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Standard Practice for Calculating Property Retention Index of Plastics ¹

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1. Scope

- 1.1 This practice covers procedures for the calculation of a property retention index (PRI) of thermoplastic and thermoset plastics after exposure to thermal aging, natural or artificial accelerated weathering, or chemical exposures.
- 1.2 This practice is not intended to establish a fixed procedure for conducting the exposure test, but it is intended to provide a set of specific procedures used to calculate the retention index of a characteristic property of the material after it has been exposed. Selection of the specific exposure test conditions depends on the material being tested and the property being measured. It is up to the user to determine which exposure test conditions are most relevant to the specific material and the service condition being used. The exposure test used must be conducted in accordance with conditions described in specific exposure standards.
- 1.3 This practice does not describe procedures for sampling the materials to be tested. These procedures are described in the standards and specifications applicable to the material being evaluated.
- 1.4 The procedure used to calculate the PRI depends on whether the test used to characterize the materials being exposed is destructive or nondestructive. The PRI can be useful in describing short-term mechanical, electrical, and other properties of plastics at specified temperatures after the materials have been subjected to an exposure test.
- 1.5 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 1—There is no similar or equivalent ISO standard. ISO DIS 11248 is significantly different since it pertains only to thermosetting resins.

2. Referenced Documents

2.1 ASTM Standards:

- D 543 Test Method for Resistance of Plastics to Chemical Reagents²
- D 618 Practice for Conditioning Plastics for Testing²
- D 883 Terminology Relating to Plastics²
- D 1435 Practice for Outdoor Weathering of Plastics²
- D 1499 Practice for Filtered Open-Flame (Carbon-Arc) Exposures of Plastics²
- D 1898 Practice for Sampling of Plastics²
- D 2565 Practice for Operating Xenon Arc-Type Light Exposure Apparatus With and Without Water for Exposure of Plastics³
- D 3045 Practice for Heat Aging of Plastics Without Load³
- D 4329 Practice for Operating Light- and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Plastics⁴
- D 4364 Practice for Performing Accelerated Outdoor Weathering of Plastics Using Concentrated Natural Sunlight⁴
- D 4459 Practice for Operating an Accelerated Lightfastness Xenon-Arc-Type (Water Cooled) Light-Exposure Apparatus for the Exposure of Plastics for Indoor Applications⁴
- D 4674 Test Method for Accelerated Testing for Color Stability of Plastics Exposed to Indoor Fluorescent Lighting and Window-Filtered Daylight⁴
- D 6360 Practice for Enclosed Carbon-Arc Exposures of Plastics⁴
- G 113 Terminology Relating to Natural and Artificial Weathering Test of Nonmetallic Materials⁵
- 2.2 ISO Standards:⁶
- ISO 291 Plastics—Standard Atmospheres for Conditioning and Testing
- ISO 877 Plastics—Methods of Exposure to Direct Weathering, to Weathering Using Glass Filtered Daylight, and to Intensified Weathering by Daylight Using Fresnel Mirrors
- ISO 4892 Plastics—Methods of Exposure to Laboratory Light Sources Part 1: General Guidance, Part 2: Xenon

¹ This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 08.03.

⁵ Annual Book of ASTM Standards, Vol 14.02.

 $^{^6}$ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

Arc Exposures, Part 3: Fluorescent UV Exposures, and Part 4: Filtered Open Flame Carbon Arc Exposures ISO DIS 11248 Plastics—Thermosetting Molding Materials, Evaluation of Short-Term Performance at Elevated Temperatures

3. Terminology

3.1 *Definitions:* The terminology given in Terminologies D 883 and G 113 is applicable to this practice.

