Long-term calibration monitoring of Spectralon diffusers BRDF in the air-ultraviolet

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Received 23 July 2007; accepted 17 September 2007; posted 3 October 2007 (Doc. ID 85538); published 8 November 2007

Long-term calibration monitoring of the bidirectional reflectance distribution function (BRDF) of Spectralon diffusers in the air-ultraviolet is presented. Four Spectralon diffusers were monitored in this study. Three of the diffusers, designated as H1, H2, and H3, were used in the prelaunch radiance calibration of the Solar Backscatter Ultraviolet/2 (SBUV/2) satellite instruments on National Oceanic and Atmospheric Administration (NOAA) 14 and 16. A fourth diffuser, designated as the 400 diffuser, was used in the prelaunch calibration of the Ozone Mapping and Profiler Suite (OMPS) instrument scheduled for initial flight in 2009 on the National Polar Orbiting Environmental Satellite System Preparatory Project. The BRDF data of this study were obtained between 1994 and 2005 using the scatterometer located in the National Aeronautics and Space Administration Goddard Space Flight Center Diffuser Calibration Laboratory. The diffusers were measured at 13 wavelengths between 230 and 425 nm at the incident and scatter angles used in the prelaunch calibrations of SBUV/2 and OMPS. Spectral features in the BRDF of Spectralon are also discussed. The comparison shows how the air-ultraviolet BRDF of these Spectralon samples changed over time under clean room deployment conditions. © 2007 Optical Society of America

OCIS codes: 290.1483, 120.0120, 120.3940, 120.5700.

1. Introduction

Spectralon diffusers, illuminated by standard irradiance lamps, are widely used as radiance sources in the prelaunch laboratory-based calibration of ultraviolet atmospheric remote sensing satellite instruments. Given a typical 5 year on-orbit lifetime of remote sensing instruments the production of a climate data record for global total ozone requires highly accurate and precise long-term measurements of the ratio of atmospherically backscattered ultraviolet radiance to incident ultraviolet solar irradiance from successive generations of temporally overlapping instruments on different platforms. For these instruments prelaunch calibration must be consistently transferred to on-orbit operation and must be monitored over each instrument’s mission lifetime. The prelaunch calibration of these instruments is greatly facilitated through the use of diffusers with stable reflectances and Lambertian scattering characteristics. In this paper we examine the long-term stability of the bidirectional reflectance distribution function (BRDF) in the air-ultraviolet of diffusers used in the prelaunch calibration of the Solar Backscatter UV/2 (SBUV/2) and Ozone Mapping Profiler Suite (OMPS) instruments. The change in Spectralon BRDF is an issue of interest to all remote sensing instruments [1] operating across the solar reflectance wavelength region.

Since 1993, the Diffuser Calibration Laboratory (DCL) located at National Aeronautic Space Administration’s (NASA’s) Goddard Space Flight Center (GSFC) has measured the ultraviolet BRDF of diffuse standards used in the prelaunch radiance calibration of a number of ultraviolet atmospheric remote sensing satellite instruments. Among these are the Total Ozone Mapping Spectrometers (TOMS) [2] on Earth probes, Advanced Earth Observing Satellite (ADEOS) and QuikTOMS, the SBUV/2 instruments on National Oceanic and Atmospheric (NOAA) 14 and 16, the Shuttle-borne SBUV (SSBUV), the OMPS on

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the National Preparatory Project (NPP) and National Polar Orbiting Environmental Satellite System (NPOESS), and the ozone monitoring instrument (OMI) on the Earth Observing System (EOS) Aura. The DCL also supports the multiwavelength scanning Cloud Absorption Radiometer (CAR) [3] instrument airborne BRDF measurements of Earth’s major biomes through laboratory measurements of natural samples. The DCL has participated in round-robin measurements with a number of domestic and international calibration institutions in the United States and abroad in the support of numerous spaceflight and nonspaceflight programs [4].

2. Methodology

Spectrafol is a well known white diffuse material with excellent reflection properties. It is the trade name of a Labsphere produced polytetrafluoroethylene (PTFE)-based material. Spectrafol is formed as a thermoplastic resin by heat treatment and pressure, and can be machined into a variety of shapes. It is thermally stable to above 350 °C, chemically inert, and can be formed into a variety of shapes. It is thermally stable to above 350 °C, chemically inert, and extremely hydrophobic. Spectralon gives the highest diffuse reflectance of any known material or coating over the UV to near infrared region of the electromagnetic spectrum. The reflectance is generally above 99% over a range from 400 to 1500 nm and above 95% from 250 to 2500 nm. It is used for reflectance diffuse standards, and its features are excellent for forming the interiors of integrating spheres.

