Designation: E 2241 – 06

Standard Test Method for Assessing the Current-Voltage Cycling Stability at Room Temperature of Absorptive Electrochromic Coatings on Sealed Insulating Glass Units[1]

This standard is issued under the fixed designation E 2241; 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 test described is a method for the accelerated aging and monitoring of the time-dependent performance of electrochromic windows (ECW). Cross sections of typical electrochromic windows have three to five-layers of coatings that include one to three active layers sandwiched between two transparent conducting electrodes (TCEs, see Section 3). Examples of the cross-sectional arrangements can be found in "Evaluation Criteria and Test Methods for Electrochromic Windows."[2] (For acronyms used in this standard, see Appendix X1, section X1.1).

1.2 The test method is applicable only for layered (one or more active 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 glass doors, windows, skylights, and exterior wall systems. The layers 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 will be subsequently exposed (see Test Methods E 2141) to solar radiation and 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, for example, photochromic and thermochromic devices.

1.5 The test method is not applicable to electrochromic windows 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. This test is intended to simulate and, possibly, to also accelerate actual in-service use of the electrochromic windows. Results from this test 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 metric (SI) units are to be regarded as the standard.

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:* [3]

2.1.1 For additional useful standards related to this standard, see Appendix X1, section X1.2.

C 168 Terminology Relating to Thermal Insulation

C 1199 Test Method for Measuring the Steady-State Thermal Transmittance of Fenestration Systems Using Hot Box Methods

E 632 Practice for Developing Accelerated Tests to Aid Prediction of the Service Life of Building Components and Materials

E 903 Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres[4]

E 1423 Practice for Determining Steady State Thermal Transmittance of Fenestration Systems

E 2094 Practice for Evaluating the Service Life of Chromogenic Glazings

E 2141 Test Methods for Assessing the Durability of Absorptive Electrochromic Coatings on Sealed Insulating Glass Units

G 113 Terminology Relating to Natural and Artificial

[1] 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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[2] Czanderna, A. W., and Lampert, C. M., "Evaluation Criteria and Test Methods for Electrochromic Windows," SERI/PR-255-3537, July 1990, Golden, CO; Solar Energy Research Institute.

[3] 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.

[4] Withdrawn.

Weathering Tests of Nonmetallic Materials

2.2 *Canadian Standard:*

CAN/CGSB 12.8 Insulating Glass Units

3. Terminology

3.1 *Definitions*—Refer to terminology in Terminology C 168, Practice E 632, and Terminology 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 photopic optical specular transmittance in the bleached state (τ_b) from that of the photopic optical specular transmittance in the colored state (τ_c).

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 to 730 nm) from that in the bleached state (τ_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, for example, Li^+ or 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 (PTR = τ_b/τ_c) between the bleached (for example, τ_b of 60 to 70 %) and colored (for example, τ_c of 12 to 14 %) states; coloring and bleaching times of a few minutes; switching with applied voltages from ~1 to 3 V; and open-circuit memory of a few hours, for example, 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.

3.3 For additional useful definitions for terminology used in this standard, see Appendix X1, section X1.3.

4. Significance and Use

4.1 This test method is intended to provide a means for evaluating the current-voltage cycling stability at ca. 22°C of ECWs as described in 1.2.[2,5] (See Appendix X1, sections X1.4-X1.7.)

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 (see Practice E 2094). While these two indicators are related, the purpose of this standard test method is to assess the current-voltage cycling stability at ca. 22°C 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 or glazings, or both, 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] Czanderna, A. W., Benson, D. K., Jorgensen, G. J., Zhang, J-G., Tracy, C. E., and Deb, S. K., "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.

