Designation: B 311 – 93 (Reapproved 2002)ε1

Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less Than Two Percent Porosity[1]

This standard is issued under the fixed designation B 311; 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.

ε[1] NOTE—Paragraphs 3.1, 4.1, 4.2, 5.4, and 6.1 were revised editorially in June 2002.

1. Scope

1.1 This test method covers the determination of density for powder metallurgy (P/M) materials containing less than two percent porosity and for cemented carbides. This test method is based on the water displacement method.

NOTE 1—A test specimen that gains mass when immersed in water indicates the specimen contains surface-connected porosity. Unsealed surface porosity will absorb water and cause density values higher than the true value. This test method is not applicable if this problem occurs.

1.2 The values stated in SI units are to be regarded as the standard. The values given 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 *ISO Standard:*

3369 Impermeable Sintered Metal Materials and Hardmetals—Determination of Density[2]

NOTE 2—The water density table in ISO 3369 differs from the table contained in this test method.

3. Summary of Test Method

3.1 Using an analytical balance, the test specimen is first weighed in air and then in water. The density is determined by calculation.

4. Significance and Use

4.1 For P/M materials containing less than two percent porosity, a density measurement may be used to determine if the part has been densified, either overall or in a critical region, to the degree required for the intended application. Density alone cannot be used for evaluating the degree of densification because chemical composition and heat treatment affect the pore-free density.

4.2 For cemented carbides, a density measurement is normally used to determine if there is any significant deviation in composition of the carbide grade. For straight tungsten carbide-cobalt grades, the relationship is straightforward. For complex carbide grades (for example, grades containing tantalum carbide or titanium carbide, or both, in addition to tungsten carbide-cobalt), the situation is more complicated. If the measured density is beyond the specified limits, the composition is outside of the specified limits. A measured density within the specified limits does not ensure correct composition; compensation between two or more constituents could result in the expected density with the wrong composition. Density alone cannot be used for evaluating a cemented carbide grade.

5. Apparatus

5.1 *Analytical Balance*, precision single-pan analytical balance that will permit readings within 0.01 % of the test specimen mass. The analytical balance shall be supported in a manner to eliminate mechanical vibrations and be shielded from air drafts.

5.2 *Weighing Liquid*—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 specimen support when immersed in water.

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

NOTE 4—A transparent container makes it easier to see air bubbles adhering to the test specimen and specimen support when immersed in water.

NOTE 5—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 2.5 mm (0.10 in.) when the test specimen is lowered into the water.

[1] This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.11 on Near Full Density Powder Metallurgy Materials.

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[2] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

5.4 *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, 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:

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	0.40 (0.015)
600 and greater	0.50 (0.020)

NOTE 6—For the most precise density determinations, it is important that the mass and volume of all supporting wires immersed in water be minimized.

5.5 *Thermometer*—A thermistor thermometer should be used to measure the temperature of the water to the nearest 0.5°C.

6. Preparation of Test Specimens

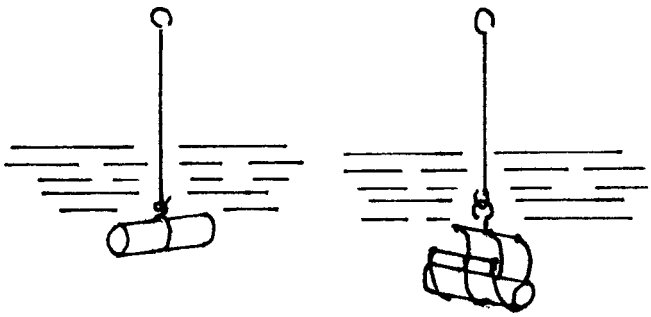
6.1 A complete part or a section of a part may be used for the test specimen. For the highest precision, the test specimen shall have a minimum mass of 5.0 g. If less precision can be tolerated, several test specimens may be used to reach the minimum mass, provided each test specimen has a mass of not less than 1.0 g.

