

## Standard Practice for Combined, Simulated Space Environment Testing of Thermal Control Materials with Electromagnetic and Particulate Radiation<sup>1</sup>

This standard is issued under the fixed designation E512; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

#### INTRODUCTION

Spacecraft thermal control coatings may be affected by exposure to the space environment to the extent that their radiative properties change and the coatings no longer control temperatures within desired limits. For some coatings, this degradation of properties occurs rapidly; others may take a long time to degrade. For the latter materials, accelerated testing is required to permit approximate determination of their properties for extended flights. The complexity of the degradation phenomena and the inability to characterize materials in terms of purity and atomic or molecular defects make laboratory exposures necessary.

It is recognized that there are various techniques of investigation that can be used in space environment testing. These range in complexity from exposure to ultraviolet radiation in the wavelength range from 50 to 400 nm, with properties measured before and after testing, to combined environmental testing using both particle and electromagnetic radiation and in situ measurements of radiative properties. Although flight testing of thermal control coatings is preferred, ground-based simulations, which use reliable test methods, are necessary for materials development. These various approaches to testing must be considered with respect to the design requirements, mission space environment, and cost.

## 1. Scope

- 1.1 This practice describes procedures for providing exposure of thermal control materials to a simulated space environment comprising the major features of vacuum, electromagnetic radiation, charged particle radiation, and temperature control.
- 1.2 Broad recommendations relating to spectral reflectance measurements are made.
- 1.3 Test parameters and other information that should be reported as an aid in interpreting test results are delineated.
- 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:<sup>2</sup>

**E275** Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

E296 Practice for Ionization Gage Application to Space

E349 Terminology Relating to Space Simulation

E434 Test Method for Calorimetric Determination of Hemispherical Emittance and the Ratio of Solar Absorptance to Hemispherical Emittance Using Solar Simulation

E490 Standard Solar Constant and Zero Air Mass Solar Spectral Irradiance Tables

E491 Practice for Solar Simulation for Thermal Balance Testing of Spacecraft

E903 Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres

#### 3. Terminology

3.1 Definitions:

<sup>&</sup>lt;sup>1</sup> This practice 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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<sup>&</sup>lt;sup>2</sup> 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.

- 3.1.1 *absorbed dose*—the amount of energy transferred from ionizing radiation to a unit mass of irradiated material.
- 3.1.2 *absorbed dose versus depth*—the profile of absorbed energy versus depth into material.
- 3.1.3 *bleaching*—the decrease in absorption of materials following irradiation because of a reversal of the damage processes. This results in a reflectance greater than that of the initially damaged material. Also referred to as *annealing*.
- 3.1.4 equivalent ultraviolet sun (EUVS)—the ratio of the solar simulation source energy to a near ultraviolet sun for the same wavelength region of 200 to 400 nm.
- 3.1.5 far ultraviolet (FUV)—the wavelength range from 10 to 200 nm. Also referred to as vacuum ultraviolet or extreme ultraviolet.
- 3.1.6 far ultraviolet sun—the spectral and energy content of the sun in the wavelength range from 10 to 200 nm. The spectrum is characterized by a continuum spectrum to approximately 160 nm and a line spectrum to 10 nm. The solar energy in the FUV fluctuates and for purposes of irradiation of thermal control coatings, the UV sun is defined as  $0.1 \text{ W/m}^2$  for the wavelength range from 10 to 200 nm (see Tables E490) at 1 AU (astronomical unit)  $(1.495 988 2 \times 10^{11} \text{ m})$  (1).<sup>3</sup>
- 3.1.7 *in situ*—within the vacuum environment. It may be used to describe measurements performed during irradiation as well as those performed before and after irradiation.
- 3.1.8 *integral flux*—the total number of particles impinged on a unit area surface for the duration of a test, determined by integrating the incident particle's flux over time. Also referred to as *fluence*.
- 3.1.9 irradiance at a point on a surface—the quotient of the radiant flux incident on an element of the surface containing the point, by the area of that element. Symbol:  $E_e$ , E;  $E_e^I = \mathrm{d}\phi_e/\mathrm{d}A$ ; Unit: watt per square metre, W/m<sup>2</sup>. (See Terminology E349.)
- 3.1.10 *near ultraviolet*—the wavelength range from 200 to 400 nm.
- 3.1.11 *near ultraviolet sun*—for test purposes only, the solar irradiance, at normal incidence, on a surface in free space at a distance of 1 AU from the sun in the wavelength band from 200 to 400 nm. Using the standard solar-spectral irradiance, the value is 8.73 % of the solar constant or 118 W/m<sup>2</sup> (see Terminology E349). This definition does not imply that any spectral distribution of energy in this wavelength band is satisfactory for testing materials.
- 3.1.12 *particle flux density*—the number of charged particles incident on a surface per unit area per unit time.
- 3.1.13 *reciprocity*—a term implying that effect of radiation is only a function of absorbed dose and is independent of dose rate.
- 3.1.14 *solar absorptance* ( $\alpha_s$ )—the fraction of total solar irradiation that is absorbed by a surface. Use the recommended spectral-solar irradiance data contained in Tables E490.
- <sup>3</sup> The boldface numbers in parentheses refer to the list of references at the end of this practice.

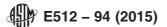
- 3.1.15 *solar constant*—the solar irradiance, at normal incidence, on a surface in free space at the earth's mean distance from the sum of 1 AU. The value is  $1353 \pm 21$  W/m<sup>2</sup> (see Tables E490).
- 3.1.16 *synergistic*—relating to the cooperative action of two or more independent causal agents such that their combined effect is different than the sum of the effect caused by the individual agents.
- 3.1.17 thermal emittance ( $\varepsilon$ )—the ratio of the thermal-radiant exitance (flux per unit area) of the radiator (specimen) to that of a full radiator (blackbody) at the same temperature.