The suitability of Spectralon as an in-flight calibration diffuser has been discussed by numerous authors with the majority of applications targeted at the visible to shortwave infrared wavelength region. Bruegge et al. [5] performed a series of environmental exposure tests to ensure the suitability of Spectralon for in-flight calibration diffuse reflectance panels. No degradation of the optical properties was apparent following proton bombardment, and visible/near infrared reflectance stability under UV illumination was satisfactory, provided that simple cleaning and handling procedures were implemented. A buildup of several thousand volts of static charge developed while simulating a pass through an auroral storm. Further testing of the charged Spectralon failed to produce arcing to the metallic housing frame, and models indicate that charge neutralization will occur after passage through the storm.

Chommeloux et al. [6] presented details on the influence of cleaning procedures on the optical stability of Spectralon diffusers under on-orbit solar illumination conditions in support of the European Space Agency’s (ESA) Medium Spectral Resolution Imaging Spectrometer (MERIS) instrument. Petroy et al. [7] discussed the flight qualification testing of Spectralon, including tests of the stability of the material under exposure to atomic oxygen and UV radiation matching the low-earth orbit environment. No degradation of optical properties was observed following atomic oxygen or proton treatment. Some degradation however was observed under UV exposure tests. This degradation has been linked to organic contaminants. Such contamination can be prevented by adopting stricter production and handling protocols. Spectralon aging was addressed by Stiegman et al. [8] through a detailed chemical analysis of diffuse standards. Spectralon was found to maintain its reflectance properties even after extensive solar UV exposure if a vacuum bake-out procedure was used to remove the impurities in the commercial material.

Moeller et al. [9] discussed the aging effects of Spectralon under low-level irradiation and in the dark. It was found that aging is mainly affected by four parameters: wavelength of radiation, irradiance level, radiant exposure (i.e., dose), and storage time of the samples.

In this paper we extend the work of previous studies by addressing the long-term BRDF properties of Spectralon in the air-ultraviolet. Since 1994 the DCL has repeatedly measured the BRDF of three Spectralon diffuse standards, namely, H1, H2, and H3, at 13 wavelengths in the spectral range from 230 up to 425 nm and at those incident and scatter angles used in the prelaunch calibration of the SBUV/2 instruments. An additional diffuser, called the 400 diffuser, used in the prelaunch calibration of the OMPS instrument was also studied at 12 wavelengths in the spectral range from 252 up to 425 nm. These diffusers were deployed only in clean rooms at Ball Aerospace in the calibration of the SBUV/2 and OMPS instruments and at NASA’s GSFC for the measurement of their BRDF. The diffusers were transported between the institutions in clean metal cases equipped with O-ring seals. The analysis of the long-term data for spectrally dependent changes in BRDF is presented in this paper. The results of this study are relevant to other remote sensing instrument programs that employ Spectralon diffuse standards in their prelaunch radiance calibrations, and to metrology studies in which radiance scales are realized using irradiance lamp standards and Spectralon diffuse standards.

The BRDF, according to the National Institute of Standards and Technology (NIST), is defined according to Nicodemus et al. [10] in radiometric terms as the ratio of the surface radiance Ls scattered by a surface into the direction \((\theta_s, \phi_s)\) to the incident surface irradiance \(E_i\) incident on a unit area of the surface at a particular wavelength:

\[
BRDF = \frac{dL_s(\theta_s, \phi_s, \theta_i, \phi_i; E_i)}{dE_i(\theta_i, \phi_i)},
\]

where the subscripts \(i\) and \(s\) denote incident and scattered, respectively, \(\theta\) is the zenith, and \(\phi\) is the azimuth angle. BRDF has units of inverse steradians and can range from very small to very large values. The large BRDF values usually correspond to specular or near specular reflection, while the small values are usually measured for low reflectance diffuse scatter.

Nicodemus et al. [10] further assumed that the beam has a uniform cross section, the illuminated area on the sample is isotropic, and all scatter comes from the sample surface. In practice, we are dealing
with real samples surfaces, which are not isotropic and the optical beams are not perfectly uniform. Hence from practical considerations the BRDF can be defined as the scattered power per unit solid angle normalized by the incident power and the cosine of the detector zenith angle, as presented by Stover [11]. This definition allows for bulk scatter in addition to surface scatter and permits nonuniform incident beam profiles as depicted in Fig. 1:

\[
\text{BRDF} = \frac{P_s}{\Omega \cos \theta_s},
\]

where \(P_s\) is the scatter power, \(\Omega\) is the solid angle of acceptance of the detector, \(P_i\) is the incident power, and \(\theta_s\) is the detector zenith angle. For a circular detector aperture, \(\Omega\) is described by

\[
\Omega = \pi r^2/R^2,
\]

where \(r\) is the radius of the detector aperture and \(R\) is the distance of the detector aperture from the sample.

