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PELA 6000 "VN" Series Accessories for the Perkin Elmer Lambda 900 Spectrophotometer

## Absolute Specular Reflectance Measurements at Fixed Angles

With the widespread use of anti-reflective optical coatings, multilayer dielectric coatings, solar controlling thin films, and laser mirrors, specular reflectance measurements have become even more important for research and QC applications. Specular reflectance measurements are used to characterize the film thickness, refractive index, and other optical properties of these materials. Labsphere has designed a series of specular reflectance accessories that allows specular reflectance measurements to be performed at five key beam incidence angles: 8°, 15°, 30°, 45°, and 60°. Called the PELA-6000 "VN" Series Accessories, this series of reflectance accessories is designed exclusively for the Perkin Elmer Lambda 900 Spectrophotometer.

The term "VN" describes the beam path shapes in the reference (V) and sample (N) configurations. (See Figure 1) The only difference between the reference (baseline) and sample scans is due to the introduction of the sample into the beam path. As long as the optical path length, mirror surfaces, and reflection angles do not change from baseline to sample scan, the measurement is absolute. Each VN reflectance accessory measures absolute specular reflectance at a well-defined, repeatable angle by a single strike method. The VN accessory design provides tremendous advantages over other available specular reflectance methods.

Traditional methods<sup>1</sup> for the analysis of specular samples at variable angles have several limitations which Labsphere's new VN reflectance accessory was designed to overcome. For instance, standard specular reflectance devices with angles ranging from near-normal at 7.5° to grazing angle at 80° are available for the measurement of relative specular reflectance. With these accessories, the user compares the sample's reflectance to that of a calibrated specular reflectance standard, such as a first surface aluminum mirror. While highly accurate measurements can be performed with these relative specular accessories, the measurement accuracy is dependent upon the calibration standard used. Any surface contamination, mirror damage, or degradation of the reflectance standard will introduce erroneous results. In addition, it is often not possible to find a suitable specular standard



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which not only has been calibrated at the same angle and beam polarization as the intended sample measurement but also has general reflectance characteristics similar to those of the sample. Measurements with the PELA 6000 VN Series accessories do not suffer from such difficulties in finding suitable calibrated reflectance standards, as none are required in this absolute measurement technique.

The VN design offers several advantages over other absolute specular reflectance devices as well. Unlike the expensive variable angle accessories, the PELA 6000 series offers measurement capability at key beam angles, which are available alone or in combination with other angles. The VN accessories are simpler to use and are better suited for a QC environment than the PELA 1030 variable angle absolute specular reflectance accessory, which has a complicated belt mechanism which drives its sphere. While the PELA 1030's collection sphere has a coating which limits NIR measurements to 2500 nm, the PELA 6000 VN measurements can extend over almost the entire wavelength range of the PE Lambda 900, out to 3300 nm with the correct lens in place on the accessory.

The PELA 6000 VN design is also advantageous over the traditional VW absolute specular reflectance accessory, which is typically available only for a near-normal angle such as 7.5 degrees. The VW optical design requires two bounces on a sample surface during the measurement. For this reason, samples often must be larger than the industry standard one inch diameter in order for both contact points in the "W" optical path to strike the sample. The use of small spot optics to reduce the beam size to fit onto a smaller sample leads to energy loss in this double bounce system, which can be a problem during some sample measurements. In addition, because two separate areas of the sample are involved in the VW measurement, errors may result if the sample is inhomogeneous. Measurement accuracy is also limited when the VW optical design is used during the measurement of very low or very high reflectance samples, such as anti-reflective coatings or laser mirrors. With two bounces on an anti-reflective coating sample, for example, the resulting spectrum is actually the square of the sample's reflectance. For very low reflectance coatings, this value is often beyond the dynamic range of the instrument upon which the measurement is performed. The VN specular accessory design overcomes such measurement problems.

