**Designation: E 2354 – 04**

An American National Standard

# Standard Guide for Assessing the Durability of Absorptive Electrochromic Coatings within Sealed Insulating Glass Units[1]

This standard is issued under the fixed designation E 2354; 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 This guide provides the recommended sequence for using the referenced ASTM test methods for assessing the durability of absorptive electrochromic coatings (ECCs) within sealed insulating glass units. Cross sections of typical electrochromic windows (ECWs) 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[2] in "Evaluation Criteria and Test Methods for Electrochromic Windows." (For a list of acronyms used in this Standard, see Appendix X1, Section X1.1).

1.2 This guide is applicable only for layered (one or more active coatings between the TCEs) absorptive ECCs on vision glass (superstrate and substrate) areas planned for use in IGUs for 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 ECCs used in this guide will ultimately be exposed (Test Method 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 This guide is not applicable to other types of coatings on vision glass with other chromogenic coatings, for example, photochromic and thermochromic coatings.

1.5 This guide is not applicable to IGUs that will be constructed from superstrate or substrate materials other than glass.

1.6 The test methods referenced in this guide are laboratory test methods conducted under specified conditions.

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

1.8 There is no comparable International Standards Organization Standard.

1.9 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:* [3]

C 168 Terminology Relating to Thermal Insulation

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

E 2188 Test Method for Insulating Glass Unit Performance

E 2190 Specification for Insulating Glass Unit Performance and Evaluation

E 2240 Test Method for Assessing the Current-Voltage Cycling Stability at 90°C (194°F) of Absorptive Electrochromic Coatings on Sealed Insulating Glass Units

E 2241 Test Method for Assessing the Current-Voltage Cycling Stability at Room Temperature (194°F) of Absorptive Electrochromic Coatings on Sealed Insulating Glass Units

E 2355 Test Method for Measuring the Uniformity of an Absorptive Electrochromic Coating on a Glazing Surface

NOTE 1—the following draft standards will be added to this guide after they have been successfully balloted.

E RRR Test Method for Measuring the Stability to Thermal Shock of Sealed Insulating Glass Units with an Operating Absorptive Electrochromic Coating

E ZZZ Test Method for Assessing the Stability in High Humidity and Cyclic Temperature Environments of an Absorptive Electrochromic Coating within Sealed Insulating Glass Units

2.2 *Canadian Standard:*

CAN/CGSB12.8 Insulating Glass Units

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[1] This guide 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*, Solar Energy Research Institute, Golden, CO, July 1990.

[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.

## 3. Terminology

3.1 *Definitions*—Refer to Terminology C 168 for definitions 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 ($\tau_b$) from that of the photopic optical specular transmittance in the colored state ($\tau_c$).

3.2.3 *chromogenic glazing*—is defined in Practice E 2094, but also see Appendix X1, Section X1.3.

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

3.2.5 *control parameters for an electrochromic coating (ECC)*—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 achieving the desired cyclic changes from the bleached state to the colored state and back to the bleached state.

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

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

3.2.8 *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 $H^+$.

3.2.9 *electrochromic window (ECW)*—a device with an ECC 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 result in changes in the solar heat gain, visible transmittance, and U-factor of the window.

3.2.10 *fenestration*—the placement of openings in a building, that is, a window, door, or skylight and its associated interior or exterior elements such as shades or blinds.

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

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

3.2.13 *performance parameters*—the photopic transmittance ratio (PTR), of at least 5:1 (PTR = $\tau_b/\tau_c$) between the bleached (for example, $\tau_b$ of 60 to 70 %) and colored (for example, $\tau_c$ 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.

3.2.14 *sealed insulating glass unit*—is defined in Test Method E 2190 but see also Appendix X1, Section X1.3.

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

3.2.16 *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 guide provides a recommended systematic sequence for using the referenced test methods for evaluating the durability of ECWs as described in section 1.2.[2,4] (See Appendix X1, Section X1.4.)

4.2 This guide provides a summary of the durability issues addressed by each of the series of standards that are necessary for establishing a service lifetime of electrochromic coatings (ECCs) in insulating glass units (IGUs). When fully implemented in buildings in the U.S., ECCs in IGUs have the potential of saving 4 to 5 % of our current energy consumption for all uses—not just buildings. Many of the standards that have been and are being developed for the durability of sealed insulating glass units are clearly relevant and important parts of the long-term national mission of replacing currently used windows with IGUs with ECCs, and these are cited in the referenced standards. IGUs with ECCs will, of necessity, have to be able to pass the applicable standards listed in Appendix X1, Section X1.4, as well as an ASTM standard on wind loading for IGUs. Passing these will not be sufficient because the operating temperatures of ECCs in IGUs is likely to be 90°C (194°F) at the center-of glass, whereas the highest temperature used in Test Methods E 773 or E 2188 is 60°C (140°F). Listings of existing and proposed standards are given in Table 1 and in Appendix X1, Section X1.4.