Equation (2) can be used for the BRDF measurement at all scatter geometries. The \(\cos \theta_s\) factor is a correction to account for the illuminated area when viewed from the scatter direction. The BRDF is often called cosine-corrected, or scatter-function, when the \(\cos \theta_s\) factor is not included. We are following the above convention through the current study; however, care is required as some publications do not follow it.

The Spectralon diffuse standards were measured in the DCL at GSFC using the laboratory's scatterometer at the source and detector angular configurations employed in the prelaunch radiance calibration of the SBUV/2 and OMPS instruments. The scatterometer shown in Fig. 2 is located in a class 10 000 laminar flow clean room. It is capable of measuring the BRDF or bidirectional transmission distribution function (BTDF) of a wide range of sample types including white diffusers, gray-scale diffusers, black painted or anodized diffusers, polished or roughened metal surfaces, clean or contaminated mirrors, transmissive diffusers, liquids, and granular solids. The scatterometer can perform in-plane and out-of-plane BRDF and BTDF measurements in the operational spectral range from 230 to 900 nm. The measurement uncertainty, \(\Delta_{\text{BRDF}}\), was evaluated in accordance with NIST guidelines [12] by Schiff et al. [13] to be less than 1% \((k = 1)\). It is regularly calibrated using facility standard diffuse standards measured for BRDF on NIST's Special Trifunction Automated Reference Reflectometer (STARR) [14].

3. Experimental

The SBUV/2 Spectralon diffusers are all the same shape and thickness, and are designated as H1, H2, and H3 (Fig. 3). The 400 diffuser is a 12 in. square Spectralon diffuser. The diffusers were mounted in a
holder on the scatterometer sample stage, and the optical surface of each diffuser was aligned with the scatterometer’s axes of rotation. The detector field-of-view was centered on the illuminated area of each diffuser for all the measurements and was under-filled by the incident beam. The light source for these measurements was a 75 W short-arc xenon lamp coupled to a Chromex 0.25 m monochromator. The spectral bandwidth of the light exiting the monochromator was 12 nm. The light source assembly was mounted on a vertical optical table that can be rotated around its horizontal axis to change the incident light zenith angle onto the horizontally mounted sample diffuser. The detector positions on the virtual hemispherical surface around the sample are defined by the scatter zenith and azimuth angles. The BRDF was calculated by dividing the net reflected signal by the product of the net incident and the projected solid angle from the sample surface to the detector. All the measurements were made for polarizations of the incident light, both parallel and perpendicular to the plane of incidence, and unpolarized scatter is reported in this paper.

In support of the flight of SBUV/2 instruments on NOAA 14 and 16, Spectralon diffusers H1, H2, and H3 were measured on multiple occasions between 1994 and 2005 permitting an investigation of their age-related BRDF change in the UV. The incident and scatter angles were determined from the incident solar and scatter angles for the NOAA 14 and 16 SBUV/2 instruments. The BRDF was measured at the following 13 wavelengths: 230, 252, 273.5, 283, 292.2, 301.9, 312.5, 331.2, 339.8, 350, 375, 400, and 425 nm.

Diffuser H1 was measured at an incident angle of 63.2° and at scatter angles from 16° to 40° in 3° steps in 1994, 1995, 1999, and 2000. The results of those measurements are shown in Fig. 4 for the 28° scatter angle. The 28° scatter zenith angle was chosen to be representative for all samples and years. Included in the figure is a 1997 measurement of the H1 diffuser by NIST using their STARR instrument. The same diffuser was also measured at an incident angle of 51.8° and scatter angles from 16° to 40° in 3° steps by NIST in 1997 and by the DCL in 1999 and 2000. The data are not shown here as they show the same tendency.

Diffuser H2 was measured in 1997 by NIST and in 1999 by the DCL at an incident angle of 51.8° and at scatter angles from 16° to 40°, in 3° steps. Diffuser H2 was also measured by the DCL in 2001, 2003, and 2005 at an incident angle of 54° and scatter angles from 22° to 52° in 3° steps, as shown in Fig. 5. Diffuser H3 was measured in 2003 and 2005 at incident angle of 54° over a range of scatter angles from 22° to 52° in 3° steps. These data are also shown in Fig. 5. H3 was new when first measured in 2003.

