Designation: D4883 – 08

Standard Test Method for Density of Polyethylene by the Ultrasound Technique[1]

This standard is issued under the fixed designation D4883; 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 covers the determination of the density of polyethylene through the utilization of ultrasound equipment.

1.2 This test method is based on the distinct behaviors of the amorphous and crystalline phases of polyethylene in response to ultrasound. Polyethylene shall be viewed as a composite structure where high-density crystalline regions are connected by lower-density amorphous material. The ratio of crystalline to amorphous material determines the final density of the material. The amorphous and crystalline phases exhibit very distinct behaviors with regard to the propagation of sound waves. The propagation characteristics in the composite will depend on the relative amount of the two phases (the degree of crystallinity).

1.3 Inorganic materials increase density as measured by Test Methods D792 and D1505, but they have little or no effect on ultrasonic density. The ultrasonic measurement is basically a base resin density.

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

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 known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D883 Terminology Relating to Plastics

D1248 Specification for Polyethylene Plastics Extrusion Materials for Wire and Cable

D1505 Test Method for Density of Plastics by the Density-Gradient Technique

D3350 Specification for Polyethylene Plastics Pipe and Fittings Materials

D4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets

D4976 Specification for Polyethylene Plastics Molding and Extrusion Materials

E494 Practice for Measuring Ultrasonic Velocity in Materials

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—The definitions given in Terminology D883, as well as in Test Methods D792 and D1505, are applicable to this test method.

4. Significance and Use

4.1 The density of polyethylene is a conveniently measurable property which is frequently useful as a means of following physical changes in a sample, as an indication of uniformity among samples, and as a means of identification.

4.2 This test method is designed to yield results with a precision of ±0.08 % or better.

5. Apparatus

5.1 Use an instrument which utilizes a sonic technique to evaluate the density of polyethylene. The DS 500 instrument[3] utilizes a sonic sensing head (transducer) which measures the velocity of sound in a molded specimen. Because sonic velocity is positively correlated to density in polyethylene, a measurement of this velocity is used to determine specimen

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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[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] The sole source of supply of the DS 500 instrument known to the committee at this time is Deguise Technologies, Inc., 11755 Rue de Guise, Quebec City (PQ), Canada, G2A 3K6; Phone: (418) 845-9064; (email:jraymond@b2b2c.ca). If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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

density. The information from this transducer then must be electronically evaluated; in the DS 500 instance this is done with a computer, and the result is reported either through a display or printout.

5.2 Equipment specified in Test Method D1505.

5.3 Equipment specified in Test Methods D792.

5.4 Equipment specified in Practice D618.

5.5 Equipment specified in Practice D4703, Annex 1.

NOTE 2—The equipment specified in 5.2 or 5.3 is required for the initial calibration of the sonic equipment. Once the equipment is calibrated, this additional equipment is no longer required. It is recommended that the standards used for the initial calibration be retained for any additional calibration when needed. It is also recommended that one or more of the calibration standards be evaluated on a routine basis for calibration verification. The absolute accuracy of data produced will not be better than this initial calibration and continued verification. Samples for initial calibration are available from various sources (such as the National Institute of Standards and Technology (NIST), resin manufacturers, and so forth).

6. Test Specimens and Materials

6.1 Test plaques shall be prepared in accordance to the molding procedure specified in Practice D4703, Annex 1, Procedure C.

6.2 The test specimen shall consist of a piece of the material under test. Mold or cut the sample specimen to the specified dimensions. When a sample piece is cut from a molded plaque, care must be taken to avoid change in density resulting from compressive stress.

	Specimen	Dimensions, mm (in.)
Length	80–100	(3.15–3.94)
Width	35–45	(1.38–1.77)
Thickness	1.5–3	(0.06–0.12)

NOTE 3—A minimum thickness of 1.5 mm is required to provide proper specimen stiffness and for the instrument to distinguish signal from echo. A maximum thickness of 3.0 mm is the thickness which the instrument sample holder allows.

NOTE 4—A sample thickness of 1.9 ± 0.2 mm shall be used in order to be in compliance with Specifications D1248, D3350 and D4976 for Polyethylene Plastics.

6.3 Use the same plaque thickness for calibration samples and testing samples.

6.4 The specimen shall be free of foreign matter and voids and shall have no surface marks or other surface flaws.

6.5 Use demineralized water for the testing equipment's water bath.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) for not less than 40 h prior to test in accordance with Procedure A of Practice D618, for those tests where conditioning is required. In cases of disagreement, specimens shall be conditioned at 23 ± 1°C and 50 ± 5 % relative humidity.

7.2 *Test Conditions*—Conduct tests in instrument's water bath at a temperature of 23 ± 2°C (73.4 ± 3.6°F) unless otherwise specified. In cases of disagreement, the tolerances shall be ±1°C (1.8°F).

NOTE 5—Testing in normal plant operations frequently calls for testing before the sample has become fully conditioned. It will be necessary to establish a correlation between the conditioning time and measured density and to apply the correlation to obtain the predicted density. If the specimens have not reached a level of stability that assures the density accuracy, the density determination shall be tested under test conditions in accordance with the test method listed in the applicable ASTM specification.

