Designation: D6683 – 14

Standard Test Method for Measuring Bulk Density Values of Powders and Other Bulk Solids as Function of Compressive Stress[1]

This standard is issued under the fixed designation D6683; 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 covers an apparatus and procedure for determining a range of bulk densities of powders and other bulk solids as a function of compressive stress.

1.2 This test method should be performed in the laboratory under controlled conditions of temperature and humidity.

1.3 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.3.1 The procedures used to specify how data are collected/recorded or calculated in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives, and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.4 *Units*—The values stated in SI units are to be regarded as standard. No other units of measure 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D6026 Practice for Using Significant Digits in Geotechnical Data

3. Terminology

3.1 *Definitions*—For common definitions of terms in this standard, refer to Terminology D653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *maximum effective head, n*—in powders, height of a column of material that has no shear stresses along its vertical walls. Used in calculation of maximum applied mass, this value can be approximated, for example, by using the height of the cylindrical section of the bin to be analyzed, m.

3.3 *Symbols:*

3.3.1 A_{cup}—inside cross-sectional area of density cup, m^2.

3.3.2 AM_{max}—calculated value of maximum applied mass, kg.

3.3.3 D_{cup}—inside diameter of density cup, m.

3.3.4 EH_{max}—maximum effective head to be applied to material in density cup, m.

3.3.5 $M_{mat'l}$—mass of material in density cup, kg.

3.3.6 V_i—calculated volume of material in density cup at i^{th} consolidation step, m^3.

3.3.7 $(\rho_b)_{approx}$—approximate value of material's bulk density used in calculation of maximum applied mass, kg/m^3.

3.3.8 $(\rho_b)_i$—calculated bulk density value at i^{th} consolidation step, kg/m^3.

3.3.9 $(\rho_b)_{initial}$—calculated initial bulk density value, kg/m^3.

3.3.10 σ_i—calculated compressive stress at i^{th} consolidation step, N/m^2.

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.24 on Characterization and Handling of Powders and Bulk Solids.

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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.

***A Summary of Changes section appears at the end of this standard**

3.3.11 σ_{max}—maximum compressive stress to be applied to material in density cup, N/m^2.

4. Summary of Test Method

4.1 Bulk density values are determined by calculating the volume of a given mass of bulk solid under increasing compressive stress.

5. Significance and Use

5.1 The data from this test can be used to estimate the bulk density of materials in bins and hoppers and for material handling applications such as feeders.

5.2 The test results can be greatly affected by the sample selected for testing. For meaningful results it is necessary to select a representative sample of the particulate solid with respect to moisture (water) content, particle-size distribution and temperature. For the tests an appropriate size sample should be available, and fresh material should be used for each individual test specimen.

5.3 Initial bulk density, $(\rho_b)_{initial}$, may or may not be used as the minimum bulk density. This will depend on the material being tested. For example, the two are often close to the same for coarse (most particles larger than about 6 mm), free-flowing bulk solids, but not for fine, aeratable powders.

5.4 Bulk density values may be dependent upon the magnitude of the applied mass increments. Traditionally, the applied mass is doubled for each increment resulting in an applied mass increment ratio of 1. Smaller than standard increment ratios may be desirable for materials that are highly sensitive to the applied mass increment ratio. An example of the latter is a material whose bulk density increases 10% or more with each increase in applied mass.

5.5 Bulk density values may be dependent upon the duration of each applied mass. Traditionally, the duration is the same for each increment and equal to 15 s. For some materials, the rate of compression is such that complete compression (no change in volume with time at a given applied compressive stress) will require significantly more than 15 s.

NOTE 1—The quality of the result produced by this standard is dependent on the competence of personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors. Practice D3740 was developed for agencies engaged in the testing or inspection (or both) of soil and rock. As such it is not totally applicable to agencies performing this standard. However, users of this standard should recognize that the framework of Practice D3740 is appropriate for evaluating the quality of an agency performing this standard. Currently there is no known qualifying national authority that inspects agencies that perform this standard.

6. Apparatus

6.1 A typical embodiment of the test apparatus is shown in Fig. 1.

6.2 *Balance,* having a capacity and readability to determine mass of the specimen and applied masses to four significant digits in accordance with Table 1 in Guide D4753.

6.3 *Stand,* to support the density cup, and to mount the dial indicator. The stand must be level and securely mounted on a vibration free base to support the test apparatus.

6.4 *Density Cup,* with cover to contain the test specimen. Density cup cover has a ball mounted in the center, which acts as a pivot point to ensure that only a vertical force is exerted on the cover by the applied mass. The density cup is to be a cylindrical cup with the minimum cell diameter of 64 mm and a minimum inside height of 21 mm or five times the diameter of the largest particle whichever results in the larger cell height. The ratio of cell diameter-to-height must be at least 3:1.

