Designation: B962 – 17

Standard Test Methods for Density of Compacted or Sintered Powder Metallurgy (PM) Products Using Archimedes' Principle[1]

This standard is issued under the fixed designation B962; 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 U.S. Department of Defense.

1. Scope*

1.1 This standard describes a method for measuring the density of powder metallurgy products that usually have surface-connected porosity.

1.2 The density of impermeable PM materials, those materials that do not gain mass when immersed in water, may be determined using Test Method B311.

1.3 The current method is applicable to green compacts, sintered parts, and green and sintered test specimens.

1.4 With the exception of the values for density and the mass used to determine density, for which the use of the gram per cubic centimetre (g/cm^3) and gram (g) units is the long-standing industry practice, the values in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

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.*

1.6 *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]

B243 Terminology of Powder Metallurgy

B311 Test Method for Density of Powder Metallurgy (PM) Materials Containing Less Than Two Percent Porosity

3. Terminology

3.1 Definitions of powder metallurgy (PM) terms can be found in Terminology B243. Additional descriptive material is available in the Related Material section of Vol. 02.05 of the *Annual Book of ASTM Standards*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *green density (D_g)*—the mass per unit volume of an unsintered PM part or test specimen.

3.2.2 *impregnated density (D_i)*—the mass per unit volume of a sintered PM part or test specimen, impregnated with oil.

3.2.3 *sintered density (D_s)*—the mass per unit volume of a sintered, non oil-impregnated PM part or test specimen.

4. Summary of Test Method

4.1 The test specimen is first weighed in air. It is then oil impregnated or some other treatment is used to seal the surface-connected porosity and the specimen is reweighed. The test specimen is then weighed when immersed in water and its density calculated based on Archimedes' principle.

5. Significance and Use

5.1 The volume of a complex shaped PM part cannot be measured accurately using micrometers or calipers. Since density is mass per unit volume, a precise method for measuring the volume is needed. Archimedes' principle may be used to calculate the volume of water displaced by an immersed object. For this to be applicable to PM materials that contain surface connected porosity, the surface pores are sealed by oil impregnation or some other means.

5.2 The green density of compacted parts or test pieces is normally determined to assist during press set-up, or for quality control purposes. It is also used for determining the compressibility of base powders, mixed powders, and premixes.

5.3 The sintered density of sintered PM parts and sintered PM test specimens is used as a quality control measure.

[1] These test methods are under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and are the direct responsibility of Subcommittee B09.04 on Bearings.

Current edition approved April 1, 2017. Published May 2017. Originally approved in 2008. Last previous edition approved in 2015 as B962 – 15. DOI: 10.1520/B0962-17.

[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.

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

5.4 The impregnated density of sintered bearings is normally measured for quality control purposes as bearings are generally supplied and used oil-impregnated.

6. Interferences

6.1 A gain in mass when a test specimen is immersed in water is an indication that the specimen contains surface-connected porosity. Unsealed surface porosity will absorb water and cause the calculated density values to be higher than the true value.

6.2 Test specimens that contain surface-connected porosity shall be oil impregnated or have the surface-connected porosity sealed by some other means prior to their immersion in water.

7. Apparatus

7.1 *Analytical Balance*—Precision single-pan balance that will permit readings within 0.01% of the test specimen mass. See Table 1.

7.2 *Water Container*—A glass beaker or other suitable transparent container should be used to contain the water.

NOTE 1—A transparent container makes it easier to see air bubbles adhering to the test specimen and specimen support when immersed in water.

NOTE 2—For the most precise density determination, the water container should be of a size that the level of the water does not rise more than 0.10 in. (2.5 mm) when the test specimen is lowered into the water.

7.3 *Water*—Distilled or deionized water to which 0.05 to 0.1 volume percent of a wetting agent has been added to reduce the effects of surface tension.

