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Standard Test Method for Calorimetric Determination of Hemispherical Emittance and the Ratio of Solar Absorptance to Hemispherical Emittance Using Solar Simulation¹

This standard is issued under the fixed designation E434; 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 measurement techniques for calorimetrically determining the ratio of solar absorptance to hemispherical emittance using a steady-state method, and for calorimetrically determining the total hemispherical emittance using a transient technique.

1.2 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

E349 Terminology Relating to Space Simulation

3. Summary of Test Method

3.1 In calorimetric measurements of the radiative properties of materials, the specimen under evaluation is placed in a vacuum environment under simulated solar radiation with cold surroundings. By observation of the thermal behavior of the specimen the thermophysical properties may be determined by an equation that relates heat balance considerations to measurable test parameters.

¹ This test method is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.04 on Space Simulation Test Methods.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2 In a typical measurement, to determine α/ϵ as defined in Definitions E349, the side of the specimen in question is exposed to a simulated solar source, through a port having suitable transmittance over the solar spectrum. This port, or window, must be of sufficient diameter that the specimen and radiation monitor will be fully irradiated and must be of sufficient thickness that it will maintain its strength without deformation under vacuum conditions. The radiant energy absorbed by the specimen from the solar source and emitted by the specimen to the surroundings cause the specimen to reach an equilibrium temperature that is dependent upon the α/ϵ ratio of its surface.

3.3 In the dynamic radiative method of measuring total hemispherical emittance, the specimen is heated with a solar simulation source and then allowed to cool by radiation to an evacuated space chamber with an inside effective emittance of unity. From a knowledge of the specific heat of the specimen as a function of temperature, the area of the test specimen, its mass, its cooling rate, and the temperature of the walls, its total hemispherical emittance may be calculated as a function of temperature.

4. Apparatus

4.1 The main elements of the apparatus include a vacuum system, a cold shroud within the vacuum chamber, instrumentation for temperature measurement, and a solar simulator.

4.2 The area of the thermal shroud shall not be less than 100 times the specimen area (controlled by the specimen size). The inner surfaces of the chamber shall have a high solar absorptance (not less than 0.96) and a total hemispherical emittance of at least 0.88 (painted with a suitable black paint),³ and shall be diffuse. Suitable insulated standoffs shall be provided for suspending the specimen. Thermocouple wires shall be connected to a vacuumtight fitting where the temperature of feedthrough is uniform. Outside of the chamber, all thermocouples shall connect with a fixed cold junction.

³ Nextel Brand Velvet Coating 401-C10 Black, available from Reflective Products Div., 3M Co., has been found to be satisfactory.

4.3 The chamber shall be evacuated to a pressure of 1×10^{-6} torr (0.1 mPa) or less at all times.

4.4 The walls of the inner shroud shall be in contact with coolant so that their temperature can be maintained uniform at all times.

4.5 A shutter shall be provided in one end of the chamber which can be opened to admit a beam of radiant energy from a solar simulator. When open, this shutter shall provide an aperture admitting the full simulator beam. When the shutter is closed, all rays emitted by the specimen shall be intercepted by a blackened surface at the coolant temperature (the shutter must be at least conductively coupled to the shroud).

4.6 The vacuum chamber shall be provided with a fused silica window large enough to admit the simulator beam and uniformly irradiate the entire specimen projected area. This window shall have high transmittance through the solar spectrum wavelength region. The chamber shall be provided with a vacuumtight sleeve for opening and closing the shutter and standard vacuum fittings for gaging, bleeding, leak testing, and pumping. If low α/ϵ specimens are to be measured, the solid angle subtended by the port from the specimen should be small (dependent upon desired accuracy). If flat specular specimens are to be measured, the port plane should be canted with respect to the specimen plane to eliminate multiple reflections of the simulator beam. Multiple reflections could result in as much as a 7 % apparent increase in α/ϵ .

