

INTRODUCTION

Over the past 43 years, Labsphere has been involved with ongoing research to improve the state of the art in diffuse reflectance coatings and materials. Our pioneering achievements in reflectance material research have produced a selection of coatings and materials that are accepted as industry standards. Our applications base is perhaps the largest in the industry, as we work closely with our customers to meet and exceed their optical and reflectance needs.

While many readers of this technical guide will have started their careers in optics dealing with traditional diffuse coatings and materials such as Opal Glass, Eastman 6080, or GE Integrating Sphere Paint™, this guide will serve as a refresher course in the materials available to spectroscopists, optical engineers and designers. For the newcomer, we hope this guide serves to aid them in selecting the proper reflectance coating and material for their application, by both outlining the physical, spectral and environmental properties, as well as presenting the limitations that are inherent in any reflectance coating or material.

This guide should be considered a "work-in-progress", as we continually work toward improving and developing innovative, new reflectance products.

We hope you will find the information presented useful, and as always, we appreciate your comments and suggestions.

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1.0 Diffuse Reflectance Coatings

For integrating spheres and many other applications that require either diffuse illumination or collection, reflectance and scattering properties are of utmost importance. An ideal coating is non-specular (to decrease geometrical effects), durable, high in reflectance and spectrally flat over a wide wavelength range to give a flat spectral response in input or output.

For some prototyping applications, a white house paint may be sufficient. Most commercial white paints, however, are not particularly white, nor particularly stable. They typically have an integrated reflectance in the 85 - 88% range over the visible region of the spectrum and drop off sharply in the blue end due to the use of titanium dioxide as a pigment. Even a flat (matte) white paint has a significant specular component that may cause problems. If low throughput is acceptable, a sandblasted aluminum surface may suffice; but with a mean reflectance of around 55% the throughput will be extremely low and may not be indicative of the performance of a component or system with a high-reflectance diffuse coating. For applications in the infrared, a sandblasted metallic surface may suffice, however the formation of oxide coatings due to atmospheric exposure may change the character of the material over time.

To design or prototype a component or integrating sphere system, the best possible coatings should be used. To that end, Labsphere has developed three standard coatings that can be applied to many substrates that give a flat spectral response over a wide wavelength range, are highly diffuse and highly reflective, and are with a single exception, quite durable for optical coatings.

These coatings, Spectraflect®, Permaflect®, and Infragold® are described in the following section.

1.1 Spectraflect Reflectance Coating

Spectraflect is a specially formulated barium sulfate coating which produces a nearly perfect diffuse reflectance surface. Spectraflect is generally used as a reflectance coating in the UV-VIS-NIR region and is most effective over the wavelength range from 300 to 2400 nm. The range can be stretched to 185 nm before binder absorption peaks begin to appear. The reflectance of Spectraflect, as with all reflectance coatings, is dependent on the thickness of the coating. At thicknesses above 0.4 mm (0.016 inches), the coating is opaque with reflectance of >98% over the wavelength range from 400 to 1100 nm. Spectraflect is thermally stable to approximately 160°C. Above that temperature, it slowly decreases in reflectance, especially in the 250 to 450 nm range. The coating outgasses slowly in high vacuum due to residual water entrapped in the binder. Spectraflect has been tested for laser damage threshold using a Q-switched YAG laser at 532 nm, the damage threshold is 1.7 J/cm². Spectraflect is an inexpensive, safe, non-toxic, high reflectance coating that is useful over a fairly wide wavelength range. The material is highly lambertian in character. Spectraflect is limited by the fact that the binder is water soluble, thus the coating is not usable in very high humidity applications. For applications where this consideration must be taken into account, Labsphere Permaflect coatings are recommended. Spectraflect is applied by spraying the coating onto a specially prepared surface. Surface preparation generally consists of degreasing. Spectraflect coating can be applied to virtually any substrate, and is an ideal reflectance coating for items such as optical components, integrating spheres, lamp housings and spectral diffuser panels.