## VN Accessory Design

The PELA-6000 VN Series accessories are designed to measure the reflectance of highly specular, non scattering, first-surface reflectors. The VN accessory utilizes the Perkin Elmer Lambda 900's photomultiplier tube and lead sulfide detector for measurements in the 190–2600 nm wavelength range. The measurements are performed in an absolute fashion, without the need for a calibrated reflectance standard, and with only one reflection off the sample surface. The VN accessory design is easy to use and is well-suited for the QC lab environment and the measurement of one inch diameter test samples.

The PELA 6000 VN Series of reflectance accessories consists of a main adapter baseplate, the PELA 6001, and five separate fixed measurement angle "Daughter Plates," the PELA 6008, 6015, 6030, 6045, and 6060. These interchangeable plates allow absolute specular reflectance scans to be performed at the key measurement angles of 8°, 15°, 30°, 45°, and 60°, respectively. PELA 6000 Series baseplates are precision machined using wire EDM technology for repeatable mirror and sample positioning. Each VN accessory fits into the standard Perkin Elmer Lambda 900 sample compartment and uses the instrument's own detectors and electronics. The baseplate mounts kinematically onto the main PELA 6001 adapter baseplate for easy installation and removal of the accessory. The optical alignment of the VN unit can be set and locked on each daughter plate so that changing between measurement angles does not require a complete optical realignment. Easily removable mirrors can be changed to convert between the V and N optical configurations.

A clip-style sample holder is provided permanently mounted to each VN accessory daughter plate. Samples which are from 1–3 inches in diameter and up to 3/8 inches thick can be measured with the standard sample holder. Sample sizes are limited by the amount of physical clearance between the edges of the sample and other accessory mirrors, especially at high beam angles. Custom sample holders may be designed if space permits. Samples must be flat, specular, first surface reflectors only.

Because light polarization can strongly influence specular reflectance measurements, especially above a 30° incidence angle, it is recommended that a common beam depolarizer be used on the Lambda 900 in conjunction with the VN reflectance accessory. To perform sample studies at known polarization, the 30°, 45°, and 60° VN accessories are fully compatible with Perkin-Elmer's motorized polarizer cassette assembly. Due to space constraints, the 8° and 15° daughter plates are not compatible with Perkin Elmer's polarizer cassette assembly. However, a manual polarizer mount is available as a custom component for the 15° VN accessory.

## **Optical Design**

The VN optical design uses the difference between the direct path to the detector and the matching path with a single reflection off the sample to determine a specular sample's reflectance. The path lengths of the baseline and reflectance scans are identical. Therefore, the measurement is absolute. This VN design is depicted below.

In the reference (V) configuration, mirrors M1, M2 and M3 are used. M1 is fixed. M2 and M3 are movable to allow for changing configurations. In the 8°, 15°, and 30° daughter plates, a lens assembly is also used to match the sample beam size to the Lambda 900 detector optics.

FIGURE 1 Reference "V" Configuration



In the sample (N) configuration, the same mirrors M1, M2 and M3 are used, but now M2 and M3 have been moved to create the "N" optical path. The specular sample is also in the sample beam path, completing it, and M3 is rotated so its reflective surface faces the sample. Because the sample "N" beam path must be the same length as the reference "V" configuration, M2 is placed closer to M1 in the sample measurement configuration.

FIGURE 2 Sample "N" Configuration

Reference Beam



Since the path length, mirror surfaces, and reflection angles remain the same during both the reference (baseline) and sample scans (except for the introduction of the sample), the measurement is absolute.

## **Additional Reading**

- 1. Springsteen, Arthur W., "Non-Standard Spectrometric Measurements—What to do when it's not a clear solution or filter." *Spectroscopy* **12** (5), 24 (1997).
- Springsteen, Arthur W., "Techniques for the Measurement of Specular Reflectance," Spectroscopy 12 (9), 24 (1997).
- Zwinkels, Joanne C., Noël, M., and Dodd, C.X., "Procedures and Standards for Accurate Spectrophotometric Measurements of Specular Reflectance," *Applied Optics* 33 (34), 7933 (1994).

