Designation: D 5208 – 09

Standard Practice for Fluorescent Ultraviolet (UV) Exposure of Photodegradable Plastics[1]

This standard is issued under the fixed designation D 5208; 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 covers the specific procedures applicable for fluorescent Ultraviolet (UV) exposure of photodegradable plastics conducted in accordance with Practices G 151 and G 154. This practice also covers the preparation of test specimens and the evaluation of test results.

NOTE 1—Previous versions of this practice referenced fluorescent UV devices described by Practice G 53, which described very specific equipment designs. Practice G 53 has been withdrawn and replaced by Practice G 151, which describes performance criteria for all exposure devices that use laboratory light sources and by Practice G 154, which gives requirements for exposing nonmetallic materials in fluorescent UV devices.

1.2 Practice D 4329 covers fluorescent UV exposures of plastics intended for long term use in outdoor applications.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety problems, 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 known ISO equivalent to this practice.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 3826 Practice for Determining Degradation End Point in Degradable Polyethylene and Polypropylene Using a Tensile Test

D 4329 Practice for Fluorescent UV Exposure of Plastics

D 5870 Practice for Calculating Property Retention Index of Plastics

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

G 53 Practice for Operating Light-and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Nonmetallic Materials (Discontinued 2001)[3]

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

G 141 Guide for Addressing Variability in Exposure Testing of Nonmetallic Materials

G 147 Practice for Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests

G 151 Practice for Exposing Nonmetallic Materials in Accelerated Test Devices that Use Laboratory Light Sources

G 154 Practice for Operating Fluorescent Light Apparatus for UV Exposure of Nonmetallic Materials

G 169 Guide for Application of Basic Statistical Methods to Weathering Tests

3. Terminology

3.1 The definitions given in Terminology G 113 are applicable to this practice.

4. Significance and Use

4.1 Materials made from photodegradable plastics are intended to show relatively rapid deterioration of chemical, physical, and mechanical properties when exposed to light, heat, and water after fulfilling their intended purpose. This practice is intended to induce property changes associated with conditions that might be experienced when the material is discarded as litter, including the effects of sunlight, moisture, and heat. The exposure used in this practice is not intended to simulate the deterioration caused by localized weather phenomena such as atmospheric pollution, biological attack, and salt water exposure.

4.2 *Cautions*—Variation in results may be expected when operating conditions are varied within the accepted limits of this practice. Therefore, no reference to the use of this practice should be made unless accompanied by a report prepared in accordance with Section 9 that describes the specific operating

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics and Biobased Products.

Current edition approved Sept. 1, 2009. Published September 2009. Originally approved in 1991. Last previous edition approved in 2001 as D 5208 - 01.

[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] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

conditions used. Refer to Practice G 151 for detailed information on the caveats applicable to use of results obtained in accordance with this practice.

NOTE 3—Additional information on sources of variability and on strategies for addressing variability in the design, execution and data analysis of laboratory accelerated exposure tests is found in Guide G 141.

4.3 Exposure of a similar material of known performance (a control) at the same time as the test specimens provides a standard for comparative purposes. Use of a control to rank the stability of test materials greatly improves agreement between different laboratories.[4,5] It is recommended that at least three replicates of each material evaluated be exposed to allow for statistical evaluation of results.

4.4 Test results will depend upon the care that is taken to operate the equipment in accordance with Practice G 154. Significant factors include regulation of line voltage, temperature of the room in which the device operates, temperature control, and condition and age of the lamps.

5. Apparatus

5.1 Use of fluorescent UV apparatus that conform to the requirements defined in Practices G 151 and G 154 is required to conform to this practice.

5.2 The spectral power distribution of the fluorescent UV lamp shall conform to the requirements in Practice G 154 for a UVA 340 lamp.

5.3 *Test Chamber Location:*

5.3.1 Locate the apparatus in an area maintained between 18 and 27°C (65 and 80°F). Measure ambient temperature at a maximum distance of 150 mm (6 in.) from the plane door of the apparatus. Control of ambient temperature is particularly critical when one apparatus is stacked above another, because the heat generated from the lower unit can interfere with the operation of the units above.

5.3.2 Place the apparatus at least 300 mm from walls or other apparatus. Do not place the apparatus near a heat source such as an oven.

