Designation: D 4883 – 03

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:* [2]

D 618 Practice for Conditioning Plastics for Testing

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

D 883 Terminology Relating to Plastics

D 1248 Specification for Polyethylene Plastics: Extrusion Materials for Wire and Cable

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

D 3350 Specification for Polyethylene Plastics: Pipe and Fittings Materials

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

D 4976 Specification for Polyethylene Plastics: Molding and Extrusion Materials

E 494 Practice for Measuring Ultrasonic Velocity in Materials

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

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 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 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 D 1505.

5.3 Equipment specified in Test Methods D 792.

[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, Section 01, Physical 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 DS 500 instrument can be obtained from R/D Tech (www.rd-tech.com) or Ultra Optec (www.ultraoptec.com).

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

5.4 Equipment specified in Practice D 618.

5.5 Equipment specified in 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 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 D 4703, 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 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 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 D 618, 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 D 4703 Annex 1, Procedure C and be conditioned in accordance with Practice D 618. 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 D 792 or D 1505. Conduct the determinations as specified by the test methods, that is, two determinations for D 792 or three determinations for D 1505. 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 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 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 For the most accurate results, test each sample four times 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: