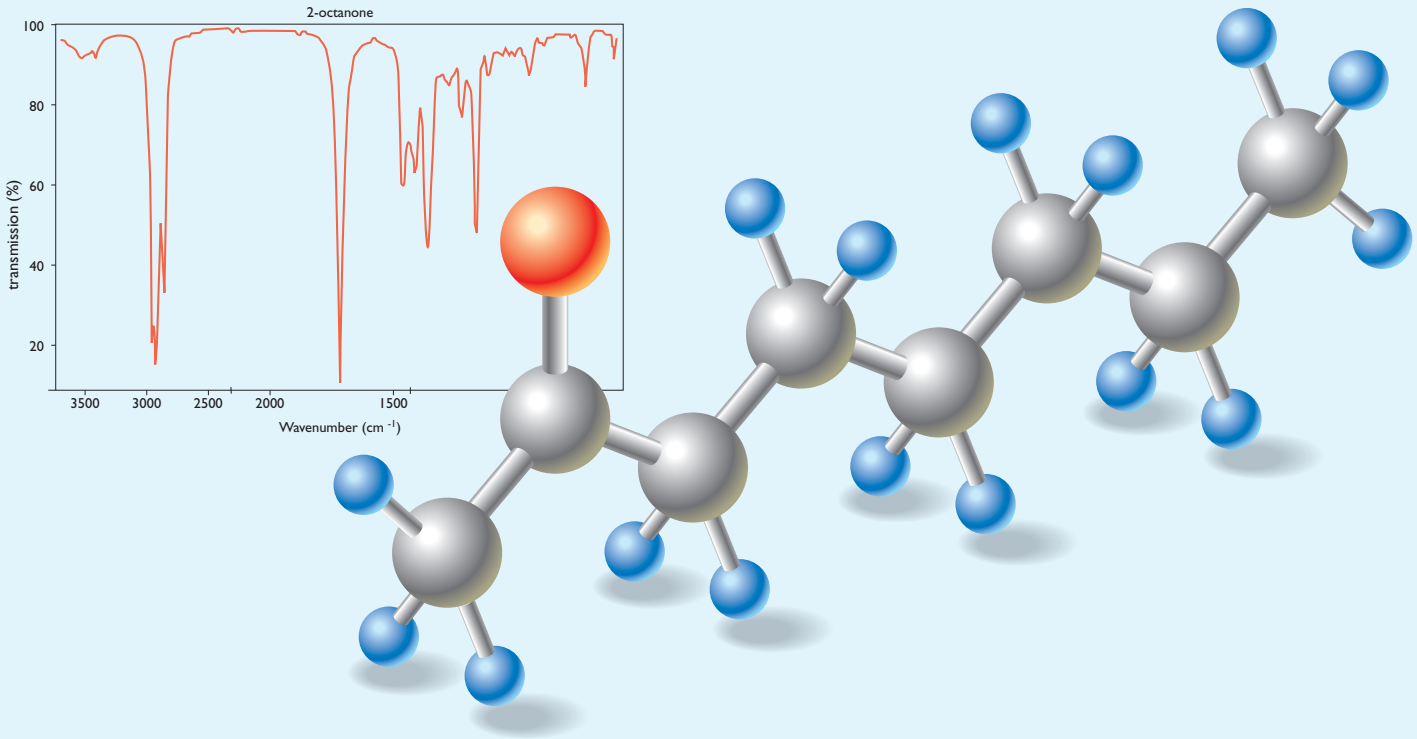
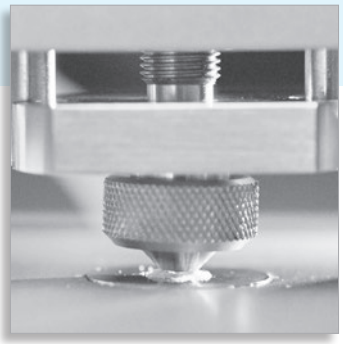


Fig. 1: Typical IR spectrum of 2-octanone.



Infrared spectroscopy

(IR)

- structural elucidation
- process kinetics
- (in)organic materials
- non-destructive

Infrared (IR) spectroscopy is used to obtain information on the molecular structure of virtual all type of samples in any physical state (solid, liquid or gas). The technique is widely spread and is applied in the polymer, pharmaceutical, medical, food and chemical industry. The infrared spectrum is related to the vibrations of molecules and is unique for each compound, like a fingerprint for a person. Using an IR microscope samples with dimensions down to 10 µm can be measured with little or no sample preparation.

