

Integrating Spheres



Integrating spheres are useful for qualitative and quantitative measurements of sample composition when morphology, particle size, surface roughness or sample flatness varies from sample to sample. PIKE Technologies offers fully integrated accessories for mid-IR and NIR applications.

Mid-IR IntegratIR

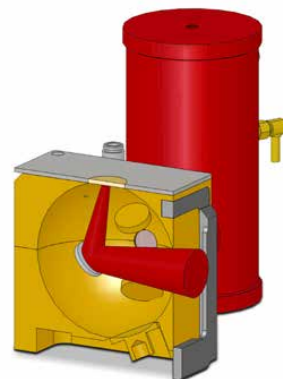
AT A GLANCE

- ▶ 3-inch sphere, gold-coated, Lambertian scatterer for high-performance measurements
- ▶ 12-degree hemispherical diffuse reflection measurement with specular exclusion port
- ▶ Diffuse transmission station for measurement of highly scattering samples
- ▶ Choice of integrated, high-performance detector MCT or DTGS for ultimate configurability
- ▶ Upward- and downward-looking optical configurations to accommodate a wide range of sample sizes and types
- ▶ In-sample-compartment design to minimize laboratory space requirements

The Mid-IR IntegratIR is an integrating sphere that is often used when studying reflection properties of solids, analyzing light-scattering and/or highly absorbing samples and collecting spectra that is difficult to obtain with standard sampling techniques.

OPTICAL DESIGNS

The Mid-IR IntegratIR™ spheres are available in upward- and downward-looking configurations and are suitable for the measurements of absolute and relative diffuse reflectance of solids, powders and opaque liquids. Both feature a 3-inch diameter, highly reflective gold-coated sphere. The spheres mount in the sample compartment of the FTIR spectrophotometer, and use a dedicated detector for maximum performance.



Optical diagram of the upward-looking IntegratIR.

The upward- and downward-looking mid-IR spheres feature a 12-degree illumination of the sample, and offer a specular exclusion port for measuring total reflection (diffuse plus specular) and reflection of the diffuse component only. For both spheres, the selection of light illumination onto the sample or onto the reference surface is done via a flipper mirror. This allows the background to be collected using either the substitution method or the Taylor method.



Gold-coated Lambertian finish sphere.



SPECIFICATIONS

Optical Design	Upward- or downward-looking sample spheres
Angle of Incidence	12 degrees
Sphere Diameter and Surface	76.2 mm (3") gold-coated Lambertian surface
Sample Port Size	20 mm
Specular Exclusion Port	Standard
Sphere Dimensions (W x D x H)	159 x 248 x 154 mm (excludes baseplate)
Sample Opening, Downward Sphere	50.8 x 35.5 x 12.7 mm
Spectral Range, MCT Detectors	Wide-band: 5000–500 cm^{-1} Mid-band: 5000–650 cm^{-1} Narrow-band: 5000–800 cm^{-1}
Extended DTGS Detector	5000–400 cm^{-1}
InGaAs Detector	12,200–3850 cm^{-1}

UPWARD-LOOKING MID-IR INTEGRATIR

For the upward-looking sphere, reflectance samples are placed directly onto the sample port located on the top of the sphere. This sphere is ideal for large and/or thick solid samples. For powders, a ZnSe window plate is available. If preferred, a KBr window can also be used with the sample plate to minimize the reflection loss compared to ZnSe.

DOWNWARD-LOOKING MID-IR INTEGRATIR

The downward-looking Mid-IR IntegratIR allows the sample to be placed underneath the sphere. This configuration is desirable for measurements of powders and particulate materials because the incidence beam strikes the sample directly, without passing through an IR transparent window.

DIFFUSE TRANSMITTANCE MEASUREMENTS

Diffuse transmittance of partially transmitting materials can be measured with both spheres. This is done by placing the sample on a standard 2 x 3" sample holder and sliding it in the mount located in the incoming beam port.

