



Designation: E 2141 – 04<sup>2</sup>

An American National Standard

## Standard Test Methods for Assessing the Durability of Absorptive Electrochromic Coatings on Sealed Insulating Glass Units<sup>1</sup>

This standard is issued under the fixed designation E 2141; 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 The tests described are methods for the accelerated aging and monitoring of the time-dependent performance of

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<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E06 on Performance of Building Constructions and is the direct responsibility of Subcommittee E06.22 on Durability of Building Constructions.

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electrochromic windows. Cross sections of typical electrochromic windows are shown<sup>2</sup> in which devices have four or five-layers of coatings that include the two or three active layers sandwiched between transparent conducting electrodes (TCEs, see Section 3).

1.2 The test methods are applicable only for multilayered (two or more coatings between the TCEs) absorptive electrochromic coatings on sealed insulating glass (IG) units fabricated for vision glass (superstrate and substrate) areas for use in buildings, such as sliding doors, windows, skylights, and exterior wall systems. The multilayers used for electrochromically changing the optical properties may be inorganic or organic materials between the superstrate and substrate.

1.3 The electrochromic coatings used in this test method are exposed to solar radiation and are deployed to control the amount of radiation by absorption and reflection and thus, limit the solar heat gain and amount of solar radiation that is transmitted into the building.

1.4 The test methods are not applicable to other chromogenic devices, e.g., photochromic and thermochromic devices.

1.5 The test methods are not applicable to electrochromic devices consisting of three layers of coatings including the two transparent conducting electrodes (see Section 3).

1.6 The test methods are not applicable to electrochromic windows that are constructed from superstrate or substrate materials other than glass.

1.7 The test methods referenced herein are laboratory tests conducted under specified conditions. These tests are intended to simulate and, in some cases, to also accelerate actual in-service use of the electrochromic windows. Results from these tests cannot be used to predict the performance with time of in-service units unless actual corresponding in-service tests have been conducted and appropriate analyses have been conducted to show how performance can be predicted from the accelerated aging tests.

1.8 The values stated in metric (SI) units are to be regarded as the standard.

1.9 *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:

C 168 Terminology Relating to Building Materials<sup>3</sup>

C 1199 Test Method for Measuring the Steady State Thermal Transmittance of Fenestration Systems Using Hot Box Methods<sup>3</sup>

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process<sup>4</sup>

E 632 Practice to Aid Prediction of Service Life of Building Components and Materials<sup>3</sup>

E 773 Test Method for Accelerated Weathering of Sealed Insulating Glass Units<sup>3</sup>

E 774 Specification for the Classification of the Durability of Sealed Insulating Glass Units<sup>3</sup>

E 891 Tables for Terrestrial Direct Normal Solar Spectral Irradiance for Air Mass 1.5<sup>3</sup>

E 892 Tables for Terrestrial Solar Spectral Irradiance at Air Mass 1.5 for a 37° Tilted Surface<sup>3</sup>

E 903 Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres<sup>3</sup>

E 1423 Practice for Determining the Steady State Thermal Transmittance of Fenestration Systems<sup>3</sup>

E 1887 Test Method for Fog Determination<sup>3</sup>

E 2094 Practice for Evaluating the Service Lifetime of Chromogenic Glazings<sup>3</sup>

G 113 Terminology Relating to Natural and Artificial Weathering Tests of Non-Metallic Materials<sup>3</sup>

G 151 Practice for Exposing Nonmetallic Materials in accelerated Test Devices that Use Laboratory Light Source<sup>3</sup>

G 155 Practice for Operating Xenon Arc Light Apparatus for Exposure of Non-Metallic Materials<sup>3</sup>

### 2.2 Canadian Standards:

CAN/CGSB12.8 Insulating Glass Units

## 3. Terminology

3.1 *Definitions*—Refer to Terminology in C 168, E 632, and G 113 for descriptions of general terms.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *accelerated aging test*—an aging test in which the rate of degradation of building components or materials is intentionally accelerated from that expected in actual service.

3.2.2 *bleached state*—a descriptor for an ECW when no ions reside in the electrochromic layer or after ions have been removed (or inserted, depending on the type of material) from the electrochromic layer(s) and if applicable, the maximum number of ions have been returned to the counterelectrode layer to restore the  $\tau_b$  from that of the photopic optical specular transmittance in the colored state ( $\tau_c$ ).

<sup>2</sup> A. W. Czanderna and C. M. Lampert, "Evaluation Criteria and Test Methods for Electrochromic Windows," SERI/PR-255-3537 (July 1990), Golden, CO: Solar Energy Research Institute.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 04.07.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

3.2.3 *colored state*—a descriptor for an ECW after ions have been inserted (or removed, depending on the type of material) into the electrochromic layer and, if applicable, removed from the counterelectrode layer to reduce the photopic optical specular transmittance (of wavelengths from 400 nm to 730 nm) from that in the bleached state ( $\tau_b$ ).

3.2.4 *durability*—the capability of maintaining the serviceability of a product, component, assembly or construction over a specified time.

3.2.5 *electrochromic coating*—the multilayered materials that include the electrochromic layers, other layers, and transparent conducting oxide layers required for altering the optical properties of the coating.

3.2.6 *electrochromic layer(s)*—the material(s) in an ECW that alter its optical properties in response to the insertion or removal of ions, e.g.,  $\text{Li}^+$  or  $\text{H}^+$ .

3.2.7 *electrochromic window (ECW)*—a window consisting of several layers of electrochromic and attendant materials, which are able to alter their optical properties in response to a change in an applied electric field. The changeable optical properties include transmittance, reflectance, and absorptance.

3.2.8 *ion conducting layer*—the material in an ECW through which ions are transported between the electrochromic layer and the ion storage layer and electron transport is minimized.

3.2.9 *ion storage layer or counter electrode layer*—the material in an ECW that serves as a reservoir for ions that can be inserted into the electrochromic layer.

3.2.10 *performance parameters*—the photopic transmittance ratio (PTR), of at least 5:1 ( $\text{PTR} = \tau_b/\tau_c$ ) between the bleached (e.g.,  $\tau_b$  of 60 % to 70 %) and colored (e.g.,  $\tau_c$  of 12 % to 14 %) states; coloring and bleaching times of a few minutes; switching with applied voltages from ~1-3 V; and open-circuit memory of a few hours, e.g., contemporary ECWs typically have open circuit memories of 6 to 24 h.

3.2.11 *serviceability*—the capability of a building product, component, assembly or construction to perform the function(s) for which it was designed and constructed.

3.2.12 *service life (of a building component or material)*—the period of time after installation during which all properties exceed minimum acceptable values when routinely maintained.

#### 4. Significance and Use

4.1 The test methods are intended to provide a means for evaluating the durability of ECWs as described in 1.2.<sup>1,2,5</sup> (See Appendix X1).

#### 5. Background

5.1 Observations and measurements have shown that some of the performance parameters of ECWs have a tendency to deteriorate over time. In selecting the materials, device design, and glazing for any application, the ability of the glazing to perform over time is an indication of that glazing's durability. The ability of the product to perform over time, at or better than specified requirements, is an indication of the service life of the glazings. While these two indicators are related, the purpose of this standard test method is to assess the durability of ECWs.

5.2 ECWs perform a number of important functions in a building envelope including: minimizing the solar energy heat gain; providing for passive solar energy gain; controlling a variable visual connection with the outside world; enhancing human comfort (heat gain), security, ventilation, illumination, and glare control; providing for architectural expression, and (possibly) improving acoustical performance. Some of these functions may deteriorate in performance over time. Solar heat gain through an ECW is decreased because of two principal processes. Energy from the visible part of the spectrum is absorbed by an ECW in the colored state. In addition, infrared radiation is either absorbed by the ECW materials or is reflected by the transparent conducting oxide layers that are used for applying the coloring or bleaching potentials across the other layers in the ECW.

5.3 It is possible, but difficult to predict the time-dependent performance of ECWs from accelerated aging tests because of the reasons listed below. Users of this document should be aware of these limitations when reviewing published performance results and their connection to durability.

5.3.1 The degradation mechanisms of ECW materials and/or glazings are complex. In some cases, however, these mechanisms may be determined and quantified.

5.3.2 The external factors that affect the performance of ECWs are numerous and may be difficult to quantify. However, in some cases, the use, the environmental factors, and other information that influence performance may be known.

