Designation: G156 – 17

Standard Practice for Selecting and Characterizing Weathering Reference Materials[1]

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1. Scope

1.1 This standard describes the criteria to be used for selection of a weathering reference material (WRM) and procedures to be used for determining within lab and between lab tolerances of changes in measured properties of weathering reference materials. This standard also describes a procedure for comparing different lots of the same type of a weathering reference material.

NOTE 1—Examples of laboratory accelerated tests in which a weathering reference material could be used to monitor consistency are exposure tests such as those described in Practices G152, G153, G154, and G155and other standards in which tests conducted according to these standards are referenced. Examples of outdoor exposures where a weathering reference material could be used to monitor consistency are those conducted according to Practices G7, G24, or G90. A reference material can also be used to monitor consistency of exposure or conditioning test that do not involve exposure to light.

1.2 Weathering reference materials are most often used to (1) monitor consistency (that is, repeatability, reproducibility, or both) of exposure tests, (2) to determine the time or radiant exposure at which test materials are evaluated, (3) as a reference material for comparing to test materials exposed at the same time. Weathering reference materials cannot be used to classify or characterize the relative severity of any exposure test because of the large variability in material responses to the effects of light, heat, and water.

1.3 This practice does not cover control materials which, by definition are selected to be of similar composition and construction to the test materials, and are exposed at the same time as test materials.

1.4 This practice provides an outline of experiments required to determine how the measured properties of the reference material change as a function of exposure to specified test conditions. It includes establishment of reproducible measurement procedures, determination of the critical spectral region in the light source causing the changes, and effects of other critical exposure stresses such as temperature and moisture.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

E1169 Practice for Conducting Ruggedness Tests

G7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials

G24 Practice for Conducting Exposures to Daylight Filtered Through Glass

G90 Practice for Performing Accelerated Outdoor Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight

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

G152 Practice for Operating Open Flame Carbon Arc Light Apparatus for Exposure of Nonmetallic Materials

G153 Practice for Operating Enclosed Carbon Arc Light Apparatus for Exposure of Nonmetallic Materials

G154 Practice for Operating Fluorescent Ultraviolet (UV) Lamp Apparatus for Exposure of Nonmetallic Materials

G155 Practice for Operating Xenon Arc Light Apparatus for Exposure of Non-Metallic Materials

G178 Practice for Determining the Activation Spectrum of a Material (Wavelength Sensitivity to an Exposure Source) Using the Sharp Cut-On Filter or Spectrographic Technique

2.2 *SAE Standard:*

SAE J2527, Accelerated Exposure of Automotive Exterior Materials using a Controlled Irradiance Water-Cooled Xenon Arc Apparatus[3]

[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.

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[2] 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] Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

SAE J2412, Accelerated Exposure of Automotive Interior Materials Using a Controlled Irradiance Water-Cooled Xenon Arc Apparatus[3]

3. Terminology

3.1 *Definitions*—The definitions listed in Terminology G113 are applicable to this standard.

4. Significance and Use

4.1 Weathering reference materials are used in laboratory accelerated exposure tests to verify consistency of tests run within the same instrument at different times (repeatability) or in different instruments or different laboratories (reproducibility), using the same exposure conditions, or both. Specifications defining consistency of exposure conditions are based on the property change of a reference material after a defined period of time. Some weathering reference materials are used to define periods of exposure. Specifications recommending the use of these materials require the material to be exposed until a defined change in the weathering reference material is achieved. Specifications are usually based on results for a single lot of the weathering reference material. When a new lot of the reference material is introduced, round-robin studies are necessary to compare the new and old lots and to establish appropriate limits for expected performance of the new lot.

NOTE 2—An example of the use of a clear polystyrene reference standard for this purpose is given in SAE J2412 and SAE J2527.

NOTE 3—Some weathering reference materials (for example blue wools) are also used to define periods of exposure. Although not specifically covered by this standard, the procedures described for characterizing a reference material used to monitor consistency of exposures are also generally applicable to characterizing reference materials used to define periods of exposure.

4.2 It is important to test the consistency of exposure in the laboratory accelerated device with a weathering reference material that responds to the test conditions similar to the way the test materials respond. Therefore, the weathering reference material should be sensitive to the spectral region of the light source mainly responsible for producing degradation in the test materials to provide the most meaningful evaluation of exposure test consistency. The weathering reference material should also provide information on consistency of temperature and humidity conditions if the latter are important factors in degradation of the test materials.

NOTE 4—Material homogeneity can also be an important factor in selection of a weathering reference material, particularly if weathering is initiated by the radiation absorbed by impurities as is the case in aliphatic type polymers exposed to radiation longer than 300 nm.