4. Significance and Use

- 4.1 The property retention index (PRI) determined by this practice is intended primarily to provide relative durability performance information on materials for design engineers. It is up to the user to ensure that appropriate sampling procedures are used for the selection of specimens to be exposed so that the PRI data obtained is actually representative of the material being evaluated.
- 4.2 The PRI obtained depends on the material being tested, property being evaluated, and exposure condition used. A PRI obtained for one property will probably not be the same as the PRI for a different property of the same material, even if the same exposure test is used.
- 4.3 Plastics exposed to a combination of environmental and thermal treatments may undergo a change in functional performance. Any laboratory-accelerated aging procedure, especially those that use only a single stress, may not realistically indicate the changes a plastic may undergo in actual use conditions. This practice provides a means for expressing the changes in properties as a function of time exposed in a wide variety of tests. The PRI data obtained is best used for comparing the performance of materials subjected to the same exposure test simultaneously.
- 4.3.1 Both laboratory-accelerated and outdoor exposure testing can be highly variable, and the PRI data will be influenced by this variability. For example, PRI data from outdoor exposures can vary depending on the exposure location and the time of year when the exposure is conducted. Variability in laboratory-accelerated exposure tests can result in large differences in PRI data from two laboratories running supposedly identical tests. PRI data obtained from exposure to laboratory-accelerated tests cannot be used to predict the PRI for exposure to natural weathering or actual use conditions unless there is a sufficient amount of data from both types of exposure to allow valid statistical comparisons.
- 4.4 A number of different exposure techniques can be used to provide information on the effects of environmental stresses such as light, heat, and water on plastics (see Practices D 1435, D 1499, D 2565, D 4329, D 4364, and D 4459; Test Method D 4674; and ISO 4892 and 877). When it is desirable to evaluate the effects of heat alone, exposures should be conducted in accordance with Practice D 3045. When it is desirable to evaluate the effects of chemical exposures, the exposures should be conducted in accordance with Test Method D 543.
- 4.5 There are a number of factors influencing the physical properties and the retention of these properties after exposure. In addition to a complete description of the exposure test conditions used, the following information shall be included in

any report referencing this practice: (1) complete description of the material tested, including the type, source, manufacturer's code number, form, and previous history; (2) methods of preparation for the material and individual test specimens; (3) procedure used for specimen conditioning prior to and after exposure; (4) complete description of the environment in which the physical properties were determined (for example, temperature and relative humidity); (5) complete description of the procedure used to determine the physical properties tested, including the rate at which specimens were tested, if applicable; (6) if applicable, void content of the specimens tested and the method used to measure void content.

Note 2—It is not the intent of this practice to require users to divulge proprietary information regarding composition. To avoid divulging proprietary information, generic descriptions may be used to provide information on material composition.

- 4.6 When destructive tests are used to determine a physical or chemical change, or both, which occurs as a result of exposure, the amount of change is expressed as a function of the value obtained for the material tested at a specified test environment (for example, temperature and humidity). The exposed and reference specimens are measured at the same time in the specified test environment.
- 4.7 When nondestructive tests are used to determine a physical or chemical change, or both, which occurs as a result of exposure, the amount of change is expressed as a function of the value obtained on the specimens prior to exposure. Property measurement tests on the specimens before and after exposure shall be conducted at the same conditions (for example, temperature and humidity).
- 4.8 The property or properties to be measured may be specified in an ASTM, ISO, or other appropriate standard for the material being tested, or by any prior agreement between interested parties. If the method used to measure the property being evaluated is not described in an ASTM, ISO, or other appropriate standard, a description of the test method shall be included in the report of test results.
- 4.9 It is realized that a material cannot be tested without specifying the method of preparation. To have any meaning in comparative testing, specimens of each material being evaluated by these test procedures should be prepared or molded from the same lot under identical processing conditions and randomized prior to testing at the conditions desired. It must be realized that lot-to-lot variation in the material may cause additional variability in results.

Note 3—For those plastics with a Tg greater than ambient, the slow collapse of free volume, with attendant significant changes in mechanical properties such as fatigue resistance, impact resistance, yield stress, and vapor transmission, etc. will be accelerated at elevated temperatures below the Tg but will be reversed at temperatures above the Tg. Therefore, incubation at elevated temperatures in the Tg range may be erratically susceptible to oven fluctuation effects.