## 4. Summary of Practice

- 4.1 The most typical approach in performing this test is to measure the radiative properties of the specimen under consideration, then to place the specimen in a vacuum chamber and expose it to the desirable simulated space environments. The specimen temperature is controlled during the period of exposure. The radiative property measurements are performed in situ without exposing the specimen to atmospheric pressure, after exposure and before measurement. Unless it has been established that the material under investigation is not affected by postexposure measurements, the in situ approach is the preferred method. Usually only the radiative property of solar absorptance,  $\alpha_s$ , is of interest, and the net result of the test is a measurement of change in solar absorptance,  $\Delta \alpha_s$ . For detailed discussions of methods of determining radiative properties, see Test Method E903 and Refs. (2), (3), and (4).
- 4.2 The most effective method is to combine the radiation components of the space environments and investigate the synergistic effects on radiative properties of the thermal control materials.

#### 5. Specimen Analysis

- 5.1 A method characterizing the behavior of thermal control materials during space environment exposure is through spectral reflectance measurements. The two parameters of engineering importance are total solar absorptance ( $\alpha_s$ ) and total hemispherical emittance ( $\epsilon_h$ ). Solar absorptance is generally determined from spectral reflectance measured under conditions of near normal irradiation and hemispherical viewing over the wavelength range from 0.25 to 2.5  $\mu$ m. For these measurements, an integrating sphere with associated spectrophotometer is commonly used. For reflectance measurements beyond 2.5  $\mu$ m, a blackbody cavity or parabolic reflectometer is frequently used.
  - 5.2 Postexposure Measurements:
- 5.2.1 Although in situ measurements are necessary, many measurements must be performed after removal of the specimen from the test chamber. The accuracy of such measurements should be verified by in situ measurements because of possible bleaching.
- 5.2.2 Postexposure measurements of properties should be accomplished as soon as possible after the exposure. Where delays allow the possibility of bleaching, it is necessary to minimize atmospheric effects by maintaining the specimens in the dark and in vacuum until measured. In the event that



evacuation is impractical, it is desirable that the specimens be maintained under a positive pressure of dry argon. Note that bleaching by diffusion of oxygen or nitrogen into the system has been observed to occur in the dark, although more slowly, than in the light.

## 5.3 In Situ Analysis:

- 5.3.1 Calorimetric measurements of thermal-radiative properties have received some attention in connection with in situ studies of thermal-radiative property changes. A calorimetric determination gives a direct measure of  $\alpha_s/\epsilon$  and therefore indicates the in situ changes in thermal-radiative properties. If edoes not change, then the change in  $\alpha_s/\epsilon$  shows the change in  $\alpha_s$ . If the electromagnetic radiation source provides a good match to the air-mass zero solar-spectral irradiance, then a will be equal to  $\alpha_s$ . The limiting factors in calorimetric  $\alpha_s/\epsilon$  determinations are the deviation of the spectral irradiance produced by the simulated solar source from that of the solar irradiance and the accuracy of the irradiance measurement (see Test Method E434).
- 5.3.2 In situ measurements allow the determination of the reflectance or absorptance in a vacuum environment. The environment maintained for in situ measurements should have no effect on the property being measured. The annealing of the specimen after irradiation may occur sufficiently fast to make the posttest measurements misleading. In situ reflectance measurements allow the investigator to plot a curve of the change in thermal radiative properties as a function of the exposure or absorbed dose. Posttest measurements limit the data to one point at the total dose.

#### 5.4 Physical Property Analysis:

- 5.4.1 The complete evaluation of thermal control coatings does not depend only on thermal-radiative property measurements; coatings must have the adhesion and stability required for retention on a specified substrate. One method used to evaluate the ability of the coating to remain firmly attached to the substrate in space is through thermal cycling of the specimens either during or after radiation exposure in a vacuum.
- 5.4.2 The loss of mass of thermal control coatings can be measured, to provide an indication of the amount of decomposition products leaving the coating during exposure. This may be important in the study of the curing, outgassing, and contamination potential of thermal control coatings.
- 5.4.3 Vacuum gas analysis (mass spectroscopy or residual gas analysis, RGA) can be used to assess the type and concentration of decomposition products.
- 5.5 Surface Analysis of Specimens—X-ray photoelectron specotroscopy (XPS), auger electron spectroscopy, and secondary ion mass spectrometry (SIMS) are some techniques that can be used to determine the composition of materials on the surface of the specimens. This information can then be used to identify any contamination that may be present on the specimens
- 5.6 Auxiliary Methods of Specimen Analysis—Several other techniques for specimen characterization and analysis are available to the investigator. As a rule, these are usually used in studies of damage mechanisms rather than engineering tests.

They are included in Table 1 to give a more complete account of methods for analysis of thermal control surfaces damaged by electromagnetic or particle irradiation, or both.

#### SIMULATION SYSTEM

## 6. Vacuum System

- 6.1 General Description—The vacuum system shall consist of the specimen test chamber, all other components of the simulation system that are joined to the chamber without vacuum isolation during specimen exposure, and the transition sections by which these components are joined to the chamber. The vacuum system must perform the following functions:
- 6.1.1 It must provide for a reduction of pressure of atmospheric gases in the test chamber to a level in which none of the constituents can react with the specimen material to affect the validity of the tests. This provision implies a pressure no greater than  $1 \times 10^{-6}$  torr (133 µPa) at the specimen position.
- 6.1.2 It must provide that the specimen area be maintained as free as possible from contaminant gases and vapors. These gases and vapors may originate anywhere in the system including from the test specimens themselves.
- 6.1.3 It must promptly trap or remove any volatiles outgassed from the test specimens.
- 6.1.4 It must provide for accurate pressure measurements in the chamber. (See Practice E296.)

