

Designation: <del>B527 - 15</del> B527 - 20

# Standard Test Method for Tap Density of Metal Powders and Compounds<sup>1</sup>

This standard is issued under the fixed designation B527; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope\*

- 1.1 This test method specifies a method for the determination of tap density (packed density) of metal powders and compounds, that is, the density of a powder that has been tapped, to settle contents, in a container under specified conditions.
- 1.2 <u>Units—</u>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<sup>3</sup>) and gram (g) units is the long-standing industry practice, the values in SI units are to be regarded as 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 safety, health, and health 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.

### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

**B215** Practices for Sampling Metal Powders

B243 Terminology of Powder Metallurgy

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

**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 Terms Specific to This Standard: Terms used in this test method are defined in B243.
- 3.1 *Definitions*—For definitions of terms used in this test method, see Terminology B243. Additional descriptive PM information is available in the *Committee Documents* section of the B09 Committee web page.

#### 4. Significance and Use

4.1 This test method covers the evaluation of the tap density physical characteristic of metal powders and related compounds. The measured tap density bears a relationship to the mass of powder that will fill a fixed volume die cavity or other container in situations where the container is tapped, vibrated, or otherwise agitated. The degree of correlation between the results of this test method and the quality of powders in use will vary with each particular application and has not been fully determined.

## 5. Apparatus

- 5.1 Balance, of appropriate capacity to satisfy the requirements shown in Table 1 and accuracy of  $\pm$  0.05 g.
- 5.2 Graduated Glass Cylinder, calibrated to contain  $\frac{100 \text{ cm}}{100 \text{ cm}}^3$  at 20 °C, the height of the graduated portion being approximately  $\frac{175 \text{ mm.}}{175 \text{ mm.}}$  The graduations shall be at 1 cm<sup>3</sup> intervals, thus allowing a measuring accuracy of  $\frac{\pm 0.5}{100 \text{ cm}}$  cm<sup>3</sup>. For apparent densities over  $\frac{4}{100 \text{ cm}}$ , do not use the  $\frac{100 \text{ cm}}{100 \text{ cm}}$  eylinder; see section 5.2.1.1.

<sup>&</sup>lt;sup>1</sup> This specification-test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.03 on Refractory Metal Powders.

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<sup>&</sup>lt;sup>2</sup> 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.

TABLE 1 Capacity and Accuracy of the Test Portion Mass and Graduated Cylinder

Apparent Density	Mass of Test Portion	Cylinder Capacity	Cylinder Accuracy
g/cm <sup>3</sup>	g	cm <sup>3</sup>	cm <sup>3</sup>
<1	50 ± 0.2	100	0.5
1-4	$100 \pm 0.5$	100	0.5
>4	$100 \pm 0.5$	25	0.2

- 5.2.1 Alternatively, the following may be used:
- 5.2.1.1 *Graduated Glass Cylinder*, calibrated to contain  $\frac{25 \text{ cm}}{25 \text{ cm}}^3$  at 20 °C, the height of the graduated portion being approximately  $\frac{135 \text{ mm.}}{25 \text{ mm.}}$  The graduations shall be at  $\frac{0.2 \text{ cm}}{25 \text{ cm}}$  intervals, thus allowing a measuring accuracy of  $\frac{135 \text{ mm.}}{25 \text{ cm}}$  cm<sup>3</sup>.
- 5.2.1.2 A 25 cm25 cm<sup>3</sup> cylinder shall be used for powders of apparent density higher than 4-4 g g/em/cm<sup>3</sup>, in particular for refractory metal powders.
- 5.3 Tapping Apparatus, which permits the tapping of the graduated cylinder against a firm base. The tapping shall be such that a densification of the powder can take place without any loosening of its surface layers. The stroke shall be 3 mm 3 mm and the tapping frequency shall be between 100 and 300300 taps taps/min./min. An example of a tapping apparatus is shown in Fig. 1.

