



Designation: **D1505—10 D1505 – 18**

## Standard Test Method for Density of Plastics by the Density-Gradient Technique<sup>1</sup>

This standard is issued under the fixed designation D1505; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

### 1. Scope\*

1.1 This test method covers the determination of the density of solid plastics.

1.2 This test method is based on observing the level to which a test specimen sinks in a liquid column exhibiting a density gradient, in comparison with standards of known density.

NOTE 1—This test method is equivalent to ISO 1183-2.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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~~ safety, health, and ~~health~~ environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[D883 Terminology Relating to Plastics](#)

[D2839 Practice for Use of a Melt Index Strand for Determining Density of Polyethylene](#)

[D4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

[E2935 Practice for Conducting Equivalence Testing in Laboratory Applications](#)

2.2 *ISO Standard:*

[ISO 1183-2 Methods for Determining the Density and Relative Density of Noncellular Plastics](#)<sup>3</sup>

### 3. Terminology

3.1 Refer to Terminology [D883](#) for definitions of other terms relating to this test method.

3.2 *Definitions:*

3.2.1 *density of plastics*—the weight per unit volume of material at 23°C, expressed as follows:

$$D^{23C}, \text{ g/cm}^3 \quad (1)$$

NOTE 2—Density is to be distinguished from specific gravity, which is the ratio of the weight of a given volume of the material to that of an equal volume of water at a stated temperature.

### 4. Significance and Use

4.1 The density of a solid 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 a means of identification.

<sup>1</sup> 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 [D20.70.01](#)).

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<sup>2</sup> 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.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, [http://www.ansi.org](#).

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

4.2 This test method is designed to yield results accurate to better than 0.05 %.

NOTE 3—Where accuracy of 0.05 % or better is desired, the gradient tube shall be constructed so that vertical distances of 1 mm shall represent density differences no greater than 0.0001 g/cm<sup>3</sup>.<sup>3</sup> The sensitivity of the column is then 0.0001 g/cm<sup>3</sup>-mm. Where less accuracy is needed, the gradient tube shall be constructed to any required sensitivity.

## 5. Apparatus

5.1 *Density-Gradient Tube*—A suitable graduate with ground-glass stopper.<sup>4</sup>

5.2 *Constant-Temperature Bath*—A means of controlling the temperature of the liquid in the tube at 23 ± 0.1°C. A thermostatted water jacket around the tube is a satisfactory and convenient method of achieving this.

5.3 *Glass Floats*—A number of calibrated glass floats covering the density range to be studied and approximately evenly distributed throughout this range.

5.4 *Pycnometer*, for use in determining the densities of the standard floats.

5.5 *Liquids*, suitable for the preparation of a density gradient (Table 1).

**TABLE 1 Liquid Systems for Density-Gradient Tubes**

System	Density Range, g/cm <sup>3</sup>
Methanol-benzyl alcohol	0.80 to 0.92
Isopropanol-water	0.79 to 1.00
Isopropanol-diethylene glycol	0.79 to 1.11
Ethanol-carbon tetrachloride	0.79 to 1.59
Toluene-carbon tetrachloride	0.87 to 1.59
Water-sodium bromide	1.00 to 1.41
Water-calcium nitrate	1.00 to 1.60
Carbon tetrachloride-trimethylene dibromide	1.60 to 1.99
Trimethylene dibromide-ethylene bromide	1.99 to 2.18
Ethylene bromide-bromoform	2.18 to 2.89

NOTE 4—It is very important that none of the liquids used in the tube exert a solvent or chemical effect upon the test specimens during the time of specimen immersion.

5.6 *Hydrometers*—A set of suitable hydrometers covering the range of densities to be measured. These hydrometers shall have 0.001 density graduations.

5.7 *Analytical Balance*, with a sensitivity of 0.0001 g or better.

5.8 *Siphon or Pipet Arrangement*, for filling the gradient tube. This piece of equipment shall be constructed so that the rate of flow of liquid may be regulated to 10 ± 5 mL/min.

