Designation: E 2240 – 02

Standard Test Method for Assessing the Current-Voltage Cycling Stability at 90°C (194°F) of Absorptive Electrochromic Coatings on Sealed Insulating Glass Units[1]

This standard is issued under the fixed designation E 2240; 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:*

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

C 168 Terminology Relating to Building Materials[3]

C 1048 Specification for Heat-Treated Flat Glass-Kind HS, Kind FT Coated and Uncoated Glass[4]

C 1199 Test Method for Measuring the Steady State Thermal Transmittance of Fenestration Systems Using Hot Box Methods[3]

E 632 Practice to Aid Prediction of Service Life of Building Components and Materials[5]

E 903 Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres[6]

E 1423 Practice for Determining the Steady State Thermal Transmittance of Fenestration Systems[7]

E 2094 Practice for Evaluating the Service Lifetime of Chromogenic Glazings[8]

E 2141 Test Methods for Assessing the Durability of Absorptive Electrochromic Coatings on Sealed Insulating Glass Units[8]

[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] *Annual Book of ASTM Standards*, Vol 04.06.

[4] *Annual Book of ASTM Standards*, Vol 15.02.

[5] *Annual Book of ASTM Standards*, Vol 14.04.

[6] *Annual Book of ASTM Standards*, Vol 12.02.

[7] *Annual Book of ASTM Standards*, Vol 04.11.

[8] *Annual Book of ASTM Standards*, Vol 04.12.

E 2188 Test Method for Insulating Glass Unit Performance[8]

G 113 Terminology Relating to Natural and Artificial Weathering Tests of Non-Metallic Materials[5]

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 90°C (194°F) of ECWs as described in 1.2.[2,9] (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 90°C (194°F) 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,

[9] 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.

in some cases, the use, the environmental factors, and other information that influence performance may be known.

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

5.4 Degradation factors (or stresses) for ECWs include the ion insertion and removal processes; temperature; solar radiation (especially UV); water vapor; atmospheric gases and pollutants; thermal stresses such as shock from sudden rain, as well as during the diurnal and annual temperature cycles; electrochemically induced stresses in the multilayer thin-film device; hail, dust, and wind; condensation and evaporation of water; and thermal expansion mismatches.[2,9] 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] 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.[2,9] 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 90°C (194°F) of ECWs at least 254 by 254 mm (10 by 10 in.).

NOTE 1—The seals in IGUs may fail at lower temperatures than those planned for testing, that is, 90°C (194°F). A seal failure will virtually guarantee failure of the ECW coating, so no assessment of the stability of the ECW coating will be made if a seal fails during the test.

NOTE 2—The test method may also be used for smaller ECWs to assess the current-voltage cycling stability at 90°C (194°F) 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.[9]

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.