NOTE 3—Degassing the water by evacuation, boiling, or ultrasonic agitation helps to prevent air bubbles from collecting on the test specimen and support when immersed in water.

7.4 *Test Specimen Support for Weighing in Water*—Two typical arrangements are shown in Fig. 1. The suspension wire may be twisted around the test specimen or the test specimen may be supported in a wire basket that is attached to the suspension wire. For either arrangement, a single corrosion-resistant wire—for example, austenitic stainless steel, copper, or nichrome—shall be used for the basket and suspension wire. The maximum recommended diameter of suspension wire to be used for various mass ranges is summarized in Table 2.

NOTE 4—For the most precise density determinations, it is important that the mass and volume of all supporting wires immersed in water be minimized.

7.5 *Oil for Oil-Impregnation*—Oil with a viscosity of 20 to 65 cSt or 100 to 300 SSU (20×10^{-6} to 65×10^{-6} m^2/s) at 100 °F (38 °C) has been found to be suitable.

7.5.1 In the case of oil-impregnated bearings, make an effort to match the oil that was originally used to impregnate them.

TABLE 1 Balance Readability

Mass, g	Balance Readable to, g
less than 10	0.0001
10 to less than 100	0.001
100 to less than 1000	0.01
1000 to less than 10 000	0.1

7.6 *Vacuum Impregnation Apparatus*—Equipment to impregnate the part or test specimen with oil.

7.7 *Thermometer*—A thermometer with an accuracy of 1.0 °F (0.5 °C) to measure the temperature of the water.

8. Preparation of Test Specimens

8.1 The mass of the test specimen shall be a minimum of 1.0 g. For small parts, several parts may be combined to reach the minimum mass.

8.2 Thoroughly clean all surfaces of the test specimen to remove any adhering foreign materials such as dirt or oxide scale. Take care with cut specimens to avoid rough surfaces to which an air bubble may adhere. A 100-grit sanding or abrasive grinding is recommended to remove all rough surfaces.

9. Procedure

9.1 The part or test specimen, the analytical balance and surrounding air shall be at a uniform temperature when weighing is performed.

9.2 For the most precise density determinations, duplicate weighings should be made for all mass measurements. Adjust the analytical balance to zero prior to each weighing. Average the mass determinations before calculating the density.

9.3 For improved repeatability and reproducibility, verify the analytical balance periodically with a standard mass that is approximately equal to the part or test specimen mass.

9.4 This standard contains three separate test methods; determination of green density, determination of sintered density, and determination of impregnated density. Each is detailed in the following sections.

Determination of Green Density

9.5 This procedure is used to determine the green density of PM parts and test specimens.

9.5.1 Determine the mass of the green part or test specimen. This is mass A. This and all subsequent weighings shall be to the precision stated in Table 1.

9.5.2 Oil impregnate the green part or test specimen as follows:

Preferred Procedure

9.5.3 Immerse the part or test specimen in oil at room temperature.

9.5.4 Reduce the pressure over the sample to 1 psi (7 kPa) or less for 30 minutes, then increase the pressure back to atmospheric pressure and keep the sample immersed for at least 30 minutes.

9.5.5 Remove excess oil by wiping gently with an absorbent, lint-free material. Take care not to extract oil absorbed within the part or test specimen.

