REFERENCES

Bailey, W. J., and Katsuki, H. (1973), ACS Div. Polym. Chem., **14**, 1169.

Bailey, W. J., Katsuki, H., and Endo, T. (1974), ACS Div. Polym. Chem., **15**,445.

Bailey, W. J. (1977), Polym. Preprints, **18**, 17.

Bailey, W.J. (1990), Mat. Sci. Eng., **A126**, 271.

Dunkers, J., Zarate, E. A., and Ishida, H. (accepted), J. Phys. Chem.

Hale, A., Macosko, C. W., and Bair, H. E. (1991), Macromolecules, **24**, 2610.

He, P., Zhou, Z., and Pan, C. (1989), J. Mat. Sci., **24**, 1528.

He, P., and Zhou, Z. (1991), J. Mat. Sci., **26**, 3792.

Holly, F. W., and Cope, A. C. (1944), J. Am. Chem. Soc., **66**, 1875.

Ishida, H., and Allen, D. J. (in press), J. Polym. Sci., Polym. Phys. Ed.

Ishida, H., and Low, H. Y. (submitted), Macromolecules.

Ning, X., and Ishida, H. (1994), J. Polym. Sci., Polym. Chem. Ed., **32**, 1121.

Ning, X., and Ishida, H. (1994), J. Polym. Sci., Polym. Phys. Ed., **32**, 921.

Pang, K. P., and Gillham, J. K. (1989), J. Appl. Polym. Sci., **37**, 1969.

Riess, G., Schwob, J.M., Guth, G., Roche, M., and Lande, B. (1986), "Advances in Polymer Science," B.M. Culbertson and J.E. McGrath, Eds., Plenum, New York p.27.

Shimbo, M., Ochi, M.,and Shigeta, Y. J. (1981), J. Appl. Polym. Sci., **26**, 2265.

Shimbo, M., Ochi, M., Inamura, T., and Inoue, M. (1985), J. Mat. Sci., **20**, 2965.

Shreiber, H., Ger. Offen. 2225504 (1973); and Ger. Offen. 2323936 (1973).

Venditti, R. A., Gillham, J. K., Chin, E., and Houlihan, F.M. (1994), J. Appl. Polym. Sci., **53**, 455.

Wisanrakkit, G., and Gillham, J. K. (1990), J. Appl. Polym. Sci., **41**, 2885.

APPENDICES

Appendix A

1) Differential Scanning Calorimetry data of B-a monomer.

Tg (°C)	Heat of reaction
26.81	316.6

2) Differential Scanning Calorimetry data of B-a cured at 155 °C.

Curing Time	Tg (°C)	Heat of reaction
30 min	41.57	314.4
40 min	39.03	309.2
1 h	52.31	297.2
2 h	73.78	277.0
3 h	109.22	229.3
4 h	122.01	200.3
5 h	125.50	196.8
6 h	132.70	171.7
7 h	129.28	178.5
8 h	136.84	157.9
9 h	136.99	155.8
10 h	141.52	138.6

3) Differential Scanning Calorimetry data of B-a cured at 165 °C.

Curing Time	Tg (°C)	Heat of reaction
30 min	40.94	314.3
40 min	43.02	315.7
1 h	60.65	280.2
2 h	125.01	190.4
3 h	133.13	166.4
4 h	143.86	134.6
5 h	145.96	129.8
6 h	146.97	129.0
7 h	152.53	103.8
8 h	151.07	118.1
9 h	159.51	75.89
10 h	154.68	82.57

4) Differential Scanning Calorimetry data of B-a cured at 175 °C.

Curing Time	Tg (°C)	Heat of reaction
30 min	47.33	307.0
40 min	52.08	293.8
1 h	105.98	239.5
2 h	138.61	149.9
3 h	157.10	87.27
4 h	157.33	89.70
5 h	160.39	73.61
6 h	161.22	73.16

Curing Time	Tg (°C)	Heat of reaction
7 h	161.36	36.86
8 h	162.97	53.05
9 h	164.50	39.66
10 h	165.96	45.23

5) Differential Scanning Calorimetry data of B-a cured at 185 °C.

