Designation: E 903 – 96

Standard Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres[1]

This standard is issued under the fixed designation E 903; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of spectral absorptance, reflectance, and transmittance of materials using spectrophotometers equipped with integrating spheres.

1.2 Methods of computing solar weighted properties from the measured spectral values are specified.

1.3 This test method is applicable to materials having both specular and diffuse optical properties. Except for transmitting sheet materials that are inhomogeneous, patterned, or corrugated, this test method is preferred over Test Method E 1084.

1.4 *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:*

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[2]

E 424 Test Methods for Solar Energy Transmittance and Reflectance (Terrestrial) of Sheet Materials[3]

E 490 Solar Constant and Air Mass Zero Solar Spectral Irradiance Tables[4]

E 772 Terminology Relating to Solar Energy Conversion[3]

E 891 Tables for Terrestrial Direct Normal Solar Spectral Irradiance for Air Mass 1.5[3]

E 1084 Test Method for Solar Transmittance (Terrestrial) of Sheet Materials Using Sunlight[3]

E 1175 Test Method for Determining Solar or Photopic Reflectance, Transmittance, and Absorptance of Materials Using a Large Diameter Integrating Sphere[3]

2.2 *Other Document:*

Federal Test Method Standard No. 141, Method 6101[5]

3. Terminology

3.1 *Definitions*—The following definitions are consistent with Terminology E 772. Additional terms appropriate to this test method are included in Terminology E 772.

3.1.1 *absorptance,* α, *n*—the ratio of the absorbed radiant flux to the incident radiant flux.

3.1.2 *diffuse*, *adj*—indicates that flux propagates in many directions, as opposed to direct beam, which refers to collimated flux. When referring to reflectance, it is the directional-hemispherical reflectance less the specular reflectance.

3.1.3 *integrating sphere*, *n*—an optical device used to either collect flux reflected or transmitted from a sample into a hemisphere or to provide isotropic irradiation of a sample from a complete hemisphere. It consists of a cavity that is approximately spherical in shape with apertures for admitting and detecting flux and usually having additional apertures over which sample and reference specimens are placed.

3.1.4 *irradiance, E*, *n*—a radiometric term for the radiant flux that is incident upon a surface ($W\cdot m^{-2}$).

3.1.5 *near normal-hemispherical*, *adj*—indicates irradiance to be directional near normal to the specimen surface and the flux leaving the surface or medium is collected over an entire hemisphere for detection.

3.1.6 *radiant flux,* Φ, *n*—a radiometric term for the time rate of flow of energy in the form of electromagnetic energy (watts).

3.1.7 *reflectance,* ρ, *n*—the ratio of the reflected radiant flux to the incident radiant flux.

3.1.8 *solar*, *adj*—(*1*) referring to radiometric quantities, indicates that the radiant flux involved has the sun as its source, or has the relative spectral distribution of solar flux, and (*2*) referring to an optical property, indicates a weighted average of the spectral property, with a standard solar spectral irradiance distribution as the weighting function.

3.1.9 *spectral*, *adj*—(*1*) for dimensionless optical properties, indicating that the property was evaluated at a specific wavelength, λ, within a small wavelength interval, $\Delta\lambda$ about λ, symbol wavelength in parentheses as L(350 nm), or as a

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

[3] *Annual Book of ASTM Standards*, Vol 12.02.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from Standardization Documents, Order Desk, Building 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5049, Attn: NPODS.

function of wavelength, symbol L(λ), and (*2*) for a radiometric quantity, the concentration of the quantity per unit wavelength (or frequency), indicated by the subscript lambda, as $L_\lambda = dL/d\lambda$; at a specific wavelength, the wavelength at which the spectral concentration was evaluated may be indicated by the wavelength in parentheses following the symbol, Lλ (350 nm).

3.1.9.1 *Discussion*—The parameters of frequency, ν, wavenumber, κ, or photon energy may be substituted for wavelength, λ , in this definition.

3.1.10 *specular*, *adj*—indicates the flux leaves a surface or medium at an angle that is numerically equal to the angle of incidence, lies in the same plane as the incident ray and the perpendicular, but is on the opposite side of the perpendicular to the surface.

