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# Standard Test Method for Density of Glass by the Sink-Float Comparator[1]

This standard is issued under the fixed designation C729; 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 glass or nonporous solids of density from 1.1 g/cm$^3$ to 3.3 g/cm$^3$. It can be used to determine the apparent density of ceramics or solids, preferably of known porosity.

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

1.3 *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:*[2]

D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

E77 Test Method for Inspection and Verification of Thermometers

F77 Test Method for Apparent Density of Ceramics for Electron Device and Semiconductor Application (Withdrawn 2001)[3]

## 3. Summary of Method

3.1 The specimen of unknown density is compared with a reference standard of known density. The specimen to be measured is placed in a test tube containing a solution whose density at 35 °C is within 0.0200 g/cm$^3$ of the density of the specimen at 25 °C. The solution is prepared using miscible liquids of known densities bracketing the desired range. The tube also contains a glass density reference standard whose density at 35 °C is close to that of the solution at 35 °C; the tube is immersed in a variable-temperature comparator bath. Initially the solutions, specimen, and standard are at a temperature near 25 °C, and both the standard and the specimen float in the solution. The temperature of the system is raised at a uniform rate. Because the volumetric expansion coefficient of the solution is much higher than those of the glass pieces, its density decreases more rapidly and eventually both the standard and the specimen will sink (settle) in the solution. The temperatures at which the specimen and standard reach the mid-point of the test tube are noted and by use of special tables, the density of the specimen is obtained.

3.2 *Range of a Given Density Solution*—A given density solution can be used to measure specimens whose density is within ±0.0200 g/cm$^3$ of the density of the solution at 35 °C, by operating the comparator bath in the range 25 °C to 45 °C.



## 4. Significance and Use

4.1 The sink-float comparator method of test for glass density provides the most accurate (yet convenient for practical applications) method of evaluating the density of small pieces or specimens of glass. The data obtained are useful for daily quality control of production, acceptance or rejection under specifications, and for special purposes in research and development.

4.2 Although this test scope is limited to a density range from 1.1 g/cm$^3$ to 3.3 g/cm$^3$, it may be extended (in principle) to higher densities by the use of other miscible liquids (Test Method F77) such as water and thallium malonate-formate (approximately 5.0 g/cm$^3$). The stability of the liquid and the precision of the test may be reduced somewhat, however, at higher densities.

## 5. Apparatus

5.1 *Single Tube and Multiple-Tube Comparators* (Test Method E77)—A single-tube comparator can be constructed from materials readily available in a typical laboratory, and useful if one wishes to measure the density of materials within a fairly narrow range, or if only a few tests need to be run each day. The multiple-tube comparator can be purchased commercially. It is useful if materials with a wide range of density must

[1] This test method is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.04 on Physical and Mechanical Properties.

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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 last approved version of this historical standard is referenced on www.astm.org.