#### 6.2 Test Chamber:

- 6.2.1 *Construction*—The specimen test chamber should be constructed of materials suitable for use in ultra-high vacuum. Metals, glasses, and ceramics are used. Tables E490 contain information on materials for vacuum applications. Austenitic-stainless steels, such as Type 304, are frequently used for vacuum-chamber construction.
- 6.2.1.1 Welding and brazing should be performed in accordance with good high-vacuum practice and the temperature requirements of the chamber. Materials to be joined must be properly cleaned so that sound, leaktight, nonporous joints can be made. Inert gas arc welding (TIG), using helium or argon, and electron beam welding have been used. Brazing materials and cleaning techniques are discussed in Refs (5) and (6). Welds should be on the vacuum side to eliminate the possibility of trapping gas in cracks and crevices, thus creating a virtual leak. Parts must be absolutely clean before welding. An oil film can cause gas to evolve and result in a porous, leaky weld.
- 6.2.1.2 Dimensions of the test chamber should be sufficiently large in relation to those of the specimen holder, so that contaminants outgassed from any of the specimens cannot be reflected back from windows or walls to the surface of other specimens.
- 6.2.1.3 The chambers should contain a cryogenic shroud, or be of an insulated double-wall (annular) construction, to provide for reducing wall temperature by the use of coolant fluids. The walls should preferably be cooled with liquid nitrogen during all tests. This feature is particularly essential if there are condensable contaminants in the test chamber arising from any part of the system or from the specimens. The temperature of the wall should always be lower than that of the test specimens to reduce the probability of contaminants

# TABLE 1 Potential Techniques Used for Specimen Analysis in Ground-Based Simulated-Solar Ultraviolet Studies on Thermal-Control Coatings

Note 1—Bidirectional reflectance is influenced by the changes in geometrical distribution of the reflected energy, as well as the change in spectral reflectance. Extreme care must be used in interpreting results for degradation evaluation.

Measurement Techniques	Laboratory Equipment	Properties Investigated <sup>A</sup>	Materials <sup>B,C</sup>
General sample analysis:			
Spectral reflectance measurements (pre- and	integrating sphere, Hohlraum, Coblentz hemisphere	α, ε	P, B, P/B
post-test)			
In-situ analysis:			
Calorimetric	vacuum, cryogenic apparatus	α, ε	P, B, P/B
Spectral	bidirectional reflectance, integrating sphere		P, B, P/B
Physical property analysis:			
Thermal cycling-mass loss (pre- and post-test	thermal cycling, apparatus, radiation exposure	flexibility, adhesion (qualitative), weight	B, P/B
and in situ)	apparatus		
Auxiliary methods of analysis:	radiation exposure apparatus or simple thermal vacuum with or without radiation		P, B, P/B
X-ray diffraction		a, b, c	
X-ray powder diffraction		a, c, e	
X-ray fluorescence		a	
Electron microscopy		b, c, g, h	
Conventional microscopy		g, h	
Metallography		c, d, e, f, h	
Particle size analyzers		g	
Particle counter analyzers		g	
Gas absorption		f, g	
Porosimeter		f, g	
Spectrograph		a	
Extensometer		i	
Profilometers		d	
Density measurement		f	
Thermal conductance		р	
Resistance measurements		j	
Electron paramagnetic resonance		k, m	
Photoconductivity		d	
Seebeck and Hall coefficients		1	
Stoichiometry		n, o	
Oxidation-reduction capacity		n	
Optical absorption		n	
Low-energy electron diffraction		d	
X-ray photoelectron spectroscopy		q	
Auger electron spectroscopy		q	
Secondary ion mass spectroscopy		q	

A a= purity
b = crystal lattice
c = physical structure
d = surface structure
e = phases
f = void volume
B P = pigment

B = binder

g = particle size
h = particle shape
i = coefficient of expansion
j = electrical resistivity
k = free radicals

I = excess carriers

m = trapped unpaired electrons or holesn = defect centers per unit volume

n = defect centers per unit vo = chemical structurep = thermal conductivityq = surface analysis

P/B = pigment/binder
<sup>C</sup> The laboratory equipment used and the types of materials investigated vary considerably and therefore will not be discussed in detail in this table.

preferentially condensing on the specimens. The use of a residual gas analyzer to measure the partial pressures of gases and vapors in the system may prove of use in interpreting the results of the tests.

6.2.1.4 The test chamber construction should also provide for bakeout to a temperature of at least 150°C and preferably to 400°C. Bakeout should be conducted before installing the test specimen for each test. Adequate bakeout can be accomplished in a shorter time at higher temperatures.

6.2.2 Chamber Pumping System—The pumping capacity of the test-chamber pumping system, including the cold wall, must be adequate not only for chamber evacuation, but to

handle outgassing loads from specimen materials and gases or vapors entering the chamber from other system components.

6.2.2.1 The pumping system should be selected or designed to maintain the test-specimen contamination at levels below those which would affect the test results.

6.2.2.2 Ion pumping, sometimes accompanied by sublimation pumping, is frequently used for optical-degradation studies of thermal control materials. This combination provides ease of operation for long-time periods with minimal attention. Other advantages of these types of pumps are that they can be baked without damage, and they do not require cryogenic baffles. Possible disadvantages of these pumps are their low

capacity for noble gases and their slow response to pressure surges. However, newer versions of ion pumps have increased their capacities for noble gases.