## 5. Background

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

[4] 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*, National Renewable Energy Laboratory, Golden, CO, May 1997; *Solar Energy Materials and Solar Cells*, 56, 1999, pp. 419-436.

TABLE 1 Recommended Sequence for Using the Referenced or Planned Test Methods or Practice to Address Questions about the Durability or Service Lifetime of ECCs within an IGU

| STM or SP | Qualification or Durability Question Addressed |
|---|---|
| | Stability of the ECC within an IGU |
| E 2355 | Will the ECC in the IGU pass initial uniformity inspection and transmittance measurements in the colored and bleached states? This test method shall also be used to demonstrate if an acceptable uniformity is maintained after the specimens have been subjected to one or more of the accelerated life tests. |
| E 2241 | Can the ECC survive 50 000 current-voltage (coloring/bleaching) cycles at room temperature without a loss in performance below an acceptable level"? |
| E 2240 | Can the ECC survive 50 000 current-voltage (coloring/bleaching) cycles at the anticipated highest operating temperature of 90°C (194°F) without a loss in performance below an acceptable level"? |
| E 2141 | Can the ECC survive 50 000 current-voltage (coloring/bleaching) cycles at 90°C (194°F) in the presence of UV without a loss in performance below an acceptable level"? |
| | Assessing the Durability of the ECC within an IGU and of the Stability and Durability of the IGU |
| E RRR | Will simulate the effect of a sudden rainstorm. Can the ECC/IGU survive (pass) a sudden exposure to a spray of water at 25°C (77°F) when the coating temperature is at 90°C (194°F) at the center-of-glass, just prior to the sudden exposure (no cracking of coatings or seal failures)? |
| E 2188 | Can the ECC in an IGU pass the "Standard Test Method for Insulating Glass Unit Performance" as given in Specification E 2190? |
| E 2189 | Can the ECW in an IGU pass the "Standard Test Method for Testing Resistance to Fogging in Insulating Glass Units" as given in Specification E 2190? |
| E ZZZ | Will the ECC and the IGU survive testing in high humidity at xx°C (yy°F) and 200 thermal cycles between –30°C (-22°F) and xx°C (yy°F)? |
| | Establishing the Service Lifetime of the ECC within an IGU |
| E 2094 | Can a service lifetime be established and what is that lifetime? |

purpose of this guide is to provide a recommended sequence for assessing the durability of absorptive ECCs within sealed IGUs.

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 and reflected 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 guide should be aware of these limitations when reviewing published performance results and their connection to durability.

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

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

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

5.4 Degradation factors (or stresses) for ECWs include the ion insertion and removal processes; temperature; solar radiation (especially UV); water vapor; atmospheric gases and pollutants; thermal stresses such as shock from sudden rain, as well as during the diurnal and annual temperature cycles; electrochemically induced stresses in the multilayer thin-film device; hail, dust, and wind; condensation and evaporation of water; and thermal expansion mismatches.[2,4] These factors may singularly or collectively limit the stability and durability of ECWs. Because the ECWs are expected to have the multilayer of coatings on one of the surfaces in the air space of double-pane or triple-pane IG units with an inert gas fill in the sealed space, many factors such as high humidity, atmospheric gases and pollutants, condensation and evaporation of water, and dust should not affect the durability of electrochromic coatings in IG units.[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, procedures, parameters, and evaluation must be used for assessing ECW stability.[2,4] 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 guide is to provide the recommended sequence for using the referenced ASTM test methods for assessing the durability of absorptive electrochromic coatings (ECCs) within sealed insulating glass units in which the ECC is on an inside glazing surface and of the preassembled permanently sealed IGUs that are at least 250 by 250-mm (10 by 10-in.).

## 6. Procedure

6.1 Study the referenced test methods and practices. Devote special attention to the sections on scope, significance and use, and an overview of each test method. Note especially that the adopted standards Practice E 2094 and Test Method E 2141 address the broad scope of establishing the service lifetime, or for assessing the durability of electrochromic coatings in insulating glass units (IGUs), respectively. The cost of testing will be considerable when implementing either of these standards.

6.2 Note that the expected in service environmental exposures for the ECW will range from temperature extremes from −30°C (−22°F) to 90°C (194°F) at the center-of-glass, relative humidities of up to 95 %, and solar irradiance of up to 1200 W per square meter. For service lifetimes of up to 20 years, it is anticipated an ECW must survive 50 000 cycles from the bleached state to the colored state and back to the bleached state. These criteria dominate the rationale for the selection of the test conditions used in the referenced test methods and practices.

