

Designation: G 178 - 03

Standard Practice for Determining the Activation Spectrum of a Material (Wavelength Sensitivity to an Exposure Source) Using the Sharp Cut-On Filter or Spectrographic Technique¹

This standard is issued under the fixed designation G 178; 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 practice describes the determination of the relative actinic effects of individual spectral bands of an exposure source on a material. The activation spectrum is specific to the light source to which the material is exposed to obtain the activation spectrum. A light source with a different spectral power distribution will produce a different activation spectrum.
- 1.2 This practice describes two procedures for determining an activation spectrum. One uses sharp cut-on UV/visible transmitting filters and the other uses a spectrograph to determine the relative degradation caused by individual spectral regions.

Note 1—Other techniques can be used to isolate the effects of individual spectral bands of a light source, for example, interference filters.

- 1.3 The techniques are applicable to determination of the spectral effects of solar radiation and laboratory accelerated test devices on a material. They are described for the UV region, but can be extended into the visible region using different cut-on filters and appropriate spectrographs.
- 1.4 The techniques are applicable to a variety of materials, both transparent and opaque, including plastics, paints, inks, textiles and others.
- 1.5 The optical and/or physical property changes in a material can be determined by various appropriate methods. The methods of evaluation are beyond the scope of this practice.
- 1.6 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.

Note 2—There is no ISO standard that is equivalent to this standard.

2. Referenced Documents

2.1 ASTM Standards:

- ¹ This practice is under the jurisdiction of ASTM Committee G03 on Weathering and Durability and is the direct responsibility of Subcommittee G03.01 on Joint Weathering Projects.
 - Current edition approved July 10, 2003. Published September 2003.

- D 256 Test Method for Determining the Izod Pendulum Impact Resistance of Notched Specimens of Plastics²
- D 638 Test Method for Tensile Properties of Plastics²
- D 822 Practice for Filtered Open-Flame Carbon Arc Exposures of Paint and Related Coatings³
- D 1435 Practice for Outdoor Weathering of Plastics²
- D 1499 Practice for Operating Light- and Water-Exposure Apparatus (Carbon Arc Type) for Exposure of Plastics²
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates³
- D 2565 Practice for Operating Xenon Arc Type Light Exposure Apparatus With and Without Water for Exposure of Plastics⁴
- D 4141 Practice for Conducting Accelerated Outdoor Exposure Tests of Coatings³
- D 4329 Practice for Operating Light- and Water-Exposure Apparatus (Fluorescent UV Condensation Type) for Exposure of Plastics⁵
- D 4364 Practice for Performing Accelerated Outdoor Weathering of Plastics Using Concentrated Natural Sunlight⁵
- D 4459 Practice for Operating an Accelerated Lightfastness Xenon Arc Type Light Exposure Apparatus for the Exposure of Plastics for Indoor Applications⁵
- D 4508 Test Method for Chip Impact Strength of Plastics⁵
- D 4587 Practice for Fluorescent UV-Condensation Exposures of Paint and Related Coatings³
- D 5031 Practice for Enclosed Carbon Arc Exposure Tests of Paint and Related Coatings³
- D 6360 Practice for Enclosed Carbon-Arc Exposures of Plastics⁴
- D 6695 Practice for Xenon-Arc Exposures of Paint and Related Coatings³
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible and Near Infrared Spectrophotometers⁶

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 08.02.

⁵ Annual Book of ASTM Standards, Vol 08.03.

⁶ Annual Book of ASTM Standards, Vol 03.06.

- E 313 Test Method for Indexes of Whiteness and Yellowness of Near-White Opaque Materials³
- E 925 Practice for Periodic Calibration for Narrow Band-Pass Spectrophotometers⁶
- G 7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials⁷
- G 24 Practice for Conducting Exposures to Daylight Filtered Through Glass⁷
- G 90 Practice for Performing Accelerated Outdoor Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight⁷
- G 113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials⁷
- G 147 Practice for Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests⁷
- G 152 Practice for Operating Open Flame Carbon Arc Light Apparatus for Exposure of Nonmetallic Materials⁷
- G 153 Practice for Operating Enclosed Carbon Arc Light Apparatus for Exposure of Nonmetallic Materials⁷
- G 154 Practice for Operating Fluorescent Light Apparatus for UV Exposure of Nonmetallic Materials⁷
- G 155 Practice for Operating Xenon Arc Light Apparatus for Exposure of Nonmetallic Materials⁷