3.1.10.1 *Discussion*—Diffuse has been used in the past to refer to hemispherical collection (including the specular component). This use is deprecated in favor of the more precise term *hemispherical*.

3.1.10.2 *Discussion*—Reversing the order of terms in an adjective reverses the geometry of the incident and collected flux respectively.

3.1.11 *transmittance,* τ, *n*—the ratio of the transmitted radiant flux to the incident radiant flux.

4. Summary of Test Method

4.1 Measurements of spectral near normal-hemispherical transmittance (or reflectance) are made over the spectral range from approximately 300 to 2500 nm with an integrating sphere spectrophotometer.

4.2 The solar transmittance, reflectance, or absorptance is obtained by calculating a weighted average with a standard solar spectral irradiance as the weighting function by either the weighted (see 8.3.3) or selected (see 8.3.4) ordinate method.

5. Significance and Use

5.1 Solar-energy absorptance, reflectance, and transmittance are important in the performance of all solar energy systems ranging from passive building systems to central receiver power systems. This test method provides a means for determining these values under fixed conditions that represent an average that would be encountered during use of a system in the temperate zone.

5.2 Solar-energy absorptance, reflectance, and transmittance are important for thermal control of spacecraft and the solar power of extraterrestrial systems. This test method also provides a means for determining these values for extraterrestrial conditions.

5.3 This test method is designed to provide reproducible data appropriate for comparison of results among laboratories or at different times by the same laboratory and for comparison of data obtained on different materials.

5.4 This test method has been found practical for materials having both specular and diffuse optical properties except for those materials that are inhomogeneous, patterned, or corrugated.

6. Apparatus

6.1 *Instrumentation*:

6.1.1 *Spectrophotometer*—A spectrophotometer with an integrating sphere attachment capable of measuring the spectral characteristics of the test specimen or material over the solar spectral region from approximately 300 to 2500 nm is required. Double beam, ratio recording instruments are recommended because of their low sensitivity to drift in source brightness or amplifier gain. Recording spectrophotometers with integrating spheres that have been found satisfactory for this purpose are commercially available.

NOTE 1—For determining extraterrestrial solar optical properties using Standard E 490, the spectral region should extend down to 250 nm.

6.1.1.1 The integrating sphere shall be either a wall-mounted type such that the specimen may be placed in direct contact with the rim of an aperture in the sphere wall for transmittance and reflectance measurements or an Edwards type such that the specimen is mounted in the center for reflectance and absorptance measurements.

NOTE 2—The interior of the integrating sphere shall be finished with a stable highly reflecting and diffusing coating. Sphere coatings having the required properties can be prepared using pressed tetrafluoroethylene polymer powder,[6] airbrushed Eastman White Reflectance Paint.[7]

NOTE 3—For high accuracy (better than ±0.01 reflectance units) measurements, the ratio of the port area to the sphere wall plus port area should be less than 0.001 (**1**).[8] In general, large spheres (> 200 mm) meet these requirements and are preferred while small spheres (< 100 mm) can give rise to large errors.

6.1.1.2 For the evaluation of near normal-hemispherical or hemispherical-near-normal reflectance, the direction of the incident radiation or the direction of viewing respectively shall be between 6 and 12° from the normal to the plane of the specimen so that the specular component of the reflected energy is not lost through an aperture. Ambient light must be prevented from entering the sphere by placing a ring of black velvet around the external rim of the specimen ports or by covering the entire sphere attachment with a light tight housing. Several acceptable system configurations are illustrated in Annex A1.

NOTE 4—The hemispherical near-normal irradiation-viewing mode is also allowed under this test method since the Helmholtz reciprocity relationship which holds in the absence of polarization and magnetic fields guarantees equivalent results are obtainable.

