Reflectance properties of pressed Algoflon F6: a replacement reflectance-standard material for Halon

Paul R. Spyak and Carole Lansard

The standard ultraviolet through short-wave infrared (200–2500-nm) diffuse-reflectance material, Halon PTFE, type G-80, is no longer available. Therefore an equivalent diffuse-reflectance standard material must be found. Algoflon F6 is shown here to be an appropriate replacement through the presentation of measurements of various spectral-reflectance properties of Halon and Algoflon F6. The measurements include spectral hemispherical reflectance, spectral bidirectional reflectance factor (BRF), sample BRF repeatability, and sample lifetime. © 1997 Optical Society of America

Key words: Hemispherical reflectance, bidirectional reflectance factor, Halon, Lambertian, polytetrafluoroethylene, reflectance standard.

1. Introduction

Pressed polytetrafluoroethylene (PTFE) powder has been used as a standard of diffuse reflectance for the ultraviolet through the short-wave infrared (200–2500 nm) for more than a decade[1–5] after being introduced as a reflectance standard in 1976.[6] Other materials that have been used as reflectance standards include smoked magnesium oxide, pressed magnesium oxide powder, pressed barium sulfate powder, and various glasses, tiles, and plastics.[7,8] Because of its outstanding reflectance properties, the PTFE powder material commonly used throughout the optics industry has been Halon PTFE, type G-80, hereafter referred to as Halon. When pressed according to the appropriate prescription, its 6° incident/hemispherical reflectance factor (for angles between 5° and 75°) is better than 0.960 for wavelengths ranging from 200 to 2500 nm and is 0.993–0.994 for 400–1250 nm.[1] In addition, its bidirectional reflectance factor (BRF) approximates that of a Lambertian surface.[1]

Halon was originally manufactured by Allied Chemical Company. In 1986 Ausimont USA, Inc.[9] purchased Allied Chemical Company and eventually discontinued production of Halon. As supplies of Halon dwindle, the optics industry must identify an equivalent alternative standard material. Some laboratories and industries have turned to a similar product manufactured by Ausimont under the trade name of Algoflon F6, hereafter referred to as Algoflon. This study investigates the potential application of Algoflon as a diffuse-reflectance standard by comparing its hemispherical reflectance, angular-reflectance, wavelength dependence, and sample repeatability with Halon's characteristics.

2. Physical Properties of Halon and Algoflon

Halon and Algoflon are manufactured with 100% PTFE. However, the manufacturing processes are slightly different. Ausimont[9] manufactures various grades of Algoflon as identified by numbers and letters following the trade name. Of the various grades, the two that seem to be most closely related to Halon are the Algoflon F5 and F6. Although these two grades are basically the same, Algoflon F6 is the better choice because it is a cleaner material and will therefore be more repeatable from batch to batch.[10] Also, contamination of the PTFE can lead to fluorescence.[1]

Two important physical properties to compare are particle size and average bulk density. For Halon these are 20–35 μm and 350 g/L, respectively. For Algoflon F6 the particle sizes range from 15 to 25 μm and the bulk density ranges from 350 to 420 g/L.

With the physical properties being so similar, it seems likely that Algoflon F6 would be comparable with Halon as a diffuse-reflectance standard.

P. R. Spyak is with the Remote Sensing Group, Optical Sciences Center, University of Arizona, Tucson, Arizona 85721. C. Lansard is with the École Nationale Supérieure de Physique de Strasbourg, Boulevard Sebastien Brant, F-67400 Illkirch, France.

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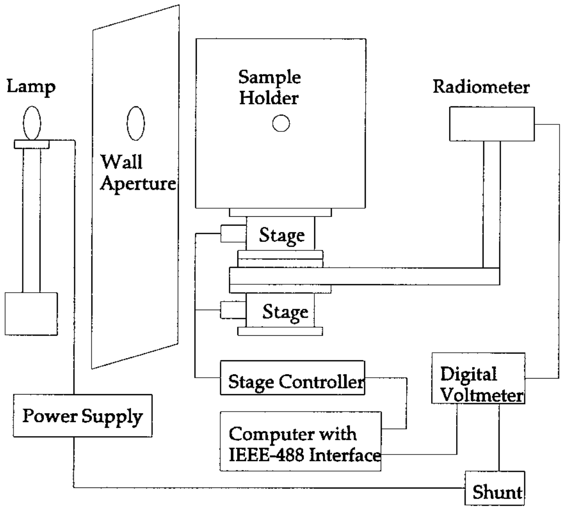


Fig. 1. Schematic of BRF instrumentation.

3. Reflectance Measurement Instrumentation and Methodology

A. Bidirectional Reflectance Factor Instrumentation

The BRF was measured with the facility shown schematically in Fig. 1. The instrumentation comprises seven components: detector arm, sample holder, lamp fixtures, stages with motion controller, data acquisition system, radiometer, and source.

The detector arm has an interchangeable mount to support various radiometers. The radiometers are situated on this arm with their entrance pupils 50 cm away from the center of the sample and their optical axes coinciding with the optical axis of the BRF instrument, that is, normal to the sample and along the axis of the incident illumination beam.