3. Terminology

- 3.1 Definitions given in Terminology G 113 are applicable to this practice.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *incremental degradation*, *n* the increase in degradation in the specimen exposed behind the shorter wavelength cut-on filter of the pair due to the addition of short UV wavelengths transmitted by the filter.
- 3.2.2 *incremental ultraviolet*, *n*—the additional short wavelength ultraviolet transmitted by the shorter wavelength cut-on filter of the pair of sharp cut-on UV/VIS transmitting glass filters. It is represented by the *spectral band* (see 3.2.4).
- 3.2.3 sharp cut-on UV/VIS transmitting glass filters, n—filters that screen out the short wavelengths and transmit radiation longer than the cut-on wavelength. The transmittance increases sharply from 5 %, the cut-on wavelength, to 72 % within a spectral range of about 20 nm. They are also referred to as longpass filters.
- 3.2.4 *spectral band*, *n*—the spectral region defined by the difference in transmittance of a pair of the sharp cut-on UV/VIS transmitting glass filters. It is also referred to as the *incremental ultraviolet*.
- 3.2.5 spectral band pass, n—the spectral range of the spectral band at the delta 20 % transmittance level. It is the spectral range of the incremental ultraviolet mainly responsible for the incremental degradation.
- 3.2.5.1 *Discussion*—The definition of this term differs from that commonly applied to the spectral bandpass, also referred to as the spectral bandwidth, of a narrow band filter or the radiant energy leaving the exit slit of a monochromator. These

- terms are defined as the full width at half-maximum, FWHM, that is, the wavelength range at one half the peak height of the spectral band.
- 3.2.6 cumulative spectral sensitivity curve, n—a plot of the cumulative effect on the optical or physical properties of a material of addition of progressively shorter wavelengths of the source to the longer wavelength exposure with progressive decrease in wavelength of the sharp cut-on UV/visible transmitting filter.

4. Significance and Use

- 4.1 The activation spectrum identifies the spectral region(s) of the specific exposure source used that may be primarily responsible for changes in appearance and/or physical properties of the material.
- 4.2 The spectrographic technique uses a prism or grating spectrograph to determine the effect on the material of isolated narrow spectral bands of the light source, each in the absence of other wavelengths.
- 4.3 The sharp cut-on filter technique uses a specially designed set of sharp cut-on UV/visible transmitting glass filters to determine the relative actinic effects of individual spectral bands of the light source during simultaneous exposure to wavelengths longer than the spectral band of interest.
- 4.4 Both the spectrographic and filter techniques provide activation spectra, but they differ in several respects:
- 4.4.1 The spectrographic technique generally provides better resolution since it determines the effects of narrower spectral portions of the light source than the filter technique.
- 4.4.2 The filter technique is more representative of the polychromatic radiation to which samples are normally exposed with different, and sometimes antagonistic, photochemical processes often occurring simultaneously. However, since the filters only transmit wavelengths longer than the cut-on wavelength of each filter, antagonistic processes by wavelengths shorter than the cut-on are eliminated.
- 4.4.3 In the filter technique, separate specimens are used to determine the effect of the spectral bands and the specimens are sufficiently large for measurement of both mechanical and optical changes. In the spectrographic technique, except in the case of spectrographs as large as the Okazaki type (1),⁸ a single small specimen is used to determine the relative effects of all the spectral bands. Thus, property changes are limited to those that can be measured on very small sections of the specimen.
- 4.5 The information provided by activation spectra on the spectral region of the light source responsible for the degradation in theory has application to stabilization as well as to stability testing of polymeric materials (2).
- 4.5.1 Activation spectra based on exposure of the unstabilized material to solar radiation identify the light screening requirements and thus the type of ultraviolet absorber to use for optimum screening protection. The closer the match of the absorption spectrum of a UV absorber to the activation spectrum of the material, the more effective the screening. The activation spectrum must be determined using a light source

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ The boldface numbers in parentheses refer to the list of references at the end of this standard.

that simulates the spectral power distribution of the one to which the material will be exposed under use conditions. However, a good match of the UV absorption spectrum of the UV absorber to the activation spectrum does not necessarily assure adequate protection since it is not the only criteria for selecting an effective UV absorber. Factors such as dispersion, compatibility, migration and others can have a significant influence on the effectiveness of a UV absorber (see Note 3).

Note 3—In a study by ASTM G03.01, the activation spectrum of a copolyester based on exposure to borosilicate glass-filtered xenon arc radiation predicted that UV absorber A would be superior to UV absorber B in outdoor use because of stronger absorption of the harmful wavelengths of solar simulated radiation. However, both additives protected the copolyester to the same extent when exposed either to xenon arc radiation or outdoors. A research report on the study and interpretation of test results is being written.

- 4.5.2 Comparison of the activation spectrum of the stabilized with that of the unstabilized material provides information on the completeness of screening and identifies any spectral regions that are not adequately screened.
- 4.5.3 Comparison of the activation spectrum of a material based on solar radiation with those based on exposure to other types of light sources provides information useful in selection of the appropriate artificial test source. An adequate match of the harmful wavelengths of solar radiation by the latter is required to simulate the effects of outdoor exposure. Differences between the natural and artificial source in the wavelengths that cause degradation can result in different mechanisms and type of degradation.
- 4.5.4 Published data have shown that better correlations can be obtained between natural weathering tests under different seasonal conditions when exposures are timed in terms of solar UV radiant exposure only rather than total solar radiant exposure. Timing exposures based on only the portion of the UV identified by the activation spectrum to be harmful to the material can further improve correlations. However, while it is an improvement over the way exposures are currently timed, it does not take into consideration the effect of moisture and temperature.
- 4.6 Over a long test period, the activation spectrum will register the effect of the different spectral power distributions caused by lamp or filter aging or daily or seasonal variation in solar radiation.
- 4.7 In theory, activation spectra may vary with differences in sample temperature. However, similar activation spectra have been obtained at ambient temperature (by the spectrographic technique) and at about 65°C (by the filter technique) using the same type of radiation source.