6.2 *Standards*:

6.2.1 In general, both reference and working (comparison) standards are required. Reference standards are the primary standard for the calibration of instruments and working standards. Reference standards that have high specular reflectance, high diffuse reflectance, and low diffuse reflectance are available from the National Institute of Standards and Technology as Standard Reference Materials (SRM).[9]

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[6] Halon, available from the Allied Chemical Co., P.O. Box 697, Pottsville, PA 17901, has been found satisfactory for this purpose.

[7] Available from Kodak Laboratory and Specialty Chemicals, Eastman Kodak Co., 343 State St., Rochester, NY 14650.

[8] The boldface numbers in parentheses refer to the list of references appended to this test method.

[9] National Institute of Standards and Technology, Office of Standard Reference Materials, Room B311, Chemistry Bldg., Washington, DC 20234. Additional details covering the appropriate SRMs(2019–2022) are available on request.

6.2.1.1 Working standards are used in the daily operation of the instrument to provide comparison curves for data reduction. In general, ceramic and vitrified enamel surfaces are highly durable and desirable. A working standard shall be calibrated by measuring its optical properties relative to the properties of the appropriate reference standard using procedures given in 8.2.

NOTE 5—Even the best standards tend to degrade with continued handling. They should be handled with care and stored in a clean, safe manner. Working standards should be recalibrated periodically and cleaned, renewed, or replaced if degradation is noticeable. Avoid touching the optical surfaces. Only clean soft cloth gloves should be worn for handling the standards. Only lens tissue or sterile cotton is recommended for cleaning. This is especially important for reference standards carrying NBS calibration.

6.2.2 For transmitting specimens, incident radiation shall be used as the standard relative to which the transmitted light is evaluated. For some applications calibrated transmittance standards are available.

6.2.3 For diffuse high-reflectance specimens, a working standard that has high reflectance and is highly diffusing over the range of the solar spectrum is required.

NOTE 6—Identified suitable working standards are tablets of pressed tetrafluoroethylene polymer, $BaSO_4$, $BaSO_4$-based paints, and white ceramic tile. The Halon **(2)** has superior reflectance at the longer wavelengths of interest but is less durable and more difficult to reproduce accurately. Magnesium oxide either pressed or smoked is no longer recommended for use as a standard.

6.2.4 For specularly reflecting specimens, a working standard that is highly specular is required. Identified suitable working standards are vacuum-deposited thin opaque films of metals. All front surface metalized working standards shall be calibrated frequently with an absolute reflectometer or relative to an NIST standard reference mirror before being acceptable in this test method. An acceptable working standard for low-specular reflectance is a flat piece of optically polished black glass.

NOTE 7—Although aluminum is most often used because of its high reflectance and ease of deposition, it is very unstable and scratches easily. Other metals such as chromium, nickel, and rhodium are much more durable. High vacuum ($\geq 10^{-10}$ torr) is required for obtaining pure films with the best optical properties **(3)**.

6.2.5 For absorber materials, a working standard that has low reflectance over the range of the solar spectrum is required in order to obtain an accurate zero line correction.

NOTE 8—Black semi-matt porcelain enameled substrates, black barbeque, stove, or wrought iron fence paints, and opaque black glass are suitable working standards. For very low-reflectance materials light traps reflecting < 0.005 can be fabricated to calibrate sphere performance.

NOTE 9—Light traps can be made by viewing the edge of a stack of razor blades, a 60° black cone, or by forming an approximate exponential horn by drawing a glass tube and painting it with a high-gloss black paint.

6.2.6 If an absolute sphere is completely free of the flux losses referred to in 10.2.1, no working standard is required. A comparison of the measured reflectance of a primary reference standard to its calibration value will give a good estimate of the error due to flux losses, if any, from a nearly absolute sphere such as described in A1.1.2 and A1.1.3.

7. Test Specimens

7.1 *Specimens for Wall-Mounting Spheres*:

7.1.1 The size of test specimens required depends on the dimensions of the integrating sphere. For wall-mounted spheres the specimen must be large enough to cover the aperture of the sphere. There may be no limit on maximum dimension. For patterned samples, either the specimen shall be large enough to make a number of measurements over different areas, or several specimens representing the different areas of the material shall be used.

