Designation: B963 – 17

Standard Test Methods for Oil Content, Oil-Impregnation Efficiency, and Surface-Connected Porosity of Sintered Powder Metallurgy (PM) Products Using Archimedes' Principle[1]

This standard is issued under the fixed designation B963; 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 three related test methods that cover the measurement of physical properties of oil-impregnated powder metallurgy products.

1.1.1 Determination of the volume percent of oil contained in the material.

1.1.2 Determination of the efficiency of the oil-impregnation process.

1.1.3 Determination of the percent surface-connected porosity by oil impregnation.

1.2 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.

1.3 *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.*

1.4 *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

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

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

E456 Terminology Relating to Quality and Statistics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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*.

4. Summary of Test Method

4.1 The part or test specimen is first weighed in air. It is then oil impregnated to fill the surface-connected porosity and the specimen is reweighed. The test specimen is then weighed when immersed in water and its volume calculated based on Archimedes' principle. The oil is then removed and the specimen is reweighed.

4.2 The *oil content* of an oil-impregnated part or test specimen is then calculated as a percentage of the volume of the specimen. This may be done for the as-received and the fully oil-impregnated specimen.

[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.

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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.

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

4.3 The *oil-impregnation efficiency* is calculated by dividing the as-received oil content by the fully impregnated oil content and expressing the result as a percentage.

4.4 The volume percentage of *surface-connected porosity* (as measured by oil impregnation) is then calculated based on the amount of oil in the fully oil-impregnated specimen.

5. Significance and Use

5.1 Oil content values are generally contained in specifications for oil-impregnated PM bearings.

5.2 The oil-impregnation efficiency provides an indication of how well the as-received parts had been impregnated.

5.3 The desired self-lubricating performance of PM bearings requires a minimum amount of surface-connected porosity and satisfactory oil impregnation of the surface-connected porosity. A minimum oil content is specified.

5.4 The results from these test methods may be used for quality control or compliance purposes.

6. Apparatus

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

6.2 *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 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.

6.3 *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.

6.4 *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 shown in Table 2.

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

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

6.5 *Oil for Oil-Impregnation*—The same type of oil that was used to impregnate the parts originally.

6.5.1 If parts are not already impregnated, 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.

6.6 *Vacuum Impregnation Apparatus*—Equipment for impregnation of the part or test specimen with oil.

6.7 *Thermometer*—A thermometer with an accuracy of 1 °F (0.5 °C) to measure the temperature of the water.

6.8 *Soxhlet Apparatus*—Glass laboratory unit consisting of a condenser, extractor, filter, flask with a suitable solvent for the oil such as petroleum ether, and a heating mantle.

7. Preparation of Test Specimens

7.1 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.

7.2 Thoroughly wipe clean all surfaces of the test specimen to remove any adhering foreign materials such as dirt or oxide scale.

7.3 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.

8. Procedure

8.1 It is important that the part or test specimen, the analytical balance and surrounding air be at a uniform temperature when weighing is performed.

8.2 For the most precise volume determinations, duplicate weighings should be made for all mass measurements. The analytical balance should be adjusted to zero prior to each weighing. Duplicate mass determinations should be averaged before performing any calculations.

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

8.4 *Determination of Oil Content, Oil-Impregnation Efficiency, and Surface-Connected Porosity:*

8.4.1 Determine the mass of the as-received part or test specimen. This is mass J. This and all subsequent weighings shall be to the precision stated in Table 1.

8.4.2 Oil impregnate the as-received part or test specimen using one of the following procedures:

Vacuum Oil Impregnation—Preferred Procedure

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

8.4.4 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.

8.4.5 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.

