



Integrating Spheres: Their Application in Reflectance Spectroscopy



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REFLECTANCE SPECTROSCOPY, INTEGRATING SPHERE,
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REFLECTANCE, SCATTER, SPECULAR, LAMBERTIAN,
REFLECTOR, GLOSS, MATTE

Introduction

Reflectance spectroscopy is a versatile analytical technique which can reveal a material's chemical composition, as well as allowing colour to be quantified in a multitude of applications in biology, pharmaceuticals, art and cultural heritage, consumer products and many more.

Reflectance measurements can be made in a variety of ways, but the preferred approach is to use an integrating sphere in combination with a light source and a spectrometer. With the sample surface illuminated, the integrating sphere captures all of the reflected light over a complete hemisphere, which is then sampled by a spectrometer coupled to the sphere. The spectral reflectance recorded allows the object's colour to be computed, while the spectral signature will be characteristic of the chemical formulation which provides an insight into the makeup of the material.

This article will review the science of integrating spheres and diffuse reflectance, explain how they are used in reflectance spectroscopy, define the various sources of error and how they can be mitigated, and compare the use of integrating

spheres to other approaches to measuring reflectance.

The Integrating Sphere

The invention of the integrating sphere is attributed to German engineer F R Ulbricht in 1894, which followed the publication of a theoretical paper in 1892 by British scientist W E Sumpner.



Figure 1: An integrating sphere used for lamp measurements



Figure 2: Labsphere RTC Integrating Sphere for Measurements of Total Hemispherical Reflectance, Total Hemispherical Transmittance and Total Hemispherical Reflectance Versus Angle of Illumination

Integrating Sphere Coatings

The very earliest integrating spheres (or Ulbricht's Kugels in German) were coated with magnesium oxide, applied to the interior of the sphere by burning magnesium over an open flame. It was only after the development of Eastman Kodak's 6080 diffuse white paint (a barium sulphate formulation) that integrating spheres started to be routinely deployed in metrology equipment. The first commercial spectrophotometer incorporating an integrating sphere was developed in 1935 by the General Electric Company and was used in colorimetry, spurred no doubt by the concurrent activity in that field by the CIE, the Commission Internationale de L'Eclairage.

Traditional integrating sphere coatings for the UV-VIS-NIR spectral region (250-2500nm) tend to be fragile and easily damaged or optically degraded with exposure to high temperatures or ultraviolet radiation. To address these limitations, American company Labsphere developed a superior integrating sphere coating called Spectralon in 1986. Being a sintered form of PTFE (polytetrafluoroethylene), Spectralon is a solid, white thermoplastic that is machined into durable integrating spheres (and other reflectors). Compared to previous sphere coatings, Spectralon is mechanically, thermally and optically stable.

Spectralon, and the similar Zenith Polymer material developed by SphereOptics in Germany, achieve the ideal of near-Lambertian diffuse reflectance combined with a high level of reflectance. At >99% in the 400-1500nm band, and >95% from 250-2500nm, the diffuse reflectance is the highest of any material in the UV-VIS-NIR band.

Labsphere's alternative sphere coating is called Spectrafect, a barium sulphate-based paint that is applied by spraying. In the visible, Spectrafect achieves 98% reflectance. The 1% higher reflectance provided by Spectralon may not sound significant, but once you factor in the multiple reflections that occur within the integrating sphere, it is easy to understand how a Spectralon sphere can have twice the throughput compared to an otherwise identical barium sulphate painted sphere. This directly translates into superior sensitivity and enhanced signal-to-noise measurements, and for these reasons, Spectralon (or Zenith Polymer) is the sphere coating of choice in reflectance spectroscopy instrumentation.

Reflectance Spectroscopy

The universal integrating sphere doesn't exist, and to function correctly, a sphere must be adapted for the type of measurement required. An integrating sphere designed for laser power measurements, for example, would perform poorly if used to measure reflectance, and vice versa.

Directional Illumination with Diffuse Collection

For spectral reflectance measurements, there are a number of sphere configurations commonly used. The traditional approach, which is still employed in "high-end" scanning spectrophotometers, is to illuminate the sample with a collimated beam of tunable, monochromatic light, and to collect the reflected light hemispherically using the integrating sphere coupled to a broadband photodetector (Figure 3). As the wavelength of the monochromator is tuned, the reflected signal is recorded at each wavelength. This measurement geometry is described as



directional illumination with diffuse collection. The angle of illumination of the sample with respect to the surface normal is usually between $0-10^\circ$, the most common being 8° . For simplicity, a reflectance measurement performed with 8° illumination and total hemispherical collection using a sphere is given the notation " $8^\circ/H$ ".