Typical 8/H Reflectance Factors of Spectraflect

1.2 Permaflect Reflectance Coatings

Permaflect is a proprietary white reflectance coating for use where hostile environments, weathering and wear may affect a coating, yet high lambertian reflectance is required. Permaflect is generally used in applications in the visible to the very near IR, approximately 350 to 1200 nm. It is stable to approximately 100°C, with slight outgassing at high vacuum. Permaflect is water resistant and durable and can be used in high humidity conditions. The coating typically has a reflectance value of >95% over the wavelength range from 350 to 1200 nm. Permaflect is not recommended for use in the UV wavelength range. Permaflect is applied by spraying the coating onto a specially prepared surface. Surface preparation generally consists of degreasing followed by sandblasting to roughen the surface. For best results, Permaflect should be applied to metal or glass substrates. Pre-testing is recommended when applied to plastic substrates. Permaflect samples left outside in New Hampshire environment showed loss of <0.5% reflectance even after multiple washings to remove dirt. Permaflect has been used at low temperatures to coat integrating cylinders used to measure snow pack and is a common coating for reflectometers used in industrial on-line processes.

Typical 8/H Reflectance Factors of Permaflect

Permaflect-94 Polar Cosine Corrected BRDF with θ Lighting = 30°

Typical Reflectance Data of Permaflect Reflectance Coatings

1.3 Infragold NIR-MIR Reflectance Coating

Infragold NIR-MIR reflectance coating is an electrochemically plated, diffuse, gold-metallic coating which exhibits excellent reflectance properties over the wavelength range from 0.7 to 20 µm. Infragold has excellent vacuum stability, with no outgassing reported. Laser damage threshold is approximately 19.3 J/cm² @ 10.6 μ m using CO₂ laser. This is considered above average for a plated surface. The threshold will increase if the material is cooled on exposure to laser, as in water-cooled integrating spheres or targets.

The typical reflectance of Infragold is >94% above 1 μ m and data is traceable to the National Institute of Standards and Technology (NIST). Infragold can be applied to metal parts, generally aluminum, nickel or steel, although it has been applied with success to copper and tungsten. It is generally used for reflectance integrating spheres and accessories for NIR to MIR applications and is suitable for many space applications.

0.5 3.0 5.5 8.0 10.5 13.0 15.5 18.0 20.5 Wavelength (µm) 0.0 $0₁$ 0.2 0.3 0.4 0.5 $_{0.6}$ 0.7 $0.8\,$ $0.9\,$ $1.0\,$ 8/H Spectral Reflectance Factor al Reflectance Factor

Typical 8/H Reflectance Factors of Infragold

2.0 Diffuse Reflectance Materials

2.1 Spectrablack® Reflectance Material

Spectrablack is a low reflectance, light absorbing material that is resistant to abrasion. The material is continuously microporous resulting in unparalleled light absorption. Typical spectral reflectance is 1.6% or less from 250 - 2500 nm. Ideal for characterizing the performance of Lidar and Time of Flight (ToF) sensing systems at low reflectance (<5%)

Typical 8/H Reflectance Factors of Spectrablack

Spectrablack Polar Cosine Corrected BRDF with θ Lighting = 30°

2.2 Spectralon® Diffuse Reflectance Material

Spectralon diffuse reflectance material is a thermoplastic resin that can be machined into a wide variety of shapes for the construction of optical components. The material has a hardness roughly equal to that of highdensity polyethylene and is thermally stable to >350°C. It is chemically inert to all but the most powerful bases such as sodium amide and organo-sodium or lithium compounds. The material is extremely hydrophobic. Gross contamination of the material or marring of the optical surface can be remedied by sanding under a stream of running water. This surface refinishing both restores the original topography of the surface and returns the material to its original reflectance. Weathering tests on the material show no damage upon exposure to atmospheric UV flux. The material shows no sign of optical or physical degradation after long-term immersion testing in sea water.

Spectralon SRM-99 material gives the highest diffuse reflectance of any known material or coating over the UV-VIS-NIR region of the spectrum. The reflectance is generally >99% over a range from 400 to 1500 nm and >95% from 250 to 2500 nm. Surface or subsurface contamination may lower the reflectance at the extreme upper and lower ends of the spectral range. The material is also highly lambertian at wavelengths from 0.257 μ m to 10.6 µm, although the material exhibits much lower reflectance at 10.6 µm due to absorbance by the material.