5.3.3 Fenestration units with tested ECWs may be different from those planned for use in service. Some companies have a database of in-service performance that can be compared to laboratory results.

5.4 Degradation factors (or stresses) for ECWs include the ion insertion and removal processes; temperature; solar radiation (especially UV); water vapor; atmospheric gases and pollutants; thermal stresses such as shock from sudden rain, as well as during the diurnal and annual temperature cycles; electrochemically induced stresses in the multilayer thin-film device; hail, dust, and

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<sup>5</sup> A. W. Czanderna, D. K. Benson, G. J. Jorgensen, J-G. Zhang, C. E. Tracy, and S. K. Deb, "Durability Issues and Service Lifetime Prediction of Electrochromic Windows for Buildings Applications," NREL/TP-510-22702, May 1997, National Renewable Energy Laboratory, Golden, CO; Solar Energy Materials and Solar Cells, 56 (1999) pp. 419-436.

wind; condensation and evaporation of water; and thermal expansion mismatches<sup>2,5</sup>. These factors may singularly or collectively limit the stability and durability of ECWs. Because the ECWs are expected to have the multilayer of coatings on one of the surfaces in the air space of double-pane or triple-pane IG units with an inert gas fill in the sealed space, many factors such as high humidity, atmospheric gases and pollutants, condensation and evaporation of water, and dust should not affect the durability of electrochromic coatings in IG units<sup>2</sup>.

5.4.1 Accelerated durability testing parameters that can be used to predict the long-term stability of the performance of ECWs are not presently accepted<sup>2</sup>. However, a rationale was recently proposed for narrowing the number of degradation factors that need to be studied. Establishing the testing criteria

5.4.1 Establishing test procedures from which ECW durability can be predicted and validated based on in-service for in-service use is an extremely crucial element for the commercialization of ECWs, even for niche markets. To reduce the number of accelerated test parameters that are required to predict the long-term performance of ECWs, accepted procedures or methods have not been established for testing ECWs.<sup>2</sup> Because no uniformly accepted procedures or methods have been established for the real-time testing of ECWs and because manufacturers and users cannot wait 20 or more years for the real-time evaluation of each window design, accelerated life testing (ALT) methods and procedures must be used for evaluating ECW stability<sup>2,5</sup>. These include (a) rapid but realistic current-voltage (I-V) cyclic tests emphasizing the electrical properties, (b) ALT parameters that are typically used in durability tests by standards organizations, (c) ALT parameters that are realistic for the intended use of large-area ECWs, and (d) how the ALT results must be related to real-time testing<sup>2</sup>. The purpose of this test method is to assess the durability of ECWs (at least  $305 \pm 6 \text{ mm} \times 305 \text{ mm}$ ; 12-in.  $250 \pm 6 \text{ mm}$  (10  $\pm$  1/4 in.  $\times$  12-0  $\pm$  1/4 in.).

NOTE 1—Caution—The seals in IGUs may fail at lower temperatures than those planned for testing, that is, 70 to 105°C. A seal failure will virtually guarantee failure of the ECW coating, so no assessment of the ECW coating will be made if a seal fails during a test.

NOTE 2—the test method may also be used for smaller ECWs to assess the durability of prototype devices. The testing parameters chosen only provide modest acceleration factors. However, the quantitative parameters discussed in (a)–(c) above are presented and include a detailed description of the procedures for using an accelerated weathering unit (AWU) (See Practice E 122).

**6. Apparatus (See Figs. 1 and 2 and Section 8.3 for Descriptive Detail)**

6.1 Accelerated Weathering Unit (AWU) consisting of properly filtered xenon-arc lamps to simulate over the applicable degradative wavelength region (UV plus visible) at least one-sun of solar irradiance at AM 1.5 (global) at sea level (E 892), a controlled temperature chamber, and humidity control in the chamber.

6.2 Voltage Cycling Unit for imposing voltage cycles to alternately and repeatedly color and bleach the ECWs from a fully bleached state to the colored state and back to the bleached state.

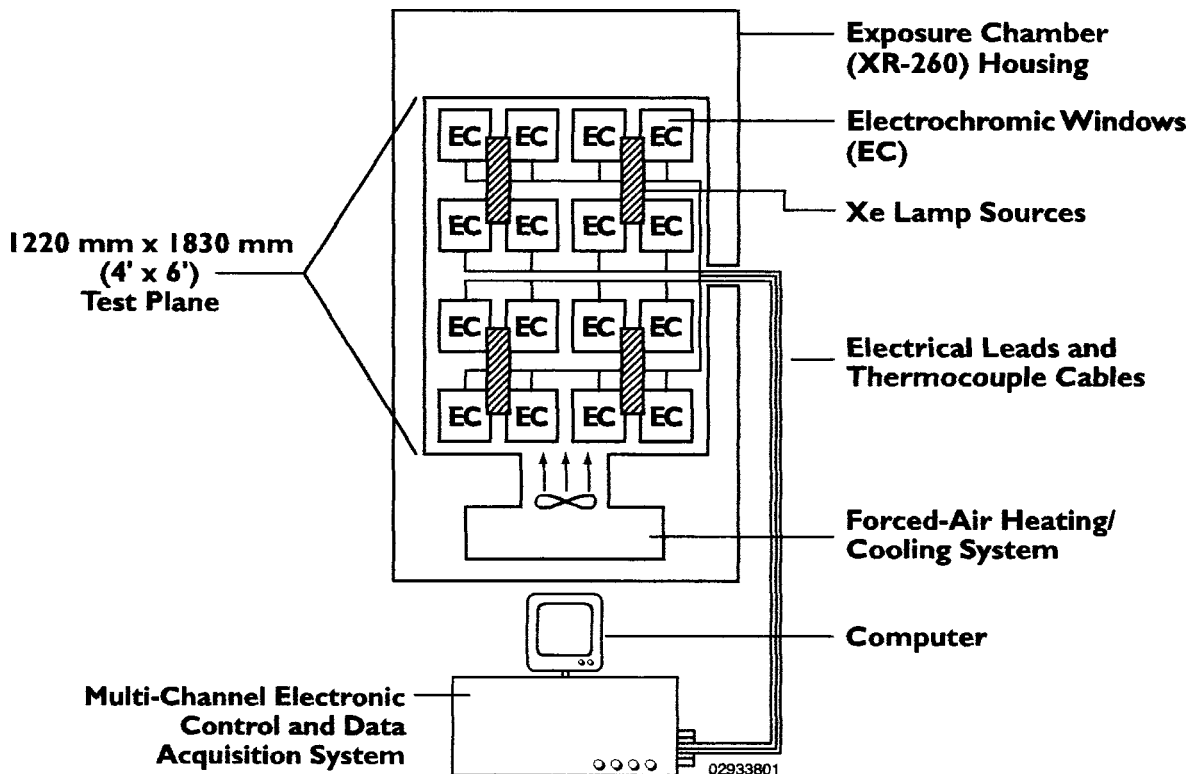
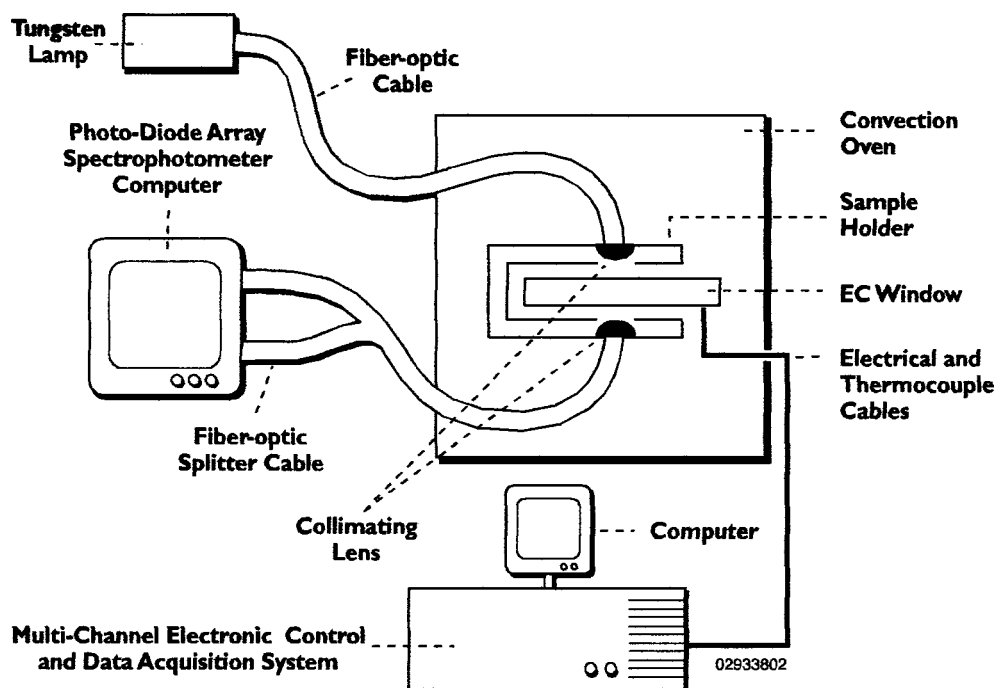


FIG. 1 Top-View Schematic Diagram of the Essential Components of an Environmental Test Chamber and Computer-Controlled Electrical Cycling and Data Acquisition System for Accelerated Weathering of Electrochromic Window Devices



Note: The measurements are used to determine the photopic transmittance ratio and record electro-optic degradation data after cyclic testing in the accelerated weathering unit.