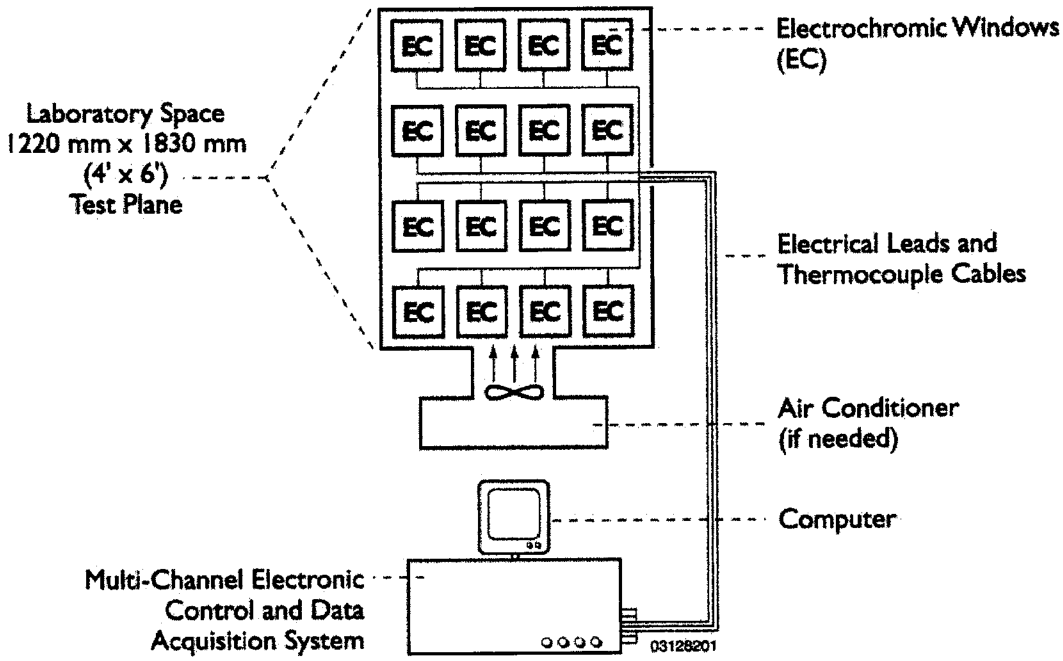


FIG. 1 Top View Schematic Diagram of the Essential Components of the Layout of Electrochromic Window Devices in Laboratory Space and Computer-controlled Electrical Cycling and Data Acquisition System for Accelerated Current-voltage Cycling at ca. 22°C

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 wind; condensation and evaporation of water; and thermal expansion mismatches.[2,5] 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 cavity 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.[2]

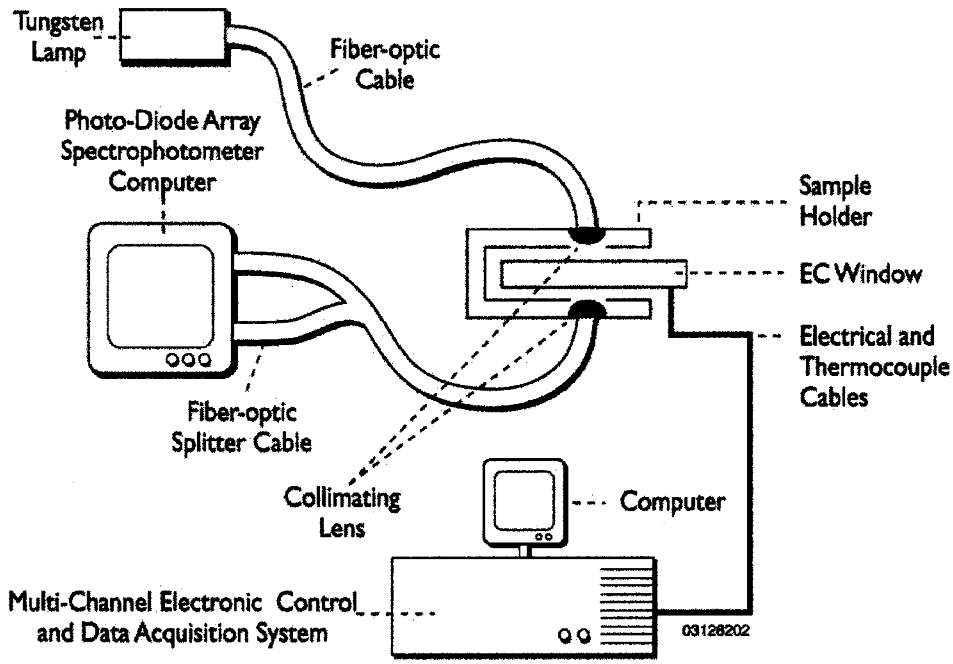
5.4.1 Establishing test procedures from which ECW durability can be predicted and validated for in-service use is an extremely crucial element for the commercialization of ECWs, even for niche markets. To reduce the number of accelerated test parameters that are required to predict the long-term performance of ECWs, accepted procedures or methods have not been established for testing ECWs.[2] However, a rationale was recently proposed for narrowing the number of degradation factors that need to be studied. Establishing the testing criteria from which ECW durability can be predicted and validated based on in service use is an extremely crucial element for the commercialization of ECWs, even for niche markets. 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.[2,5] 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.[2] The purpose of this test method is to assess the current-voltage cycling stability at ca. 22°C of ECWs at least 254 by 254 mm (10 by 10 in.).

