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Standard Test Method for Normal Spectral Emittance at Elevated Temperatures[1]

This standard is issued under the fixed designation E 307; 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.

ε[1] NOTE—Section 11 was added editorially in May 1996.

1. Scope

1.1 This test method describes a highly accurate technique for measuring the normal spectral emittance of electrically conducting materials or materials with electrically conducting substrates, in the temperature range from 600 to 1400 K, and at wavelengths from 1 to 35 μm.

1.2 The test method requires expensive equipment and rather elaborate precautions, but produces data that are accurate to within a few percent. It is suitable for research laboratories where the highest precision and accuracy are desired, but is not recommended for routine production or acceptance testing. However, because of its high accuracy this test method can be used as a referee method to be applied to production and acceptance testing in cases of dispute.

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

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 349 Terminology Relating to Space Simulation[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *spectral normal emittance*—the term as used in this specification follows that advocated by Jones **(1)**,[3] Worthing **(2)** and others, in that the word emittance is a property of a specimen; it is the ratio of radiant flux emitted by a specimen per unit area (thermal-radiant exitance) to that emitted by a blackbody radiator at the same temperature and under the same conditions. Emittance must be further qualified in order to convey a more precise meaning. Thermal-radiant exitance that occurs in all possible directions is referred to as hemispherical thermal-radiant exitance. When limited directions of propagation or observation are involved, the word directional thermal-radiant exitance is used. Thus, normal thermal-radiant exitance is a special case of directional thermal-radiant exitance, and means in a direction perpendicular (normal) to the surface. Therefore, spectral normal emittance refers to the radiant flux emitted by a specimen within a narrow wavelength interval centered on a specific wavelength and emitted in a direction normal to the plane of an incremental area of a specimen's surface. These restrictions in angle occur usually by the method of measurement rather than by radiant flux emission properties.

NOTE 1—All the terminology used in this test method has not been standardized. Definitions E 349 contain some approved terms. When agreement on other standard terms is reached, the definitions used herein will be revised as required.

4. Summary of Test Method

4.1 The principle of the test method is a direct comparison of the radiant flux from a specimen at a given temperature to the radiant flux of a blackbody at the same temperature and under the same environmental conditions of atmosphere and pressure. The details of this test method are given by Harrison et al **(3)** and Richmond et al **(4).**

4.2 The essential features of the test method are the use of a double-beam ratio-recording infrared spectrophotometer with variable slit widths, which combines and compares the signals from the specimen and the reference blackbody through a monochromator system which covers the wavelength range from 1 to 35 μm (Note 2). According to Harrison et al **(3)** a differential thermocouple with suitable instrumentation is used to maintain a heated specimen and the blackbody at the same temperature.

NOTE 2—An electronic-null, ratio-recording spectrophotometer[4] is preferred to an optical-null instrument for this use. It may be difficult to obtain and maintain linearity of response of an optical-null instrument if the optical paths are not identical to those of the instrument as manufactured.

5. Significance and Use

5.1 The significant features are typified by a discussion of

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[2] *Annual Book of ASTM Standards*, Vol 15.03.

[3] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[4] The Perkin-Elmer Model 13U has been found satisfactory for this purpose.

the limitations of the technique. With the description and arrangement given in the following portions of this test method, the instrument will record directly the normal spectral emittance of a specimen. However, the following conditions must be met within acceptable tolerance:

5.1.1 The effective temperatures of the specimen and blackbody must be within 1 K of each other. Practical limitations arise, however, because the temperature uniformities are often not better than a few degrees Kelvin.

5.1.2 The optical path length in the two beams must be equal, or the instrument should operate in a nonabsorbing atmosphere or a vacuum, in order to eliminate the effects of differential atmospheric absorption in the two beams. Measurements in air are in many cases important, and will not necessarily give the same results as in a vacuum, thus the equality of the optical paths for dual beam instruments becomes very critical.

