Designation: B 328 – 96

Standard Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Metal Structural Parts and Oil-Impregnated Bearings[1]

This standard is issued under the fixed designation B 328; 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.

1. Scope

1.1 This test method covers determination of the density, oil content, and interconnected porosity of sintered bearings and structural parts with or without oil impregnation.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

B 243 Terminology of Powder Metallurgy[2]

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[3]

D 1298 Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method[3]

3. Terminology

3.1 Definitions of powder metallurgy (P/M) terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material section of Vol 02.05 of the *Annual Book of ASTM Standards*.

4. Significance and Use

4.1 The volume of an arbitrary P/M shape cannot be accurately measured by standard techniques such as by micrometers or calipers. Since density is mass/volume, a precise method to measure the volume is needed. For nonporous objects, the volume of water displaced by the immersed object is determined by Archimedes principle. For porous P/M parts, a method is required to seal surface connected pores. If the pores are not sealed or the part is not oil impregnated, the part will absorb some of the water and decrease its buoyancy and exhibit an erroneously high density.

4.2 Density and oil content values are generally contained in the specifications for oil-impregnated bearings and other self-lubricating P/M parts. Desired lubrication requires sufficient interconnected porosity and satisfactory oil impregnation of the porosity.

4.3 For a particular P/M material, the mechanical properties of P/M structural parts are directly related to their density. Density values are therefore generally contained in the specifications for P/M parts.

5. Apparatus

5.1 *Analytical Balance*, of sufficient capacity and accurate to 0.01 % of the test specimen mass.

5.2 Device for weighing the test piece in air and in liquid (water); the water is distilled or deionized and preferably degassed. A wetting agent[4] is added to the water, 0.05 to 0.1 % by weight, to reduce surface tension effects.

5.3 *Soxhlet Extractor*, with oil solvent. Extractors may be purchased from most laboratory supply companies.[5]

5.4 Apparatus for vacuum impregnation of the test piece with oil.

5.5 *Beaker and Wires*, of various sizes. A wire basket may be used in place of wires (see Figs. 1 and 2).

5.6 *Thermometer*—Capable of reading temperature in the range of 10 to 38°C (50 to 100°F) to an accuracy of 0.5°C (1°F).

[1] This specification is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.04 on Bearings.

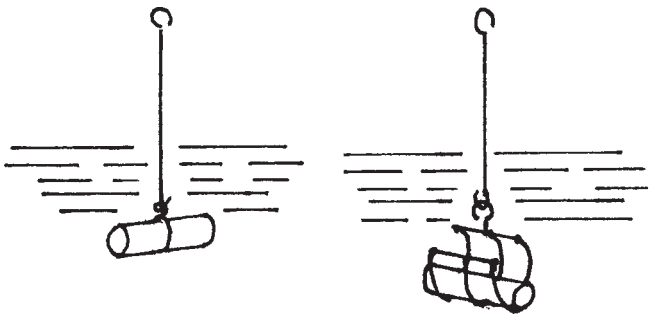
Current edition approved Sept. 10, 1996. Published November 1996. Originally published as B 328 – 58T. Last previous edition B 328 – 94.

[2] *Annual Book of ASTM Standards*, Vol 02.05.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] Kodak Photo-Flo 200, available from Momentum Graphics, 400 D. Pierce St., Somerset, NJ 08873, or its equivalent, has been found suitable.

[5] Extractors may be purchased from Fisher Scientific, 585 Alpha Drive, Pittsburgh, PA 15238; Cole-Parmer, 7425 North Oak Ave., Niles, IL 60714; V.W.R., P.O. Box 15646, Philadelphia, PA 19105-5645; or Thomas Scientific, P.O. Box 99, Swedesboro, NJ 08085-6099.



a. Twisted wire arrangement b. Basket support arrangement

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

5.7 *Lubricant*, of 20 to 65 cSt (100 to 300 SSU) at 38°C (100°F).