## 6. Test Specimen

6.1 The test specimen shall consist of a piece of the material under test. The piece shall be cut to any shape convenient for easy identification, but shall have dimensions that permit the most accurate position measurement of the center of volume of the suspended specimen (Note 5). Care shall be taken in cutting specimens to avoid change in density resulting from compressive stress.

NOTE 5—The equilibrium positions of film specimens in the thickness range from 0.025 to 0.051 mm (0.001 to 0.002 in.) may be affected by interfacial tension. If this effect is suspected, films not less than 0.127 mm (0.005 in.) in thickness shall be tested.

6.2 The specimen shall be free of foreign matter and voids and shall have no cavities or surface characteristics that will cause entrapment of bubbles.

## 7. Preparation of Density-Gradient Columns

7.1 *Preparation of Standard Glass Floats*<sup>5</sup>—Prepare glass floats by any convenient method such that they are fully annealed, approximately spherical, have a maximum diameter less than one fourth the inside diameter of the column, and do not interfere with the test specimens. Prepare a solution (400 to 600 mL) of the liquids to be used in the gradient tube such that the density of the solution is approximately equal to the desired lowest density. When the floats are at room temperature, drop them gently into the solution. Save the floats that sink very slowly, and discard those that sink very fast, or save them for another tube. If necessary to obtain a suitable range of floats, grind selected floats to the desired density by rubbing the head part of the float on a glass plate

<sup>4</sup> Tubes similar to those described in Refs (1) and (2) may also be used.

<sup>5</sup> Manufactured certified glass floats may be purchased.

on which is spread a thin slurry of 400 or 500-mesh silicon carbide (Carborundum) or other appropriate abrasive. Progress shall be followed by dropping the float in the test solution at intervals and noting its change in rate of sinking.

**7.2 Calibration of Standard Glass Floats** (see **Appendix X1**):

**7.2.1** Place a tall cylinder in the constant-temperature bath maintained at  $23 \pm 0.1^\circ\text{C}$ . Fill the cylinder about two thirds full with a solution of two suitable liquids selected from **Table 1**, the density of which can be varied over the desired range by the addition of either liquid to the mixture. After the cylinder and solution have attained temperature equilibrium, place the float in the solution, and if it sinks, add the denser liquid by suitable means with good stirring until the float reverses direction of movement. If the float rises, add the less dense liquid by suitable means with good stirring until the float reverses direction of movement.

**7.2.2** When reversal of movement has been observed, reduce the amount of the liquid additions to that equivalent to  $0.0001\text{-g/cm}^3$  density. When an addition equivalent to  $0.0001\text{-g/cm}^3$  density causes a reversal of movement, or when the float remains completely stationary for at least 15 min, the float and liquid are in satisfactory balance. The cylinder must be covered whenever it is being observed for balance, and the liquid surface must be below the surface of the liquid in the constant-temperature bath. After vigorous stirring, the liquid will continue to move for a considerable length of time; make sure that the observed movement of the float is not due to liquid motion by waiting at least 15 min after stirring has stopped before observing the float.

**7.2.3** When balance has been obtained, fill a freshly cleaned and dried pycnometer with the solution and place it in the  $23 \pm 0.1^\circ\text{C}$  bath for sufficient time to allow temperature equilibrium of the glass. Determine the density of the solution by normal methods and make “in vacuo” corrections for all weighings. Record this as the density of the float. Repeat the procedure for each float.

**7.3 Gradient Tube Preparation** (see **Annex A1** for details):

**7.3.1 Method A**—Stepwise addition.

**7.3.2 Method B**—Continuous filling (liquid entering gradient tube becomes progressively less dense).

**7.3.3 Method C**—Continuous filling (liquid entering gradient tube becomes progressively more dense).

**8. Conditioning**

**8.1** Test specimens whose change in density on conditioning is greater than the accuracy required of the density determination shall be conditioned before testing in accordance with the method listed in the applicable ASTM material specification.

**9. Procedure**

**9.1** Wet three representative test specimens with the less dense of the two liquids used in the tube and gently place them in the tube. Allow the tube and specimens to reach equilibrium, which will require 10 min or more. Thin films of 1 to 2 mils in thickness require approximately  $1\frac{1}{2}$  h to settle, and rechecking after several hours is advisable (**Note 4**).