NOTE 3—Very careful optical alignment of the spectrophotometer is required to minimize differences in absorptance along the two paths of the instrument, and careful adjustment of the chopper timing to reduce "cross-talk" (the overlap of the reference and sample signals) as well as precautions to reduce stray radiation in the spectrometer are required to keep the zero line flat. With the best adjustment, the "100 % line" will be flat to within 3 %; both of these measurements should be reproducible within these limits (see 7.3, Note 6).

5.1.3 Front-surface mirror optics must be used throughout, except for the prism in prism monochromators and the grating in grating monochromators, and it should be emphasized that equivalent optical elements must be used in the two beams in order to reduce and balance attenuation of the beams by absorption in the optical elements. It is recommended that optical surfaces be free of SiO_2 and SiO coatings; MgF_2 may be used to stabilize mirror surfaces for extended periods of time. The optical characteristics of these coatings are critical, but can be relaxed if all optical paths are fixed during measurements or the incident angles are not changed between modes of operation (during "0 % line," "100 % line," and sample measurements). It is recommended that all optical elements be adequately filled with energy.

5.1.4 The source and field apertures of the two beams must be equal in order to ensure that radiant flux in the two beams compared by the apparatus will pertain to equal areas of the sources and equal solid angles of emission. In some cases it may be desirable to define the solid angle of the source and sample when comparing alternative measurement techniques.

5.1.5 The response of the detector-amplifier system must vary linearly with the incident radiant flux.

6. Apparatus

6.1 The spectrophotometer used for the measurement of spectral normal emittance is equipped with a wavelength drive that provides automatic scanning of the spectrum of radiant flux and a slit servomechanism that automatically opens and closes the slits to minimize the variations of radiant flux in the comparison beam. For most materials the wavelength band-pass of the instrument is generally smaller than the width of any absorption or emission band in the spectrum to be measured. Operation of the spectrophotometer at a higher sensitivity level or in a single-beam mode can be used to evaluate band-pass effects. In a prism instrument, several prisms compositions can be used to cover the complete wavelength range; however, a sodium chloride prism is typically used to cover the spectral range from 1.0 to 15 µm, and a cesium bromide prism used to cover the spectral range from 15 to 35 µm. As a detector, a vacuum thermocouple with a sodium chloride window is used in the spectral range from 1 to 15 µm, and a vacuum thermocouple with a cesium bromide window in the spectral range from 1 to 35 µm. A black polyethylene filter is used to limit stray radiation in the 15 to 35-µm range.

6.2 In order to reduce the effects of absorption by atmospheric water vapor and carbon dioxide, especially in the 15 to 35-µm range, the entire length of both the specimen and reference optical paths in the instrument must be enclosed in dry air (dew point of less than 223 K) by a nearly gas-tight enclosure maintained at a slight positive pressure relative to the surrounding atmosphere.

6.3 The design of the reference blackbody is very critical when accurate measurements are to be made. Several designs are possible and a complete description of the one used at the National Institute of Standards and Technology is presented in Ref **(3).** Several points should be emphasized in the design of the blackbody reference. The temperature of the blackbody furnace is measured by means of a platinum, platinum-10 % rhodium thermocouple, the bare bead of which extends about 6 mm (¼ in.) into the cavity from the rear. The thermocouple leads are insulated from the core by high-alumina refractory tubing, which is surrounded by a grounded platinum tube to prevent pickup by the thermocouple of spurious signals due to electrical leakage from the winding. The effective emittance of any blackbody furnace which is to be used as a reference, computed by the DeVos' **(5)** or the Gouffé **(6)** equation as the situation dictates, should not be less than 0.995 assuming that the interior of the cavity is at a uniform temperature, within 3° and is a completely diffuse reflector.

6.4 The National Institute of Standards and Technology uses specimens in the shape of strips, 6 mm (¼ in.) wide by 200 mm (8 in.) long, of any convenient thickness. These specimens are heated by passing a current through the length of the strip. Specimen geometry is such that temperature uniformity can be adequately maintained.

6.5 The specimen enclosure should have certain design characteristics to allow for accurate and precise measurements.

6.5.1 The enclosure should be water cooled when measurements are being made at the higher end (1400 K) of the temperature range. Provisions should be made to cool the enclosure to 200 K or liquid nitrogen temperatures during measurements at the low end (600 K) of the temperature range especially when measuring low emittance specimens.