5.3.3 Ventilate the room in which the apparatus is located to remove heat and moisture.

6. Test Specimens

6.1 The size and shape of specimens to be exposed will be determined by the specifications of the particular test method used to evaluate the effects of the exposure on the specimens; the test method shall be determined by the parties concerned. Where practical, it is recommended that specimens be sized to fit specimen holders and racks supplied with the exposure apparatus. Unless supplied with a specific backing as an integral part of the test, specimens shall be mounted so that only the minimum specimen area required for support by the holder shall be covered. This unexposed surface must not be used as part of the test area.

6.2 For specimens of insulating materials, such as foams, maximum specimen thickness is 20 mm in order to allow for adequate heat transfer for condensation.

6.3 To provide rigidity, attach flexible specimens to a backing panel made of aluminum, 0.635 mm (0.025 mm) thick. Suggested aluminum alloys are 5052, 6061, or 3003.

6.4 Seal any holes in specimens larger than two mm and any openings larger than one mm around irregularly shaped specimens to prevent loss of water vapor. Attach porous specimens to a solid backing such as aluminum that can act as a vapor barrier.

6.5 Unless otherwise specified, expose at least three replicate specimens of each test and control material.

6.6 Follow the procedures described in Practice G 147 for identification and conditioning and handling of specimens of test, control, and reference materials prior to, during, and after exposure.

6.7 Do not mask the face of a specimen for the purpose of showing on one panel the effects of various exposure times. Misleading results may be obtained by this method, since the masked portion of the specimen is still exposed to temperature and humidity cycles that, in many cases, will affect results.

6.8 Since the thickness of a specimen may markedly affect the results, thickness of test and control specimens shall be within ±10 % of the nominal dimensions.

NOTE 4—This is especially important when mechanical properties are being investigated.

6.9 Retain a supply of unexposed file specimens of all materials tested.

6.10 Specimens should not be removed from the exposure apparatus for more than 24 h and then returned for additional tests, since this may not produce the same results on all materials as tests run without this type of interruption. Any elapsed time should be reported as noted under Section 9.

NOTE 5—Since the stability of the file specimen may also be time-dependent, users are cautioned that over prolonged exposure periods, or where small differences in the order of acceptable limits are anticipated, comparison of exposed specimens with the file specimen may not be valid. Instrumental measurements are recommended whenever possible.

7. Procedure

7.1 When the test and control specimens do not completely fill the specimen racks, fill all empty spaces with blank panels to maintain the test conditions within the chamber.

7.2 Unless otherwise specified, control irradiance at 0.89 ± 0.02 W/(m^2 · nm) at 340 nm.

NOTE 6—In devices without irradiance control operated at 50 ± 3°C uninsulated black panel temperature the typical irradiance at 340 nm is 0.89 W/(m^2 · nm). (See Note 1 of Table X2.1 in Practice G 154 for a full explanation of the current default irradiance.)

7.2.1 During equilibrium operation, the allowed deviation from the 340 nm set point is ±0.02 W/(m^2 • nm). If the indicated irradiance is outside the tolerance, stop the test and correct the problem before continuing.

7.3 Unless otherwise specified, program the device to one of the following test cycles.

[4] Fischer, R., "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 Douglas Grossman, Eds., American Society for Testing and Materials, Philadelphia, 1993.

[5] Ketola, W., and Fischer, R., "Characterization and Use of Reference Materials in Accelerated Durability Tests," VAMAS Technical Report No. 30. Available from NIST, Gaithersburg, MD.

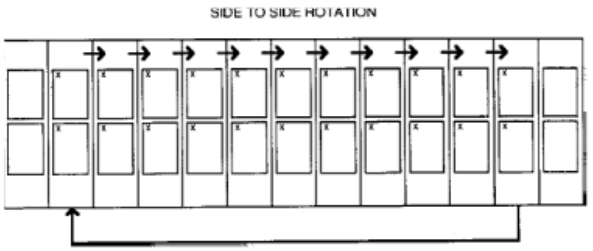

FIG. 1 Sample Rotation

7.3.1 *Cycle A*—20 h UV (light only) with uninsulated black panel temperature controlled at 50°C.

4 h Dark/condensation with uninsulated black panel temperature controlled at 40°C.