In March, April, and October 2004 a new Spectralon diffuse standard was measured by the DCL. This diffuser was used in the calibration of the OMPS instrument scheduled to fly NPOESS and NPOESS NPP platforms. This diffuser was designated as number 400 and was measured at normal incidence from 22° to 52° in 3° steps in scatter zenith angles. The OMPS 400 diffuser was new when initially measured in March 2004 and its BRDF showed interesting spectral features (Fig. 6).
4. Results and Discussion

The BRDF of H1, H2, H3, and 400 diffusers has been analyzed by computing the difference of each measurement set from the average BRDF. For example, the average for diffuser H1 is

\[
\text{BRDF}_{H1}^{\text{Average}} = \left( \frac{\text{BRDF}_{H1}^{1994} + \text{BRDF}_{H1}^{1995} + \text{BRDF}_{H1}^{1997} + \text{BRDF}_{H1}^{1999} + \text{BRDF}_{H1}^{2000}}{5} \right),
\] (4)

and the percent difference, for example, of the 1994 measurement from the average would be

\[
\text{BRDF}_{H1}^{\text{Diff}}^{1994} = \left( \frac{\text{BRDF}_{H1}^{1994} - \text{BRDF}_{H1}^{\text{Average}}}{\text{BRDF}_{H1}^{1994}} \right) \times 100.
\] (5)

Diffuser H1 had the longest history of BRDF measurements in the DCL. The percent difference between the BRDF measurements acquired between 1994 and 2000 and the average BRDF for the same period for diffuser H1 is shown in Fig. 7. The 1997 NIST measurement is also included in this figure. The BRDF measurements made in 1995 are clearly lower than those made in other years between the wavelengths of 273 and 400 nm. The disagreement of the 1995 measurements with the average BRDF at these wavelengths is larger than the scatterometer measurement uncertainty of 1%. This could be due to scatterometer-related errors in the 1995 measurement of the incident power onto the diffuser or to a potential mispositioning of an order sorting filter following the monochromator exit slit during the 1995 scatter power measurements. We quote an uncertainty of 1% for BRDF, and we suspect it to be smaller (0.7%) for specific wavelengths. The larger disagreement at 230 nm was primarily due to lower incident light intensity at this wavelength, hence poorer scatterometer signal to noise ratio. The difference at 230 nm between the BRDF data when measured in the DCL is up to 2.2%. The NIST results from 1997 are also included, showing an approximate 3.8% difference between the average and the NIST measurements indicating similar problems. In an effort to examine whether the source of the 1995 measurement difference at 230 nm was due to a geometric measurement problem in the DCL scatterometer, the
reciprocity of the DCL BRDF measurements at 230 nm was quickly checked by comparing its measurements at 63.2° incidence and 28° scatter versus those at 28° incidence and 63.2° scatter. The difference between these measurements was within the scatterometer measurement uncertainty of 1%, effectively eliminating this as a potential source of this difference. Recognizing the 1995 measurements as outliers and taking those data out, the 1994, 1999, and 2000 data (Fig. 8) show agreement to within ±0.5% for wavelengths of 252 nm and well above the measurement uncertainty. The NIST measurements are also excluded from Fig. 8 to eliminate the setup to setup differences. The BRDF difference at 230 nm (Fig. 8) is still higher than the measurement uncertainty between 1% and 1.4%.

The BRDF percent differences between diffuser H1 acquired at 51.8° incident angle in 1999 and 2000 by the DCL and the average BRDF are less than ±0.5% for the spectral range of 230–425 nm (Fig. 9). This is well within the DCL measurement uncertainty.

The BRDF percent differences for diffuser H2 acquired at a 54° incident angle in 2001, 2003, and 2005 by the DCL and the average BRDF are shown in Fig. 10. The data show agreement to within ±0.7% over the complete wavelength range with the exception of the 2003 measurements at 273 and 282 nm where the difference is ±0.9%. A BRDF reciprocity check was done for 252 nm by comparing the BRDF data at 54° incidence and 28° scatter versus those at 28° incidence and 54° scatter. The difference between these measurements was within the scatterometer measurement uncertainty of 1%.