**9.2** Read the height of each float and each specimen by a line through the individual center of volume and averaging the three values. When a cathetometer is used, measure the height of the floats and specimens from an arbitrary level using a line through their center of volume. If equilibrium is not obtained, the specimen may be imbibing the liquid.

**9.3** Remove old samples without destroying the gradient by slowly withdrawing a wire screen basket attached to a long wire (**Note 6**), which is conveniently done by means of a clock motor. Withdraw the basket from the bottom of the tube and, after cleaning, return it to the bottom of the tube. It is essential that this procedure be performed at a slow enough rate (approximately 30 min/300-mm length of column) so that the density gradient is not disturbed.

**NOTE 6**—Whenever it is observed that air bubbles are collecting on samples in the column, a vacuum applied to the column will correct this.

**10. Calculation**

**10.1** The densities of the samples may be determined graphically or by calculation from the levels to which the samples settle by either of the following methods:

**10.1.1 Graphical Calculation**—Plot float position versus float density on a chart large enough to be read accurately to  $\pm 1$  mm and the desired precision of density. A minimum correlation factor of 0.995 shall be obtained to show the column is acceptable. Plot the positions of the unknown specimens on the chart and read their corresponding densities.

**10.1.2 Numerical Calculation**—Calculate the density by interpolation as follows:

$$\text{Density at } x = a + [(x - y)(b - a)/(z - y)] \quad (2)$$

where:

$a$  and  $b$  = densities of the two standard floats,

$y$  and  $z$  = distances of the two standards,  $a$  and  $b$ , respectively, bracketing the unknown measured from an arbitrary level, and

$x$  = distance of unknown above the same arbitrary level.

**11. Report**

**11.1** Report the following information:

**11.1.1** Density reported as  $D^{23^\circ\text{C}}$ , in grams per cubic centimetre, as the average for three representative test specimens,

- 11.1.2 Number of specimens tested if different than three,
- 11.1.3 Sensitivity of density gradient in grams per cubic centimetre per millimetre,
- 11.1.4 Complete identification of the material tested, and
- 11.1.5 Date of the test.

## 12. Precision and Bias<sup>6</sup>

12.1 *Specimens Molded in One Laboratory and Tested in Several Laboratories*—An interlaboratory test was run in 1981 in which randomized density plaques were supplied to 22 laboratories. Four polyethylene samples of nominal densities of 0.92 to 0.96 g/cm<sup>3</sup> were molded in one laboratory. The data were analyzed using Practice E691, and the results are given in Table 2.

12.2 *Specimens Molded and Tested in Several Laboratories:*

12.2.1 *Samples Prepared Using Practice D4703 in Each Laboratory*—Table 3 is based on a round robin<sup>6</sup> conducted in 1994 in accordance with Practice E691, involving seven materials tested by 7 to 11 laboratories. For each material, all of the samples were prepared by each laboratory, molded in accordance with Procedure C of Annex A1 of Practice D4703, and tested using this test method. The data are for comparison with the data of the same samples tested by Practice D2839. Each test result is an individual determination. Each laboratory obtained six test results for each material.

12.2.2 *Samples Prepared Using Practice D2839 in Each Laboratory*—Table 4 is based on a round robin<sup>6</sup> conducted in 1994 in accordance with Practice E691, involving seven materials tested by 10 to 15 laboratories. For each material, all of the samples were prepared by each laboratory in accordance with Practice D2839. Each test result is an individual determination. Each laboratory obtained six test results for each material.

12.3 *Concept of  $r$  and  $R$* —**Warning**—The following explanations of  $r$  and  $R$  (12.3 – 12.3.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Tables 2-4 shall not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and cannot be 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 12.3 – 12.3.3 will then be valid for each 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:

12.3.1 *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 value below which the absolute difference between two individual test results obtained under repeatability conditions may be expected to occur with a probability of approximately 0.95 (95 %).  $r$  value for that material.

12.3.2 *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 value below which the absolute difference between two individual test results obtained under reproducibility conditions may be expected to occur with a probability of approximately 0.95 (95 %).  $R$  value for that material.