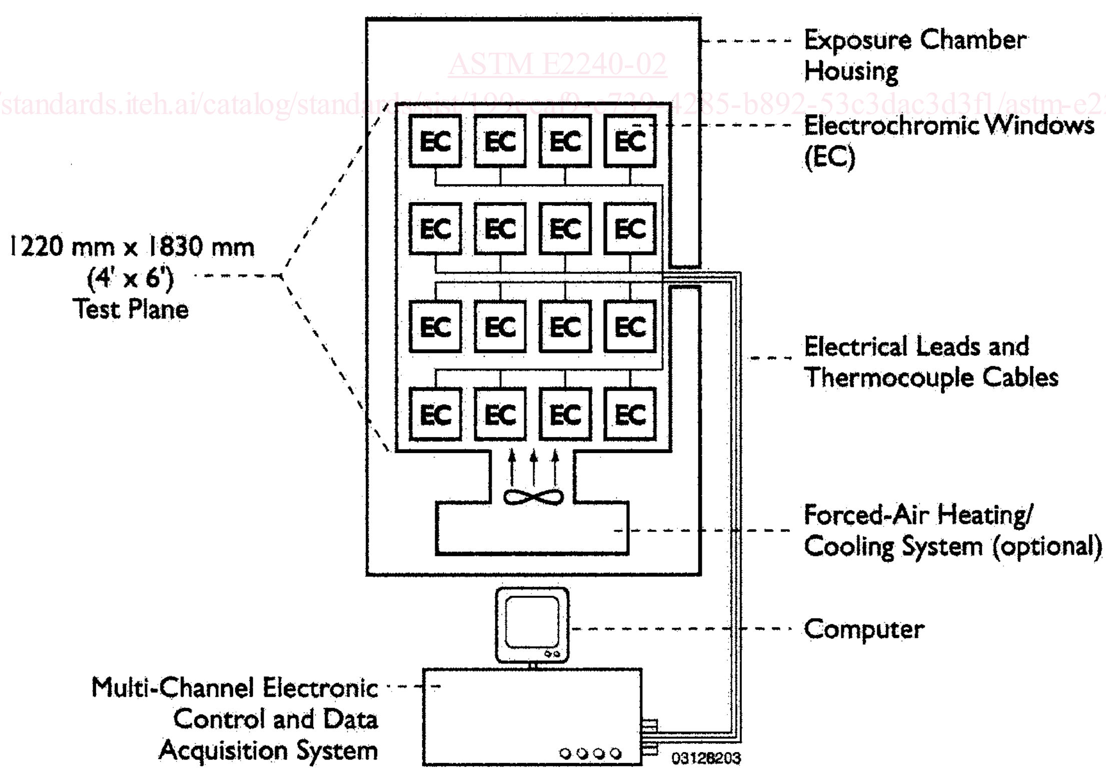
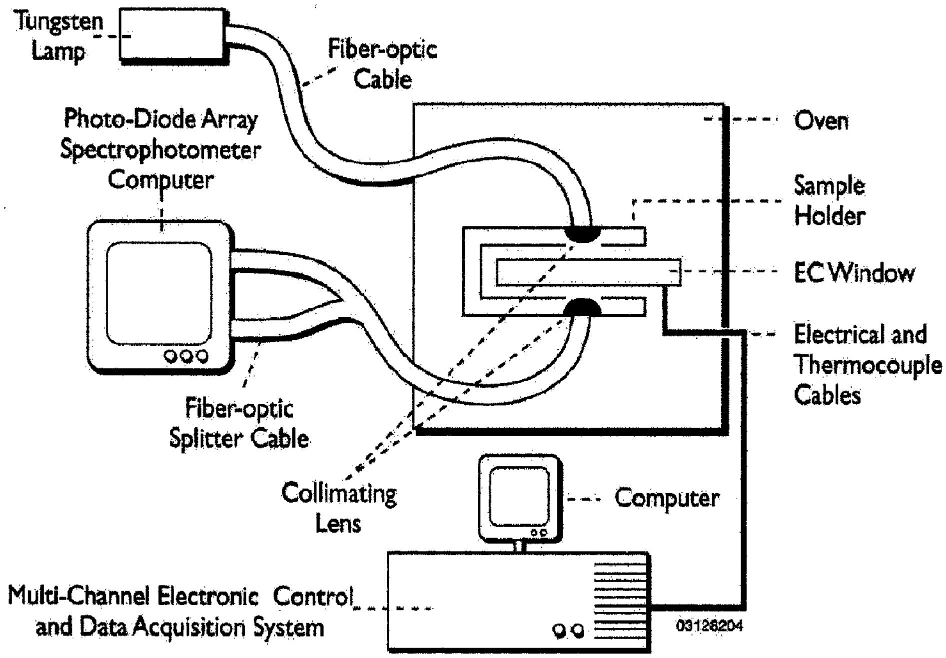


FIG. 1 Top View Schematic Diagram of the Essential Components of an Oven and Computer-Controlled Electrical Cycling and Data Acquisition System for Accelerated Current-Voltage Cycling at 90°C of Electrochromic Window Devices



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 Controlled Temperatures

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.

6.3 *Oven*, that is large enough for the largest ECW to be tested and that can reach the ECW testing temperature. The oven must also be designed to permit using the equipment in 6.2 for optical measurements while the ECW is maintained at the temperature chosen for testing.