The sample holder situates the sample surface coincident with the vertical axis of stage rotation and can accommodate samples as large as 61 cm on a side.

The lamp that provides illumination is mounted in a fixture that allows three orthogonal translations, two orthogonal tilts, and a direct rotation of the source. A wall separates the lamp room from the sample room. A 5-cm-diameter hole in a plate mounted on the wall allows the light to enter the sample room and restricts the illumination into the sample room.

All hardware and the walls are either black anodized or painted flat black to reduce stray reflections.

Angular positioning of the radiometer arm and the sample holder is achieved through the use of two rotary stages that are stacked such that the sample holder and the detector arm may move to any angular position with respect to one another for any illumination angle. The stages feature 0.1 arc min resolution and are controlled by a microprocessor-based controller. We accomplished angular alignment by mounting a flat mirror in the sample holder and laser aligning the system. By aligning the reflected laser beam upon the laser aperture we could achieve an angular alignment of better than ±0.1°.

The radiometer output is measured with a high-accuracy digital voltmeter (Hewlett Packard, Model HP3457A) with remote programming capabilities through an external computer. An MS-DOS personal computer configured with an IEEE-488 interface is used for data acquisition and motion control, with software written in C.

The radiometer used in this experiment was manufactured locally. It uses a silicon detector, has a nominal 1° full field of view, and has nine interchangeable narrow-bandpass filters: 450, 500, 550, 650, 700, 750, 860, 948, and 1040 nm.

The source is a 1 kW quartz halogen DXW lamp. The lamp is powered by two Hewlett Packard 6274B dc power supplies, and the current is monitored by measuring the voltage across a precision 0.01-Ω shunt resistor from Leeds and Northrup. To maintain a constant source output, we maintained the nominal 8-A current to within 1 mA, which results in a radiometer output voltage change of 0.079% at 450 nm, 0.052% at 700 nm, and 0.038% at 1040 nm.

B. Derivation of the Bidirectional Reflectance Factor

Following the derivation for normally incident light,[11] we can calculate the hemispherical reflectance factor $R(0°/h)$ of a diffuser from the reflectance factor $R(0°/\theta)$ (the first term in parentheses describes the angle of illumination and the second term describes the angle of detected radiance, and h implies hemispherical and θ implies angular):

$$R(0°/h) = \frac{2\pi \int_0^{\pi/2} R(0°/\theta)\cos\theta \sin\theta \, d\theta}{2\pi \int_0^{\pi/2} \cos\theta \sin\theta \, d\theta}. \quad (1)$$

Dividing Eq. (1) by $R(0°/45°)$ and simplifying, we may rewrite it as

$$\frac{R(0°/h)}{R(0°/45°)} = 2 \int_0^{\pi/2} \frac{R(0°/\theta)}{R(0°/45°)} \sin\theta \cos\theta \, d\theta. \quad (2)$$

Rewriting Eq. (2) in terms of measurable quantities, one obtains

$$\frac{R(0°/h)}{R(0°/45°)} = 2 \int_0^{\pi/2} B(0°/\theta)\sin\theta \cos\theta \, d\theta, \quad (3)$$

where

$$B(0°/\theta) = \frac{\Phi(0°/\theta)/\cos\theta}{\Phi(0°/45°)/\cos 45°}, \quad (4)$$

and Φ is the reflected flux from the sample.

$B(0°/\theta)$ can then be approximated by an nth order polynomial in θ:

$$B(0°/\theta) = \sum_{i=0}^{n} b_i \theta^i. \quad (5)$$

Substituting Eq. (5) into Eq. (3), one finds

$$\frac{R(0°/h)}{R(0°/45°)} = 2 \sum_{i=0}^{n} b_i I_i, \qquad (6)$$

where

$$I_i = \int_0^{\pi/2} \theta^i \sin\theta \cos\theta \, d\theta. \qquad (7)$$

For this study, the radiometer output voltages, which are proportional to the reflected flux, are fitted to a fifth order polynomial in Eq. (5), and Eq. (6) is solved for $R(0°/45°)$. Knowing the voltage and the reflectance for one data point is sufficient to calculate the reflectances $R(0°/\theta)$ for all θ. This study also assumes that the Helmholtz reciprocity principle holds such that $R(0°/\theta) = R(\theta/0°)$.

C. Bidirectional Reflectance Factor Measurement Procedure

Prior to and following each sample measurement, the radiometer entrance aperture is covered to measure the dark current (the input offset voltage of the amplifier). The sample is then measured at a given wavelength for incident angles ranging between 10° and 85°, at 5° intervals. For these measurements the detector is always normal to the sample surface. For each position 100 readings are taken and averaged; and a standard deviation is calculated. The dark value then is subtracted from the average, the voltages are converted to $B(0°/\theta)$ with Eq. (4), and $B(0°/\theta)$ is fitted with a weighted least-squares method. Typically, an individual datum does not deviate from the fitted curve by more than 0.001. The value used for $R(0°/h)$ is obtained from the National Bureau of Standards (now the National Institute of Standards and Technology) published value[1,2] and $R(0°/45°)$ is calculated with Eq. (6). From this, $R(0°/\theta)$ is calculated.

