Application Note No. 03

Written by: Shannon L. Storm, Ph.D.,
Reflectance Spectroscopy Product Specialist
Art Springsteen, Ph.D., *Principal Scientist*

Choosing the Right Sphere Size for Your Application

The Integrating Sphere Reflectance Accessory

This article is designed to aid UV-VIS and UV-VIS-NIR Spectrophotometer users in determining the most suitable integrating sphere accessory for their sample measurement needs. It will present the operating features of both the 60 mm and the 150 mm diameter integrating sphere reflectance accessories.

Sphere coatings, sphere efficiency, signal throughput, spectrum noise level, port fraction, measurement accuracy, and design options are discussed. A chart detailing the advantages and limitations for each sphere size is presented on page 6.

Integrating Sphere Reflectance Accessories

Integrating sphere reflectance accessories offer a simple method to extend the measurement capabilities of a UV-VIS or UV-VIS-NIR spectrophotometer. By adding a sphere reflectance accessory to a spectrophotometer, one is able to expand from traditional transmittance measurements of liquid samples and non-scattering solids to incorporate both reflectance measurements of opaque solids, powders, or pastes, as well as total transmittance scans of translucent films and scattering creams. Minimal preparation is required for most samples when a reflectance accessory is used, as samples can often be measured in an unprocessed form.

With integrating sphere accessories, most of the light scattered by a translucent or opaque sample is collected by the sphere. In many integrating sphere accessories the detector is placed directly on the sphere's surface to improve detection efficiency. This design ensures accurate, quantitative measurements for diffuse samples. Reflectance accessories are specially designed for each UV-VIS or UV-VIS-NIR spectrophotometer so that they are compatible with the optics, electronics, and software of the host instrument.

Traditionally, integrating spheres were fabricated from metal spheres that were internally coated with a highly reflective white paint or packed powder such as magnesium oxide, barium sulfate, or PTFE (polytetrafluoroethylene). While these materials are highly reflective when first applied to a sphere, the coating reflectance degrades over time. Labsphere offers a diffuse reflectance material which exhibits the high reflectance properties found in the white paint coatings and packed powders but also offers unmatched long term stability of reflectance. This material, a white thermoplastic with the trade name Spectralon,® offers very high diffuse reflectance over the range of 190-2500 nm and is extremely durable.[1]

labsphere®

Unlike the traditional integrating sphere coatings which need to be recoated or repacked on a yearly basis to maintain high reflectivity, Spectralon maintains its reflectance indefinitely under normal laboratory conditions.

Given the advantages of integrating sphere reflectance accessories for the measurement of diffuse and total reflectance or transmittance, it is easy to see that such an accessory is useful for many laboratory applications. However, many spectrophotometers have available both internal sample compartment accessories, with 50-60 mm diameter spheres, and external reflectance accessories, with 150 mm diameter spheres. How does a user decide between the large and small diameter integrating spheres when choosing an accessory? Let's take a look at some of the principal advantages and limitations of each.

Integrating Sphere Efficiency and Throughput

Smaller integrating spheres are efficient collectors of light. The throughput of the system, that is, the amount of incident light which reaches the detector, is governed by the laws of probability. In a small integrating sphere, a photon will have to take a certain number of bounces before it reaches the detector. In a larger sphere system, however, statistics dictate that a photon will have to take more bounces than were required in the smaller integrating sphere to reach the same detector. In the larger sphere, since more "bounces" are required, the photon must undergo many more interactions with the sphere wall and, therefore, is more likely to be absorbed before it actually reaches the detector. Thus, a large 150 mm integrating sphere acts as an attenuator of signal and is inherently less efficient than a 60 mm diameter sphere. The larger the sphere, the greater the attenuation which results.[2] A rule of thumb, all other factors being equal, is that the relative attentuation is roughly equal to the square of the ratio between the two sphere diameters. As we'll present, things are not always equal, so this rule can only be used as a rough guide.

Spectrum Noise Level

A sphere's efficiency may play an important role in the measurement of highly absorbing samples. When the high absorbance sample being tested limits the amount of light reaching the sphere's detector, a high throughput, small diameter sphere will generate better spectra with less noise than a larger sphere mounted to the same spectrophotometer and using the same detector. Thus, spectral results of small spheres tend to have greater signal-to-noise levels than those of larger spheres. In addition, the small sphere does not attenuate the instrument's sample beam as significantly as a large diameter sphere, thereby only slightly affecting the overall linearity range of the instrument.[3]

Port Fraction

Ideally, the integrating sphere accessory design should be flexible enough to allow the user to optimize the sphere diameter based on the application's required size, number of port openings and peripheral devices. While this is often true for those who purchase stand-alone integrating spheres as general equipment for optical laboratories, customers of spectrophotometer reflectance accessories generally have access only to generic sphere designs which cannot be modified to fit individual needs. In this case, it is important to understand the effects which the sphere's diameter and port fraction have on the performance of the integrating sphere accessory.