- 6.2.2.3 Sputter-ion pumps cause stray magnetic fields that may interfere with tests using low-energy protons or electrons. The orbitron and diffusion-type pumps do not present this problem. Hydrocarbons tend to build up in ion pumps when the high voltage is turned off, particularly if the system has recently been exposed to air. Under normal pump operating conditions, the hydrocarbon buildup is either minimal or does not occur. The emission of previously trapped gases may occur when the pump is started or operated at above-normal pressures. The electronic pumps may have a lesser capability of operating under pulsed-gas loads than do diffusion pumps.
- 6.2.2.4 Since the basic pumping mechanism of the orbitron is one of titanium sublimation, it has a disadvantage, in common with ordinary titanium-sublimation pumps, in that new sources of titanium must be frequently provided.
- 6.2.2.5 Care must be taken in the design of chambers using ion pumps and titanium sublimation pumps. Sublimed and sputtered material must be kept from the specimen area. Specimens must also be protected from electromagnetic radiation generated by the discharge in the ion pumps especially upon ignition. The intensity of the startup discharges depends upon the pressure to which the system was rough pumped, and roughing to approximately 10<sup>-3</sup> torr (13 mPa) or lower is recommended. Rough pumping can be accomplished by either sorption pumps, mechanical pumps, or a combination of both.
- 6.2.2.6 When mechanical pumps are used, proper equipment and procedures are required to minimize the backstreaming of oil into the chamber. Dry nitrogen may be used to maintain the roughing pressure in the viscous-flow regime if sorption pumps are used for the final rough pumping. Properly sized molecular sieves or cold traps should be used if roughing pressures are below approximately 1 torr (133 Pa).
- 6.2.2.7 Oil- and mercury-diffusion pumps may be used if their construction and operation provide reduction of backstreaming of pump fluids to levels below which affect test results. Reduction of backstreaming may be accomplished by using optically dense, anticreep traps or baffles that are cryogenically cooled, or both. A closed-cycle refrigeration system may be advantageous from the standpoint of extended-test periods and cost of operation. Thermoelectrically cooled baffles may also be used. Silicone, polyphenyl ether, or other low-vapor pressure fluids are recommended for use as pump fluids because of their stability and lower backstreaming rates. The advantages of diffusion-pumped systems are the ability to pump all common gases well and the ability to handle pulsed gas loads.
- 6.2.2.8 Cryopumps or turbomolecular pumps may also be used in simulation systems and are the preferred pumping systems of many. These pumps do not use pumping fluids and they pump all common gases well. Startup and shutdown procedures are critical, as with other types of pumps. Cryopumps have an advantage in that they pump water incredibly well. Sublimation pumps can be used in conjunction with diffusion and turbomolecular pumps to handle large gas loads and provide selective pumping.

6.2.3 Demountable Seals—Many standard materials used to seal openings in walls or at flanges in vacuum systems are a major source of contamination. Metal-to-metal demountable seals are recommended whenever they are feasible in a system. Where metal-to-metal seals are not practical, as when a part of the system must be electrically isolated, organic materials may be used, but the type should be carefully selected. Fluoroelastomers, fluorocarbons, and polyimides have been used.<sup>4</sup> The design must provide for protection if organic seals from electromagnetic or particulate radiations are used. It is recommended that organic seals be vacuum baked at 250°C before installation to remove volatile materials. If a system is to be baked at temperatures in excess of 150°C, means should be provided to prevent excessive heating of the seals. No vacuum grease should be used on the seals or any other parts of the system.

## 6.3 Auxiliary Simulation Components:

- 6.3.1 Certain components of the simulation system, which operate at pressures that are high in relation to that of the test chamber, may have to be attached to the chamber without complete vacuum isolation. Particle accelerators are generally in this category. Basic pressures of accelerators are usually in the 0.5 to  $5\times 10^{-6}$ -torr (167- to 665-µPa) range with operating pressures of 0.1 to  $1.5\times 10^{-5}$  torr (133 to 2000 µPa), particularly for positive-ion accelerators. Vacuum isolation, even with thin foils, is not feasible, except for higher-energy particles, and even then this leads to energy straggling.
- 6.3.2 Commercial accelerators and other components may provide sources of contamination through the use of elastomer seals or by virtue of a poorly designed vacuum system. Replacement of inadequate seals and modifications of the vacuum system are recommended when feasible.

#### 6.4 Transition Sections:

6.4.1 The flow rate of gases and vapors from auxiliary components into the test chamber must be reduced to a minimum. This is usually accomplished by means of transition sections that limit the "leak" rate solely by conductance limiting and differential pumping. This latter method usually consists of mounting a vacuum-pumped section in the transition line between the offending component and the test chamber and limiting the gas conductance from the component into the pumped section and from the pumped section into the chamber. The differential-pumping method is recommended because transition sections can be substantially shorter to produce the same reduction in "leak" rate. This not only conserves space, but if small diameter metal tubing is used for conductance limiting from particle accelerators and the tubing is at ground potential, the resultant beam spreading for lowenergy charged particles may be a problem.

6.4.2 All comments in 6.2 pertinent to vacuum techniques apply to the transition sections. Use of in-line cryogenic traps in transition sections are advantageous in pumping condensable vapors, but are of little value in removing gases such as

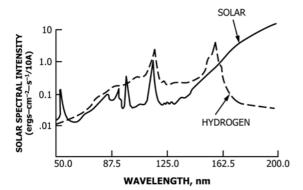
<sup>&</sup>lt;sup>4</sup> Viton-A, available from E.I. Dupont de Nemours and Co., Inc. has been found satisfactory for this purpose.

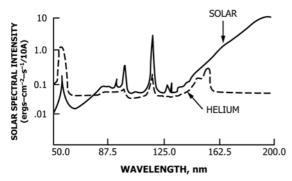
hydrogen from proton accelerators or ultraviolet-gaseous discharge sources. The effects of relatively high partial pressures of such gases on test results have not been evaluated.