FIG. 2 Schematic of the (Essential) Elements of the Electro-Optic Measurement System Used for Recording 300–1100 Nm Transmittance Spectra for a Color/Bleach Cycle of EC Window Devices at Controlled Temperatures

6.3 *Computer Controlled Photodiode Array Spectrophotometer* e.g., for obtaining and storing data from the electro-optical characterization of the optical transmittance in the colored and bleached state and measuring the rate of coloring and bleaching.

6.4 *Oven* that is large enough for the largest ECW to be tested and that can reach the ECW testing temperature. The oven must also be designed to permit using the equipment in 6.3 for optical measurements while the ECW is maintained at the temperature chosen for testing in the AWU described in 6.1.

6.5 *Tungsten Lamp*. A spectrum from the source must be compatible with the fiber optic illumination of the photodiode array spectrophotometer described in 6.3.

6.6 *Digital Camera*.

6.7 *Video Camera and Recorder*.

6.8 *Calibrated Thermocouples*.

6.9 *Electrical Leads* from the Unit in 6.2 to each ECW in the AWU described in 6.1.

## 7. Test Specimens

7.1 Test specimen size, design, and construction shall be established and specified by the user of this standard, except the specimens shall be at least  $250 \pm 6 \text{ mm} \times 250 \pm 6 \text{ mm}$  ( $10 \pm 1/4 \text{ in.} \times 10 \pm 1/4 \text{ in.}$ ).

7.2 Six to 10 test specimens that are represented to be “identical” shall be the minimum number used to assess the durability of a particular design and construction.<sup>4</sup> (See Item 2 in Appendix X1).

## 8. Procedure<sup>6</sup>

8.1 *Overview*. Expose the ECWs to simulated solar irradiance in a temperature- and humidity-controlled chamber at selected sample temperatures ranging from 70°C (158°F) to 105°C (221°F) while the ECWs are cyclically colored and bleached with the ability to pause during the duty cycles, depending on the control strategy prescribed by the manufacturer. The “testing” temperature shall be that in the colored state; the average ECW temperature will be less because of a typical decrease of about 5°C (9°F) when the ECW is in the bleached state. Accept the prevailing relative humidity in the chamber (e.g., 5 % to 20 %) because the prototype multilayer coatings will be sealed inside double-pane or triple-pane IG units for in-service use. Measure transmittances in a manner analogous to that described in E 903.

8.2 *Electro-optical Characterization of ECWs* is accomplished by using a computer-controlled, multichannel potentiostat and a photodiode array spectrophotometer. The optical transmittance of all ECWs is initially measured at room temperature (ca. 22°C),

<sup>6</sup> The procedure is based in part on the paper by A. Czanderna, et al., in “Optical Materials Technology for Energy Efficiency and Solar Energy Conversion XV,” C. M. Lampert, C. Granqvist, M. Grätzel, and S. K. Deb, eds., SPIE Vol. 3138, 68 (1997).

as shown schematically in Fig. 2. The fiber optic cables are routed from the tungsten lamp source into the ECW sample holder. The convection oven shown in Fig. 2 is simply allowed to equilibrate with room temperature for measurements at ca. 22°C (72°F). The temperature of the ECW is monitored by a thermocouple (or other appropriate surface temperature probe or device) attached to the device surface facing the xenon-arc lamps with a highly reflective tape (e.g., (for example, aluminum or silver) having an emissivity close to that of glass. One optical fiber guides the incident light from the tungsten lamp to one side of the sample; another optical fiber guides the transmitted light to the photodiode array spectrometer attached to a computer. The fibers shall be optically coupled by properly aligned collimating lens assemblies attached to both the illuminating and the collection fibers. Reference spectra for 100 % and 0 % transmittance are taken before each measurement. The magnitudes of the coloring and bleaching voltages (typically <3 volts), as specified by the ECW manufacturer, are then applied. To minimize degradation caused by large current surges that occur at the beginning of the coloring or bleaching process, a trapezoidal voltage (ramp rate – 0.05 V/s) instead of a step voltage may be used. A typical voltage (V) waveform and the corresponding current (i) are plotted in Fig. 3 as a function of time. The optical transmittance of the sample is measured over an appropriate spectral range in successive intervals during the coloring and bleaching processes. The time interval between the recorded spectra can be as small as one second. In typical testing experiments, a time interval of a fraction of the total cycle time for taking each spectrum should be adequate for recording the optical properties of each ECW, e.g., for  $t_{\text{cycle}} = t_c + t_b$ , spectra taken at time intervals between  $t_{\text{cycle}}/20$  to  $t_{\text{cycle}}/60$  will probably be adequate. Typical transmittance spectra recorded during a coloring and bleaching cycle are shown in Fig. 4, in which the optical spectra of the devices are plotted as a function of wavelength. The time constants used in the voltage profile are determined by monitoring the time to reach an optical PTR ( $\tau_b/\tau_c$ ) of 5 at 550 nm. The photopic transmittance of the devices can be obtained by integrating the spectra in the wavelength range of 400 to 730 nm using the spectral photopic efficiency  $I_p(\lambda)$  (CIE, 1924) as the weighting factor<sup>7</sup> (see also E 1423, C 1199, CAN/CGSB 12.8).

8.2.1 A trapezoidal voltage profile similar to the one used at room temperature (ca. 22°C) is also used for the long-term cycling tests at temperatures ranging from 70°C (158°F) to 105°C (221°F). Each ECW is heated in a convection oven at T and the coloring and bleaching times are determined for obtaining the specified PTR, e.g., 5 at 550 nm. These data are then used to program the multichannel potentiostat with specific voltage profiles (for each device type) for cyclic testing at T in the AWU. After cycling for the desired time period, (for example, 4,000 to 10,000 cycles), the samples are cooled to room temperature and removed from the AWU. These samples are then electro-optically recharacterized at room temperature (ca. 22°C) using the voltage profile determined temperature (ca. 22°C) during the pretest procedure and compared to the initial values as shown in Fig. 4. The initial photopic transmittance for a typical ECW is shown in Fig. 5 as open circles, and the open squares and solid-circles indicate the typical photopic transmittance of an ECW at 22°C (72°F) after 5,000 and 10,000 cycles at an elevated temperature, respectively.

8.3 Accelerated Weathering Environmental Exposure Chamber and Irradiance Calibration. Fig. 1 shows a top-view schematic diagram of the essential features of using the environmental test chamber including the layout of the ECWs on a 1220 mm x 1830

<sup>7</sup> R. Kingslake, "Applied Optics and Optical Engineering," in Vol. 1, *Light: Its Generation and Modification*, Academic Press, New York, NY, 1965, Table II, Chapter 1.

