



Designation: C1770 – 21

Standard Test Method for Determination of Loose and Tapped Bulk Densities of Small Quantities of Plutonium Oxide¹

This standard is issued under the fixed designation C1770; 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.

1. Scope

1.1 This test method specifies a method for the determination of loose and tapped bulk density of plutonium oxide (PuO_2) powder.

1.2 This test method is applicable when limited quantities of powder are available for performance of the measurements. Alternative test methods, such as Test Methods [B527](#) or [D7481](#), may be used when sufficient quantities are available.

1.3 This test method contains notes that are explanatory and are not part of the mandatory requirements of the method.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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. Some specific hazards statements are given in Section 7 on Hazards.*

1.6 *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

[B527 Test Method for Tap Density of Metal Powders and Compounds](#)

¹ This test method is under the jurisdiction of ASTM Committee [C26](#) on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee [C26.05](#) on Methods of Test.

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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.

[C859 Terminology Relating to Nuclear Materials](#)

[D3766 Terminology Relating to Catalysts and Catalysis](#)

[D6393 Test Method for Bulk Solids Characterization by Carr Indices](#)

[D7481 Test Methods for Determining Loose and Tapped Bulk Densities of Powders using a Graduated Cylinder](#)

[E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

[E1272 Specification for Laboratory Glass Graduated Cylinders](#)

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method but not defined herein, refer to Terminology [C859](#) and Terminology [E135](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bulk density, n*—ratio of the mass of a collection of discrete pieces of solid material to the sum of the volumes of the solids in each piece, the voids within the pieces, and the voids among the pieces of the particular collection. [D3766](#)

3.2.2 *loose bulk density, n*—bulk density that results from pouring the powder into a heap or container in the absence of any applied compression. [D7481](#)

3.2.3 *tapped bulk density, n*—bulk density resulting from the application of compression, for example, impact or vibration. [D7481](#)

4. Summary of Test Method

4.1 Loose bulk density is determined by measuring the volume of a known mass of PuO_2 powder poured into a graduated cylinder with a capacity of 10 mL, 25 mL, 50 mL, or 100 mL. The cylinder size should be selected based on the amount of PuO_2 powder available for the test.

4.2 The cylinder is then mechanically tapped at a dropping height of 3 mm for 2 min at 250 taps per minute, and the resulting volume is then measured. This process is repeated for cycles of 2 min until the difference in tapped volumes is less than 4 % (this corresponds to a change of less than one graduation on the 10 mL, 25 mL, and 50 mL graduated cylinders and two graduations on the 100 mL graduated cylinder).

5. Significance and Use

5.1 This test method is intended for determination of loose or tapped bulk density or both for PuO₂ powders or compounds in the nuclear industry. It is intended for use when the quantity of available material for performing the measurements is limited because of reasons such as nuclear safety or laboratory scale limits on nuclear inventory.

5.2 This test method can be applied to other metal powders in the nuclear industry provided that appropriate validation has been performed.

5.3 Values of loose bulk density obtained using this test method should be used with caution since they can vary considerably depending on factors such as the initial state of dispersion of the test specimen, height-to-diameter ratio of the specimen in the graduated cylinder, the dryness of the powder, and operator-to-operator variation (for example, the speed with which the sample is poured into the cylinder).

5.4 The data from the tapped bulk density test can be used to estimate the needed volume of small containers holding a fixed mass of powder that has been compacted.

5.5 This test method may be useful for the determination of the Carr Compressibility Index as described in Test Method D6393.

6. Apparatus

6.1 *Balance*, analytical, 0.1 mg sensitivity.

6.2 *Graduated glass cylinder*, with a capacity of 10 mL, 25 mL, 50 mL, or 100 mL, Class A, meeting the requirements of Specification E1272.

6.3 *Tapping Apparatus*, with the following features (see Fig. 1).

