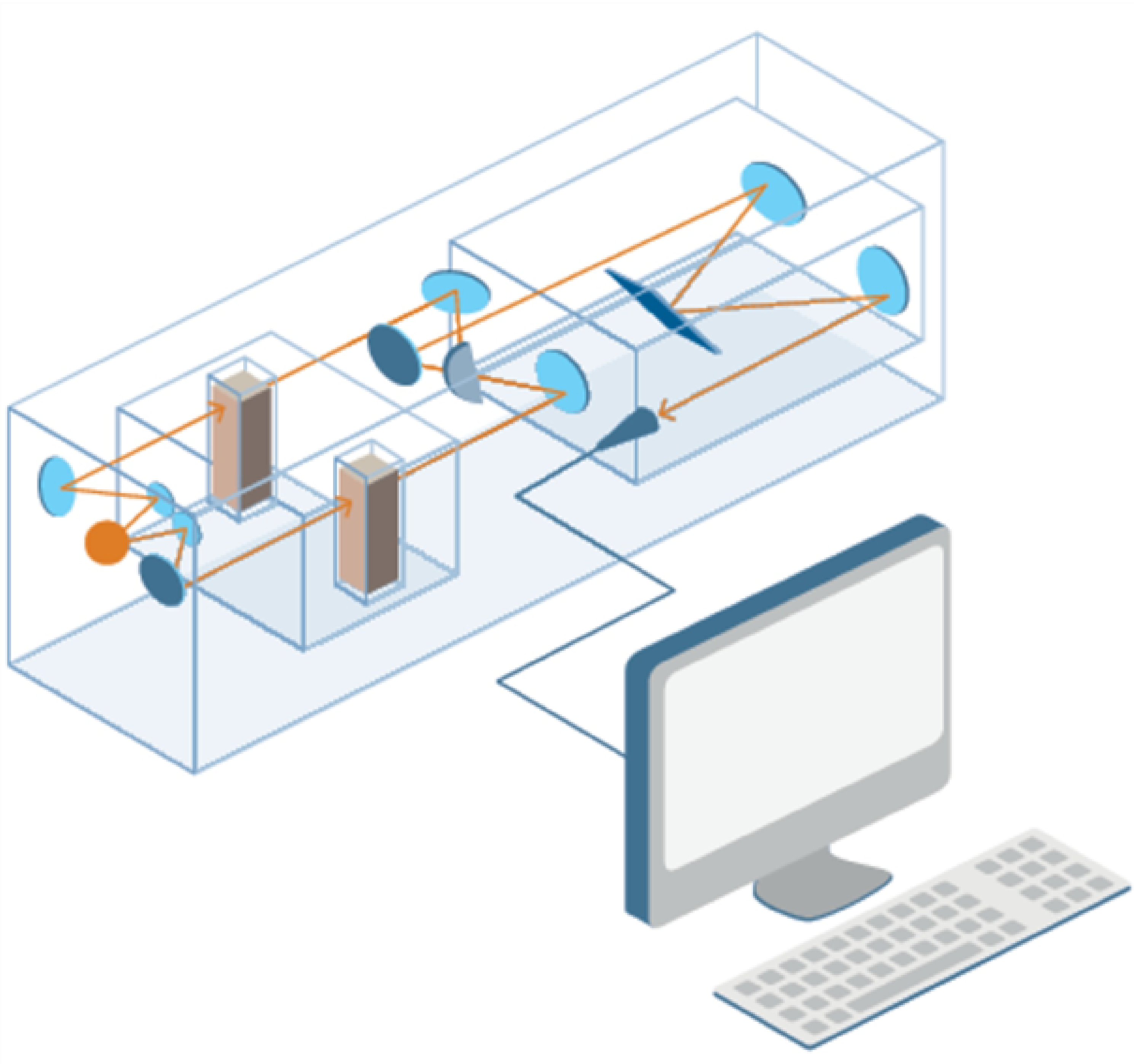
Infrared spectroscopy

Infrared spectroscopy is the measurement of the wavelength and intensity of the absorption of mid-infrared light by a sample.



