AMERICAN NATIONAL STANDARD ANSI/ASTM D 2342 - 68 (Reapproved 1972)

Standard Test Method for LOT-TO-LOT STABILIZATION UNIFORMITY OF PROPYLENE PLASTICS[1]

This Standard is issued under the fixed designation D 2342; 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.

1. Scope

1.1 This method covers two procedures for determining the stability of propylene plastics when exposed to air in the molten state for a relatively brief period of time:

1.1.1 *Procedure A*—without the presence of copper.

1.1.2 *Procedure B*—with the presence of copper.

NOTE 1—The values stated in SI units are to be regarded as the standard.

2. Summary of Method

2.1 Plastic pellets are exposed in aluminum or copper dishes in a circulating-air oven at 185 C for definite time periods. Thermal oxidative stability under these conditions is determined visually by comparing the cooled disk of heated plastic with a standard or by measuring any change in its flow rate by ASTM Method D 1238, Measuring Flow Rates of Thermoplastics by Extrusion Plastometer.[2]

3. Significance

3.1 These procedures are intended primarily as a means for rapid quality control. They serve to indicate the uniformity of the thermal oxidative stability of a material as produced from lot-to-lot in a particular composition by a single manufacturer.

3.2 The test can detect differences among materials having different levels and types of stabilizers. Procedure A is applicable to all propylene plastics. The use of Procedure B is generally limited to materials that are specifically designed for applications in which there is direct contact of the plastic with copper or its alloys.

3.3 Due to the complexities of polymer oxidative degradation and its retardation and their dependence on such factors as temperature and time, the data obtained by these procedures for materials of various polymer and stabilization types may not correlate with behavior under actual use conditions. Therefore, these procedures are not recommended for rating or qualifying such materials in the absence of other long-term aging information.

4. Apparatus

4.1 *Oven*—Air-circulating oven capable of maintaining a uniform temperature and an air flow of 75 to 140 m/min (approximately 250 to 450 fpm) measured at the inlet side. A convenient method of checking uniformity of oven exposure conditions consists of filling the oven shelf to be used with the aluminum dishes described in 4.2, after charging each with 15 g of a general-purpose (moderately stabilized) polypropylene. Heat for 16 h at 185 ± 2 C. Remove the dishes, cool, and examine. The polypropylene in each of the dishes should be similar in color. Variation in polypropylene color between dishes indicates nonuniformity of oven temperature or air flow, or both; if variation occurs, the oven is

[1] This method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D-20.12 on Olefin Plastics.

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[2] *Annual Book of ASTM Standards*, Part 35.

unsatisfactory and must not be used.

NOTE 2—A Freas oven, Model 625, gives satisfactory results when used with the air intake and exhaust vents wide open and the air flow dial set at the maximum air flow.

4.2 *Dishes*—Aluminum dishes and copper dishes, each approximately 0.14 mm (0.0055 in.) thick, 70 mm (2.76 in.) in diameter, and 17 mm (0.67 in.) deep.

NOTE 3—Satisfactory aluminum dishes are flat bottom aluminum foil dishes used for milk analysis, made from aluminum alloy 1145-O. At present there is no commercial source for similar copper dishes, but for the purpose of this test these may be easily fabricated by hand or stamping-tool from annealed ETP copper sheet (ASTM Specification B 152, for Copper Sheet, Strip, Plate, and Rolled Bar[3]).

4.3 *Extrusion Plastometer*—Apparatus and accessories as specified in ASTM Method D 1238.

5. Test Specimen

5.1 The test specimen shall be in the form of pellets or granules as manufactured for molding or extrusion use.

6. Conditioning

6.1 The specimens shall be tested in the as-received condition unless otherwise specified.

7. Procedure A—Aluminum Dish Test

7.1 Set the oven temperature at 185 ± 2 C and regulate the air flow to be in the range from 75 to 140 m/min (approximately 250 to 450 fpm).

7.2 Clean the aluminum dishes before use with a 1+1 mixture of acetone and toluene. It is necessary for the bottoms of the dishes to be flat. Discard any dishes that are not satisfactory or reparable.

7.3 Place 3.5 ± 0.1 g of the sample into each of two aluminum dishes. Spread the pellets to a uniform depth in the dish and take precautions to avoid any disturbance of the pellets when transferring the dishes to the oven.

7.4 Place the specimen dishes onto the tray in the oven, taking care that they rest horizontally, and expose for the specified time. Time of exposure shall be as agreed upon between the seller and the purchaser.

NOTE 4—Testing exposure time is not specified by this procedure in order to provide flexibility for use with materials of widely differing stabilization characteristics. The exposure for a particular product should be of sufficient duration to detect any real lot-to-lot abnormality but should not be too excessive since inherent variability of the method increases with the degree of degradation. A recommended technique for determining a suitable exposure time for control purposes with a given product is as follows: Make flow rate determinations on a representative number of replicate samples after several intervals of exposure to establish the longest time of exposure that still gives significantly reproducible results. For example, 1, 2, 4, 8, and 16 h may be tried for each particular resin. If the reproducibilities of replicate determinations for the 8 and 16-h intervals are poor and those for the 1, 2, and 4-h exposures are acceptable and of the same order of magnitude, a 4-h exposure time might be specified.

7.5 Remove the specimens from the oven.

7.6 For control purposes, by agreement, a "go-no-go" method of visual inspection may be used. This is applicable to light-colored plastic only. An approved lot of plastic representing the maximum color limit is exposed for the specified time in air at 185 ± 2 C in the same manner as the material under test. For acceptance, the color of the material under test should be no greater than that of the approved material. If any question of acceptability is presented, complete the testing under Procedure A.

7.7 Cut the cooled wafers of melted polymer into pellets of 3 to 6 mm ($^1/_8$ to $^1/_4$ in.) and mix thoroughly.

7.8 Determine the flow rates of the exposed specimen and the unexposed plastic in accordance with Method D 1238 at Condition L. If the wafers of exposed plastic (see 7.7) are brittle when cut, or show visible degradation, the following modifications apply when testing its flow rate: Use a smaller load during the initial 5 min in the extrusion plastometer. Then replace with the test load. Cut off and discard the extrudate of the next minute. Shorten this time to 15 s if the amount of material extruded is excessive. The time of extrusion of the sample to be weighed will vary from 5 s to 3 min, depending on the amount of material being extruded.

7.9 Calculate the percentage change in flow rate as follows:

$$A_x = [(a_x - a_0)/a_0] \times 100$$

where:

[3] *Annual Book of ASTM Standards*, Part 6.