The surface and immediate subsurface structure of Spectralon exhibits highly lambertian behavior. The porous network of thermoplastic produces multiple reflections in the first few tenths of a millimeter of Spectralon. Although it is extremely hydrophobic, this "open structure" readily absorbs non-polar solvents, greases and oils. Impurities are difficult to remove from Spectralon; thus, the material should be kept free from contaminants to maintain its reflectance properties.

The use of Spectralon should be limited to the UV-VIS-NIR. Spectralon exhibits absorbances at 2800 nm, then absorbs strongly (<20% reflectance) from 5.4 to 8 µm. Plated metal surfaces, such as the Labsphere Infragold-IR standards, are recommended as diffuse reflectance standards for the MIR.

Three grades of Spectralon are available: optical-grade, extreme physics and vacuum (EPV), and space-grade. Optical-grade Spectralon is characterized by its Lambertian behavior over the UV-VIS-NIR wavelength region. The optical-grade materials include our highly reflective white, greyscale, color, and wavelength reference materials, and fluorescent pigment-doped materials. EPV Spectralon offers a purified output product for ground-based applications. Space-grade Spectralon combines high reflectance with an extremely Lambertian reflectance profile and is the material of choice for terrestrial remote sensing applications. EPV and space-grade Spectralon are only available in 99% white.

2.3 Reflectance Properties of Spectralon

Spectralon exhibits relatively flat spectral distribution over most of the UV-VIS-NIR. From 250 to 2500 nm, Spectralon SRM-99 exhibits a reflectance variance of <5% and from 360 - 740 nm (VIS) the variance in reflectance is <0.5%. These spectral properties exceed those of most paints, which show strong absorbances in the UV due to absorbances by TiO2 or similar pigments. The hydrophobic nature of Spectralon also leads to exclusion of water overtone bands in the NIR which may occur in barium-sulfate-based materials. The open structure of Spectralon causes both reflectance and transmittance, but not absorbance of light.

Typical 8/H Reflectance Factors of Spectralon

99% Spectralon Polar Cosine Corrected BRDF with $θ$ Lighting = 30°

2.4 Spectralon Gray Scale Material

Spectralon can be doped with black pigment to produce spectrally flat gray scale standards and targets. Spectralon gray materials have physical and spectral properties similar to Spectralon and are useful as standards for calibration of various optical instruments, including those used in blood analysis, CCD arrays and night vision devices.

2.5 Typical Reflectance Data of Spectralon Reflectance Materials

2.6 Physical, Thermo-Optical and Electronic Properties of Spectralon

2.7 Reflectance Properties of Thin Sections of Spectralon

The reflectance of Spectralon decreases with decreasing thickness over most of the spectrum. The figures below illustrate the reflectance properties of thin sections of Spectralon.

Typical Reflectance Data of Thin Sections of Spectralon SRM-99

2.8 Environmental Testing of Spectralon Material

Spectralon was exposed to atomic oxygen from an ERC plasma stream, with a fluence of \approx 5.3 x 10²⁰ oxygen ions per square centimeter, with a vacuum in the range of 10-5 torr. Post-exposure measurements of the Spectralon showed no change in either the reflectance or the BRDF of the material.⁽¹⁾

Spectralon was bombarded with low energy protons at a current density of 10¹² protons cm⁻² at 40 KeV in a vacuum of ≤10⁻⁶ torr. As with the atomic oxygen exposure, no change was seen in either the reflectance or BRDF of the material from pre-exposure measurements.⁽¹⁾

Spectralon test samples were exposed to deep and mid-UV (unfiltered Hg arc lamp) at a vacuum of ≤10-6 torr with the equivalent of 2 suns for 500 equivalent sun hours. At 110 sun hours, a lowering of reflectance of between 5 - 10 % in the UV was noted; at 500 sun hours, a slight yellowing in the VIS was noted, along with a 20% total drop in the UV (250 nm). However, upon returning to atmospheric conditions, the material returned to near original values, presumably due to oxidation and loss of the surface contaminants that caused the discoloration. (1) Data from another source indicates that the loss of reflectance in the UV and subsequent yellowing does not occur if Spectralon is subjected to a vacuum bakeout procedure. Spectralon has undergone extensive testing for UV-VUV exposure, proton bombardment, atomic oxygen exposure an α -Lyman radiation. Please contact Labsphere for a list of published articles for results of this testing.