8. Calibration

8.1 Refer to instrument's operating manual for details on operating the instrument.

NOTE 6—The cleanliness of the demineralized water used in the water bath shall be monitored and the water be replaced on a regular basis to avoid erroneous testing results.

8.2 Resins to be utilized for calibration shall be molded into plaques in accordance with Practice D4703 Annex 1, Procedure C and be conditioned in accordance with Practice D618. Specimens to be used for calibration shall undergo full conditioning.

NOTE 7—One method of ensuring full conditioning is by aging at 70°C for 24 h.

8.3 Determine the density value of the specimen in accordance with Test Methods D792 or D1505. Conduct the determinations as specified by the test methods, that is, two determinations for D792 or three determinations for D1505. Calculate a mean density value for the sample plaque.

8.4 Evaluate each plaque on ultrasound instrument and use the mean density obtained in 8.3 for calibration. Use either the same sample plaque used in Section 8.3 or different plaques. This is considered as one data point. Six data points are recommended per resin sample. Ensure that the molded samples acquired for Test Methods D792 or D1505 accurately represent the molded samples utilized for the ultrasonic calibration.

8.5 The absolute accuracy of the data acquired is directly correlated to the accuracy of the calibration curve. This curve shall be made up of as many data points as possible and cover the entire density range of interest. A minimum of 30 data points per calibration curve is required. More data points are recommended if a broad density range is to be measured. These data points shall be evenly spread throughout the density range.

NOTE 8—Numerous product attributes such as product family, reactor geometry, catalyst, comonomer, additives and fillers, have been known to influence instrument calibration. Use different calibration curves for different products when needed. Verify the calibration curve when any change of this nature is made to the product.

NOTE 9—Because this test method is based on electronic techniques as compared to physical methods, it is imperative that the electronics be calibrated correctly. The electronics shall be re-calibrated when the transducer or the board is replaced.

9. Sample Testing

9.1 Place the test specimen in the instrument's water bath and allow it to condition for approximately 10 minutes prior to measurement to allow the specimen to become properly wetted and at the proper temperature.

9.2 For the most accurate results, test each sample four times (from different locations on the specimen) and determine the average. If the density of one determination is equal or greater than ±0.0004 g/cm^3 from the average, discard this

determination. If two determinations are equal or greater than ± 0.0004 g/cm^3 from the average, make a new plaque. If data has demonstrated that the resin samples have good uniformity, one determination per sample will be sufficient.

NOTE 10—In the case that deviation from the average is caused by inadequate conditioning of the sample in the bath, place the sample back to the bath for an additional five minutes then re-measure the density.

10. Report

10.1 Report the following information:

10.1.1 Complete identification of the material or product tested, including method of specimen preparation and conditioning.

10.1.2 Average specific gravity for all specimens from a sampling unit, reported as sp gr 23/23°C = ___, or average density reported as D23°C = ___ g/cm^3.

10.1.3 A measure of the degree of variation of specific gravity or density within the sampling unit such as the standard deviation and number of determinations.

10.1.4 Date of test.

11. Precision and Bias[4]

11.1 *Precision*—Table 1 is based on a round robin conducted in 1987 in accordance with Practice E691, involving four materials tested by six laboratories. Each material was molded, with all specimens being prepared in one laboratory. Each material tested was represented by four specimens, and each specimen was evaluated six times. This procedure yielded 24 test results for each material under evaluation from each laboratory.

[4] Supporting data are available from ASTM Headquarters. Request RR:D20-1157.

TABLE 1 Precision Data

Material	Average	S_r^A	S_R^B	r^C	R^D
1	0.9216	0.00029	0.00128	0.00082	0.00362
2	0.9187	0.00047	0.00107	0.00133	0.00302
3	0.9341	0.00073	0.00148	0.00207	0.00419
4	0.9516	0.00039	0.00127	0.00110	0.00359

[A] S_r = within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of 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_R = between-laboratories reproducibility limit = 2.8 S_R.

11.2 *Concept of r and R*—

Warning—The following explanations of *r* and *R* (11.2 through 11.2.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 shall not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and are not to be considered representative of other lots, conditions, materials, or laboratories. Users of this test method shall apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 11.2 through 11.2.3 shall then be valid for such data.

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:

11.2.1 *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 shall be judged not equivalent if they differ by more than the *r* value for that material.

11.2.2 *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 shall be judged not equivalent if they differ by more than the *R* value for that material.

11.2.3 Any judgment in accordance with 11.2.1 or 11.2.2 has an approximate 95 % (0.95) probability of being correct.

11.3 *Bias*—There are no recognized standards by which to estimate the bias of this test method.

12. Keywords

12.1 amorphous; crystalline; density; molded; plaques; polyethylene; sonic; ultrasonic; ultrasound

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4883 - 03) that may impact the use of this standard. (November 1, 2008)

(1) Removed permissive language in 1.2 and in Section 11.
(2) Revised wording of ISO statement in Note 1 for consistency with the latest standards.
(3) Updated the contact information for the sole supplier known for the instrument in Footnote 3.
(4) Added informational verbiage in 9.1 and 9.2 for clarification.