

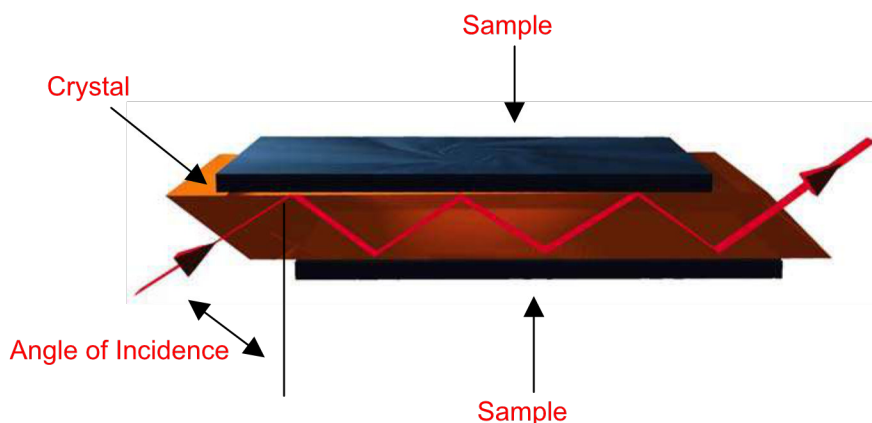
Keywords:- Reflectance, ATR, Diffuse, Specular, Solids, Liquids.

Reflectance Spectroscopy

Reflectance spectroscopy is used for samples that are difficult to analyze by transmission. The samples can usually be analyzed as is without the need for preparation or any modification.

Reflectance measurements can be divided into two basic categories – internal or external reflectance. The technique of Attenuated Total Reflection (ATR) is employed for internal reflectance measurements as the beam of infrared radiation passes through an ATR element (crystal) in contact with the sample. For external reflectance measurements, the infrared beam of radiation is reflected directly from the sample surface. This type of external reflectance measurement can also be divided into two types – Diffuse or Specular.

Attenuated Total Reflectance (ATR)

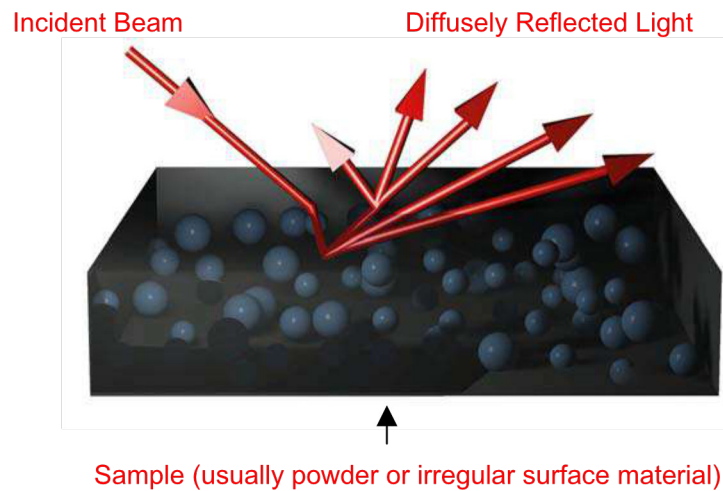


The diagram above shows a typical type of ATR experimental set up. In the example a parallelogram shaped ATR crystal is covered on two surfaces with a black sample material. The beam of infrared light traversing through the ATR crystal at a particular angle of incidence is being internally reflected at the top and bottom surfaces and in this instance there are 6 points of contact with the sample material (3 at the top and 3 below).

Internal reflection occurs when infrared radiation enters an ATR crystal made of a highly refractive infrared transmitting material. The crystal is designed to enable internal reflection, creating an evanescent wave at the crystal surface. This wave extends into a sample that is held in intimate contact with the crystal material and so absorption spectra can be recorded as a result. The depth of penetration of the evanescent wave into the sample is a function of the crystal material and the angle of incidence for the infrared beam traversing through the crystal to the sample surface interface. Deeper penetration into a sample is achieved with either a smaller incident angle or a lower refractive index ATR crystal. In general the depth of penetration into a sample increases with increasing wavelength of light.