NOTE 1—The test method may also be used for smaller ECWs to assess the current-voltage cycling stability at ca. 22°C of prototype devices. The testing parameters chosen may 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.[5]

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

6.1 *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.

6.2 *Computer Controlled Photodiode Array Spectrophotometer*, for example, 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.



NOTE—The measurements are used to determine the photopic transmittance ratio and record electro-optic degradation data after cyclic testing.

FIG. 2 Schematic of the (Essential) Elements of the Electro-optic Measurement System Used for Recording 300 to 1100 nm Transmittance Spectra for a Color/Bleach Cycle of EC Window Devices at Room Temperature

6.3 *Laboratory Space*, that is large enough for the largest ECW to be tested and that maintains the constant ECW testing temperature of ca. 22°C. The space must permit using the equipment in 6.2 for optical measurements while the ECW is maintained at ca. 22°C.

6.4 *Tungsten Lamp*, a spectrum from the source must be compatible with the fiber optic illumination of the photodiode array spectrophotometer described in 6.2.

6.5 *Digital Camera*.

6.6 *Video Camera and Recorder*.

6.7 *Calibrated Thermocouples*.

6.8 *Electrical Leads*, from the unit in 6.1 to each ECW in the laboratory space described in 6.3.

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 254 by 254 mm (10 by 10 in.).

NOTE 2—Consideration should be given to the ultimate requirement for testing specimens that are 355 ± 6 mm by 505 ± 6 mm (14 ± 1/4 in. by 20 in. ± 1/4 in.) such as those used in Test Method E 2188 and for using heat-strengthened or tempered glass (Specification C 1048). Consult Section 5 in Test Method E 2188 and Section 12.1 in CAN/CGSB 12.8 for a description of test specimens and their preparation.

7.2 Six test specimens that are represented to be "identical" shall be the minimum number used to assess the current-voltage cycling stability at room temperature of a particular design and construction.[5] (See Appendix X1, section X1.5.)

7.3 The manufacturer shall provide control parameters and other information that are needed by the testing laboratory for carrying out this test.

NOTE 3—Control parameters for an ECW are the time dependent voltage or current profile that is supplied by the manufacturer of the ECW in which the voltage or current is applied to the ECC for the cyclic coloring to achieve the desired PTR and bleaching of the device.

7.4 The testing laboratory shall retain two of the supplied units as control specimens.

8. Procedure [6]

8.1 *Overview*—Expose the ECWs to a constant temperature of ca. 22°C in the absence of light 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 at ca. 22°C. Accept the prevailing relative humidity in the laboratory space because the prototype EC 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 Test Method 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 ca. 22°C, as shown schematically in Fig. 2. The fiber optic cables are routed from the tungsten lamp source into the ECW sample holder. The temperature of the ECW is established by monitoring the temperature of the room by a thermocouple (or other appropriate temperature probe or device). One optical fiber guides

[6] 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, 1997, p. 68.