Curing Time	Tg (°C)	Heat of reaction
30 min	86.76	269.6
40 min	130.11	199.8
1 h	144.4	124.2
2 h	163.76	59.75
3 h	168.22	34.78
4 h	169.88	23.66
5 h	166.28	21.66
6 h	167.66	12.32
7 h	168.08	5.268
8 h	168.26	4.034
9 h	168.45	3.796
10 h	167.98	1.633

Appendix B

- American Society for Testing and Materials (ASTM D792)

Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[1]

This standard is issued under the fixed designation D 792; 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 to replace method 5011 and 5012 of Federal Test Method Standard 406 and 14011 and 14021 of FTMS 601. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods describe the determination of the specific gravity (relative density) and density of solid plastics in forms such as sheets, rods, tubes, or molded items.

1.2 Two test methods are described:

1.2.1 *Test Method A*—For testing solid plastics in water, and

1.2.2 *Test Method B*—For testing solid plastics in liquids other than water.

NOTE 1—Alternatively, Test Method D 1505 may be applied to many such forms, as well as to films and sheeting.

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

D 891 Test Methods for Specific Gravity of Liquid Industrial Chemicals[3]

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

D 1622 Test Method for Apparent Density of Rigid Cellular Plastics[2]

D 1898 Practice for Sampling of Plastics[2]

E 1 Specification for ASTM Thermometers[4]

E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[5]

E 380 Practice for Use of the International System of Units (SI)[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *specific gravity (relative density)*—the ratio of the mass in air of a unit volume of the impermeable portion of the material at 23°C to the mass in air of equal density of an equal volume of gas-free distilled water at the same temperature. The form of expression shall be:

Specific gravity (relative density) 23/23°C
(or sp gr 23/23°C)

NOTE 2—This definition is essentially equivalent to the definition for apparent specific gravity and apparent density in Terminology E 12, because the small percentage difference introduced by not correcting for the buoyancy of air is insignificant for most purposes.

3.1.2 *density*—the mass in air in kilograms per cubic metre of impermeable portion of the material at 23°C. The form of expression shall be:

D^{23}, kg/m^3 (Notes 2, 3, 4)

NOTE 3—The SI unit of density, as defined in Practice E 380 is kg/m^3. To convert density in g/cm^3 to density in kg/m^3, multiply by 1000.

NOTE 4—Specific gravity 23/23°C can be converted to density 23°C, mg/m^3, by use of the following equation:

$$D^{23\,C},\ kg/m^3 = \text{sp gr } 23/23°C \times 997.6$$

4. Summary of Test Methods

4.1 Determine the mass of a specimen of the solid plastic in air. It is then immersed in a liquid, its apparent mass upon immersion is determined, and its specific gravity (relative density) calculated.

5. Significance and Use

5.1 The specific gravity or density of a solid is a property that can be measured conveniently to identify a material, to follow physical changes in a sample, to indicate degree of uniformity among different sampling units or specimens, or to indicate the average density of a large item.

5.2 Changes in density of a single specimen may be due to changes in crystallinity, loss of plasticizer, absorption of solvent, or to other causes. Portions of a sample may differ in density because of difference in crystallinity, thermal history, porosity, and composition (types or proportions of resin, plasticizer, pigment, or filler).

NOTE 5—Reference is made to Test Method D 1622.

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[1] These test methods are under the jurisdiction of ASTM Committee D-20 on Plastics and are the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

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

[3] *Annual Book of ASTM Standards*, Vol 15.05.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 15.05.

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

5.3 Density is useful for calculating strength-weight and cost-weight ratios.

6. Sampling

6.1 The sampling units used for the determination of specific gravity (relative density) shall be representative of the quantity of product for which the data are required, in accordance with Practice D 1898.

6.1.1 If it is known or suspected that the sample consists of two or more layers or sections having different specific gravities, either complete finished parts or complete cross sections of the parts or shapes shall be used as the specimens, or separate specimens shall be taken and tested from each layer. The specific gravity (relative density) of the total part cannot be obtained by adding the specific gravity of the layers, unless relative percentages of the layers are taken into account.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at 23 ± 2°C 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 of disagreement, the tolerances shall be 1°C and ±2 % relative humidity.

7.2 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C and 50 ± 5 % relative humidity, unless otherwise specified in the test methods or in this specification. In cases of disagreement, the tolerances shall be 1°C and ±2 % relative humidity.