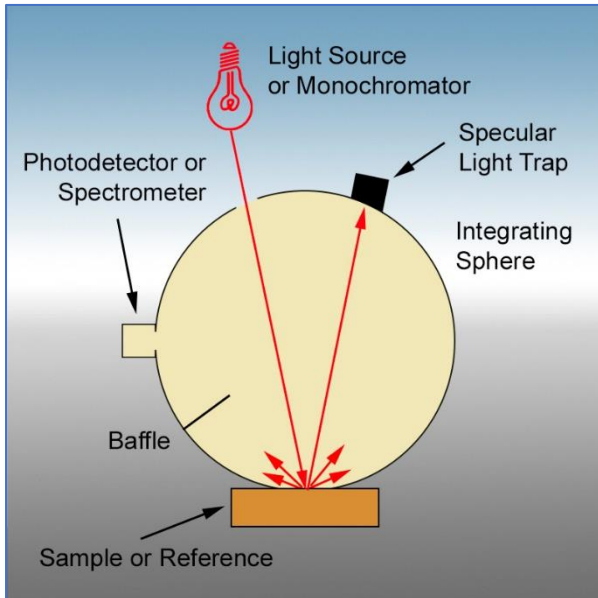


Figure 3: Integrating Sphere for Reflectance Measurements with Directional Illumination and Hemispheric Collection

With the exception of mirror-like surfaces, the reflectance from most materials is mainly diffuse but with a gloss (or specular) component. The reflectance integrating sphere can be equipped with a so-called "gloss trap". This is either a port opening, or highly absorbent chamber positioned on the sphere wall so as to exclude or absorb the specular component of the light reflected from the sample. With the specular port open, or fitted with a gloss trap, the light that remains within the integrating sphere is the diffuse-only component of reflectance. The notation " $8^\circ/D$ " is used for this measurement condition. It follows that if one were to measure the $8^\circ/H$ hemispherical reflectance and then subtract the $8^\circ/D$ diffuse reflectance, the magnitude of the specular reflectance at 8° could be computed. One refers to the measurement of

specular-included versus specular-excluded reflectance.

Diffuse Illumination with Directional Collection

The reciprocal geometry to that described above is where the sample is placed at a port on a sphere and illuminated hemispherically (diffusely) by a broadband light source placed at the wall of the sphere (Figure 4). A spectrometer is constrained to view the sample directionally from an angle between $0-10^\circ$ from the surface normal. Again a measurement geometry of 8° is often used. This geometry is described as being diffuse illumination with directional collection, and is given the notation " $H/8^\circ$ ". This geometry is commonly employed when using the latest generation of CCD array spectrometers, which are usually coupled to the integrating sphere using an optical fibre. Array spectrometers have the advantage of capturing all wavelengths at once, making for much faster measurements compared to a scanning monochromator that measures one wavelength at a time.

An $H/8^\circ$ diffuse illumination integrating sphere can also be equipped with a specular exclusion port (or light trap), positioned at the opposite 8° direction from which the sample is viewed. In that case, measurements are of the diffuse-only component of reflectance, and the notation used is " $D/8^\circ$ ". Subtracting the diffuse-only from the total hemispherical reflectance again yields the value of the specular reflectance.

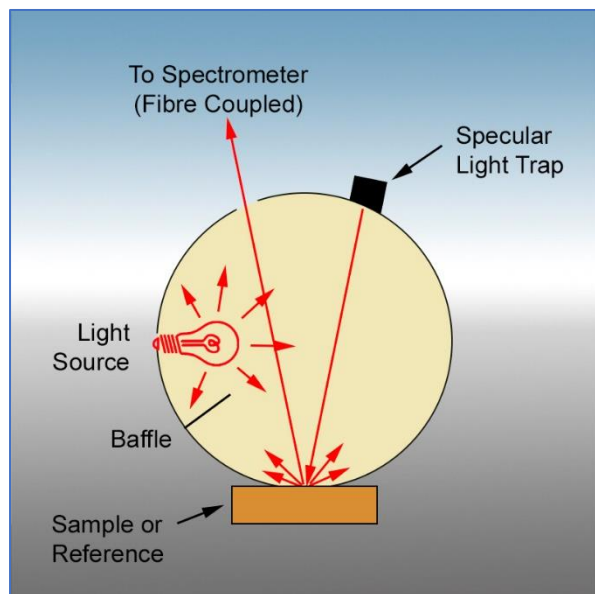


Figure 4: Integrating Sphere for Reflectance Measurements with Diffuse Illumination and Directional Collection

Measurements performed using directional illumination with hemispheric collection are perfectly equivalent to those made in the reverse configuration, using diffuse illumination with directional collection. This relies upon a fundamental law of physics known as the Helmholtz Reciprocity Theorem.