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 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, for example, for $t_{cycle} = t_c + t_b$, spectra taken at time intervals between $t_{cycle}/20$ to $t_{cycle}/60$ 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 (τ_b/τ_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

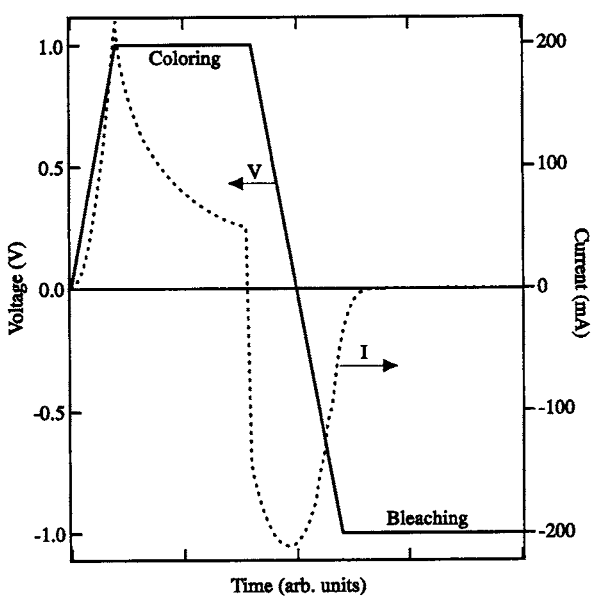


FIG. 3 Voltage and Current as a Function of Time During Coloring and Bleaching Processes for a Typical ECW

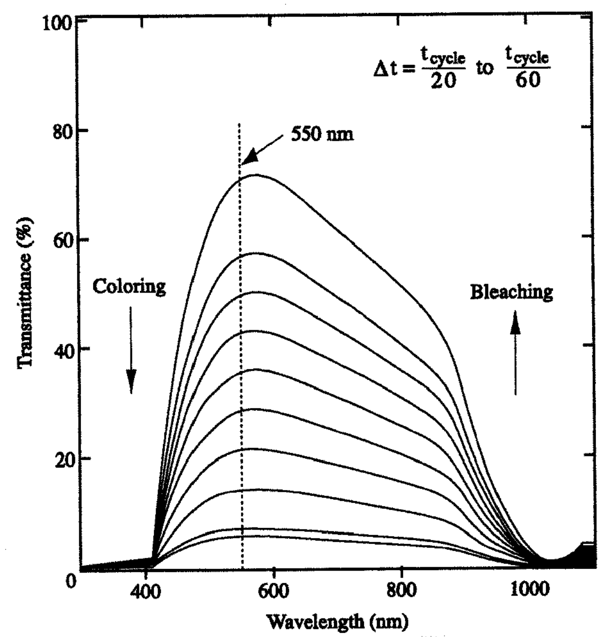


FIG. 4 Transmittance Spectra During a Coloring and Bleaching Process at Intervals Ranging from $t_{cycle}/20$ to $t_{cycle}/60$ for a Typical ECW

spectral photopic efficiency I_p (λ) (CIE, 1924) as the weighting factor[7] (see also, Practice E 1423, Test Method C 1199, and CAN/CGSB 12.8).

8.2.1 A trapezoidal voltage profile similar to the one used at ca. 22°C is also used for the long-term cycling tests at ca. 22°C. Each ECW is maintained at ca. 22°C and the coloring and bleaching times are determined for obtaining the specified PTR, for example, 5 at 550 nm. These data may then used to program the multichannel potentiostat with specific voltage profiles (for each device type) for cyclic testing at the temperatures chosen when using an accelerated weathering unit (AWU) as in Test Methods E 2141. After cycling for the desired time period, (for example, 4000 to 10 000 cycles), the samples are then electro-optically recharacterized at 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 5000 and 10 000 cycles at an elevated temperature, respectively.