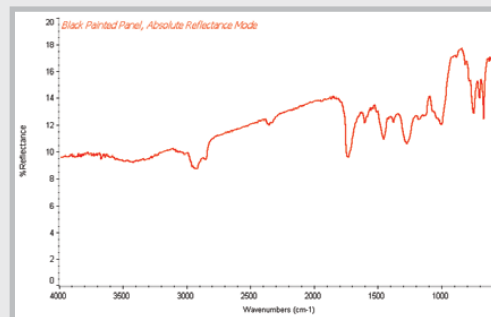
DETECTOR OPTIONS

A selection of mercury cadmium telluride (MCT) or deuterated triglycine sulfate (DTGS) detectors is offered with the IntegratIR spheres. The wide-band MCT is the commonly configured detector while the less sensitive DTGS is an option for users who require the convenience of a room temperature detector. The MCT detector is approximately 50 times more sensitive compared to the DTGS detector. All detectors are interchangeable. For those FTIR spectrometers with near-IR spectral capabilities, a sensitive InGaAs detector is available.

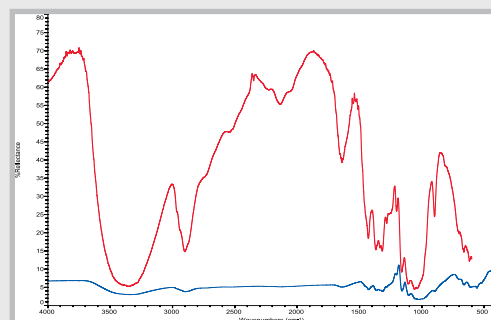
Downward-looking IntegratIR.

APPLICATION

An integrating sphere is ideal for measuring reflectance and transmittance of diffusely scattering material, as the accessory enables the hemispherical collection of scattered IR beam caused by the sample.



Absolute reflectance spectrum of a painted black panel measured using the Mid-IR IntegratIR.