4.10 The results depend on which side of the test specimen is exposed with some tests. In bending tests, for example, different results are obtained in accordance with whether the exposed surface or the unexposed surface of the test specimen is placed under tension. Care must be taken to ensure that all specimens being exposed have the same orientation in the test

fixture used to hold the specimens during exposure. In addition, the results also depend on the orientation of test specimens during the procedure used to measure the property being monitored. This is especially true with impact tests. During the procedure used to measure the characteristic property, care must be taken to ensure that all specimens are oriented the same way in the test fixture.

4.11 Before proceeding with this practice, reference should be made to the specification of the material being tested. Any test specimen preparation, conditioning, or dimensions, or some combination thereof, and testing parameters covered in the material's specification shall take precedence over those mentioned in this practice. The default conditions described in this practice apply if there are no material specifications.

5. Apparatus

5.1 The apparatus used for exposure and measurement of the property desired will depend on the particular exposure used and property being measured. Refer to the appropriate ASTM or ISO standards for requirements on the apparatus needed.

6. Sampling

- 6.1 Sampling of materials for testing is covered under applicable standards or specifications for the material being tested.
- 6.2 It is important to select samples for testing that are representative of the material being evaluated. Procedures for sampling plastics are described in Practice D 1898.

7. Test Specimens

- 7.1 The test specimens shall be in accordance with the appropriate test method used for the properties being measured.
- 7.2 All test specimens shall be prepared in accordance with the pertinent material standards and other relevant ASTM standards.
- 7.3 Annealing of thermoplastic materials and post-curing of thermosetting materials has a significant effect on many properties. To minimize errors caused by these effects, annealing and post-curing shall be conducted accurately in accordance with pertinent material standards or, if not available, in accordance with the material manufacturer's recommendations. The conditions used for any annealing or post curing of specimens shall be reported.
- 7.4 The number of specimens tested shall be in accordance with the test method for the particular properties being measured. Large numbers of test specimens should be used in cases of extreme variability, in which the standard deviation of test results is more than 20 % of the mean value. If the test method used does not require a specific number of test specimens, a minimum of five replicate specimens of each material shall be used.

8. Procedure

- 8.1 Exposure of Test Specimens
- 8.1.1 When determining the PRI for materials exposed to natural weathering, conduct exposures in accordance with Practice D 1435.

- 8.1.2 When the PRI for exposure to concentrated natural sunlight is to be determined, conduct exposures in accordance with Practice D 4364.
- 8.1.3 When the PRI for exposure to laboratory light sources is to be determined, conduct exposures in accordance with one of the following ASTM standards:
- (1) Practice D 1499 for exposures to filtered open-flame carbon-arc light sources;
- (2) Practice D 2565 for exposures to xenon-arc light sources;
- (3) Practice D 4329 for exposure to fluorescent ultraviolet (UV) light sources; and
- (4) Practice D 6360 for exposures to enclosed carbon-arc light sources;
- (5) Procedures for exposure to these and other types of light sources are also described in ASTM or ISO standards applicable to specific types of materials; these can also be used when mutually agreed upon by all interested parties.
- 8.1.4 When the PRI for exposure to heat onermined, conduct exposures in accordance with Practice D 3045. Exposures used to determine the PRI will typically be conducted at one or two temperatures. The procedures covered in Practice D 3045, which describe the calculation of a time to fail, would not be used for tests to determine the PRI.
- 8.1.5 When the PRI for chemical exposure is to be determined, conduct exposures in accordance with Test Method D 543.
- 8.2 Material Properties Measured with Nondestructive Tests—When material properties are measured using non-destructive methods (for example, gloss, haze, and transparency), determine the PRI as follows:
- 8.2.1 Determine the initial level of the measured property, $P_{i,o}$, for each replicate specimen of the materials being tested.
- 8.2.2 Expose all replicate test specimens to the environment desired, conducting exposures in accordance with the applicable ASTM standards.
- 8.2.3 Measure the desired property after exposure, $P_{i,x}$, using the same test method used to determine the level of the initial property.
- 8.2.4 Determine the PRI for each replicate specimen, z_i , as follows:

$$z_i = \frac{P_{i,x}}{P_{i,o}} \tag{1}$$

where:

 $P_{i,o}$ = initial value of measured property, and

 $P_{i,x}$ = property at exposure time, x.