TEST METHOD A FOR TESTING SOLID PLASTICS IN WATER (SPECIMENS 1 TO 50 g)

8. Scope

8.1 This test method involves weighing a one-piece specimen of 1 to 50 g in water, using a sinker with plastics that are lighter than water. This test method is suitable for plastics that are wet by, but otherwise not affected by water.

9. Apparatus

9.1 *Analytical Balance*—A balance with a precision within 0.1 mg, accuracy within 0.05 % relative (that is, 0.05 % of the mass of the specimen in air), and equipped with a stationary support for the immersion vessel above the balance pan ("pan straddle").

NOTE 6—Assurance that the balance meets the performance requirements should be provided by frequent checks on adjustments of zero point and sensitivity and by periodic calibration for absolute accuracy, using standard masses.

9.2 *Wire*—A corrosion-resistant wire for suspending the specimen.

9.3 *Sinker*—A sinker for use with specimens of plastics that have specific gravities less than 1.000. The sinker shall: (*1*) be corrosion-resistant; (*2*) have a specific gravity of not less than 7.0; (*3*) have smooth surfaces and a regular shape; and (*4*) be slightly heavier than necessary to sink the specimen. The sinker should have an opening to facilitate attachment to the specimen and wire.

9.4 *Immersion Vessel*—A beaker or other wide-mouthed vessel for holding the water and immersed specimen.

9.5 *Thermometer*—A thermometer with an accuracy of ±1°C is required if the test is not performed in the standard laboratory atmosphere of Practice D 618, (refer to 17.4).

10. Materials

10.1 *Water*—The water shall be substantially air-free, distilled, or demineralized water.

NOTE 7—Water may be rendered substantially air-free by boiling and cooling or by shaking under vacuum in a heavy-walled vacuum flask. (Precaution: Use gloves and shielding.) If the water does not wet the specimen, a few drops of a wetting agent shall be added. If this solution does not wet the specimen, Method B shall be used.

11. Test Specimens

11.1 The test specimen shall be a single piece of the material under test of any size and shape that can conveniently be prepared and tested, provided that its volume shall be not less than 1 cm^3 and its surface and edges shall be made smooth. The thickness of the specimen should be at least 1 mm for each 1 g of weight. A specimen weighing 1 to 5 g usually will be found convenient, but specimens up to approximately 50 g may be used (Note 8). Care should be taken in cutting specimens to avoid changes in density resulting from compressive stresses or frictional heating.

NOTE 8—Specifications for certain plastics require a particular method of specimen preparation and should be consulted if applicable.

11.2 The specimen shall be free from oil, grease, and other foreign matter.

12. Procedure

12.1 Weigh the specimen in air to the nearest 0.1 mg for specimens of mass 1 to 10 g or to the nearest mg for specimens of mass more than 10 to 50 g.

12.2 Attach to the balance a piece of fine wire sufficiently long to reach from the hook above the pan to the support for the immersion vessel. Attach the specimen to the wire such that it is suspended about 25 mm above the vessel support.

NOTE 9—The specimen may be weighed in air after hanging from the wire. In this case, record the mass of the specimen, a = (mass of specimen + wire, in air) − (mass of wire in air).

12.3 Mount the immersion vessel on the support, and completely immerse the suspended specimen (and sinkers, if used) in water (10.1) at a temperature of 23 ± 2°C. The vessel must not touch wire or specimen. Remove any bubbles adhering to the specimen, wire, or sinker, paying particular attention to holes in the specimen and sinker. Usually these bubbles can be removed by rubbing them with another wire. If the bubbles cannot be removed by this method or if bubbles are continuously formed (as from dissolved gases), the use of vacuum is recommended (Note 11). Determine the mass of the suspended specimen to the required precision (12.1) (Note 10). Record this apparent mass as b (the mass of the specimen, sinker, if used, and the partially immersed wire in liquid). Unless otherwise specified, weigh rapidly in order to minimize absorption of water by the specimen.

NOTE 10—It may be necessary to change the sensitivity adjustment of the balance to overcome the damping effect of the immersed specimen.

NOTE 11—Some specimens may contain absorbed or dissolved gases or irregularities which tend to trap air bubbles; any of these may affect the density values obtained. In such cases, the immersed specimen may

be subjected to vacuum in a separate vessel until evolution of bubbles has substantially ceased before weighing (see Test Method B). It must also be demonstrated that the use of this technique leads to results of the required degree of precision.