5. Activation Spectrum Procedure Using Sharp Cut-On Filter Technique

- 5.1 Spectral Bands of Irradiation:
- 5.1.1 Select glass types for the sharp cut-on UV/visible transmitting glass filters which provide a spectral shift of approximately 10 nm at 40 % transmittance between filter pairs when ground to appropriate thicknesses. It may be necessary to use filters from more than one source. The exact thickness to which each filter is ground is governed by the incremental ultraviolet transmitted by the shorter wavelength filter of the

pair. Adjust the thicknesses so that the incremental ultraviolet is within 10 % of the average of the incremental ultraviolet of all filter pairs. The method for determining the incremental ultraviolet is described in 5.1.3.

Note 4—Typically, 12 or 13 filters with cut-on wavelengths ranging from 265 to 375 nm are used to determine the effects of 10 spectral bands, each approximately 20 nm wide, in the solar UV region. A larger set of filters can be used to reduce the width of each spectral band, but it would extend the time required to produce degradation by each of the spectral regions. The filter size is normally 2 by 2 in., but other sizes up to 6 by 6 in. can be used.

Note 5—The spectral transmittance curves of a typical set of filters are shown in Figs. X1.1 and X1.2 in the Appendix.

Note 6—Due to variations in the melt of each glass type, the filter types and thicknesses used for one filter set may not be applicable to other sets.

5.1.2 Spectral Transmittance Data:

- 5.1.2.1 Use a UV/visible spectrophotometer that produces either digital data or an analog curve to measure the spectral transmittance of each filter from the spectral region of complete blocking at the short wavelength end to maximum transmittance at the long wavelength end.
- 5.1.2.2 Determine the wavelength calibration and linearity of the spectrophotometer as described in either Practice E 275 or E 925. Check the 0 % and 100 % baselines and adjust, if necessary, according to manufacturer's recommendations. If the 100 % baseline is not flat in the spectral region in which the filters are measured, correct the data. In the case of analog curves, use sufficient chart expansion to allow accurate transmittance values to be read from the chart at 2 nm intervals.
 - 5.1.3 Incremental Ultraviolet:

5.1.3.1 From Digitized Data:

(1) The delta % transmittance for each filter pair and resultant spectral bands can often be obtained instrumentally when using a computerized spectrophotometer for the digitized data

5.1.3.2 From Analog Data:

- (1) Tabulate the % transmittance of each filter at 2 nm intervals and calculate the delta % transmittance for each pair at each wavelength tabulated by subtracting the % transmittance of the longer wavelength cut-on filter from that of the shorter wavelength cut-on filter.
- (2) For each filter pair, plot the delta % transmittance versus wavelength on linear graph paper to produce a spectral curve referred to as the incremental UV. It represents the added portion of the ultraviolet transmitted by the shorter wavelength filter of the pair.

Note 7—Fig. X1.3 is a plot of delta % transmittance from analog data of the transmittance of filters 5 and 6 shown in Fig. X1.1.

5.1.3.3 *Effective Incremental Ultraviolet*:

(1) Determine the areas of the spectral bands above the delta 10 % transmittance line by any suitable technique, including computerized calculations based on digitized data or such as by use of a planimeter or other technique for curves based on analog data. Take the average of at least two measurements by any of the techniques. The areas are used to obtain the normalization factors described in 5.1.4.

Note 8—Using the area of the spectral band above delta $10\,\%$ transmittance instead of the full spectral band for the effective incremental

ultraviolet gives the most meaningful comparison of the incremental ultraviolet for all filter pairs. Since some of the spectral bands for the long wavelength pairs of filters are very broad below the delta 10 % level, inclusion of these areas in the normalization step would require greater adjustment of the degradation caused by the shorter wavelengths. It would result in an apparent greater sensitivity of the material to shorter wavelengths.

5.1.4 *Normalization Factors*:

5.1.4.1 Calculate the normalization factors by dividing the average of all the measured areas by the area measured for each filter pair.

Note 9—Normalization factors are used to adjust the measured incremental degradation so that it represents the effects of equal spectral portions of the radiation. Although the filters are designed to provide nearly equal areas above the delta $10\,\%$ transmittance level for all filter pairs, the areas are not identical. This step can be omitted if refinement of the measured data is not important for the application.

Note 10—The normalization factors for the filter set shown in Figs. X1.1 and X1.2 are given in Fig. X1.7. The areas above 10 % transmittance were obtained by counting squares of the curves plotted on linear graph paper.

5.1.5 Spectral Band Pass:

5.1.5.1 The spectral band pass represents the spectral range mainly responsible for the degradation caused by the incremental ultraviolet radiation defined by each filter pair. It is the wavelength range of the spectral band at delta 20 % transmittance. Fig. X1.3 shows the spectral band pass for the incremental UV of filter pair 5/6. It ranges from 317 nm to 338 nm.

