

# Standard Test Method for Accelerated Aging of Electrochromic Devices in Sealed Insulating Glass Units<sup>1</sup>

This standard is issued under the fixed designation E2141; 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.

#### 1. Scope

- 1.1 This test method covers the accelerated aging of electrochromic devices (ECD) integrated in insulating glass units.
- 1.2 The test method is applicable for any electrochromic device incorporated into sealed insulating glass units (IGUs) fabricated for vision glass (superstrate and substrate) areas for use in buildings, such as sliding doors, windows, skylights, and exterior wall systems. The layers used for constructing the EC device and electrochromically changing the optical properties may be inorganic or organic materials.
- 1.3 The electrochromic (EC) glazings used in this test method are exposed under use conditions 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 method is not applicable to other chromogenic devices, such as, photochromic and thermochromic devices which do not respond to electrical stimulus.
- 1.5 The test method is not applicable to electrochromic (EC) glazings that are constructed from superstrate or substrate materials other than glass.
- 1.6 The test method referenced herein is a laboratory test conducted under specified conditions. The test is 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.
- 1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
  - 1.7.1 Exception—Inch-pound units are used 7.6.2.

1.8 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>

C168 Terminology Relating to Thermal Insulation

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E631 Terminology of Building Constructions

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E2953 Specification for Evaluating Accelerated Aging Performance of Electrochromic Devices in Sealed Insulating Glass Units

G113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials

G173 Tables for Reference Solar Spectral Irradiances: Direct Normal and Hemispherical on 37° Tilted Surface

2.2 ISO Standard:<sup>3</sup>

ISO 9050 Glass in building - Determination of light transmittance, solar direct transmittance, total solar energy transmittance, ultraviolet transmittance and related glazing factors

#### 3. Terminology

- 3.1 *Definitions*—For definitions of general terms used in this test method related to building construction, thermal insulating materials and natural and artificial weathering tests for nonmetallic materials, refer to Terminologies E631, C168 and G113, respectively.
  - 3.2 Definitions of Terms Specific to This Standard:

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

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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.

<sup>&</sup>lt;sup>3</sup> Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.



- 3.2.1 accelerated aging test, n—a test in which the rate of degradation of building components or materials is intentionally increased from that expected in actual service.
- 3.2.2 electrochromic device (ECD), n—a combination of materials that include materials in which the transmittance, reflectance and absorptance properties can be altered, and other layers, such as transparent conducting oxide (TCO) layers for altering the optical properties (for example, transmittance, reflectance, absorptance) of the device, in response to an applied electrical voltage or current.
- 3.2.3 electrochromic (EC) glazing, n—in a prepared opening of a building, the material installed which consists of an ECD with layer(s) of materials in which the optical properties can change in response to an applied electrical field, attendant materials, and one or more lites of glass.
- 3.2.4 *electrochromic layer(s)*, *n*—in an ECD, the layer(s) of material(s) in which the optical properties can change in response to application of an electrical voltage and/or current.
- 3.2.5 *electro-optic cycling, n*—the process of applying repetitive positive and negative voltages and/or currents to an ECD for the purpose of reversibly changing the optical properties of the EC glazing from the highest to the lowest transmittance state.
- 3.2.6 highest transmittance state, n—also referred to as the clear state or bleached state, a descriptor for an EC glazing when it is in the transmittance state with the highest photopic specular light transmittance.
- 3.2.7 *lateral uniformity, n*—the degree of variation in the amount of irradiance in the x and y directions in the test plane used for exposing an EC glazing.
- 3.2.8 *lowest transmittance state*, *n*—also referred to as the tinted state, dark state or colored state, a descriptor for an EC glazing when it is in the transmittance state with the lowest photopic specular light transmittance.
- 3.2.9 photopic transmittance ratio (PTR), n—photopic transmittance ratio or PTR =  $\tau_{\rm H}/\tau_{\rm L}$ . The photopic transmittances ( $\tau_{\rm H}$ ,  $\tau_{\rm L}$ ) are obtained by integrating the spectra in the wavelength range of 380 to 780 nm using the spectral photopic efficiency Ip (CIE, 1924) as the weighting factor.
  - 3.2.10 room temperature, n—ca 22°C
- 3.2.11 *serviceability*, *n*—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 *solar irradiance*, *n*—as related to natural weathering of materials, the irradiance of the sun incident on the earth's surface, having wavelengths between 295 nm and 4050 nm.
- 3.2.13 *specular (regular) transmittance, n*—the optical transmittance that does not include light with a diffuse component.
- 3.2.14 *switching cycle, n*—a transition in visible light transmittance through the whole or part of the EC-IGU's visible light transmittance range starting at one end of the range (at  $\tau_H$  or  $\tau_L$ ) and ending back at the same point. For example, one switching cycle can be a transition from the highest transmittance state to the lowest transmittance state and back to the

highest transmittance state, or a transition from the lowest transmittance state to the highest transmittance state and back to the lowest transmittance state.