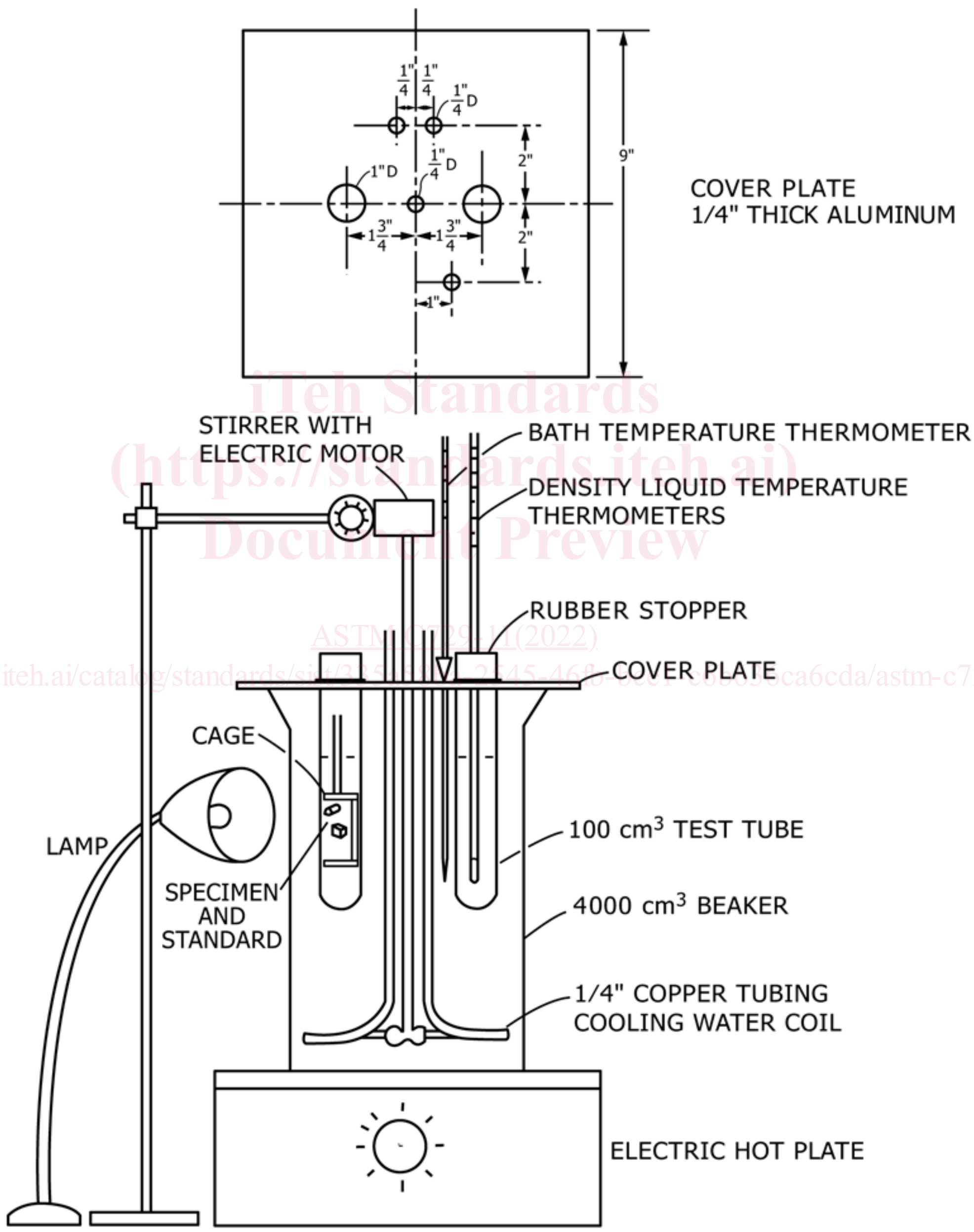
be tested or if many specimens must be tested each day. The comparators shall consist of the following:

5.1.1 *Single-Tube Comparator* (Fig. 1):

5.1.1.1 *Circulating Water Bath,* consisting of a 4000 $cm^3$ beaker, a cover plate supporting test tubes and thermometer, a cooling water coil made from copper tubing, an electrically driven stirrer, and containing an immersion heater with rheostat for controlling heating rate, or heated by an external heat source such as a hot plate.

5.1.1.2 *Test Tubes,* two, 100 $cm^3$ capacity. The cover plate supports the test tubes, which extended into the water bath. One tube contains the density solution, the test specimen, the standard, and a glass or TFE-fluorocarbon cage (Fig. 2) that keeps the specimens immersed in the solution. The second test tube contains density solution and a thermometer; both test tubes employ rubber stoppers for supporting the cage or thermometer.