D. Bidirectional Reflectance Factor Instrument Repeatability

The BRF instrument repeatability was determined as follows. For a given sample and wavelengths of 450, 700, and 1040 nm, five sets of BRF measurements were made on each of two consecutive days. Between days the entire system was shut off and the entire measurement procedure repeated. The mean standard deviation in BRF for all 10 sets of measurements was 0.001498, 0.002764, and 0.002715, respectively, for the three wavelengths. For these calculations, the 85° values were not used because of their large standard deviations. The standard deviation in the mean reflectance for 85° was 0.01491, 0.022365, and 0.023732, respectively. These are larger because samples are not Lambertian, leading to a decrease in the signal-to-noise ratio; and there is some shadowing of the incoming light prior to illuminating the panel. This variation can be seen throughout the data. Although the standard deviation increases significantly for incident angles greater than about 70°, the measurements are quite repeatable from 10° to 80°.

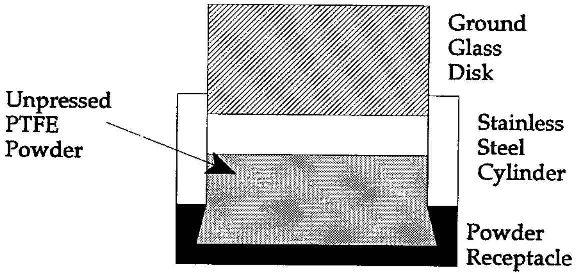


Fig. 2. PTFE press used to make standard diffuse reflectance samples.

4. Sample Preparation

The samples were prepared following the prescriptions outlined in publications by the National Bureau of Standards[1,2] and by the American Society for Testing and Materials (ASTM)[12]:

(1) Clean and dry the hardware thoroughly.

(2) Coat the sample holder with a thin layer of high-vacuum silicone grease to improve the PTFE adherence.

(3) Handle the PTFE with only stainless steel or glass tools to minimize contamination.

(4) Pulverize the PTFE to a fine, uniform powder with a glass blender with stainless steel blades.

(5) Spoon approximately 25–30 g of PTFE from the blender to a glass dish located on a calibrated weight scale.

(6) Accurately weigh the PTFE and transfer it to the aluminum–stainless steel sample holder/pressing fixture, Fig. 2. The sample holder has a depth of 10 mm.

(7) Repeat steps 3. through 6. until the amount of PTFE transferred to the sample holder equals 1 g/cm^3 of sample volume.

(8) Press the PTFE until it is flush with the sample holder surface, using a glass disk that has been finely ground with a 40-μm grit. The density should be 1 g/cm^3 as the reflectance of the material is a strong function of density.[1] This is achieved quite easily with a fixture as shown in Fig. 2.

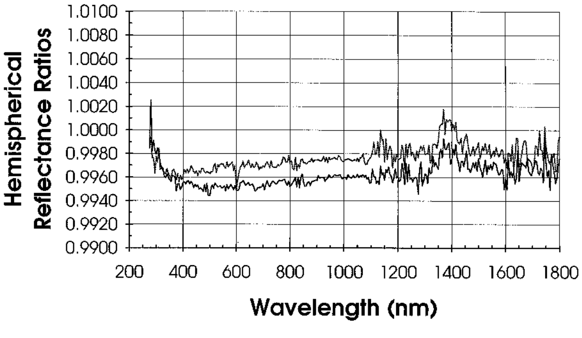


Fig. 3. Hemispherical reflectance ratios of Algoflon to Halon.