Note 11—The spectral band pass is approximately the width at half the peak height for most of the spectral bands. Thus, it is related to, but not identical, to the spectral bandwidth of narrow band filters or radiant energy leaving the exit slit of a monochromator (see the Discussion in 3.2.5).

5.2 Change in Filter Characteristics:

5.2.1 Obtain transmittance measurements of the filter sets periodically to determine if solarization has occurred. Until further information on the rate of solarization is available, check the transmission of the filters after a maximum of 5,000 h exposure when used for solar radiation or for light sources that simulate solar UV spectral irradiance and after a maximum of 2,000 h when used for light sources that emit shorter UV wavelengths than solar radiation.

Note 12—After more than two years of nearly constant exposure of a filter set to radiation in a borosilicate glass-filtered (B/B) 6500 watt water-cooled xenon arc type exposure device plus several months exposure in a single enclosed carbon arc type exposure device, there was no detectable change in transmission.

- 5.2.2 Use of the filters with sources of radiation such as the quartz-filtered xenon or mercury arcs or the unfiltered open flame carbon arc is not recommended without prior investigation of their rates of solarization by these sources.
- 5.2.3 It is not necessary to check the full spectral transmission curves of the filters if the original transmittance at two wavelengths in the region between 10 and 20 % transmittance has not changed by more than 1.5 % absolute. If the change is greater than 1.5 %, obtain full spectral curves with determination of the ultraviolet incremental data of the filter pairs and normalization factors. If any of the normalization factors exceed 15 %, discontinue use of the filter set. Complete

recharacterization of the entire filter set is required following attempts to reverse solarization by heat treatment or other means.

- 5.2.4 If possible, use the same spectrophotometer for transmission measurements before and after use since differences in the band passes of spectrophotometers can alter transmittance values in the sharp cut-on spectral region.
- 5.2.5 Document the exposure history of each filter set, recording the type of exposure source, its filters, irradiance level, exposure time and, if measured, radiant exposure and spectral region in which it is measured. Document the filter transmission data versus exposure history to determine the monitoring frequency required. Any attempt at reversing solarization by heat treatment and its success or failure shall also be documented.
- 5.2.6 If one or more filters in the set requires replacement because of solarization or damage, replace with a filter that has the same transmission properties as the original.

5.3 Specimens:

- 5.3.1 *Number of Specimens*—A set of replicate specimens equal in number to the filters in the set are the minimum number required for a single test. However, unless duplicate specimens can be exposed behind each filter, repeat the test and produce the activation spectrum based on the average of the two separate tests. Expose one or more additional specimens unfiltered for estimating exposure time required for the set (see 5.4.3).
- 5.3.2 Specimen Size and Markings—The maximum specimen size is limited by the size of the filters. The minimum specimen size is limited by the size required for optical or physical property measurements. The specimen is scribed with the filter number in an unexposed area on the front surface, that is, the surface exposed to the light source, or on the back surface. Follow the procedures described in Practice G 147 for identification, conditioning and handling of specimens prior to, during and after exposure.

5.3.3 Mounting of Specimens:

- 5.3.3.1 One or more specimens are mounted behind each of the filters. If multiple specimens are required for replicate analyses and if the size required is smaller than the filter size, a single filter of each type may be adequate for the procedure. For example, 5 microtensile bars having 3/8 in. maximum width can be mounted behind a 2 by 2 in. filter.
- 5.3.3.2 It is desirable to allow a gap of about ½16 in. between the specimen and filter to prevent etching of the filters by corrosive samples. Shims or other means can be used to separate the sample from the filter. A larger air gap is needed for forced air cooling of the specimens such as during exposure to concentrated sunlight. However, caution is advised against excessive separation of the filter and specimen to avoid irradiating the specimen with unfiltered light through the sides unless baffles are used to prevent it.
- 5.3.3.3 To prevent unfiltered light from irradiating the specimens from the back side, an opaque backing is used which has a non-reflective black coating on the side toward the sample. Black paper supported by a stiff backing has been found to be suitable. The unfiltered specimens are backed and mounted identically to the filtered specimens.

5.4 Exposure Procedure:

5.4.1 Test Sources—Various types of exposure sources routinely used for stability testing are applicable to determination of activation spectra and cumulative spectral sensitivity curves by the sharp cut-on filter technique. The only limitations are avoidance of water spray and condensation on the filters during the test and solarization of the filters by sources emitting high intensity radiation in the short wavelength ultraviolet region. The following are some applicable exposure tests described by ASTM practices:

Direct outdoor exposure: D 1435; G 7
Outdoor exposure under glass: G 24
Concentrated natural sunlight: D 4141; D 4364; G 90
Xenon arc exposures: D 2565; D 4459; D 6695; G 155
Enclosed carbon arc exposures: D 5031; D 6360; G 153
Filtered Open flame carbon arc exposures: D 822; D 1499; G 152
Fluorescent UV exposures: D 4329; D 4587; G 154

5.4.2 Placement of Specimens:

- 5.4.2.1 The filtered specimens plus one or more unfiltered specimens are placed in sample holders suitable for the laboratory accelerated test device or for outdoor exposure.
- 5.4.2.2 The sample holders are placed in the laboratory test device in positions that assure uniformity of irradiance on the surface of the filters. The irradiance shall not vary by more than 10 % from the average irradiance on all filters. The specimens are exposed in the absence of water spray or condensation.
- 5.4.2.3 Outdoor exposures may be carried out at any exposure angle normally used for samples or on an equatorial mount or a Fresnel reflector apparatus.

