Standard Practice for Transfer Standards for Reflectance Factor for Near-Infrared Instruments Using Hemispherical Geometry[1]

INTRODUCTION

The internationally accepted standard of reflectance is the perfect reflecting diffuser. This ideal reflecting surface reflects 100 % of the radiant power incident on it, such that the radiance is the same for all directions within the hemisphere of solid angles. No physical realization of this standard exists. Optical properties of standards prepared from pressed plaques of barium sulfate ($BaSO_4$) or polytetrafluoroethylene (PTFE), as well as commercially available samples of sintered PTFE **(1-4)**,[2] can approximate those of a white material. For further information, see CIE Publication No. 46 **(5)** and Specification D 1457. Additional transfer standards are required that have a very stable reflectance factor that is constant with wavelength and that have a range of values from near zero to close to that of the perfect reflecting diffuser. Such materials as carbon-black doped sintered PTFE **(6-8)** fulfill this requirement. The principle uses of a reflectance factor standard are for transfering an absolute scale of reflectance to a more durable material or for calibrating near-infrared (NIR) spectrophotometers for linearity of reflectance scale. In theory, this transfer, conducted from first principles, should be quite easy. In practice, values are likely to be required for parameters that are unknown, proprietary, or require a highly sophisticated level of skill. Some, but not all, of these parameters are discussed in this practice.

1. Scope

1.1 This practice covers procedures for the preparation and use of acceptable transfer standards for NIR spectrophotometers. Procedures for calibrating the reflectance factor of materials on an absolute basis are contained in CIE Publication No. 44 (**9**). Both the pressed powder samples and the sintered PTFE materials are used as transfer standards for such calibrations because they have very stable reflectance factors that are nearly constant with wavelength and because the distribution of flux resembles closely that from the perfect reflecting diffuser.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1457 Specification for Polytetrafluoroethylene (PTFE) Molding and Extrusion Materials[3]

E 131 Terminology Relating to Molecular Spectroscopy[4]

E 259 Practice for Preparation of Pressed Powder White Reflectance Factor Transfer Standards for Hemispherical Geometry[5]

E 284 Terminology of Appearance[5]

3. Terminology

3.1 *Definitions*—Terms and definitions in Terminology E 284 are applicable to this practice.

3.2 *Descriptions of Terms Specific to This Standard*—The following definitions are particularly important to this practice.

3.2.1 *linearity*—the ability of a photometric system to yield a linear relationship between the radiant power incident on its detector and some measureable quantity provided by the system. **(E 131)**

3.2.2 *near-infrared*, *adj*—the region of the electromagnetic spectrum for radiation of wavelengths between 780 and 2500 nm (0.78 and 2.50 µm).

[1] This practice is under the jurisdiction of ASTM Committee E-13 on Molecular Spectroscopy and is the direct responsibility of Subcommittee E13.11 on Chemometrics.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this practice.

[3] *Annual Book of ASTM Standards*, Vol 08.01.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] *Annual Book of ASTM Standards*, Vol 06.01.

3.2.3 *perfect reflecting diffuser*—ideal reflecting surface that neither absorbs nor transmits light, but reflects diffusely, with the radiance of the reflecting surface being the same for all reflecting angles, regardless of the angular distribution of the incident light.

3.2.4 *reflectance, r, n*—ratio of the reflected radiant or luminous flux to the incident flux in the given conditions **(1)**.

3.2.4.1 The term reflectance is often used in a general sense or as an abbreviation for reflectance factor. Such usage may be assumed unless the definition is specifically required by the context.

3.2.5 *reflectance factor, R, n*—ratio of the flux reflected from the specimen to the flux reflected from the perfect reflecting diffuser under the same geometric and spectral conditions of measurement **(2)**.

4. Summary of Practice

4.1 Procedures for the preparation of packed powder samples of barium sulfate and PTFE can be found in Practice E 259. Sintered PTFE samples are commercially available.[6] Reflectance data for this material are given in Table 1. These materials provide close approximation to the optical properties of the perfect reflecting diffuser and may be used to transfer a scale of reflectance factor to another material or instrument.

TABLE 1 6°/Typical Diffuse Reflectance for Sintered (Labsphere Spectralon™ SRS-99)^A

Wavelength, nm	Reflectance Factor
250	0.940
300	0.977
400	0.991
500	0.991
600	0.991
700	0.990
800	0.991
900	0.991
1000	0.990
1100	0.990
1200	0.989
1300	0.988
1400	0.986
1500	0.988
1600	0.987
1700	0.984
1800	0.984
1900	0.978
2000	0.970
2100	0.950
2200	0.963
2300	0.955
2400	0.944
2500	0.940

Density = 1500 kg/m^3; thickness ≥ 7 mm.