9.5.6 Do not place or store parts on porous surfaces such as paper, cloth, or cardboard as these will absorb oil.

9.5.7 Proceed to 9.5.13.

Alternative Procedure

9.5.8 Immerse the part or test specimen in oil at a temperature of 180 ± 10 °F (82 ± 5 °C) for at least 4 hours.

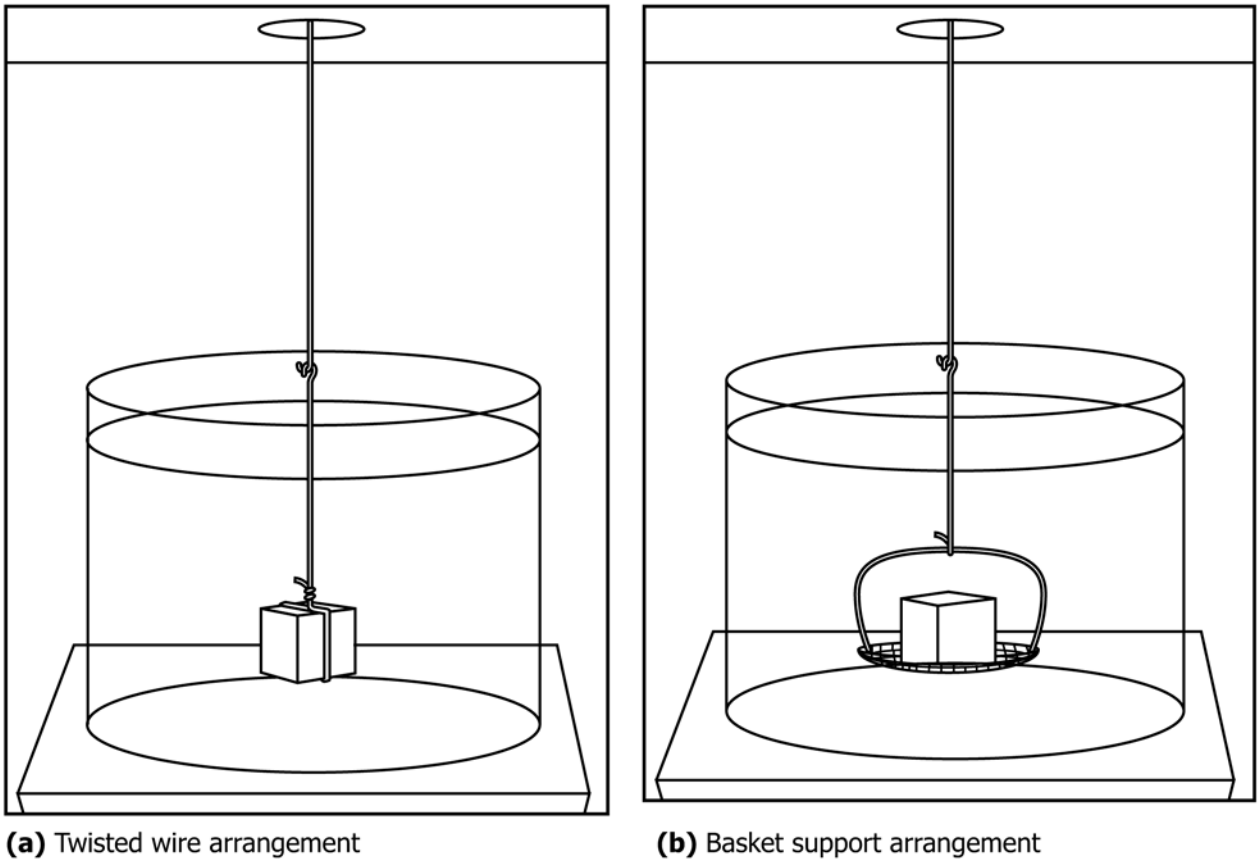

FIG. 1 Methods for Holding the Test Specimen When Weighing in Water

TABLE 2 Maximum Recommended Wire Diameters

Mass, g	Wire Diameter, in. (mm)
less than 50	0.005 (0.12)
50 to less than 200	0.010 (0.25)
200 to less than 600	0.015 (0.40)
600 and greater	0.020 (0.50)

9.5.9 Cool by immersing in a bath of the same oil held at room temperature and keep in this oil for at least 30 minutes.

9.5.10 Remove excess oil by wiping gently with an absorbent, lint-free material. Take care not to extract oil absorbed within the part or test specimen.