8.2.5 Calculate the mean PRI, \bar{Z} , as follows:

$$\bar{Z} = \frac{\sum_{i=1}^{n} z_i}{n} \tag{2}$$

where:

 z_i = PRI for individual test specimen, and

n = number of replicate specimens.

8.2.6 Calculate the standard deviation for the PRI, \mathbf{s}_z , as follows:

$$s_{z} = \sqrt{\frac{\sum_{i=1}^{n} (z_{i} - \bar{Z})^{2}}{n-1}}$$
 (3)

8.3 Material Properties Measured with Destructive Tests—Measurement of mechanical properties of materials often involves destructive tests on individual specimens. When destructive tests are used to measure mechanical or other properties, calculate the PRI as follows:

8.3.1 Prepare sufficient test specimens to provide for measurement of the desired property on a set of file specimens and on a set of exposed specimens for each exposure period used. It is recommended that test specimens be prepared individually or machined from a larger sample prior to exposure. Test specimens may be machined from larger pieces after they have been exposed, if specifically permitted in a given material standard or if specifically recommended by a material manufacturer. If this is done, it may be necessary to precondition the sheets of exposed material prior to cutting or machining in order to facilitate test specimen preparation. A PRI determined on specimens machined prior to exposure will probably show significant difference from a PRI determined on specimens machined after exposure. Prepare all test specimens and file specimens used for determination of the reference value of the measured property at the same time, using the same cutting or machining procedure. It is very important that all tests be conducted under the same conditions.

8.3.2 Unless otherwise specified, store unexposed file specimens used for determination of the reference property level in the dark, using one of the standard atmospheres specified in Practice D 618 or ISO 291.

8.3.3 After exposure of the test specimens as desired, determine the PRI as follows. Repeat this process for each exposure increment used.

8.3.3.1 Determine the level of the reference property using the number of file specimens required by the standard describing the procedure used for measuring the desired property. Use the following equation to calculate the mean of reference property,

$$\bar{p}_{o} = \frac{\sum_{i=1}^{n} p_{o,i}}{n} \tag{4}$$

where:

 $p_{o,i}$ = measured initial property of each replicate, and n = number of replicate file specimens.

8.3.3.2 After the desired exposure period has been completed, determine the level of the measured property, $p_{i,x}$, on each specimen. Prior to property measurement, condition the exposed specimens using one of the atmospheres and conditioning periods described in Practice D 618 or ISO 291. The atmosphere and conditioning time used shall be agreed upon by all interested parties in agreement with 8.3.2 and shall be included in the test report.

8.3.3.3 Determine the PRI, z_i , for each exposed specimen using the following equation:

$$z_i = \frac{\bar{p}_{i,x}}{p_o} \tag{5}$$

8.3.3.4 Determine the mean PRI, \bar{Z} , using the following equation:

$$\bar{Z} = \frac{\sum_{i=1}^{n} z_i}{n} \tag{6}$$

where:

 z_i = PRI for individual exposed specimen, and

n =number of exposed specimens.

8.3.3.5 Determine the standard deviation of the PRI, s_z , using the following equation:

$$s_z = \sqrt{\frac{\sum_{i=1}^{n} (z_i - \bar{Z})^2}{n-1}}$$
 (7)

8.3.4 When destructive property measurement tests are used, there are three options for determining the PRI on materials subjected to thermal aging exposures.