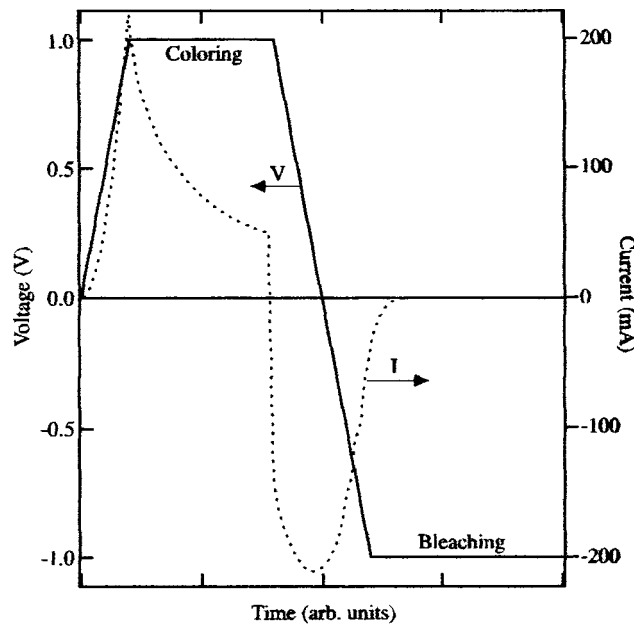


FIG. 3 Voltage and Current as a Function of Time During Coloring and Bleaching Processes for a Typical ECW

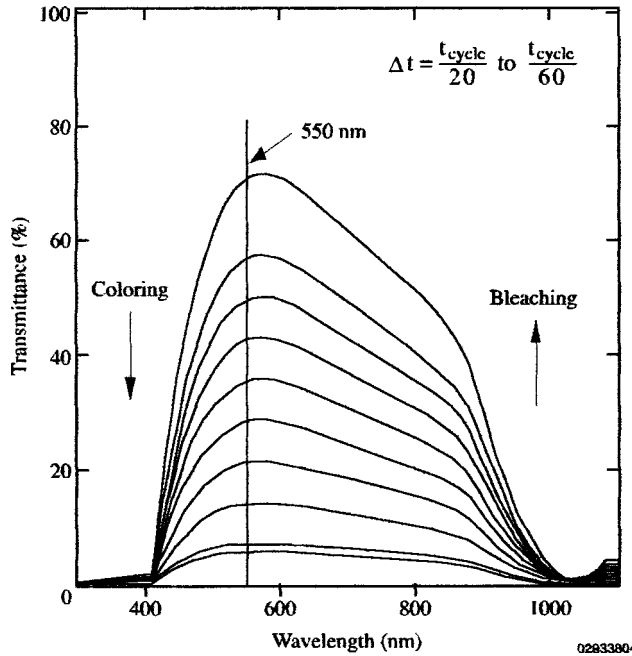


FIG. 4 Transmittance Spectra During a Coloring and Bleaching Process at Intervals Ranging from  $t_{cycle}/20$  to  $t_{cycle}/60$  for a Typical ECW

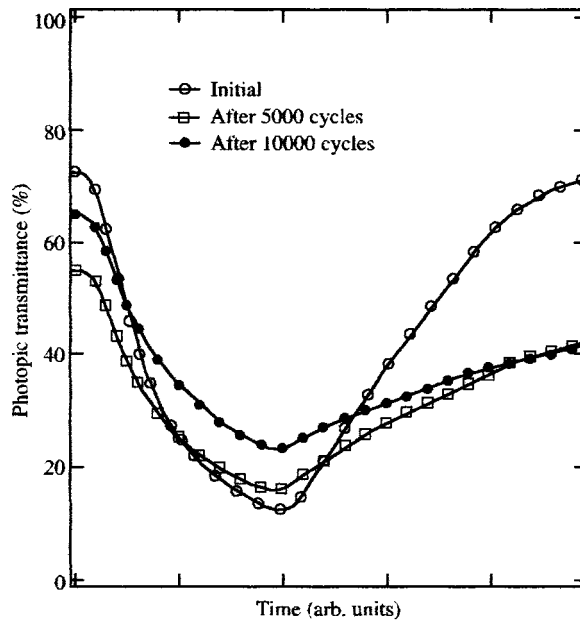


FIG. 5 Photopic Transmittance as a Function of Time Measured at Various Stages of Cycling of a Typical ECW

mm (4-ft × 6-ft) test plane, the location of four xenon-arc lamps above the test plane, and the necessary connecting cables from the ECWs to the computer-controlled cycling and data acquisition system. (The XR-260 is 2440 mm (8 ft) high, 2650 mm (8.7 ft) deep, and 4480 mm (14.7 ft) wide and is specifically designed for indoor accelerated weathering tests of large samples.) The test plane is vertically adjustable and the user should adjust the distance from the lamps to the sample to obtain the desired light intensity and lateral uniformity within the guidelines of this document. Conditions inside the closed space can be controlled for temperatures from -40°C to 95°C (-40°F to 203°F) and relative humidities ranging from 5 % to 95 %. Temperature and relative humidity control within the test chamber are provided with a humidifier and by an integral forced-air heating and cooling system. Simulated solar irradiance is provided by four spectrally filtered and water-cooled 6500-W, long-arc xenon lamps housed within a reflector system in the ceiling of the test chamber. The ECWs are located on the vertically moveable test plane beneath the xenon

lamps. Monitoring of the coloring and bleaching processes and of the sample temperatures are accomplished with a computerized electronic control and data acquisition system via 9 m (30-ft.) long cables through access ports in the rear chamber wall.

8.3.1 The xenon lamps are powered to yield roughly a 1-sun illumination at the sample exposure plane from 290 nm through 600 nm. The lamps at these wavelengths shall be suitably filtered to provide an excellent match of an AM 1.5 solar spectrum from 300 nm to 900 nm (See Note 23). The water-cooled lamps are surrounded by an inner IR-absorbing filter and an outer IR-absorbing filter, which prevent an excessive heat load. Calculate the total accumulated energy dose. For example, the AM 1.5 irradiance of 1-sun in 24 h yields the equivalent of a total accumulated solar energy dose of 86.4 MJ/m<sup>2</sup> (2,592 MJ/m<sup>2</sup> per 30-day month) to each of the ECW samples. The four Xe lamps are typically operated at about 5400 W. At this power, the filters and lamps only need to be replaced after about 2000 h of operation. Adjust the chamber temperature to obtain the desired surface temperature of the ECW. For example, with a chamber air temperature of  $39^{\circ}\text{C} \pm 1^{\circ}\text{C}$  ( $102 \pm 2^{\circ}\text{F}$ ), the average ECW surface temperature reaches a steady-state temperature of about  $60^{\circ}\text{C} \pm 5^{\circ}\text{C}$  ( $140 \pm 9^{\circ}\text{F}$ ) depending on the sample size, optical PTR-ratio reached during voltage cycling, location of the device in the test plane, and the ECW construction. When the relative humidity is not controlled during testing, it may range from 5 % to 20 % at a 60°C (140°F) chamber temperature.

NOTE 23—At longer wavelengths, the fit to the solar spectrum is at variance with the AM 1.5 spectrum because of thermal emission lines associated with the xenon-arc lamp, but this part of the spectrum is generally not an important cause of photolytically induced degradation.

8.3.2 The remainder of this section is a description of a suitable procedure for deducing the spatial uniformity of the irradiance in the test plane<sup>8</sup>. A uniformity measurement system (UMS) can be constructed using a thick aluminum plate as a base that is 610 mm × 1220 mm × 3.2 mm (2 ft. × 4 ft. × 0.125 in.). Sensors can be mounted in a 305 mm (1-ft.) center-to-center spacing scheme with a 3-row, 5-column configuration. To reduce adverse heating effects, all sensors should be spaced about 3 mm (0.125 in.) above the plate to allow for proper air circulation around the entire device. The plate itself should be painted flat black to minimize return reflections. The sensors are connected via a multiconductor data cable to an electronic enclosure that houses 15 miniature digital panel meters (DPM). Calibration is achieved by referencing all the sensors to a pyranometer under natural daylight conditions. All DPM readouts are then adjusted to agree with the PSP at a 1-sun (1000 W/m<sup>2</sup>) reading, which typically results in a normal absolute error of about ±3 % and a maximum uncertainty of ±5 %.

8.3.3 The procedure for determining the irradiance in the test plane then involves laying the UMS assembly inside the AWU (e.g., an XR-260) chamber on the test plane, with the sensors facing the xenon lamps. External connections are made to the DPM enclosure via the multiconductor data cable through the access port to the chamber. The chamber is then set to operate at 25°C (77°F) and a relative humidity of 20 %. These are arbitrary operating set points because no defined procedures or specifications exist for calibrating solar simulator lamps. The Li-C or sensors used are not recommended for an absolute calibration measurement because of their slow response time and limited wavelength response. However, the procedure permits approximating the relative spectral uniformity across a defined test plane even though the absolute values are not known.

