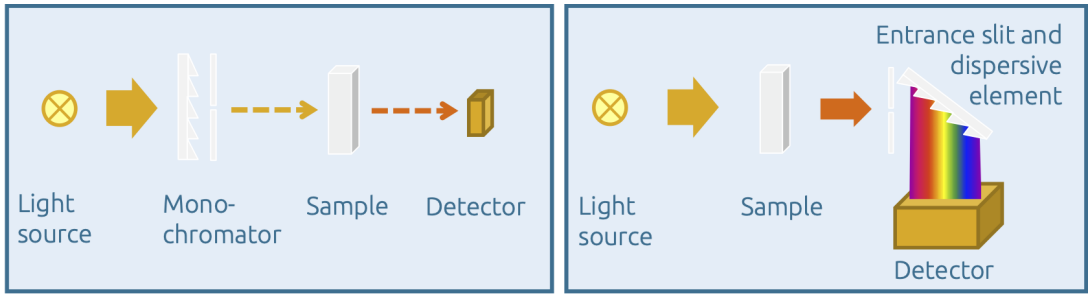
Measurement arrangements

The beam path of optical spectrometers is always based on the same principles. The radiation first passes through a monochromator, then the sample and is then recorded by the detector. Due to the scanning process of the monochromator through the spectral range, measurements with such spectrometers are time-consuming. If, on the other hand, the device has a detector array to detect all wavelengths simultaneously, the entire measurement can be performed instantaneously. In this case, the dispersive element is positioned after the sample.



Principal setup of optical spectrometers

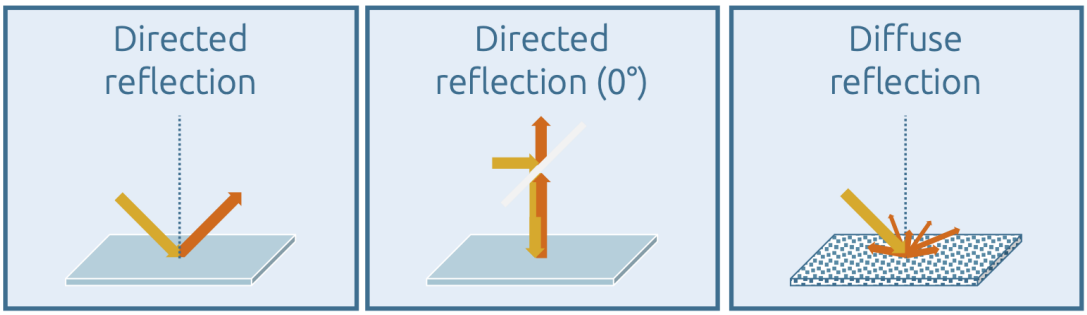
All current hyperspectral imaging spectrometers also work according to the last principle presented. Depending on the spectroscopy method, the type and wavelength of the excitation, the sample detection (transmission, reflection, etc.) and the arrangement of the detector in the beam path naturally differ. Additional lenses, optics and filters (polarization filters, grey filters, etc.) can also be placed in the beam path. Microscopes are suitable for realizing the smallest possible examination areas or examination of small structure widths. In addition to the UV/VIS range, such microscopes are also available for the IR range, although they require special optical components such as lenses for the IR spectral range.

Transmission

Transmission measurements are well suited for (semi-)transparent solids such as glasses and films, but also for gases and liquids in particular. With the latter, care must be taken to ensure that the layer thickness is not too great, as otherwise complete absorption can occur and the spectra will be distorted. For a quantitative evaluation, precise knowledge of the path length is also required. Liquid cuvettes are commercially available in large numbers and layer thicknesses. Transmission measurements can also be carried out using microscope optics to resolve smaller structure widths.