5.1.1.3 *Thermometers,* two, mercury, readable to 0.1 °C between 20 °C and 50 °C. One thermometer passes through a rubber stopper supported by the cover plate into the water bath. The second thermometer passes through a rubber stopper into the test tube that contains density solution only. Thermistors,






**Metric Equivalents**

| in. | ¼ | ½ | 1 | 1¾ | 2 | 9 |
|---|---|---|---|---|---|---|
| mm | 6.4 | 12.7 | 25.4 | 44.4 | 51 | 229 |

**FIG. 1 Single Tube Sink-Float Density Apparatus**

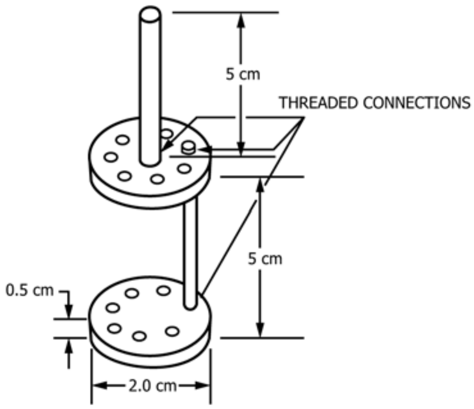



**FIG. 2 TFE-Flourocarbon Cage for 100-mL Test Tube**

resistive thermal devices (RTD), or thermocouples capable of measuring and displaying at least 0.1 °C accuracy between 20 °C and 50 °C can be used in lieu of mercury thermometers.

5.1.2 *Multiple-Tube Comparator*—The commercially obtainable multiple-tube comparator employs the same principle as the single-tube comparator, except that the multiple-tube type contains additional specimen tubes. These specimen tubes may contain similar density solutions if a large number of specimens with similar density are to be measured; they may contain density solutions of differing density if a number of specimens with a range of densities are to be measured.

## 6. Reagents and Materials

6.1 *Density Reference Standards*—The reference standard shall be a solid piece of glass with a volume between 0.10 $cm^3$ and 0.15 $cm^3$, and a ratio of major to minor dimensions not exceeding 2.0. It shall have a smooth surface and be free of seeds, cords, and cracks. A quantity of such standards may be cut from a 20-g piece of glass similarly free of defects, with density at 25 °C ($\rho_{25}$) known to ±0.0001 g⁄$cm^3$. The density of such a standard glass can be determined to ±0.00001 g/$cm^3$ by a precise buoyancy method.[4] Determine the settling temperature of each reference standard to the nearest 0.1 °C and discard any that deviate more than 0.1 °C from mean temperature. Less precise density standards are commercially available.

6.2 *Density Solution*—The following organic liquids[5] are mixed to provide a solution of the desired density:

6.2.1 *Isopropyl Salicylate,* density (25 °C) approximately 1.10 g/$cm^3$ or alpha-bromonaphthalene, density (25 °C) approximately 1.49 g/$cm^3$.

6.2.2 *sym-Tetrabromoethane,* density (25 °C) approximately 2.96 g/$cm^3$.

6.2.3 *Methylene Iodide,* density (25 °C) approximately 3.32 g⁄$cm^3$.

NOTE 1—Methylene iodide, *sym*-tetrabromoethane, and alpha-bromonaphthalene are light-sensitive. These liquids should be stored in light-protective containers. A piece of copper wire in the methylene iodide container will help retard decomposition.

6.2.4 The density solution consists of mixtures of isopropyl salicylate and *sym*-tetrabromoethane for densities between 1.10 g⁄$cm^3$ and 2.96 g/$cm^3$, and of *sym*-tetrabromoethane and methylene iodide for densities between 2.96 g⁄$cm^3$ and 3.32 g⁄$cm^3$. Proper amounts of the two liquids to be used are found by simultaneous solution of:

$$\rho_s V_s = \rho_1 V_1 + \rho_2 V_2 \quad (1)$$

$$V_s = V_1 + V_2 \quad (2)$$

$$\rho_s = (\rho_1 V_1 + \rho_2 V_2)/(V_1 + V_2) \quad (3)$$

where:

$\rho_s$ = density of solution − density of standard at 35 °C,
$V_s$ = volume of solution to be prepared,
$\rho_1$ and $\rho_2$ = densities of the component liquids at 35 °C, and
$V_1$ and $V_2$ = volumes of the component liquids at 35 °C.