6.5.2 The inner surface of the enclosure should have a reflectance of less than 0.05 at the operating temperature of the water cooled walls. Several black paints[5] may be used; or alternatively, the inner surface may be constructed from a

[5] Parson's black, manufactured and sold by Thos. Parsons and Sons, Ltd., England; 3M Black Velvet, available from Reflective Products Division, Minnesota Mining and Manufacturing Co.; and Platinum Black have been found satisfactory for this purpose.

nickel-chromium-iron alloy which has been threaded with a No. 80 thread and then oxidized in air at a temperature above 1350 K for 6 h to obtain the desired reflectance.

6.5.3 For cylindrically shaped enclosures the specimen should be positioned off-center so that any radiant flux specularly reflected from the walls will be reflected twice before hitting the specimen.

6.5.4 With resistance heating techniques, the electrodes holding the specimen are water cooled and insulated from the ends of the enclosure. The lower electrode and enclosure configuration are designed to permit the specimen to expand without buckling when heated.

6.5.5 Adjustable baffles above and below the viewing window are used to reduce convection and the resulting temperature fluctuations and thermal gradients. Adjustable telescoping cylindrical reflectors surround the specimen at each end to reduce heat loss at the ends of the specimen, and the thermal gradients along the specimen.

6.6 The temperatures of the specimen and blackbody are adjusted to be equal within 1 K over the temperature range from 800 to 1400 K by means of a differential thermocouple. One bead of the differential thermocouple is located in the cavity of the blackbody furnace and the other is attached in such a manner as to be in intimate contact (Note 4) with the back of the specimen, in the center of the area being viewed. In the most common method of automatic control the signal from the differential thermocouple is amplified by a d-c amplifier and fed to a center-zero recorder-controller. The output of the recorder-controller is fed to a current-actuating-type controller, the output of this unit being fed to the coil of a saturable core reactor which varies the power input to the specimen. Other automatic, semiautomatic or manual methods of temperature control can be used if they maintain the above accuracy of the differential signal. Since temperature measurement can be a major source of error in making emittance measurements, welding or direct mechanical attachment of the differential thermocouple to a metallic specimen is desirable. However, such methods are not adequate for nonmetallic or coated metallic specimens unless temperature corrections based on the coating thickness and thermal conductivity are used.

NOTE 4—Intimate contact implies that the thermocouple bead assumes the same temperature as that of the specimen in the vicinity of the attachment.

7. Preparation of Apparatus and Procedure

7.1 Provide an adequate warm-up time of approximately 30 min for all equipment for all measurements of spectral normal emittance. In addition, purge the instrument and specimen enclosure for several hours with dry nitrogen or dry air, free from carbon dioxide, until the dew point in the system is less than 223 K in order to avoid serious absorption in the 15 to 35-μm range. Because of this relatively long period required for purging, it is recommended that the dry atmosphere be maintained continuously, except when the enclosure must be opened to permit adjustment of equipment or insertion of a new specimen.

NOTE 5—When standardizing the measurements using emittance standards, the nitrogen purge should be accomplished before the standard is heated. Atmospheric air passed through a drying tower filled with a CO_2 absorber then dried to a dew point of 173 K may be used instead of the dry nitrogen.

7.2 In making a wavelength calibration of the monochromator use standard calibration techniques in accordance with Plyler et al **(7)** and Blaine **(8)**. Typical techniques use the emission spectra of a helium arc, a mercury arc, and the absorption spectra of didymium glass or the atmosphere **(9)**, and a polystyrene film. The emission and absorption peaks having known wavelengths are identified in the respective curves, and for each peak the observed chart indication or wavelength drum position at which the peak occurred is plotted as a function of the known wavelength of the peak.