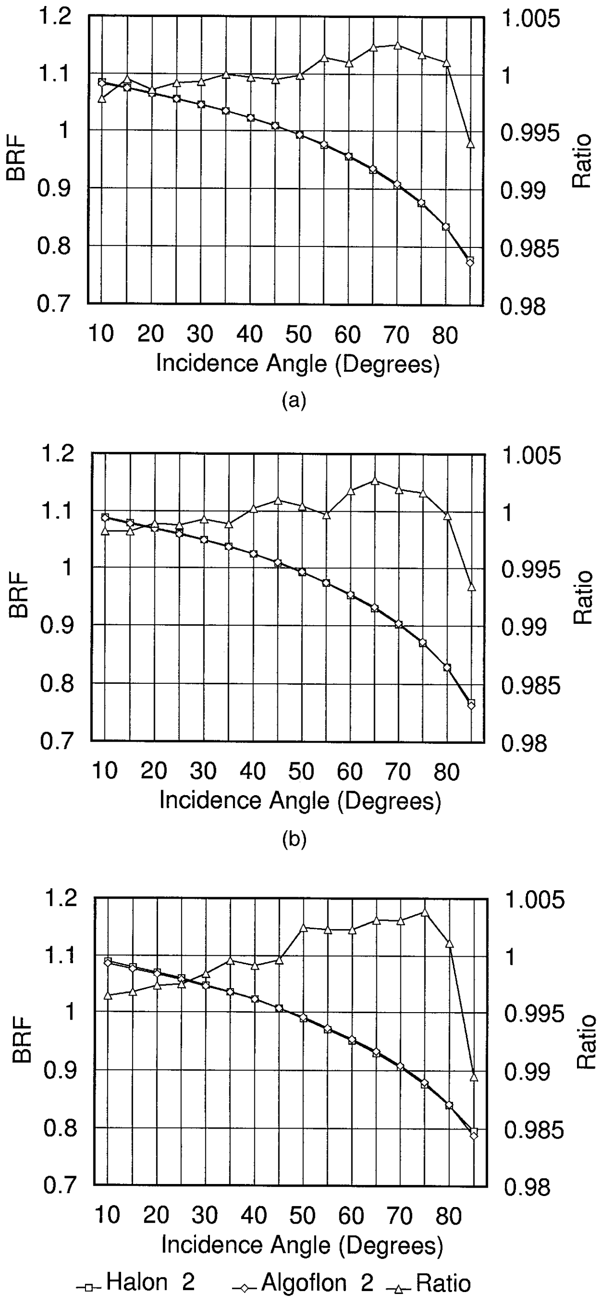


Fig. 4. BRF comparison of a good Halon sample and a good Algoflon sample with a varying incidence angle and the detector fixed normal to the sample. Wavelengths are (a) 450 nm, (b) 700 nm, (c) 1040 nm.

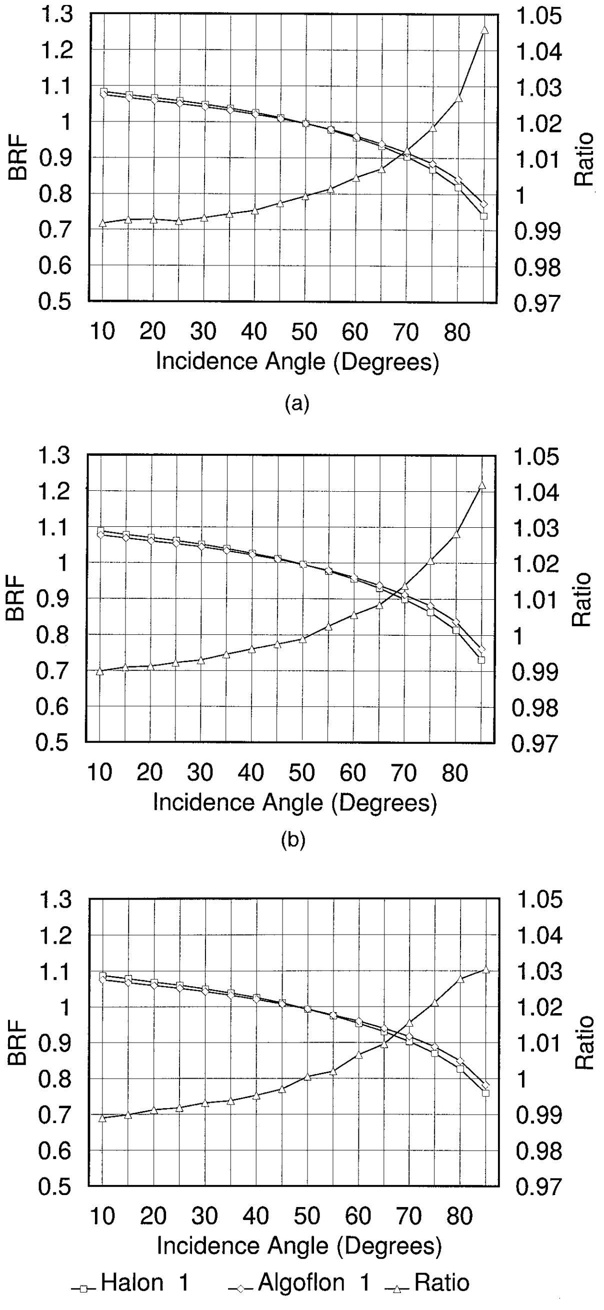


Fig. 5. BRF comparison of an acceptable Halon sample and an unacceptable Algoflon sample with a varying incidence angle and the detector fixed normal to the sample. Wavelengths are (a) 450 nm, (b) 700 nm, (c) 1040 nm.

(9) Inspect the sample in a darkened room by illuminating it with a bright light at near grazing incidence. A good sample is one with a smooth, uniform surface. Often bumps appear as a result of PTFE clumping or nonuniform distribution of PTFE before packing; such samples are not used.

(10) Cover sample with a stainless steel cover that provides a Viton O-ring seal to the sample holder. This is essential to maintain sample cleanliness.