6. Test Specimen

6.1 The specimen mass shall be a minimum of 1.0 g. Several specimens may be used to reach the minimum mass.

7. Procedure

7.1 Using an analytical balance, obtain the mass of the as received oil-containing specimen (Mass J), the oil-free specimen (Mass A), and the fully impregnated specimen (Mass B). These, and all subsequent weighing, should be to 0.01 % of the mass of the part, for example:

Specimen Mass, g	Balance Sensitivity, 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

7.2 To determine Mass A, remove any oil from the samples by extracting it in a Soxhlet apparatus of suitable size using toluol or petroleum ether as a solvent. After extracting for approximately 1 h, remove the residual solvent by heating samples 1 h at 120°C (250°F) and weigh upon cooling. Continue alternate extractions and drying until the dry mass in air is constant to 0.05 % of the mass of the part.

7.2.1 For large parts or for a faster method, but not as accurate and with no concern for subsequent metallurgical properties, the oil can be removed by heating the specimen in a protective atmosphere in the temperature range of 430 to 650°C (800 to 1200°F). This method may be used if agreed upon by both parties. The selection of a proper burnout temperature may be critical in the case of sintered 90/10 Cu/Sn materials, depending upon the sintering temperature and time used by the fabricator. The normal sintering temperature range for bronze is 815 to 870°C (1500 to 1600°F), depending on the desired shrinkage, strength, and porosity relationships. This method is also applicable to sintered aluminum materials if the temperature does not exceed 540°C (1000°F).

7.3 For the purpose of determining the mass of oil-impregnated specimens in air (Mass B) or in water (Mass C), either of the following two methods may be used to impregnate the test specimen. The vacuum method is preferred.

7.3.1 At room temperature, using a suitable evacuating method, reduce the pressure over the immersed specimen to not more than 7 kPa (2 in. mercury) pressure for 30 min. Then permit the pressure to increase to atmospheric pressure and the specimen to remain immersed in oil 20 to 65 cSt (100 to 300 SSU) 38°C (100°F) at room temperature and pressure for 10 min.

7.3.2 Immerse the specimen in oil, viscosity of 20 to 65 cSt (100 to 300 SSU) at 38°C (100°F), hold at a temperature of 82°C ± 5°C (180°F ± 10°F) for at least 4 h, and then cool to room temperature by immersion in oil at room temperature.

7.4 To weigh the specimen in water, select a fine wire for supporting the specimen. Suspend the wire from the beam hook, while the specimen is immersed in a beaker of distilled water. Support the beaker of water over the pan of the balance, using a suitable bridge. The container of water may also be supported below the balance for weighing specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. Use a wetting agent (in the amount of 0.05 to 0.1 % by weight) to reduce the effects of surface tension. The recommended diameter of wire (copper or stainless steel) to be used for various weight range is as follows:

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

In place of attaching the specimen on a wire, the use of a wire basket suspended in water may be used as an alternate method (see Fig. 1b).

7.5 Twist the wire around the specimen and suspend it from the beam hook so that the specimen is completely immersed in the water. The water should cover the specimen by at least 6 mm (0.25 in.) and the wire twist should be completely submerged. Immersion should be to the same point each time. Take care to ensure that no air bubbles adhere to the specimen or to the wire. If a wire basket is used as an alternate method, completely immerse the wire basket in the water.

7.6 Weigh the specimen and wire in water. This is Mass C. If a wire basket is used as an alternate method, weigh the specimen and wire basket in water.

7.7 Remove the specimen and reweigh the wire in water immersed to the same point as before. This is Mass E. Some balances are capable of being tared automatically, which eliminates the need for reweighing the wire for correction. The mass of the oil impregnated specimen in water with the mass of wire tared is Mass F. Excess oil should be removed from the surface of the specimen before weighing. Care should be taken not to remove oil from the porosity of the part. Water density, D_w, is found in Table 1. Measure the temperature of the water to the nearest 1°C.

7.7.1 If a wire basket is used as the alternate method, follow the same procedure but substitute the basket for the wire.

7.8 For oil content and interconnected porosity, determine the density of the impregnant, which is (D_o). Oil content is defined as the percent oil content by volume in the part as received. The interconnected porosity is the percent oil content by volume in the part as it is impregnated under specific laboratory conditions.

NOTE 1—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 . Refer to