9.5.11 Do not place or store parts on porous surfaces such as paper, cloth, or cardboard as these will absorb oil.

9.5.12 Proceed to 9.5.13.

NOTE 5—It may not be necessary to oil impregnate the green part with oil. There may be enough admixed lubricant present in the surface-connected pores to prevent the absorption of water. If the test specimen gains mass when immersed in water it is an indication that the specimen contains surface-connected porosity and that it needs to be sealed by oil impregnation or some other means.

9.5.13 Determine the mass of the oil-impregnated green part or test specimen to the precision stated in Table 1. This is mass B.

9.5.14 Support the container of water over the pan of the balance using a suitable bridge as shown in Fig. 2a. Take care to ensure that the bridge does not restrict the free movement of the balance pan. The container of water may also be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. If this arrangement is used, it is important to shield the weighing system, including the wire, from the effect of air drafts.

9.5.15 Suspend the test specimen support along with the part or test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least ¼ in. (6 mm) to minimize the effect of surface tension forces on the weighing.

9.5.16 The test specimen support and test specimen shall hang freely from the balance beam hook, be free of air bubbles when immersed in the water, and be at the same temperature as the water and the balance.

9.5.17 The surface of the water shall be free of dust particles.

9.5.18 Weigh the part/test specimen and specimen support immersed in water. This is mass C.

9.5.19 Remove the part/test specimen from the support.

9.5.20 Weigh the test specimen support immersed in water at the same depth as before. This is mass E. The suspension support shall be free of air bubbles and the suspension wire shall not be immersed below its normal hanging depth, as a change in depth will change the measured mass.

NOTE 6—Some balances are capable of being tared. This automatically removes the necessity of reweighing the specimen support every time. In this case, tare the specimen support alone, immersed in water to the same depth as with the specimen, before weighing the specimen support and