5.4.3 Length of Exposure:

- 5.4.3.1 The set of filtered specimens is exposed until the incremental degradation by most of the spectral bands, that is, the difference in degradation in the two specimens exposed behind the filter pairs, can be determined with sufficient statistical significance. For some materials, particularly as thin specimens, spectral bands longer than about 360 nm may have a negligible effect.
- 5.4.3.2 The exposure time can be estimated from the time required to produce a change in the unfiltered specimen that is 5 to 10 times larger than the change required for statistical significance by the spectral bands that cause most of the degradation. For example, for most polymeric materials, the time to produce a 10 unit change in yellowness index in the unfiltered specimen yields a well defined activation spectrum showing a change of several yellowness index units caused by the spectral bands that have the greatest actinic effect.
- 5.4.3.3 If statistically significant data for differences in degradation in the pairs of samples exposed behind the filter pairs cannot be obtained in a practical exposure time, the exposure shall be at least of sufficient length to provide data for a cumulative spectral sensitivity curve (see 8.3).

5.4.4 Cleaning of Filters:

- 5.4.4.1 Clean filters at appropriate intervals during each test. Daily cleaning is recommended for outdoor tests unless it is a "behind glass" test. During laboratory accelerated tests, examine the filters at least weekly and clean when the surface of the filters is visibly coated.
- 5.4.4.2 Cleaning can be accomplished with a mild detergent solution on a cotton swab followed by alcohol and final buffing to visible clarity with a lint-free cloth.

5.4.4.3 When specimens are removed for measurement, clean both sides of the filters. Prior to cleaning, examine the side of the filters opposite the specimens for any exudate from the specimens. If the filters are visibly coated and the deposit from the test specimens has caused a change in transmission of the filters, the test must be repeated with more frequent cleaning of the backside of the filters.

6. Activation Spectrum Procedure Using Spectrographic Technique

6.1 Type of Spectrograph:

- 6.1.1 Select a spectrograph with sufficient optical speed for adequate intensity of the spectrally dispersed radiation on the sample and adequate separation of the spectral bands at the focal plane so that each is incident on a section of the sample wide enough to allow for measurement of the degradation of interest by the individual bands.
- 6.1.2 While either a prism or grating spectrograph can be used, the latter is preferable because it disperses the spectral bands linearly. Thus, it gives a more accurate representation of the relative degradation caused by the different spectral bands during exposure to the polychromatic radiation of the source. The prism spectrograph spectrally disperses shorter wavelengths more than longer wavelengths, resulting in fewer wavelengths per mm at the short wavelength end. Therefore, the short wavelength spectral bands will have a smaller effect relative to the effect of the long wavelength bands than they do under field exposures.

Note 13—Spectrographs used for activation spectra have ranged from a small double quartz prism type that disperses spectral bands between 280 and 410 nm across a 10 mm section of the sample (2) to the Okazaki Large Spectrograph (OLS) with a double blazed plane grating that projects wavelengths between 250 and 1000 nm onto a 10 meter long focal plane (1). The wavelength dispersion of the former ranges from 10 nm/mm at 280 nm to 30 nm/mm at 410 nm. The wavelength dispersion of the OLS is about 0.8 nm/10 mm.

- 6.2 Exposure of Specimen(s) to Spectrally Dispersed Radiation:
- 6.2.1 Using a spectrograph other than a large one, such as the OLS, place a single specimen in the focal plane where it is exposed to the dispersed radiation of a light source that is focused on the entrance slit of the spectrograph. In the OLS or similar spectrograph, place individual specimens at appropriate positions on the focal curve to expose it to the spectral bands of interest.
- 6.2.2 Identify the wavelength positioning on the focal plane of the smaller spectrographs by a suitable technique and transcribe it to the irradiated portion of the specimen. A technique that had been used successfully exposed a photographic plate with the same dimensions as the specimen in the focal plane to the well-defined wavelengths of a low pressure mercury arc focused on the entrance slit of the spectrograph (2). In the case of the large spectrograph, identify the spectral band that irradiates each specimen.
- 6.2.3 Expose the specimen(s) until the changes produced by the actinic spectral bands are sufficient to produce a well-defined activation spectrum.

7. Measurement of Degradation

7.1 Measure the changes in optical and/or physical properties of the materials resulting from exposure to the individual spectral bands. The applicable techniques depend on the size of the specimen area to be measured. Exposures using the sharp cut-on filters and the large spectrograph provide sufficient size areas exposed to each spectral band for mechanical as well as optical property changes to be determined. Generally, smaller spectrographs only allow for determination of optical property changes.