6.2.5 *Solution Preparation*—Approximate volumes of liquids required to supply desired density $\rho_s$ are shown in Table 1. Mix the two required volumes of liquids 1 and 2 (6.2.4) in a beaker, set on a hot plate, and warm to 35 °C. Place a density standard in the solution. Adjust the mixture by adding one or

**TABLE 1 Volumes of Liquids for Solutions of Various Densities**

| $\rho_s$ g/$cm^3$ at 35°C | Volume of Material Used, $cm^3$ | | |
|---|---|---|---|
| | Isopropyl Salicylate | *sym*-Tetra-bromo-ethane | Methylene Iodide |
| 2.103 | 135 | 165 | ... |
| 2.136 | 127 | 173 | ... |
| 2.190 | 120 | 180 | ... |
| 2.222 | 115 | 185 | ... |
| 2.236 | 113 | 187 | ... |
| 2.257 | 109 | 191 | ... |
| 2.291 | 104 | 196 | ... |
| 2.315 | 100 | 200 | ... |
| 2.335 | 95 | 205 | ... |
| 2.363 | 92 | 208 | ... |
| 2.403 | 85 | 215 | ... |
| 2.434 | 80 | 220 | ... |
| 2.448 | 78 | 222 | ... |
| 2.473 | 74 | 226 | ... |
| 2.495 | 70 | 230 | ... |
| 2.511 | 68 | 232 | ... |
| 2.529 | 65 | 235 | ... |
| 2.560 | 60 | 240 | ... |
| 2.589 | 56 | 244 | ... |
| 2.596 | 54 | 246 | ... |
| 2.619 | 50 | 250 | ... |
| 2.633 | 48 | 252 | ... |
| 2.669 | 42 | 258 | ... |
| 2.702 | 37 | 263 | ... |
| 2.728 | 33 | 267 | ... |
| 2.757 | 28 | 272 | ... |
| 2.812 | 19 | 281 | ... |
| 2.847 | 13 | 287 | ... |
| 2.863 | 10 | 290 | ... |
| 2.893 | 6 | 294 | ... |
| 2.933 | ... | 300 | 1 |
| 2.960 | ... | 277 | 23 |
| 2.999 | ... | 248 | 52 |
| 3.035 | ... | 214 | 86 |
| 3.054 | ... | 198 | 102 |
| 3.096 | ... | 168 | 132 |



[4] Bowman, H. A., and Schoonover, R. M., "Procedure for High Precision Density Determinations by Hydrostatic Weighing," *Journal of Research*, National Bureau of Standards, 71 C, 3, 1967, p. 179.

[5] These liquids are available from most chemical supply companies.

more drops of either component until the density standard settles at 35 °C ± 0.2 °C in the well-stirred solution.

## 7. Preparation of Density-Temperature Tables

7.1 Tables are prepared from the equations of this section to relate the specimen density at 25 °C to its settling temperature. These tables are prepared once for each glass reference standard-density solution system. Subsequent supplies of density solutions prepared for use with the same glass reference standard will be sufficiently similar in expansion and density characteristics so that the same table can be used.

7.2 *Determination of Temperature Coefficient of Density*—Measure the density of the solution at approximately 25 °C and 45 °C using the Bingham pycnometer, Test Method D1217, or equivalent pycnometer method. Calculate the temperature coefficient of density, $C\rho$, as follows:

$$C_\rho = (\rho_{T1} - \rho_{T2})/(T_1 - T_2) \tag{4}$$

where:

$C_\rho$ = temperature coefficient of the solution, g/cm$^3$·°C, and

$\rho_{T1}$ and $\rho_{T2}$ = density of the solution at temperature $T_1$ and $T_2$, g/cm$^3$.

7.3 *Equations for Determination of Density:*

NOTE 2—Alternative equations or method of calculation may be used in conjunction with different density tables and standard settling temperatures.

7.3.1 These equations relate the specimen density to its settling temperatures. Express the exact relationship:

$$\rho_T = \rho_s + C_\rho (T - T_s) \tag{5}$$

where:

$\rho_T$ = density of specimen at its settling temperature, $T$, and

$\rho_s$ = density of standard at its settling temperature, $T_s$, approximately 35 °C.