6.3.1 The apparatus permits the tapping of the graduated cylinder against a firm base.

6.3.2 The apparatus shall be capable of a tapping rate of 250 + 15 taps per minute.

6.3.3 The minimum stroke or dropping height shall be 3 mm.

6.3.4 The apparatus shall have an adjustable counter that can be pre-set to deliver between 1 and 9999 taps.

7. Procedure

7.1 Ensure that the balance is set on a sturdy surface, leveled, and zeroed.

7.2 Ensure that the graduated glass cylinder is dry and clean of any and all foreign material before starting each test.

7.3 Determine and record the mass of the graduated cylinder to the nearest 0.1 mg.

7.4 Transfer the powder to the graduated cylinder as follows:

7.4.1 Using a powder funnel, slowly pour the sample into the graduated cylinder.

7.4.2 Ensure that the volume of the loose bulk powder is at least 40 % of the volume of the cylinder (for example, when using a 10 mL cylinder, the minimum volume is 4.0 mL.)

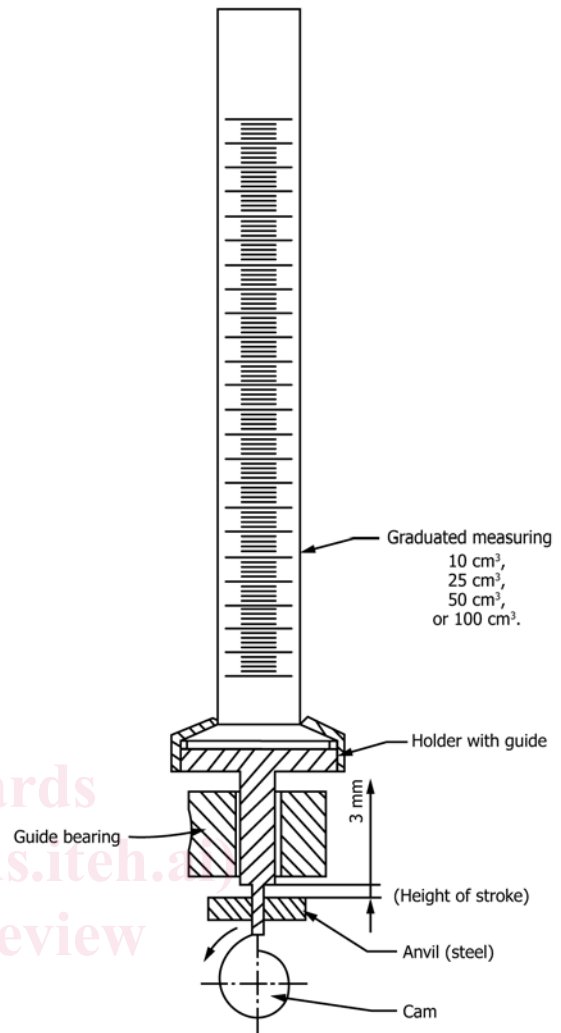


FIG. 1 Example of Tapping Apparatus (Adapted from Test Method B527)

NOTE 1—These volumes are necessary to determine whether the difference in tapped volumes is less than 4 % (see 7.8).

7.4.3 Avoid attempting to level the surface since PuO₂ powder will self-compress yielding an artificially high loose bulk density.

7.5 Record the loose bulk powder volume, V_L . If the surface is level, read the volume directly. If the surface is not level, determine the volume by calculating the mean value between the highest and the lowest reading of the surface. Record the volume to the nearest cylinder graduation.

7.6 Place the graduated cylinder containing the sample onto the balance. Determine and record the mass to the nearest 0.1 mg.

7.7 Load the graduated cylinder containing the sample into the tapping apparatus. Tap the cylinder as described in either 7.8 or 7.9:

7.8 *Tapping procedure for processes not well characterized:*

7.8.1 Tap the cylinder for 2 min at 250 taps per minute for a total of 500 taps.

7.8.2 Read and record the tapped bulk powder volume, V_T , directly to the nearest cylinder graduation.

7.8.3 Tap the cylinder for an additional 2 min at 250 taps per minute.

7.8.4 Read and record the volume, V_T .

7.8.5 If the difference between the two tapped volumes in 7.8.2 and 7.8.4 is less than 4 %, use the smaller of the two measurements in 7.8.2 and 7.8.4 for the tapped volume. Otherwise, repeat 7.8.3 and 7.8.4 until the difference between succeeding measurements is less than 4 %.