Note 14—Methods used to measure property changes by different spectral bands incident on a single specimen are given in Refs (3) and (4).

7.2 Some ASTM methods applicable to measurement of changes in the exposed specimens include color difference (Test Method D 2244), yellowness index (Test Method E 313), tensile properties (Test Method D 638), impact resistance (Test Method D 256), and chip impact strength (Test Method D 4508).

8. Data Analysis

- 8.1 Activation Spectra by the Sharp Cut-On Filter Technique:
- 8.1.1 Determine the difference in the measured change in property for the two specimens exposed behind each pair of filters in the set. This is the incremental degradation, that is, the degradation due to the added portion of shorter wavelength radiation transmitted by the shorter wavelength filter of the pair, the incremental ultraviolet radiation.
- 8.1.2 Multiply the incremental degradation by the normalization factor for each filter pair to obtain the adjusted incremental degradation for each pair of specimens.
- 8.1.3 Plot the adjusted incremental degradation versus the spectral band pass of the filter pair to produce an activation spectrum in bar graph form. The wavelength scale in nm increases linearly from left to right.
- 8.1.4 Alternatively, plot the adjusted incremental degradation versus the midpoint of the spectral band for each filter pair and connect the points to produce an activation spectrum as a smooth curve.

Note 15—An example of an activation spectrum based on increase in yellowness index of a polysulfone film exposed to the xenon arc with daylight filters is shown in Fig. X1.4. The data used to graph the activation spectrum is given in Fig. X1.8. The filter set differed from the one shown in Figs. X1.1 and X1.2. Filter pair 7-8 was not used because it does not meet the criterion for filter pairs, that is, a shift of about 10 nm between the two curves at 40 % transmittance. For the same reason, the longer wavelength filters that were paired were not necessarily the two filters having sequential numbers.

Note 16—The spectral energy absorbed from the source by the polysulfone film is superimposed on the activation spectrum in Fig. X1.4. For this polymer the profiles are similar, showing that the wavelengths most strongly absorbed are responsible for most of the yellowing. However, for many polymeric materials the spectral sensitivity to the light source differs from the spectral energy absorbed because degradation is a function of both the energy absorbed and quantum efficiencies of the degradation processes initiated by the absorbed wavelengths.

- 8.2 Activation Spectra by the Spectrographic Technique:
- 8.2.1 Determine the change in optical or mechanical property caused by each of the isolated spectral bands and plot the

data versus wavelength of the midpoint of the spectral band to produce the activation spectrum.

Note 17—Fig. X1.5 is an example of an activation spectrum obtained by the spectrographic technique. It represents the wavelength sensitivity of a film of polycarbonate exposed to xenon arc radiation through a daylight type filter based on measurement of increase in absorbance at 300 nm, 350 nm, and 400 nm. The latter is a measure of yellowing.

- 8.3 Cumulative Spectral Sensitivity Curve Using Sharp Cut-on Filters:
- 8.3.1 When exposure in a practical time frame does not produce statistically significant differences in degradation of the specimens exposed behind pairs of filters, plot the measured property change in each specimen versus the 10 % transmittance wavelength of the filter behind which the sample was exposed. It represents the cumulative increase in degradation of the material with addition of progressively shorter wavelengths. It provides information on the long wavelength limit of the radiation responsible for the degradation.

Note 18—Fig. X1.6 is an example of cumulative spectral sensitivity curves of the increase in yellowness index and decrease in chip impact strength of a 100 mil thick specimen of unstabilized acrylonitrile-butadiene-styrene (ABS) exposed to xenon arc radiation through daylight filters.

9. Report

- 9.1 Report the following information:
- 9.1.1 Description of sample including type of material, and any non-proprietary identifying information, such as the source of the material, the presence and type of additives, processing history, thickness of specimen and any differences between the front and back of specimen.
 - 9.1.2 Activation spectrum technique used.
- 9.1.3 For the sharp cut-on filter technique report the following:
- 9.1.3.1 Band passes at delta 20 % transmittance for the filter pairs and the normalization factors.
- 9.1.3.2 Type of mounting, including distance between specimens and filters and backing used for specimens.
 - 9.1.3.3 For outdoor exposure report the following:
- (1) Type of exposure, that is, direct, behind glass, heated or unheated black box and air and black panel temperature, if controlled, angle and direction, geographic location and approximate latitude.
- (2) The dates the test was started and completed and any elapsed down time, total solar radiation dose in MJ/m² and total ultraviolet dose below 385 nm in MJ/m², if obtained.
- 9.1.3.4 For exposure in artificial weathering devices report the following:
 - (1) Type and model of exposure device.
- (2) Type of light source and associated filters, if used, age of source and filters, if applicable.
- (3) Operating conditions including cycles of light and dark, relative humidity and black panel temperature.
- (4) Type of sample rack and placement of specimens with respect to light source.
- (5) Duration of exposure and elapsed down time, UV irradiance in $W/(m^2 \cdot nm)$ and radiant exposure in $kJ/(m^2 \cdot nm)$ at 340 nm, if available.