8.3.4 Once the chamber has reached the set-points, the lamps are ignited at 5400 W and are allowed to stabilize for whatever length of time the manufacturer recommends, which is usually 3 to 7 min. While watching the DPM readouts, the test-plane height is raised or lowered until the DPM readings average 1-sun (~1000 W/m<sup>2</sup>). The DPM readouts will not agree with each other unless the intensity is the same at all positions; the uniformity differences in the test plane are readily apparent. The procedure from this point is to adjust the lamp intensity or reflectors, or both, to improve uniformity of the lateral intensity within the desired area in the test plane. This is done by noting the readings until they agree as close as possible with each other but produce an overall lateral uniformity of ±8 %.

8.3.5 Depending on the size of the desired test-plane area, the sensor plate is moved incrementally up or down, or left or right, for the number of measurements desired to establish the test-plane uniformity. The uniformity in the test-plane area is typically ±7 % to 8 %.

8.4 *Mounting ECWs in the AWU Test Chamber.* Each manufacturer of ECWs must provide the coloring and bleaching voltages for room temperature characterization and for operating their ECWs at a selected test temperature. When received, inspect the ECWs visually, take photographs of any obvious defects or aberrations of the EC samples in the bleached state or colored state, and note and record your observations. Make electrical connections, e.g., solder friction-fit, bullet-type, make/break or some other suitable connector, to the wires of each EC device. Mate the connectors with those on the cables, e.g., 9 m (30 ft.) long, leading to the computer-controlled ECW testing electronics. Record successive optical transmittance measurements for color/bleach cycles at room temperature (ca. 22°C or 72°F) using the voltages supplied by the manufacturer to verify the electrical protocols for achieving a 5:1 PTR. Compare subsequent optical and electrical data with these data as a measure of degradation of each ECW after environmental exposure. Characterize the samples optically and electrically in a convection oven (8.2) at the intended temperature of testing to determine the bleaching and coloring times required to achieve a 5:1 PTR at the elevated temperature. This measurement establishes what voltage and time protocols will be used for the accelerated voltage cycling of the samples at the test temperature. For example, the ECW samples may be electro-optically cycled for durability testing at 85°C (185°F) using the 85°C (185°F) protocol and periodically characterized for transmittance changes at room temperature (ca. 22°C) using the 22°C (72°F) protocol.

<sup>8</sup> A description of a recently improved procedure is given in Appendix 1.

8.4.1 Place the ECW samples horizontally onto the test plane and connect them to the cables leading to the remote electronics via the connectors, e.g., bullet-type, quick-disconnect terminals, described earlier. Tape thermocouples (0.13 mm or 5-mil diameter) to the center surface of the samples (facing the light source) with 8-mm square (0.3-in. square) pieces of 0.05-mm (0.002 in.)-thick aluminum tape. The thermocouple leads may be taped about 75 mm (3 in.) away from the center of the sample to provide strain relief. Mate the thermocouples to the appropriately thicker extension wires leading to the remote electronics via subminiature connectors.

NOTE 34—Before cycling at the test temperature, it is prudent to electro-optically cycle all the ECW samples at room temperature (ca. 22°C) to verify the integrity of the electronic control and data acquisition system, as well as the continuity of the electrical and thermocouple connections.

8.5 *Voltage Cycling the ECWs at an Elevated Temperature.* With the sample in the bleached state, illuminate the Xe lamps and adjust the dry-bulb temperature (DBT) to provide control of the chamber air temperature. The DBT serves as the controlling parameter for the internal forced-air heating and cooling system and is set at a temperature less than T so as to achieve a sample temperature of T, which is reached by the combined heat transfer from conduction and exposure to the irradiance sources. The sample temperature depends on the manufactured ECW construction, PTR during voltage cycling, sample size, and location in the AWU test plane, as well as whether the ECW is in the colored or bleached state. When the bleached EC sample approaches the desired testing temperature, begin the voltage cycling of the ECW using the electrical protocol at T obtained by earlier oven pretesting at T. Make minor adjustments to the DBT setting to account for the inevitable rise in temperature of the samples resulting from absorption in the colored state. Sample temperatures may increase or decrease by up to several degrees Celsius during cycling the sample through the colored and bleached states, respectively. Continually record and monitor periodically the temperatures and electro-optic cycling data using the data acquisition system to ensure proper operation of the AWU, associated experimental apparatus, and the computer.

8.5.1 Program the electro-optic cycling of the EC devices and the functions of the AWU to shut down periodically after a predetermined number of coloring and bleaching cycles; these may be typically  $6,000 \pm 2,000$  cycles for testing the ECWs. After the first shut down, disconnect the thermocouple and electrical leads to the sample from the cabling, remove the samples, and remeasure the optical transmittance at room temperature (ca. 22°C). Visually inspect the ECW samples and photograph any detectable degradation with the digital camera. Note and record any visually detectable degradation of the samples in the bleached or colored state. Record the electro-optic measurements and other observations, and reinsert the ECW samples into the AWU for the next series of cyclic testing, e.g., another 4,000 to 10,000 coloring and bleaching cycles. Repeat this procedure until a total of 50,000 cycles are achieved or a PTR of less than 4 is obtained at room temperature (ca. 22°C), whichever result comes first. If a PTR below 4 is reached before 50,000 cycles are completed or if  $\tau_b$  of less than 50 % is measured, the ECWs fail the durability test.

NOTE 5—As ECWs age from exposure to accelerated weathering (8.5), the times to color and bleach usually become longer (Fig. 5). Rigidly using the coloring and bleaching times for the new device on an aged device may result in a PTR of less than 4, but the device still may be suitable for conserving energy in buildings. Before an ECW is deemed a failure, the times to color and bleach should be extended for up to 30 min or up to the time it takes for the rate of change of the transmittance to become less than about 0.4% of the transmittance per minute in the colored or bleached state, respectively, whichever yields the shorter time to color or bleach. If a PTR of less than 4 is still obtained when using times of up to 30 min to color or bleach, then the device fails this performance criterion.

NOTE 6—The visual uniformity tests should be made when the ECW is held at a constant transmittance. To establish a given transmittance state for assessing the uniformity of the ECW, the manufacturers should be asked to provide control information (voltage, current, time) that will result in a constant transmittance of the ECW in the colored and bleached state, and this information should be used.

8.6 *Video Documentation.* After the final cycling series in 8.5, record the dynamic response of the ECWs at room temperature (ca. 22°C). Mount each ECW that has been aged as in 8.5 next to an unaged witness specimen from the same lot of all those tested. Record the dynamic response for 5 coloring and bleaching cycles using the video camera.

8.7 *Final Visual Inspection.* After the final cycling series in 8.5, perform the final visual inspection, take photographs, and record all evidence of visually detectable degradation.

## 9. Analysis for Performance Losses

9.1 *Coloration Efficiency.* The coloration efficiency may be useful as an indicator of performance losses. The PTR of a typical ECW can be calculated from the transmittance spectra such as those shown in Fig. 4. These ratios are shown in Fig. 6 as a function of the cycle number (see open squares). The change in the coloring efficiency ( $\eta$ ) at any time  $t$  of the ECWs can be also calculated from these ratios and the charge ( $Q$ ) passed through the ECW during the coloring process as given in the following:

$$\Delta\eta(t) = \frac{\Delta O.D.}{Q} = \frac{\log\left(\frac{\tau_b}{\tau_c}\right)}{\int_0^t i(t) dt}$$

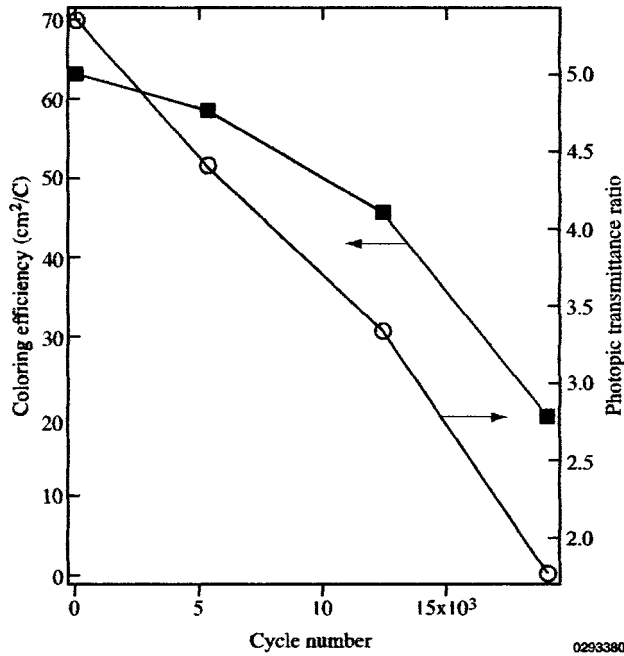


FIG. 6 Photopic Transmittance Ratio and Coloring Efficiency as a Function of Cycle Number for a Typical ECW

where:

$O.D.$  = the optical density,

$i$  = the ion current,

$t$  = the time, and

$t_c$  = is the time to color the ECW.