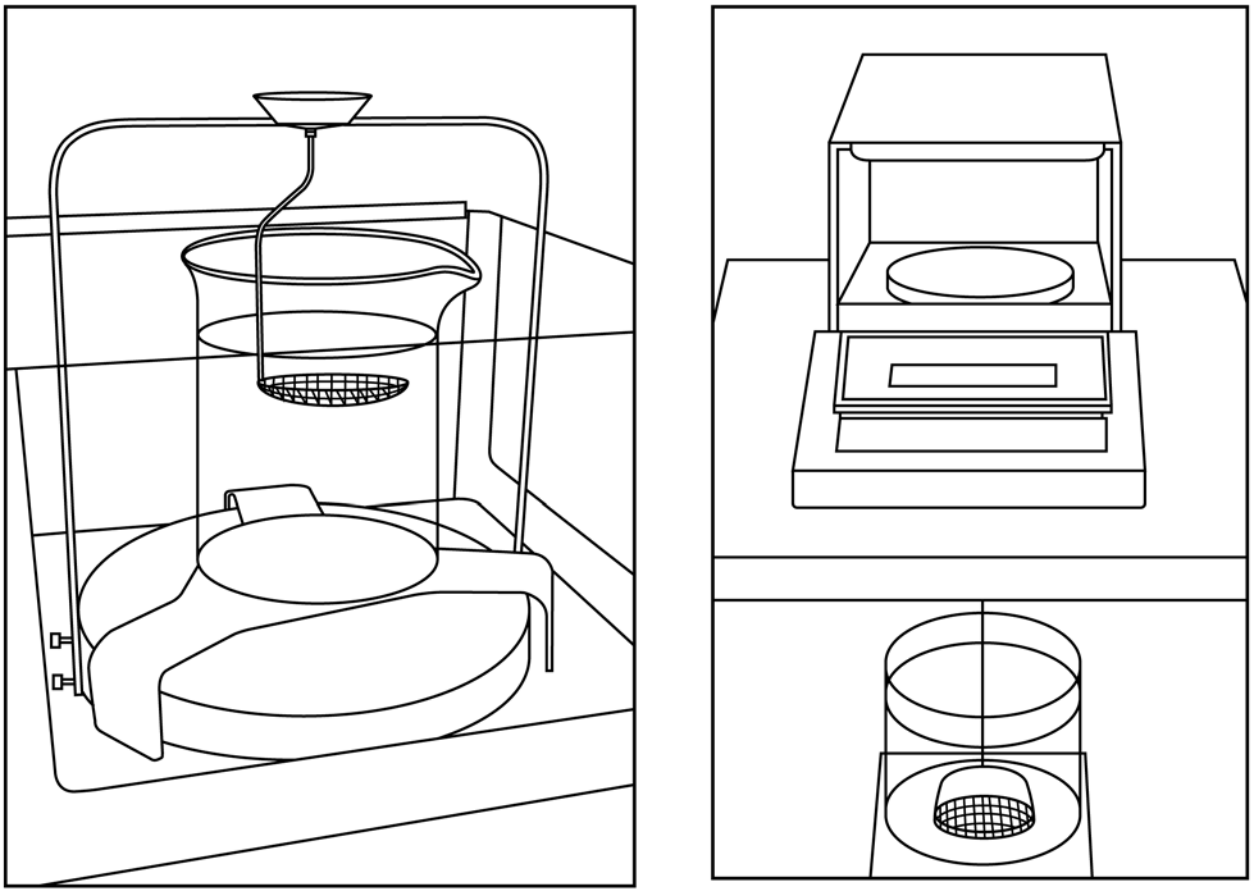

FIG. 2 Methods for Weighing in Water

part/test specimen immersed in water. The mass of the specimen support and specimen immersed in water is mass F, which replaces mass C minus mass E.

9.5.21 Measure the temperature of the water to the nearest 2 °F (1 °C) and record its density ρ_w, at that temperature, from Table 3.

9.5.22 Calculate the green density of a part or test piece from the following formula:

$$\text{Green Density, } D_g = \frac{A\rho_w}{B-(C-E)} \quad (1)$$

or

$$\text{Green Density, } D_g = \frac{A\rho_w}{B-F} \quad (2)$$

where:

A = the mass of the green part or test piece in air, g,
B = the mass of the oil-impregnated green part or test piece, g,
C = the mass of the oil-impregnated part/test specimen and specimen support immersed in water, g,
E = the mass of the oil-impregnated part/test specimen support immersed in water, g,
F = the mass of the oil-impregnated part/test specimen in water with the mass of the specimen support tared, g, and
ρ_w = the density of the water, g/cm^3.

If the green part did not need to be oil impregnated then use the following formula:

TABLE 3 Effect of Temperature on the Density of Air-Free Water[A]

Temperature		Density
°F	(°C)	g/cm^3
59.0	(15)	0.9991
60.8	(16)	0.9989
62.6	(17)	0.9988
64.4	(18)	0.9986
66.2	(19)	0.9984
68.0	(20)	0.9982
69.8	(21)	0.9980
71.6	(22)	0.9978
73.4	(23)	0.9975
75.2	(24)	0.9973
77.0	(25)	0.9970
78.8	(26)	0.9968
80.6	(27)	0.9965
82.4	(28)	0.9962
84.2	(29)	0.9959
86.0	(30)	0.9956

[A] *Metrological Handbook 145*, "Quality Assurance for Measurements," National Institute of Standards and Technology, 1990, pp. 9-10.

$$\text{Green Density, } D_g = \frac{A\rho_w}{[A - (B - C)]} \quad (3)$$

Determination of Sintered Density

9.6 This procedure is used to determine the sintered density of PM parts and test pieces.

9.6.1 Determine the mass of the sintered part or test specimen to the precision stated in Table 1. This is mass A. This and all subsequent weighings shall be to the precision stated in Table 1.

