Designation: D 4883 – 99

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

This standard is issued under the fixed designation D 4883; 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 can 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 D 792 and D 1505, 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 similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

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

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[2]

D 883 Terminology Relating to Plastics[2]

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[2]

D 1898 Practice for Sampling of Plastics[2]

D 4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

3. Terminology

3.1 *Definitions:* The definitions given in Terminology D 883, as well as in Test Methods D 792 and D 1505, 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 Tecrad instrument[5] utilizes a sonic sensing head (transducer) which measures the velocity of sound in a molded specimen, approximately 1.9 mm in thickness. Because sonic velocity is positively correlated to density in polyethylene, a measurement of this velocity can be used to determine specimen density. The information from this transducer then must be electronically evaluated; in the Tecrad 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 D 1505.

5.3 Equipment specified in Test Methods D 792.

5.4 Equipment specified in Practice D 618.

5.5 Equipment specified in Annex A1 of Practice D 4703.

NOTE 2—The equipment specified in 5.2 or 5.3 is required for the initial calibration of the sonic equipment. Once the sonic 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 which may be required. It is also recommended that one or more of the calibration standards be evaluated on a routine basis for

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 08.03.

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

[5] The Tecrad instrument can be obtained from Tecrad U.S.A., 132 Boston Post Rd., East Lyme, CT 06333.

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

calibration verification. The absolute accuracy of data produced will be no better than this initial calibration and continued verification. Samples for initial calibration may be acquired from various standards sources (National Institute of Standards and Technology, manufacturer, etc.).

6. Test Specimens and Materials

6.1 Material for testing shall be acquired by procedures specified in Practice D 1898.

6.2 Test plaques shall be prepared in accordance to the molding procedure specified in Procedure C of Annex A1 of Practice D 4703.

6.3 The test specimen shall consist of a piece of the material under test. The piece may be cut from a molded plaque in the dimensions required for testing. Care should be taken in cutting specimens 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)

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

6.5 The water required by the testing equipment should be demineralized.

7. Calibration

7.1 Resins to be utilized for calibration should be molded into plaques in accordance with Procedure C of Annex A1 of Practice D 4703, be conditioned in accordance with Practice D 618, and have the density value determined in accordance with Test Methods D 792 or D 1505. Conduct the determinations using Test Methods D 792 or D 1505 a minimum of six times, allowing a mean value to be determined for that sample. Calibration should be performed with LLDDE C4, C6, and C8 copolymers; HDPS; and HP-LDPE homopolymers with no inorganic pigments, fillers, antiblock, etc.

7.2 Evaluate each ultrasonic sample a minimum of six times to obtain a mean density value.

7.3 Take care to acquire molded samples for Test Methods D 792 or D 1505 which accurately represent the molded samples utilized for the ultrasonic calibration.

NOTE 3—Because this test method is based on electronic techniques as compared to physical methods it is mperative that the electronics be calibrated correctly.

NOTE 4—The absolute accuracy of the data acquired is directly correlatable to the accuracy of the calibration curve. This curve should be made up of as many data points as possible (25 to 40) and should comprise as broad a density range as possible (from 0.900 to 0.970 g/cm^3).

NOTE 5—Specimens to be used for calibration should undergo full conditioning. One method of ensuring this is by aging at elevated temperatures (24 h at 70°C), to ensure that the specimen density has become stable.

8. Conditioning

8.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618, for those tests where conditioning is required. In cases where between laboratory data disagree, the tolerances shall be 1°C (1.8°F) and ±2 % relative humidity.

8.2 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity, unless otherwise specified in this test method. In cases where between laboratory data disagree, the tolerances shall be 1°C (1.8°F) and ±2 % relative humidity.

NOTE 6—Testing in normal plant operations frequently calls for testing before the sample can become fully conditioned. It will be necessary to develop correlations between measured density and conditioning time and to apply them to obtain the desired predicted density.

NOTE 7—Test specimens whose change in density during testing may be greater than the accuracy required of the density determination shall be tested under test conditions in accordance with the test method listed in the applicable ASTM specification.

9. Procedure (for Tecrad)

9.1 Switch the system on and observe the warm-up countdown.

9.2 After the machine has fully stabilized activate the“ F1” function key.

9.3 Insert a sample and enter the sample identification (15 characters maximum) after the machine displays “Please specify sample name.”

9.4 Wait while the machine displays “Please stand by while processing.”

9.5 A 10-s countdown will start when “Please remove sample” is displayed. The sample must be removed during this time interval to allow reference acquisition.

9.6 The density of the sample is then displayed, and can either be recorded on separate paper or printed on the associated printer.

9.7 Repeating 9.3 through 9.6 will allow the evaluation of additional samples.

9.8 To quit density measurements, the operator must activate the “F10” function key.

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 D 23°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 [6]

11.1 *Precision*—Table 1 is based on a round robin conducted in 1987 in accordance with Practice E 691, 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

[6] 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 reproduciblity limit = 2.8 S_R.

each specimen was evaluated six times. This procedure yielded 24 test results for each material under evaluation from each laboratory.

NOTE 8—**Caution:** 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 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method 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 11.2 through 11.2.3 would then be valid for such data.

11.2 *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:

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 should 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 should 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 would have 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

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D-20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 4883 – 96:
(*1*) Added an ISO equivalency statement and sections on keywords and precision and bias.

D 4883 – 99:
Added Note 7.