5. Results

A. Hemispherical Reflectance of Algoflon F6

The 10°/hemispherical reflectance of several Halon and Algoflon samples was measured on a spectroradiometer system manufactured by Optronic Laboratories, Inc., over the wavelength range of 275–1800 nm. The ratio of the Algoflon-to-Halon hemispherical reflectance is shown in Fig. 3 for two sets of measurements. The data illustrate that the two

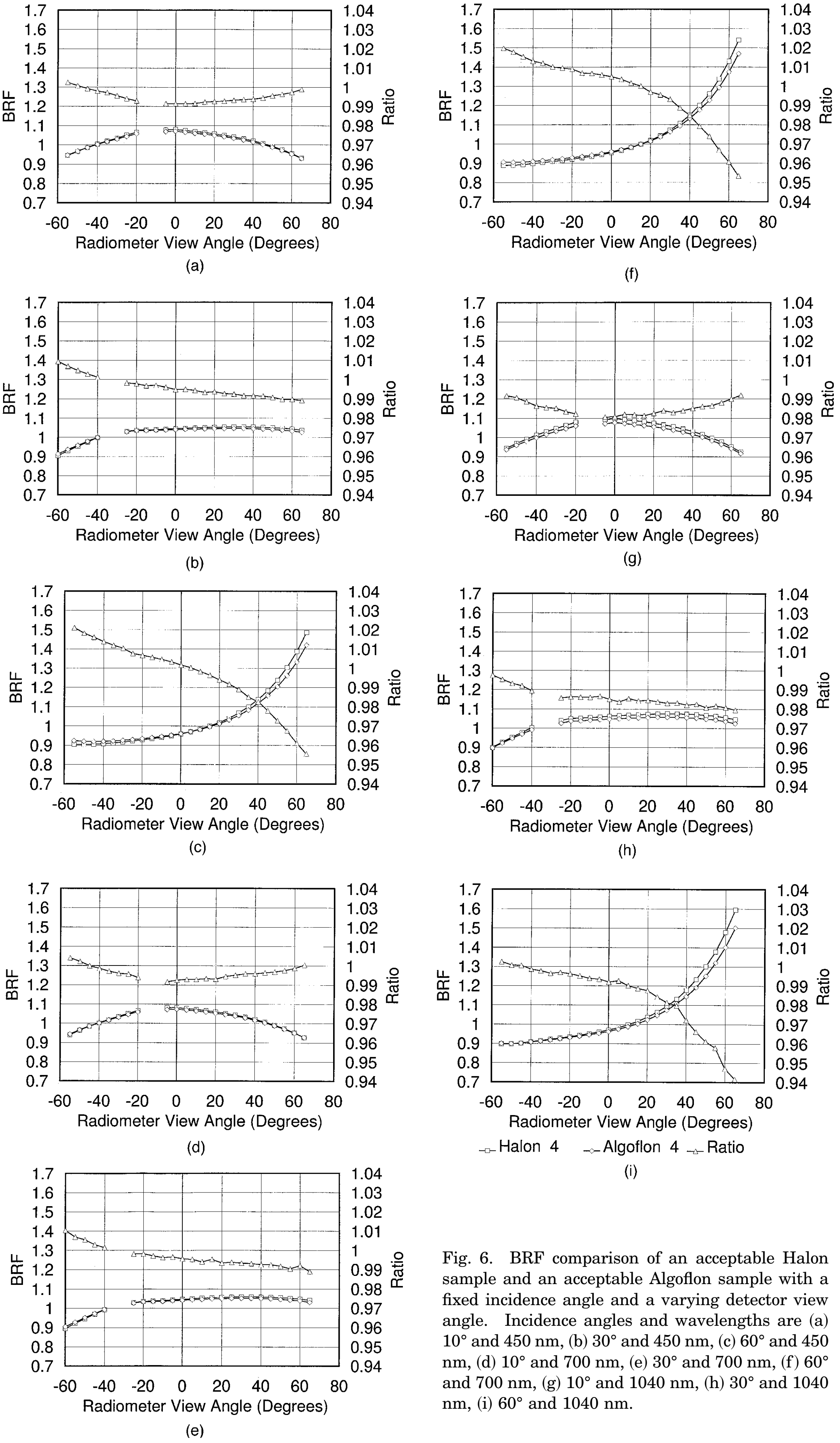


Fig. 6. BRF comparison of an acceptable Halon sample and an acceptable Algoflon sample with a fixed incidence angle and a varying detector view angle. Incidence angles and wavelengths are (a) 10° and 450 nm, (b) 30° and 450 nm, (c) 60° and 450 nm, (d) 10° and 700 nm, (e) 30° and 700 nm, (f) 60° and 700 nm, (g) 10° and 1040 nm, (h) 30° and 1040 nm, (i) 60° and 1040 nm.