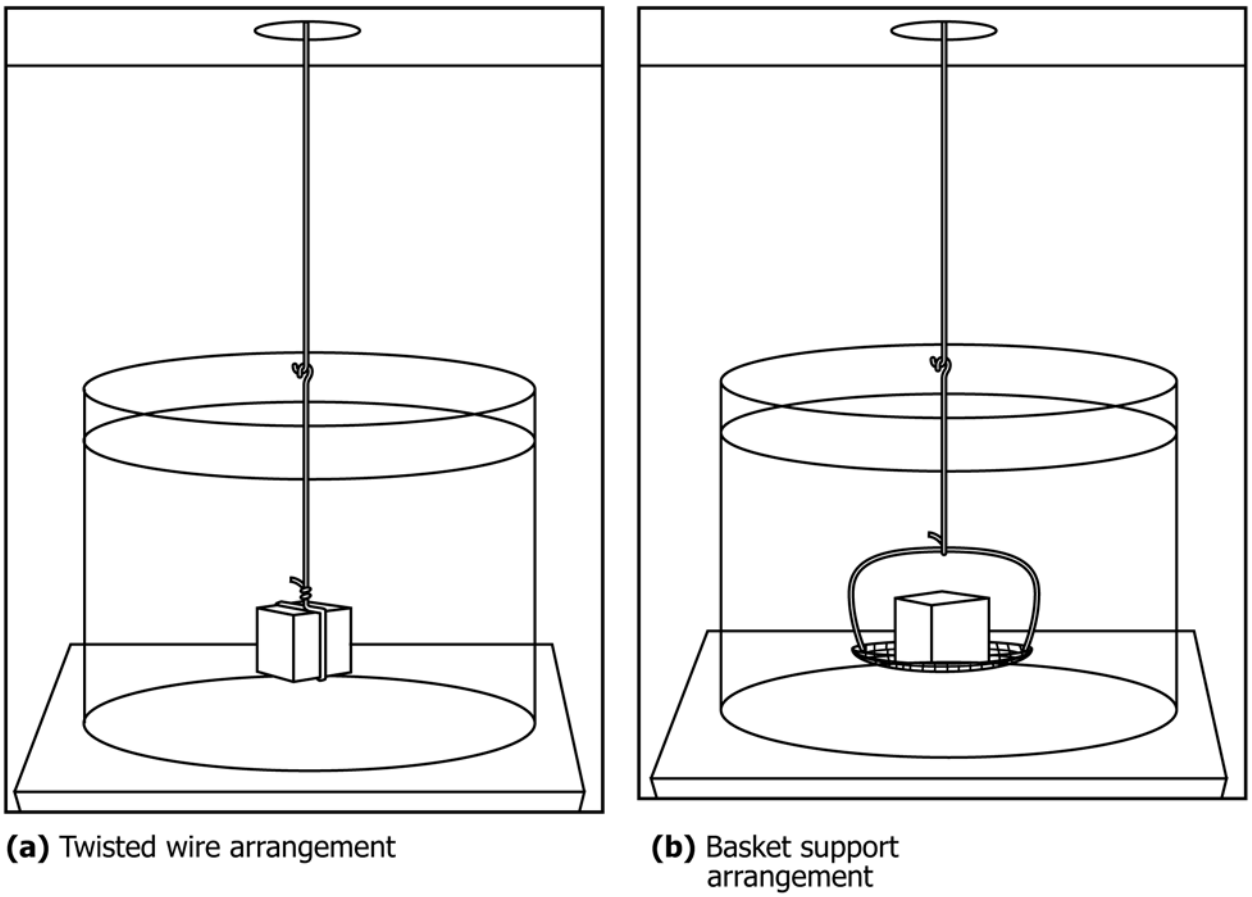

FIG. 1 Methods for Holding the Test Specimen When Weighing in Water

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)

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

8.4.7 Proceed to 8.4.13.

Immersion Oil Impregnation—Alternative Procedure

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

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

8.4.10 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.

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

8.4.12 Proceed to 8.4.13.

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

8.4.14 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, shield the weighing system, including the wire, from the effect of air drafts.

8.4.15 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.

8.4.16 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.

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

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

8.4.19 Remove the part/test specimen from the support.

8.4.20 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 5—Some balances are capable of being tared. This automatically