The typical trend for coloring efficiency of an ECW as a function of cycle number is shown in Fig. 6 as solid squares. Both the PTR and coloring efficiency decrease with cycle number for the device shown.

NOTE 7—The coloration efficiency may be difficult to assess for solid state devices in which the measured current is the sum of the ionic and electronic currents and not just the ion current,  $i$ .

9.2 *Photopic Transmittance Ratio*. The PTR is calculated from the optical transmittance data in the bleached and colored states. This is especially important because interference effects can distort the real changes in the PTR when only a single wavelength is used. The following may be used to calculate  $\tau_b(p)$ , the photopic transmittance in the bleached state:

$$\tau_b(p) = \frac{\int_{\lambda_{min}}^{\lambda_{max}} \tau_b(\lambda) I_p(\lambda) d\lambda}{\int_{\lambda_{min}}^{\lambda_{max}} I_p(\lambda) d\lambda}$$

where:

$\lambda_{max}$  = 730 nm,

$\lambda_{min}$  = 400 nm,

$\tau_b(\lambda)$  = the transmittance in the bleached state at any wavelength, and

$\lambda$ , and  $I_p(\lambda)$  = the photopic intensity function.

A similar expression is used to calculate  $\tau_c(p)$ , the photopic transmittance in the colored state. Then, the PTR is simply  $\tau_b(p)/\tau_c(p)$ . A plot (Fig. 6) of the PTR versus cycles completed is also useful for assuring performance degradation of the ECWs.

9.3 *Additional Analyses*. The  $t_c$  to a given  $\tau_c(p)$  or  $t_b$  to the original or maximum  $\tau_b(p)$  may also be useful for assessing performance degradation. Typically, an ECW exhibits larger  $t_c$  or  $t_b$  that are too long. Measurements of the PTR at different sample locations, e.g., at the center, corners, and between the center and edge of the ECW, may be useful for assessing performance degradation. Lateral variations in the PTR may also result as the ECW ages. Any PTR below 4.0 is considered a failure.

## 10. Observations

10.1 Observe and document the following:

10.1.1 Specimen breakage.

10.1.2 Specimen failure that is indicated by a PTR < 4.

10.1.3 Specimen degradation that is evident visually in the bleached state.

- 10.1.4 Specimen degradation that is evident visually in the colored state.
- 10.1.5 Specimen degradation that is evident from photographs in the bleached state.
- 10.1.6 Specimen degradation that is evident from photographs in the colored state.
- 10.1.7 Specimen degradation that is evident from the videos in the bleached state.
- 10.1.8 Specimen degradation that is evident from the videos in the colored state.

**11. Report**

11.1 The following information shall be provided in the test report.

11.1.1 A complete description of the test specimen(s).

11.1.2 The tests performed on the specimen(s) and the results of those tests both before and after accelerated tests were conducted.

11.1.3 The type of accelerated tests conducted.

11.1.4 The number of dynamic cycles completed before failure occurs.

11.2 Additional information such as technical drawings and videos may be included in the test report.

11.3 Prepare a report for each set of ECWs after each series of cycles and optical characterization. The report shall provide the PTR after each series of cycles including the initial and final PTR. The reports may provide tables with the appropriate entries for the initial characterization data and the same data after electro-optical voltage cycling, as shown by the simulated data in Table 1. The photopic transmittance data may be provided only in the final report. Plots of the voltage ramps similar to Fig. 3, transmittance changes during coloring and bleaching similar to Fig. 4 (but as separate plots), the transmittance during coloring and bleaching similar to Fig. 5, and the PTR similar to Fig. 6 may be provided for each set of ECWs tested. The time interval between plots of the transmittance spectra similar to Fig. 4 was specified for their use. Finally, use longer coloring and bleaching times, e.g., 5 to 10 times longer than used during cycling for room temperature characterization, to determine the time to reach PTRs of 5 after each series of cycles.

11.3.1 As with the quantitative data, summarize and include the visual observations for the ECWs with the custom report prepared for each supplier. When possible, summarize the observations in general for all ECWs and then make appropriate additional comments for each sample. After final cycling and electro-optic characterization, place each exposed ECW side by side with the control sample (not subjected to exposure in the AWU) and make a video-camera recording to capture visually the effects of any degradation during a color/bleach cycle using the 22°C (72°F) electro-optical coloring/bleaching protocols. Carefully pack and store the tested ECW samples subjected to the durability testing. The time-consuming final analyses involve assembling and integrating the original electro-optical cycling data and subsequent periodic cycling results into a series of coherent and independent final reports.

**12. Additional Needs**

12.1 As reported in 1999<sup>5</sup> a great deal remains to be learned about how to carry out durability testing correctly. During the initial efforts,<sup>5,6</sup> additional improvements were identified that urgently need to be made. These include (a) using a higher sample temperature e.g., 85°C and 107°C (185°F and 225°F), (b) using enhanced irradiance (e.g., 2-3-sun) conditions, (c) optimizing the trapezoidal voltage waveform to minimize the damage during coloring and bleaching, (d) establishing an appropriate duty cycle for the coloring and bleaching voltages, (e) improving the measurements of transmittance changes by using fiber optic cables at more locations on each ECW, and (f) securing testing apparatus that can operate reliably for longer periods of time than required to complete the ECW durability tests. From (a) and (b), the testing time will be reduced, assuming the increased temperature and irradiance produce more accelerated degradation. From (c) and (d), the testing time may be increased, but simulating the in- service use will be more realistic. From (e), better statistics and quantitative evaluation will be obtained about the overall non-uniformity of coloring and bleaching after any degradation occurs, e.g., a single defect at the spot of transmittance measurement will greatly skew the results for the entire ECW. The video comparisons help reduce this problem, but video results are visual and qualitative. Item (f) is especially important because the calendar time for completing the testing was about three times the actual time for testing and electro-optical characterization. The major reliability problems resulted from failures in the potentiostat-galvanostat testing unit, storm-related power surges to the computer, and an inability of the AWU to provide sufficient cooling for the xenon lamps and chamber. These have all been corrected for future testing, but additional equipment reliability issues can be expected

**TABLE 1 Electro-optical Test Results at 22°C (72°F) Before and After Exposure in an AWU**

Device Number	V <sub>c</sub> (V)	V <sub>b</sub> (V)	t <sub>c</sub> (s)	t <sub>b</sub> (s)	% τ <sup>A</sup>		% τ		Cycles at 60°C/85 ± 5°C and 1-sun		
					Max	Min	Max	Min			
D-1	w	y	x	z	aa	bb	aa/bb	ee	ff	ee/ff	0
D-1	w	y	x	z	cc	dd	cc/dd	gg	hh	gg/hh	V.VVV

<sup>A</sup> PTR is the photopic transmittance ratio % τ<sub>b</sub>/% τ<sub>c</sub>.

as a generic problem in long-term testing. Clearly, controlled testing of ECWs for 20,000 to 50,000 cycles requires that all the testing equipment functions flawlessly for up to a year, depending on the coloring/bleaching time for one voltage cycle. Constructive feedback to the manufacturers is essential to improve the reliability of their testing equipment.

### **13. Precision and Bias**

13.1 Precision—The precision of the procedures in this test method is being determined.

13.2 Bias—Because there are no accepted reference materials suitable for determining the bias for the procedures in this test method, bias has not been determined.