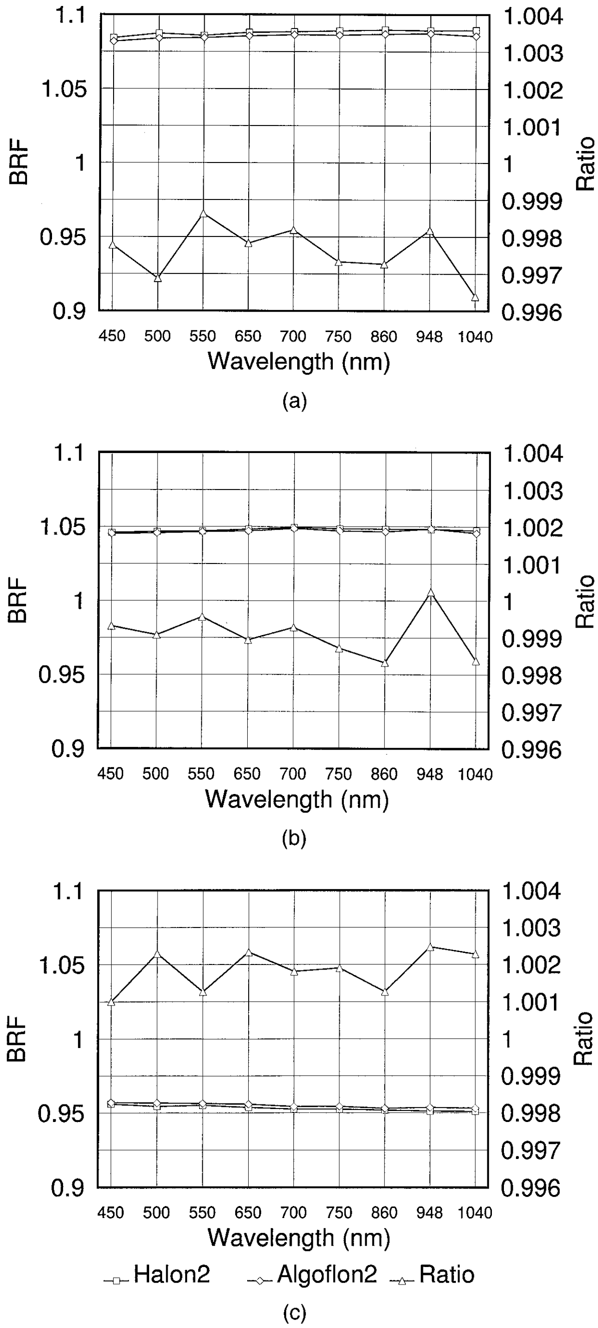
materials are within approximately 0.5% of each other, well within the relative precision of the measurements. Thus it is assumed here that the hemispherical reflectance of the two materials is equal, and the value given by Ref. 1 is used here as the hemispherical reflectance of Algoflon.

B. Bidirectional Reflectance Factor Comparisons

Four Halon and four Algoflon samples were made and their BRF's measured as a function of incidence angle, with the radiometer remaining normal to the sample surface for all incidence angles. The results described below are representative of the comparisons of all four samples and for all the measured wavelengths of 450, 500, 550, 650, 700, 750, 860, 948, and 1040 nm. Data for only three wavelengths will be presented here as they are representative of all the measured data and the entire wavelength range. The comparison of a visually good Halon sample and an Algoflon sample is shown in Fig. 4 for wavelengths of 450, 700, and 1040 nm. Shown in the figure are sample 2 Halon and Algoflon measured BRF's and the ratio of Algoflon to Halon BRF's. For all wavelengths Algoflon compares well with Halon, having a difference between the two BRF's of less than 0.4% for incidence angles between 10° and 80°. For 85° incidence, the difference is less than about 1.25%; it is largest for the longer wavelengths.

Figure 5 shows BRF's for the same three wavelengths for a visually acceptable Halon sample and a visually unacceptable Algoflon sample. The Algoflon sample was considered unacceptable because it had what appeared to be a rather large nonuniformity near the center of the sample. It appeared as though this was due to clumping or uneven distribution of the material during compaction. By far, this was visually the worst of all eight samples; and for typical calibration measurements, a sample such as this would be rejected. For incidence angles from 10° to 65° the difference is less than ~1% for all wavelengths. For larger incidence angles the difference is as large as 4.5% with the largest difference occurring for the shorter wavelengths. However, in this case the unacceptable Algoflon sample is more Lambertian than the good Halon sample.

The data presented thus far do not contain information regarding the specular direction. Figure 6 presents such data showing the BRF's and ratios for Halon and Algoflon samples, 4. For these data the radiometer was scanned from approximately −65° to +65° about the sample normal while the incidence angle was fixed at 10°, 30°, and 60°. Data are shown for 450 [Figs. 6(a)–6(c)], 700 [Figs. 6(d)–6(f)], and 1040 nm [Figs. 6(g)–6(i)]. These were not ideal samples but, typically, they would be visually acceptable. The curves are discontinuous because data collected where the incoming beam is partially blocked by the detector were deleted from the graphs. Again Algoflon compares well with Halon. Generally speaking, for wavelengths of 450 and 700 nm and incidence angles of 10° and 30°, the ratios are less than ~1%. For 60° and these same two wavelengths, the difference approaches 5% for large radiometer angles; however, the Algoflon is slightly more Lambertian than the Halon. The 1040-nm data are somewhat discrepant with respect to other BRF data. The magnitudes of the reflectance seem appropriate, but the Algoflon-to-Halon ratio is too small compared with other measurements. The cause of this is unclear, but we suspect that there was a signal change resulting from the silicon detector's temperature sensitivity that begins beyond ~900 nm. Regardless, there is no evident specular component.



(a)

(b)

(c)

Fig. 7. BRF of Halon and Algoflon as a function of wavelength with a normal detector viewing angle. The incidence angles are (a) 10°, (b) 30°, (c) 60°.