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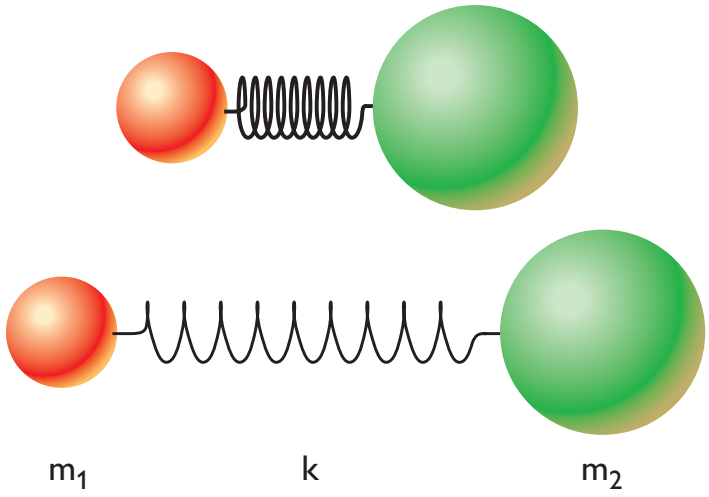

Fig. 2: A two-atomic vibrating system.

Principle of Infrared Spectroscopy

Infrared spectroscopy is based on the fact that all molecules vibrate and can absorb energy in the infrared region. Most of the vibrational absorption states correspond to the wavelength in the order of 2.5 to 25 µm (4000-400 cm^{-1}).

For a two atomic system consisting of two masses m_1 and m_2 (fig. 2) the vibrational frequency (ν) is related to the force constant (k) and the reduced mass (µ) by the following equation:

$$\nu = \frac{1}{2\pi}\sqrt{\frac{k}{\mu}} \quad \text{with } \mu = \frac{m_1 m_2}{m_1 + m_2}$$

An infrared spectrum shows transmission versus wavenumber (cm^{-1}), i.e. the frequency (ν) divided by the speed of light. Absorption is only possible at specific frequencies at which higher vibrational states can be reached. If energy is absorbed by a molecule the signal at this frequency decreases, leading to a peak in the spectrum.

The formula shows that if the force constant (k) of the bond between two atoms is larger, the frequency of the vibration is higher. This means that a C=C bond will absorb at a higher frequency than a C-C bond, resulting in a peak at 1600 cm^{-1} and 1000 cm^{-1} respectively. The lighter the atoms involved in the vibration the higher the frequency will be. The stretching vibrations of the C-H bond can be found in the 3000 cm^{-1} region of the spectrum.

Collecting an FT-IR spectrum

With FT-IR (Fourier Transform - InfraRed) instruments (fig. 3) all IR wavelengths of the polychromatic source are measured at the same time. A so-called interferogram is collected by the detector, which is transferred to an IR spectrum by means of a Fourier transformation.
A routine FT-IR spectrum is normally acquired in seconds but if a fast reaction needs to be followed, spectra can be easily recorded at a rate of 100 per second.