Repeat this 24-hour cycle continuously until the desired total exposure is reached.

7.3.2 *Cycle B*—4 h UV (light only) with uninsulated black panel temperature controlled at 50°C.

4 h Dark/condensation with uninsulated black panel temperature controlled at 40°C.

Repeat this 8-hour cycle continuously until the desired total exposure is reached.

7.3.3 *Cycle C*—continuous UV with uninsulated black panel temperature controlled at 50°C. Operate continuously until the desired total exposure is reached.

7.3.4 During equilibrium operation, the maximum allowable deviation from the uninsulated black panel temperature set point is ±3°C. If the indicated temperature of the uninsulated black panel is outside these limits, stop the test and correct the problem before continuing.

NOTE 7—The set points and tolerances for 7.2 and Cycles A, B, and C represent an operational control point for equilibrium conditions at a single location in the cabinet which may not necessarily represent the uniformity of those conditions throughout the cabinet. ASTM Committee G03 is working to refine these tolerances and address the uniformity issue.

7.3.5 Use Cycle C for materials that will be used for toxicity testing after exposure. This is essential because cycles that use condensation may wash away by-products of photochemical degradation.

7.4 Unless otherwise specified, reposition specimens as follows in order to minimize any effects from temperature or UV light variation. Figure 1 shows a diagram of the specimen repositioning.

7.4.1 Reposition the specimens horizontally at least every third day by (1) moving the two extreme right hand holders to the far left of the exposure area, and (2) sliding the remaining holders to the right.

7.4.2 Reposition the specimens vertically so that each specimen spends the same amount of exposure time in each vertical position within the specimen holder. For instance, if two specimens are stacked vertically in each holder, then the top and bottom specimens should switch places halfway through the test. If four specimens are stacked vertically, then the specimens should be repositioned vertically three times during the test.

7.5 Identification of any control specimen used shall accompany the report.

8. Periods of Exposure and Evaluation of Test Results

8.1 If a standard or specification for general use requires a definite property level after a specific time or radiant exposure in an exposure test conducted in accordance with this practice, base the specified property level on results from round-robin experiments run to determine the test reproducibility from the exposure and property measurement procedures. Conduct these round-robins in accordance with Practice E 691 and include a statistically representative sample of all laboratories or organizations who would normally conduct the exposure and property measurement. The precision and bias section contains results from such a round-robin.

8.1.1 If a standard or specification for use between two or three parties requires a definite property level after a specific time or radiant exposure in an exposure test conducted in accordance with this practice, base the specified property level on two independent experiments run in each laboratory to determine the reproducibility for the exposure and property measurement process. The reproducibility of the exposure/property measurement process is then used to determine the minimum level of property after the exposure that is mutually agreeable to all parties.

8.2 When reproducibility in results from an exposure test conducted in accordance with this practice have not been established through round-robin testing, specify performance requirements for materials in terms of comparison (ranked) to a control material. The control specimens shall be exposed simultaneously with the test specimen(s) in the same device. All concerned parties must agree on the specific control material used.

8.2.1 Conduct analysis of variance to determine whether any differences between test materials and control materials are statistically significant. Expose replicates of the test specimen and the control specimen so that statistically significant performance differences can be determined.

NOTE 8—Fischer illustrates use of rank comparison between test and control materials in specifications.[6]

NOTE 9—Guide G 169 includes examples showing use of analysis of variance to compare materials.

8.3 In most cases, periodic evaluation of test and control materials is necessary to determine the variation in magnitude and direction of property change as a function of exposure time or radiant exposure.

8.4 The time or radiant exposure necessary to produce a defined change in a material property can be used to evaluate or rank the stability of materials. This method is preferred over evaluating materials after an arbitrary exposure time or radiant exposure.

8.4.1 Exposure to an arbitrary time or radiant exposure may be used for the purpose of a specific test if agreed upon by the parties concerned. When a single exposure period is used, select a time or radiant exposure that will produce the largest

[6] Fischer, R., Ketola, W., "Impact of Research on Development of ASTM Durability Testing Standards," *Durability Testing of Non-Metallic Materials, ASTM STP 1294*, Robert Herling, Editor, American Society for Testing and Materials, Philadelphia, 1995.

performance differences between the test materials or between the test material and the control material.