BRDF measurements were acquired in the DCL of two new Spectralon diffusers, designated as H3 and 400, in order to further investigate the Spectralon aging. The H3 diffuser is a SBUV/2 diffuser with the same shape and thickness as H1 and H2. The H3 diffuser was cleaned by Ball Aerospace in a vacuum chamber before the measurements. Diffuser 400 is a 12 in. diffuser used in the prelaunch calibration of the NPOESS OMPS instrument. The BRDF of H3 is included in Fig. 5 for 2001, 2003, and 2005 measurements. The BRDF of H2 measured in 2003 was compared to the BRDF of H3 measured in the same year at 28° scatter zenith angle and at a 54° incident angle. The BRDF of H2 in 2001 was also compared to the BRDF of H3 in 2003; the idea was to see the difference in the BRDF of a new and a 12 year old Spectralon that had been kept in a clean room environment. Figure 11 shows the experimental data and the fifth degree polynomial fitting. The BRDF difference in the visible is 0.9% or better; however, it can be as high as 17%–18% at 230 nm. The advantage of this comparison is to determine what decrease in the BRDF should be expected after 12 years in Spectralon that has been kept in a clean room environment. The statement above assumes that H2 had a similar BRDF to H3 at one time. We do not know that for sure as we did not measure H2 in its infancy. However we want to caution users of Spectralon that for new samples the BRDF needs to be closely monitored in order to track rapid reflectance changes in the UV.

The short-term BRDF difference from the average of diffuser 400 from 2004 is presented in Fig. 12. The BRDF measured in April is seen to decrease between 273 and 301 nm, while the BRDF at 252 nm remains largely unchanged. The BRDF measured in October increases between 273 and 301 nm, while the BRDF at 252 nm is about the same. The observed relatively rapid change in BRDF indicates that it is most probably due to an absorbing hydrocarbon molecular species that leaves the sample over time. The diffuser 400 was not vacuum baked before measurements, which support the thesis of absorbing molecular species. These conclusions are in accordance with the subsequent contamination analysis on Spectralon by Petrovy et al. [7], and Stiegman et al. [8]. Their results identify the presence of a hydrocarbon impurity in the commercial material, which could be removed by a vacuum bake-out procedure. The Spectralon changes its optical properties when exposed to UV radiation if the contaminant was not removed.
5. Conclusions
The ability to construct an ozone climate data record from measurements of the UV albedo of the Earth’s atmosphere depends on the accurate and consistent prelaunch calibration of successive generations of satellite instruments. With the exception of a spurious measurement in 1995, the percent change in BRDF of diffuser H1 measured in 1994, 1999, and 2000 was less than ±0.7% from 252 to 425 nm. The percent difference in the measured BRDF of the second diffuser, designated as H2, was also within ±0.7% over the same spectral range between 1997 and 2003. DCL measurements of both diffusers at 230 nm showed spreads of ±1.25% and ±0.6% for H1 and H2, respectively. NIST measurements of H1 and H2 in 1997 showed very good agreement with the DCL measurements with the exception of the 230 nm data. At 230 nm the H1 and H2 BRDF measured in the DCL show larger differences primarily due to reduced signal to noise ratio. The overall degradation of BRDF seems to be related to the age of the diffusers relative to when they were manufactured. In order to study this effect further, BRDF data were acquired on two additional new diffusers, H3 and 400. The same change in BRDF when compared to the average BRDF was seen in both diffusers. Diffuser 400 was measured in March, April, and October 2004. Its BRDF was seen as decreasing between 252 and 300 nm with the 252 nm data unchanging.

In general, our results indicate a rapid decrease in the BRDF of new Spectralon at 230 nm. The BRDF decrease is slower from 252 to 300 nm. There is also some decrease from 300 to 400 nm. The BRDF is practically stable at 400 nm and 425 nm, although wavelengths above 425 nm were not part of this study. It was found that the aging effect of Spectralon laboratory samples used for calibration purposes and kept in a clean room environment in tightly closed containers depends mainly on the storage time of the samples. This study indicates a very small overall degradation in the BRDF when compared year after year for both UV and visible spectral ranges. However a significant difference is observed when the BRDF of a new Spectralon diffuser, H3, is compared to the BRDF of the old one, H2 (Fig. 11). This difference is most pronounced in the UV at 230 nm and gradually decreases toward the visible. We can state from the data of our long-term stability study of the H1 and H2 that these diffusers have reached a stable reflectance, and the method of storing and transporting these diffusers has contributed to their stability. For the newer diffusers, we have not seen a decrease in the BRDF. Yet, it could happen. Hence the Spectralon would not be recommended for space applications in the UV, below 400 nm, especially with the possible contamination problems in mind. These have important implications for the use of Spectralon diffusers in the prelaunch calibration of UV satellite instruments.

The authors thank Kevin Kelly and Steve Bennett of Ball Aerospace and Jim Mentall of NASA’s Goddard Space Flight Center for making the samples available to us for measurements, and Bob Barnes of Science Applications International Corporation for the critical reading and recommendations. This work was supported by the National Aeronautics and Space Administration under agreement NAS5-02041 issued through the Science Mission Directorate.

Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Aeronautics and Space Administration nor does it imply that the materials or equipment identified are the best available for the purpose.

References