Calibration of the Integrating Sphere

Regardless of the measurement geometry chosen, your reflectance integrating sphere needs to be calibrated. This is performed using a certified standard of known spectral reflectance. Both Labsphere and SphereOptics offer reflectance standards fabricated from Spectralon and Zenith Polymer, respectively, for this purpose.

The transfer of the calibration from the reflectance standard to the sphere is performed by substitution. The reflectance standard is positioned at the sample port on the integrating sphere, then illuminated by the light source, and the detector (or spectrometer) signal recorded. This is commonly referred to as the "baseline" or 100% reflectance measurement. The reference tile is then substituted by (swapped for) the sample material whose reflectance you wish to determine.

The ratio of the two detector signals is equal to ratio of the reflectance of the sample compared to the reference tile. Thus, the unknown sample reflectance can be easily computed.

Single-Beam Substitution Error

The calibration method described above does suffer from an important source of error, referred to as the single-beam substitution error. This error arises due to the fact that the reflectance of the standard and the reflectance of the sample are (usually) not the same, and the mean sphere throughput changes between the reference and the sample measurement steps because of the multiple reflections and sample reillumination. The substitution error can be estimated by calculation, and is zero for the case of the sample reflectance equalling the reflectance of the reference tile. The substitution error is at a maximum when the sample has half the reflectance of the standard. It is possible to purchase sets of reference reflectance standards, which allows for the sample and reference levels to be more closely matched.

The more elegant solution is to design the integrating sphere with an additional, substitution correction port (Figure 5). This allows the sample and the reference tile to be mounted on the sphere at the same time, and one simply swaps their positions between the calibration and measurement steps. This maintains the mean sphere throughput between the reference and sample measurements, thus eliminating the substitution error.

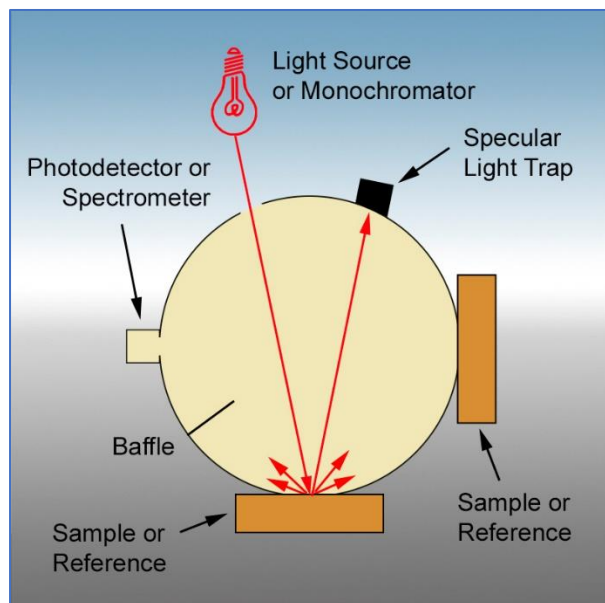


Figure 5: Reflectance Integrating Sphere with Single Beam Substitution Error Correction Port

An even more elegant solution is to employ a double-beam, ratio-recording spectrophotometer (Figure 6). This applies to the case of an integrating sphere deployed in a directional illumination with hemispherical collection geometry. Whereas the previous approach requires the operator to swap the position of the sample and reference tiles during the measurement, a double-beam sphere allows the sample and reference tiles to be left in place. The sample beam from the monochromator is split into two beams, known as the sample and reference beams. The sample beam is directed onto the sample, held at the sample port on the sphere. The reference beam is directed onto the reference tile, held at a separate reference port on the sphere. An optical chopper causes the sample and reference beams to alternately illuminate the sample and reference ports, while a lock-in technique is used to decode the detector signal.

Diffuse Transmittance Measurements

Integrating spheres can also be configured for measuring diffuse transmittance from scattering and translucent solids and liquids (Figure 7). In

many instances, a knowledge of total transmittance is required, this being the sum of the regular transmittance (equivalent to the specular component of reflectance) and the diffuse (or scattered) component of transmittance. A standard spectrophotometer configured without an integrating sphere will only be capable of measuring the regular transmittance; any light that is scattered by the sample will not be detected, and the transmittance under-recorded.

A certified standard of transmittance is not usually required. The 100% transmittance (baseline) level is established with the sample port open and no sample present.