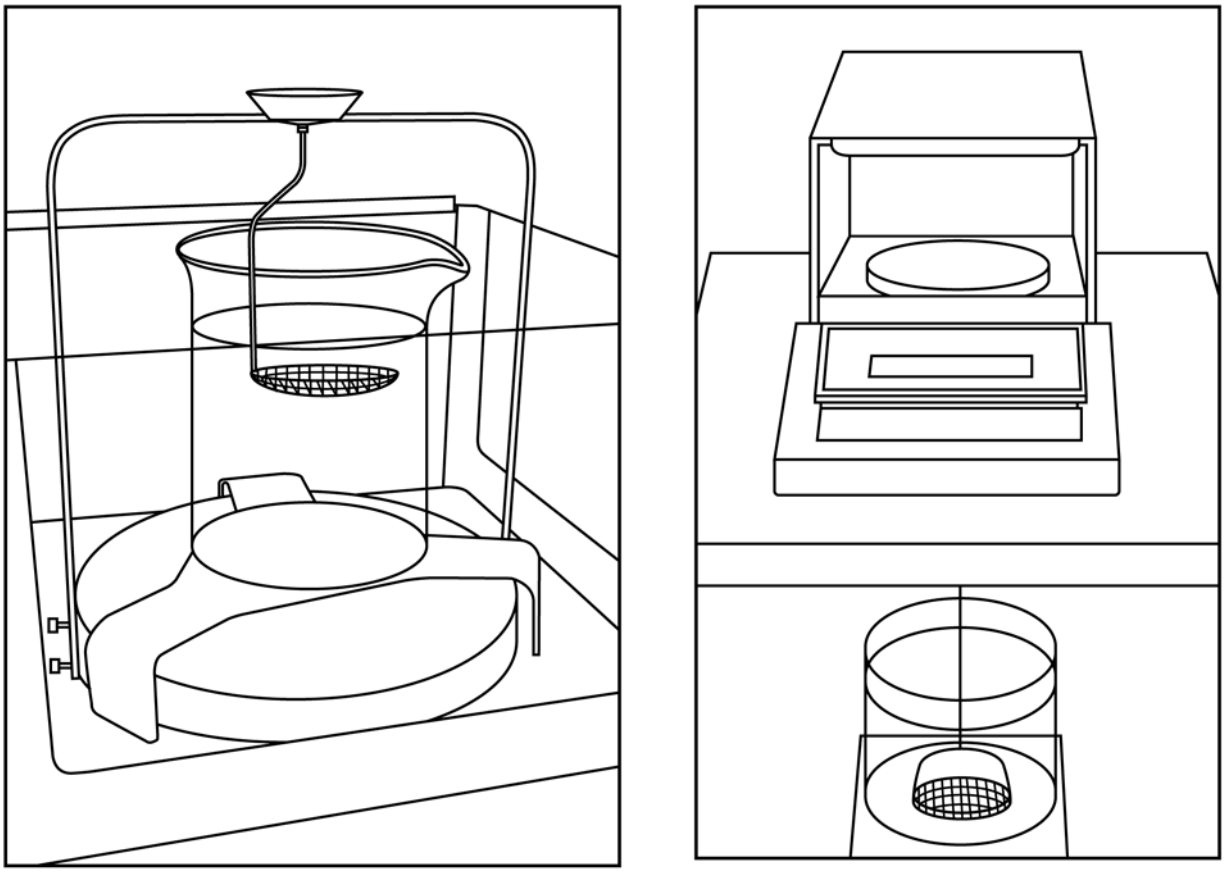

FIG. 2 Methods for Weighing in Water

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.

8.4.21 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.

8.4.22 Remove the oil from the part or test specimen in a Soxhlet apparatus using a solvent such as toluene or petroleum ether in order to determine the dry mass of the part or test specimen.

8.4.23 After extraction of the oil, remove residual solvent by heating the part or test specimen to 36 °F (20 °C) above the boiling point of the selected solvent.

8.4.24 Continue to alternate extraction and drying until the mass of the part or test specimen is constant to within 0.05%. Weigh the part to the precision stated in Table 1 to determine the dry mass. This is mass A.

8.4.25 A practical and fast method of oil removal for most materials consists of heating the part or test specimen in a protective atmosphere to a temperature in the range of 800 to 1600 °F (425 to 870 °C). The method is applicable only if metallurgical properties are not a point of concern and all concerned parties agree upon its use.

NOTE 6—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 °F (815 to 870 °C) for bronze, depending on the sintering temperature that was used; and 1000 °F

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.

(540 °C) should not be exceeded for aluminum alloys.

8.4.26 If the oil density is not already known, determine the density of the oil that was used to impregnate the part or test specimen in accordance with Test Method D1217 or Test Method D1298. This density is ρ_o.

NOTE 7—The typical density of petroleum-type lubricants is 0.880 g/cm^3 and for synthetic lubricants it ranges from 0.910 to 1.000 g/cm^3.

9. Calculation

As-Received Oil Content

9.1 Calculate the as-received oil content (volume %) from the following formula:

$$\text{As} - \text{received oil content } P_1 \text{ (volume \%)} = \left(\frac{J - A}{(B - (C - E))\,\rho_o} \times 100 \right) \rho_w \quad (1)$$

or

$$\left(\frac{J - A}{(B - F)\,\rho_o} \times 100 \right) \rho_w \quad (2)$$

where:

P_1 = as-received oil content by volume, %,
J = the mass of as-received part/test specimen, g,
B = the mass of oil-impregnated part/test specimen, g,
C = 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,
A = the mass of the oil-free part/test specimen, g,
ρ_o = the density of the oil used to impregnate the part/test specimen, g/cm^3, and
ρ_w = the density of the water, g/cm^3.