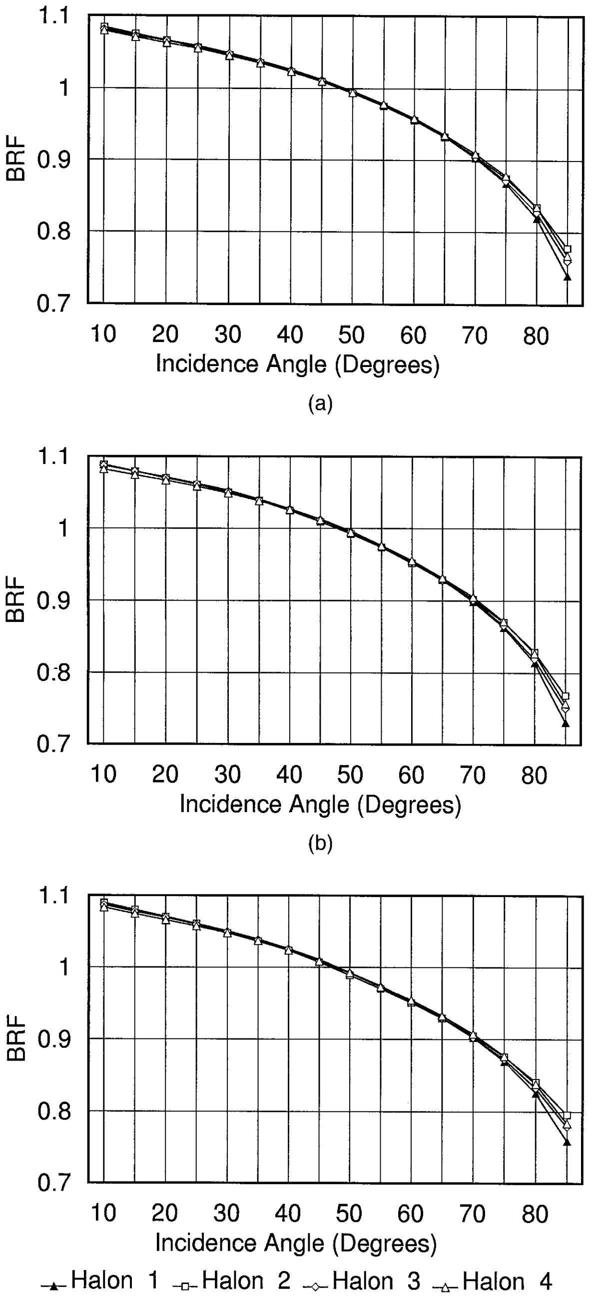


Fig. 8. BRF comparison of four Halon samples with a varying incidence angle and the detector fixed normal to the sample. Wavelengths are (a) 450 nm, (b) 700 nm, (c) 1040 nm.

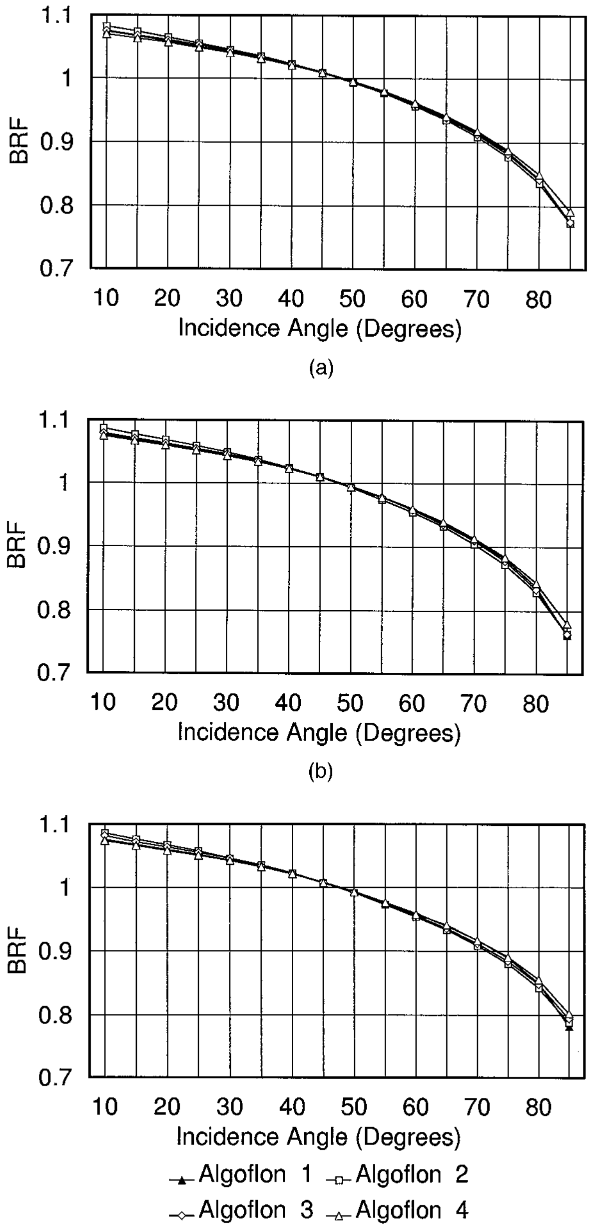


Fig. 9. BRF comparison of four Algoflon samples with a varying incidence angle and the detector fixed normal to the sample. Wavelengths are (a) 450 nm, (b) 700 nm, (c) 1040 nm.