NOTE 7—Oil impregnated specimens or specimens that contain any oil are to be free of lubricant for determining mass A. Remove the oil in a Soxhlet apparatus using a suitable solvent, such as petroleum ether. After extraction, residual solvent shall be removed by heating specimens at 250 °F (120 °C) for 1 hour. Alternate extraction and drying shall be continued until the dry mass, A, is constant to 0.05%.

NOTE 8—A practical and fast method of oil removal is to heat the specimen in a protective atmosphere in the temperature range of 800 to 1600 °F (425 to 870 °C). This method, which results in values in close agreement with those obtained using the Soxhlet apparatus, may be used if agreed upon by both parties. The selection of the appropriate temperature is very important and care should be taken not to exceed the melting point of any material that is tested. For example, 1500 to 1600 ºF (815 to 870 ºC) for bronze, depending on the sintering temperature that was used. This method also is applicable to sintered aluminum materials if the temperature does not exceed 1000 °F (540 °C).

9.6.2 In order to seal the surface-connected porosity the parts/test pieces are oil impregnated or the pores are filled with a suitable material. If using oil impregnation, oil impregnate the part or test specimen using one of the procedures described in sections 9.5.2 – 9.5.12.

9.6.3 Determine the mass of the oil-impregnated part or test specimen to the precision stated in Table 1. This is mass B.

9.6.4 Support the container of water over the pan of the balance using a suitable bridge as shown in Fig. 2a. Take care to ensure that the bridge does not restrict the free movement of the balance pan. The container of water may also be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. If this arrangement is used, it is important to shield the weighing system, including the wire, from the effect of air drafts.

9.6.5 Suspend the test specimen support along with the part or test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least ¼ in. (6 mm) to minimize the effect of surface tension forces on the weighing.

9.6.6 The test specimen support and test specimen shall hang freely from the balance beam hook, be free of air bubbles when immersed in the water, and be at the same temperature as the water and the balance.

9.6.7 The surface of the water shall be free of dust particles.

9.6.8 Weigh the part/test specimen and specimen support immersed in water. This is mass C.

9.6.9 Remove the part/test specimen from the support.

9.6.10 Weigh the test specimen support immersed in water at the same depth as before. This is mass E. Take care to ensure that the suspension support is free of air bubbles and that the suspension wire is not immersed below its normal hanging depth, as a change in depth will change the measured mass.

NOTE 9—Some balances are capable of being tared. This automatically removes the necessity of reweighing the specimen support every time. In this case, tare the specimen support alone, immersed in water to the same depth as with the specimen, before weighing the specimen support and part/test specimen immersed in water. The mass of the specimen support and specimen immersed in water is mass F, which replaces mass C minus mass E.

9.6.11 Measure the temperature of the water to the nearest 2 °F (1 °C) and record its density ρ_w, at that temperature, from Table 3.

9.6.12 Calculate the sintered density from the following formula:

$$\text{Sintered Density, } D_s = \frac{A\rho_w}{B - (C - E)} \quad (4)$$

or

$$\text{Sintered Density, } D_s = \frac{A\rho_w}{B - F} \quad (5)$$

where:

A = the mass of the sintered part or test piece in air, g,
B = the mass of the oil-impregnated part or test piece, g,
C = the mass of the oil-impregnated part/test specimen and specimen support immersed in water, g,
E = the mass of the oil-impregnated part/test specimen support immersed in water, g,
F = the mass of the oil-impregnated part/test specimen in water with the mass of the specimen support tared, g, and
ρ_w = the density of the water, g/cm^3.

Determination of Impregnated Density

9.7 This procedure is used to determine the density of oil-impregnated PM bearings or parts/test pieces.

9.7.1 Oil impregnate the specimen using one of the procedures described in sections 9.5.2 – 9.5.12 to ensure that the bearing, part, or test piece is fully oil impregnated.

9.7.2 Determine the mass of the oil-impregnated green part or test specimen to the precision stated in Table 1. This is mass B.