### **14. Keywords**

134.1 chromogenic glazing; durability; electrochromic windows; fenestration; fenestration products

## **APPENDIX**

(Nonmandatory Information)

### **X1. ADDITIONAL INFORMATION**

#### **X1.1 Acronyms Used in This Test Method**

- X1.1.1 *ALT* = accelerated life testing
- X1.1.2 *AWU* = accelerated weathering unit
- X1.1.3 *AM* = air mass
- X1.1.4  $\eta$  = coloring efficiency
- X1.1.5 *I-V* = current-voltage
- X1.1.6 *DPM* = digital panel meters
- X1.1.7 *DBT* = dry-bulb temperature
- X1.1.8 *ECW* = electrochromic window
- X1.1.9 *IG* = insulating glass
- X1.1.10 *IGUs* = insulating glass unit(s)
- X1.1.11 *IR* = infrared (radiation)
- X1.1.12 *PTR* = photopic transmittance ratio or  $PTR = \tau_b/\tau_c$
- X1.1.13  $\tau_c$  = specular transmittance in the colored state
- X1.1.14  $\tau_b$  = specular transmittance in the bleached state
- X1.1.15 *UV* = ultraviolet (radiation)
- X1.1.16 *UMS* = uniformity measurement system
- X1.1.17 *V* = voltage

#### **X1.2 Additional Useful Definitions for Terminology Used in This Standard**

- X1.2.1 *accelerated life testing*— is a protocol that results in accelerated aging of materials or devices.
- X1.2.2 *anomalous hot spot*—(in the lateral uniformity) is a region of unexpected elevated temperature.
- X1.2.3 *coloration efficiency*— is the change in optical density (OD) per unit of charge (Q) inserted into an EC device or material.
- X1.2.4 *counter electrode layer*— is the ion storage material in an ECW that serves as a reservoir for ions that can be inserted into or received from the electrochromic layer.
- X1.2.5 *degradation factors*—refer to conditions, imposed or natural, that influence or cause a degradation mechanism, effect, or mode.
- X1.2.6 *electro-optic characterization*— refers to the process of recording optical changes (transmittance, reflectance, absorbance, etc.) in an ECW as a function of electrical protocols (voltage, current).
- X1.2.7 *electro-optic cycling*— refers to the electrochemical cycling process of applying repetitive positive and negative voltages to an ECW for the purpose of reversibly changing the optical properties of the ECW device from the bleached to the colored state.
- X1.2.8 *integrated irradiance*— refers to the cumulative amount of irradiance within a spectral bandwidth of interest.
- X1.2.9 *lateral uniformity*—refers to the degree of variation in the amount of irradiance in the x and y directions in the test plane used for exposing an ECW.
- X1.2.10 *light intensity*—is the areal density of irradiance on a surface illuminated with a light source.
- X1.2.11 *optical density*—refers to the attenuation in the amount of transmitted light by absorptive or reflective processes in the material being irradiated. OD is the base 10 logarithm of the reciprocal of the transmittance ( $\tau$ ):  $OD = -\log_{10}(\tau)$ .
- X1.2.12 *optical photopic transmittance ratio*—refers to the ratio of the bleached state transmittance ( $\tau_b$ ) to the colored state transmittance ( $\tau_c$ ) where  $\tau_b$  and  $\tau_c$  are both weighted by a spectral photopic response curve.

X1.2.13 *optical transmittance*— is the ratio of the radiant energy transmitted by a body to the total radiant energy incident upon the body.

X1.2.14 *photodiode array spectrophotometer*— is an optical detector system that uses an array of photodiodes coupled to CCDs to facilitate UV-VIS-NIR spectroscopic measurements.

X1.2.15 *photolytic-induced degradation*— refers to the decomposition or deterioration of properties of a material that results from the action of light.

X1.2.16 *solar irradiance*—refers to exposing a material or device to the sun or a simulated solar light source, i.e., from 295 nm to 2600 nm.

X1.2.17 *spatial uniformity*—is the same as lateral uniformity as used in this standard.

X1.2.18 *spectral irradiance*—refers to exposing a material or device to a light source over a specific bandwidth of radiation, e.g., from 400 nm to 730 nm.

X1.2.19 *spectral photopic response*— refers to the relative response of the human eye in its light-adapted state (daylight) to radiation of a given wavelength ( $\lambda \sim 410\text{-}720$  nm).

X1.2.20 *spectral radiometer*—is a device for measuring the irradiance in  $\text{W}/\text{m}^2$  or a comparable areal density unit.

X1.2.21 *specular transmittance*— refers to the optical transmittance that does not include light with a diffuse component.

X1.2.22 *trapezoidal voltage profile*— is the geometric shape generated by plotting the voltage versus time applied to an ECW with a slope in V/s up to a constant voltage and then a negative slope in V/s back to zero voltage (See Fig. 4).

X1.2.23 *uniformity measurement system*— is used for measuring the lateral uniformity of the irradiance in the test plane.

X1.2.24 *UV acceleration factor*— refers to the ratio of the amount of UV irradiance received by a material from a UV light source compared with that from terrestrial exposure to the sun for the same spectral bandwidth (typically 300-400 nm).

X1.2.25 *X-Y gantry stage*—refers to an arrangement that moves an object in two perpendicular directions in a test plane.

X1.3 The suitability of the test methods will be further evaluated as the analyses of test results are completed.

X1.4 The total number of specimens supplied shall be three more than the number listed in 6.2 and shall serve as control specimens or to allow for breakage of two specimens.

X1.5 The test methods are to simulate the in-use conditions of an EC coating in an IG unit.

X1.6 The tests may be carried out in parallel or separately.

### **X1.7 Description of an Alternate Automated Procedure for Deducing the Spatial Uniformity of Irradiance in the Test Plane**

X1.7.1 To map the spectral and spatial uniformity of the light intensity experienced by samples exposed within our Atlas XR-260 exposure chamber, a programmable X-Y gantry stage was constructed that can be assembled and operated inside the chamber. Fig. X1.1 shows the hardware arrangement. There are four 6500W xenon-arc lamps with a CIRA inner and soda-lime outer filter system designed to provide a close spectral match to a terrestrial air-mass (AM) 1.5 global solar spectrum. (See Tables E 892.) Samples are exposed on a vertically adjustable horizontal plane having dimensions roughly 1.2 m by 1.8 m (4' x 6'). The nominal vertical distance from the light sources to the exposure plane is 770 mm (30.5"). Each of the 4 lamps is controlled to 0.12  $\text{W}/\text{m}^2$  at 340 nm. The X-Y gantry stage allows a light detector to be positioned at a number of programmed locations (for example, at a regular grid) in the sample exposure plane without having to turn off the lights, open the doors, and manually move the detector.

X1.7.2 Two types of detectors have been used. The first is a fiber optic probe that couples a mini-integrating sphere detector with an Oriel Merlin spectral radiometer and allows complete spectral characterization between 250-1100 nm. The other (faster / more convenient) detector is an Atlas Xenosensitive unit having an integrated spectral response between 300-400 nm only. A comparison of the ASTM standard spectrum [1] and the spectral irradiance measured in the sample plane at the center of the chamber is shown in Fig. X1.2. Very good agreement is seen in the UV part of the spectrum (between 300-400 nm), which is generally the part of the solar spectrum that can cause materials degradation.

X1.7.3 Changing the power to the xenon lamps will shift the intensity of the measured spectrum up or down but the shape will remain the same. (For a given filter system, the spectral shape is primarily effected by the age of the lamps, which is why controlling irradiance at 340 nm, i.e., roughly midway within the UV band is the usual practice.) For example, the measured irradiance at 340 nm (Fig. X1.2) is 0.542  $\text{W}/\text{m}^2/\text{nm}$  and the ASTM standard value [1] is 0.420  $\text{W}/\text{m}^2/\text{nm}$ . The ratio of these numbers yields a UV acceleration factor of  $\sim 1.3$ . However, a more precise quantity is the ratio of the integrated irradiance within the spectral bandwidth of interest. Between 300-400 nm, the ASTM standard specifies an irradiance of 44.49  $\text{W}/\text{m}^2$ . The ratio of the Xenosensitive detector to this value will therefore provide the UV acceleration factor.

X1.7.4 The spatial uniformity within the XR-260 chamber was mapped by positioning the Xenosensitive detector at 96 regular grid positions within the sample plane (+ signs in Fig. X1.3). Excellent uniformity was found; the irradiance was measured to be  $52.5 \pm 4$   $\text{W}/\text{m}^2$  between 300 to 400 nm. This corresponds to a 1.2 sun UV acceleration factor. The anomalous hot spot at  $x=1$  000 mm,  $y=700$  mm results from aluminum foil used to shield a cable feedthrough at that location; samples are not exposed in proximity to this position.