6.3 Note that the need for a series of standards, which will permit manufacturers of electrochromic coatings in insulating glass units (IGUs) to determine if their products are ready for the more comprehensive Test Method E 2141 and subsequently Practice E 2094, has resulted in the adoption of additional standards. These additional test methods may be defined as “qualification” standards, that is, if the product passes a rather simple, less expensive qualification test, then it is ready for the more-comprehensive scrutiny inherent in Test Method E 2141 and subsequent Practice E 2094.

6.4 Refer to Table 1 for a summary of the durability questions addressed from using each of the referenced methods and practices.

6.5 Evaluate what is already known about each particular ECW for which a service lifetime needs to be established.

6.6 Revise the sequence of testing to fit the needs for assessing the durability of any particular absorptive ECC within an IGU.

6.7 Implement the revised sequence of testing.

## 7. Keywords

7.1 chromogenic glazing; durability; electrochromic windows; fenestration; insulating glass units; sealed insulating glass units

# APPENDIX

**(Nonmandatory Information)**

## X1. ADDITIONAL INFORMATION

### X1.1 Acronyms Used in This and Related Test Methods

X1.1.1 ALT = accelerated life testing

X1.1.2 I-V = current-voltage

X1.1.3 ECC (s) = electrochromic coating(s)

X1.1.4 ECW (s) = electrochromic window(s)

X1.1.5 IG = insulating glass

X1.1.6 IGU (s) = insulating glass unit(s)

X1.1.7 IR = infrared (radiation)

X1.1.8 PTR = photopic transmittance ratio or $transmittance_{bleached}/transmittance_{colored}$

X1.1.9 TCE (s) = transparent conducting electrode(s)

X1.1.10 UV = ultraviolet (radiation)

X1.1.11 V = voltage

### X1.2 Additional Useful Standards Related to This Standard

X1.2.1 ASTM Standards (Refer to Practice E 632 and Terminology G 113 for a Description of General Terms):[3]

C 1036 Specification for Flat Glass

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

C 1172 Specification for Laminated Architectural Flat Glass

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

E 122 Practice for Calculating Sample Size to Estimate, with a Specified Tolerable Area, the Average for Characteristic of a Lot or Process

E 546 Test Method for Frost Point of Sealed Insulating Glass Units

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

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

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

E 1887 Test Method for Fog Determination

E 2189 Test Method for Testing Resistance to Fogging in Insulating Glass Units

G 113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials

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

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

X1.3.2 *chromogenic glazing*—a glazing consisting of one or more layers of chromogenic materials, which are able to alter their optical properties in response to a change in ambient conditions such as illumination intensity, termperature, applied electric field, and so forth. The changeable optical properties include transmittance, reflectance, absorptance, and emittance.

X1.3.3 *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.

X1.3.4 *degradation factors*—refer to conditions, imposed or natural, that influence or cause a degradation mechanism, effect, or mode.

X1.3.5 *electro-optic characterization*—refers to the process of recording optical changes (transmittance, reflectance, absorptance, and so forth.) in an ECW as a function of electrical protocols (voltage, current).

X1.3.6 *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.

X1.3.7 *optical photopic transmittance ratio*—refers to the ratio of the bleached state transmittance ($\tau_b$) to the colored state transmittance ($\tau_c$) where $\tau_b$ and $\tau_c$ are both weighted by a spectral photopic response curve.

X1.3.8 *optical transmittance*—the ratio of the radiant energy transmitted by a body to the total radiant energy incident upon the body.

X1.3.9 *photodiode array spectrophotometer*—an optical detector system that uses an array of photodiodes coupled to CCDs to facilitate UV-VIS-NIR spectroscopic measurements.

X1.3.10 *sealed insulating glass unit*—a preassembled unit, comprising lites of glass, which are sealed at the edges and separated by dehydrated spaces(s), intended for vision areas of buildings. The unit is normally used for windows, window walls, picture windows, sliding doors, patio doors, or other types of fenestration.

X1.3.11 *spectral photopic response*—refers to the relative response of the human eye in its light adapted state (daylight) to radiation of a given wavelength in the spectral region of ~410 to 720 nm.

X1.3.12 *specular transmittance*—refers to the optical transmittance that does not include light with a diffuse component.

X1.3.13 *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.

## X1.4 Additional Published Standards under the Jurisdiction of E06.22 (Task Group 22.05)[3]

E 546 Test Method for Frost Point of Sealed Insulating Glass Units

E 576 Test Method for Frost Point of Sealed Insulating Glass Units in the Vertical Position

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 2188 Test Method for Insulating Glass Unit Performance

E 2189 Test Method for Testing Resistance to Fogging in Insulating Glass Units

E 2190 Specification for Insulating Glass Unit Performance and Evaluation

E 2269 Test Method for Determining Argon Concentration in Sealed Insulating Glass Units Using Gas Chromatography