12.3.3 *Any judgment Conducting equivalence testing on numerical data from two sources shall be conducted in accordance with Practice E2935 or 12.2.2 would have an approximate 95 % (0.95) probability of being correct.* any known method for judging the equivalence of two means, for example, a t-test.

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

## 13. Keywords

13.1 ~~density;~~ density; film; gradient; plaque; polyolefins; polyethylene; polypropylene; preparation

**TABLE 2 Precision Data Summary—Polyethylene Density**

Material	Average Density, g/cm <sup>3</sup>	$S_r^A$	$S_R^B$	$r^C$	$R^D$
1	0.9196	0.00029	0.00106	0.00082	0.0045
2	0.9319	0.00012	0.00080	0.00034	0.0023
3	0.9527	0.00033	0.00116	0.00093	0.0033
4	0.9623	0.00062	0.00114	0.00180	0.0033

<sup>A</sup> $S_r$  = 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.

<sup>B</sup> $S_R$  = between-laboratories reproducibility, expressed as standard deviation, for the indicated material.

<sup>C</sup> $r$  = within-laboratory repeatability limit = 2.8  $S_r$ .

<sup>D</sup> $R$  = between-laboratories reproducibility limit = 2.8  $S_R$ .

<sup>6</sup> Supporting data are available from ASTM Headquarters. Request RR:D20-1123.

**TABLE 3 Precision Data—Density, g/cm<sup>3</sup>**

Material	Number of Laboratories	Density, g/cm <sup>3</sup>	$S_r^A$	$S_R^B$	$r^C$	$R^D$
B	7	0.9139	0.00029	0.00088	0.00081	0.00245
F	8	0.9177	0.00018	0.00079	0.00051	0.00221
G	8	0.9220	0.00028	0.00071	0.00078	0.00197
A	11	0.9356	0.00036	0.00105	0.00100	0.00294
E	11	0.9528	0.00046	0.00118	0.00129	0.00331
C	10	0.9619	0.00100	0.00100	0.00103	0.00281
D	9	0.9633	0.00036	0.00137	0.00101	0.00384

<sup>A</sup>  $S_r$  = 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.

<sup>B</sup>  $S_R$  = between-laboratories reproducibility, expressed as standard deviation, for the indicated material.

<sup>C</sup>  $r$  = within-laboratory repeatability limit = 2.8  $S_r$ .

<sup>D</sup>  $R$  = between-laboratories reproducibility limit = 2.8  $S_R$ .

**TABLE 4 Density, g/cm<sup>3</sup>, Samples Prepared in Accordance With Practice D2839**

Material	Number of Laboratories	Density, g/cm <sup>3</sup>	$S_r^A$	$S_R^B$	$r^C$	$R^D$
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

<sup>A</sup>  $S_r$  = 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.

<sup>B</sup>  $S_R$  = between-laboratories reproducibility, expressed as standard deviation, for the indicated material.

<sup>C</sup>  $r$  = within-laboratory repeatability limit = 2.8  $S_r$ .

<sup>D</sup>  $R$  = between-laboratories reproducibility limit = 2.8  $S_R$ .

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## ANNEX

### (Mandatory Information)

#### A1. GRADIENT TUBE PREPARATION

##### A1.1 Method A—Stepwise Addition:

A1.1.1 Using the two liquids that will give the desired density range, and sensitivity ( $S$ ) in grams per cubic centimetre per millimetre, prepare four or more solutions such that each differs from the next heavier by 80  $S$  g/cm<sup>3</sup>. The number of solutions will depend upon the desired density range of the column and shall be determined as follows:

Numbers of solutions to prepare density-gradient column (Note A1.2) =

$$(1 + D_2 - D_1) / 80 S \quad (\text{A1.1})$$

where:

$D_2$  = upper limit of density range desired,

$D_1$  = lower limit of density range desired, and

$S$  = sensitivity, in grams per cubic centimetre per millimetre.