- 3.2.15 switching times  $T_H$  and  $T_L$ , n—switching time,  $T_H$ , is the time it takes for the EC glazing to transition from its highest transmittance state over 80% of its initial dynamic range (where dynamic range is  $\tau_{H^-}\tau_{L}$ ). Switching time ( $T_L$ ) is the time it takes for the EC glazing to transition from its lowest transmittance state across 80% of its dynamic range (where dynamic range is  $\tau_{H^-}\tau_{L}$ ).
  - 3.3 Acronyms:
  - 3.3.1 *AM*—air mass
  - 3.3.2 AWU—accelerated weathering unit
  - 3.3.3 *DBT*—dry-bulb temperature
  - 3.3.4 DPM—digital panel meters
  - 3.3.5 ECD—Electrochromic device
  - 3.3.6 IG—insulating glass
  - 3.3.7 *IGU(s)*—insulating glass unit(s)
  - 3.3.8 NIR—near-infrared (radiation)
  - 3.3.9 PTR—photopic transmittance ratio
- 3.3.10  $\tau_H$ —specular (regular) transmittance in the *highest* transmittance state
- 3.3.11  $\tau_L$ —specular (regular) transmittance in the *lowest* transmittance state
- 3.3.12  $T_H$ —time to switch from the highest transmittance state to a transmittance of  $\tau_H 0.8 \ (\tau_H \tau_L)$
- 3.3.13  $T_L$ —time to switch from the lowest transmittance state to a transmittance of  $\tau_L$  + 0.8 ( $\tau_H$ - $\tau_L$ )
  - 3.3.14 TCO—transparent conducting oxide
  - 3.3.15 *UV*—ultraviolet (radiation)

## 4. Significance and Use

- 4.1 EC glazings 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. It is therefore important to understand the relative serviceability of these glazings.
- 4.2 This test method is intended to provide a means for evaluating the relative serviceability of EC Glazings as described in Section 1.
- 4.3 The procedures in this test method include (a) rapid but realistic current-voltage cycling tests emphasizing the electrical properties, and (b) environmental test parameters that are typically used in weatherability tests by standards organizations and are realistic for the intended use of large-area EC IGUs.



#### 5. Apparatus (See Figs. 1 and 2)

5.1 Accelerated Weathering Unit (AWU), consisting of a temperature controlled chamber with properly filtered xenonarc lamps to simulate the spectral power distribution of solar radiation over the UV/visible and NIR wavelength regions (G173).

5.1.1 Fig. 1 shows a top-view schematic diagram of the essential features of the environmental test chamber including the layout of the EC IGUs on a 1220 by 1830 mm test plane, the location of four xenon-arc lamps above the test plane, and the necessary connecting cables from the EC IGUs to the computer-controlled cycling and data acquisition system. Chamber dimension shall be large enough to accommodate all specimens. It has been found that a chamber of size of 2400 mm high by 2650 mm deep by 4480 mm wide has been useful.<sup>4</sup>

5.1.2 The test plane shall be vertically adjustable and the user shall adjust the distance from the lamps to the specimens to obtain the desired light intensity and lateral uniformity within the guidelines of this document. Temperature control within the test chamber shall be provided. Conditions inside the closed space shall be controlled for air temperatures from

20 to 95°C. Humidity within the test chamber shall be monitored and shall not exceed 60 %.

5.1.3 Simulated solar radiation shall be provided by spectrally filtered and water-cooled 6500-W, long-arc xenon arc lamps housed within a reflector system in the ceiling of the test chamber. The lamps shall be suitably filtered to provide a match of an AM 1.5 solar spectrum from 300 to 900 nm (see Note 1). The water-cooled lamps are surrounded by an NIR-absorbing filter, which reduces the heat load. The EC IGU specimens are located on the vertically moveable test plane beneath the xenon arc lamps. The AWU shall have a means for allowing electrical connections to pass from inside to outside the unit to allow temperature monitoring and electrical control of the EC IGUs.

Note 1—At longer wavelengths, the xenon arc emission is at variance with the AM 1.5 solar spectrum because the intensities relative to those in the UV/visible region are higher than in solar radiation. However, this part of the spectrum does not cause photolytically induced degradation.

- 5.2 EC Cycling Unit, for imposing voltage and/or current cycles to alternately and repeatedly change the transmittance of the EC IGUs.
- 5.3 *Electrical Leads*, from the EC cycling unit in 5.2 to each EC IGU in the AWU described in 5.1.
- 5.4 *Computer Controlled Spectrometer*, for obtaining and storing data from the optical characterization of the specimens.
- 5.4.1 *Spectrometer Light Source*, a tungsten lamp or other suitable lamp source that provides illumination from 380 to 780 nm.

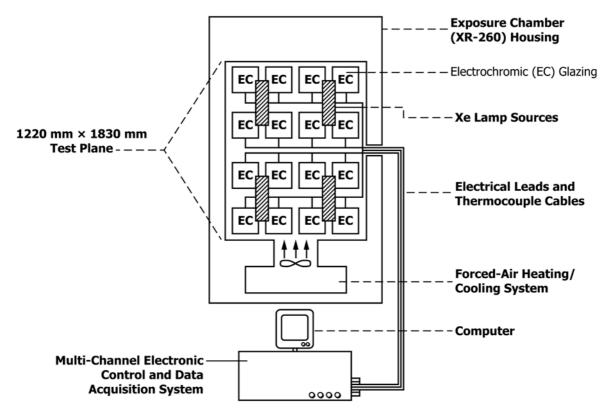


FIG. 1 Top-View Schematic Diagram of the (Essential) Components of an Environmental Test Chamber and Computer-Controlled Electrical Cycling and Data Acquisition System for Accelerated Aging of Electrochromic Glazings

