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Designation: D 3012 – 00

Standard Test Method for Thermal-Oxidative Stability of Polypropylene Using a Specimen Rotator Within an Oven[1]

This standard is issued under the fixed designation D 3012; 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 test method provides a means for estimating the resistance of polypropylene, in molded form, to accelerated aging by heat in the presence of air using a forced draft oven.

1.2 The stability determined by this test method is not directly related to the suitability of the material for use when different environmental conditions prevail and shall not be used to predict performance.

NOTE 1—The specified thermal levels in this test method are considered sufficiently severe to cause failure of commercial grades of heat-stable polypropylene within a reasonable period of time. If desired, lower temperatures can be applied to estimate the performance of polypropylene with lower heat stabilities.

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

1.4 *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—This test method and ISO 4577–1983 are technically similar but different in preparation of test specimens, thickness of test specimen, measurement of the number of air flow changes in the ovens, and the number of air changes per hour required.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 883 Terminology Relating to Plastics[2]

D 3641 Practice for Injection Molding Test Specimens of Thermoplastic Molding and Extrusion Materials[3]

D 4101 Specification for Polypropylene Injection and Extrusion Materials[3]

D 5374 Test Methods for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation[4]

D 5423 Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation[4]

E 1 Specification for ASTM Thermometers[5]

E 77 Test Method for Inspection and Verification of Thermometers[5]

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques[5]

E 608 Specification for Metal-Sheathed Base-Metal Thermocouples[5]

E 644 Test Methods for Testing Industrial Resistance Thermometers[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

E 1137 Specification for Industrial Platinum Resistance Thermometers[5]

2.2 *ISO Standard:*[7]

ISO 4577–1983 Plastics—Polypropylene and Propylene Copolymers—Determination of Thermal Oxidative Stability in Air-Oven Method

ISO 1873 Plastics—Polypropylene and Propylene-Copolymer Thermoplastics:

Part 1: Designation

Part 2: Determination of Properties

3. Terminology

3.1 *Definitions*—The definitions of plastics used in this test method are in accordance with Terminology D 883 unless otherwise indicated.

4. Summary of Test Method

4.1 Aging is accelerated by exposing the specimens to an elevated temperature in a forced draft oven equipped with a biaxial or uniaxial rotating specimen holder.

4.2 Visual examination is used to determine the time to failure. The time to failure of the material is taken as the number of days after which the specimen shows localized crazing, crumbling, or discoloration, or a combination thereof.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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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 10.02.

[5] *Annual Book of ASTM Standards*, Vol 14.03.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

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5. Significance and Use

5.1 Under the severe conditions of this test method, the specimens undergo degradation at a rate that is dependent upon the thermal endurance of the polypropylene material under examination.

5.2 The thermal level of this test method is considered sufficiently severe to cause failure of commercial grades of heat-stable polypropylene within a reasonable period of time. If desired, lower temperatures can be applied to estimate the performance of polypropylene materials with lower heat stability.

5.3 The technique of specimen rotation described in this test method provides an estimate of the life-temperature relationship of polypropylene. If this test method is conducted at different temperatures on the same material, a more reliable estimate of the life-temperature relationship of polypropylene is determined. This test method can be conducted at several temperatures and the data interpreted through use of the Arrhenius relation, by plotting the logarithms of times to failure against the reciprocals of the temperatures in kelvins (K). Temperatures in the range from 100 to 150°C, with intervals of 10°C, are suggested for this purpose.

5.4 The stability as determined under the prescribed test method is not directly related to the suitability of the compound for a use where different conditions prevail.

5.5 The specimen rotation technique of thermal aging increases the probability that all specimens will be exposed similarly and that the effect of temperature gradients in an oven will be minimized.

6. Apparatus

6.1 *Oven*, mechanical convection type for controlled circulation of air, with adjustable air intake and exhaust facilities, and designed for air velocities around ± 250 1000 mm/s (197 ± 49 ft/min).[8] The oven shall meet the requirements of Specification D 5423.

6.1.1 The oven shall be equipped with a temperature-control system designed to maintain the test temperature range from 150 ± 1°C (302 ± 1.8°F) and a device to prevent temperature override. With the oven adjusted to the nominal test temperature of 150°C, the override shall be set at 154°C (309°F). A bimetallic-strip temperature switch has been found satisfactory.

6.2 *Oven Temperature Measurement System*, consisting of a thermocouple, thermometer, or resistance thermometer as the sensor, together with its associated conditions and readout instrumentation covering at least the temperature range from 0 to 200°C (32 to 372°F).

6.2.1 The thermometer must cover the range in one-degree subdivisions. It must be tested for bulb stability and standardized, in accordance with Test Method E 77.