4.3 The measurement of the characteristic property of a weathering reference material can be subject to error depending on the instrument and the procedure used to measure the property. It is important to use measurement procedures that are clear and which minimize chances for operator misinterpretation. It is also important to determine the level of variability caused by measurement of the characteristic property.

4.4 When a reference material is used to monitor or specify the consistency of an exposure test, it is important that any specification limits defined by changes in the reference material be based on a sound statistical analysis of results from a properly designed round-robin experiment. This practice provides a procedure which can be followed to set up the round-robin, analyze results, and establish reasonable limits of change in the characteristic property of the reference material that can be used in specifications.

4.4.1 The results obtained according to this practice are valid only for the exposure cycle used for the round-robin and cannot be applied to the same weathering reference material used in different exposure cycles.

4.5 The change in characteristic property of a reference material may be affected by the placement of the reference material in the exposure device. This is often due to variations in light intensity and temperature within the allowed exposure area. Random placement of replicate specimens of the weathering reference material throughout the allowed exposure area provides an indication of the uniformity of conditions within the exposure area.

NOTE 5—In some cases, procedures require exposure of a weathering reference material at a specific location within the exposure device or chamber. Results for a reference material used in this way may not provide an accurate representation of the exposure conditions in other positions within the device.

5. Procedure

5.1 Select a weathering reference material that exhibits a significant change in a characteristic property when exposed for an acceptable period of time, to the exposure conditions described in the applicable test procedure.

5.2 Determine an evaluation procedure that can best measure the change in characteristic property of the reference material.

5.2.1 Conduct a series of experiments to determine the effect of important factors in the measurement procedure. It is recommended that this be done using a ruggedness test according to Guide E1169 to determine which factors significantly affect results. The results from this test can be used to tighten the test measurement procedures.

5.2.2 When the results from the ruggedness testing are complete, write a set of instructions for measuring the characteristic property that is unambiguous and clearly understood by operators who will be making the measurement. Have two operators in one laboratory conduct the measurement on at least three replicate specimens of the reference material. Interview the operators to determine whether the procedure provides clear and easy to understand directions. Modify the procedure to remove any ambiguity in instructions.

5.3 Determine the homogeneity of the weathering reference material by measuring the property change of randomly selected replicate specimens that have been exposed to very tightly controlled test conditions, or by appropriate chemical analysis techniques such as ultraviolet and/or infrared spectroscopy.

5.4 For any new weathering reference material, determine the response of the reference material to critical exposure stresses. These results are important to determining whether

the material being considered is appropriate for monitoring the specific conditions of the exposure test being used. Tests to determine the materials response to exposure stresses should be done in a single laboratory or a series of laboratories agreed upon by all interested parties.

5.4.1 Conduct experiments to determine the spectral region of the light source to which the characteristic property is most sensitive, i.e. the activation spectrum of the weathering reference material. This can be accomplished by techniques to isolate the effects of individual narrow bands of the actinic region of the light source. It is recommended that several determinations of the activation spectrum be conducted.

NOTE 6—Practice G178 describes two procedures for determination of activation spectra of polymers.

5.4.2 Conduct experiments to determine how the rate of change of the characteristic property of the reference material is affected by temperature. This can be done by exposing a series of reference material specimens to the light source of choice at a series of temperatures (with irradiance and humidity held constant).

5.4.3 Conduct a series of experiments to determine how the rate of change of the characteristic property of the reference material is affected by moisture. This can be done by conducting exposure tests at constant light intensity and temperature where the time of wetness or relative humidity is varied.

5.4.4 A factorially designed experiment can be used to determine the effects of moisture and humidity. Use at least two levels of temperature and relative humidity in the designed experiment conducted at a constant light intensity.

5.5 The procedure for conducting the exposure test in which the weathering reference material is used should clearly specify the levels of light intensity, optical filters used with the light source, chamber and insulated/uninsulated black panel temperature, and moisture conditions. These conditions must be specified for each different light/dark period used in the exposure cycle chosen.

5.6 Determine the stability of the measured property of the reference material after it has been removed from the exposure. The post exposure stability of the characteristic property can be determined by measurements made at a series of times after removal from the exposure. Typically, these measurements will be made at successively long time intervals. Some materials (for example polysulfone) show a continuing change in measured property after they are removed from the exposure. If the characteristic property shows a change after being removed from the exposure, the instructions for use must clearly specify the time after exposure when the characteristic property is to be measured. It is strongly recommended that the characteristic property of the reference material be reasonably stable after removal from the exposure test.

5.6.1 Property measurements must be made during the period when the weathering reference material is stable after being exposed.

NOTE 7—Tests to determine wavelength sensitivity do not provide information about the effects of temperature, moisture, or variation in irradiance. Variations in these other exposure stresses may alter the results of test used to determine wavelength sensitivity.