Fully Impregnated Oil Content

9.2 Calculate the fully impregnated oil content (volume %) from the following formula:

$$\text{Fully impregnated oil content } P_1 \text{ (volume \%)} = \left(\frac{B - A}{(B - (C - E))\rho_o} \times 100 \right) \rho_w \quad (3)$$

or

$$\left(\frac{B - A}{(B - F)\,\rho_o} \times 100 \right) \rho_w \quad (4)$$

Oil-Impregnation Efficiency

9.3 Calculate the oil impregnation efficiency (%) from the following formula:

$$\text{Oil impregnation efficiency, (\%)} = (P_1/P) \times 100 \quad (5)$$

Surface-Connected Porosity

9.4 Calculate the surface-connected porosity (based on the extent of oil impregnation) as follows:

$$\text{Surface} - \text{Connected Porosity, } P \text{ (volume \%)} = \left(\frac{B - A}{(B - (C - E))\,\rho_o} \times 100 \right) \rho_w \quad (6)$$

or

$$\left(\frac{B - A}{(B - F)\,\rho_o} \times 100 \right) \rho_w \quad (7)$$

10. Report

10.1 Report the method used for oil impregnation and the following to the nearest 0.1 %:

10.1.1 The as-received oil content.
10.1.2 The fully impregnated oil content.
10.1.3 The oil-impregnation efficiency.
10.1.4 The surface-connected porosity.

11. Precision and Bias

11.1 An interlaboratory study of the oil content and impregnation efficiency was run by the MPIF Standards Committee in 2010. Each of thirteen laboratories tested two materials at two different densities. The design of the study followed Practice

TABLE 4 Repeatability and Reproducibility Data for Oil Content and Oil Impregnation Efficiency (courtesy of MPIF)

Bronze (CT-1000)		Sintered Density (g/cm^3)		Iron Graphite (FG-0308)		Sintered Density (g/cm^3)	
		6.23	6.49			5.72	6.10
As-Received Oil Content (%)	P_1 average	27.9	24.7	As-Received Oil Content (%)	P_1 average	23.7	18.9
	r	2.0	1.1		r	0.8	1.2
	R	2.1	1.3		R	1.8	1.7
Fully Impregnated Oil Content (%)	P average	28.9	25.4	Fully Impregnated Oil Content (%)	P average	24.7	19.3
	r	2.2	0.9		r	0.4	0.5
	R	2.5	1.3		R	1.2	1.2
Oil Impregnation Efficiency (%)	P_1/P average	96.4	97.2	Oil Impregnation Efficiency (%)	P_1/P average	95.9	97.7
	r	2.7	2.6		r	3.4	1.0
	R	4.5	3.7		R	6.5	4.1

E691 and a within-between analysis of the data are given in an MPIF Research Report[3]. The data are reported here with the permission of MPIF.

11.2 The precision information presented herein has been calculated for the comparison of three results from each of the thirteen laboratories for each of two materials and two densities, each of which is an individual test determination.

11.3 *Precision:*

11.3.1 *95% Repeatability Limit (within a laboratory)*—The within laboratory repeatability limit, *r*, as defined by Terminology E456, is listed for each of the two materials and for each density in Table 4. At the 95% confidence level, duplicate oil content or impregnation efficiency test results from the same laboratory should not be considered to be different unless they differ by more than *r*.

[3] The precision for this test method was developed by the standards committee of the Metal Powder Industries Federation (MPIF) and is used herein with their permission.

11.3.2 *95% Reproducibility Limit, (between laboratories)*—The between-laboratories reproducibility limit, *R*, as defined by Terminology E456 is listed for each of the two materials and for each density in Table 4. At the 95% confidence limit, duplicate oil content or impregnation efficiency test results from different laboratories should not be considered different unless they differ by more than *R*.

11.4 *Bias*—No information can be presented on the bias of the procedures in Test Methods B963 for measuring oil content and oil impregnation efficiency because no material having an accepted reference value is available.

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

12. Keywords

12.1 impregnation efficiency; oil content; oil-impregnated bearings; oil-impregnation efficiency; oil-impregnated PM parts; surface-connected porosity

SUMMARY OF CHANGES

Committee B09.04 has identified the location of selected changes to this standard since the last issue (B963-14) 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.”