7.3 The linearity of response of the spectrophotometer must be established (within the varying wavelength interval encompassed by the exit slit) when the instrument is operated double-beam in ratio mode. In order to check linearity, two blackbody furnaces, controlled very closely to the same temperature (about 1400 K), are used as sources for the two beams. Adjust the instrument for the "100 % curve" operation. Then introduce sector-disk (see Table 1 and Note 6) attenuators into the specimen beam near the blackbody furnace each in turn to obtain "75, 50, 25, 12.5, and 5 % curves" over the wavelength range of interest. The height of each curve above the experimentally obtained zero for the pertinent wavelength is plotted against the percentage of the flux in the specimen beam passed by the attenuator. The height of each curve above this zero line, divided by the height of the "100 % line" above the zero line shall not deviate from the measured transmittance of the disk by more than 0.5 % at any wavelength.

NOTE 6—The sector-disk attenuator consists of a variable-speed motor, 0 to 4000 rpm, with an attenuator disk mounted on its shaft. The attenuator is normally operated at about 1300 rpm, and the direction of rotation is opposite to that of the chopper of the spectrophotometer. The disks are machined from sheet aluminum on a precision milling machine. The transmittance of each disk shall be determined by measuring to an accuracy of 0.1 % the width of each notch and blade along three circles corresponding to the positions of the two edges and centers of the beam at the position where the attenuator is used. The transmittance of the disk at each circle shall be computed as the total width of the notches divided by the sum of the total width of the notches and blades. The three measured transmittances shall agree to 0.15 %, and the transmittance of the disk shall be taken as the average of the three values.

7.4 To record the "100 % line," two blackbody furnaces, whose temperatures differ by less than 1 K, are placed in positions to act as sources for the reference and sample beams of the spectrophotometer. The spectrophotometer is then adjusted to scan the spectrum very slowly. The amplifier gains should be set to such a value that response and resolution can

TABLE 1 Sector-Disk Attenuators

Percent Attenuation	Disk Diameter, mm	No. of Notches	Width of Each Notch in Angular Degrees	Length of Each Notch from Rim, mm
75	229	8	33¾	64
50	229	8	22½	64
25	229	8	11¼	64
12.5	229	8	5⅝	64
5	254	4	4½	76

be adequately maintained. After recording the "100 % line" the chart is rerolled or the pen reset to the initial wavelength. The specimen beam is then blocked near the source and a "0 % line" similarly recorded over the same wavelength range, after which the chart paper is rerolled or pen reset to the initial wavelength. Extreme care should be taken to ensure that the initial recording points are on the same ordinate. The specimen is next substituted for the reference blackbody furnace, in such a position to act as a source for the specimen beam of the spectrophotometer. The temperature of the specimen is brought to and held at the temperature of the comparison blackbody furnace. An adequate "soak" or heating period should also be observed to assure that no thermal gradients exist. The specimen beam is unblocked, and the "specimen line" is recorded over the wavelength range of interest.

8. Calculation of Results

8.1 The heights of the respective curves are measured at preselected wavelengths and the normal spectral emittance is computed for each such wavelength. If Z_λ is the height of the "0 % line" (where all measurements are in arbitrary units), S_λ the height of the "specimen line," and H_λ the height of the "100 % line," at same wavelength λ, the normal spectral emittance, is given by:

$$\epsilon_{N\lambda} = (S_\lambda - Z_\lambda)/(H_\lambda - Z_\lambda) \quad (1)$$

Values of $\epsilon_{N\lambda}$ are computed for each preselected wavelength in the range from 1 to 35 μm, and $\epsilon_{N\lambda}$ plotted as a function of wavelength. A curve drawn through the plotted points represents the spectral normal emittance of the specimen.

9. Report

9.1 The spectral normal emittance of materials is influenced to various degrees by a wide variety of parameters which are incompletely understood. It is therefore necessary to specify as many specimen parameters as possible to increase the future value of the reported data. Those parameters of particular importance are the following:

9.1.1 *Intrinsic Properties*:

9.1.1.1 Index of refraction,

9.1.1.2 Scattering coefficient,

9.1.1.3 Absorption coefficient,

9.1.1.4 Direct-current electrical resistivity,

9.1.1.5 Particle size, shape, and distribution,

9.1.1.6 Composition, and

9.1.1.7 Density

9.1.2 *Surface Properties*:

9.1.2.1 Chemistry, and

9.1.2.2 Thickness of reaction-product films (for example, oxide layers).

9.1.3 *Specimen preparation*, such as thermal history and application techniques.