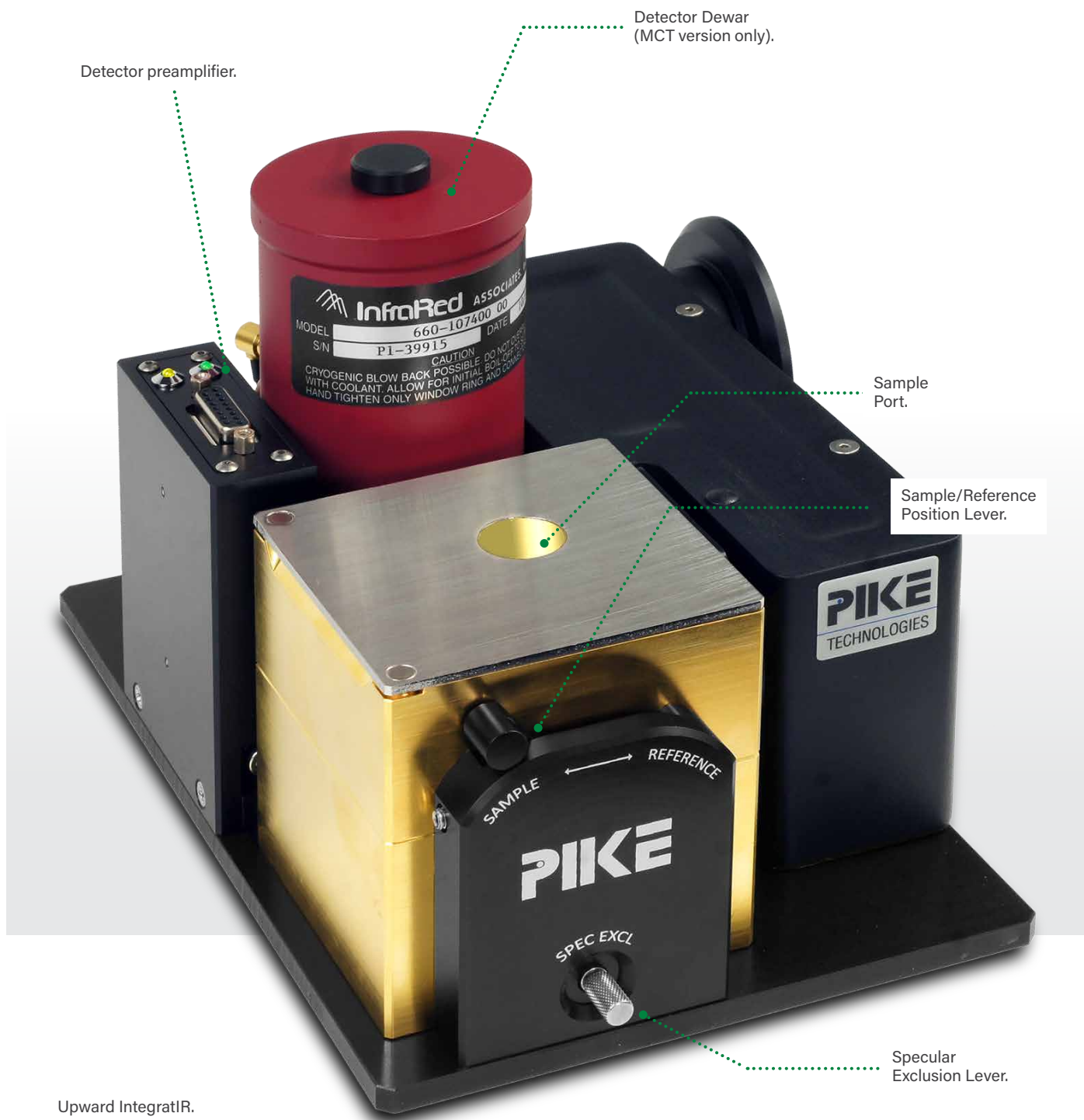


Comparison of transmittance spectra of diffusely scattering paper collected using an IntegratIR (red) versus without a sphere (blue).



PART NUMBER	DESCRIPTION
048-12XX	Upward Mid-IR IntegratIR Integrating Sphere Includes sphere, purge enclosure and tubing, diffuse gold reference and sample plate with ZnSe window
048-11XX	Downward Mid-IR IntegratIR Integrating Sphere Includes sphere, purge enclosure and tubing, one diffuse gold reference and powder sample cup Notes: replace XX with your spectrometer's Instrument Code listed in the back of the catalog. Your FTIR spectrometer must be capable of interfacing with an external detector.
Detector Choice for IntegratIR (<i>must select one</i>)	
048-3350	Wide-band MCT Detector
048-3250	Mid-band MCT Detector
048-3150	Narrow-band MCT Detector
048-3550	InGaAs Detector
Notes: Detector includes preamplifier electronics. MCT detectors require liquid nitrogen for cooling.	

PART NUMBER	DESCRIPTION
Replacement Parts and Sampling Options	
048-0108	Sample Plate with 20 x 2 mm ZnSe Window for Upward IntegratIR
048-0208	Sample Plate with 20 x 2 mm KBr Window for Upward IntegratIR
048-3000	Diffuse Gold Reference for Upward IntegratIR
048-3001	Diffuse Gold Reference for Downward IntegratIR
048-2020	Powder Sample Cup for Downward IntegratIR



Upward IntegratIR.

External Integrating Sphere

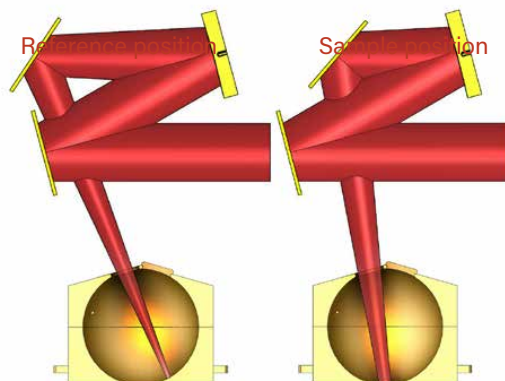
AT A GLANCE

- ▶ Accommodates large-sized samples
- ▶ 4-inch sphere, gold-coated, Lambertian scatterer for high performance measurements
- ▶ 8-degree hemispherical diffuse reflection measurement with specular exclusion port
- ▶ Manual translation mirror to switch between the reference and sample position
- ▶ Integrated, high-performance MCT detector
- ▶ Utilizes external spectrometer beam to allow for the analysis of oversized samples positioned under the sphere
- ▶ Fully purgeable enclosure

The External Integrating Sphere utilizes the external beam of the spectrometer. It is ideal for large samples because it is not limited to the confines of the sample compartment. This sphere is great for highly precise reflectivity measurements.