<sup>&</sup>lt;sup>4</sup> The sole source of supply of the apparatus known to the committee at this time is Atlas Material Testing Technology LLC, 4114 North Ravenswood Avenue, Chicago, IL 60613, Phone: +1-773-327-4520 | Fax: +1-773-327-5787, Email: atlas.info@ametek.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

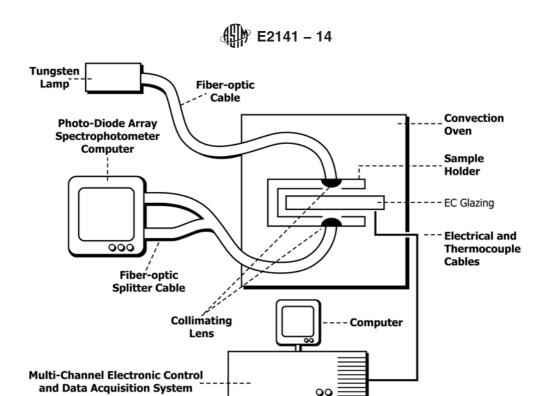


FIG. 2 Schematic of the (Essential) Elements of the Optical Measurement System Used for Recording 300 to 780 nm Transmittance Spectra for a Switching Cycle of EC Glazings at Controlled Temperatures

- 5.4.2 Fiber Optic Cables, which shall be routed from the lamp source into the EC IGU specimen holder and from the EC IGU specimen holder to the spectrometer. One optical fiber guides the incident light from the lamp source to one side of the specimen; another optical fiber guides the transmitted light to the 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.
- 5.5 Computer-Controlled, Multichannel Potentiostat, for switching to and from highest and lowest transmittance states during spectrometer transmittance measurements.
- 5.6 *Oven*, capable of heating the test specimens to the selected test temperature. The oven will be used to carry out optical measurements of the EC-IGUs at the selected test temperature. It shall be large enough for the largest EC IGU to be tested and shall be able to reach the EC IGU testing temperature. The oven must also be designed to permit using the equipment in 5.4 for optical measurements while the EC IGU is maintained at the temperature chosen for testing in the AWU described in 5.1.
  - 5.7 Digital Camera.
  - 5.8 Video Camera and Recorder.
- 5.9 *Verified Thermocouples*, for use to measure specimen and chamber temperatures in the AWU and the oven.

### 6. Test Specimens

6.1 Test specimen size, design, and construction shall be established and specified by the manufacturer, except that the specimens shall be at least 250  $\pm$  6 mm  $\times$  250  $\pm$  6 mm.

6.2 Refer to Specification E2953 for details on specimen quantity and size.

### 7. Procedure<sup>5</sup>

7.1 Overview—The EC IGUs are exposed to simulated solar radiation in a temperature controlled chamber at selected specimen temperatures ranging from 70 to 105°C while the EC IGUs are cyclically switched between low and high transmittance states with the ability to pause during the duty cycles (see Note 2 for further information), depending on the control strategy prescribed by the manufacturer. Accept the prevailing relative humidity in the chamber but ensure that it does not exceed 60 %. The EC IGU specimens are initially characterized optically at room temperature to determine initial performance. Then they are optically characterized in an oven at the selected test temperature in order to determine the time during which the specimens will be in or switching to the lowest transmittance state and the time during which the specimens will be in or switching to the highest transmittance state. This is because some EC products have temperature dependent switching times. The same equipment is used for both room and high temperature optical characterization measurements. After exposure in the AWU, the specimens are again optically characterized at room temperature as they were initially to provide after-aging EC-IGU performance data.

Note 2—Control duty cycle refers to the fraction of the total cycle time

<sup>&</sup>lt;sup>5</sup> The procedure is based in part on the paper by Czanderna, A., et al., in "Optical Materials Technology for Energy Efficiency and Solar Energy Conversion XV," Lampert, C. M.. Granqvist, C., Grätzel, M., and Deb, S. K.. eds., SPIE Vol. 3138, 68 (1997).

over which the specimen has a voltage and/or current applied which reduces the transmittance of the specimen. A 50 % duty cycle means that a voltage and/or current which causes a reduction in transmittance is applied for 50 % of the total cycle time. During the remaining 50 % of the cycle time a voltage and/or current which causes an increase in the transmittance is applied. The applied voltages and/or currents to increase and decrease transmittance are as specified in 7.3.4.

- 7.2 When received, inspect the EC IGUs visually, take photographs of any obvious defects or aberrations of the EC specimens in the highest transmittance state or lowest transmittance state whichever is the unpowered state, and note and record your observations.
- 7.3 Carry out the initial optical characterization of the EC IGUs at room temperature:
- 7.3.1 Measure the optical transmittance between 380 and 780 nm of all EC IGUs at room temperature in the convection oven, as shown schematically in Fig. 2.
- 7.3.2 The convection oven shall be allowed to equilibrate with room temperature for measurements at ca. 22°C. The temperature of the EC IGU shall be monitored with a thermocouple (or other appropriate surface temperature probe or device) attached to the lite that contains the EC layers. The placement should be located on every sample on the center of the outer surface of the glass containing the EC layers with a highly reflective tape (for example, aluminum or silver) having an emissivity close to that of glass.
- 7.3.3 Measure transmittances per the spectrometer manufacturer's instructions ensuring that reference spectra for 100 % and 0 % transmittance are taken before each measurement and using at least 5 nm increments. The sample should be positioned or marked at the center of the outer surface of the lite containing the EC device such that the same spot on the sample is measured before and after exposure.
- 7.3.4 The magnitudes of the voltages and/or currents used for switching the EC glazing between transmittance states, shall be applied as specified by the EC glazing manufacturer.