6.2 All test specimen surfaces shall be thoroughly cleaned of all adhering foreign materials, such as, dirt, grease, oil, oxide scale, metal powders or assembly materials. For cut specimens, care must be used to avoid rough surfaces to which an air bubble can adhere. A100-grit sanding or abrasive grinding is recommended to remove all rough surfaces.

7. Procedure

7.1 Weigh the test specimen in air using an analytical balance. This is mass A. This and all subsequent weighings shall be to 0.01% of the test specimen mass, for example:

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



a. Twisted wire arrangement b. Basket support arrangement

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

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

NOTE 7—For the most precise density determination, duplicate weighings should be made for all mass determinations. The analytical balance should be adjusted to zero prior to each weighing. Duplicate mass determinations should be averaged before calculating the density.

NOTE 8—For improved reproducibility, the analytical balance should be periodically calibrated with a standard mass that is approximately equal to the test specimen mass.

7.2 Support the container of water over the pan of the balance using an suitable bridge as shown in Fig. 2. 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 suspension wire between the container of water and the bottom of the balance from air drafts.

7.3 Suspend the test specimen support with the test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least 6 mm (¼ in.) to minimize the effect of surface tension forces on the weighing. Care should be taken to ensure that the test specimen and specimen support hang freely from the balance beam hook, are free of air bubbles where immersed in the water and are at the same temperature as the water and balance. Care should also be taken to ensure the surface of the water is free of dust particles.

7.4 Weigh the test specimen and specimen support immersed in water. This is mass B.

7.5 Remove the test specimen. Weigh the test specimen support immersed in water at the same depth as before. This is mass C. Care should be taken 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 specimen immersed in water. The mass of the specimen support and specimen immersed in water is mass F, which replaces mass B minus mass C.

7.6 Measure the temperature of the water to the nearest 0.5°C and record its density E, at that temperature, from Table 1.