#### 7. Solar Simulation

- 7.1 Radiation Above 200 nm—There are several radiation sources that can be used as ultraviolet energy sources for thermal control coatings evaluation work. The source should duplicate the spectral irradiance of the extraterrestrial sun as closely as possible (see Tables E490) even though uncertainties in measurements as large as 10 % may exist below 250 nm. Common sources that have been used extensively by investigators are the xenon-arc lamps, but mercury-arc, mercury-xenon-arc, and carbon-arc lamps have also been used. Generally, there is agreement among investigators that filtered xenon arc lamps provide the best source. The filtering is necessary to remove the excess amounts of heat that these lamps generate.
  - 7.1.1 Xenon Arc Lamp:
- 7.1.1.1 The xenon arc lamp should be manufactured with a UV-grade, high-purity fused-silica envelope with a transmission of at least 70 % at 0.20  $\mu m.$
- 7.1.1.2 The lamp output shall be monitored periodically within each individual lamp life to determine nominal total irradiance changes. Lamp irradiance will vary as much as 35 % in total radiant flux over the life of the lamp. Therefore, the irradiance must be monitored and controlled by varying the source-to-specimen distance or increasing the operating current within the lamp manufacturer's specified limits, or both.
- 7.1.2 Filtering Techniques—There are a couple of recommended methods of filtering the infrared (IR) radiation to closely match the zero air mass solar spectrum. Common absorption filters are not generally suitable for these xenon arc lamps because of the high energy densities usually associated with the higher wattage lamps. Thus, other filtering options are usually used.
- 7.1.2.1 One filtering option is a water filter. The water filter consists of a tube with UV-grade fused quartz windows on either end which has filtered and softened water circulating through it. This filter greatly reduces the IR content of the beam.
- 7.1.2.2 Another filtering method is through the use of dichroic filters. These filters are designed to reflect only UV and visible light while transmitting the IR. Dichroic mirrors are available with different wavelength characteristics.
- 7.1.3 Lamp Power Supply—The lamp operates on highly regulated dc power supply, which are regulated by either current, voltage, power, or optional output power control. A timer is incorporated in the power supply output circuit to record the lamp's operating time.
- 7.1.4 *Useful Life of Sources*—The criteria for changing lamps should involve a consideration of ultraviolet irradiance rather than total irradiance. Note that for xenon short arc lamps, the UV output of the lamp decreases more rapidly than that of the rest of the lamp's spectrum.
- $^{5}\,\mbox{General}$  Electric A-H6 and B-H6 lamps have also been found satisfactory for this purpose.

- 7.1.5 Radiation Detector—The total irradiance at the specimen can be measured with a National Institute of Standards and Technology (NIST) traceable, calibrated total radiation detector. The filters should be periodically recalibrated. For calibration of ultraviolet detectors in the near ultraviolet, refer to Practice E275.
- 7.1.6 Spectral Irradiance Measurement—The relative spectral distribution of the source can be determined accurately with the use of a UV spectroradiometer and a NIST traceable, calibrated source. Spectral measurements are useful for the determination of the degree to which ultraviolet sources degrade with time.
- 7.2 Radiation Below 200 nm—There are several types of sources that provide FUV radiation. For simulating the solar FUV radiation to irradiate thermal control coatings, hydrogen or deuterium lamps and capillary-type windowless-discharge sources are used.
- 7.2.1 Hydrogen or deuterium lamps are readily available from commercial sources. They generally use  $MgF_2$  windows, but other materials are available. They generate approximately 0.14 W/m<sup>2</sup> at a distance of 10 cm, and their operating life is on the order of 500 h.
- 7.2.2 Typical capillary-type windowless-discharge sources are the Hinterrigger and Tanaka sources. These sources operate windowless to the vacuum system. They each can handle power inputs of approximately 1 kW. These capillary-type discharge sources are commercially available.
- 7.2.2.1 *Source Power Supply*—The sources operate on either an ac- or dc-regulated power supply, with line-voltage fluctuation controlled.
- 7.2.2.2 Discharge Gases—Hydrogen or helium gas is used in simulating the solar radiation in the wavelength range from 200 to 90 nm and 50 to 160 nm, respectively. A typical scan of the hydrogen and helium spectrum obtained with a suitable capillary-type discharge source is shown in Fig. 1 compared to the solar specimen (7). Gases used in the discharge can be used directly from the commercial pressurized bottles without any special purity requirements.
  - 7.2.3 FUV Radiation Detectors:
- 7.2.3.1 *General*—There are a number of detector types that are sensitive to vacuum and extreme ultraviolet radiation. The most acceptable type is the photomultiplier detector. This type of detector must be operated windowless to the vacuum system or be provided with a phosphor coating on the face of the photomultiplier detector.
- 7.2.3.2 Coatings—The phosphor coating deposited over the window of the photomultiplier functions as a light transformer, which is excited by the vacuum and extreme ultraviolet and emits longer wavelengths that can be measured with the photomultiplier. Sodium salicylate is considered an ideal phosphor for use in measuring irradiances in the ultraviolet region because its constant quantum efficiency is essentially independent of wavelength in the vacuum and extreme ultraviolet.
- 7.2.3.3 *Calibration*—The detectors are calibrated in the ultraviolet to the wavelength limit of approximately 185 nm, and this value is used to extrapolate the energy measurement into the vacuum and extreme ultraviolet.





Note 1—The sunspot maximum curve is smoothed over most spectral lines. Flare radiation is not shown.

FIG. 1 Comparison of UV Sources and Solar Ultraviolet Spectrum at 1 AU

#### 8. Charged Particle Sources

#### 8.1 Particle Accelerators:

8.1.1 Particle accelerators are the most commonly used means of simulating the space-charged particle environment (8). Van de Graaff or other types of accelerators can be used to produce energetic protons or electrons in various energy ranges from about 0.1 MeV to several MeV. Other types of accelerators that are commercially available use resonant transformers or various transformer-rectifier systems to produce the high voltages required for particle acceleration. These types of power supplies are used to accelerate particles up to the 100-kV energy range and higher. Electron guns are also available. It is also possible for the investigator to build his own system. Ion sources and other components are readily available.