NOTE 8—In general, weathering reference materials that are stable for at least 10 days after removal from exposure will be the most useful.

5.7 After selecting the reference material, characteristic property, property measurement procedure, and the desired exposure conditions, set up a round-robin study to determine the level of within lab repeatability and between lab reproducibility for the change in property of the reference material after specific exposure periods.

5.7.1 Conduct the round-robin according to Guide E691. At least six laboratories should participate in the round robin.

5.7.2 Prepare a clear set of instructions for all round-robin participants. The instructions should cover specimen placement in the exposure device, including how the reference material is to be mounted in specimen holder, exact exposure cycle to be used, the exact procedure for measuring characteristic property, and how data will be recorded.

NOTE 9—Round-robin studies can be designed to determine reproducibility and repeatability limits for a candidate weathering reference material.[4]

5.7.3 At least three exposure periods should be used for the round-robin, with separate sets of the reference material to be used for each exposure period. Use at least three replicate specimens of the reference material each exposure period.

5.7.4 After all specimens have been returned to the round-robin coordinator, data should be entered into a spreadsheet for analysis. Plot the data to get a preliminary look at trends, patterns, and possible outlier data. For example, a time series plot of the data with the sample ID as the x-axis can give an indication of any repeating patterns in results such as the effect of exposure position. A histogram of the data at a particular exposure time can give an indication of the spread of the data and possible outliers.

5.7.5 Analyze data according to Guide E691 to determine repeatability standard deviation and reproducibility standard deviation. Use the repeatability and reproducibility standard deviations to determine the "difference two sigma limit" for repeatability and reproducibility as defined in Practice E177.

5.7.6 The data from the round-robin shall be used as follows for establishing specification limits for the reference material:

5.7.6.1 Repeatability within a laboratory: grand mean from round-robin $\pm 2 \times$ repeatability standard deviation

5.7.6.2 Reproducibility between laboratories: grand mean from round-robin $\pm 2 \times$ reproducibility standard deviation

NOTE 10—The three primary sources of variability in results from the round robin test are from the exposure, the property measurement, and between replicate specimens. If desired, a components of variance analysis can be conducted on the results from the round-robin to try to determine the relative contribution of each to overall variability.

5.8 When the original lot of a reference material must be replaced with a new lot, at least two laboratories shall conduct the following experiment to compare the original and new lots in order to determine whether a complete round-robin study must be conducted to determine repeatability and reproducibility limits for a new lot of the reference material.

[4] Ketola and Fischer, "Use of Reference Materials in Accelerated Durability Tests", VAMAS Technical Report Number 30, ISSN 1016-2186, Versailles Project on Advanced Materials and Standards, available from National institute of Standards and Technology, Gaithersburg, MD.

TABLE 1 Layout of experiment comparing new lot of a reference material with an original lot that has already been characterized by conducting a round-robin study according to section 5.7

Exposure Time	Original Lot	New Lot	Difference between randomly selected pair
one	x_1	a_1	$D1_1$
	x_2	a_2	$D1_2$
	x_3	a_3	$D1_3$
	...	...	...
	x_n	a_n	$D1_n$
two	y_1	b_1	$D2_1$
	y_2	b_2	$D2_2$
	y_3	b_3	$D2_3$
	...	...	...
	y_n	b_n	$D2_n$
three	z_1	c_1	$D3_1$
	z_2	c_2	$D3_2$
	z_3	c_3	$D3_3$
	...	...	...
	z_n	c_n	$D3_n$

5.8.1 Each laboratory shall simultaneously expose the original and new lot of the reference material in one device. Measurement of the characteristic property must be determined at a minimum of three exposure times with at least three replicate specimens of both lots evaluated at each time. For each exposure time, calculate the difference in characteristic property between randomly selected pairs of the original and new lots. Table 1 illustrates how this experiment is laid out for each laboratory.

5.8.2 Each laboratory shall conduct a *t*-test of the differences between the original and new lots with mu = 0 as the null hypothesis and a 95 % *t*-confidence interval.

5.8.2.1 If either lab rejects the null hypothesis, a full round robin study must be conducted to establish repeatability and reproducibility limits for the new lot of the reference material.

5.8.2.2 If both labs accept the null hypothesis, the repeatability and reproducibility limits for the original lot can also be used for the new lot.

6. Report

6.1 The report shall include a complete description of the exposure test cycle and the procedure used to measure the property of interest.

6.2 Report the results from the round-robin according to Guide E691.

6.3 Report observations of trends from graphing the data (include the graphs in the report).

7. Keywords

7.1 accelerated-aging; analysis of variance; exposure; round-robin; weathering reference material