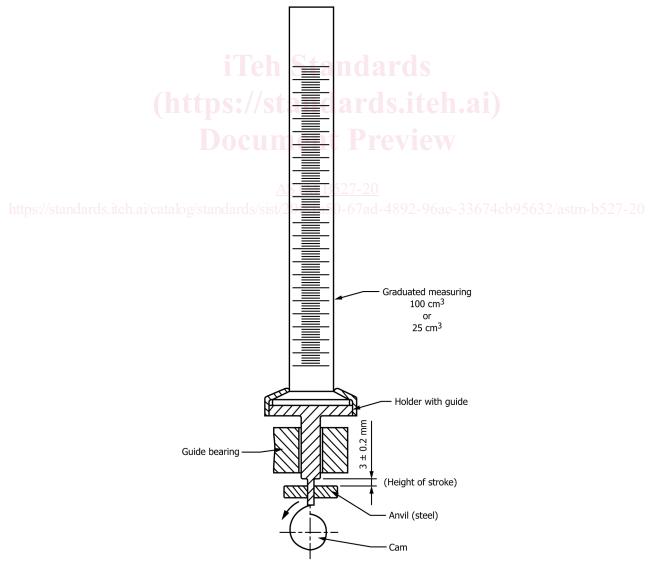


FIG. 1 Example of Tapping Apparatus

# 6. Test Specimen

- 6.1 For the quantities of powder required for each test, see Table 1. Obtain test portions according to Practices B215.
- 6.2 In general, the powder should be tested in the as-received condition. In certain instances the powder may be dried. However, if the powder is susceptible to oxidation, the drying shall take place in a vacuum or in inert gas. If the powder contains volatile substances, it shall not be dried.
  - 6.3 The test shall be carried out on three test portions, if possible.

### 7. Procedure

- 7.1 Clean the inside wall of the graduated cylinder (see 5.2) with a suitable clean brush or, if necessary, by rinsing with a solvent, such as acetone. If a solvent is used, thoroughly dry the cylinder before reuse.
  - 7.2 Weigh, to the nearest 0.1 g, the mass of the test portion as indicated in Table 1, using a balance (see 5.1).
- 7.3 Pour the test portion into the graduated cylinder. Take care that a level surface of the powder is formed. Place the cylinder in the tapping apparatus (see 5.3). Tap the cylinder until no further decrease in the volume of the powder takes place (see Note 1).

Note 1—In practice, the minimum number of taps, N, such that no further change in volume takes place would be determined. For all further tests on the same type of powder, the cylinder would be subjected to 2N taps, except where general experience and acceptance have established a specific number of taps (no less than N taps) as being satisfactory. For fine refractory metal powders, 3000 taps has been found to be satisfactory for all sizes.

7.4 If the tapped surface is level, read the volume directly. If the tapped surface is not level, determine the tap volume by calculating the mean value between the highest and the lowest reading of the tapped surface. Read the final volume to the nearest 0.5 cm<sup>3</sup> when using a 100 cm<sup>3</sup> cylinder and to the nearest 0.2 cm<sup>3</sup>when using a 25 cm<sup>3</sup> cylinder.

### 8. Calculation

8.1 The tap density is given in the following equation

(https://standards.iteh.ai)

TD =  $\frac{M}{V}$  (1)

where:

9. Report

 $TD = \text{tap density, g/cm}^3$ ,

M = mass of powder, g, and

V = volume of tapped powder, cm<sup>3</sup>.

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- 9.1 Report the following information: tandards/sist/20f81a50-67ad-4892-96ae-33674cb95632/astm-b527-20
- 9.1.1 Reference to this test method, method;
- 9.1.2 All details necessary for identification of the test sample;
- 9.1.3 The drying procedure, if the powder has been dried, dried;
- 9.1.4 Cylinder capacity, mass of test portion, and method <del>used,</del>used;
- 9.1.5 All results obtained. Report tap density to the nearest 0.1 g/cm<sup>3</sup> for powders with apparent densities up to and including 4 g/cm<sup>3</sup>. For powders with apparent densities higher than 4 g/cm<sup>3</sup>, report to the nearest 0.2 g/cm<sup>3</sup>;
  - 9.1.6 All operations not specified in this test method or regarded as optional; and
  - 9.1.7 Details of any occurrence that may have affected the result.

# 10. Precision and Bias

- 10.1 Precision—The precision of this test method is based on an intralaboratory interlaboratory study of Test Method B527, conducted in 2011–2012. A single laboratory with two operators analyzed three different powders for tap density. Either 10 or 20 replicates were reported for each material in this study. Except for testing in just one lab, B527 conducted in 2018. Each of 8 laboratories tested 9 different materials. Every "test result" represents an individual determination, and all participants reported triplicate test results. Practice E691 was followed for the study design; the design and analysis of the data; the details are given in ASTM Research Report No.\_RR:B09-1020RR:B09-1025. The precision statement was determined through statistical examination of 50 results, from a single laboratory, testing three materials. The materials were designated in the study as:
- 1. Cermet (TiC based), apparent density 1.02 g/cm<sup>3</sup>
- 2. Iron (Ancorsteel 1000), apparent density 2.92 g/cm<sup>3</sup>
- 3. Tungsten carbide (WC), apparent density 6.91 g/cm<sup>3</sup>

<sup>&</sup>lt;sup>3</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1020 RR:B09-1025. Contact ASTM Customer Service at service@astm.org.