8.1.2 Some form of mass separation must generally be used with accelerators of positive ions, particularly protons. The proton content of the ion beams may vary from 30 to 90 % for different accelerators, depending primarily upon the type of ion source used. Different ion species of the same energy produce different degrees of degradation in the sample materials. The relative damage as a result of the different species is a function of ion energy and probably of the sample material. The energy of the charged particles must be well known to yield useful information.

8.2 Radioactive Sources—Radioactive beta-particle sources present another means for simulation of the space electron environment. A wide selection of radioactive isotopes is available. These can be combined or used individually to provide simulation in various energy ranges. This method of simulation has the advantage of affording a reasonable fit to the electron spectra of space, but lacks the capability provided by accelerators of obtaining electron damage data as a function of discrete energies. Spectra, geometric, and intensity monitoring of the electrons must be provided at specimen position.

#### 8.3 Charged-Particle Flux Density Determination:

8.3.1 Several methods are available for irradiating required sample areas with accelerator-produced charged particle beams. If the beam area is at least as large as the area to be irradiated, the specimen need only be placed in the beam path. If an area larger than the beam is to be irradiated, such as frequently occurs for multiple-specimen irradiations, the beam can be scanned or rastered across the required area by electrostatic or magnetic techniques. Scattered-beam techniques can also be applied. Scattering of molecular hydrogen ions through thin foils serves as a way of obtaining protons over large areas.

8.3.2 Particle fluxes are usually determined for accelerator produced beams by measuring a beam current incident upon a known area. Several methods are available for making these measurements. Faraday cups may be used to measure the beam current before testing or continuously or intermittently during a test. Solid-state detectors used with a multichannel analyzer can provide data on both flux density and energy profile when used at the specimen position. When measurements are made directly of the current striking the target, a collimator may be mounted on insulators in front of the target holder to define the irradiated area. In measurements made by the latter method, care must be taken to prevent secondary electrons from either the collimator or the specimen and the specimen holder from interfering with the current measurement. This can be accomplished by inserting a negatively biased electron suppressor between the collimator and specimen holder to force the secondaries back to their respective sources. Alternatively, if the specimen holder is essentially at infinite impedance with respect to ground, the collimator and specimen holder may be individually biased to restrain their own secondaries.

8.3.3 The problem of charging the surface of the insulating specimen materials can also present a problem, since it results in repulsion of the incident beam, arcing, and nonuniform specimen exposure. Accurate, direct specimen-current measurements are not possible under these conditions. One method of alleviating this problem is to place a conducting grid across the specimen surface. Five or six parallel strands of 0.0254-mm wire spaced across the surface of a 25-mm diameter specimen is usually adequate and results in masking less than 1 % of the area. If this method is used, care should be taken in orienting the specimen when reflectance measurements are made so that the masked (unirradiated) areas do not interfere with the measurement.

## 9. Specimen Thermal Control

- 9.1 *General*—Specimen temperature control can be maintained in one of two ways: the specimen can be insulated from its surroundings or the specimen can be thermally "connected" to a sink.
- 9.2 Insulated Specimens—These specimens come to temperature equilibrium with their environment. If this method of thermal control is chosen, the specimen temperature is a function of the following: lamp irradiance (which may change with time), the chamber wall temperature, and the spectral characteristics of the specimen.

## 9.3 Connected Specimens:

- 9.3.1 The other method of thermal control is to establish good thermal contact between the specimen and the specimen support. The specimen support is maintained at a specific temperature within the prescribed limits. The specimen table can be maintained at a temperature greater than room temperature by using a heater. When temperatures below room temperature are desired, the table can be cooled by circulating fluids or gases.
- 9.3.2 The assurance of good thermal contact between the specimen and the specimen support is critically important. If an aluminum substrate is clamped to a stainless steel (or steel) support table with an average contact pressure of approximately 345 kPa (50 psi), good thermal contact is developed. A control specimen, with a thermocouple attached, is recommended for use in assuring that the desired temperatures are maintained in vacuum during irradiation.
- 9.3.3 Another way of developing good thermal contact is to bond the specimen to the substrate with a low-vapor pressure conductive adhesive. If an adhesive is used, it must be thoroughly cured and must not be exposed to the irradiation components. Tests should be performed to determine the adequacy of any adhesives used.
- 9.3.4 An example of a heat-sink type of specimentemperature control apparatus consists of the following:
- 9.3.4.1 Specimen Holder—The specimen holder should be capable of supporting and thermally isolating the specimen from the exposure chamber and adjacent specimens. It is desirable for the holder to provide for cooling or heating the specimen. Typical specimens are 25 mm (1 in.) in diameter and from 0.8 to 3.2 mm (½2 to ½ in.) in thickness. The body of the holder is constructed of oxygen-free high-conductivity (OFHC) grade copper and has dimensions of approximately 35 mm (13/8 in.) in diameter and 9.5 mm (3/8 in.) thick with a 26-mm (1.025-in.) diameter counterbore about 1.6 mm (½16 in.) deep.
- 9.3.4.2 Temperature Control—Two 6-mm (1/4-in.) diameter austenitic stainless steel (Type 304 has been found satisfactory) tubes are brazed to the back of the holder. The tubes are aligned with a passageway drilled into the holder to permit temperature control of the holder. The holder is supported and positioned by the stainless steel tubing for the desired irradiation level. The tubing is brazed to a vacuum flange and protrudes through the flange to permit external control of the flow rate or the type of heating or cooling required. Both liquid and gaseous methods can be used. When operating at cryogenic temperatures, vacuum-jacketed feedthroughs are required.