The Specac accessories that are used for the analysis of solids and liquids by the ATR technique are the Golden Gate and Silver Gate single reflection ATR accessories, the Gateway 6 reflection ATR system and the 25 reflection ATR accessory.

Diffuse Reflectance

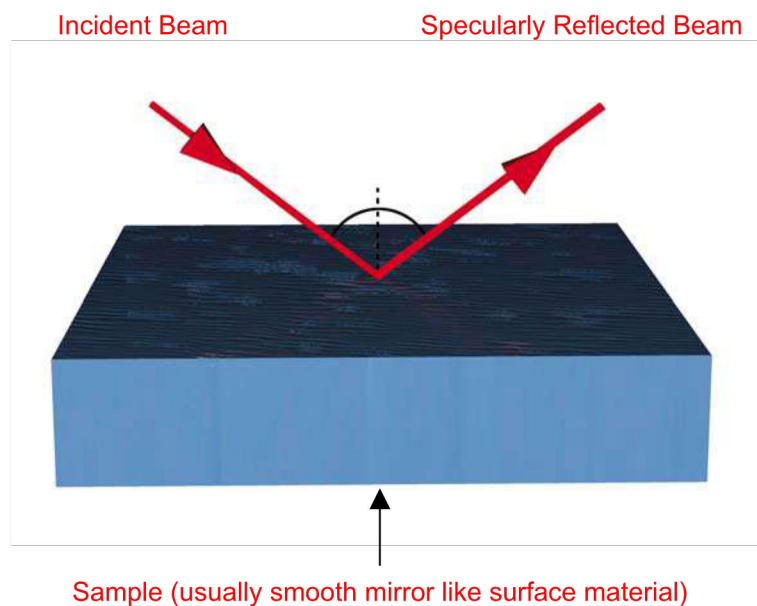


In external reflectance, incident radiation is focused onto the sample and two forms of reflection can occur – diffuse and specular. Energy from the incident beam that penetrates one or more particles is reflected in all directions and this component of light is called diffuse reflectance.

On a rough or irregular surface material, such as a powder, specularly reflected light is a minor contributor to the overall signal. Therefore Specac accessories are optimised to increase collection of the diffuse reflectance component and decrease the specular component. Collection of the diffusely scattered light can be made directly from a sample or by using an abrasive sampling pad for intractable samples. Many samples will give diffusely reflected spectra including powders, fibers or matt surfaced samples such as textiles.

There are two types of Specac diffuse reflectance accessories, the Minidiff Plus and the Selector. Specifically, the Selector has an “off axis” optical design to select for diffusely reflected light. It can also be extended in its capabilities for the study of samples at elevated temperatures and pressurised conditions by using with its complementary Environmental Chamber accessory.

Specular Reflectance



Specular reflectance is a non-destructive method for surface measurements using a mirror-like reflection from the shiny (or matt) surface of a sample.

Specular reflectance occurs when the reflected angle of infrared radiation equals the angle of incidence. The amount of light reflected depends on the angle of incidence, the refractive index, surface roughness and absorption properties of the sample.

A particularly useful application for specular reflectance is the study of surface coatings, such as surface treated metals, paints and polymers. Increased pathlengths through thin coatings can be achieved using grazing angles of incidence (typically 85° angle) and this gives increased sensitivity. Thicker coatings in the micrometer thickness range can be studied using angles typically at 30° incidence. The band intensities depend on the type and degree of polarization of light.

The Specac range of specular reflectance accessories includes both fixed angle and variable incidence angle accessories. The monolayer/grazing angle accessory can be used for range of incident angles from 8 to 85° and with both liquid and solid samples.