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Standard Test Method Practice for Use of a Melt Index Strand for Determining Density of Polyethylene¹

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This revision includes the addition of an ISO equivalency statement and precision and bias data. D 2839 – 96.

1. Scope *

1.1 This test method practice covers the preparation of a sample for polyethylene density determination in accordance with Test Method D 1505. The sample consists of a strand produced by extrusion of the polyethylene in accordance with Test Method D 1238, Condition 190/2.16 (Melt Index).

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

1.3 *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.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer²

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique²

~~D 1928 Practice 4703 Practice for Preparation of Compression-Molded Polyethylene~~ Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets and Test Specimens^{2,3}

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

3. Terminology

3.1 *Definitions:*

3.1.1 *melt index strand*—the extrudate produced when polyethylene is extruded in accordance with Test Method D 1238, Condition 190/2.16.

4. Significance and Use

4.1 This test method practice has been found to be very useful for preparing polyethylene samples suitable for determination of density by Test Method D 1505, for quality control purposes, especially in a resin manufacturing facility where fast, reproducible, comparative results are needed. It is not necessarily recommended for resin specifications which may be a part of a sales contract between the buyer and the seller.

4.2 The density of a polyethylene sample is highly dependent on the preparation and thermal history of the specimens. The level of density results obtained by this test method practice of sample preparation differs from that obtained by other methods as described in Practice ~~D 1928~~ D 4703.

4.3 Before proceeding with this test method practice, reference should be made to the specification of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters, or some combination thereof, covered in the

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol ~~14.02~~ 08.03.

⁴ Annual Book of ASTM Standards, Vol 14.02.

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

materials specification shall take precedence over those mentioned in this ~~test method~~ practice. If there are no material specifications, the default conditions apply.

5. Apparatus

- 5.1 *Extrusion Plastometer*, as described in Test Method D 1238.
- 5.2 *Hot Plate*, to boil water.
- 5.3 *Beakers*, 250-mL low form, graduated, with watch-glass covers.

6. Sample

- 6.1 *Polyethylene*, in any form suitable for test in accordance with Test Method D 1238.

7. Procedure

7.1 Prepare a Melt Index Strand by extruding the sample in accordance with Test Method D 1238, Condition 190/2.16, dropping the strand on a cool metal plate after cutting off. When Procedure B of Test Method D 1238 is used, cut off the extrudate at about the time the timer is actuated and discard. Save the portion extruded during the timed interval.

NOTE 2—The conditioning procedure as described in 7.2 and 7.3 may be omitted, if desired; in such case, after a 10-min cooling period, cut off the density specimen as described in 7.4 and determine the density in accordance with Test Method D 1505.

7.2 Drop the strand into a 250-mL beaker containing at least 200 mL of briskly boiling water, and cover with a watch-glass. Keep a large beaker full of water boiling along with this so that the amount of water can be maintained at 200 mL for the whole period without interrupting the boiling. Let the strand condition in the boiling water for 30 min.

NOTE 3—A time interval of 5 days at room temperature between extrusion of the strand and dropping into boiling water has not been found critical.

7.3 At the end of the boiling time, remove the beaker from the hot plate, making sure the water level is at approximately 200 mL, and allow to stand on the bench at standard laboratory temperature for 1 h.

7.4 Remove the strand from the water and cut off a 5-mm (1/4-in.) length from the thicker end of the strand. Discard this portion, and, from the next 10 mm (1/2 in.) of strand, cut at least three short pieces of convenient size for density determination. If the sample whitens when cutting, use a very sharp razor blade, and cut very slowly. Cutting under water may avoid whitening. This whitening appears to affect sample density and should be avoided. Discard any specimens with bubbles or visible roughness.

7.5 Determine the density of the specimen in accordance with Test Method D 1505 within 24 h of the conditioning described in 7.2 and 7.3.

NOTE 4—Considerable periods of time at room temperature between conditioning and density determination have little effect on results, but should be held to less than a day.

8. Report

- 8.1 Report the following information:
 - 8.1.1 Any deviations from the procedure, including omission of the conditioning.
 - 8.1.2 The density according to the Report Section of Test Method D 1505.

9. Precision and Bias

9.1 *Precision*—Table 1 is based on a round robin conducted in 1994, in accordance with Practice E 691, involving 7 materials tested by 10 to 15 laboratories. For each material, all of the samples were prepared by each laboratory in accordance with this ~~test method~~ practice. Each test result is an individual determination. Each laboratory obtained six test results for each material.

NOTE 5—**Caution:** The following explanations of r and R (9.2-9.2.3) are only intended to present a meaningful way of considering the approximate precision of this ~~test method~~ practice. The data in Table 1 should not be rigorously applied to acceptance or rejection of material, as those data are specific

TABLE 1 Precision Data

Material	N	Average	S_r^A	S_R^B	r^C	R^D
B	10	0.9139	0.00026	0.00078	0.00072	0.00219
F	12	0.9179	0.00020	0.00078	0.00055	0.00220
G	13	0.9222	0.00030	0.00073	0.00085	0.00206
A	15	0.9357	0.00041	0.00080	0.00115	0.00225
E	14	0.9530	0.00039	0.00092	0.00109	0.00258
C	11	0.9615	0.00030	0.00073	0.00085	0.00206
D	10	0.9626	0.00053	0.00109	0.00148	0.00305

^A S_r = within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations or the test results from all of the participating laboratories.

^B S_R = between-laboratories reproducibility, expressed as standard deviation, for the indicated material.

^C r = within-laboratory repeatability limit = $2.8 S_r$.

^D R = between-laboratories reproducibility limit = $2.8 S_R$.

to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this ~~test method~~ practice should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 9.2-9.2.3 would then be valid for such data.

9.2 *Concept of r and R* —If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing one specimen:

9.2.1 *Repeatability Limit, r* — (Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results should be judged not equivalent if they differ by more than the r value for that material.

9.2.2 *Reproducibility Limit, R* , (Comparing two test results for the same material, obtained by different operators using different equipment in different laboratories)—The two test results should be judged not equivalent if they differ by more than the R value for that material.

9.2.3 Any judgement in accordance with 9.2.1 or 9.2.2 would have an approximate 95 % (0.95) probability of being correct.

9.3 *Bias*—At the 95 % confidence level, there is no evidence of difference between the mean values of density tests prepared by this ~~test method~~ practice and those prepared using Practice ~~D 1928~~, D 4703, Annex 1, Procedure C.

10. Keywords

10.1 density; melt index strand; PE; polyethylene

SUMMARY OF CHANGES

This section identifies the location of selected changes to this practice. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this practice. This section may also include descriptions of the changes or reasons for the changes, or both.

D 2839 – 02:

(1) Change title from “Test Method” to “Practice.”

(2) Replaced Practice D 1928 with Practice D 4703 throughout practice.

(3) Changed subcommittee from D20.12 to D20.15 (D20.15.01) in Footnote 1.

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