- 9.3.4.3 Temperature Measurement—Specimen temperature is measured with a suitable thermocouple. The thermocouple is attached to the specimen holder by spot-welding or peening when metallic substrates are used. Specimens exposed with dielectric substrates have at least a 0.080-mm (40-gage) thermocouple imbedded in the material. Preferably, the thermocouple is located 0.25 mm (0.010 in.) or less from the exposed surface. A suitable potentiometer is used for measurement of the thermocouple emf.
- 9.3.4.4 *Specimen Attachment*—The specimen is attached to the holder by spring clips or other suitable means, 0.812-mm (20-gage) piano wire or beryllium-copper tabs have been successfully used.

#### 10. Safety Precautions

- 10.1 Particle Accelerators:
- 10.1.1 Both positive-ion and electron accelerators can present a personal hazard because of X-ray generation. With electron accelerators, a source of X-rays exists wherever any of the primary beam is intercepted. With positive-ion accelerators, the X-rays arise from secondary electrons, which are accelerated toward the high-voltage terminal, and strike in the area of the ion source. Personnel must be made aware of this hazard and adequate protective measures must be taken.
- 10.1.2 High-energy electron accelerators can present an additional hazard from the primary particles themselves and protective measures must be taken in regard to the design of the system.
- 10.2 Radioactive Sources—Radioactive beta sources present hazards similar to those of electron accelerators. In addition, these sources may contaminate the test chamber if they are not adequately sealed.
- 10.3 Solar Simulators—Safety precautions must be taken against several hazards which exist with solar simulators. The gas pressure in the arc lamps is high, particularly when in operation, and danger always exists of the bulb exploding. Protective covering must be worn over all parts of the body when working with the lamps. The ultraviolet radiation can cause severe skin burns and serious eye damage in minutes unless protection is worn. The ultraviolet radiation will ionize the air surrounding the simulator and produce ozone, which must be exhausted from the area. If a tube containing mercury is broken or explodes, the mercury presents a potential hazard. Practice E491 reviews arc-lamp safety precautions.
- 10.4 *High Voltages*—Much of the equipment in a combined simulation system operates with dangerously high voltages against which personnel must be guarded.
- 10.5 *Vacuum System*—Two potential hazards exist in vacuum systems, as follows:
- 10.5.1 Mercury-diffusion pump systems must be properly designed and operated to prevent the escape of mercury vapor into the room. Spillage of mercury, and vapor escape during component cleaning and repair, represent hazards. For additional, more recent information on decontamination, see Ref (9).
- 10.5.2 Possible explosion hazards that exist from operating oil-diffusion pumps in conjunction with solar simulators are discussed in Ref (10).

#### 11. Contamination Control

- 11.1 Contamination of test specimens during testing must be minimized to prevent erroneous results. Contamination can be reduced so that it has a negligible contribution to the test results.
- 11.2 Organic contaminants are of particular importance in the testing of thermal control materials. Typical sources of organic contaminants (which are discussed below) include the following:
- (a) Residues on the specimen surface before installation in the test chamber,
- (b) Outgassing from materials that are part of the vacuum chamber and pumping system,
- (c) Contaminants remaining in the chamber from previous tests,
  - (d) Cross contamination from other test specimens, and
  - (e) Contaminants from the radiation sources.
- 11.3 Residues on the Specimen Surface—Protection of the specimens before installation and adequate cleaning techniques are required to eliminate this type of contaminant. The cleaning procedures that are used must be compatible with the type of specimen. In addition, cleaning solvents may have high nonvolatile residue levels, because of their initial formulation or storage in containers such as polyethylene, which contain plasticizers or other soluble components. Suitable container materials are glass, TFE-fluorocarbon, and corrosion-resistant metals.
- 11.4 *Test Chamber and Vacuum System*—Section 6.2 discusses many aspects of the materials used in test chambers and vacuum systems, as well as the types of vacuum pumps.
- 11.5 Cross Contamination—Specimen holders should be designed so as to minimize the transfer of contaminants from one specimen to another. Baffles, maintained at temperatures below those of the specimens, can be used to prevent line-of-sight transfer. Other factors to be considered include surface migration and reflection from warm surfaces, such as windows, within the chamber.
- 11.6 Radiation Sources—Radiation sources that are within the chamber vacuum are potential sources of contaminants. Radiation sources may have materials that outgas or use vacuum systems that produce organic contaminants. Where appropriate, windows can be used. Another approach that can minimize contamination is the use of differential vacuum pumping and cryogenically cooled surfaces when a windowless connection is required between the source and the test chamber.

## 11.7 Contamination Monitoring:

11.7.1 The probability for contamination always exists. Therefore, it is necessary to determine if contamination has occurred. Contamination monitors can be passive or active types. A passive device collects contaminants but does not measure the contaminant or the effect of the contaminant. An example of a passive monitor is a witness plate. Following a test, the witness plate is removed and measured (for reflectance, transmittance, and mass change). An active monitor measures some property. Examples of active monitors are

QCMs (quartz crystal microbalances) that measure changes in mass and Lyman- $\alpha$  reflectometer. The Lyman- $\alpha$  reflectometer measures the changes in reflectance of a front-surface mirror using light at the wavelength of 121.6 nm.

