SmartNote | SN1821

NIR in a nutshell

Introduction to integrating spheres and their benefits to near-infrared spectroscopy

Integrate more light into your analysis

Near-infrared (NIR) light can interact with materials in several ways, including transmission and reflection. Light can be reflected by materials in a specular manner, meaning the light is reflected from the material at an angle matching the angle of incidence, or in a diffuse manner, wherein the light is reflected at angles other than the angle of incidence. The amount reflected in each manner depends on the properties of the sample: A smooth, glossy surface will reflect more in a specular geometry, while a rough or matte surface will reflect more diffusely. The latter description is applicable to powders and many solid materials tested with NIR instrumentation; because of this, these types of samples are best analyzed with an integrating sphere due to its efficiency in the collection of diffusely reflected light.

A common challenge in diffuse reflection measurements is the collection of light which is reflected across a wide range of angles. Simply orienting a detector towards the sample would provide poor measurements, as the detector element is small compared to the "cone" of diffusely reflected light. A mirror could be used to capture light reflected from the sample, collecting more light, though it would still only be a fraction of the diffuse radiation. This limited collection translates to higher sensitivity to sample packing, texture, and optical properties, which hinders quantitative efforts. A third, and arguably the most effective configuration to overcome this challenge of diffuse reflection, is the use of an integrating sphere—a spherical cavity atop which the sample is positioned.

A sphere for efficiency

The spherical shape, illustrated in Figure 1, allows collection of diffuse reflected light with very high efficiency. Integrating spheres in the Thermo Scientific™ Antaris™ II FT-NIR Spectrometer can collect more than 95% of diffuse reflected light. Efficiency of light collection is critical for samples such as grains, seeds, or polymer pellets, where the sample's shape is irregular, or the material is not highly reflective. Additionally, the integrating sphere is equipped with an automated internal diffuse gold reference flag, positioned beneath the top port window. This feature streamlines sample analysis while also protecting the reference material from contamination, damage, or loss.

While the integrating sphere is an ideal solution for analysis of solids and powders, it can also be configured for analysis of liquids, gels, and pastes. Thermo Fisher Scientific has engineered a solution for measurement of challenging samples known as the "viscous liquid sampling accessory," which sits atop the integrating sphere. The device consists of a base plate and an arm which contains a diffuse gold mirror behind a protective window. The accessory includes three arms which provide controlled gaps of 0.5, 1.0, and 2.0 mm for pathlengths of 1.0, 2.0, and 4.0 mm, respectively. Materials which are difficult to clean may be analyzed through packaging such as baggies; these liquid-filled baggies can be placed under the arm which provides a pathlength appropriate for measurement. The sample and baggie can be disposed of following analysis, preventing measurement errors from sample carryover that might occur if the viscous liquid were analyzed in a cuvette.

While samples may be analyzed in single-use vials to eliminate the possibility of carryover, it may be challenging to effectively fill the vial with sample. Other materials such as translucent rubber or other solids may be analyzed directly on the viscous sampling accessory. Such materials are often too transparent for a “typical” diffuse reflectance measurement, but too thin or irregularly-shaped for a transmission measurement.

Increased efficiency with a Thermo Scientific integrating sphere

Integrating spheres offer superior collection of diffuse reflected light compared to mirror- or lens-based interfaces. Improved optical efficiency reduces sensitivity to sample morphology and packing, resulting in more repeatable measurements and more robust NIR methods. The Thermo Scientific™ Integrating Sphere for the Antaris II can be outfitted with viscous sampling accessories which extend utility of the system to measurements of translucent solids, gels, and liquids which would otherwise prove too challenging for other instruments. These capabilities make the Antaris II spectrometer adaptable to the diverse analytical demands of modern laboratories.

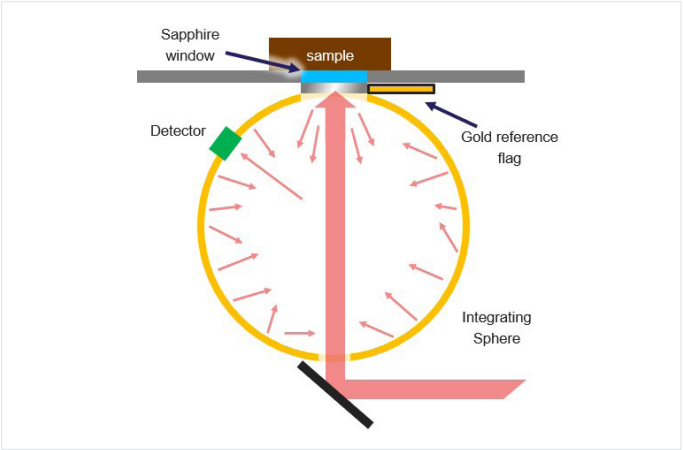

Figure 1. Diagram illustrating the spectral measurements with the integrating sphere sampling module.

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