6.5 *Applied masses,* to be used with the hanger for applying compressive stress.

6.6 *Hanger,* to support applied masses and guide load onto the density cup cover. A thin, short rod extends between the hanger and cover to prevent the hanger from coming in contact with the density cup or its cover.

6.7 *Dial or Digital Displacement Indicator,* to measure change in height. Indicator should be able to read in 0.01 mm increments and apply negligible (if any) force on the test specimen in the density cup. The spring force from the dial indicator is assumed to be negligible in this test.

6.8 *Plug,* gauge block used to zero the dial indicator. Its length should be equal to the inside height of the density cup.

6.9 *Mass Support,* to support applied masses as they are added to compress the material.

6.10 *Scraper,* used to scrape off excess material from top of cup. It should be straight and flat, with a length in excess of cup diameter and a width of at least 15 mm.

7. Preparation of Apparatus

7.1 Check that the balance is set on a sturdy table or bench, level and zeroed, and its calibration/verification sticker is within requirements.

7.2 Make sure that the density cup and cover are clean and free of foreign material prior to starting each new test.

7.3 Check that the applied masses are clean of foreign material and have a known mass.

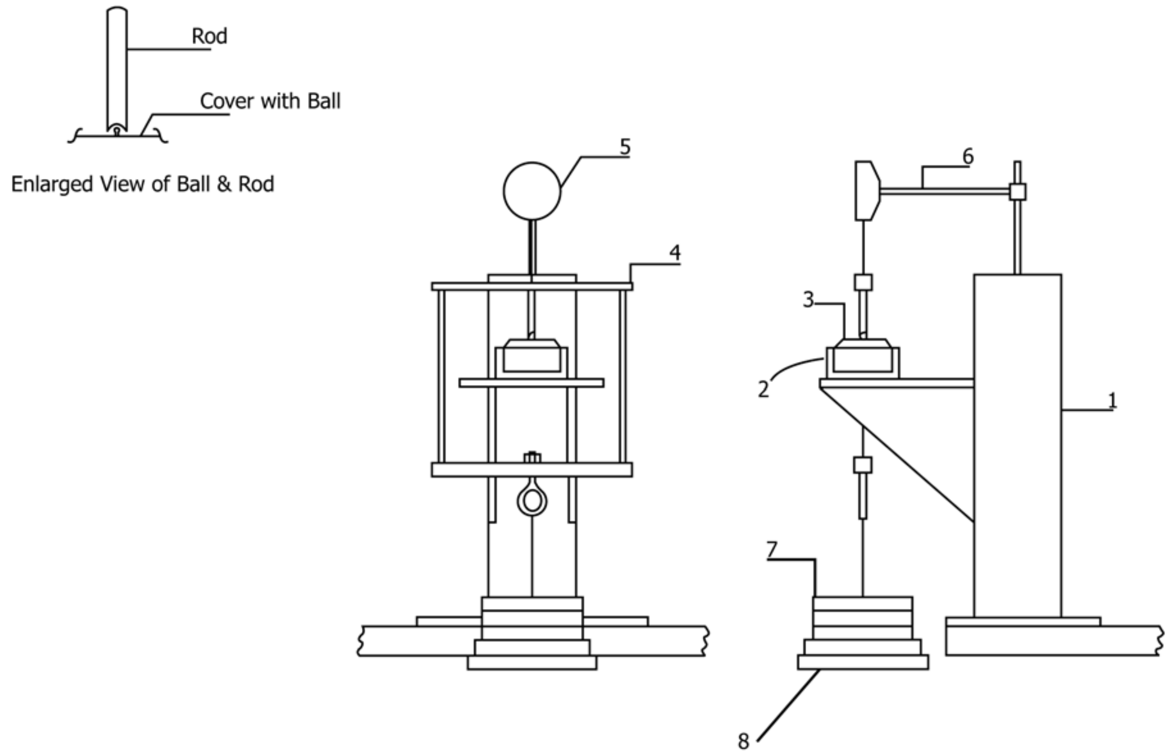
7.4 Select a minimum of five applied masses to be used according to the following procedure. Additional applied masses may be used if more data points are desired or required.

7.4.1 Calculate the maximum applied mass, AM_{max} by multiplying the material's approximate bulk density, $(\rho_b)_{approx}$ (kg/m^3) by maximum effective head to be applied, EH_{max} (m) times the inside cross-sectional area of the density cup, A_{cup}.

$$A_{cup} = \pi (D_{cup})^2/4,\ m^2$$

$$AM_{max} = (\rho_b)_{approx} (EH_{max}) (A_{cup}),\ kg$$

7.4.2 Alternatively, if the maximum compressive stress to be applied to the material, σ_{max} has been specified, the maximum applied mass, AM_{max} is calculated by multiplying σ_{max} by the inside cross-sectional area of the density cup, A_{cup}, and then dividing the product by the acceleration of gravity (g), where $g = 9.81\ m/s^2$



1. Stand
2. Density cup
3. Cover
4. Hanger
5. Dial indicator
6. Dial indicator holder
7. Applied masses
8. Support

FIG. 1 Test Apparatus

$$AM_{max} = \sigma_{max}\left(A_{cup}\right)/g,\ kg$$

7.4.3 Divide the maximum applied mass, AM_{max}, in half then in half again and continue until at least five applied masses have been identified.