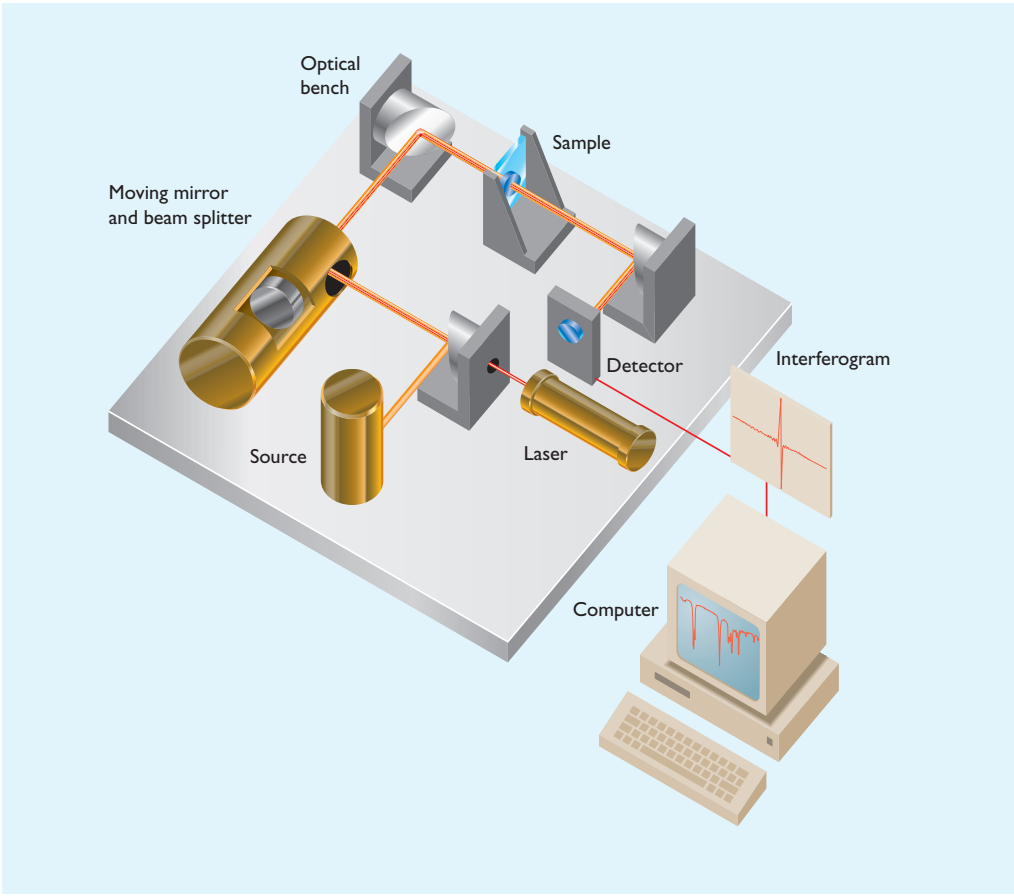

Fig. 3: Schematic of a FT-IR spectrometer.

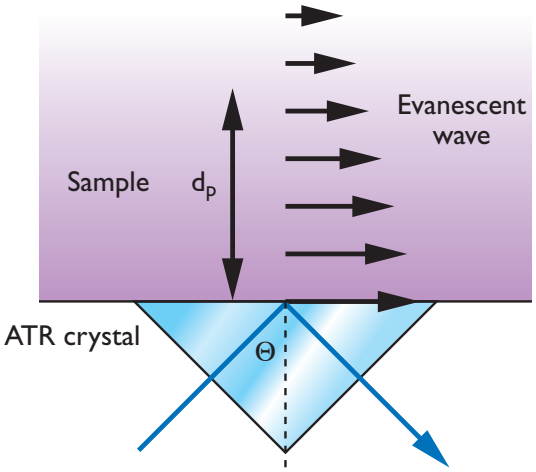

Fig. 4: Schematic of ATR principle.

ATR

For a good transmission IR spectrum of a solid or liquid, a sample should not be thicker than 10 µm. A very popular and easy to use reflection technique can be applied to thicker samples. This technique is called ATR, which stands for Attenuated Total Reflection. The principle of this method is based upon total reflection of IR radiation that occurs within a high refractive index crystal (fig. 4) in contrast with a material with a lower refractive index.

When radiation is incident on an IR transparent material at an angle that exceeds the critical angle, total internal reflection takes place. When this occurs the radiation also penetrates the sample. It then returns in the crystal as if it is reflected within it. If an absorbing sample is placed in contact with the surface of the crystal at the point of the internal reflection, energy is absorbed by the sample. The reflected beam now contains spectral information of the sample. The depth of penetration (d_p) is in the order of several micrometers depending on the angle of incidence of the beam (θ), the wavelength of the light (λ) and on the refractive indices of the sample and ATR crystal:

$$d_p = \frac{\lambda}{2\pi\, n_1 \sqrt{\sin^2(\theta) - (n_1/n_0)^2}}$$

A typical ATR application is the surface analysis of a plastic or rubber compound to look for contaminant components segregating to the surface.

