

Integrating Spheres – Introduction and Theory

Measuring Sample Reflectance

Reflectance sampling accessories rely upon a light beam coming from the spectrometer to be focused upon the sample. In order to achieve the best signal-to-noise ratio (SNR), the smaller the focus is, the easier it is to refocus the illuminated sample spot back onto the detector. In order to measure light reflected at a larger angle, optical designs will allow only a small area of the sample to be projected onto the detector. This arrangement serves well if the sample is microscopically homogeneous, but will result in a larger sample position error. When the sample is moved, the focused beam will see a different portion of the sample resulting in measurement-to-measurement differences. This is called insertion error because the spectrum will be slightly different each time the sample is inserted.

Some industrial or natural samples are inhomogeneous either because they are mixtures of different substances or because they have a particle size comparable to the probing beam diameter. Clearly, if the probing beam could be larger and the reflected light could all be collected, a more representative spectrum could be measured.

Some other samples develop a directional scattering. For example, fibers wound on a mandrel are highly oriented, not just macroscopically as parallel, unidirectional filaments, but also in many cases the molecules of the drawn fibers are oriented within the fiber itself. Such a sample, when placed in a reflectance accessory will generate different results depending on the angle from which the detector is “viewing” the sample. When the overall reflectance needs to be measured reproducibly, for example to measure the concentration of a minor ingredient in the sample, only isotropic optical systems, insensitive to such directionalities could be utilized.

Furthermore, in some cases, not just the reflectance in a small solid angle but the reflectance in all angles is sought. Most reflectance accessories measure at fixed or variable angles, narrower or wider collection angles, but there is a need for a device that uniformly collects all reflected light from a sample. In other words it measures the total reflectance of the sample.

Therefore the main reasons for using integrating spheres for the measurement of sample reflectance are the following:

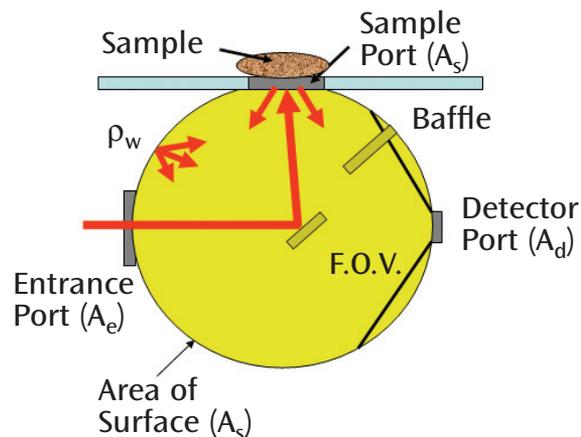
- Efficient measurement of combined diffuse and specular reflectance
- Uniform detection of reflectance even when sample is inhomogeneous
- Isotropic detection of reflectance even on samples that reflect in preferred directions
- Reduction of polarization effects from the illuminating beam and the sample
- Measurement of absolute reflectance (with special integrating spheres)

All of the above concerns are addressed with integrating sphere based reflectometers.

Integrating Sphere Optics

Integrating spheres are highly reflective enclosures that are placed in close proximity to the sample, such that the reflected light enters the sphere, bounces around the highly reflective diffuse surface of the sphere wall and finally impinges upon the detector – usually part of the integrating sphere assembly. The name, integrating sphere, refers to one of the main functions of the device, namely that it spatially integrates the light flux, in our application the light reflected from a sample. In spite of the long history of engineering and development of the sphere, the applications and further developments continue to this day. Advances in the theory, detector and electronics development and most of all, new applications, drive the progress.

As the name implies, the main part of the device is a sphere with a very highly reflecting inner surface. The surface should approach the ideal Lambertian scatterer, which means that the light falling on the surface is evenly scattered in all directions and the scattered light intensity is proportional to the cosine of the angle of observation.