4.7 The solar simulator should duplicate the extraterrestrial solar spectrum as closely as possible. A beam irradiance of at least 7000 W/m^2 at the specimen plane shall be available from the solar simulator (~ 5 solar constants). This irradiance may be required to raise the temperature of certain specimens to a desired level.

5. Coating Requirements

5.1 Any type of coating may be tested by this test method provided its structure remains stable in vacuum over the temperature range of interest.

5.2 For high emittance specimens the accuracy of the measurements is increased if only one surface of the substrate is coated with the specimen coating in question. The remaining area of the substrate shall be coated with a low emittance material of known hemispherical emittance (such as evaporated aluminum or evaporated gold).

5.3 The thickness and density of the coating shall be measured and its heat capacity calculated from existing references (see Refs (1) and (2)).⁴

6. Specimen Preparation

6.1 The substrates used for the measurements described here shall be of a material whose specific heat as a function of temperature can be found in standard references (for example, OFHC copper or a common aluminum alloy such as 6061-T6) (Ref (1)).

6.2 The substrate shall be machined from flat stock and to a size proportioned to the working area of the chamber.

6.3 Each specimen shall be drilled with a set of holes, near the edge, through which suspension strings are to be inserted.

6.4 Each substrate shall be drilled with two small shallow holes in the back for thermocouples.

6.5 Ideally the back and sides of the substrate shall be buffed and polished and one uninsulated thermocouple inserted in the back of the specimen (one wire in each hole). One of these wires shall be peened into each hole.

6.6 A low-emittance coating shall be applied to the back and sides of the substrate and to the thermocouple wires for several inches at the specimen end.

6.7 The substrates shall be coated with the material in question. The coating shall be of sufficient thickness so as to be opaque. (This will avoid any substrate effects.)

6.8 The specimens shall be suspended from the top of the shroud by means of thread or string. These strings shall be of small diameter, low thermal conductivity, and low emittance in order to minimize heat losses through the leads.

6.9 An alternative method of specimen mounting (mass dependent) shall be to suspend the specimens by their own small wire thermocouple leads. In this case the thermocouple holes shall be drilled as before but radially around the edge. The suspension holes may also be eliminated in this case.

7. Procedure

7.1 Suspend the test specimen in the chamber normal to the incident solar radiation, but geometrically removed from the central axis of the chamber so that radiation from the specimen to the chamber walls is not specularly reflected back to the specimen. Since the chamber walls are designed to be cold and highly absorbing, first reflections from the walls are usually all that need be considered.

7.2 Determine the simulated solar irradiance incident on the specimen with a suitable radiometric device such as a commercial thermopile radiometer or a black monitor sample of known α/ϵ which may be suspended similarly to the test specimen within the incident beam of simulated solar radiation. Take care in the latter case that the irradiance and spectral distribution of the incident energy is the same for both specimen and monitor.

7.3 Then close the system and start the evacuation and cooling of the shroud (see Ref (3) for a typical system). Maintain a pressure of 1×10^{-6} torr (0.1 mPa) or less and the walls of the chamber must be at coolant temperature. Record the specimen, monitor, and shroud temperatures.

7.4 When the specimen has reached thermal equilibrium, that is, when the specimen temperature becomes constant with constant surrounding conditions, shut off the solar simulator. When specimens of large thermal mass are used, carefully evaluate the $\Delta T/\Delta t = 0$ conditions, that is, the Δt chosen should be dependent on the specimen time constant.

7.5 Close the moveable door in the shroud and allow the specimens to cool to a desired temperature. Measure the

⁴ The boldface numbers in parentheses refer to the list of references appended to this method.

specimen temperature as a function of time and calculate the rates of change of the temperature.

