Designation: D 4883 – 9903

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:*

[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, Section 01, Physical Methods.

Current edition approved July 10, ~~1999.~~ 2003. Published September ~~1999.~~ 2003. Originally ~~published as D 4883 – 89.~~ approved in 1989. Last previous edition approved in 1999 as D 4883 – 9~~6~~9.

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

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 1248 Specification for Polyethylene Plastics: Extrusion Materials for Wire and Cable[2]
D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[2]
D ~~1898 Practice~~ 3350 Specification for ~~Sampling of Plastics~~[2]
~~D 4703 Practice for Compression Molding Thermoplastic~~ Polyethylene Plastics: Pipe and Fittings Materials ~~into Test Specimens, Plaques, or Sheets~~[3]
D 4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets[3]
D 4976 Specification for Polyethylene Plastics: Molding and Extrusion Materials[4]
E 494 Practice for Measuring Ultrasonic Velocity in Materials[5]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

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~~ DS 500 instrument[7] utilizes a sonic sensing head (transducer) which measures the velocity of sound in a molded ~~specimen, approximately 1.9 mm in thickness.~~ specimen. Because sonic velocity is positively correlated to density in polyethylene, a measurement of this velocity ~~can be~~ is used to determine specimen density. The information from this transducer then must be electronically evaluated; in the ~~Tecrad~~ 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 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, 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 ~~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.~~ 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 ~~no~~ better than this initial calibration and continued verification. Samples for initial calibration ~~may be acquired~~ are available from various ~~standards~~ sources (such as the National Institute of Standards and Technology (NIST), resin manufacturers, ~~e~~ and so fort~~c~~h).

6. Test Specimens and Materials

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

~~6.2 Test~~

6.1 Test plaques shall be prepared in accordance to the molding procedure specified in ~~Procedure C of Annex A1 of~~ Practice D 4703, Annex 1, Procedure C.

6.~~3~~2 The test specimen shall consist of a piece of the material under test. ~~The~~ Mold or cut the sample specimen to the specified dimensions. When a sample piece ~~may be~~ is cut from a molded ~~plaque in the dimensions required for testing. Care should~~ plaque, care must be taken ~~in cutting specimens~~ to avoid change in density resulting from compressive stress.

~~Specimen Dimensions, mm (in.)~~
Specimen Dimensions, mm [in.]

[2] *Annual Book of ASTM Standards*, Vol 08.01.
[3] *Annual Book of ASTM Standards*, Vol 08.0~~3~~2.
[4] *Annual Book of ASTM Standards*, Vol ~~14.02.~~ 08.03.
~~[5] The Tecrad instrument can be obtained from Tecrad U.S.A., 132 Boston Post Rd., East Lyme, CT 06333.~~
[5] *Annual Book of ASTM Standards*, Vol 03.03.
~~[6] Supporting data are available from~~
[6] *Annual Book of ASTM ~~Headquarters. Request RR: D20-1157.~~ Standards*, Vol 14.02.
[7] The DS 500 instrument can be obtained from R/D Tech (www.rd-tech.com) or Ultra Optec (www.ultraoptec.com).

~~Length~~	~~80–100~~	~~(3.15–3.94)~~
~~Width~~	~~35–45~~	~~(1.38–1.77)~~
~~Thickness~~	~~1.5–3~~	~~(0.06–0.12)~~
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 D 1248, D 3350 and D 4976 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 ~~Th~~Use demineralized water ~~required by~~ for the testing ~~equipment should be demineralized.~~ equipment's water bath.

7. ~~Calibration~~

~~7.1 Resins to be utilized~~Conditioning

7.1 *Conditioning*—Condition the test specimens at 23 ± 2°C [73.4 ± 3.6°F] for ~~calibration should be molded into plaques~~ not less than 40 h prior to test in accordance with Procedure ~~C~~ A of ~~Annex A1 of~~ Practice ~~D 4703,~~ D 618, for those tests where conditioning is required. In cases of disagreement, specimens shall be conditioned ~~in accordance with Practice D 618,~~ at 23 ± 1°C and ~~have the density value determined~~ 50 ± 5 % relative humidity.

7.2 *Test Conditions*—Conduct tests in ~~accordance with Test Methods D 792 or D 1505. Conduct the determinations using Test Methods D 792 or D 1505~~ instrument's water bath at a ~~minimum~~ temperature 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~~ 23 ± 2°C [73.4 ± 3.6°F] unless otherwise specified. In cases 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~~ disagreement, the ~~molded samples utilized for the ultrasonic calibration.~~ tolerances shall be ±1°C [1.8°F].

NOTE ~~3—Because this test method is based on electronic techniques as compared~~ 5—Testing in normal plant operations frequently calls for testing before the sample has become fully conditioned. It will be necessary to ~~phy~~ establiesh a correl~~m~~ation between the cond~~s~~itis~~o~~ning 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 ~~electronics~~ 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 R~~ed c~~efer to instrument's operating manual for de~~c~~tail~~y~~s on operating the instrument.