- 9.1.3.5 Type of degradation measured, referencing an ASTM or other applicable method. Refer to report section of ASTM or other standard used for report requirements.
- (1) The measured property change in each specimen and the difference in property change in the two specimens exposed behind each pair of filters that define a spectral band. Also, if the degradation data is normalized, report the adjusted data.
 - 9.1.4 For the spectrographic technique, report the following:
- 9.1.4.1 Type of spectrograph and spectral dispersion at the focal plane. For a prism spectrograph report the dispersion at the short and long wavelength limits of exposure.
- 9.1.4.2 The spectral range of wavelengths incident on the specimen and the width of the irradiated portion.
 - 9.1.4.3 Type of light source and filters, if any.
 - 9.1.4.4 Duration of exposure.
- 9.1.4.5 Method of identifying the position of the spectral bands on the specimen.

- 9.1.4.6 Type of degradation measured and method of measurement.
- 9.1.5 The activation spectrum either in bar graph form or as a smooth curve.

10. Precision and Bias

- 10.1 *Precision*—The repeatability and reproducibility of results obtained according to this practice will vary with the materials tested, the material property measured and the specific exposure conditions. No precision statement is applicable to this practice.
- 10.2 *Bias*—Bias can not be determined because no acceptable standard weathering reference materials are available.

11. Keywords

11.1 activation spectrum; cumulative spectral sensitivity curve; degradation; light exposure; sharp cut-on filter technique; spectrographic technique; ultraviolet radiation

APPENDIX

(Nonmandatory Information)

X1.

X1.1 The following figures show filter spectral transmittance curves, the incremental ultraviolet of a filter pair, activation spectra by the sharp cut-on filter technique and spectrographic technique, cumulative spectral sensitivity

curves and tabulated data for activation spectra.

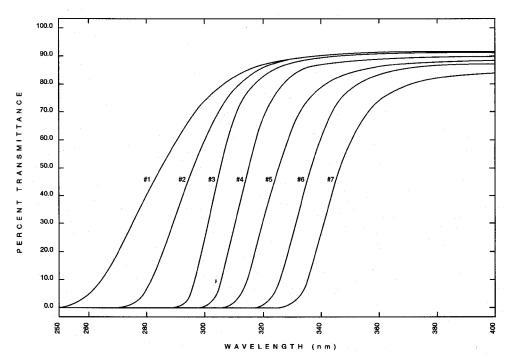


FIG. X1.1 Transmittance Curves of Sharp Cut-On UV/VIS Transmitting Filters 1 through 7 of a Typical Set

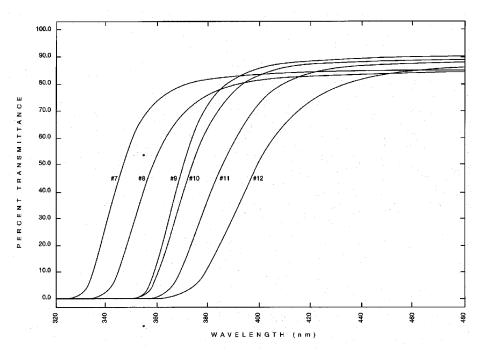


FIG. X1.2 Transmittance Curves of Sharp Cut-On UV/VIS Transmitting Filters 7 through 12 of a Typical Set

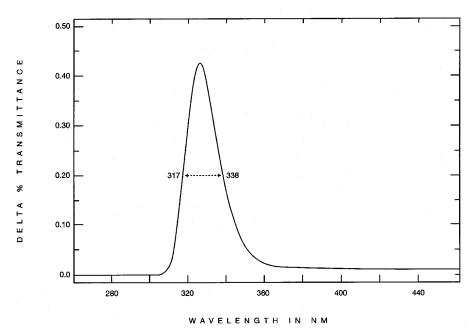
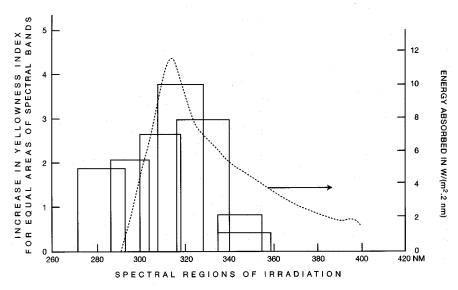


FIG. X1.3 Delta % Transmittance (Incremental UV) of Filters 5 minus 6 Shown in Fig. X1.1



(BAR WIDTH = SPECTRAL BAND AT DELTA 20% TRANSMISSION OF FILTER PAIRS)

FIG. X1.4 Activation Spectrum of Yellowing of 2 mil Polysulfone Film Exposed Behind Sharp Cut-On Filters to Xenon Arc Radiation
Through Daylight Type Filters Compared with the Spectral Energy Absorbed by the Polymer