8. Calculation

8.1 Calculate the $\alpha_{es}/\varepsilon (T_1)$ ratio from the following equation:

$$\frac{\alpha_{es}}{\varepsilon (T_1)} = \frac{A_t}{A_p E} \sigma \left(T_1^4 - \frac{\varepsilon (T_0)}{\varepsilon (T_1)} T_0^4 \right) \quad (1)$$

where:

- α_{es} = Effective solar absorptance relative to the illuminating source,
- $\varepsilon (T_0)$ = hemispherical emittance of the specimen at Temperature T_0 ,
- $\varepsilon (T_1)$ = hemispherical emittance of the specimen at Temperature T_1 ,
- σ = Stefan-Boltzmann constant,
- A_p = projected area of the specimen exposed to solar radiation,
- E = incident total irradiance,
- T_1 = specimen equilibrium temperature with simulated solar radiation,
- T_0 = chamber wall temperature with solar source off, and
- A_T = total radiating area of the specimen.

8.2 This equation is derived in the following manner: If a specimen coated on all sides with the material in question, with a projected area as viewed in the direction of irradiation, A_p , a total area, A_T , effective simulated solar absorptance, α_{es} , emittance at T_1 , $\varepsilon (T_1)$, and specific heat c_p is suspended in an evacuated high absorptance isothermal cold-walled chamber and exposed to a simulated solar irradiance, E , the rate of temperature change can be determined by evaluating the heat balance equation. The energy balance of an irradiated specimen emitting radiant energy in a vacuum is given by the following equation (assuming parasitic heat losses can be ignored):

$$m c_p \left(\frac{dT}{dt} \right) = A_p \sigma E_p + E_p - A_t \varepsilon (T_1) \sigma T_1^4 + A_t \alpha_{tr} (T_0) \sigma T_0^4 \quad (2)$$

where $E_p = A \varepsilon \sigma T_2^4$, the thermal radiation from the port. To determine the incident thermal radiation, E_p , see Ref (3). The last term, $A_t \alpha_{tr} (T_0) \sigma T_0^4$, is the amount of heat energy absorbed by the sample from the chamber walls. Kirchoff's law tells us that at a given temperature the infrared absorptance is equal the infrared emittance. This means that it will emit as much heat as it absorbs from a black body at the same temperature as the sample. Therefore, to know how much energy is absorbed by the sample from the shroud walls we must know the infrared absorptance (and hence the emittance) of the sample at the temperature of the shroud wall. The infrared absorptance at T_0 , $\alpha_{tr} (T_0)$, by Kirchoff's law is equal to the infrared emittance of the sample at that temperature so we can write:

$$m c_p \left(\frac{dT}{dt} \right) = A_p \sigma E_p + E_p - A_t \varepsilon (T_1) \sigma T_1^4 + A_t \varepsilon (T_0) \sigma T_0^4 \quad (3)$$

If E_p is eliminated from Eq 2 when an equilibrium temperature is reached, $m c_p (dT/dt) = 0$, and, From Eq 2, solving for the α/ε ratio we obtain

$$\frac{\alpha_{es}}{\varepsilon (T_1)} = \frac{A_t}{A_p E} \sigma \left(T_1^4 - \frac{\varepsilon (T_0)}{\varepsilon (T_1)} T_0^4 \right) \quad (4)$$

Eq 4 is used to calculate the $\alpha_{es}/\varepsilon (T_1)$ ratio when the parameters A_T , E , and A_p are determined and the equilibrium temperature is measured.

8.3 If the source is blocked by the shutter and the specimen loses energy only by radiation, the energy balance equation becomes:

$$m c \left(\frac{dT}{dt} \right) = A_t \varepsilon (T_1) \sigma T_1^4 - A_t \alpha_{tra} (T_0) \sigma T_0^4 + Q_{ll} + Q_{rg} - Q_{ts} \quad (5)$$

Where Q_{ll} and Q_{rg} represent the heat losses from the support leads and the heat lost from the residual gasses in the vacuum chamber, respectively. The last term Q_{ts} is any heat input from the temperature sensor. See Ref (4) and Ref (5) for a treatment of the lead loss and residual gas heat loss terms.