OPTICAL DESIGN

The internal optics of the External Integrating Sphere focus light from the external beam of the spectrometer into a 4-inch gold-plated integrating sphere. A translation mirror is moved manually using the mirror position lever, located on the enclosure of the accessory for precise movement between the sample and reference positions. In the sample position, incident light is 8° from normal. Specular reflection may be excluded by opening a port at the top of the sphere. The detector port is 90° from the sample port.



Optical diagram of the FTIR external beam path for reference and sample positions.

Accurate measurement of both solid- and liquid-phase samples is possible with the 4-inch External Integrating Sphere. By utilizing highly-accurate Taylor methodology for measurement, high-quality components, and sensitive MCT detection, the External Integrating Sphere offers low-noise, highly accurate measurements for a wide range of samples.

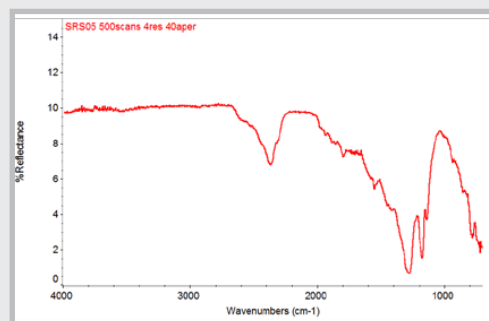


SPECIFICATIONS

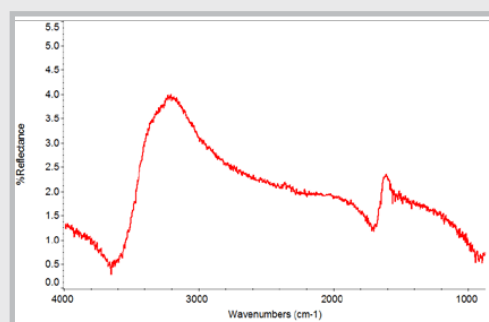
Optical Design	Downward-looking sample sphere
Angle of Incidence	8 degrees
Sphere Diameter and Surface	101.6 mm (4") gold-coated Lambertian surface
Sample Port Size	20 mm
Specular Exclusion Port	Standard
Spectral Range, MCT Detectors	Wide-band: 5000–500 cm^{-1} Mid-band: 5000–650 cm^{-1} Narrow-band: 5000–800 cm^{-1}
FTIR Placement	Right or Left Side

APPLICATION

The External Integrating Sphere offers precision measurements for samples spanning from high to low reflectance. By using the external beam of the spectrometer, this accessory can accommodate small to large samples.



Reflectivity standard spectrum, highlighting the quality data of a low-reflectivity sample.



Water reflectivity spectrum, collected using the External Integrating Sphere.

PART NUMBER	DESCRIPTION
048-13XXL	Mid-Infrared External Integrating Sphere Accessory — Left Includes sphere, purge enclosure and tubing.
048-13XXR	Mid-Infrared External Integrating Sphere Accessory — Right Includes sphere, purge enclosure and tubing.
Notes: Replace XX with your spectrometer's Instrument Code listed in the back of the catalog. Accessory uses the spectrometer's external beam. Spectrometer must be equipped to accept an external detector.	
Detector (<i>must select one</i>)	
048-3360	Wide-Band MCT Detector for External Sphere
048-3260	Mid-Band MCT Detector for External Sphere
048-3160	Narrow-Band MCT Detector for External Sphere
Options	
048-3000	Diffuse Gold Reference

