Designation: D 1505 – 98$^{\epsilon 1}$

Standard Test Method for Density of Plastics by the Density-Gradient Technique[1]

This standard is issued under the fixed designation D 1505; 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.

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

ϵ^1 Note—Editorially changed 7.2.1 to clarify the location for liquid selection in April 2002.

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—The comparable ISO method is R1183-1987.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer[2]

D 2839 Practice for Use of a Melt Index Strand for Determining Density of Polyethylene[3]

D 4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

2.2 *ISO Standard:*

R 1183-1987 Methods for Determining the Density and Relative Density of Noncellular Plastics[6]

3. Terminology

3.1 *Definition:*

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

$$D^{23C},\ \text{g/cm}^3 \tag{1}$$

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.

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.[3] The sensitivity of the column is then 0.0001 g/cm^3·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.[7]

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).

Note 4—It is very important that none of the liquids used in the tube

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastic and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

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[2] Discontinued; see *1992 Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 08.02.

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

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

[6] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

[7] Tubes similar to those described in Refs **(6)** and **(12)** may also be used.

TABLE 1 Liquid Systems for Density-Gradient Tubes

System	Density Range, g/cm^3
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

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 should have 0.001 density graduations.

5.7 *Analytical Balance*, with a sensitivity of 0.001 g.

5.8 *Siphon or Pipet Arrangement*, for filling the gradient tube. This piece of equipment should 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 may be cut to any shape convenient for easy identification, but should have dimensions that permit the most accurate position measurement of the center of volume of the suspended specimen (Note 5). Care should 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 affect is suspected, films not less than 0.127 mm (0.005 in.) in thickness should 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*[8]—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 on which is spread a thin slurry of 400 or 500-mesh silicon carbide (Carborundum) or other appropriate abrasive. Progress may 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 ± 0.1°C. Then 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-g/cm^3 density. When an addition equivalent to 0.0001-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 may 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 ± 0.1°C bath for sufficient time to allow temperature equilibrium of the glass. Determine the density of the solution by normal methods (Test Method D 941) 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 appendix 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 may be 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 1½ 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

[8] Glass floats may be purchased from American Density Materials, Inc., RD2 Box 38E, Belvidere, NJ 07823.

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 Old samples can be removed without destroying the gradient by slowly withdrawing a wire screen basket attached to a long wire (Note 6). This can be 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 ±1 mm and the desired precision of density. 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^{23C}, 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 [9]

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^3 were molded in one laboratory. The data were analyzed using Practice E 691, and the results are given in Table 2.

12.2 *Specimens Molded and Tested in Several Laboratories*:

12.2.1 *Samples Prepared Using Practice D 4703 in Each Laboratory*—Table 3 is based on a round robin[9] conducted in 1994 in accordance with Practice E 691, 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 D 4703, and tested using this test method. The data are for comparison with the data of the same samples tested by Practice D 2839. Each test result is an individual determination. Each laboratory obtained six test results for each material.

12.2.2 *Samples Prepared Using Practice D 2839 in Each Laboratory*—Table 4 is based on a round robin[9] conducted in 1994 in accordance with Practice E 691, 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 D 2839. Each test result is an individual determination. Each laboratory obtained six test results for each material.

NOTE 7—**Caution:** 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 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 12.3-12.3.3 would then be valid for each data.

12.3 *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:

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 r value for that material.

[9] Supporting data are available from ASTM Headquarters. Request RR:D20-1123.

TABLE 2 Precision Data Summary—Polyethylene Density

Material	Average Density, g/cm^3	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

^{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 = between-laboratories reproducibility limit = 2.8 S_R.