- 10.1.1 Test Results—Repeatability Limit (r)—The precision information presented herein has been calculated for the comparison of three materials, each of which is the mathematical average of ten or twenty individual test determinations: difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.
- 10.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.
- 10.1.1.2 95 % Repeatability Limit (r)—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the r value for that material; r is the interval representing the critical difference between two test results for the same material, using the same mass and appropriate volumetric cylinder capacity according to Table 1. Repeatability limits are listed in Table 2. The within-laboratory repeatability limit, r, as defined by Terminology E456, is dependent on the apparent density of the tested material (see Table 2). At the 95 % confidence level, duplicate tap density results from the same laboratory should not be considered to be different unless they differ by more than r.
- 10.1.1.2 Reproducibility Limit (R)—The reproducibility limit of this test method is being determined and will be available in December, 2019. Reproducibility results cannot be determined from a single laboratory study.
- 10.1.2 Reproducibility Limit (R)—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.
- 10.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.
  - 10.1.2.2 Reproducibility limits are listed in Table 2.
  - 10.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.
- 10.1.4 Any judgment in accordance with statements 9.1.1 and 9.1.2 would have an approximate 95 % probability of being correct.
- 10.2 Bias—No information can be presented on the bias of the procedure in Test Method B527 for measuring tap density because no material having an accepted reference value is available.
  - 10.3 The above terms ("repeatability limit" and "reliability limit") are used as specified in Practice E177.
- 10.2 <u>Bias—Any judgment in accordance with statement At the time</u> 10.1.1 would normally have an approximate 95 % probability of being correct, however because of the limited number of participants in this study, consider the precision noted as an estimate of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.
  - 10.3 The precision statement was determined through statistical examination of 195 results, from 8 laboratories, on 9 materials.
- 10.4 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

## TABLE 2 Tap Density (g/cm<sup>3</sup>)

<del>Material</del>		Average <sup>A</sup>	Repeatability	Repeatability Limit
(100 gm per run)	Cylinder Capacity	<del>x</del> -	<del>Standard</del>	f
	<del>cm<sup>3</sup></del>		<del>Deviation</del>	
	_		<del>S</del> r	
Cermet – 20 replicates	<del>100</del>	<del>1.6</del>	0.02	0.06
<del>Iron – 20 replicates</del>	<del>100</del>	<del>3.7</del>	<del>0.10</del>	<del>0.29</del>
Tungsten Carbide (WC) -	<del>25</del>	<del>8.2</del>	0.07	0.20
10 replicates				

## TABLE 2 Tap Density (TD), g/cm<sup>3</sup>

<u> </u>							
Material	Average <sup>A</sup>	Repeatability	Reproducibility	Repeatability Limit	Reproducibility Limit		
	_	<u>Standard</u>	Standard Deviation	<u>r</u>	<u> </u>		
		Deviation	<u>s</u>				
		$s_r$					
Ancorsteel 1000 Powder	3.90	0.04	<u>0.10</u>	<u>0.11</u>	0.29		
Ancorsteel 85HP Powder	3.95	0.06	0.13	0.18	0.37		
Coarse 316L Stainless Steel Powder	4.73	0.05	0.17	0.15	0.48		
Coarse Tungsten Carbide Powder	7.05	0.13	0.39	0.35	1.10		
Coarse Tungsten Powder	9.29	0.18	1.32	0.49	3.71		
Fine 316L Stainless Steel Powder	4.79	0.08	0.24	0.23	0.69		
Fine Tungsten Carbide Powder	4.62	0.06	0.23	0.15	0.63		
Fine Tungsten Powder	4.16	0.05	0.29	0.15	0.82		
Molybdenum Powder	2.52	0.03	0.09	0.07	0.25		

 $<sup>^{</sup>A}\overline{\text{The}}$  The average of the laboratories' calculated averages.