NOTE A1.1—Correct the value of  $(1 + D_2 - D_1) / 80 S$  to the nearest whole number. To prepare these solutions, proceed as follows: Using the hydrometers,

mix the two liquids in the proportions necessary to obtain the desired solutions. Remove the dissolved air from the solutions by gentle heating or an applied vacuum. Then check the density of the solutions at  $23 \pm 0.1^\circ\text{C}$  by means of the hydrometers and, if necessary, add the appropriate air-free liquid until the desired density is obtained.

NOTE A1.2—Where aqueous mixtures are used, 0.5 % aqueous sodium acetate shall be used to prepare the mixture. This reduces the formation of bubbles from dissolution.

NOTE A1.3—In order to obtain a linear gradient in the tube, it is very important that the solutions be homogeneous and at the same temperature when their densities are determined. It is also important that the density difference between the solutions consecutively introduced into the tube be equal.

A1.1.2 By means of a siphon or pipet, fill the gradient tube with an equal volume of each liquid starting with the heaviest, taking appropriate measures to prevent air from being dissolved in the liquid. After the addition of the heaviest liquid, very carefully and slowly pour an equal volume of the second heaviest liquid down the side of the column by holding the siphon or pipet against the side of the tube at a slight angle. Avoid excess agitation and turbulence. In this manner, the “building” of the tube shall be completed.

NOTE A1.4—Density gradients may also be prepared by reversing the procedure described in A1.1.1 and A1.1.2. When this procedure is used, the lightest solution is placed in the tube and the next lightest solution is very carefully and slowly “placed” in the bottom of the tube by means of a pipet or siphon, which just touches the bottom of the tube. In this manner the “building” of the tube shall be completed.

A1.1.3 If the tube is not already in a constant-temperature bath, transfer the tube, with as little agitation as possible, to the constant-temperature bath maintained at  $23 \pm 0.1^\circ\text{C}$ . The bath level will be equal to or greater than the solution in the tube, and provision shall be made for vibrationless mounting of the tube.

A1.1.4 For every 254 mm of length of tube, dip a minimum of five clean calibrated floats, spanning the effective range of the column, into the less dense solvent used in the preparation of the gradient tube and add them to the tube. By means of a stirrer (for example, a small coiled wire or other appropriate stirring device) mix the different layers of the tube gently by stirring horizontally until the least dense and most dense floats span the required range of the gradient tube. If, at this time, it is observed that the floats are “bunched” together and not spread out evenly in the tube, discard the solution and repeat the procedure. Then cap the tube and keep it in the constant-temperature bath for a minimum of 24 h.

A1.1.5 At the end of this time, plot the density of floats versus the height of floats to observe whether or not a fairly smooth and nearly linear curve is obtained. Some small irregularities may be seen, but they ~~should~~ shall be slight. A minimum correlation factor of 0.995 shall be obtained to prove linearity of the column. Whenever an irregular curve is obtained, the solution in the tube shall be discarded and a new gradient prepared. In the event a column is disturbed in a manner which causes a bead or beads (top or bottom, one or two) to give a bad correlation factor, that bead or beads height may be removed from the correlation chart as long as the sample to be analyzed does not fall within that range. There must be four consecutive beads in correlation. The minimum correlation factor shall be 0.995.

NOTE A1.5—Gradient systems may remain stable for several months.

## A1.2 Method B—Continuous Filling with Liquid Entering Gradient Tube Becoming Progressively Less Dense:

A1.2.1 Assemble the apparatus as shown in Fig. A1.1, using beakers of the same diameter. Then select an appropriate amount of two suitable liquids which previously have been carefully deaerated by gentle heating or an applied vacuum. Typical liquid systems for density-gradient tubes are listed in Table 1. The volume of the more dense liquid used in the mixer (Beaker B shown in Fig. A1.1) must be equal to at least one half of the total volume desired in the gradient tube. An estimate of the volume of the less dense liquid required in Beaker A to establish flow from A to B can be obtained from the following inequality:

$$V_A > d_B V_B / d_A \quad (\text{A1.2})$$

where:

- $V_A$  = starting liquid volume in Beaker A,
- $V_B$  = starting liquid volume in Beaker B,
- $d_A$  = density of the starting liquid in Beaker A, and
- $d_B$  = density of the starting liquid in Beaker B.