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 oven 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 3—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 ± 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 90°C (194°F) of a particular design and construction.[9] (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 4—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 [10]

8.1 *Overview*—Expose the ECWs to a constant temperature of 90 ± 2°C (194 ± 4°F) 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 90 ± 2°C (194 ± 4°F). Accept the prevailing relative humidity in the oven 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

[10] 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.

transmittance of all ECWs is initially measured at room temperature (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 convection oven shown in Fig. 2 is simply allowed to equilibrate with room temperature for measurements at ca. 22°C. The temperature of the ECW is monitored by a thermocouple (or other appropriate surface temperature probe or device) attached to the device surface that will face the sun (see Test Methods E 2141) with a highly reflective tape (for example, aluminum or silver) having an emissivity close to that of glass. One optical fiber guides the incident light from the tungsten lamp to one side of the sample; another optical fiber guides the transmitted light to the photodiode array spectrometer attached to a computer. The fibers shall be optically coupled by properly aligned collimating lens assemblies attached to both the illuminating and the collection fibers. Reference spectra for 100 % and 0 % transmittance are taken before each measurement. The magnitudes of the coloring and bleaching voltages (typically < 3 volts), as specified by the ECW manufacturer, are then applied. To minimize degradation caused by large current surges that occur at the beginning of the coloring or bleaching process, a trapezoidal voltage (ramp rate–0.05 V/s) instead of a step voltage may be used. A typical voltage (V) waveform and the corresponding current (i) are plotted in Fig. 3 as a function of time. The optical transmittance of the sample is measured over an appropriate spectral range in successive intervals during the coloring and bleaching processes. The time interval between the recorded spectra can be as small as one second. In typical testing experiments, a time interval of a fraction of the total cycle time for taking each spectrum should be adequate for recording the optical properties of each ECW, 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 spectral photopic efficiency I_p (λ) (CIE, 1924) as the weighting factor[11] (see also, Practice E 1423, Test Method C 1199, and CAN/CGSB 12.8).

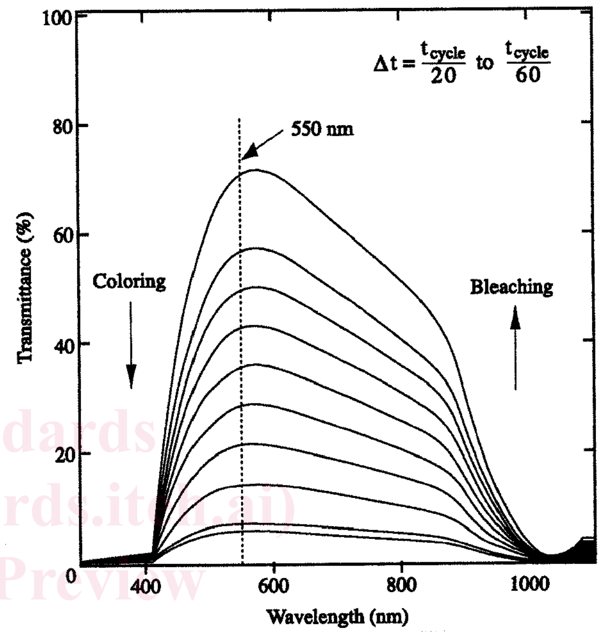


FIG. 4 Transmittance Spectra During a Coloring and Bleaching Process at Intervals Ranging from $t_{cycle}/20$ to $t_{cycle}/60$ for a Typical ECW

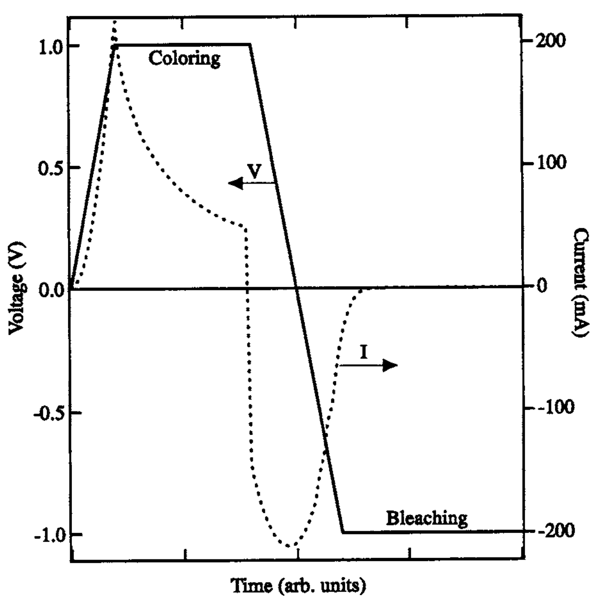


FIG. 3 Voltage and Current as a Function of Time During Coloring and Bleaching Processes for a Typical ECW