^{A} Available from Labsphere, Inc., P.O. Box 70, North Sutton, NH 03260-0070; uncertainty of measurement ±0.002.

4.2 Sintered carbon-black doped PTFE samples are also commercially available[7] and are described in Table 2. These materials provide close approximation to the optical properties of a perfect reflecting diffuser with spectrally neutral absorbance features and may be used to transfer a scale of linearity in reflectance factor to another material or instrument.

TABLE 2 6°/Typical Diffuse Reflectance for Three Sintered Carbon-Black Doped PTFE (Labsphere Spectralon™ SRS-80, SRS-10, and SRS-02)^A

Wavelength, nm	SRS-80	SRS-10	SRS-02
250	0.774	0.106	0.015
300	0.793	0.099	0.016
400	0.795	0.097	0.017
500	0.796	0.099	0.017
600	0.797	0.101	0.017
700	0.799	0.103	0.017
800	0.802	0.105	0.018
900	0.803	0.105	0.017
1000	0.805	0.106	0.018
1100	0.806	0.108	0.017
1200	0.807	0.109	0.018
1300	0.808	0.111	0.018
1400	0.808	0.112	0.018
1500	0.810	0.113	0.020
1600	0.811	0.114	0.021
1700	0.812	0.115	0.023
1800	0.813	0.116	0.024
1900	0.811	0.118	0.026
2000	0.814	0.117	0.027
2100	0.809	0.114	0.030
2200	0.812	0.110	0.032
2300	0.813	0.110	0.035
2400	0.809	0.103	0.034
2500	0.809	0.101	0.038
	thickness ≤7 mm	thickness ≤5 mm	thickness ≤3 mm

^{A} Available from Labsphere, Inc., P.O. Box 70, North Sutton, NH 03260-0070. Materials are available in nominal reflectance factor values at 600 nm from between 0.02 and 0.99.

5. Significance and Use

5.1 Most commercial reflectometers and spectrophotometers with reflectance capability measure relative reflectance. The instrument reading is the ratio of the measured radiation reflected from the reference specimen to the measured radiation reflected by the test specimen. That ratio is dependent on specific instrument parameters.

5.2 National standardizing laboratories and some research laboratories measure reflectance on instruments calibrated from basic principles, thereby establishing a scale of absolute reflectance as described in CIE Publication No. 44 **(5)**. These measurements are sufficiently difficult and of prohibitive cost that they are usually left to laboratories that specialize in them.

5.3 A standard that has been measured on an absolute scale could be used to transfer that scale to a reflectometer. While such procedures exist, the constraints placed on the mechanical properties restrict the suitability of some of the optical properties, especially those properties related to the geometric distribution of reflected radiation. Thus, reflectance factor standards that are sufficiently rugged or cleanable to use as permanent transfer standards, with the exception of the sintered PTFE standards, depart considerably from the perfect diffuser in the geometric distribution of reflected radiation.

5.4 The geometric distribution of reflected radiance from such standards is sufficiently diffuse that such a standard can provide a dependable calibration of a directional-hemispherical or certain directional-directional reflectometers. Although pressed powder standards are subject to contamination and

[6] Such material is available under the trade name Spectralon SRS-99 from Labsphere, Inc., P.O. Box 70, North Sutton, NH 03260-0070.

[7] Such material is available under the trade name Spectralon SRS-XX from Labsphere, Inc., P.O. Box 70, North Sutton, NH 03260-0070.

breakage, the reflectance factor of pressed powder can be sufficiently reproducible from specimen to specimen from a given lot of powder to allow the assignment of absolute reflectance factor values to all of the powder in a lot.

5.5 Sintered PTFE materials exhibit sufficient reproducibility from within the same specimen after resurfacing or cleaning the specimen to allow the assignment of absolute reflectance factor values.

5.6 Preparation of packed powder reflectance standards is covered in Practice E 259. This practice describes the spectral and physical properties of these materials and of the sintered PTFE materials.

6. Apparatus

6.1 The basic apparatus for preparing pressed powder standards includes a powder press, powder containers, and an analytical balance. Powder presses suitable for the production of standards are commercially available.[8]

6.2 Sintered PTFE specimens, both white and for linearity testing, are commercially available.[6,7]

7. Handling Procedures

7.1 Pressed plaques should be kept in a dessicator when not in use. Pressed powder samples of both barium sulfate and PTFE are prone to particulate contamination and electrostatically attract airborne particles. Packed PTFE powder is also susceptible to absorbing vapors from organic solvents. All such contaminants can make these materials slightly luminescent and reduce reflectance in the ultraviolet and NIR regions. Typical reflectance data for pressed barium sulfate and pressed PTFE powder are given in Table 3.