6.2.2 The secondary standard shall be ASTM Thermometer 67C of Specification E 1.

6.2.3 Thermocouples shall comply with the requirements of Specification E 608 and shall be calibrated in accordance with Method E 220.

6.2.4 Resistance thermometers shall comply with the requirements of Test Methods E 644 and Specification E 1137, and be calibrated in accordance with NIST Special Publication 250-22.[9,10]

6.3 *Molding Press*, designed to operate at 200 ± 5°C (392 ± 9°F).

6.4 *Injection Molding Unit*, meeting the requirements of Practice D 3641.

6.5 *Mold*:

6.5.1 *Compression Mold*, comprised of the following:

6.5.1.1 *Compression Molding Chase*, having a blanked-out area of suitable size (Note 3) and capable of producing a plaque 1.00 ± 0.05 mm (0.039 ± 0.002 in.) thick.

NOTE 3—A 152.4 by 152.4-mm (6 by 6-in.) blanked-out section has been found satisfactory.

6.5.1.2 *Backing Plates*, large enough to cover this chase and strong enough to resist warping or distortion, under the molding conditions. Polished steel plates, 3 mm (0.1 in.) thick, are satisfactory.

6.5.2 *Injection Mold*, designed using the guidelines specified in Practice D 3641. The mold shall be capable of producing either a plaque having a thickness of 1.00 ± 0.05 mm (0.039 ± 0.002 in.) from which 50 by 10 by 1.00-mm (2 by 0.4 by 0.039-in.) specimens may be die cut, or mold a standard test specimen, 50 by 10 by 1.00 mm.

6.6 *Parting Sheets*—Fluoropolymer, polyester, or other film that will not affect the long-term thermal stability of polypropylene, 0.05 to 0.20 mm (0.002 to 0.008 in.) thick. The film must be free of wrinkles and foreign matter, such as lubricants and oils.

6.7 *Cutting Die*, to produce 50 by 10-mm (2 by 0.4-in.) specimens from either a compression-molded or injection-molded plaque. The die must be sharp and free of nicks.

6.8 *Specimen Holder*—The specimen holder may be biaxially rotated or uniaxially rotated provided that the test specimens are in a stream of air having a relative velocity about 1000 mm/s (197 ft/min). Illustrations of suitable apparatus for biaxially and uniaxially rotated specimen holders are shown in Figs. 1 and 2, respectively. Biaxial rotation increases the probability that all specimens will be exposed similarly. In the case of dispute, the use of biaxial rotation shall be the reference method.

6.8.1 *Biaxially Rotated Specimen Holder* (see Fig. 1)[11]. The frequency of rotation about the horizontal and vertical axes shall be 1 to 3 min^{-1}.

6.8.2 *Uniaxially Rotated Specimen Holder* (see Fig. 2). The drum peripheral velocity shall be such that the air stream impinges on the flat section of the test specimens at about 1 m/s.

6.9 *Air Velocity Meter*, nondirectional resistance wire type for measuring the air velocity in the oven.

[8] A Precision Scientific Freas Model 835 B, or Blue M POM-206C-1, or equivalent, can be used.

[9] Mangum, B. W., "Platinum Resistance Thermometer Calibration," NBS Special Publication 250-22 (1987).

[10] Available from National Institute of Standards Technology, Gaithersburg, MD.

[11] Standard Scientific Supply Company, Model CS191, or equivalent, can be used.