8.3 *Laboratory Space*—Fig. 1 shows a top-view schematic diagram of the essential features of using laboratory space for multiple samples including the layout of the ECWs on a 1220 by 1830 mm (4 by 6 ft) test plane and the necessary connecting cables from the ECWs to the computer-controlled cycling and

[7] 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.

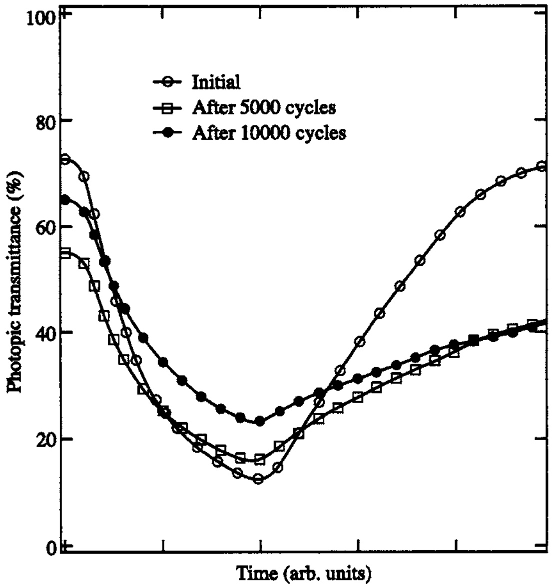


FIG. 5 Photopic Transmittance as a Function of Time Measured at Various Stages of Cycling of a Typical ECW

data acquisition system. Temperature control of the laboratory space is provided and the temperature shall be recorded and reported. Monitoring of the coloring and bleaching processes and of the sample temperatures are accomplished with a computerized electronic control and data acquisition system via cables through access ports in the chamber.

NOTE 4—The sample layout shown in Fig. 1 is only for illustrative purposes, and other sample arrangements are acceptable, for example, stacking the samples vertically.

8.4 *Mounting ECWs in the Laboratory Space*—Each manufacturer of ECWs must provide the coloring and bleaching voltages for characterization at 22°C and for operating their ECWs at 22°C. 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 record your observations. Make electrical connections, for example, 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, for example, 9 m (30 ft) long, leading to the computer-controlled ECW testing electronics. Record successive optical transmittance measurements for color/bleach cycles at ca. 22°C 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 periods of long-term current-voltage cycling. Characterize the samples optically and electrically in the laboratory space at ca. 22°C (see 8.2) which is the intended temperature of testing, to determine the bleaching and coloring times required to achieve a 5:1 PTR at 22°C. This measurement establishes what voltage and time protocols will be used for the accelerated voltage cycling of the samples at ca. 22°C. For example, the ECW samples may be further tested using Test Methods E 2141 by being 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 ca. 22°C using the 22°C (72°F) protocol.

8.4.1 Place the ECW samples horizontally onto the test plane and connect the cables leading to the remote electronics via the connectors, for example, bullet-type, quick-disconnect terminals, described earlier. Tape thermocouples (0.13 mm or 5-mil diameter) to the center surface of the samples (that will face the xenon-arc light source when further testing is done using Test Methods E 2141) 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 5—Before cycling extensively at ca. 22°C, it is prudent to electro-optically cycle all the ECW samples at 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 ca. 22°C*—Program the electro-optic cycling of the EC devices to shut down periodically after a predetermined number of coloring and bleaching cycles; these may be typically 6000 ± 2000 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 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 oven for the next series of cyclic testing, for example, another 4000 to 10 000 coloring and bleaching cycles. Repeat this procedure until a total of 50 000 cycles and at least 5000 hours are achieved or a PTR of less than 4 is obtained at room temperature (ca. 22°C), whichever result comes first. The duty cycle shall be 50 %, with a bleaching voltage applied or in the bleached state, and 50 % with a coloring voltage applied or in a colored state. If a PTR below 4 is reached before 50 000 cycles and 5000 hours are completed or if τ_b of less than 50 % is measured, the ECWs fail the durability test.