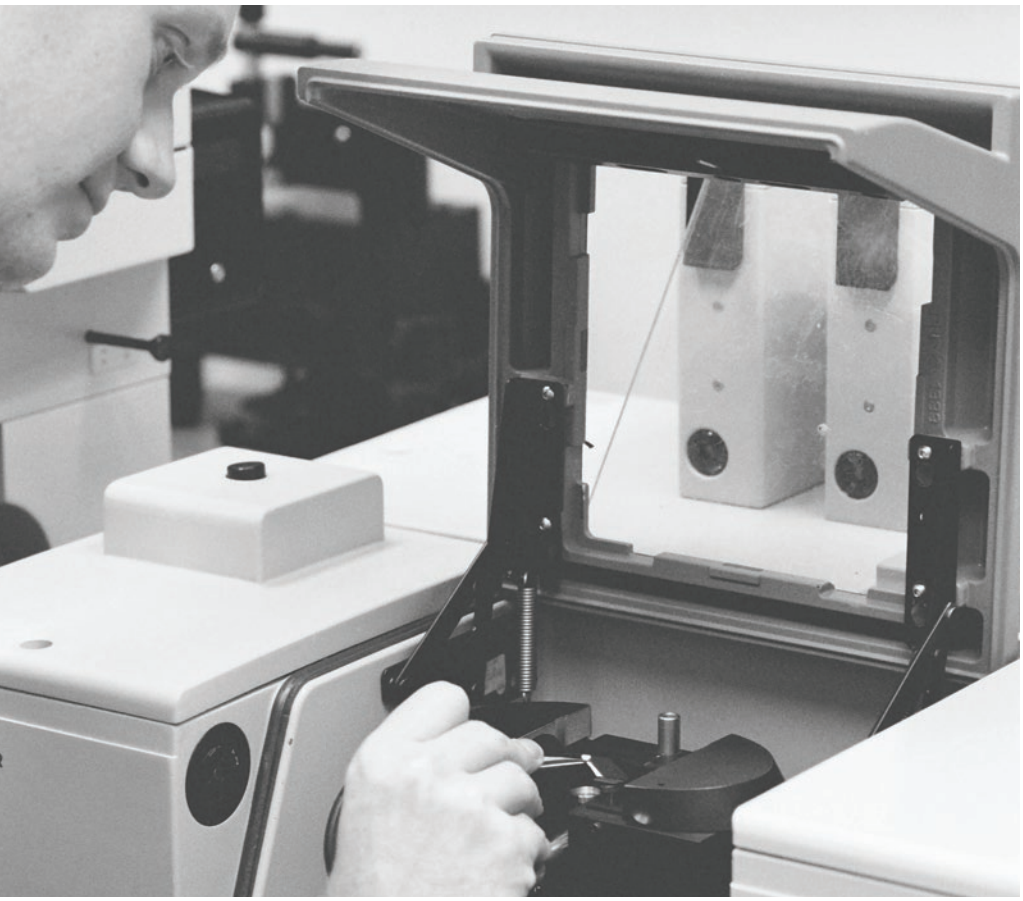

Fig. 5: Sample chamber FT-IR spectrometer.

FT-IR microscopy

The sensitivity of an FT-IR spectrometer may not be sufficient for the smallest sample sizes. For this reason IR microscopes have been developed which can be used for samples down to the diffraction limit of the IR radiation, e.g. about 10 µm. Spectra can be recorded in transmission as well as in reflection.

An FT-IR microscope consists of a modified visible-light microscope and viewing optics with 150 times magnification. By switching mirrors, the IR radiation follows the same optical path as the visual light used for viewing, so that the selected sample part can be analysed. Positioning of the sample is done with a computer controlled adjustable stage. Besides collecting single spectra also a mapping of the sample can be performed.

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Technical Note 16
August 2016

Applications

- identification of inorganic and organic materials
- purity control of materials
- reaction kinetics e.g. conversion of polymers, curing acrylics, hybrid systems
- trouble shooting
- identification of monomers and polymers; resins, hardeners, stabilisers, plasticizers, fillers, adhesives, oils and waxes
- identification of solvents and extracts
- identification and quantification of contaminants on surfaces

Characteristics

Obtained information

- functional groups, identification, orientation, crystallinity, molecular structure
- qualitative and quantitative when calibration standards are available

Sample type

- solids, liquids and gases
- organic and inorganic
- bulk and thin films (monolayer)
- powder, single crystals

Sample requirements

- no or little sample preparation required

Spatial resolution

- infrared microscopy allows 10 µm spatial resolution
- the surface sensitive ATR technique allows analysis of the upper few microns

Detection limit

- strongly depending on material and matrix (100 ppm to 1%)

Accuracy

- quantitative 1-5% when calibration standards are available

Analytical range

- traces to main components

Routine analysis

- non destructive and very useful in quality control and on-line analysis because of its speed and ease of use

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