- 7.3.5 Determine the highest  $(\tau_H)$  and lowest  $(\tau_L)$  transmittance and the switching speed of the EC-IGUs.
- 7.3.5.1 The optical transmittance of the specimen shall be measured over a spectral range covering at least 380 to 780 nm in successive intervals during the process of cycling between highest and lowest transmittance states. 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 EC IGU.
- 7.3.5.2 The photopic transmittance of the devices shall be obtained by integrating the spectra in the wavelength range of 380 to 780 nm using the spectral photopic efficiency Ip  $(\lambda)$  (CIE, 1924) as the weighting factor. The procedure for integration can be found in ISO 9050.
- 7.3.5.3 Allow the samples to reach their lowest transmittance and highest transmittance states during these measurements. Wait for 30 min from the start of the transition for the specimens to reach their extreme states or until the rate of change of transmittance is less than 0.4 %T per min whichever yields the shortest time.
- 7.3.5.4 Plot the transmittance spectra measured during switching as a function of wavelength. Typical transmittance spectra recorded during a full switching cycle are shown in Fig. 3, in which the optical spectra of the glazings are plotted as a function of wavelength.
- 7.4 Use the oven and spectrometer to measure the optical transmittance as a function of time of the specimens at the selected test temperature.
- 7.4.1 The magnitudes of the voltages and/or currents used for switching the EC glazing between transmittance states

<sup>&</sup>lt;sup>6</sup> Kingslake, R., "Applied Optics and Optical Engineering," in Vol. 1, Light: Its Generation and Modification, Academic Press, New York, NY, 1965, Table II, Chapter 1.

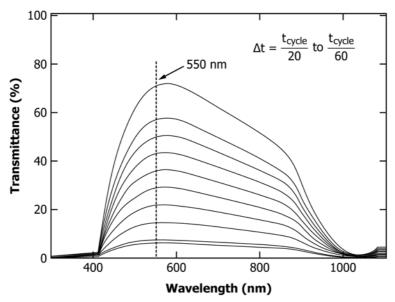


FIG. 3 Example Transmittance Spectra When Transitioning Reversibly Between Highest and Lowest Transmittance States at Intervals Ranging from t<sub>cvcle</sub>/20 to t<sub>cvcle</sub>/60 for an Example EC IGU

during the long-term cycling tests at temperatures ranging from 70 to 105°C, shall be applied as specified by the EC glazing manufacturer.

- 7.4.2 Heat each EC IGU in a convection oven at the specified specimen test temperature. Determine the switching times for obtaining a PTR of at least 5, starting from and returning to the highest transmittance state by measuring the transmittance as a function of time using the spectrometer as described in 7.3.5. For specimens that have a PTR lower than 5, determine the switching time to transition through 85 % of the specimen's dynamic range (where dynamic range is  $\tau_{\rm H}$ - $\tau_{\rm L}$ ).
- 7.4.3 Use these switching times to program the multichannel potentiostat with specific voltage and/or current profiles for cyclic testing at the test temperature in the AWU. The control duty cycle shall be 50 % with a voltage and/or current applied which causes an increase in transmittance or in the highest transmittance state, and 50 % with a voltage and/or current applied which causes a decrease in transmittance or in the lowest transmittance state (see Note 2).
  - 7.5 Set up the AWU.
- 7.5.1 Adjust the lamp to specimen distance to obtain a reading of 1000 W/m<sup>2</sup> over the spectral range 300 to 3000 nm on the digital panel meter (DPM) to represent a 1 sun illumination. Ensure that the uniformity of the illumination at the test plane of the specimens is at least  $\pm 8$  %. See Annex A1 for a description of how to adjust the illumination uniformity.
- 7.5.2 Adjust the chamber temperature to obtain the desired testing temperature of the EC IGU (see 7.7 for definition of testing temperature). For example, with a chamber air temperature of 60°C, the average EC IGU surface temperature reaches a steady-state temperature of about 85°C depending on the specimen size, optical PTR-ratio reached during EC cycling, location of the glazing in the test plane, and the EC IGU construction. Ensure that the relative humidity does not exceed 60 %.
  - 7.6 Mount the EC IGUs in the AWU.
- 7.6.1 Place the EC IGU specimens horizontally onto the test plane. The IGU shall be oriented such that the exterior lite, as installed in the field, is facing the light source. Make suitable electrical connections from the EC cycling unit and data acquisition system to the wires of each EC glazing.
- Note 3—Specimens may be raised from the surface of the test plane to allow for proper air circulation if required to maintain adequate temperature control. The test plane must be lowered an equal distance in order to maintain the proper incident light energy on the lite closest to the light source.
- 7.6.2 Tape thermocouples (0.13 mm diameter) to the center surface of the specimens (to the lite containing the EC) with 8-mm square pieces of 0.05-mm thick aluminum tape. The thermocouple leads may also be taped away from the center of the specimen using up to two additional 8-mm square pieces of aluminum tape to provide strain relief to prevent the thermocouple from being pulled off the glass surface. Mate the thermocouples to thicker extension wires leading to the remote electronics via subminiature connectors. If the EC IGU is oriented with the EC containing lite furthest away from the light source, the thermocouple on that lite shall be shielded from the incident radiation by a 2 by 2 in.-piece of aluminized

tape applied to the surface closest to the lamp array to prevent it from heating up due to absorbing the radiation from the lamp array.