NIR IntegratIR

AT A GLANCE

- ▶ Optimized 2-inch gold-coated integrating sphere with high signal-to-noise ratio
- ▶ Fully integrated InGaAs detector for greater sensitivity and faster measurements
- ▶ 10-mm horizontal sampling port for easy sample placement
- ▶ Excellent qualitative and quantitative NIR analysis tool
- ▶ Optional rotating stage for averaging of heterogeneous samples
- ▶ Spectral range 12,200–3850 cm^{-1}
- ▶ In-sample-compartment design

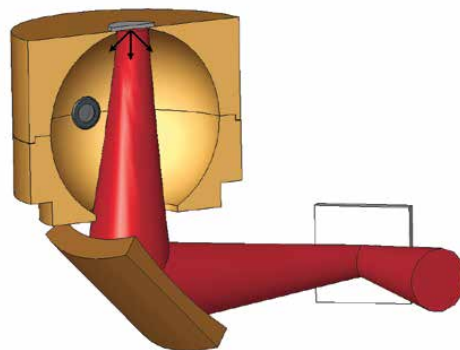


The NIR IntegratIR™ is a near-infrared (NIR) integrating sphere for quantitative and qualitative measurements of a wide variety of solids and paste materials.

The NIR IntegratIR collects reflected energy from a spherical perspective thereby capturing complete and quantitative response from the sample. Using near-infrared chemometrics, qualitative product identification and quantitative analysis may be performed on pharmaceutical, nutraceutical, chemical, polymer, textile, food, agricultural and other samples.

OPTICAL DESIGN

The NIR IntegratIR accessory features a 2-inch reflective gold-coated integrating sphere and an extended range, high-speed, low-noise indium gallium arsenide (InGaAs) detector, transfer optics and interface electronics. The accessory fits into the sample compartment of most commercial FTIR spectrometers, which are able to interface with an external detector. A 10-mm diameter horizontal sampling port makes the placement of samples onto the accessory easy. An optimized borosilicate window serves as a sampling port at the top of the integrating sphere. The window is bonded and sealed to protect the sphere from corrosive materials and contamination.



Optical diagram of the NIR IntegratIR.

SAMPLING OPTIONS

High-quality spectra are produced quickly using the NIR IntegratIR, making qualitative and quantitative analysis of a wide variety of sample types efficient and reliable. Sampling of tablets, packaging materials and plastics is easily accomplished by placing the sample directly on the window of the upward-looking sphere. Powders, creams, pastes or liquids containing reflective particles may be placed in disposable flat-bottom vials—eliminating the need for any sample cleanup. The vials may be held in place by a sample-positioning vial holder, resulting in more repeatable measurements.

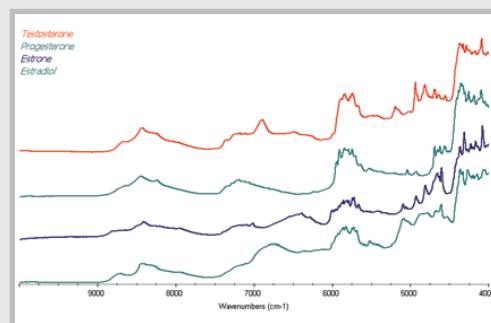
SPECIFICATIONS

Sphere Diameter	50.8 mm (2")
Internal Surface	Lambertian gold
Illumination Angle	Normal
Input Beam Port Diameter	18.8 mm
Detector	Integrating InGaAs, 2 mm diameter
Spectral Range	12220-3850 cm ⁻¹ ; 0.82-2.6 micron
Sample Port	10 mm diameter
Sampling Mode	Diffuse Reflection
Background Collection	Substitution
Specular Rejection	Standard
Automation Options	Rotating stage, automated tablet analyzer
Accessory Dimensions	167 x 210 x 157 mm (excludes FTIR baseplate and mount)

For heterogeneous samples, PIKE Technologies offers a rotating stage for the NIR IntegratIR. With this option, one obtains an averaged result to eliminate variations in quantitative measurements for the chosen sample area.