8.2.1 A trapezoidal voltage profile similar to the one used at room temperature (ca. 22°C) is also used for the long-term cycling tests at a temperature of 90°C (194°F). Each ECW is heated in a convection oven at 90°C (194°F) 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 cooled to room temperature. The samples are then electro-optically recharacterized at room temperature (ca. 22°C) using the voltage profile determined temperature (ca. 22°C) during the pretest procedure and compared to the initial values as shown in Fig. 4. The initial photopic transmittance for a typical ECW is shown in Fig. 5 as open circles, and the open squares and solid-circles indicate the typical photopic

[11] 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.

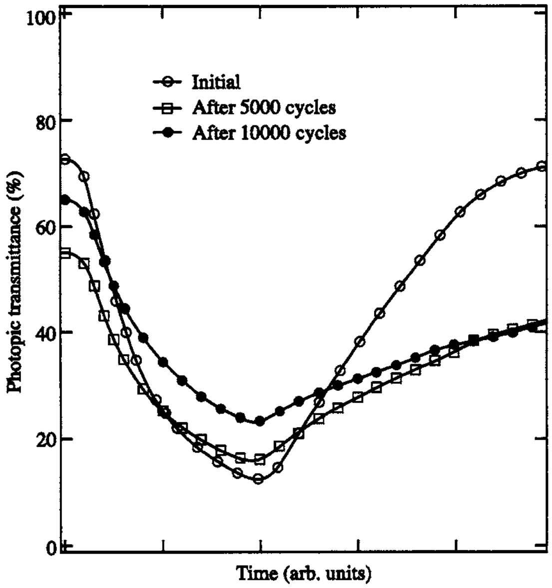


FIG. 5 Photopic Transmittance as a Function of Time Measured at Various Stages of Cycling of a Typical ECW

transmittance of an ECW at 22°C (72°F) after 5000 and 10 000 cycles at an elevated temperature, respectively.

8.3 *Thermal Exposure Chamber (Oven)*—Fig. 1 shows a top-view schematic diagram of the essential features of using an oven with 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 data acquisition system. Temperature control of the oven is provided. 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 5—The sample layout shown in Fig. 1 is only for illustrative purposes, and other sample arrangements are acceptable, for example, stacking the samples vertically on shelves in an oven.

8.4 *Mounting ECWs in an Oven*—Each manufacturer of ECWs must provide the coloring and bleaching voltages for room temperature characterization and for operating their ECWs at 90°C (194°F). 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 room temperature (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 environmental exposure. Characterize the samples optically and electrically in a convection oven (see 8.2) at the intended temperature of testing to determine the bleaching and coloring times required to achieve a 5:1 PTR at 90°C (194°F). This measurement establishes what voltage and time protocols will be used for the accelerated voltage cycling of the samples at the test temperature. For example, the ECW samples may be 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 room temperature (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 6—Before cycling at 90°C (194°F), it is prudent to electro-optically cycle all the ECW samples at room temperature (ca. 22°C) to verify the integrity of the electronic control and data acquisition system, as well as the continuity of the electrical and thermocouple connections.

8.5 *Voltage Cycling the ECWs at 90°C (194°F)*—Program the electro-optic cycling of the EC devices and the oven 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 room temperature (ca. 22°C). Visually inspect the ECW samples and photograph any detectable degradation with the digital camera. Note and record any visually detectable degradation of the samples in the bleached or colored state. Record the electro-optic measurements and other observations, and reinsert the ECW samples into the 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 are achieved or a PTR of less than 4 is obtained at room temperature (ca. 22°C), whichever result comes first. If a PTR below 4 is reached before 50 000 cycles are completed or if τ_b of less than 50 % is measured, the ECWs fail the durability test.

NOTE 7—As ECWs age from exposure to the accelerated weathering (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 an 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 room