Spectralon plates were subjected to electron beam bombardment with a beam energy of 10 KeV at densities of 0.5, 1.0, and 5.0 nA cm-2. The Spectralon was uniformly charged to a potential of -6000 V. Investigation of the discharge phenomenon over extended periods showed no discharge at any current density or charging.⁽¹⁾

Spectralon has undergone two types of weathering and environmental tests. After measuring the initial reflectance of several samples, they were exposed to the outside environment of central New Hampshire for up to two years. At three month intervals, the samples were cleaned and gently sanded under a stream of tap water to restore the original surface finish. Measurements taken at 50 nm intervals throughout the visible wavelength region revealed essentially no change in reflectance. The results of those tests are shown below.

Environmental Exposure

In a second test, samples of the same material were immersed in sea water. After six months, no change in reflectance was noted. No surface preparation or cleaning was necessary as the samples were not wetted by sea water, neither initially or after six months immersion.

(1) MERIS Activities Report, Doc. No. PO.RP.LSP.ME.0008 10-28-93. This work was performed by Lockheed in conjunction with flight qualification of Spectralon Reflectance Material for use on the European Space Agency MERIS sensor, launched in 1997.

2.9 Spectralon Color Materials

Several colorimetric standards are available today, including Spectralon color materials, ceramic tiles, painted chips and Carrera glass. Spectralon color materials have the same physical properties as Spectralon. Therefore, they solve many of the problems associated with other standards. Available in an endless range of colors, Spectralon color materials offer the durability typically lacking in painted chips and ceramic tiles. Unlike ceramic tiles, Spectralon color materials are not subject to the restriction of a specific measurement geometry.

Slight translucency in supposedly opaque material can cause errors due to undetected light losses. Spectralon color standards exhibit significantly less translucency error than Carrera glass standards. Spectralon color standards are also less temperature sensitive than previously available standards and thus less subject to chromatic drift when warmed under intense illumination.

Reflectance Data of Spectralon Color Standards Red, Yellow, Green, Blue, Cyan, Purple, Violet, and Orange

2.10 Typical Reflectance Data of Spectralon Color Reflectance Standards

2.11 Spectralon Wavelength Standards

Spectralon wavelength calibration standards are formulated by impregnating a Spectralon substrate with the oxide of a rare earth element which displays sharp absorption spikes at specific wavelengths. Complete absorption spectral data is supplied with each standard. Durable, washable, and chemically inert without loss of surface texture, Spectralon wavelength calibration standards retain uniformity throughout making them ideal for calibration of spectrophotometers, reflectometers, and other spectral instruments. Calibration data for peak absorbance wavelengths relative to maximum absorbance across the UV-VIS-NIR is provided with each calibrated standard. Calibration is traceable to National Institute of Standards and Technology (NIST).

Spectralon wavelength calibration standards are available in either 1.25 or 2.00 inch diameters and are doped with one of the following: Holmium Oxide, Dysprosium Oxide, or Erbium Oxide. A multi-component wavelength calibration standard is also available and combines the three rare earth oxides.