7.1.2 Opaque specimens shall have at least one surface that is essentially plane over an area large enough to cover the aperture of the sphere.

7.1.3 Transparent and slightly translucent specimens shall have two surfaces that are essentially plane and parallel. In order to reduce light scattered out the edges of translucent specimen, the minimum distance between the edge of the beam and the edge of the aperture shall be ten times the thickness of the specimen.

7.1.4 The transmittance of highly scattering translucent samples is not easily measured with an integrating sphere instrument, because a significant portion of the incident flux will be scattered outside the aperture. For such materials the standard test method using the sun as a source (Test Methods E 1084 or E 1175) is preferred.

NOTE 10—If such a sample must be measured, the edge losses can be greatly reduced by using a circular sample of diameter slightly less than that of the aperture, and coating the edge with silver, using the wet mirror process. Alternatively small stops can be cemented to the edges of the sample, so that it can be suspended in the aperture with about half of its thickness extending outside the aperture.

7.2 *Specimens for Edwards Sphere*—The area of the specimen shall be limited to 0.01 of the surface area of the sphere.

NOTE 11—For a 200-mm diameter sphere, the required specimen size would be |Lq 20 mm in radius.

8. Procedure

8.1 *Calibration*—Calibrate linearity and wavelength scales of the spectrophotometers as recommended by the manufacturer or in accordance with Practice E 275. Check on calibration annually.

8.2 *Measurement*:

8.2.1 *Correction for 100 % and Zero Line Errors*:

8.2.1.1 Record 100 % and zero line curves at least twice a day during testing.

NOTE 12—Variations in signal from the two beams are normal, usually wavelength dependent, and give rise to nonideal 100 % lines. Similarly beam cross talk, light scattering or leaks, and detector noise give rise to a nonideal zero line. These effects produce errors in the measured ratio of the flux reflected by the specimen and the working standard.

8.2.1.2 For spheres with separate sample and reference ports, record the 100 % line curves using identical high-reflectance specimens in both ports. The specimens are identical in reflectance if the recorded curve does not change when the two specimens are interchanged.

8.2.1.3 For reflectance measurements, record the zero line with a perfect absorber or light trap in the sample port.

NOTE 13—The practice of recording the zero line with the same beam blocked at the entrance port is discouraged because the effect of scattered light incident on the sphere wall is not included.

8.2.1.4 For transmittance measurements, record the zero line with the sample beam blocked, preferably as far in front of the entrance port as convenient.

8.2.2 *Reflectance of Opaque Specimen—ComparisonType Sphere:*

8.2.2.1 Record the spectral 100 % and zero lines as indicated in 8.2.1.

8.2.2.2 Record the spectral reflectance of specimen relative to the working standard by placing the specimen on the sample port and the standard on the reference port. Include the specular component in the measurement.

8.2.2.3 Compute the spectral reflectance, ρ(λ), for the specimen, at wavelength λ using:

$$\rho(\lambda) = (S_\lambda - Z_\lambda)/(100_\lambda - Z_\lambda)\rho'(\lambda) \tag{1}$$

where:

S_λ = recorded specimen reading,
Z_λ = zero line reading,
100_λ = 100 % line reading, and
$\rho'(\lambda)$ = calibrated spectral reflectance for the working standard or reference, all at wavelength λ.

8.2.3 For the reflectance of opaque specimen of an absolute sphere, compute the spectral reflectance as:

$$\rho(\lambda) = (S_\lambda - Z_\lambda)/(100_\lambda - Z_\lambda) \tag{2}$$

where:

100_λ = 100 % correction obtained with the specimen port replaced by a sample having a coating and a curvature identical to the sphere wall. The zero line correction for an absolute sphere is usually so small that it can be neglected.

NOTE 14—Slightly different procedures may be required for other sphere designs.

8.2.4 For reflectance of transparent or translucent materials or specimens having transmittance greater than 0.001, back the specimen by a light trap or black material having a low reflectance (< 0.02) over the 300 to 2500-nm spectral range. For these measurements, the zero line shall be recorded with the specimen removed but the light trap or backing still in place. Obtain the spectral reflectance following 8.2.2.