7.2 Sintered PTFE plaques or standards should be kept in a clean, dust-free environment when not in use. Higher reflectance specimens are prone to particulate contamination and electrostatically attract airborne particles. Sintered PTFE samples are also susceptible to absorbing vapors from organic solvents. Such contaminants can make these materials slightly luminescent and reduce reflectance in the ultraviolet and NIR regions. Typical reflectance data for sintered PTFE materials are given in Table 1 and for a range of sintered carbon-black doped PTFE are given in Table 2.

TABLE 3 6°/Diffuse Reflectance Factor of Eastman-Kodak White Reflectance[A]

Wavelength, nm	Reflectance Factor
Standard Pressed Barium Sulfate Powder[B,C]	
300	0.968
350	0.979
400	0.987
450	0.991
500	0.991
550	0.992
600	0.992
650	0.992
700	0.992
750	0.992
800	0.992
850	0.991
900	0.990
950	0.988
1000	0.986
Pressed PTFE Powder[D,E]	
300	0.984
400	0.993
500	0.994
600	0.994
700	0.994
800	0.994
900	0.994
1000	0.994
1100	0.994
1200	0.993
1300	0.992
1400	0.991
1500	0.992
1600	0.992
1700	0.990
1800	0.990
1900	0.985
2000	0.981
2100	0.968
2200	0.977
2300	0.972
2400	0.962
2500	0.960

[A] Reflectance data for packed PTFE over certain wavelengths in the NIR have been called into question. The data presented are those currently certified and used by NIST.

[B] Density = 2000 kg/m^3; thickness = 5 mm.

[C] Available from Eastman-Kodak Corp. as Kodak White Reflectance Standard 6091, Eastman-Kodak Laboratory and Research Products, Rochester, NY 14650.

[D] Density = 1000 kg/m ^{3}; thickness ≥ 7 mm.

[E] The following PTFE powders have been found acceptable: PTFE-M-12 from Daikin Industries, Ltd., 1-1 Nishihitotsuya Yodogawa, Siesakusho, Setto-Shi, Osaka (Japan); Teflon 7-A from E. I. Dupont de Nemours & Co., Barley Mill Plaza, Wilmington, DE 19880; and Ausimont Algoflon F-5 and F-6, available from Ausimont USA, Inc., CN-1838-T, Morristown, NJ 07960.

8. Precision and Bias

8.1 The National Institute for Standards and Technology (NIST) and the Intersociety Color Council Project Committee 22, Materials for Instrument Calibration, have conducted collaborative tests to determine the precision and bias of the preparation of PTFE reflectance factor standards **(6-8)**. The standard deviation of three determinations of the reflectance factor of PTFE by NIST ranged from 0.0002 to 0.0008 over the spectral range from 300 to 1000 nm. The measured reflectance of PTFE from two manufacturers exhibited differences of from −0.002 to +0.004 over the same range, with the largest differences near the ends of the range and a constant measurement uncertainty of ±0.005.

8.2 The CIE Publication Number 46 **(1)** cites literature references on the reproducibility of barium sulfate pressings that range from 0.05 to 1.0 %, with the most common value being 0.2 %. This puts the reproducibility of the plaque preparation near the level of the reproducibility of the international standardizing laboratories' ability to characterize the absolute reflectance of the material.

8.3 Collaborative studies have not been conducted on the sintered PTFE or carbon-black doped PTFE materials. However, these materials are now being supplied as calibrated transfer standards by both the National Physical Laboratory

[8] Powder presses for making acceptable standards are available from Carl Zeiss Canada, Ltd., 45 Valleybrook Drive, Don Mill, Ontario M3B 2S6, Canada, Part No. 505866; Technidyne Corp., 100 Quality Avenue, New Albany, IN 47150-2272, Part No. 176601; and Labsphere, Inc., P.O. Box 70, North Sutton, NH 03260-0070, Part No. PSH-020.

(United Kingdom) and the NIST, with the standard blanks provided by Labsphere, Inc.[6,7]

9. Keywords

9.1 hemispherical optical measurement system; integrating sphere; materials standards; reflectance and reflectivity; Spectralon™; transfer standards

REFERENCES

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