Directed and diffuse reflection

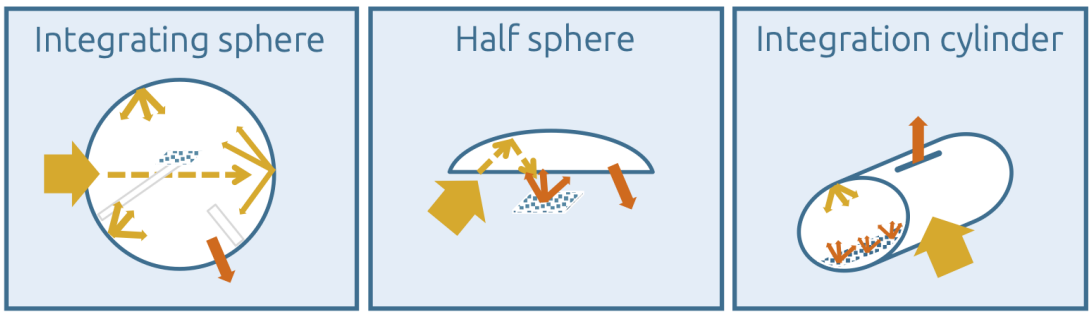
Once the sample under investigation is no longer (semi-)transparent, transmission measurements can no longer be carried out. Reflection methods in optical spectroscopy provide a remedy. They can be used in a wide range of applications and sometimes allow very different insights into the samples.



Overview of directed and diffuse reflection

With directional reflection, the angle of the observation direction must be equal to the angle of the incident radiation; this is realized by additional mirrors in the beam path. A special case of directional reflection is the measurement at 0° in relation to the sample standard (perpendicular incidence on the sample). To realize such a beam path, with identical direction of excitation and detection, a semi-transparent mirror must be used. Microscopes generally use this approach for reflection measurements. Sample surfaces should be very smooth or reflective for directional reflection. The wavelength-dependent reflection of the sample is measured, which is determined by the refractive index and absorption coefficient. In addition, the penetration depth is also dependent on the wavelength. In the case of thin layers (usually with low absorption), the interference pattern can also be recorded, which, together with other information about the layer, can be used to determine the layer thickness or the optical functions of the layer. The homogeneity of a coating can thus be checked independently of the knowledge of any coating parameters.

Diffuse reflection is very suitable for characterizing powders and rough surfaces. If there are many scattering centers (i.e. small particles on which the light is scattered), an undirected, diffuse light cone is created. This reflected, diffuse light cone also contains the absorption of the sample. To make this measurable, an integrating sphere is often used. It can be used to determine the total reflection of a sample, as the directional reflection is also recorded. If data on the directional reflection is also available, a sample can be characterized very precisely with regard to its reflection properties. In simpler setups, hemispheres are used for integration or, in the case of hyperspectral imaging measurements, integration cylinders. The integrating sphere and the integrating cylinder must have a rough but highly reflective inner coating, either made of gold, barium sulphate or PTFE. The half-spheres, on the other hand, are constructed in such a way that they capture the diffuse reflection of the sample after coupling in the radiation and focus the beam path towards the detector via directional reflection. The spectra obtained from the diffuse reflection can also be specified as absorption according to the Kubelka-Munk transformation.



Possibilities for implementing diffuse reflection measurements

Attanuated total reflection

Attenuated total internal reflection is based on the fact that the excitation radiation is guided in a crystal or an optical fiber and is completely reflected at its edges. The angle of incidence (in relation to the normal) must be very large, and total internal reflection only occurs if the difference between the refractive indices of the guiding medium and the cladding or surrounding matter is large enough (otherwise the radiation is coupled out). ATR spectroscopy is widely used in IR spectroscopy. A distinction is also made between single-reflection ATR and multi-reflection ATR. At the positions where the ATR crystal or fiber openly meets the sample, a so-called 'evanescent field' is formed and the excitation radiation interacts with the sample.[9] The advantages of ATR spectroscopy are the very high surface sensitivity and the small sample quantities required for the investigation (at least this applies to single-reflection ATR). However, ATR techniques play a subordinate role in hyper- and multispectral imaging.

References

[9] For a complete description, please refer to further literature, i. e. M. Otto, „Analytische Chemie“, Wiley-VCH, 4. Auflage 2011, ISBN 9783527328819

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