7.3.2 If the thermal expansions of a specimen and standard are similar, express their densities at 25 °C as follows:

$$\rho_{25} = \rho_{s25} + (C_\rho + 3\alpha_s \rho_s)(T - T_s) \tag{6}$$

where:

$\rho_{25}$ = specimen density at 25 °C,

$\rho_{s\,25}$ = standard density at 25 °C, and

$\alpha_s$ = linear expansion of standard ≈ expansion of specimen.

7.3.3 It is convenient to fix 35 °C as the settling temperature of the standard, as it will vary slightly with heating rate, operator, and liquid density. The specimen settling temperature must be corrected as follows:

$$T_c = T + (35 - T_s) \tag{7}$$

where:

$T_c$ = corrected specimen settling temperature,

$T$ = observed specimen settling temperature, and

$T_s$ = observed standard settling temperature.

Eq 6 then becomes:

$$\rho_{25} = \rho_{s25} + (C_\rho + 3\alpha_s \rho_s)(T_c - 35) \tag{8}$$

7.4 *Density Table*—This table is prepared and used when many routine densities are to be determined. Eq 8 is solved for $T_c$ between 25 °C and 45 °C in 0.1 °C increments, and specimen density at 25 °C is tabulated with corrected specimen settling temperature $T_c$. A typical density table is shown in Table 2.

7.5 *Density Equation for Unlike Expansions*—If the thermal expansions of specimen and standard differ, specimen density will be in error by approximately 0.0001 g/cm$^3$ for every $20 \times 10^{-7}$/°C mismatch in expansion. This error is greater if the specimen settles above 35 °C and less if it settles below 35 °C. Use the following equation, which is accurate to ±0.0001 g/cm$^3$:

$$\rho_{25} = \rho_{s25}[(1.0000 - 30\,\alpha_s) + 3\alpha(T_c - 25)] + C_\rho(T_c - 35) \tag{9}$$

where: $\alpha$ = linear expansion coefficient of specimen.

## 8. Procedure for Determining Density of Test Specimens

8.1 Prepare the specimen for testing by cutting from the sample a piece comparable in size with the standard. The test specimen should be smooth and free of bubbles and cracks. Identify the specimen using a diamond-point marking pencil or by cutting it to a distinctive shape. Clean the specimen in reagent grade alcohol or acetone and wipe dry with silicone-free lens tissue. Place specimen in the solution (Note 3) that contains the standard. The bath and solution temperatures should be approximately 25 °C ± 3 °C, and both specimen and standard should float.

NOTE 3—Adsorbed moisture on the specimen surface will lower the measured density. Moisture, from condensation, on the solution surface should be removed by periodically filtering the solution through coarse filter paper.

8.2 Place all the tubes, thermometers, stoppers, etc., in their proper location in the bath, and rapidly heat the bath (1 to 2 °C ⁄min), noting the temperature of the density solution at which the test specimen (or the standard) begins to settle.

8.3 Adjust the bath temperature by cooling to 2 °C to 4 °C *below* the expected settling temperature of the specimen (or standard). Allow the bath and solutions to come to equilibrium for 10 min, then heat the bath at a rate of 0.1 ± 0.02 °C ⁄min. Heating rates can be controlled by adjusting the power to the hotplate or immersion heater and the cooling water flow rate. Cooling water is used as a fine adjustment of heating rate.

8.4 As either the specimen or standard begin to settle in the solutions, note the temperature at which either is halfway between upper and lower cage disks. The bath and density solution temperatures must agree within 0.4 °C when the temperature is recorded, with the bath temperature being higher. Record $T$ and $T_s$.

8.5 Calculate the corrected specimen settling temperature, $T_c$, by Eq 7. From an appropriate density table prepared from Eq 4 and 8, read the density that corresponds to the corrected specimen settling temperature, $T_c$. This density is the density of the specimen *at 25 °C*, $\rho_{25}$.

8.6 Up to three test specimens can be run in a single tube simultaneously.