Absorbance Spectrum - Multi-Component Wavelength Calibration Standard

Holmium Oxide Wavelength Calibration Standard

Dysprosium Oxide Wavelength Calibration Standard

Erbium Oxide Wavelength Calibration Standard

Multi-Component Wavelength Calibration Standard

Wavelength (nm) Spectralon Spectrablack Spectraflect Permaflect Infragold 250 0.91 0.01 0.94 0.64 - 0.96 0.01 0.96 0.85 - 0.98 0.01 0.97 0.91 0.35 0.98 0.01 0.98 0.93 0.32 0.99 0.01 0.98 0.94 0.36 0.99 0.01 0.98 0.94 0.39 0.99 0.01 0.98 0.94 0.57 0.99 0.01 0.98 0.94 0.75 0.99 0.01 0.98 0.93 0.84 0.99 0.01 0.97 0.93 0.92 0.99 0.01 0.97 0.93 0.93 0.99 0.01 0.97 0.93 0.93 0.99 0.01 0.97 0.93 0.93 0.99 0.01 0.97 0.92 0.94 0.99 0.01 0.97 0.92 0.94 0.99 0.01 0.97 0.92 0.95 0.99 0.01 0.96 0.92 0.95 0.99 0.01 0.96 0.92 0.95 0.99 0.01 0.96 0.91 0.95 0.99 0.01 0.95 0.90 0.95 0.99 0.01 0.95 0.91 0.95 0.98 0.01 0.95 0.91 0.95 0.98 0.01 0.94 0.90 0.95 0.98 0.01 0.93 0.88 0.95 0.98 0.01 0.91 0.88 0.95 0.98 0.01 0.92 0.89 0.95 0.98 0.01 0.92 0.90 0.95 0.98 0.01 0.92 0.89 0.95 0.98 0.01 0.92 0.88 0.95 0.98 0.01 0.92 0.82 0.95 0.98 0.01 0.91 0.83 0.95 0.98 0.01 0.91 0.85 0.95 0.98 0.01 0.91 0.86 0.95 0.97 0.01 0.86 0.82 0.95 0.97 0.01 0.83 0.80 0.95 0.97 0.01 0.85 0.83 0.95 0.95 0.01 0.86 0.84 0.96 0.95 0.01 0.86 0.83 0.96 0.94 0.01 0.87 0.81 0.96 0.96 0.01 0.87 0.81 0.96 0.96 0.01 0.86 0.63 0.96 0.95 0.01 0.84 0.60 0.96 0.94 0.01 0.83 0.65 0.96 0.94 0.01 0.82 0.64 0.96 0.94 0.01 0.80 0.65 0.96 0.93 0.01 0.77 0.69 0.96

Typical Reflectance Data of Labsphere Reflectance Coatings and Materials

Typical Reflectance Data of Labsphere Reflectance Coatings and Materials

General References

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ANNEX - Labsphere BRDF Testing - 94% Permaflect

1. Overview

Bi-Directional Reflectance Distribution Function (BRDF) is defined as the ratio of the radiance (L) of a sample to the irradiance (E) upon that sample, for a given direction of incidence and direction of scatter.

$$
BRDF(\theta_d, \phi_d, \theta_i, \phi_i) = \frac{L(\theta_d, \phi_d)}{E_0(\theta_i, \phi_i)} \quad [sr^{-1}] \qquad 1
$$

 θ_d = Detector Elevation Angle, "Theta Measure"

 Φ_d = Detector Azimuth Angle, "Phi Measure"

 θ_i = Incident Lighting Elevation Angle, "Theta Lighting"

 Φ_i = Incident Lighting Azimuth Angle, "Phi Lighting"

Characterizing the BRDF of a planar sample requires constant illumination of a spot and a detector that has the ability to collect the spatial information of the reflected light from a hemispherical array of points above the sample.

This report details the BRDF measurement procedure and results of a sample of 94% Permaflect. The angles of illumination used for the measurements are 0° (normal) 20° and 30°. More angles are available upon request.

2. System Details

Bidirectional Reflection Distribution Function (BRDF) is measured using Labsphere's Light-Tec REFLET-180 Goniophotometer, shown in Figures 1 and 2. The system is built on an optical bench that incorporates an illumination module, photodiode detector module, sample holder, and rotation mechanisms. The illumination module receives light from a 50-watt quartz tungsten-halogen (QTH) lamp via a fiber optic connected to the external Light Box. The incident light (θi) can be set to any angle of incidence from normal (90°) to grazing (0°) in 1 degree increments. The light box has a 6-filter wheel that accepts 1" diameter filters. The illumination module includes a diaphragm for adjusting the illuminance levels from 100 to 25,000 lux and an iris for setting the spot size from 1 to 13mm diameter. Polarizing filters can be mounted on both the illumination and detector modules. The polarization is easily adjustable to S or P orientation. For this report, the samples are illuminated with unfiltered broadband light from the QTH lamp.

The detector module is connected to the electronics box via an optical fiber to the 2 photodiodes in the electronics cabinet. The two detectors each have dual, auto-adjusting gain settings. This provides a wide dynamic range of 1:109. The software automatically switches between detectors and the gain settings to optimize the signal to noise level.