NOTE 6—A 50 % duty cycle means that a coloring voltage is applied for 50 % of the total cycle time. During the remaining 50 % of the cycle time a clearing voltage is applied. Since $t_{cycle} = t_c + t_b$ as defined in 8.2, with a 50 % duty cycle, $t_c = 0.5 * t_{cycle}$. The applied coloring and clearing voltages are as specified in 8.2.

NOTE 7—As ECWs age during accelerated life testing (see 8.5), the times to color and bleach usually become longer (see 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 a 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 the longer times to color and bleach, then the device fails this performance criterion.

8.6 *Video Documentation*—After the final cycling series in 8.5, record the dynamic response of the ECWs at ca. 22°C.

Mount each ECW that has been aged as in 8.5 next to an unaged specimen from the same lot of all those tested. Record the dynamic response for 5 coloring and bleaching cycles using the video camera.

NOTE 8—The 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 or bleached state, and this information should be used.

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 (η) 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:

$$\Delta\eta(t) = \frac{\Delta \text{O.D.}}{Q} = \frac{\log\left(\frac{\tau_b}{\tau_c}\right)}{\int_0^{t_c} i(t)\,dt} \quad (1)$$

in which O.D. is the optical density, i is the ion current, t is the time, τ_c and τ_b are as defined in 3.2.2, 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 9—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.

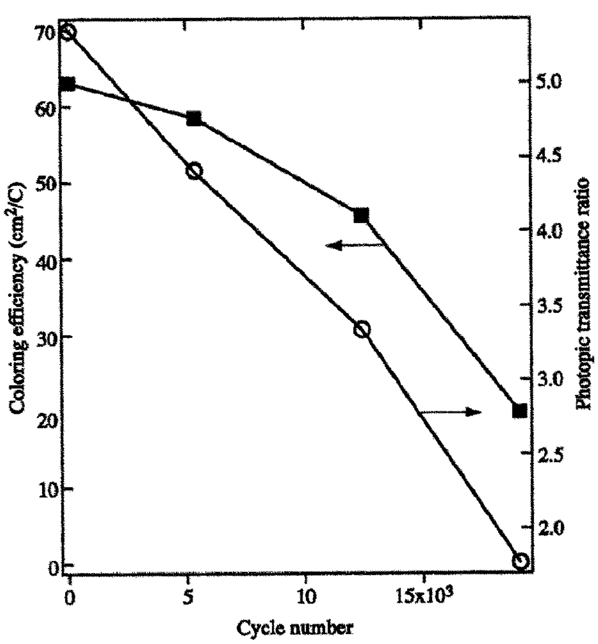


FIG. 6 Photopic Transmittance Ratio and Coloring Efficiency as a Function of Cycle Number for a Typical ECW

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:

$$\tau_b(p) = \frac{\int_{\lambda_{min}}^{\lambda_{max}} \tau_b(\lambda)\, I_p(\lambda)\, d\lambda}{\int_{\lambda_{min}}^{\lambda_{max}} I_p(\lambda)\, d\lambda} \quad (2)$$

in which λ_{max} is 730 nm, λ_{min} is 400 nm, $\tau_b(\lambda)$ is the transmittance in the bleached state at any wavelength, λ, and $I_p(\lambda)$ is 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 (see 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, for example, 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, and

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) including the observations of the as-received specimens,

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, and

11.1.4 The number of dynamic cycles completed before failure occurs, and

11.1.5 The number of hours 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 and accumulated test hours 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, for example, 5 to 10 times longer than used during cycling for characterization at ca. 22°C, to determine the time to reach PTRs of 5 after each series of cycles.