The port fraction is defined as the ratio of the total port area relative to the total internal surface area of the sphere.[2,4] All beam entrance ports, sample ports, and detector ports which are filled with material of lower reflectance than the Spectralon sphere wall contribute to the calculated port fraction. The port fraction is significantly lower for 150 mm diameter spheres than it is for 60 mm spheres. For example, the port fraction of a representative Labsphere 150 mm double beam integrating sphere accessory is 2.5 %, while a 60 mm sphere for the same instrument has a port fraction of 11.3%. The design of both accessories includes sample and reference beam transmittance and reflectance ports, as well as PMT and PbS detector ports.

In order to adhere to many ASTM and CIE methods for measurements using integrating spheres, the port fraction of the sphere must be minimized. For instance, CIE recommends[4] that the sphere's port fraction be lower than 10% for color reflectance measurements, whereas ASTM D1003-95 requires[5] the sphere to have a total port fraction less than 4% for haze measurements on transparent plastics. Thus, 150 mm diameter integrating spheres can be used for these methods. A 60 mm diameter integrating sphere with the standard transmittance, reflectance, and detector ports is often unable to meet these strict port fraction requirements.

A low port fraction ensures good integration of the sample signal before it reaches the sphere's detector. The influence of port fraction on sphere radiance is discussed further in the section entitled "Measurement Accuracy."

Measurement Accuracy

Obviously, the integrating sphere's design will affect its measurement accuracy. The size and location of ports, detectors, and baffles will influence how the light bounces around the sphere. As will be discussed in this section, large 150 mm diameter spheres have better light integration and their measurements are less likely to be affected by hot spots. The signal integration is not as good in smaller spheres, and the large port fraction typically found in 60 mm spheres can introduce significant errors in measurement due to flux loss.[2,6] All of these factors must be considered when choosing an integrating sphere accessory which is appropriate to the user's application.

Sphere Baffle Design

When designing integrating spheres for sample measurements, it is important that the detector's field of view does not include any portion of the sphere surface directly irradiated by the sample beam or the first reflection from the sample.[2,6,7] This would introduce a false response into the measurement. Baffles are typically made from thick pieces of Spectralon or from metal which has been coated with the same material as the integrating sphere wall. Baffles are used to block the detector's view of light which has not undergone at least two reflections from the sphere surface. Thus, the baffle is positioned to prevent the so-called 'first-strike' reflections from entering the field of view of the detector.

The size and position of baffles within the integrating sphere are very important factors which influence the system's measurement accuracy. As described in ASTM E903, "large errors can arise if the angular distribution of the light reflected from the specimen is different from that reflected by the standard.[6]" An example is given in transmittance measurements of translucent samples. Measurement errors may occur since the translucent sample, which scatters light, is measured relative to the nonscattering open port (air), which is used for the background correction as a standard. Careful baffle design can substantially reduce errors due to the different light scattering distribution of samples and standards. However, baffle design must always be performed with respect to the overall radiance characteristics of the sphere. The balance between baffle design and sphere flux is an important consideration when choosing an integrating sphere design.

The Perfect Sphere a balance between proper baffle design and light integration

The distribution of light within an integrating sphere will drastically affect its measurement accuracy. While small spheres do have higher energy efficiency than their 150 mm diameter counterparts, large integrating spheres will yield measurements with greater accuracy since the light in large systems can be 'integrated' or distributed evenly about the sphere's surface. The large internal surface area and the small overall port fraction of 150 mm spheres allows the light to reflect properly around the sphere, creating a homogeneous flux. However, in the design of small integrating sphere accessories, sphere flux homogeneity must often be compromised in an attempt to reduce the effects of hot spots.

Hot spots are areas within the sphere which appear brighter to the sphere's detectors than other portions of the sphere. Measurement errors, sometimes termed[7] regular reflectance screening errors, can result from hot spots, especially when measuring glossy or specular, mirror-like samples. The reflectance of mirrors may appear higher than the true value if the sphere's detectors are not baffled from the spot on the sphere wall where the first-strike radiation hits. As described above, spheres are designed with baffles between the detectors and each sample port to minimize such occurrences. A sphere's baffles are intended to eliminate hot spots or to shield the sphere's detectors from viewing them directly.[2,6,7]

Hot spots are more prevalent in small 60 mm diameter spheres. In these spheres, it is often impossible to design baffles to the proper dimensions needed to adequately shield the sphere's detector from view of a sample port or another bright spot. In small spheres, the sizes of baffles must often be minimized. The problem is one of real estate—there is just not enough space to fit properly sized baffles into the 60 mm diameter sphere in the proper places. Often the final integrating sphere baffle design is a compromise between the ideal baffle size which will fully shield the detector and a smaller baffle size which will interfere less with the sphere's flux homogeneity.