8.3.4.1 Option 1—Materials are exposed at temperature T_e , for a specified length of time, t, and the material property determination is determined at the same temperature, immediately upon completion of the exposure period. The reference value of the file specimens is determined at the same temperature at which the exposure is conducted. If this option is chosen, the PRI shall be designated PRI(E).

8.3.4.2 Option 2—Materials are exposed at temperature T_e , for a specified length of time, t, and then are removed from the exposure chamber and conditioned at a comparison temperature for a specified time, t_c . The reference value of the file specimens is also determined at the comparison temperature. If this option is chosen, the PRI shall be designated PRI(C).

8.3.4.3 Option 3—Materials are exposed at temperature T_e , for a specified length of time, t, and then are removed from the exposure chamber and conditioned at standard laboratory conditions of $25 \pm 2^{\circ}\text{C}$ and 50 ± 5 % relative humidity for a specified time, t_{cl} . The reference value of the material property determined on the file specimens is determined at the same standard laboratory temperature and humidity. If this option is chosen, the PRI shall be designated PRI(A).

9. Report

- 9.1 Report the following information:
- 9.1.1 Reference to this ASTM practice.
- 9.1.2 Complete identification of the product tested, including the following:
- 9.1.2.1 Complete description of the material tested, including the type, source, manufacturer's code number, form, and previous history;
- 9.1.2.2 Methods of preparation for the material and individual test specimens; and
- 9.1.2.3 If applicable, void content of the specimens tested and the method used to measure void content.
 - 9.1.3 Complete description of the exposure test used:

- 9.1.3.1 For accelerated exposure tests, including the following:
 - (1) Type of exposure device;
- (2) If specimens are exposed to light, the light source and filters used;
- (3) Complete description of the exposure cycle, including temperatures, period of light exposure, dark exposure, moisture exposure, etc.
 - (4) Length of exposure period; and
- (5) Reference to all relevant ASTM or ISO standards, including the year of issue.
 - 9.1.3.2 For exterior exposure tests, include the following:
 - (1) Location of exposure;
 - (2) Start and end dates for the exposure;
 - (3) Angle at which specimens were exposed; and
- (4) Reference to relevant ASTM or ISO standards, including the year of issue.
- 9.1.4 Complete description of the test methods used to measure the reported properties, with reference to the appropriate ASTM or ISO standard, where appropriate.
- 9.1.5 Complete description of the environment in which the physical properties were determined (for example, temperature and relative humidity).
- 9.1.6 Conditioning procedures used (where appropriate, by reference to the relevant ASTM or ISO standard).
- 9.1.7 The PRI for each test specimen, mean property retention index, and standard deviation of PRI.

10. Precision and Bias

10.1 Precision:

10.1.1 The repeatability and reproducibility for results obtained from this practice will vary with the materials being tested, type of exposure and specific exposure cycle used, and specific procedure used to measure the property of interest. In round-robin studies⁷ conducted by ASTM Subcommittee G03.03, the 60° gloss values of replicate specimens exposed in different laboratories were highly variable and exhibited very poor reproducibility. This result restricts the use of PRI data in absolute specifications that require a specific PRI after a specific exposure period.

10.1.2 The same round-robin studies demonstrated that the gloss values for a series of materials could be ranked with a high level of reproducibility between laboratories. This indicates that performance requirements based on the PRI of materials exposed in laboratory-accelerated tests should be specified in terms of comparison or ranking with a reference or control test specimen. The control test specimens must be exposed simultaneously in the same device as the test specimens of the material being evaluated. The specific control materials used must be agreed upon by all interested parties.

10.2 *Bias*—Bias in this practice cannot be determined because no standard reference materials exist for the various material weathering property responses described herein.

11. Keywords

11.1 accelerated test; aging; exposure; weathering

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⁷ Fischer, R. M., "Results of Round-Robin Studies of Light- and Water-Exposure Standard Practices," *Accelerated and Outdoor Durability Testing of Organic Materials*, ASTM STP 1202, Warren D. Ketola and Douglass Grossman, eds., ASTM, Conshohocken, PA, 1993.