



Designation: B963 – 24

Standard Test Methods for Oil Content, Oil-Impregnation Efficiency, and Surface-Connected Porosity of Sintered Powder Metallurgy (PM) Products Using Archimedes' Principle¹

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 *Units*—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 SI units are to be regarded as standard. The values given in parentheses after SI units 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, health, and environmental 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.*

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. 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 under “General Information on PM” on the ASTM B09 web page.

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.

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**. The analytical balance shall be supported in a manner to eliminate mechanical vibrations and be shielded from air drafts.

6.2 *Water*—Distilled or deionized and preferably degassed water to which 0.05 to 0.1 volume percent of a wetting agent has been added to reduce the effects of surface tension. The density of distilled water changes as a function of water temperature and therefore should be accounted for when calculating the density of the specimen. See **Table 2**.

NOTE 1—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.

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

NOTE 2—A transparent container makes it easier to see air bubbles adhering to the test specimen and specimen support when immersed in water.

NOTE 3—For the most precise determination, the water container should be of a size that the level of the water does not rise more than 2.5 mm (0.10 in.) when the test specimen is lowered into the 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.

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

TABLE 2 Effect of Temperature on the Density of Air-Free Water^A

Temperature °C	ρ_w g/cm ³	Temperature °F	ρ_w g/cm ^{3*}
15.0	0.9991	60	0.9990
15.5	0.9990	61	0.9989
16.0	0.9989	62	0.9988
16.5	0.9988	63	0.9987
17.0	0.9988	64	0.9986
17.5	0.9987	65	0.9985
18.0	0.9986	66	0.9984
18.5	0.9985	67	0.9983
19.0	0.9984	68	0.9982
19.5	0.9983	69	0.9981
20.0	0.9982	70	0.9980
20.5	0.9981	71	0.9978
21.0	0.9980	72	0.9977
21.5	0.9979	73	0.9975
22.0	0.9978	74	0.9974
22.5	0.9976	75	0.9973
23.0	0.9975	76	0.9972
23.5	0.9974	77	0.9970
24.0	0.9973	78	0.9969
24.5	0.9972	79	0.9967
25.0	0.9970	80	0.9966
25.5	0.9969	81	0.9964
26.0	0.9968	82	0.9963
26.5	0.9966	83	0.9961
27.0	0.9965	84	0.9959
27.5	0.9964	85	0.9958
28.0	0.9962	86	0.9956
28.5	0.9961		
29.0	0.9959		
29.5	0.9958		
30.0	0.9956		

*Interpolated from
°C Data

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

For the maximum recommended diameter of suspension wire to be used for various mass ranges, see **Table 3**.

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

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 20×10^{-6} to 65×10^{-6} m²/s (20 to 65 cSt (centistokes) or 100 to 300 Saybolt Universal Seconds (SUS)) at 38 °C (100 °F) has been found to be suitable.

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

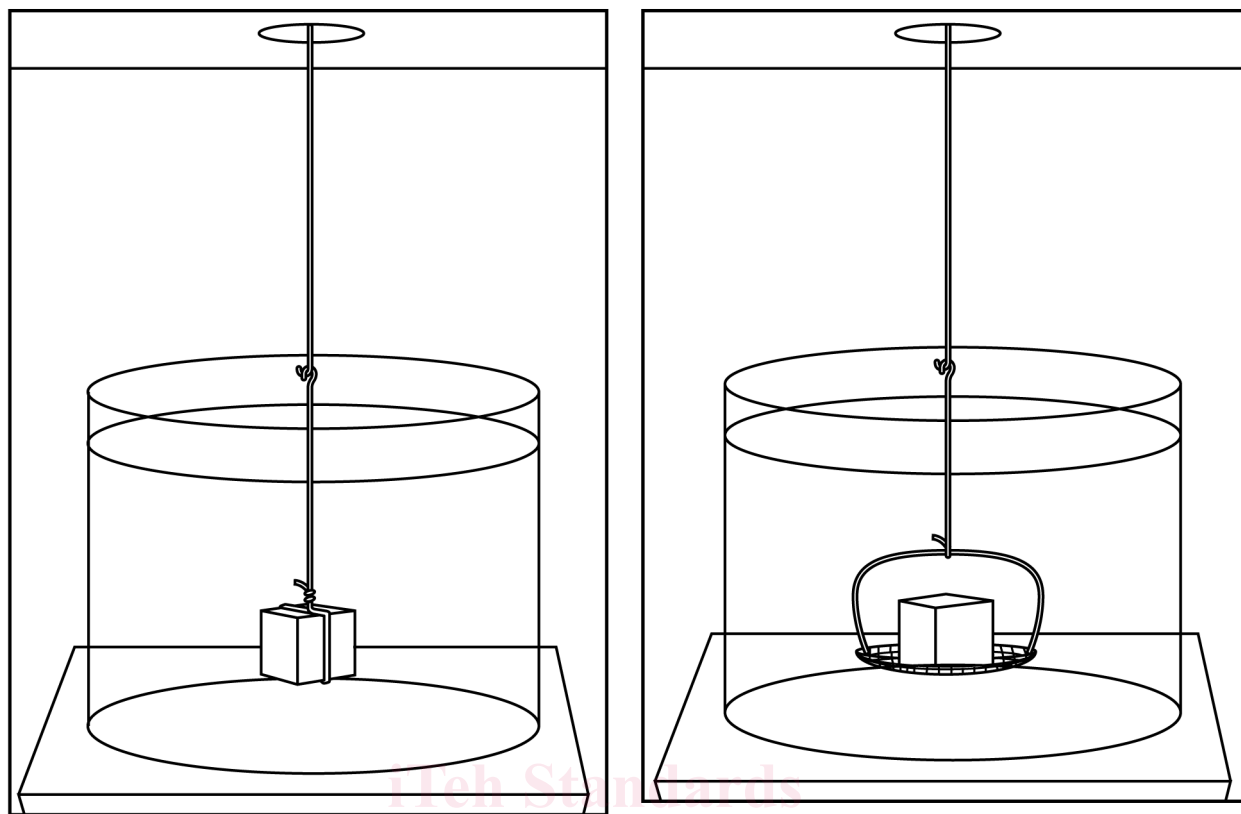
6.7 *Thermometer*—A thermometer to measure the temperature of the water to the nearest 0.5 °C (1 °F).

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 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.



(a) Twisted wire arrangement

(b) Basket support arrangement

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

TABLE 3 Maximum Recommended Wire Diameters

Mass, g	Wire Diameter, mm (in.)
less than 50	0.12 (0.005)
50 to less than 200	0.25 (0.010)
200 to less than 600	0.38 (0.015)
600 and greater	0.50 (0.020)

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 as follows:

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

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

NOTE 5—If the oil that had been used originally to impregnate the part had a viscosity greater than $65 \times 10^{-6} \text{ m}^2/\text{s}$ (65 cSt (centistokes) or 300 SUS (Saybolt Universal Seconds)), then the oil shall be heated to a temperature of $82 \pm 5 \text{ }^\circ\text{C}$ ($180 \pm 10 \text{ }^\circ\text{F}$).

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 $82 \pm 5 \text{ }^\circ\text{C}$ ($180 \pm 10 \text{ }^\circ\text{F}$) for at least 4 h.

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 min.