Substitution Errors

Small integrating spheres which are used on single or dual beam (beamsplitter-based) spectrophotometers are subject to sample substitution errors. Due to the dual beam instrument's characteristics, only the sample beam can be placed into the integrating sphere, and the reference beam must remain outside. The single beam substitution error which occurs during color measurements using such spheres is the systematic, predictable, and non-random error inherent in single beam integrating spheres measuring reflectance or transmittance. The error is caused by the difference in the throughput of the sphere when the reference makes up a portion of the sphere wall and when the sample is substituted for the reference.[7,8] Integrating sphere accessories that are used on double beam spectrophotometers, or those instruments which utilize a beam chopper, are not subject to this error since both the sample and reference beams can be located in the sphere at the same time.

Single beam substitution errors can approach 10%R for diffuse reflectance samples.[8] In quality control applications where a threshold value is used, this may not be a concern, as the error can simply be built into the threshold. This is also true if only peak position information is required, as single beam correction only concerns the photometric scale. When a sample and a reference are of similar reflectance, the substitution correction is very small. If the sphere is made sufficiently large and the area of the sample is minimized, the error diminishes. However, the use of a large sphere is usually not practical in low cost spectrophotometer systems due to signal-to-noise concerns as well a price considerations. Consult the references for further explanation of single beam substitution error and methods of correction.[3,4,7,8]

Sample Beam Size

Due to their size, 150 mm integrating sphere accessories have proportionally larger sample beam spot sizes, typically 50% larger than those of small 60 mm spheres. A large spot size is an advantage for inhomogeneous samples, where large beam coverage ensures representative reflectance measurements over the entire surface of the test sample. However, the typical large beam size of the 150 mm integrating sphere accessory is not optimized for the measurement of samples smaller than one inch in diameter. For such small samples, the sample beam must be reduced so that it does not overfill the sample. Two methods can be used to reduce the size of the sample beam so it better matches small samples; either a lens can be used (in conjunction with a small spot kit) to focus the beam down, or the sample can be masked so that the correct portion of the beam strikes the sample. Both beam reduction methods result in sometimes considerable loss in beam energy, which can increase the noise in the scan and will require slower scan times to compensate for the energy loss.

Design Options

While all sizes of integrating sphere accessories are able to measure diverse samples such as powders, liquids in cuvettes, and translucent or opaque solids such as fabrics or syringes, the 150 mm diameter integrating sphere has extended sampling options that are unavailable on its smaller counterpart. Integrating spheres of 150 mm diameter or greater are able to accept center mount sample holders. These center mounts enable variable angle reflectance measurements of opaque samples, absorptance scans of thin films or translucent samples, or fixed angle liquid measurements to be performed. In addition, the standard 150 mm sphere accessories have removable reflectance port covers for the measurement of large or bulky samples. Other sampling or customization options are available only for 150 mm integrating spheres designs, such as downward viewing reflectance ports or large transmittance sample compartments for bulky samples, adapters for polarizers, or sample beam attenuation for small beam spot sizes.

Conclusion

While consideration is needed in determining the proper sphere size required to achieve optimal performance for your spectrophotometer, the process is made easier when one understands the advantages and limitations of the accessories. We hope this article has provided a useful tool to aid in your selection process.

Comparison of Sphere Accessories

150 mm Diameter Integrating Sphere

ADVANTAGES

Availability
- Available for high-end, double beam spectrophotometers.

Port Fraction
- Low port fraction—typically 2–4%. Meets CIE color measurement specifications.

Measurement Accuracy
- Highest measurement accuracy is achieved with large integrating spheres since sphere errors can be minimized, resulting in very homogeneous light flux and minimal hot spots in sphere.

Substitution Errors
- Double beam integrating spheres—no sample substitution errors.

Sample Beam Size
- Large sample beam size—good coverage of inhomogeneous samples.

Design Options/Description
- Accessory is more flexible—can accommodate various sphere designs such as center mount sample holder, small spot optics, or downward viewing reflectance port.
- External compartment accessory—can measure reflectance of large samples using the external ports with removable covers.
- More room to design custom sample holders for reflectance or transmittance samples—not limited by size of spectrophotometer's sample compartment.
- Uses sensitive PMT (200–850 nm) and PbS (850–2500 nm) detectors.