8.5 Evaluate or rate changes in exposed test specimens in accordance with applicable ASTM test methods.

8.5.1 When testing degradable polyethylene and polypropylene, conduct tensile tests in accordance with Practice D 3826 to determine the degradation end point.

8.5.2 In accordance with EPA regulation 40 CFR Part 238, a polyethylene or polypropylene material cannot be considered as photodegradable when tested in accordance with this practice if an exposure of longer than 250 light hours using Cycle A is needed to produce degradation end point determined in accordance with Practice D 3826.

NOTE 10—For some materials, changes may continue after the specimen has been removed from the exposure apparatus. Measurements (visual or instrumental) should be made within a standardized time period or as agreed upon between interested parties. The standardized time period needs to consider conditioning prior to testing.

9. Report

9.1 Report the following information:

9.1.1 Type and model of exposure device.

9.1.2 If exposure is conducted in a device without irradiance control, record the age of fluorescent lamps used at the start of the exposure, and whether any lamps were changed during the period of exposure.

9.1.3 If required, report irradiance measured at a single wavelength in $W/(m^2 \cdot nm)$ and radiant energy measured at a single wavelength in $J/(m^2 \cdot nm)$. For measurements made over a broad band, such as 300–400 nm, report irradiance in W/m^2 and radiant exposure in J/m^2, specifying the spectral region measured.

9.1.3.1 Do not report spectral irradiance or radiant exposure unless direct measurement of spectral irradiance was made during the exposure.

9.1.4 Elapsed exposure time,

9.1.5 Light and dark-water-condensation or humidity cycle employed,

9.1.6 Operating uninsulated black panel temperature,

9.1.7 If required, operating relative humidity,

9.1.8 Specimen repositioning procedure, if different from the procedure described in 7.3, and

9.1.9 Type of property tests and results. Calculate retention of characteristic property in accordance with Practice D 5870 when it is reported.

10. Precision and Bias [7]

10.1 *Precision*

10.1.1 The repeatability and reproducibility of results obtained in exposures conducted in accordance with this practice will vary with the materials being tested, the material property being measured, and the specific test conditions and cycles that are used. It is essential to determine reproducibility of the exposure/property measurement process when using the results from exposures conducted in accordance with this practice in product specifications.

10.1.2 A round-robin conducted and analyzed in accordance with Practice E 691 for exposures of three degradable polyolefin polymers produced the repeatability and reproducibility results for tensile elongation shown in Table 1.

NOTE 11—For this round-robin, six laboratories exposed three different materials supplied by two laboratories. Each participating laboratory exposed five replicate specimens. After exposure, the specimens were returned to the originating laboratory for tensile testing.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D20-1205.

TABLE 1 Results from Round-Robin Test

Material Tested	Tensile Elongation Determined in Accordance with Practice D 3826				
	Average	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
ECO[A], unexposed	187.0	67.7	185.5	189.6	519.5
ECO, exposed 240 h[B]	1.7	0.8	1.1	2.3	3.0
white LL[C], exposed 24 h[B]	364.7	64.5	110.0	180.6	308.1
white LL, exposed 240 h[B]	4.0	2.1	4.6	5.8	12.8
clear LL, exposed 24 h[B]	531.3	110.2	110.2	308.5	308.5
clear LL, exposed 240 h[B]	75.1	26.7	152.7	74.8	427.5

[A] ECO material is ethylene/carbon monoxide copolymer which is known to degrade under UV exposure.
[B] The exposure used for this round-robin was Cycle A of this standard.
[C] LLDPE and white LLDPE are blown film low density polyethylene with an additive to promote degradation by solar radiation. Clear LLDPE was natural in color, while white LLDPE had some TiO_2 added to provide the white color.

10.2 *Bias*

10.2.1 Bias cannot be determined because no acceptable standard weathering reference materials are available.

11. Keywords

11.1 degradation; exposure; fluorescent UV; light exposure; ultraviolet

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D 5208 - 01) that may impact the use of this standard. (September 1, 2009)

(*1*) Removed references to discontinued standards.
(*2*) Updated default irradiance in Procedure section to 0.89 W/(m^2 • nm) and added detail to Cycles A, B and C to more clearly provide the desired control point for the light only and dark cycles.
(*3*) Updated 9.1.2 for devices that operate without irradiance control.