11.7.2 For thermal control system testing, it is convenient to use passive contamination monitors. The monitors are placed in the test chamber and are measured following the test and at least one monitor should be exposed to the radiation environment. Therefore, the monitors should be made of materials that show little or no change when exposed to the test-chamber irradiation. Fused-silica, second-surface mirrors, using silver or aluminum, and aluminized front-surface mirrors are suitable. The monitors exposed to the radiation will defect contaminants that form as a result of radiation induced polymerization on the surface. The monitors should be maintained at the same temperature as the test specimens. Active monitors should be considered when in situ, real-time measurements are required. A further check on contamination is to clean the monitor or thermal control coating surface following the test to determine if either reflectance or bulk-property changes are caused by surface deposition. The cleaning procedure should be one that will not affect the coating or monitor in any way.

## 12. Interpretation of Results

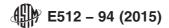
- 12.1 *Reciprocity*—Reciprocity, as used in this section, implies that the effect of radiation is only a function of absorbed dose and is independent of dose rate.
- 12.1.1 Testing methods using reciprocity as an acceleration factor have been documented by theoretical and experimental evidence. Experience has shown the upper limit for accelerated UV (both NUV and FUV) testing to be no more than three times the intensity of the sun (three EUVS) in those spectral regions. For particulate radiation, there is more room to use reciprocity testing. Investigators frequently use acceleration factors of 100 or more times the expected on orbit fluence. In either case, it is recommended that each coating system be investigated for dose-rate effects if long-term performance is to be predicted from short-term exposures. Various exposure techniques and data for many materials are reported in Refs (3), (4), and (11).
- 12.1.2 Other than the intrinsic properties of the material, the factors that may give rise to an apparent nonreciprocity effect include flux measurement, spatial and temporal uniformity of flux over specimen surface, solar spectral matching, specimen temperature, vacuum level, source constancy, and so forth. Practically, the ability to control and reproduce test environments and parameters may be as important as a demonstrable intrinsic rate effect, in terms of reciprocity correlation. At very high acceleration, nonuniform irradiance can become a problem. This nonuniformity may cause at least two problems: one associated with the average or "effective" irradiance at the specimen; and the other, with the different rate responses across the specimen. The damage developed by the lower irradiance may differ appreciably from that developed by the higher irradiance. Considering the size of the port openings of the usual spectral reflectance instruments, these differences in reflectance spectra depend upon the relative amount of highirradiance area versus low irradiance area seen by the specimen-viewing port.

- 12.2 The influence of operating temperature will vary with material. Organic materials may show significant differences in degradation rate with an 8°C (15°F) difference in temperature change. Inorganic constituents generally show negligible differences with temperature changes of this magnitude. Most materials exhibit increases in solar absorptance ( $\alpha_s$ ) with temperature when irradiated with simulated solar ultraviolet radiation and particulate radiation. Therefore, it is recommended that the temperature should be known and controlled to simulate the projected mission.
- 12.3 Solar spectral mismatch may produce "nonsimulating" effects for at least the two following reasons:
- 12.3.1 If the quantum efficiency for exciting defects, which are important in causing  $\Delta a_s$ , is wavelength-dependent then a departure from the solar UV spectrum may result in a different concentration of such defects.
- 12.3.2 If the induced absorption corresponds to the annihilation of this defect (bleaching), then their concentration will be dependent upon the ratio of UV to the total energy intensities. Thermal processes usually assist the removal of these defects; so there is a rather high probability of a rate effect which is temperature dependent.

#### 13. Report

- 13.1 To permit interpretation of the results of combined environmental tests, record and report at least the following data:
  - 13.1.1 *Vacuum:*
- 13.1.1.1 Description of test chamber including type of pumping.
- 13.1.1.2 Description of other components and elements comprising the total vacuum system,
- 13.1.1.3 Description of chamber preparation including cleaning method and temperature and time of bakeout.
  - 13.1.1.4 Chamber wall temperature,
- 13.1.1.5 Chamber pressure before initiation of test including the method of measurement.
- 13.1.1.6 Chamber pressures during test (if different than at initiation), and
  - 13.1.1.7 General remarks.
  - 13.1.2 Sample Temperature:
  - 13.1.2.1 Temperature level,

- 13.1.2.2 How maintained,
- 13.1.2.3 Method of measurement, and
- 13.1.2.4 General remarks.
- 13.1.3 Charged Particle Radiation:
- 13.1.3.1 Source of particles,
- 13.1.3.2 Particle energy or energy spectrum at sample position,
- 13.1.3.3 Flux density including uncertainty and how determined,
  - 13.1.3.4 Integral flux including uncertainty,
  - 13.1.3.5 Uniformity,
- 13.1.3.6 Absorbed dose and absorbed dose versus depth appropriate,
  - 13.1.3.7 Angle of incidence, and
  - 13.1.3.8 General remarks.
  - 13.1.4 Electromagnetic Radiation:
  - 13.1.4.1 Type of source used,
- 13.1.4.2 Spectral irradiance at specimen position including method of measurement and changes with time,
- 13.1.4.3 Irradiance at specimen position including spectral range of measurement and method of measurement,
  - 13.1.4.4 Monitoring during test and method used,
  - 13.1.4.5 Integrated exposure (including uncertainty),
- 13.1.4.6 Absorbed doses and absorbed dose versus depth, as required, and
  - 13.1.4.7 General remarks.
  - 13.1.5 Test Chamber Gas Analysis:
  - 13.1.5.1 Method used.
  - 13.1.6 Optical Property Measurement:
  - 13.1.6.1 Type of measurement and spectral range,
  - 13.1.6.2 Change in spectral property,
  - 13.1.6.3 Change in total property, and
- 13.1.6.4 Measurement conditions including description of apparatus and temperature of specimen during measurement.
  - 13.1.7 Specimen Description:
  - 13.1.7.1 Material type, characteristics, and source,
- 13.1.7.2 Physical properties (thickness, density, and so forth)
  - 13.1.7.3 Number of specimens per test,
  - 13.1.7.4 Size of specimens, and
  - 13.1.7.5 Distribution of specimens in chamber.



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