Mid-infrared is energetic enough to excite molecular vibrations to higher energy levels. The wavelength of infrared absorption bands are characteristic of specific types of chemical bonds, and infrared spectroscopy finds its greatest utility for identification of organic and organometallic molecules.
The high selectivity of the method makes the estimation of an analyte in a complex matrix possible. This method involves examination of the twisting, bending, rotating and vibrational motions of atoms in a molecule.

Infrared Spectrometry - Instrumentation

An infrared spectrophotometer is an instrument that passes infrared light through an organic molecule and produces a spectrum that contains a plot of the amount of light transmitted on the vertical axis against the wavelength of infrared radiation on the horizontal axis. In infrared spectra the absorption peaks point downward because the vertical axis is the percentage transmittance of the radiation through the sample. Absorption of radiation lowers the percentage transmittance value. Since all bonds in an organic molecule interact with infrared radiation, IR spectra provide a considerable amount of structural data.

There are four types of instruments for infrared absorption measurements available:
-Dispersive grating spectrophotometers for qualitative measurements
-Nondispersive (/en/analytical_methods/Infrared_spectroscopy/non_dispersive_infrared.html) photometers for quantitative determination of organic species in the atmosphere
-Reflectance photometers for analysis of solids
-Fourier transform infrared (FT-IR) (/en/analytical_methods/Infrared_spectroscopy/fourier_transform_infrared.html) instruments for both qualitative and quantitative measurements.

Infrared spectrometry - Infrared light sources

Instruments for measuring infrared absorption all require a source of continuous infrared radiation and a sensitive infrared transducer, or detector.
Infrared sources consist of an inert solid that is electrically heated to a temperature between 1,500 and 2,200 K. The heated material will then emit infra red radiation.

The Nernst glower
The Nernst glower is constructed of rare earth oxides in the form of a hollow cylinder. Platinum leads at the ends of the cylinder permit the passage of electricity. Nernst glowers are fragile. They have a large negative temperature coefficient of electrical resistance and must be preheated to be conductive.

The globar source
A globar is a rod of silicon carbide (5 mm diameter, 50 mm long) which is electrically heated to about 1,500 K. Water cooling of the electrical contacts is needed to prevent arcing. The spectral output is comparable with the Nernst glower, execept at short wavelengths (less than 5 mm) where it's output becomes larger.

The carbon dioxide laser
A tuneable carbon dioxide laser is used as an infrared source for monitoring certain atmospheric pollutants and for determining absorbing species in aqueous solutions.

Infrared Spectrometry - Detectors

The detectors can be classified into three categories, thermal detectors, pyroelectric detectors and photoconducting detectors.

Thermal detectors

Thermal detectors can be used over a wide range of wavelengths and they operate at room temperature. Their main disadvantages are slow response time and lower sensivity relative to other types of detectors.

Thermocouple

A thermocouple consists of a pair of junctions of different metals; for example, two pieces of bismuth fused to either end of a piece of antimony. The potential difference (voltage) between the junctions changes according to the difference in temperature between the junctions. Several thermocouples connected in series are called a thermopile.

Bolometer

A bolometer functions by changing resistance when heated. It is constructed of strips of metals such as platinum or nickel or from a semiconductor.