12.4 Weigh the wire (and sinker, if used) in water with immersion to the same depth as used in the previous step (Notes 12 and 13). Record this height as w (mass of the wire in liquid).

NOTE 12—It is convenient to mark the level of immersion by means of a shallow notch filed in the wire. The finer the wire, the greater the tolerance which may be permitted in adjusting the level of immersion between weighings. With wire Awg No. 36 or finer, disregard its degrees of immersion and, if no sinker is used, use the mass of the wire in air as w.

NOTE 13—If the wire is left attached to the balance arm during a series of determinations, the mass a may be determined either with the aid of a tare on the other arm of the balance or as in Note 11. In such cases, care must be taken that the change of mass of the wire (for example, from visible water) between readings does not exceed the desired precision.

12.5 Repeat the procedure for the required number of specimens. Two specimens per sample are recommended. Determine acceptability of number of replicate test specimens by comparing results with precision data given in Tables 1 and 2 of Section 23. Additional specimens may be required to give the desired precision.

13. Calculation

13.1 Calculate the specific gravity of the plastic as follows:

$$\text{Sp gr } 23/23°C = a/(a + w - b)$$

where:

a = apparent mass of specimen, without wire or sinker, in air,

b = apparent mass of specimen (and of sinker, if used) completely immersed and of the wire partially immersed in liquid, and

w = apparent mass of totally immersed sinker (if used) and of partially immersed wire.

13.2 Calculate the density of the plastic as follows:

$$D^{23C}, \text{ kg/m}^3 = \text{sp gr } 23/23°C \times 997.6$$

14. Report

14.1 Report the following information:

14.1.1 Complete identification of the material or product tested, including method of specimen preparation and conditioning,

14.1.2 Average specific gravity (relative density) for all specimens from a sampling unit, reported as sp gr 23/23°C = _____, or average density reported as D^{23C} = _____ kg/m^3,

14.1.3 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 on a homogeneous material or the averages plus these measures of dispersion on different layers or areas of a nonhomogeneous product,

14.1.4 Any evidence of porosity of the material or specimen,

14.1.5 The method of test (Method A of Methods D 792), and

14.1.6 Date of test.

15. Precision and Bias

15.1 See Section 23.

TEST METHOD B FOR TESTING SOLID PLASTICS IN LIQUIDS OTHER THAN WATER (SPECIMENS 1 TO 50 g)

16. Scope

16.1 Test Method B uses a liquid other than water for testing one-piece specimens, 1 to 50 g, of plastics that are affected by water or which are lighter than water.

17. Apparatus

17.1 The apparatus shall include the balance, wire, and immersion vessel of Section 8, and, optionally, the following:

17.2 *Pycnometer with Thermometer*—A 25-mL specific gravity bottle with thermometer, or

17.3 *Pycnometer*—A pycnometer of the Weld type, preferably with a capacity of about 25 mL and an external cap over the stopper.

17.4 *Thermometer*—A thermometer having not fewer than four divisions per °C over a temperature range of not less than 5°C or 10°F above and below the standard temperature, and having an ice point for calibration. A thermometer short enough to be handled inside the balance case will be found convenient. ASTM Thermometer 23C (see Specification E 1) and Anschutz-type thermometers have been found satisfactory for this purpose.

17.5 *Constant-Temperature Bath*—An appropriate constant-temperature bath adjusted to maintain a temperature of 23 ± 0.1°C.

18. Materials

18.1 *Immersion Liquid*—The liquid used shall not dissolve, swell, or otherwise affect the specimen, but should wet it and should have a specific gravity less than that of the specimen. In addition, the immersion liquid should be nonhygroscopic, have a low vapor pressure, a low viscosity, and a high flash point, and should leave little or no waxy or tarry residue on evaporation. A narrow cut distilled from kerosine meets these requirements for many plastics. The specific gravity 23/23°C of the immersion liquid shall be determined shortly before and after each use in this method to a precision of at least 0.1 % relative, unless it has been

TABLE 1 Test Method A Specific Gravity Tested in Water

Material	Mean	S_r	S_R	I_r	I_R
Polypropylene	0.9007	0.00196	0.00297	0.00555	0.00841
Cellulose Acetate Butyrate	1.1973	0.00232	0.00304	0.00657	0.00860
Polyphenylene Sulfide	1.1708	0.00540	0.00738	0.01528	0.02089
Thermoset	1.3136	0.00271	0.00313	0.00767	0.02171
Polyvinyl Chloride	1.3396	0.00243	0.00615	0.00688	0.01947