9.7.3 Support the container of water over the pan of the balance using a suitable bridge as shown in Fig. 2a. Take care to ensure that the bridge does not restrict the free movement of the balance pan. The container of water may also be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. If this arrangement is used, it is important to shield the weighing system, including the wire, from the effect of air drafts.

9.7.4 Suspend the test specimen support along with the part or test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least ¼ in. (6 mm) to minimize the effect of surface tension forces on the weighing.

9.7.5 The test specimen support and test specimen shall hang freely from the balance beam hook, be free of air bubbles when immersed in the water, and be at the same temperature as the water and the balance.

9.7.6 The surface of the water shall be free of dust particles.

9.7.7 Weigh the part/test specimen and specimen support immersed in water. This is mass C.

9.7.8 Remove the part/test specimen from the support.

9.7.9 Weigh the test specimen support immersed in water at the same depth as before. This is mass E. Take care to ensure that the suspension support is free of air bubbles and that the suspension wire is not immersed below its normal hanging depth as a change in depth will change the measured mass.

NOTE 10—Some balances are capable of being tared. This automatically removes the necessity of reweighing the specimen support every time. In this case, tare the specimen support alone, immersed in water to the same depth as with the specimen, before weighing the specimen support and part/test specimen immersed in water. The mass of the specimen support and specimen immersed in water is mass F, which replaces mass C minus mass E.

9.7.10 Measure the temperature of the water to the nearest 2 °F (1 °C) and record its density ρ_w, at that temperature, from Table 3.

9.7.11 Calculate the impregnated density of an oil-impregnated part or test piece from the following formula:

$$\text{Impregnated Density, } D_i = \frac{B\rho_w}{B-(C-E)} \quad (6)$$

or

$$\text{Impregnated Density, } D_i = \frac{B\rho_w}{B-F} \quad (7)$$

where:

B = the mass of the oil-impregnated part or test piece, g,
C = the mass of the oil-impregnated part/test specimen and specimen support immersed in water, g,
E = the mass of the oil-impregnated part/test specimen support immersed in water, g,
F = the mass of the oil-impregnated part/test specimen in water with the mass of the specimen support tared, g, and
ρ_w = the density of the water, g/cm^3.

10. Report

10.1 Report the green density, sintered density, or the impregnated density rounded to the nearest 0.01 g/cm^3.

10.2 For the green density measurement report if the green part/test specimen was impregnated and which method was used.

11. Precision and Bias

11.1 The results of an interlaboratory study to determine the precision of this test method are available in an ASTM Research Report.[3] The study involved six laboratories and covered identical samples of both iron and copper-based sintered parts.

11.2 For ferrous and copper-based sintered parts, the repeatability interval, r, is 0.05 g/cm^3 for sintered or impregnated density. Duplicate results from the same laboratory should not be considered suspect at the 95% confidence level unless they differ by more than r.

11.3 There are no data available for the repeatability of this test method for green density. An interlaboratory study to determine repeatability is planned within the next five years.

11.4 For ferrous and copper-based sintered parts, the reproducibility interval, R, is 0.06 g/cm^3 for sintered or impregnated density. The results from two laboratories should not be considered suspect at the 95% confidence level unless they differ by more than R.

11.5 There are no data available for the reproducibility of this test method for green density. An interlaboratory study to determine reproducibility is planned within the next five years.

11.6 There is no estimate of bias because there is no accepted porous reference material.

11.7 *Measurement Uncertainty*—The precision of this test method shall be considered by those performing the test when reporting the results.

12. Keywords

12.1 density; green density; impregnated density; PM products; powder metallurgy products; sintered density

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1008. Contact ASTM Customer Service at service@astm.org.

SUMMARY OF CHANGES

Committee B09.04 has identified the location of selected changes to this standard since the last issue (B962 – 15) that may impact the use of this standard.

(1) Changed the heading of the right-hand column in Table 1 from "Balance Sensitivity, g" to "Balance Readable to, g."