- 7.7 Cycle the EC IGUs in the AWU at elevated temperature and under simulated solar exposure. Note that the "testing" temperature shall be that of the lite containing the ECD in the lowest transmittance state; the average EC IGU temperature will be less because of a typical decrease of about 5°C when the EC IGU is in the highest transmittance state.
- 7.7.1 Before cycling at the test temperature, electrooptically cycle all the EC IGU specimens in the AWU at room temperature to verify the integrity of the electronic control and data acquisition system, as well as the continuity of the electrical and thermocouple connections.
- 7.7.2 With the specimen in the highest transmittance state, activate the Xenon arc lamps and adjust the dry-bulb temperature (DBT) to adjust the chamber air temperature and thus provide adjustment of the specimen temperature.
- 7.7.2.1 The DBT serves as the controlling parameter for the internal forced-air heating and cooling system. It is set at a temperature less than the specified specimen temperature, so as to achieve the latter, which is reached by the combined heat transfer from absorption of the radiant energy of the source and conduction. The specimen temperature depends on the manufactured EC IGU construction, PTR during EC cycling, specimen size, and location in the AWU test plane, as well as whether the EC IGU is in the lowest or highest transmittance state.
- 7.7.2.2 When the EC specimen in the highest transmittance state approaches the desired testing temperature, begin the EC cycling of the EC IGU using the voltage/current profile at the specified test temperature obtained by earlier oven pretesting (7.4). Make minor adjustments to the DBT setting to account for the inevitable rise in temperature of the specimens resulting from absorption in the lowest transmittance state. Specimen temperatures may increase or decrease by several degrees Celsius during cycling the specimen through the lowest and highest transmittance states, respectively.
- 7.7.2.3 Record the specimen temperatures and electro-optic cycling data using the data acquisition system and periodically monitor to ensure proper operation of the AWU, associated apparatus, and the computer.
  - 7.8 Interim Visual and Optical Characterizations:
- 7.8.1 Program the electro-optic cycling of the EC glazings and the functions of the AWU to shut down periodically after 2000 h and 4000 h and finally after 50 000 cycles and at least 5000 h have elapsed. Periodic shutdowns at other times are also allowed. After each shut down, disconnect the thermocouple and electrical leads to the specimen from the cabling, remove the specimens, and re-measure the optical transmittance in the highest and lowest transmittance states at room temperature as in 7.3. (See Note 4.)

Note 4—Because of difference in switching cycle times of different specimen sets based on for example, sample size or material type, exposure times may exceed the minimum of 5000 h in order to achieve 50 000 cycles. Variation in exposure time should be considered when interpreting the results of this test method.

- 7.8.2 Visually inspect the EC IGU specimens and photograph any detectable degradation with the digital camera. Note and record any visually detectable degradation of the specimens in the highest and lowest transmittance states. (See Note 5.)
- Note 5—The visual uniformity tests should be made when the EC IGU is held at a constant transmittance. To establish a given transmittance state for assessing the uniformity of the EC IGU, the manufacturers should be asked to provide control information (voltage and/or current, time) that will result in a constant transmittance of the EC IGU in the lowest and highest transmittance states, and this information should be used.

Note 6—The seals in an IGU can degrade if the test temperature is too high resulting in a visible obstruction to the glass compared to the original sample. In this case, no assessment of the EC glazing shall be made.

- 7.8.3 Record the optical measurements and other observations, and reinsert the EC IGU specimens into the AWU for the next series of cyclic testing, that is, another 2000 h or 1000 h as appropriate.
- 7.8.4 Repeat the procedure in 7.7 and 7.8 until a total of 50 000 switching cycles and at least 5000 h under test conditions are achieved.
  - 7.9 Final Characterization:
- 7.9.1 Final Optical Characterization—After the specimens have amassed 50 000 switching cycles and 5000 h under test conditions, optically characterize the specimens at room temperature following the procedure in 7.3.
- 7.9.2 *Video Documentation*—Mount each EC IGU that has been aged next to an un-tested specimen from the same lot of all those tested. Record the dynamic response for 5 switching cycles between highest and lowest transmittance states using the video camera.
- 7.9.3 Final Visual Inspection—Perform the final visual inspection, take photographs, and record all evidence of visually detectable degradation.

#### 8. Analysis for Performance

- 8.1 *Photopic Transmittance*—Use 3.3 (Light Transmittance) and Table 1 (Weighting Functions) of ISO 9050 to determine the photopic transmittance of the lowest and highest transmittance states.
- 8.2 Switching Speed—Calculate the switching time from the highest transmittance state ( $\tau_H$ ) to the lowest transmittance state ( $\tau_L$ ) and from the lowest ( $\tau_L$ ) to the highest transmittance state ( $\tau_H$ ).