C. Wavelength Dependencies

For sample 2, Fig. 7 shows the BRF as a function of wavelength for incidence angles of 10°, 30°, and 60° and the radiometer view being normal to the sample surface. Like Halon, Algoflon essentially has no wavelength dependence.

D. Sample Variability

A major goal in finding a replacement reflectance standard is achieving confidence that the standard can be repeated from time to time. The BRF's of the four Halon and the four Algoflon samples are compared in Figs. 8 and 9, respectively, for wavelengths of 450, 700, and 1040 nm. The sample-to-sample variability of Algoflon is comparable with that of Halon, even with the visually unacceptable Algoflon sample included (sample 1). Note that the Algoflon samples are generally more Lambertian than the Halon samples.

E. Sample Lifetime

Once a sample is pressed one would like to know how long the sample will be good. An Algoflon sample was made and its BRF measured on days 1, 2, 13, 20, 28, and 268 (approximately 9 months). Figure 10 shows a comparison of the day one data with the 9-month data for wavelengths of 450, 700, and 1040 nm. This

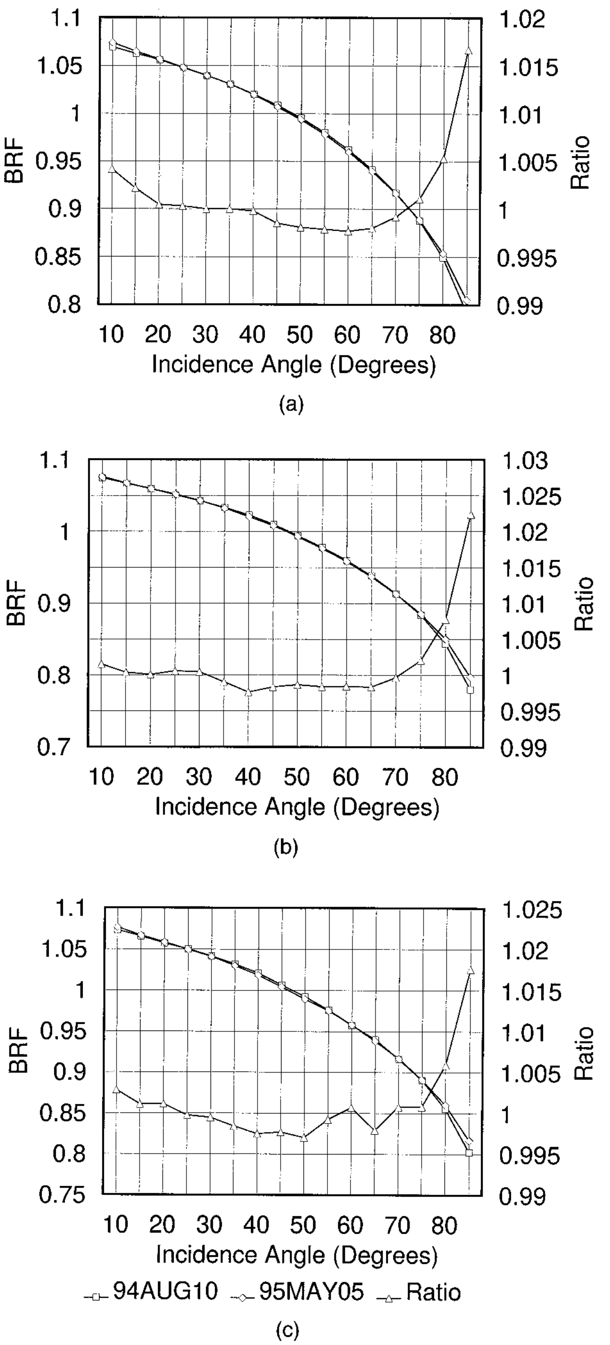


Fig. 10. Two BRF measurements of the same Algoflon sample spaced in time by approximately 9 months. Wavelengths are (a) 450 nm, (b) 700 nm, (c) 1040 nm.

figure illustrates that for a 9-month period the sample change was not measurable, with the two measurements being within a few tenths of a percent for all but the two largest angles of incidence. The intermediate days also show no evidence of change.

6. Conclusions

Hemispherical reflectance and BRF measurements were performed on pressed Algoflon F6 and Halon to determine the possibility of using Algoflon as a new diffuse reflectance standard. Algoflon's hemispherical reflectance was found to be equal (within measurement error) to that of Halon, and the BRF measurements indicate that the two materials are essentially identical for wavelengths from 450 to 1040 nm. If anything, Algoflon appears to be more Lambertian than Halon. The BRF's are essentially wavelength independent, very repeatable from sample to sample, and have a lifetime greater than approximately 9 months. Algoflon is unquestionably an excellent replacement for Halon for the wavelength range of 450–1040 nm.

However, there is a downside. During the course of reducing the data and writing this paper, the production of Algoflon F6 was discontinued. The manufacturer has indicated that it is considering reinstating this material as a standard product but there is no assurance that it will ever be available. The usefulness of this work is that it illustrates that PTFE powders, if chosen appropriately, can be used as a consistent reflectance-material standard, and these results are useful for those who have a stock of the material.

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