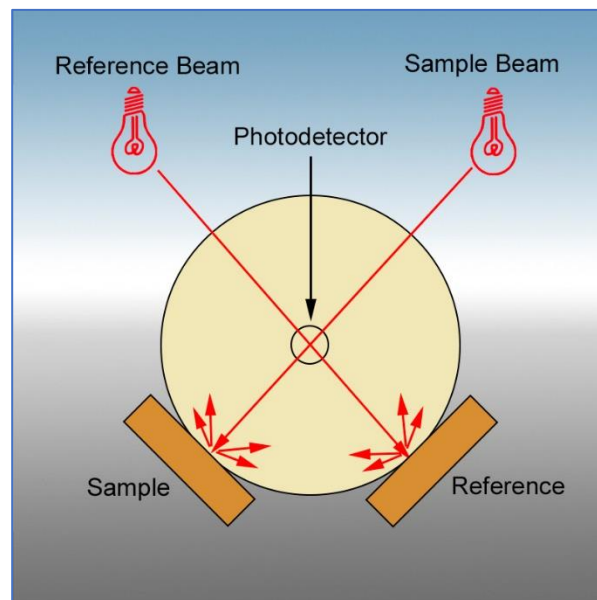


Figure 6: Reflectance Integrating Sphere with Double Beam Substitution Error Correction Geometry

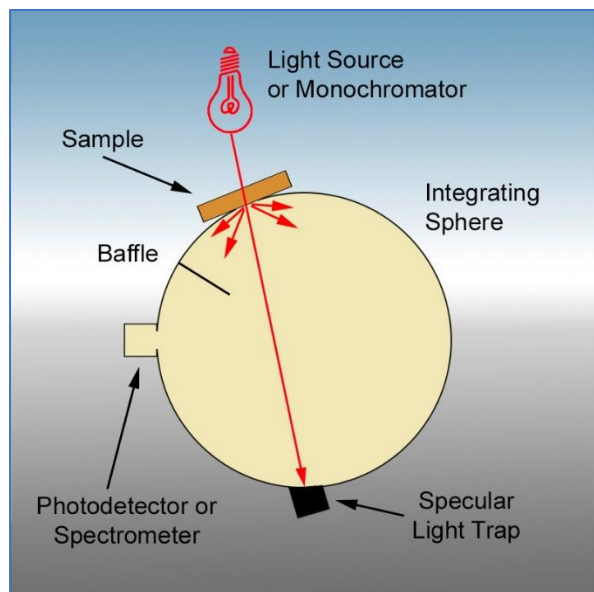


Figure 7: Integrating Sphere for Total Hemispherical Transmittance Measurements

Absolute Versus Relative Reflectance

In certain quality control applications, knowledge of the total reflectance of a material is not important. Instead, we may simply wish to track differences in reflectance compared to an in-house reference standard. Such measurements are considered qualitative, or relative, and while an integrating sphere can be used, it is not essential for comparative analysis.

Relative reflectance measurements can be performed using alternative measurement geometries. Compared to the measurement of absolute (total) hemispherical reflectance using an integrating sphere, comparative measurements employ both directional illumination with directional collection of the reflected light, and as such, yield instrument-specific values of reflectance. A common geometry is "0/45°", whereby the sample is illuminated at normal incidence, and the reflectance sampled at 45°.

While relative measurements are perfectly adequate if all that matters is to ensure long term consistency of colour in production, it should be noted that the measured reflectance values are unique to that instrument and to the particular measurement geometry employed. If instead you need to know the absolute or total reflectance, an integrating sphere with hemispherical illumination or hemispheric collection needs to be used.

Further Reading

Pro-Lite is the parent company of SphereOptics GmbH and also serves as the distributor for Labsphere Inc, and supplies integrating spheres, light metrology instrumentation, and calibrated diffuse reflectance standards and targets on behalf of both companies.

Pro-Lite also supplies instruments for measuring reflectance and BRDF/scatter from Avantes (spectrometer systems), Surface Optics Corp (reflectometers) and from The Scatter Works (scatterometers).

[Pro-Lite Integrating Spheres](#)

[Pro-Lite Reflectance Standards](#)

[Pro-Lite Spectrometers](#)

[Pro-Lite Scatterometers](#)

[Surface Optics Reflectometers](#)

In addition, Pro-Lite also provides independent measurement services for scatter (BRDF/BTDF), as well as spectral reflectance and transmittance (250-2500nm and 800nm-15µm), the latter being accredited to ISO 17025.

[Pro-Lite Measurement Services](#)