The Light-Tec REFLET-180 BRDF instrument measures the intensity of the light reflected from a stationary sample surface at any location in the hemisphere above the sample.

Figure 1. Reflet-180 BRDF System

Figure 2. Optical Bench Components

4. BRDF Uncertainty and Conservation of Energy

An analysis of measurement uncertainty associated with this instrument (including positioning uncertainty, instrument noise and lamp decay) has been done for custom BRDF measurements and is less than 2%. Conservation of energy when measuring a 99% Spectralon sample was experimentally proven to capture nearly all the irradiance of the beam: 180° sweeps were taken every 2 degrees in the Phi direction. The numerically integrated hemispherical data array (99% irradiance) was then compared to a measurement of the beam directly (radiance upon the sample) using the same detector. The resulting radiance and irradiance values matched within the expected 2%.

5. Results

The REFLET-180 Goniophotometer records intensity in 0.1° increments of each scan. These intensity values are relative and must be normalized to yield BRDF values.

For a hemispherical representation of the BRDF of a sample, the broadband incident light is set to a specified incident angle while the receiver is swept in θ from -90° to 90° in all four Phi azimuth angles. To normalize the data, a sample of Spectralon is placed on the sample stage and measured at the home position for Phi (-90°) and 30° elevation.

To normalize the data, the following equation is used:

$$
BRDF = \frac{Intensity_{DUT}}{\cos (\phi_{detector\;Elevation\;Angle})} \times \left[\frac{\cos (30^{\circ})}{Normal\;Lighting, \; Intensity\; at\; 30^{\circ} \pm 0.2^{\circ}} \times \frac{1}{\pi} \right]
$$

Normalization Factor, Measured with Spectralon ($R\% \approx 1$)

The three plots at each incident angle demonstrate three different methods of representing the same data:

- 1. "Straight BRDF" values. These plots do not include the natural cosine-losses at the lower viewing angles (the circular beam area gets geometrically smaller at lower angles), and thus looks less than ideal at angles past 45°. In order to correct for this, we use a cosine correction.
- 2. The cosine-corrected BRDF plots include this cosine response as the detector sweeps over the surface.
- 3. The polar plots takes the cosine correction to the next level which allows comparison to an easily identifiable shape: a circle.

The ideal Lambertian is represented by the black line in each of the plots. The "V-shaped" indentation in the plots are where the scanning detector eclipses the light source. This is particularly apparent in the 0° plots. A linear fit can be added to smooth these obscurations, though they are useful for confirming proper alignment between the detector and the light source.

A small (<3" diameter) sample is placed on the sample stage. The stage is height adjustable and has a fine adjustment of tip and tilt to ensure the measured surface is level when compared to the rotational axes of the instrument. The instrument is designed to collect constant-speed "sweeps" by rotating the detector module so that the detector field of view points at the sample from all elevation angles from -90° to 90° in 0.1 degree increments.

The illumination module angle can also be adjusted in integer degree steps below the sample (from 0° to -90°) for Bidirectional Transmittance Distribution Function measurements.

3. Standard BRDF Measurements

Labsphere has developed a Standard BRDF measurement that includes four equally-spaced "sweeps" of the sample to create a four-slice representation of the reflection distribution in the hemisphere above the sample. The geometry and coordinate definitions for Labsphere's Standard BRDF measurements is shown in Figure 3:

Figure 3. BRDF Measurement Geometry Figure 3. BRDF Measurement Geometry

This graphical representation of the standard BRDF measurement scans shows the path that the detector module follows, as well as the relative orientation of the illumination module. The detector "sweeps" along the edges of the teal, blue, red and green arcs (see 3D representation in top left) while maintaining a concentric field of view on the illuminated portion of the sample for all locations. The illuminated spot size is set so that it always underfills the area observed by the detector. The detector scans begin at θd = -90° and end at θd = 90°. **The BRDF sweep plots at the end of this report match the color scheme of the sweeps in Figure 3 to aid in** See the arrows on each of the arcs for the direction of the semi-circular scans at each azimuthal angle of phi.

MEASURE any light source

CREATE any spectrum

REFLECT any wavelength