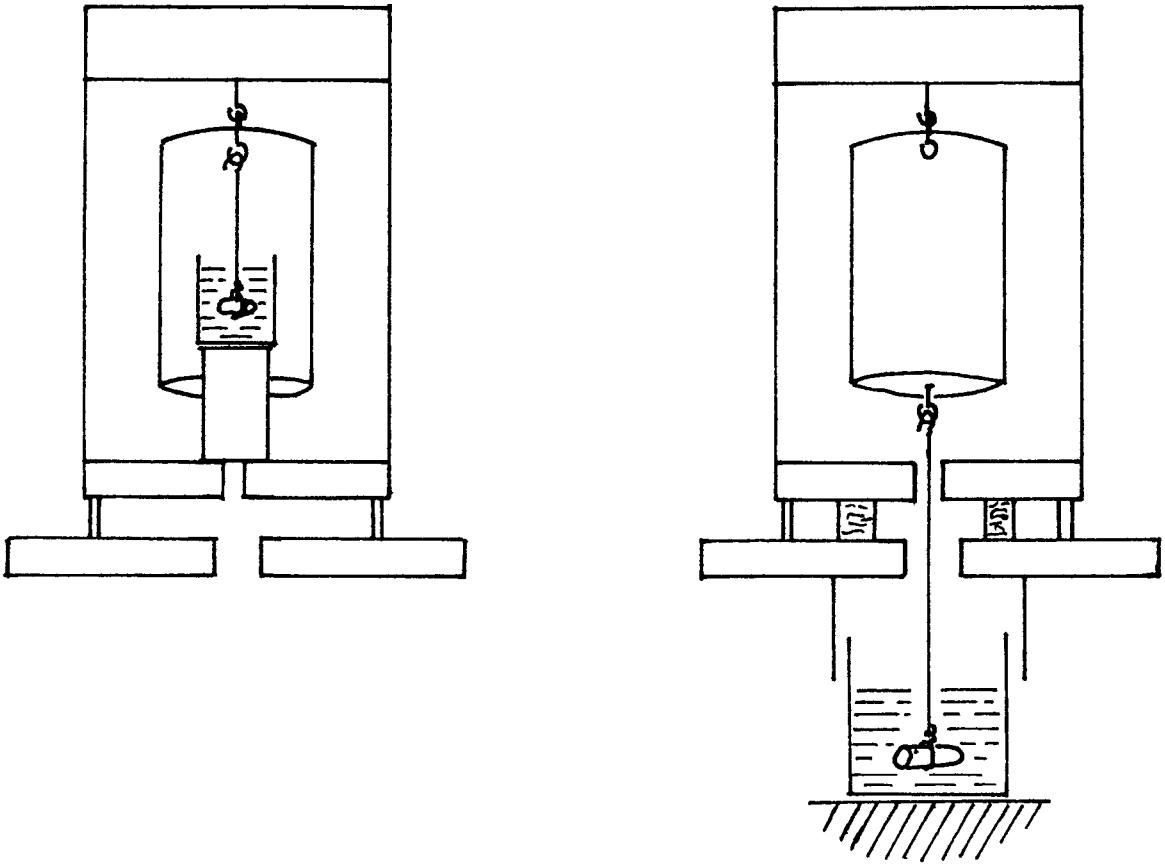
8. Calculation

8.1 Calculate the density as follows:

$$\text{Density} = D = \text{Mass/Volume} \tag{1}$$

$$D = A/\frac{[A - (B - C)]}{E} \tag{2}$$

$$D = (A \times E)/(A - B + C) = (A \times E)/(A - F) \tag{3}$$



a. Weighing a small specimen within the balance

b. Weighing a large specimen below the balance

FIG. 2 Methods for Weighing in Water

TABLE 1 Density of Air-Free Water[A]

Temperature (°C)	Density (g/cm^3)
18.0	0.9986
18.5	0.9985
19.0	0.9984
19.5	0.9983
20.0	0.9982
20.5	0.9981
21.0	0.9980
21.5	0.9979
22.0	0.9978
22.5	0.9976
23.0	0.9975
23.5	0.9974
24.0	0.9973
24.5	0.9972
25.0	0.9970
25.5	0.9969
26.0	0.9968
26.5	0.9966
27.0	0.9965
27.5	0.9964
28.0	0.9962
28.5	0.9961
29.0	0.9959
29.5	0.9958
30.0	0.9956

[A] *Metrological Handbook 145*," Quality Assurance for Measurements," National Institute of Standards and Technology, 1990, p. 9.10.

where:

D = density of test specimen, g/cm^3,

A = mass of test specimen in air, g,

B = apparent mass of test specimen and specimen support in water, g,

C = mass of specimen support immersed in water, g,

F = mass of test specimen in water with mass of specimen support tared, g, and

E = density of water in g/cm^3.

9. Report

9.1 Report the density rounded to the nearest 0.01 g/cm^3.

10. Precision and Bias

10.1 The following precision and bias data were developed using the procedures contained in Test Method B 311 – 86. An interlaboratory study is in-progress using the procedures in this revised test method.

10.2 For test specimens over 5 g mass, the repeatability interval, r, is 0.025 g/cm^3. Duplicate results from the same laboratory should not be considered suspect at the 95 % confidence level unless they differ by more than r.

10.3 For test specimens of over 5 g mass, the reproducibility interval, R, is 0.03 g/cm^3. Test results from two different laboratories should not be considered suspect at the 95 % confidence level unless they differ by more than R.

10.4 For test specimens of 1 to 5 g mass, the repeatability interval, r, is 0.025 g/cm^3.

10.5 For test specimens of 1 to 5 g mass, the reproducibility

interval, *R*, is 0.05 g/cm^3.

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

11. Keywords

11.1 cemented carbides; density; hard metals; metal injection molded (MIM) parts; powder metallurgy (P/M); powder forged (P/F) parts; powder metallurgy