TABLE 2 Test Method B Specific Gravity Tested in Liquids Other Than Water

Material	Mean	S_r	S_R	I_r	I_R
Polypropylene	0.9023	0.00139	0.00239	0.00393	0.00676
LDPE	0.9215	0.00109	0.00195	0.00308	0.00552
HDPE	0.9678	0.00126	0.00189	0.00356	0.01007
Thermoset	1.3130	0.00160	0.00217	0.00453	0.01282

established experimentally in the particular application that a lesser frequency of determination can be used to assure the desired precision.

NOTE 14—For the determination of the specific gravity of the liquid, the use of a standard plummet of known volume (Note 15) or of Method A, C, or D of Test Methods D 891, using the modifications required to give specific gravity 23/23°C instead of specific gravity 60/60°F, is recommended. One suggested procedure is the following:

If a constant-temperature water bath is not available, determine the mass of the clean, dry pycnometer with thermometer to the nearest 0.1 mg on an analytical balance. Fill the pycnometer with water (10.1) cooler than 23°C. Insert the thermometer-stopper, causing excess water to be expelled through the side arm. Permit the filled bottle to warm in air until the thermometer reads 23.0°C. Remove the drop of water at the tip of the side arm with a bit of filter paper, taking care not to draw any liquid from within the capillary, place the cap over the side arm, wipe the outside carefully, and determine the mass of the filled bottle again to the nearest 0.2 mg. Empty the pycnometer, dry, and fill and determine the mass with the other liquid in the same manner as was done with the water. Calculate the specific gravity 23/23°C of the liquid, d, as follows:

$$d = (b - e)/(w - e)$$

where:

e = apparent mass of empty pycnometer,
w = apparent mass of pycnometer filled with water at 23.0°C, and
b = apparent mass of pycnometer filled with liquid at 23.0°C.

If a constant-temperature water bath is available, a pycnometer without a thermometer may be used (compare 30.2).

NOTE 15—One standard object which has been found satisfactory for this purpose is the Reimann Thermometer Plummet. These are normally supplied calibrated for measurements at temperatures other than 23/23°C, so that recalibration is necessary for the purposes of these methods.

19. Test Specimens

19.1 See Section 11.

20. Procedure

20.1 The procedure shall be similar to Section 12, except for the choice of immersion liquid, and the temperature during the immersed weighing (12.3) shall be 23 ± 0.5°C.

21. Calculation

21.1 The calculations shall be similar to Section 13, except that d, the specific gravity 23/23°C of the liquid, shall be placed in the numerator:

$$\text{Sp gr } 23/23°C = (a \times d)/(a + w - b)$$

22. Report

22.1 See Section 14.

23. Precision and Bias[7]

23.1 Tables 1 and 2 are based on round robins conducted in 1985 involving five materials tested by six laboratories for Test Method A. Four materials were tested with Test Method B by six laboratories. Each test result was based on two individual determinations. Each laboratory obtained four test results for each material.

23.2 In Tables 1 and 2, for the materials indicated, and for mean values that are derived from testing two specimens:

23.2.1 S_r is the within-laboratory standard deviation of the mean and $I_r = 2.83\ S_r$. (See 23.2.3 for application of I_r.)

23.2.2 S_R is the between-laboratory standard deviation of the mean, and $I_R = 2.83\ S_R$. (See 23.2.4 for application of I_R.)

23.2.3 *Repeatability*—In comparing two mean values for the same material obtained by the same operator using the same equipment on the same day, the means should be judged not equivalent if they differ by more than the I_r value for that material and condition.

23.2.4 *Reproducibility*—In comparing two mean values for the same material obtained by different operators using different equipment on different days, the means should be judged not equivalent if they differ by more than the I_R value for that material and condition. (This applies between different laboratories or between different equipment within the same laboratory.)

23.2.5 The judgments per 23.2.3 and 23.2.4 will have approximate 95 % (0.95) probability of being correct.

23.2.6 Other materials may give somewhat different results.

23.3 For further information on the methodology used in this section, see Practice E 691.

24. Keywords

24.1 density; relative density; specific gravity

[7] Supporting data are available from ASTM Headquarters. Request RR:D 20 - 1133

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