7.9 *Tapping Procedure for Well-characterized Processes:*

7.9.1 Tap the cylinder for a minimum of 1000 taps.

7.9.2 Read and record the tapped bulk powder volume, V_T , directly to the nearest cylinder graduation.

8. Calculation

8.1 Calculate the loose bulk density using Eq 1:

$$\rho_L = \frac{(M_{(C+S)} - M_C)}{V_L} \quad (1)$$

Where:

ρ_L = loose bulk density, g/mL,

$M_{(C+S)}$ = mass of cylinder plus sample, g,

M_C = mass of cylinder, g, and

V_L = volume of loose bulk powder, mL.

8.2 Calculate the tapped bulk density using Eq 2:

$$\rho_T = \frac{(M_{(C+S)} - M_C)}{V_T} \quad (2)$$

Where:

ρ_T = tapped bulk density, g/mL³,

$M_{(C+S)}$ = mass of cylinder plus sample, g,

M_C = mass of cylinder, g, and

V_T = volume of tapped bulk powder, mL³.

NOTE 2—The difference between $M_{(C+S)}$ and M_C is the mass of the powder sample.

9. Test Data Sheet

9.1 *General Information*—Record as a minimum the following:

9.1.1 Requesting agency or client,

9.1.2 Analyst, and

9.1.3 Date.

9.2 *Test Specimen Information*—Record as a minimum the following:

9.2.1 Unique sample identification, and

9.2.2 Sample description.

9.3 *Test Results*—Record as a minimum the following:

9.3.1 Volume of graduated cylinder,

9.3.2 Mass of empty graduated glass cylinder, M_C ,

9.3.3 Mass of cylinder plus sample, $M_{(C+S)}$,

9.3.4 Volume of loose bulk powder, V_L ,

9.3.5 Calculated loose bulk density, ρ_L ,

9.3.6 Number of taps and corresponding values of tapped bulk powder volume, V_T , for each cycle of tapping performed,

9.3.7 Calculated tapped bulk density, ρ_T , and

9.3.8 Identification for tapping apparatus used.

10. Precision and Bias

10.1 *Precision*—The repeatability standard deviation (1σ) of the method as determined by a single laboratory has been determined to be between 4 % for loose bulk density and 2 % for tapped bulk density. These standard deviations were calculated from repeated measurements (ten measurements) of two different samples (see Tables 1 and 2). The reproducibility of this test method is not provided at this time because relatively few laboratories perform this analysis. In addition, the cost and complexity of the logistics associated with the transfer of PuO₂ between laboratories is considered to be prohibitive. As a result, it is not likely that reproducibility data will be available in the future. If any user is aware of repeatability data from other laboratories, please contact the technical committee having jurisdiction over this standard, and those data will be considered for future versions of the standard.

10.2 *Bias*—The bias of the test method cannot be tested reliably because of the lack of suitable matrix matched reference materials.

11. Keywords

11.1 apparent density; bulk density; loose bulk density; nuclear materials; packed density; plutonium oxide; poured density; powder metallurgy; tapped bulk density

TABLE 1 Repeatability Data for Loose Bulk Density and Tapped Bulk Density Measurement of PuO₂ (Sample 1)

NOTE 1—All units for cylinders and weights are in grams (g), volumes in millilitres (mL), and densities are in g/mL.

Sample ID	Empty Cylinder	Full Cylinder	Net Weight	Bulk Volume	Loose Bulk Density	Tapped Volume	Tapped Bulk Density
Sample 1 Measurement 1	37.8	68.0	30.2	6.50	4.6	5.90	5.1
Sample 1 Measurement 2	37.8	68.3	30.5	6.80	4.5	5.85	5.2
Sample 1 Measurement 3	37.8	67.8	30.0	6.90	4.3	6.05	5.0
Sample 1 Measurement 4	37.8	69.2	31.4	7.05	4.5	6.30	5.0
Sample 1 Measurement 5	37.8	69.0	31.2	7.70	4.1	6.45	4.8
Sample 1 Measurement 6	37.8	68.8	31.0	7.20	4.3	6.10	5.1
Sample 1 Measurement 7	37.8	68.7	30.9	7.15	4.3	6.15	5.0
Sample 1 Measurement 8	37.8	68.6	30.8	7.00	4.4	6.20	5.0
Sample 1 Measurement 9	37.8	68.1	30.3	7.05	4.3	6.00	5.1
Sample 1 Measurement 10	37.8	70.0	32.2	7.65	4.2	6.45	5.0
				Average	4.4		5.0
				St. Dev.	0.2		0.1