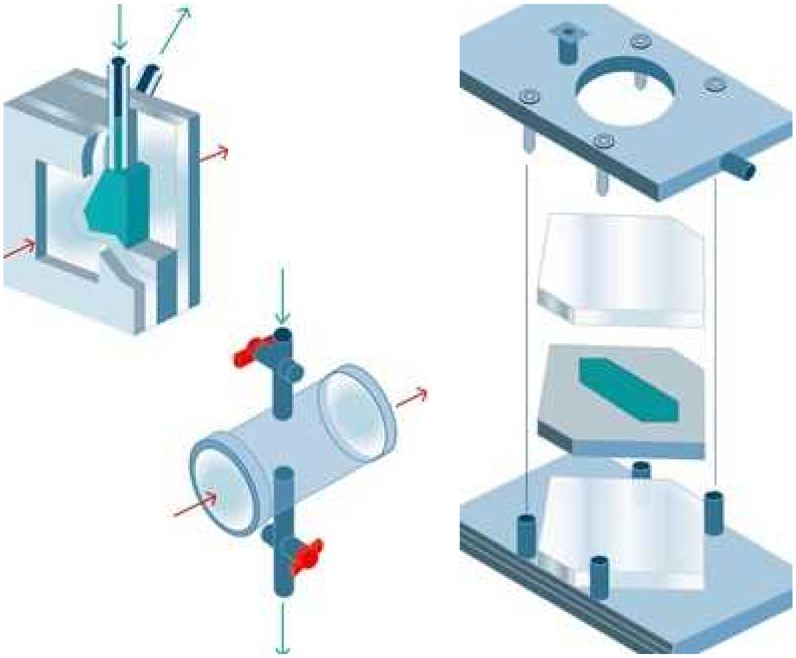
Pyroelectric detectors

Pyroelectric detectors consists of a pyroelectric material which is an insulator with special thermal and electric properties. Triglycine sulphate is the most common material for pyroelectric infrared detectors. Unlike other thermal detectors the pyroelectric effect depends on the rate of change of the detector temperature rather than on the temperature itself. This allows the pyroelectric detector to operate with a much faster response time and makes these detectors the choice for Fourier transform spectrometers where rapid response is essential.

Photoconducting detectors

Photoconducting detectors are the most sensitive detectors. They rely on interactions between photons and a semiconductor. The detector consists of a thin film of a semiconductor material such as lead sulphide, mercury cadmium telluride or indium antimonide deposited on a nonconducting glass surface and sealed into an evacuated envelope to protect the semiconductor from the atmosphere. The lead sulphide detector is used for the near-infrared region of the spectrum. For mid- and far-infrared radiation the mercury cadmium telluride detector is used. It must be cooled with liquid nitrogen to minimize disturbances.

Infrared Spectrometry - Sample Handling



Gas samples

The spectrum of a gas can be obtained by permitting the sample to expand into an evacuated cell, also called a cuvette.

Solutions

Infrared solution cells consists of two windows of pressed salt sealed and separated by thin gaskets of Teflon, copper or lead that have been wetted with mercury. The windows are usually made of sodium chloride, potassium chloride or cesium bromide. Samples that are liquid at room temperature are usually analysed in pure form or in solution. The most common solvents are Carbon Tetrachloride (CCl_4) and Carbon Disulfide (CS_2). Chloroform, methylene chloride, acetonitrile and acetone are useful solvents for polar materials.

Solids

Solids reduced to small particles can be examined as a thin paste or mull. The mull is formed by grinding a few milligrams of the sample in the presence of one or two drops of a hydrocarbon oil. The resulting mull is then examined as a film between flat salt plates. In the reference beam path a window of the same thickness is placed. Another technique is to ground a milligram or less of the sample with about 100 milligram potassium bromide. The mixture is then pressed in an evacuable die to produce a transparent disk. In the reference beam path a disk of pure potassium bromide is placed.

Infrared Spectrometry - ATR & FT-IR

ATR - Attenuated total reflectance

Attenuated total reflectance uses a property of total internal reflection called the evanescent wave. A beam of infrared light is passed through the ATR, which reflects it at least once off the internal surface in contact with the sample. This forms an evanescent wave which extends into the sample. The beam is then collected by a detector as it exits the crystal. The evanescent effect works best if the crystal is made of an optical material with a higher refractive index than the sample being studied. With a liquid sample, pouring a shallow amount over the surface of the crystal is sufficient. If it is a solid sample, it is pressed into direct contact with the crystal. Because the evanescent wave into the solid sample improves with intimate contact, solid samples are usually clamped against the ATR crystal so that trapped air does not distort the results.

FT-IR - Fourier transform infrared

Fourier transform infrared, more commonly known as FT-IR, is the preferred method for infrared spectroscopy. Developed in order to overcome the slow scanning limitations encountered with dispersive instruments, with FT-IR the infrared radiation is passed through a sample. The measured signal is referred to as an interferogram. Performing a Fourier transform on this signal data results in a spectrum identical to that from conventional (dispersive) infrared spectroscopy, but results are much faster with results in seconds, rather than minutes.

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