#### 9. Observations

- 9.1 Observe and document the following:
- 9.1.1 Specimen breakage.
- 9.1.2 Specimen degradation that is evident visually in the highest transmittance state.
- 9.1.3 Specimen degradation that is evident visually in the lowest transmittance state.
- 9.1.4 Specimen degradation that is evident from photographs in the highest transmittance state.
- 9.1.5 Specimen degradation that is evident from photographs in the lowest transmittance state.
- 9.1.6 Specimen degradation that is evident from the videos in the highest transmittance state.

9.1.7 Specimen degradation that is evident from the videos in the lowest transmittance state.

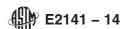
#### 10. Report

- 10.1 The following information shall be provided in the test report.
- 10.1.1 A complete description of the test specimen(s) including size, the make-up of the EC insulating glass unit tested, including a description of the EC pane and the non-EC pane, the cavity dimension and any additional films and/or coatings, product name, code/identifier, manufacturer, date received, date testing was completed.
  - 10.1.2 The number of samples.
- 10.1.3 The orientation of the EC lite with respect to the light source during testing (that is, was it tested in an exterior EC lite or room side EC lite configuration).
- 10.1.4 The photopic transmittance in the lowest and highest transmittance states of the specimen(s) both before and after the accelerated test was conducted.
  - 10.1.5 Switching times  $T_L$  and  $T_H$  before and after test.
- 10.1.6 Additional measurements and analyses performed on the specimen(s) and the results.
  - 10.1.7 The number of switching cycles completed.
  - 10.1.8 The number of hours completed.
- 10.1.9 The average test temperature and range (this is the average of the specimen temperatures in the lowest transmittance states).
  - 10.1.10 Any observations made in Section 9.
- 10.2 Additional information such as technical drawings, photographs, and videos shall be included or referenced in the test report.

#### 11. Precision and Bias<sup>7</sup>

- 11.1 Precision—The precision of this test method is based on an interlaboratory study of ASTM E2141-06, Standard Test Methods for Assessing the Durability of Absorptive Electrochromic Coatings on Sealed Insulating Glass Units, conducted in 2012. A single laboratory participated in this study, testing 14 sealed tungsten oxide-based electrochromic insulating glass units from two manufacturers (five from one manufacturer and nine from another). Every "test result" represents an individual determination. Except for the use of only one laboratory, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:E06-1005
- 11.1.1 Repeatability (r)—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.
- 11.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability

<sup>&</sup>lt;sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E06-1005. Contact ASTM Customer Service at service@astm.org.



conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method

- 11.1.1.2 The repeatability limits are estimated in Table 1.
- 11.1.2 Reproducibility (R)—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material.
- 11.1.2.1 Reproducibility limits could not be determined as there is currently only one laboratory that performs this testing and more than one laboratory is needed to determine reproducibility.
- 11.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.
- 11.1.4 Any judgment in accordance with statement 11.1.1 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results, and the unavoidable sample to sample variation, essentially guarantees

TABLE 1 Change in Percentage Transmittance After Testing to Test Method E2141 (5,000 h, 50 000 Cycles)

	Standard Deviation	Repeatability Limit
Highest transmittance state	1.1	3.2
Lowest transmittance state	0.3	0.7

that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Note that the repeatability limit determined and reported here is a combination of both sample to sample variation and test method repeatability. While all efforts were made to use sample sets which minimized sample to sample variation, based on the experience of the test laboratory, sample to sample variation remains a significant factor in the variation measured. Unfortunately, sample to sample variation cannot be separated from test method variation. Therefore, the repeatability limit reported here is higher than the actual repeatability limit of the test method and represents an upper bound. Consider the repeatability limit as a general guide, and the associated probability of 95 % as only a rough indicator of what can be expected

- 11.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.
- 11.3 The precision statement was determined through statistical examination of 14 results, from a single laboratory, on the sealed units from two manufacturers.

#### 12. Keywords

12.1 accelerated aging; chromogenic glazing; electrochromic glass

#### ANNEX

(Mandatory Information)

#### A1. METHOD FOR MEASURING LATERAL UNIFORMITY OF THE LIGHT INTENSITY AT THE TEST PLANE

A1.1 A description of two suitable procedures for deducing the lateral uniformity of the irradiance in the test plane<sup>6</sup> is given in A1.1.1 and A1.1.2.

A1.1.1 Point by Point Mapping Measurement: A uniformity measurement system (UMS) shall be constructed using a thick aluminum plate as a base that is 610 mm by 1220 mm by 3.2 mm. Sensors shall be mounted in a 305 mm center-tocenter spacing scheme with a 3-row, 5-column configuration. To reduce adverse heating effects, all sensors shall be spaced about 3 mm above the plate to allow for proper air circulation around the entire glazing. The plate itself shall be painted flat black to minimize return reflections. The sensors are connected via a multi-conductor 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  $\pm 3$  % and a maximum uncertainty of  $\pm 5$  %.