11.4 As with the quantitative data, summarize and include the visual observations for the ECWs in a 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 oven) 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,[5] a great deal remains to be learned about how to carry out durability testing correctly. During the initial efforts,[5,6] additional improvements were identified that urgently need to be made. These include (*a*) using a higher sample temperature for example, 85 and 107°C (185 and 225°F), (*b*) using enhanced irradiance (for example, 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, for example, 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 as a generic problem in long-term testing. Clearly, controlled testing of ECWs for 50 000 cycles and at least 5000 hours 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

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

TABLE 1 Electro-optical Test Results at 22°C (72°F) Before and After Current-Voltage Cycling at Room Temperature

Device Number	V_c (V)	V_b (V)	t_c (s)	t_b (s)	%τ[A] Max	%τ[A] Min	PTR[A]	%τ Max	%τ Min	PTR[A]	Cycles at 22 ± 2°C (70 ± 4°F)	Hours Under Test Conditions
D-1	w	y	x	z	aa	bb	aa/bb	ee	ff	ee/ff	0	ZZZZ
D-1	w	y	x	z	cc	dd	cc/dd	gg	hh	gg/hh	V, VVV	YYYY

[A] PTR is the photopic transmittance ratio % τ_b /% τ_c.

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 η—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 = τ_b/τ_c
X1.1.13 τ_c—specular transmittance in the colored state
X1.1.14 τ_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 Standards Related to This Standard

X1.2.1 *ASTM Standards*[3]:
C 1036 Specification for Flat Glass
E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process
E 546 Test Method for Frost Point of Sealed Insulating Glass Units
E 773 Test Method for Accelerated Weathering of Sealed Insulating Glass Units
E 774 Specification for the Classification of the Durability of Sealed Insulating Glass Units
E 1887 Test Method for Fog Determination
E 2189 Test Method for Testing Resistance to Fogging in Insulating Glass Units
E 2190 Specification for Insulating Glass Unit Performance and Evaluation
G 159 Tables for References Solar Spectral Irradiance at Air Mass 1.5: Direct Normal and Hemispherical for a 37° Tilted Surface[8]

X1.3 Additional Useful Definitions for Terminology Used in This Standard

X1.3.1 *accelerated life testing*—a protocol that results in accelerated aging of materials or devices.

X1.3.2 *anomalous hot spot—in the lateral uniformity*, is a region of unexpected elevated temperature.

X1.3.3 *coloration efficiency*—the change in optical density (OD) per unit of charge (*Q*) inserted into an EC device or material.

X1.3.4 *counter electrode layer*—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.

X1.3.5 *degradation factors*—refer to conditions, imposed or natural, that influence or cause a degradation mechanism, effect, or mode.

X1.3.6 *electro-optic characterization*—refers to the process of recording optical changes (transmittance, reflectance, absorptance, etc.) in an ECW as a function of electrical protocols (voltage, current).

X1.3.7 *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.

X1.3.8 *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 (τ): OD = $-\log_{10}(\tau)$.

X1.3.9 *optical photopic transmittance ratio*—refers to the ratio of the bleached state transmittance (τ_b) to the colored state transmittance (τ_c) where τ_b and τ_c are both weighted by a spectral photopic response curve.

X1.3.10 *optical transmittance*—the ratio of the radiant energy transmitted by a body to the total radiant energy incident upon the body.

X1.3.11 *photodiode array spectrophotometer*—an optical detector system that uses an array of photodiodes coupled to CCDs to facilitate UV-VIS-NIR spectroscopic measurements.

X1.3.12 *trapezoidal voltage profile*—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).

X1.3.13 *spectral photopic response*—refers to the relative response of the human eye in its light adapted state (daylight) to radiation of a given wavelength (~410 to 720 nm).

X1.3.14 *specular transmittance*—refers to the optical transmittance that does not include light with a diffuse component.

X1.4 The suitability of the test methods will be further evaluated as the analyses of test results are completed.

X1.5 The total number of specimens supplied shall be three more than the number listed in 7.2 and shall serve as control specimens or to allow for breakage of two specimens.

X1.6 The test methods are to simulate the in-use conditions of an EC coating in an IGU.

X1.7 The tests may be carried out in parallel or separately.

[8] G 159 replaced E 891 and E 892, which were removed in 1998.