8.4 If the term T_0^4 is neglected, and the parasitic heat losses and gains can be ignored, the above equation can be integrated and expanded into:

$$\varepsilon (T_1) = \frac{(m_s c_s + m_c c_c)}{3 \sigma A_t \Delta t} \left(\frac{1}{T_1^3} - \frac{1}{T_2^3} \right) \quad (6)$$

where:

- m_s = mass of the substrate,
- m_c = mass of the coating,
- c_s = thermal capacitance of the substrate,
- c_c = thermal capacitance of the coating,
- T = temperature of the specimen, and
- Δt = change in time from T_1 to T_2 and magnitude such that c_s and c_c may be assumed constant over small temperature ranges.

When the temperature decay is recorded with time, then the total hemispherical emittance of the sample can be determined with Eq 5 or Eq 6. The use of Eq 6 is preferable since Eq 5 involves the experimental determination of two quantities (dT/dt and T^4), thereby introducing more possible errors than in Eq 6.

8.5 Data from specimens which are coated on one side only shall be reduced by use of the following equation:

$$\varepsilon (T)_c = \frac{(m_s c_s + m_c c_c)}{3 \sigma A_c \Delta t} \left(\frac{1}{T_1^3} - \frac{1}{T_2^3} \right) - \frac{\varepsilon_s (A_T - A_c)}{A_c} \quad (7)$$

where:

- ε_s = total hemispherical emittance of substrate,
- A_c = area of coating, and
- ε_c = total hemispherical emittance of coating.

8.6 To obtain an α/ε measurement or an effective solar absorptance, α , for a specimen coated only on one side, one must consider the following expression:

$$A_T \varepsilon_T = A_c \varepsilon_c + A_s \varepsilon_s \quad (8)$$

where:

- A_T , A_c , A_s = total area, area of the coating, and uncoated area of the substrate, respectively, and
- ε_T , ε_c , ε_s = total hemispherical emittance of the specimen, coating, and substrate respectively.

Rearrangement shows that:

$$\varepsilon_T = (A_c \varepsilon_c + A_s \varepsilon_s) / A_T \quad (9)$$

Multiplying the α/ε value obtained from Eq 4 by ε_T (at the same temperature of equilibrium) obtained from Eq 9 will give the solar absorptance, α . In order to acquire the (α/ε) coating, divide the α_s value by ε_c (already measured in a transient cool down).

9. Report

9.1 The report should include the methods used for temperature and irradiance measurements, and the actual data used for the calculations.

9.2 A complete characterization of the specimen shall be given whenever possible. This shall include specimen dimensions, specimen composition, coating thickness and composition, surface roughness, and surface contamination, and any other conditions which may be considered pertinent.

9.3 In an α/ε type of measurement, the total exposure time and level of irradiance, and spectral distribution of the incident flux shall also be reported.

10. Uncertainty Analysis

10.1 Many potential errors exist in the calorimetric determination of radiative properties. If it is assumed that the major uncertainties encountered in these calorimetric measurements are systematic rather than random, they will add in a linear manner and the total uncertainty can be expressed as:

$$\delta\varepsilon_s/\varepsilon_s = (\delta\varepsilon_s/\varepsilon_s)_{conv} + (\delta\varepsilon_s/\varepsilon_s)_q + (\delta\varepsilon_s/\varepsilon_s)_R + (\delta\varepsilon_s/\varepsilon_s)_{HL} \quad (10)$$

for emittance, and as

$$\frac{\delta\alpha/\varepsilon}{\alpha/\varepsilon} = \left(\frac{\delta\alpha/\varepsilon}{\alpha/\varepsilon} \right)_{conv} + \left(\frac{\delta\alpha/\varepsilon}{\alpha/\varepsilon} \right)_R + \left(\frac{\delta\alpha/\varepsilon}{\alpha/\varepsilon} \right)_{HL} + \left(\frac{\delta\alpha/\varepsilon}{\alpha/\varepsilon} \right)_s \quad (11)$$

for the ratio of solar absorptance to hemispherical emittance. The terms on the right of the emittance uncertainty equation can be defined as *conv* the conventional error, *q* the heat measurement error, *R* the extraneous radiation error, and *HL* the heat loss error, respectively. In the uncertainty equation for α/ε the last term, *s*, is defined as the error due to solar simulation where all fractional errors have been previously defined. These uncertainties are discussed in the following paragraphs.