A1.1.1.1 The procedure for determining the irradiance in the test plane then involves laying the UMS assembly inside the AWU (that is, 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 multi-conductor data cable through the access port to the chamber. The chamber is then set to operate at 25°C. These are arbitrary operating set points because no defined procedures or specifications exist for calibrating solar simulator lamps. The Li-Cor 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 uniformity across a defined test plane even though the absolute values are not known.

A1.1.1.2 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/m²). 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  $\pm 8\%$ .

A1.1.1.3 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  $\pm 7$  to 8%.

A1.1.2 Five-Point Uniformity Measurement of Solar Irradiance: This is appropriate for use if no changes have been made to the reflective surfaces inside the test chamber and requires the use of a pyranometer that has been calibrated to the ASTM AM1.5 Solar Irradiance Spectrum using an appropriate reference standard.

A1.1.2.1 Fixed pyranometers must be installed in the center and on each corner of the test plane to use this method. For

each fixed pyranometer, position the calibrated pyranometer  $(C_P)$  next to the fixed pyranometer  $(F_P)$ . Turn on all lamps and allow the radiation to stabilize for at least 10 min. Record the voltage output of the  $C_P$  and the  $F_P$ . The calibration factor is then calibrated using the equation:

$$V_{\text{offset}} = V_{\text{cp}} - V_{\text{fp}} \tag{A1.1}$$

Record V<sub>offset</sub> for later reference.

A1.1.2.2 To determine the uniformity, calculate the real irradiance for each of the 5 points using the equation:

$$V_{Calc} = V_{cp} + V_{offset} \tag{A1.2}$$

Calculate uniformity using the following equation:

$$Uniformity = (Max(V_{calc}) - Min(V_{calc}))/Avg(V_{calc}) (A1.3)$$

#### APPENDIX

(Nonmandatory Information)

## X1. ADDITIONAL INFORMATION

# X1.1 Additional Useful Definitions for Terminology Used in This Appendix

X1.1.1 Definitions:

X1.1.1.1 *anomalous hot spot*, *n*—(in the lateral uniformity) is a region of unexpected elevated temperature.

X1.1.1.2 *spectral irradiance, n*—the radiant power per unit area as a function of wavelength, typically reported in watts per square meter per nm,  $(W/(m^2 \cdot nm))$ .

X1.1.1.3 *spectral radiometer, n*—a device for measuring radiation as a function of wavelength in energy or power units.

X1.1.1.4 *uniformity measurement system*—used for measuring the lateral uniformity of the irradiance in the test plane.

X1.1.1.5 X-Y gantry stage, n—refers to an arrangement that moves an object in two perpendicular directions in a test plane.

# X1.2 Additional Data Reporting During the Test Duration

X1.2.1 A report may also be prepared for each set of EC IGUs after each series of switching cycles and optical characterization between test start and final test completion. The report shall provide the initial and final photopic transmittances after each series of switching cycles and accumulated test hours. The reports may provide tables with the appropriate entries for the initial characterization data and the same data

after electro-optical EC cycling, as shown by the simulated data in Table X1.1. The photopic transmittance data may be provided only in the final report. Plots of the voltage and/or current ramps similar to Fig. X1.1, transmittance changes during cycling between states similar to Fig. 3 (but as separate plots), and the transmittance during switching between transmittance states similar to Fig. X1.2 may be provided for each set of EC IGUs tested.

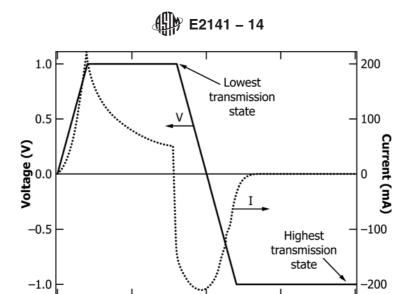
X1.2.2 As with the quantitative data, summarize and include the visual observations for the EC IGUs with the custom report prepared for each supplier. When possible, summarize the observations in general for all EC IGUs and then make appropriate additional comments for each specimen. After final cycling and optical characterization, place each exposed EC IGU side by side with the un-tested specimen (not subjected to exposure in the AWU) and make a video-camera recording to capture visually the effects of any degradation during a switching cycle using the room temperature electro-optical switching protocols.

# X1.3 Description of an Alternate Automated Procedure for Deducing the Lateral Uniformity of Irradiance in the Test Plane

X1.3.1 To map the spectral and lateral uniformity of the light intensity experienced by specimens exposed within the

TABLE X1.1 Optical Test Results at Room Temperature Before and After Exposure in an AWV

Specimen Number	Test Temp (°C)	T <sub>H</sub> (initial) S	T <sub>L</sub> (initial) S	T <sub>H</sub> (after) exposure) S	T <sub>L</sub> (after) exposure) S	% τ <sub>H</sub> Max (initial)	% τ <sub>L</sub> Min (initial)	% τ <sub>H</sub> Max (after exposure)	% τ <sub>L</sub> Min (after exposure)	Cycles at 85 ± 5°C and 1-sun	Hours under Test Conditions
D-1	c1	W1	Y1	X1	Z1	a1	b1	d1	f1	g1	ZZZZ
D-2	c2	W2	Y2	X2	Z2	a2	b2	d2	f2	g2	YYYY
D-3	сЗ	W3	Y3	Х3	Z3	a3	b3	d3	f3	g3	XXXX



Time (arb. units)