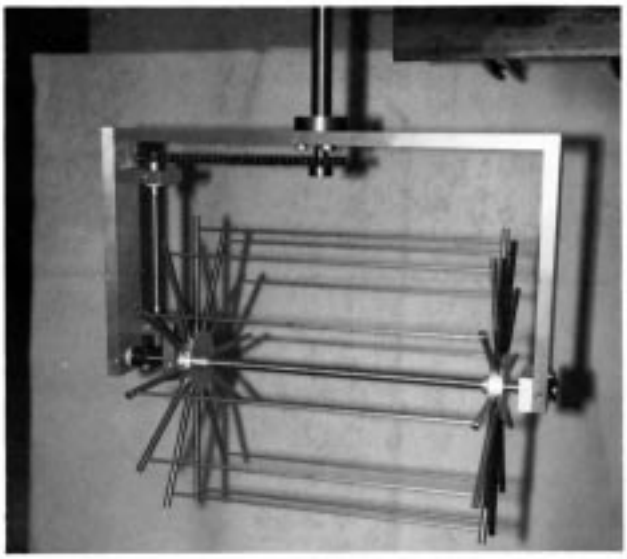

FIG. 1 Biaxial Ferris-Wheel-Type Rotator

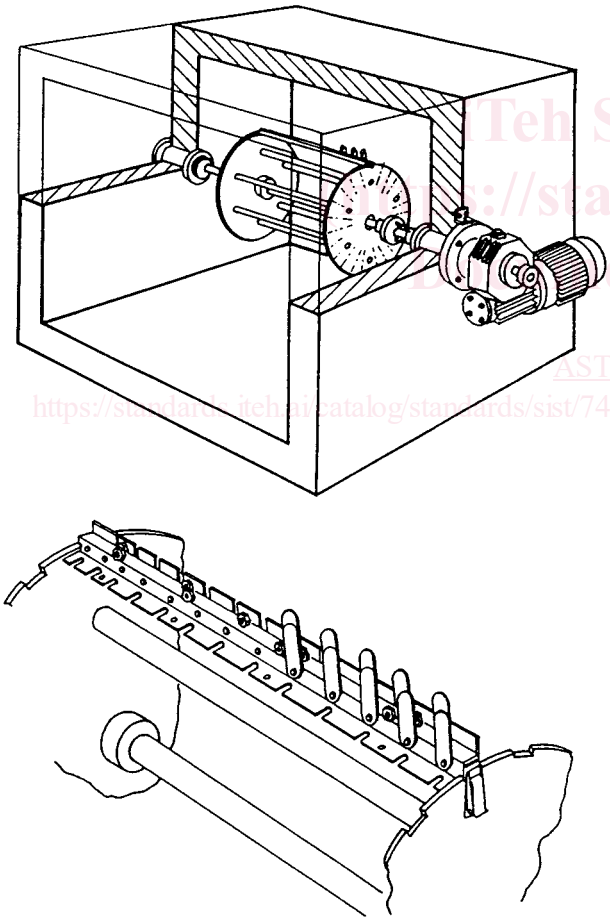

FIG. 2 Uniaxially Rotated Specimen Holder—Drum Rotator

6.10 *Anemometer*, with a circular vane, for determining the frequency of air changes in the oven. The anemometer shall be positioned directly in front of the oven outlet. Based on the design of the outlet and the anemometer, every effort shall be made to position the anemometer's electronics in a way as to minimize the degree of exposure to the heated air exiting the oven.

6.11 *Metal Clips*, lined with fluorocarbon film or other materials that have no adverse effect on the oxidative thermal stability of polypropylene.

7. Specimen Preparation

7.1 The test specimens shall be cut from either a compression–molded plaque or injection-molded plaque prepared from granules or other homogeneous molding material. Plaques shall be prepared as follows:

7.2 *Compression Molding*:

7.2.1 Adjust the temperature of the platens to 200 ± 5°C (392 ± 9°F).

7.2.2 Place a smooth, clean parting sheet on a backing plate and center the chase on it. Put enough of the sample into the cavity to fill it completely when molded. A slight excess of material is desirable. Cover the loaded chase first with a clean parting sheet and then a backing plate. Finally, put the assembled mold on the lower platen and close the press carefully until both platens are in contact with the assembly. When the material has melted, apply sufficient pressure to form a void-free plaque in the 1.00 ± 0.5 mm (0.039 ± 0.002 in.) thick and record this pressure. Leave the polypropylene in the heated press under pressure for 3 to 4 min at 200 ± 5°C (392 ± 9°F). Flash cool the mold assembly by transferring to a water-cooled press or by water quenching.

7.3 *Injection Molding*:

7.3.1 Plaques or test specimens shall be injection-molded in accordance with the requirements of Specification D 4101.

7.4 Prepare a minimum of five specimens per material sample by die-cutting specimens from the plaque or directly molding the standard test specimen. The standard specimen shall be 10 mm wide, 50 mm long, and 1.00 ± 0.05 mm thick. Edges shall be smoothed, if necessary, to remove imperfections introduced by cutting.

7.5 Test specimens prepared directly by injection molding without die cutting or specimens cut from polypropylene products can be used as agreed upon between the interested parties. In all cases the referee method will be based on compression molded die cut specimens.

NOTE 4—Failure test times for compression-molded and injection-molded specimens may vary due to the skin surface effect and the distinctness or sharpness of the specimen edge.

7.6 Specimens of other thicknesses can be used as agreed upon between the interested parties.

7.7 In cases of dispute, the referee specimens shall be die-cut only from compression-molded specimens, unless the test specimen is otherwise agreed upon.

7.8 In handling the plaques and cut specimens, use clean gloves or tongs to prevent contamination of the test specimens.

8. Conditioning

8.1 Condition the test specimens in accordance with Specification D 4101. When unsure of the material formulation, condition the test specimen at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in

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