10.2 *Conventional Error*—The conventional error contribution to the total uncertainty involves errors in the measurement of the basic physical quantities of a sample such as the area of the sample, the temperature of the sample, and the enclosure temperature. This error can be expressed as:

$$\left(\frac{\delta\varepsilon/\varepsilon}{\alpha/\varepsilon} \right)_{conv} = \left(\frac{\delta\varepsilon_s}{\varepsilon_s} \right)_{conv} = \frac{\delta A_T}{A_T} \quad (12)$$

$$+ 4 \left(\frac{T_1^4}{T_1^4 - T_0^4} \right) \frac{\delta T_1}{T_1} + 4 \left(\frac{T_0^4}{T_1^4 - T_0^4} \right) \frac{\delta T_0}{T_0}$$

In a given system, each of the quantities in Eq 10 is subject to a varying degree of accuracy; however, the most significant uncertainty occurs in measurement of specimen temperature. The magnitude of the uncertainties of specimen and enclosure temperatures can be determined when thermocouples with known calibrations are utilized. Thermocouples used in these

investigations should have a maximum deviation of ± 0.5 K and should read on the 1968 International temperature scale. From Eq 10 a total maximum uncertainty of calculated emittance values is about 2.0 %.

10.3 *Heat Measurement Error*—The error in determination of heat radiation by the test specimen in the enclosure walls includes the uncertainties in the measurement of Δt , specimen mass, and specific heat of the test specimen which can be expressed as follows:

$$(\delta\varepsilon_s/\varepsilon_s)_q = (\delta m/m) + (\delta c_p/c_p) + (\delta\Delta t/\Delta t) \quad (13)$$

10.3.1 To determine the uncertainty of specimen mass, it is necessary to know the balance manufacturers stated uncertainty. A precision balance from any reputable manufacturer will yield a purely negligible error (on the order of ± 0.003 % for any metallic substrate). The uncertainties of specific heat values as published in the technical literature are dependent upon the material of the substrate; stated values of specific heat are known to a higher degree of accuracy for elemental materials than for most alloys. The published values of c_p for the substrates suggested for use in these investigations are known to within ± 1 %. These uncertainties in c_p can be considered as maximum and result in like uncertainties in calculated values of emittance. For emittance measurements of coatings having an unknown c_p , an elemental substrate should be used. It is evident that the thermal mass of the coating shall not be a significant percentage of the thermal mass of the substrate (less than 2 %). This procedure will minimize the error due to the uncertainty of the thermal capacitance of the coating. For the method described here, the uncertainty in the measurement of time intervals, Δt , is on the order of ± 0.3 s. The maximum uncertainty involved would occur for high ε materials (greater than 0.93). This results in a maximum uncertainty of approximately 3.5 % for calculated values of emittance. (This occurs at relatively high temperatures.)

10.4 *Extraneous Radiation*—Extraneous radiation depends to a great extent on the chamber geometry and the position of the specimen within the chamber. Several possible sources of extraneous radiation that could exist in these measurements are:

- (1) Thermal radiation from the chamber walls,
- (2) Thermal radiation from the port and through the port from the ambient environment,
- (3) Thermal radiation from the specimen reflected back to the specimen from the chamber walls,
- (4) Solar source radiation reflected by the chamber walls onto the specimen,
- (5) Solar source radiation reflected from the specimen to the chamber walls or port and back to the specimen, and
- (6) Radiation from pumps and other heat sources internal to the system which may be viewed by the specimen.

The design of an apparatus for measurement of radiative properties by the calorimetric method must minimize the errors associated with reflection by the enclosure walls of the specimen-emitted energy and by the radiation of energy from the enclosure walls. This minimization of errors is accomplished by making the ratio of the wall area to the specimen area as large as practical, by coating the enclosure walls with