APPLICATION

The NIR IntegratIR is great for making quantitative and qualitative measurements of biological, chemical and pharmaceutical products. The NIR spectral region often requires no sample preparation. Samples may often be analyzed through a vial or plastic packaging films.



Steroids spectra for quantitative analysis measured through glass vials using the NIR IntegratIR.

PART NUMBER	DESCRIPTION
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048-60XX	NIR IntegratIR Integrating Sphere Accessory Includes 2" diffuse gold-coated integrating sphere, InGaAs detector, detector preamplifier, diffuse gold reference, vial holder, and 25 glass vials.
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Notes: Replace XX with your spectrometer's Instrument Code listed in the back of the catalog. Your spectrometer must be capable of interfacing with an external detector.

Sampling Options

048-3000	Diffuse Gold Reference
162-5483	NIST Traceable NIR Reference Standard
162-5484	NIST Traceable NIR Reference Standard – Recertification
044-3010	Glass Vial Holder for 19-mm vials
048-2999	Glass Sample Vials, 19 x 65 mm (25 ea.)
048-0150	Rotating Stage for Petri dish for heterogeneous samples Includes 100 x 20 mm Petri Dish
048-0151	Rotating Stage Adapter for 500 mL beaker

Note: Please contact us for other options. Stage rotates counterclockwise.



Rotating stages available for beakers and Petri dishes.

Integrating Spheres

Theory and Applications

Integrating spheres are useful sampling accessories for the hemispherical collection of scattering light caused by reflection off or transmission through diffuse material.

WHY USE AN INTEGRATING SPHERE?

When measuring sample reflectance, the main reasons for using integrating spheres are:

- ▶ Efficient measurement of combined diffuse and specular reflection.
- ▶ Uniform detection of reflectance even when the sample is inhomogeneous.
- ▶ Isotropic detection of reflection 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).

MEASURING REFLECTION

Traditional reflection 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. 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

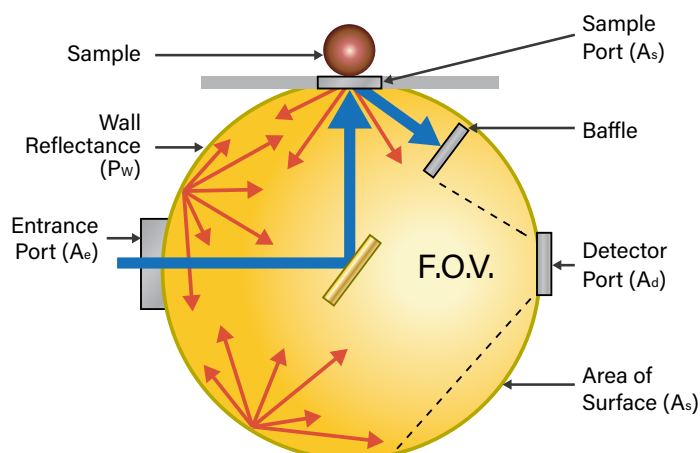
“viewing” the sample. When the overall reflectance needs to be measured reproducibly, for example to measure the concentration of a minor ingredient in the sample, only isotropic optical systems, insensitive to such directionalities could be utilized.

In some cases, not just the reflectance in a small solid angle is sought, but the reflectance in all angles. Most reflection 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.

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 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.



In an upward-sample-positioning sphere the infrared beam from the interferometer is directed through an entrance port onto the sample, placed on top of the sphere. 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, 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 nearly all the 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 gold 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.

INTEGRATING SPHERE THEORY

The throughput of a single sphere may be defined as a function of the hemispherical reflectance to the average spherical wall reflectance ratio. 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 sphere wall 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:

$$\tau = \frac{A_d}{A_s} \times \frac{\rho_w}{(1 - \rho_{w, \text{avg}})}$$

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).

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

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

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

where Φ_i is the input light flux.

For the sphere the area depends on the sphere diameter (D), and thus the formula shows that a smaller sphere is brighter than one with a larger diameter.

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

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.