Optical geometry of an integrating sphere

In an upward sample positioning sphere the infrared beam from the interferometer is directed through an entrance port onto the sample placed behind the sample port (shown above). Samples can be directly touching the sphere or separated from the sphere by a thin, infrared transparent window. The detector is placed close to the sphere, so that it can view the integrating sphere with a large solid angle. In order to improve the isotropy (non-directionality) of the detection, the detector is not directly in the line of sight of the sample. A small, also highly reflective and scattering baffle is placed in the sphere such that it blocks the first reflection of the sample from reaching the detector.

A well-designed sphere has the sample close to the sphere geometry so that the sphere will collect close to the full available hemispherical reflectance (2π steradians). A window to separate the sphere and sample may be important in some cases, but it will place the sample a small distance from the sphere, thereby somewhat reducing the collected high-angle reflectance. The PIKE Technologies integrating spheres are coated with the highest possible reflective surface for the desired wavelength region. The coating of the surface of the sphere has to be uniform and close to being a perfect Lambertian scatterer. These characteristics allow the light falling in the sphere to be uniformly distributed over the entire surface of the sphere. It is also important how much of this light is actually collected on the detector surface.

Sphere Throughput

The throughput of the sphere is defined as the ratio of the incoming light impinging on the detector. The closer the sphere surface is to ideal reflectance, the higher the throughput. The detector, the sample and the illumination require that a portion of the wall of the complete sphere be removed. Smaller cutouts for beam input and output result in higher energy throughput. Due to other considerations, such as reduction of light scatter from the edges of the sphere cutouts, called ports, these have to be optimized and cannot be too small.

The throughput can be expressed with these sphere design parameters:

$$\tau = \frac{A_d}{A_s} \times \frac{\rho_w}{\rho_{w, \text{avg}}}$$

Where A_d is the detector area, A_s is the sphere area, ρ_w is the sphere wall hemispherical reflectance, $\rho_{w, \text{avg}}$ is the average sphere wall reflectance.

The sphere throughput is higher if the light falling on the detector is increased by the multiple reflections of the light. Another way of looking at the integrating sphere is that it enhances the detector signal by collecting the light, and if the wall surface is reflective enough, bounce it around until it illuminates the detector. The factor that is used to express this gain is called the sphere multiplier (M), which is a function of the wall reflectance (ρ_w), the proportion of the total area of ports to the surface of the sphere (f).

$$M = \frac{\rho_w}{1 - \rho_w(1 - f)}$$

The brightness of the sphere, using the same amount of input light flux, is dependent on the wall reflectivity, the port-to-sphere surface ratio and the size of the sphere surface.

$$L_s = \frac{\Phi_i}{\pi A_s} \frac{\rho_w}{1 - \rho_w(1 - f)}$$

where Φ_i is the input light flux and A_s is the area of the sphere wall surface.

For the sphere the area of the sphere obviously depends on the sphere diameter, and thus the formula shows that a smaller sphere is brighter than a larger diameter one.

$$L_s \sim \frac{M}{D^2}$$

The sphere diameter cannot be reduced too far however, because the sample diameter will also have to be decreased proportionally when the sphere is smaller. For typical spectroscopic applications the optimum sphere diameter is influenced by the beam size coming from the FTIR spectrometer and the typical sample size of 3-25 mm. Most integrating sphere modules use a 2-4 inch diameter sphere to accommodate the above design parameters. In a practical design, the openings of the sphere need to be kept around 5% for optimum throughput. Wall reflectance is usually between 95-99% and results in a sphere gain of 10-30.

Integrating Spheres for Mid-IR and NIR

Integrating spheres, although much more efficient than an optical system with an equivalent detector position, still have lower throughput than the direct imaging optics. In the visible and NIR spectral region, where there are very good sources and excellent, high-speed detectors are readily available, the SNR is usually not limited by the reduced light level. In the mid-IR spectral region, in order to utilize the above discussed advantages and benefits of integrating spheres, the reduced throughput needs to be offset by the use of the high sensitivity, cooled detectors, such as the liquid nitrogen cooled MCT detector utilized by PIKE Technologies. The near-infrared and mid-infrared measurements using integrating sphere optics have different analytical and measurement goals as well as different features. PIKE Technologies offers both mid-IR and NIR versions.