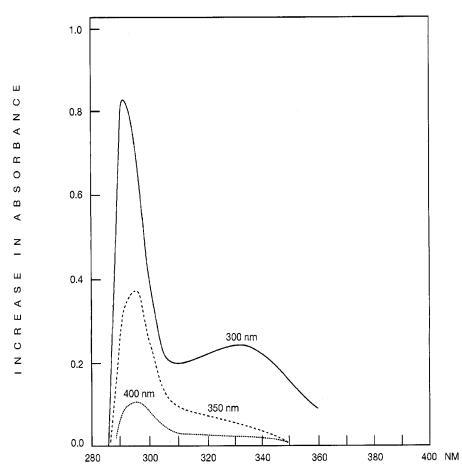


FIG. X1.5 Activation Spectrum of Increase in 300nm, 350nm, and 400nm Absorbance of a Polycarbonate Film Exposed to Xenon Arc Radiation Through Daylight Type Filters Spectrally Separated by a Quartz Prism Spectrograph

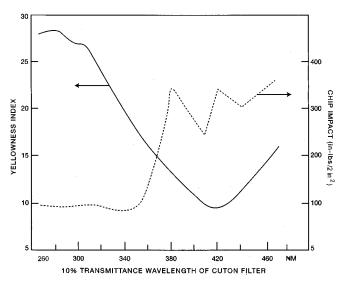


FIG. X1.6 Cumulative Spectral Sensitivity to Yellowing and Reduction in Impact Strength of a 100 mil Plaque of Unstabilized ABS with Progressive Addition of Short Wavelengths of Xenon Arc Radiation Through Daylight Type Filters with Decrease in Cut-On Wavelength of UV/Visible Transmitting Filter

Filter Pair	Band Pass at Delta 20% Transmittance	Area Above Delta 10% Transmittance (# Squares)	Normalization Factor# (39.6/area)
1 minus 2	23.5 nm (271-294.5 nm)	40.6	0.98
2 minus 3	19.0 nm (286-305 _{nm})	40.2	0.99
3 minus 4	18.0 nm (299-317nm)	37.7	1.05
4 minus 5	21.0 nm (308-329 _{nm})	40.4	0.98
5 minus 6	21.0 nm (317-338nm)	43.3	0.91
6 minus 7	20.0 nm (329-349 _{nm})	37.0	1.07
7 minus 8	21.5 nm (338.2-359.7nm)	38.4	1.03
8 minus 9	21.0 nm (348-369 _{nm})	42.9	0.92
10 minus 11	22.0 nm (365-387nm)	34.9	1.13
11 minus 12	26.0 nm (376-402 _{nm})	40.8	0.97

[#] Used to adjust the measured incremental degradation to incremental degradation based on equal areas above delta 10% transmittance for all filter pairs.

FIG. X1.7 UV Spectral Band Passes and Normalization Factors for Filter Pairs of the Filter Set Shown in Figs. X1.1 and X1.2

Filter No.	40% Trans.	<u> YI*</u>	Filter Pair	Delta YI	Normaliz. <u>Factor</u>	Delta YI Adjusted	Spectral Band Pass at Delta 20% Trans
2	(NM) 282	20.8	2-3	1-7	1.10	1.9	272 - 293 rm
3	292	19.1	3-4	1.9	1.08	2.1	286 - 304 rm
4	302	17.2	4-5	2.4	1.13	2.7	300 - 318 rm
5	313	14.8	5-6	3.9	0.97	3.8	308 - 329 rm
6	322	10.9	6-7	3.5	0.87	3.0	317 - 340 rm
7	332.5	7.4	0-7	3.5	0.07	3.0	317 - 340 1111
8	339	6.5	0.0	0.0	4.40	0.0	005 055
9	349.4	5.9	8-9	0.6	1.10	0.8	335 - 355 rm
11	362	5.6	9-11	0.3	0.87	0.3	344 - 367 rm
12	370.4	5.5	11-13	0.1	1.01	0.1	359 - 381 rm
13	372	5.5	12-14	0	1.00	0	366 - 391 rm
14	382	5.4					
15	395.5	5.5	14-15	-0.1	1.01	-0.1	377 - 402 rm

Exposed 12 hours

FIG. X1.8 Data Used for the Activation Spectrum of Polysulfone Shown in Fig. X1.4

REFERENCES

- (1) Watanabe, M., et. al., "Design and Performance of the Okazaki Large Spectrograph for Photobiological Research," *Photochem. Photobiol.*, 36, 1982, pp. 491-498.
- (2) Searle, N. D., "Activation Spectra of Polymers and Their Application to Stabilization and Stability Testing," *Handbook of Polymer Degra*dation, 2nd Ed., S. H. Hamid, Ed., Marcel Dekker, NY, 2000, Chap. 16
- (3) Fischer, R. M., et. al., "Analytical Studies of Spectrally Dispersed Polyethylene-naphthalate," *Polymer Preprints*, 83, ACS Nat'l Meeting, August 2000, pp. 136-139.
- (4) Hirt, R. C., Searle, N. D., and Schmitt, R. G., "Ultraviolet Degradation of Plastics and the Use of Protective Ultraviolet Absorbers," SPE Transactions, 1, No. 1, 1961, pp. 21-25.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

YI = Yellowness Index of Polysulfone

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).