NOTE ~~4—The absolute accuracy~~ 6—The cleanliness of the ~~data acquired is directly correlatable to~~ demineralized water used in the ~~accuracy of~~ water bath shall be monitored and the ~~calibration curve. This curve should~~ water be ~~made up of as many data points as possible (25~~ replaced on a regular basis to ~~40)~~ avoid erroneous testing results.

8.2 Resins to be utilized for calibration shall be molded into plaques in accordance with Practice D 4703 Annex 1, Procedure C and ~~should comprise as broad a density range as possible (from 0.900~~ be conditioned in accordance with Practice D 618. Specimens to ~~0.970 g/cm^3).~~ be used for calibration shall undergo full conditioning.

NOTE ~~5—Specimens to be used for calibration should undergo full conditioning. One~~ 7—One method of ensuring ~~this~~ full conditioning is by aging at ~~elevated temperatures (24 h at 70°C), to ensure that~~ 70°C for 24 h.

8.3 Determine the ~~specimen~~ density ~~has become stable.~~

~~8. Conditioning~~

~~8.1 *Conditioning*—Condition~~ value of 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~~ specimen in accordance with ~~Procedure A of Practice D 618,~~ Test Methods D 792 or D 1505. Conduct the determinations as specified by the test methods, that is, two determinations for D 792 or three determination~~s~~e for D 1505. Calculate a mean density value for the s~~w~~ample 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 differengt plaques. This is ~~required. In cases where between laboratory~~ considered as one data point. Six data points agre re~~,~~commended per resin sample. Ensure that the molded samples acquired for Test Methods D 792 or D 1505 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 ~~1°C (1.8°F)~~ made up of as many data points as possible and ~~±2 % relative humidity.~~

~~8.2 *Test Conditions*—Conduct tests in~~ cover the ~~standard laboratory atmosphere~~ entire density range 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~~ interest. A minimum of 30 data ~~disagree, the tolerances~~ 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 ~~1°C (1.8°F) and ±2 % relative humidity.~~ evenly spread throughout the density range.

NOTE ~~6—Testing in normal plant operations frequently calls~~ 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 ~~testing before~~ different products when needed. Verify the ~~sample can become fully conditioned. It will be necessary~~ calibration curve when any change of this nature is made to ~~develop correlations between measured density and conditioning time and to apply them to obtain~~ the ~~desired predicted density.~~ product.

NOTE ~~7—Test specimens whose change in density during testing may be greater than~~ 9—Because this test method is based on electronic techniques as compared to physical methods, it is imperative that the ~~accuracy required of the density determination~~ electronics be calibrated correctly. The electronics shall be ~~tested under test conditions in accordance with~~ re-calibrated when the ~~test method listed in~~ transducer or the ~~applicable ASTM specification.~~ board is replaced.

9. ~~Procedure~~ (for Tecrad)

~~9.1 Switch~~Sample Testing

9.1 For the mo~~s~~yt accurate results, test each sample fo~~n~~ur times and ~~observe~~ determine the ~~warm-up countdown.~~

~~9.2 After~~ average. If the ~~machine has fully stabilized activate the" F1" function key.~~

~~9.3 Insert~~ 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 ~~sample and enter~~ new plaque. If data has demonstrated that the resin samples have good uniformity, one determination per sample ~~identification (15 characters maximum) after~~ will be sufficient.

NOTE 10—In the ~~machine displays "Please specify sample name."~~

~~9.4 Wait while~~ case that deviation from the ~~machine displays "Please stand by while processing."~~

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

~~9.6 The density~~ caused by inadequate conditioning of the sample ~~is then displayed, and can either be recorded on separate paper or printed on~~ in the ~~associated printer.~~

~~9.7 Repeating 9.3 through 9.6 will allow~~ bath, place the ~~evaluation of~~ sample back to the bath for an additional ~~samples.~~

~~9.8 To quit density measurements,~~ five minutes then re-measure the ~~operator must activate the "F10" function key.~~ 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 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 [8]

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 each specimen was evaluated six times. This procedure yielded 24 test results for each material under evaluation from each laboratory. ~~NOTE 8—**Caution:**~~

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

[8] 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.

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~~

If S_r and S_R have been calculated from a large enough body of dat0a, 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 – 03:
(*1*) Added to Referenced Documents: D 1248, D 3350, D 4703, D 4976, and E 494.
(*2*) Added Note 3, 4, 8, 10 and Section 9.1.
(*3*) Combine Notes 6 and 7 of the previous revision into Note 5.
(*4*) Deleted the relative humidity requirement for testing.
(*5*) Clarified the number of determinations needed for calibration and sample testing.
(*6*) Deleted instrument's operating procedures.
(*7*) Removed non-mandatory languages.
D 4883 – 96:
(*1*) Added an ISO equivalency statement and sections on keywords and precision and bias.
D 4883 – 99:
Added Note 7.