9.1.4 *Measurement temperature and atmosphere*.

9.1.5 *Description of optics geometry*, such that solid angles can be evaluated.

9.2 The test method described merely measures the spectral normal emittance. Test methods for the other parameters are not defined or are being formulated. When the other properties are indicated, and it is desirable to have most of them when reporting data, the test method used to obtain such properties should be indicated. For example, surface characteristics influence emittance strongly. An actual surface profile is needed for some emittance studies. Where there are no data or data such as "as received" are all that are available, arithmetic average (AA) values can provide some information; however, it should be pointed out that they have serious limitations for emittance studies. Surface contamination layers should be described along with their exact thicknesses. An adequate description of the thermal history of the specimens should be indicated since the intrinsic and surface properties may be functions of the thermal history as well as temperature. Temperature measurement methods must be described and temperature corrections indicated. The magnitude of the temperature difference between the specimen and reference should be indicated.

10. Precision and Bias

10.1 The precision and bias of the measurement method are based on the precision and bias of the spectral normal emittance determinations for the working standards (see Appendix). The precision and bias of the working standards vary with wavelength and temperature. The spectral normal emittance of these standards is given by Harrison **(3)** and Richmond **(10)** along with the spectral distribution of the standard deviations due to differences between specimens and due to random error of measurement. The present standards are accurate and the measurements repeatable to about 1 % in emittance units.

11. Keywords

11.1 emittance; infrared emittance; spectral normal emittance; radiative heat transfer; thermal radiation; material radiative property; spacecraft thermal control

APPENDIX

(Nonmandatory Information)

X1. STANDARDS FOR EMITTANCE MEASUREMENTS

X1.1 In order to standardize emittance measurements by different laboratories, working standards of thermal emittance having high, intermediate, and low emittance can be purchased in various sizes, and shapes as indicated below:

X1.1.1 *Strips*, 6 mm (¼ in.) by 200 mm (8 in.), 19 mm (¾ in.) by 254 mm (10 in.), 25 mm (1 in.) by 254 mm (10 in.) in size,

X1.1.2 *Squares*, 51 mm (2 in.) by 51 mm (2 in.) in size, and

X1.1.3 *Disks*, 13 mm (½ in.), 22 mm (⅞ in.), 25 mm (1 in.), 29 mm (1⅛ in.), and 32 mm (1¼ in.) in diameter.

X1.2 The following materials were selected on the basis of being stable on heating in air at temperatures up to 1400 K for times of several hundred hours:

X1.2.1 For standards of low spectral normal emittance, polished platinum-13 % rhodium.

X1.2.2 For standards of intermediate spectral normal emittance, oxidized Kanthal.

X1.2.3 For standards of high spectral normal emittance, oxidized Inconel.

X1.3 These standards are available from the Office of Standard Reference Materials, Room B311, Chemistry Building, at the National Institute of Standards and Technology, Gaithersburg, MD 20899.

X1.4 Extreme precautions should be taken to prevent contamination or damage to the surface of the standards during use. Handling should be kept to the absolute minimum. Clean surgical rubber gloves should be worn when handling specimens, in order to avoid fingerprints, and should be touched at the ends or edges only. The flat areas to be viewed should never be touched or permitted to come in contact with a bench or desk top. If a specimen must be laid down, it should be returned to its holder, or supported by the ends or edges only on clean glass or stainless steel. Particularly, care should be taken to avoid contamination by oil, grease, dust, or condensed volatilized materials.

X1.5 The standards were heated in air during calibration, and should be heated only in a clean air atmosphere at atmospheric pressure. The Kanthal and Inconel specimens have been oxidized in air, and heating in other atmospheres may significantly change the character of the oxide layer, and hence the emittance. While there is no visible oxide layer on the platinum standards, they were calibrated in air, and may change in emittance if heated in other atmospheres. When the standards are not in use they should be replaced in their individual containers and stored in a clean, dry place at room temperature.

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