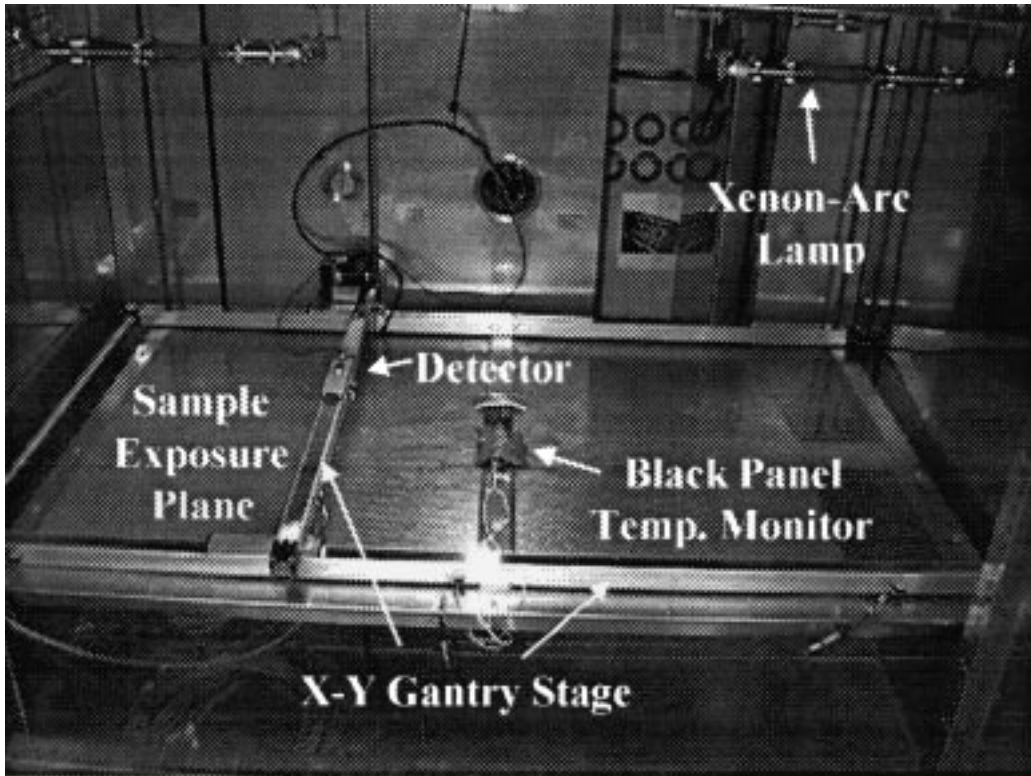


FIG. X1.1 X-Y Gantry Stage Inside XR-260

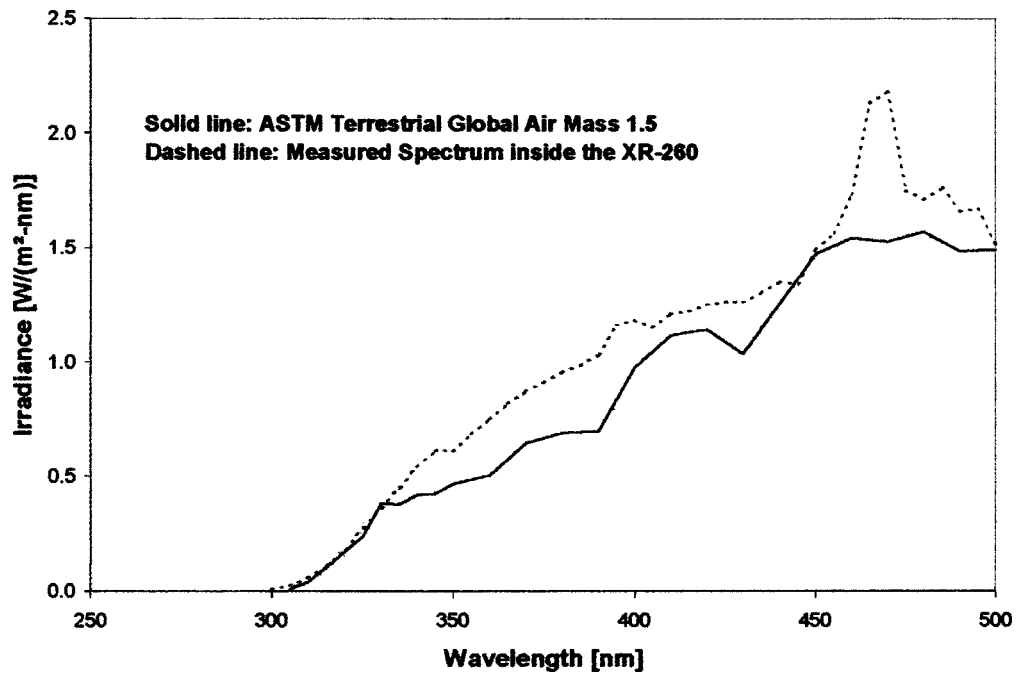


FIG. X1.2 Spectral Irradiance of XR-260 vs. ASTM Standard Terrestrial Global Air Mass 1.5 Spectrum

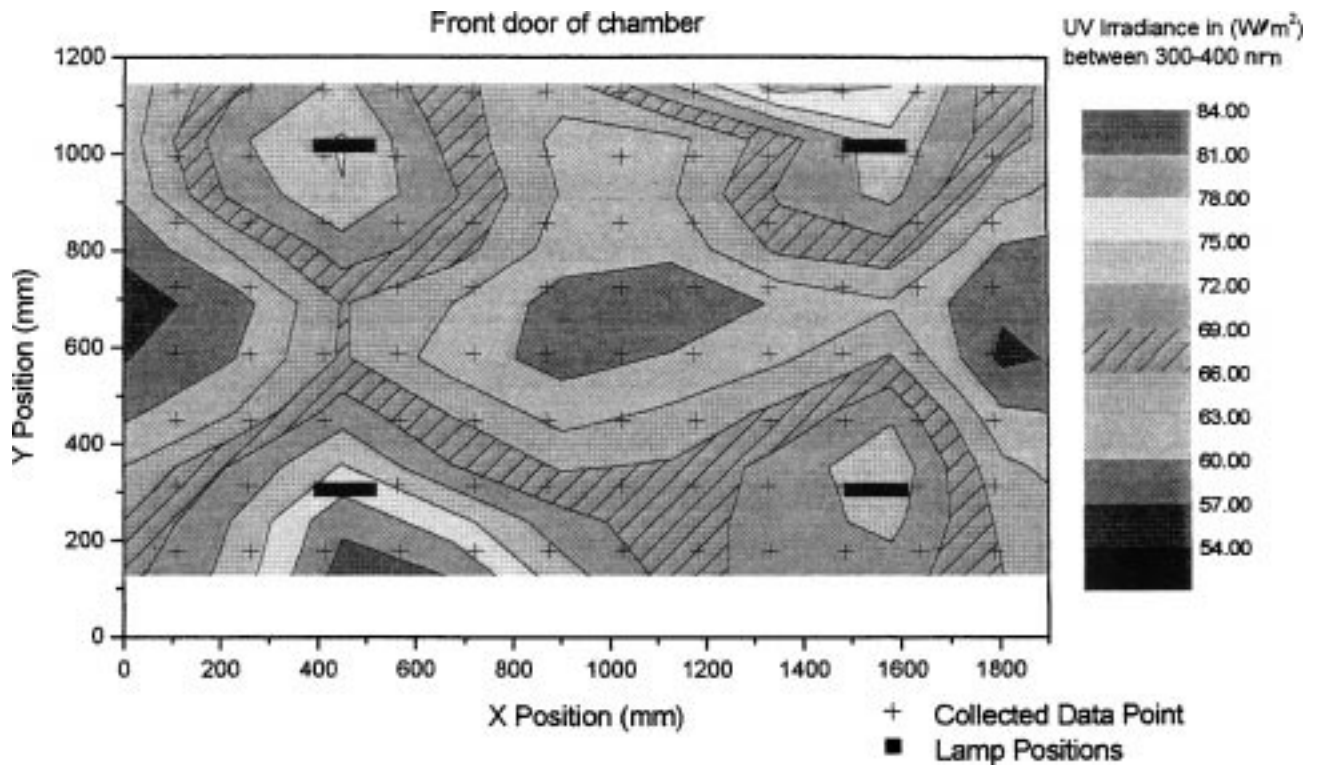


FIG. X1.3 Spatial Distribution of UV Irradiance Within XR-260 Exposure Chamber

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