LIMITATIONS

Availability
- Not available for many moderately priced single or dual beam spectrophotometers.

Sphere efficiency
- Not as efficient as smaller spheres—large sphere diameter attenuates the sample beam energy more than a small sphere of similar design.

Noise Level
- Signal-to-noise may be lower for highly absorbing samples (may have to perform scans at larger slit widths, slower scan speeds, or with reference beam attenuation to compensate).

Sample Beam Size
- Large sample beam spot size overfills small test samples, requiring masking or small spot kits which lead to additional energy loss.

Design Options/Description
- Out-of-compartment accessory—may be subject to stray light effects.
- Often more difficult to use—may be harder to install, optical alignment may be more complicated.
- Usually cannot be equipped with silicon photodiode (200–1100 nm) detector.

Cost
- More expensive (more optics, typically two detectors and preamplifier boards).

60 mm Diameter Integrating Sphere

ADVANTAGES

Availability
- Available for moderately priced single, dual, and double beam spectrophotometers.

Sphere efficiency
- Smaller spheres are more efficient collectors—in general, more energy reaches detector.

Noise Level
- Higher throughput systems, therefore signal-to-noise is usually better, especially for highly absorbing samples (Note—also depends on linearity range of instrument).

Sample Beam Size
- Smaller sample beam spot size better matches small test samples, no need to mask or use small spot kits which lead to throughput loss.

Design Options/Description
- In compartment accessory—generally not subject to light leaks.
- Simple to use—easy to install and remove accessory (once electronics are in place, if applicable), optical alignment is more straightforward.
- Accessories with small spheres can be equipped with either PMT (200–850 nm) or silicon photodiode (200–1100 nm) detectors, depending on spectrophotometer.

Cost
- Less expensive (fewer optics, typically one detector only).

LIMITATIONS

Availability
- Often not available for high-end, double beam spectrophotometers.

Port Fraction
- High port fraction—typically above 10%. Sphere does not meet CIE color measurement specifications.

Measurement Accuracy
- Sphere errors or hot spots may occur in small spheres—errors may not be completely corrected by a sphere's baffles due to space constraints, which often prohibit placement of correctly sized baffles into a small sphere's interior.

Substitution Errors
- Small integrating spheres on single or dual beam spectrophotometers are subject to sample substitution errors, which can approach 10%R for diffuse samples.

Sample Beam Size
- Small sample beam size means multiple locations must be measured on inhomogeneous samples.

Design Options/Description
- Accessory design is less flexible—cannot accommodate special sphere designs such as center mount sample holder or downward viewing reflectance port.
- Cannot measure reflectance or transmittance of extremely large samples inside the spectrophotometer's sample compartment.
- Limited ability to design custom sample holders for reflectance or transmittance samples—limited by size of sample compartment.

References

1. Spectralon® A proprietary diffuse reflecting material ideal for applications ranging from the UV-VIS to the NIR wavelength region. For additional technical information about Spectralon, consult the Labsphere Diffuse Reflectance Coatings and Materials Catalog.

2. Carr, K.F., "A Guide to Integrating Sphere Theory and Applications," Labsphere Technical Guide, 1997.

3. Springsteen, A., "A Guide to Reflectance Spectroscopy," Labsphere Technical Guide, 1992.

4. CIE 15.2, Colorimetry, International Commission on Illumination, Second Edition (1986).

5. ASTM D 1003-95, "Standard Test Method for Haze and Luminous Transmittance of Transparent Plastics."

6. ASTM E 903-82 (Reapproved 1992), "Standard Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres."

7. Clarke, F.J.J. and Compton, J.A., "Correction Methods for Integrating-Sphere Measurement of Hemispherical Reflectance," Color Research and Application 11 (4), 253 (1986).

8. Springsteen, A., Ricker, T.M., Blanchard, S.S., and Dwyer, K.A., "Quantitation of Single Beam Substitution Correction in Reflectance Spectroscopy Accessories," Labsphere Application Note 01 (1996).

UNITED STATES
Labsphere, Inc.
Tel: (603) 927-4266
Fax: (603) 927-4694
Email: labsphere@labsphere.com

GERMANY
Labsphere-Germany
Tel (49) 0 7556 50107
Fax: (49) 0 7556 50108
Email:wolfboehme@T-Online.de

FRANCE
Labsphere-France
Tel: (33) 01 60 92 05 04
Fax (33) 01 60 92 04 95
Email: labsphere@wanadoo.fr

UNITED KINGDOM
Labsphere Ltd.
Tel: (44) 0 1625 871188
Fax: (44) 0 1625 871778
Email: labsphereuk@compuserve.com

0002-500