FIG. X1.1 Voltage and Current as a Function of Time when Transitioning Reversibly Between Highest and Lowest States for an Example EC IGU

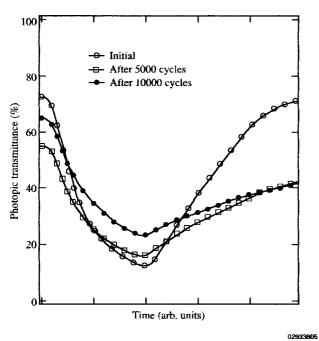


FIG. X1.2 Photopic Transmittance as a Function of Time Measured at Various Stages of Cycling of a Typical EC IGU

Atlas XR-260 exposure chamber, a programmable X-Y gantry stage was constructed that can be assembled and operated inside the chamber. Fig. X1.3 shows the hardware arrangement. There are four 6500 W xenon-arc lamps with a quartz inner filter and CIRA/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 G173.) Specimens are exposed on a vertically adjustable horizontal plane having dimensions roughly 1.2 by 1.8 m. The nominal vertical distance from the light sources to the exposure plane is 770 mm. Each of the 4 lamps is controlled to 0.12 W/(m²·nm)

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 specimen exposure plane without having to turn off the lights, open the doors, and manually move the detector.

X1.3.2 Two types of detectors have been used. The first is a fiber optic probe that couples a mini-integrating sphere detector with a spectral radiometer and allows complete spectral characterization between 300 to 780 nm. The other (faster/more convenient) detector is an Atlas Xenosensitive unit<sup>4</sup> having an integrated spectral response between 300 to 400 nm only. A comparison of the ASTM standard spectrum and the spectral irradiance measured in the specimen plane at the center of the chamber is shown in Fig. X1.4. Very good agreement is seen in the UV part of the spectrum (between 300 to 400 nm), which is generally the part of the solar spectrum that causes the most severe degradation to materials.

X1.3.3 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 affected by the age of the lamps, which is why controlling irradiance at 340 nm, that is, roughly midway within the UV band is the usual practice.) For example, the measured irradiance at 340 nm (Fig. X1.4) is 0.542 W/(m²·nm) and the ASTM standard value is 0.420 W/(m²·nm).

X1.3.4 The lateral uniformity within the XR-260 chamber was mapped by positioning the Xenosensive detector at 96 regular grid positions within the specimen plane (+ signs in Fig. X1.5). Excellent uniformity was found; the irradiance was measured to be  $52.5 \pm 4 \text{ W/m}^2$  between 300 to 400 nm. The anomalous hot spot at x=1000 mm, y=700 mm results from aluminum foil used to shield a cable feedthrough at that location; specimens are not exposed in proximity to this position.

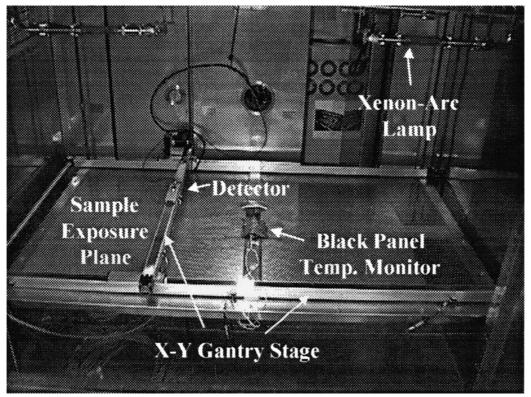


FIG. X1.3 X-Y Gantry Stage Inside XR-260

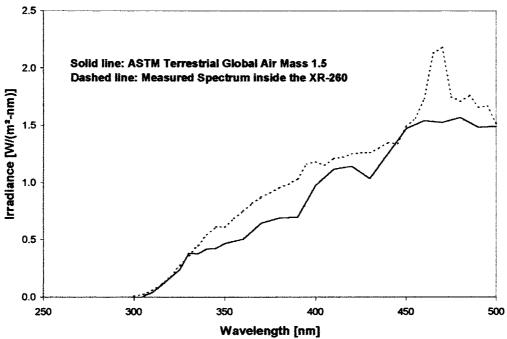


FIG. X1.4 Spectral Irradiance of XR-260 Versus ASTM Standard Terrestial Global Air Mass 1.5 Spectrum

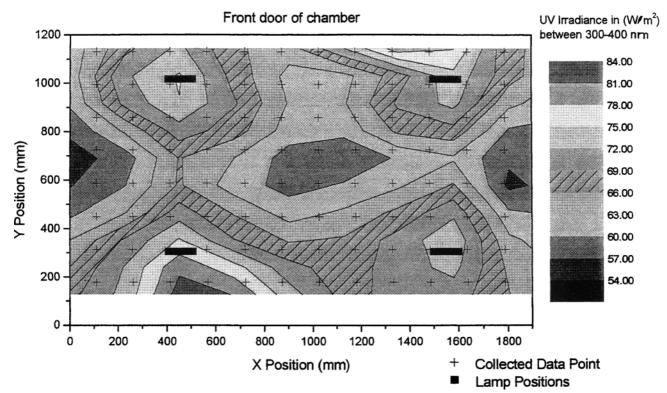


FIG. X1.5 Spatial Distribution of UV Irradiance Within XR-260 Exposure Chamber

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