8.2.5 *Transmittance*—Cover the specimen and reference ports at the rear of the sphere with surfaces having the same coating and optical properties as the sphere walls when measuring transmittance (Note 15). Record spectral curves without any specimen in place and then with the specimen over the specimen beam entrance port of the sphere. Calculate the spectral transmittance as:

$$\tau(\lambda) = (S_\lambda - Z_\lambda)/(100_\lambda - Z_\lambda) \tag{3}$$

where:

S_λ = signal recorded with the specimen over the entrance port,
Z_λ = zero line reading with the specimen beam blocked with an opaque material, and
100_λ = line recorded with no specimen over the specimen beam entrance port.

NOTE 15—The working standards, 6.2.3, could be used with only a small error.

8.2.6 *Absorptance*—For opaque samples record the reflectance spectrum as in 8.2.2. The solar absorptance is calculated by first obtaining the solar reflectance as in 8.3 and subtracting from 1, that is, $\tau_s = 0$ in the Kirchoff relationship:

$$\alpha_s + \tau_s + \rho_s = 1 \tag{4}$$

8.2.6.1 For non-opaque samples, either obtain both the solar reflectance and solar transmittance using the described techniques and calculate the solar absorptance by using the Kirchoff relationship, or use an Edwards-type integrating sphere instrument with the specimen mounted so that the beam that exits through the back of the specimen is free to fall on the sphere wall. In this case the sum $\tau(\lambda) + \rho(\lambda)$ is measured directly. Then use 8.3 and the Kirchoff relationship to determine the solar absorptance.

8.3 *Computation of Solar Properties*— Solar energy transmittance or reflectance is computed by the weighted ordinate or 50 selected ordinate method.

8.3.1 *Solar Spectral Irradiance Distribution*:

8.3.1.1 For terrestrial applications, Standard E 891 shall be used. Calculate the optical properties using the 50-point selected ordinate method.

8.3.1.2 For extraterrestrial applications, Standard E 490 shall be used. Calculate the optical properties using the weighted ordinate method.

8.3.2 *Product of Optical Properties*— When calculating solar optical efficiency of a complicated system such as a reflecting concentrator with an absorber in a transparent envelope, the product of ρ , τ, and α is required. The appropriate procedure is to measure the spectral optical properties of each component ρ(λ), α(λ), and τ(λ) respectively and form the product $\eta(\lambda) = \rho(\lambda)\alpha(\lambda)\tau(\lambda)$ before solar weighting. Calculate η_s as described in 8.3.3 or 8.3.4. Calculation of η_s from individually weighted properties can lead to substantial error, that is, $\eta_s \neq \rho_s\alpha_s\tau_s$**(4)**.

8.3.3 *Weighted Ordinates*—Obtain the solar reflectance ρ_s by integrating the spectral reflectance over the standard spectral irradiance distribution, *E*λ, as follows:

$$\rho_s = \left(\sum_{i=1}^{n} \rho(\lambda_i)E_{\lambda i}\right)\Delta\lambda_i \Big/ \left(\sum_{i=1}^{n} E_{\lambda i}\Delta\lambda_i\right) \tag{5}$$

Solar transmittance τ_s or absorptance α_s is obtained from a similar expression with ρ(λ) replaced by τ(λ) or α(λ) respectively. Here *n* is the number of wavelengths for which *E*gl is known. The $\Delta\lambda_i$ are not constant but are given by:

$$\Delta\lambda_i = (\lambda_{i+1} - \lambda_{i-1})/(2) \tag{6}$$

For $i = 1$ and $i = n$, one assumes a Δλ equal to the last interval, that is, $\Delta\lambda_1 = \lambda_2 - \lambda_1$ and $\Delta\lambda_n = \lambda_n - \lambda_{n-1}$.

8.3.4 *Selected Ordinates*:

8.3.4.1 In the selected ordinate method, the solar irradiance distribution is divided into *n* wavelength intervals each containing 1/*n* of the total irradiance. The spectral reflectance or