8. Procedure

8.1 Determine the mass of the density cup and record this value to the nearest 0.1 g or better on a test data sheet as the tare mass.

8.2 Determine the mass of the cover and the hanger (this becomes the initial mass). Be sure this mass is less than the smallest applied mass to be used. These are to be recorded to the nearest 0.1 g or better on a test data sheet, and the total of these will be used to calculate the first compressive stress.

8.3 Insert the plug (gauge block) inside the density cup. Place the cover on the plug, next place the density cup on the test stand, and then place the hanger on the cover. Position the dial indicator on the top of the weight hanger, then set the dial indicator to zero. Now carefully lift the plunger of the dial indicator far enough to remove the hanger and slide the density cup out from under the dial indicator so when the material is put into the density cup, the dial indicator won't be accidentally bumped.

8.4 Remove the cover and plug.

8.5 Place the test specimen into a mixing bowl, and stir the material with a spatula to be assured the material hasn't agglomerated from transit. Avoid agglomeration and segregation of material.

8.6 Carefully spoon the material into the density cup as loosely as possible to the point of overflowing. Scrape off all excess material using a straight flat scraper. The angle of the scraper as it is drawn across the density cup should be such that it does not compress the material in the cup. Allow the test specimen to set for approximately one minute. If it settles below the top of the density cup add additional material to bring the level above the cover and scrape again. Determine the mass of the density cup again with the material in the cup. Be careful not to spill any material. Record this value to the nearest 0.1 g or better on the test data sheet.

8.7 Place the density cup back on the stand, then carefully place the cover so it is centered on the density cup. Place the centering rod of the hanger over the ball which is mounted to the center of the cover, and is used as a pivot for the weight hanger. Slowly lift the plunger of the dial indicator, and slide the cup, cover and hanger under the plunger. Allow the plunger to come down and rest on the hanger. Record the change in height indicated by the dial indicator to the nearest 0.01 mm on the test data sheet.

8.8 Hang the mass support on the bottom of the hanger. Record the change in height due to this additional mass to the nearest 0.01 mm on the test data sheet.

8.9 Record the change in height to the nearest 0.01 mm with each applied mass that is added. Allow 15 s between each change in applied mass or until there is no noticeable change in height, as indicated by the dial indicator.

NOTE 2—At the completion of the test, visually inspect the cover's position with respect to the density cup. If it is visibly tilted, the test is not valid and should be repeated, paying particular attention to procedure steps 8.5 and 8.6. Tilting of the lid is most often caused by non-uniform initial bulk density in the density cup.

9. Calculation

9.1 Determine the mass of the material in the density cup, $M_{mat'l}$ (kg) by subtracting the mass of the density cup from the value obtained of the density cup with material in it. This mass is in kg.

9.2 Determine the initial bulk density, $(\rho_b)_{initial}$ (kg/m^3) by dividing the mass of material in the density cup, $M_{mat'l}$ (kg) by the volume of the cup determined by multiplying the inside cross-sectional area of the density cup, A_{cup}, by the height of the cup.

9.3 Determine the force (N) applied by the cover, hanger and mass support by multiplying the sum of these masses (kg) by acceleration of gravity (g), where g = 9.81 m/s^2.

9.4 Determine the force (N) applied by each applied mass (kg) by multiplying it by acceleration of gravity (g) where g = 9.81 m/s^2.

9.5 Determine the compressive stress (N/m^2) corresponding to each force (N) by dividing the calculated force by the inside cross-sectional area of the density cup, A_{cup} (m^2).

9.6 For each consolidation step, sum the compressive stress (N/m^2) caused by the cover, hanger and mass support with that caused by the sum of the applied masses. This is σ_i (N/m^2).

9.7 For each consolidation step, determine the volume of material in the density cup (m^3) by subtracting the change in height of the dial indicator (m) from the plug (gauge block) height (m), and multiplying the value by the inside cross-sectional area of the density cup, A_{cup} (m^2).

9.8 For each calculated volume of material in the density cup, V_i (m^3), determine the corresponding bulk density, $(\rho_b)_i$ (kg/m^3) by dividing the mass of material in the density cup, $M_{mat'l}$ (kg) by the volume of material, V_i.

10. Report: Test Data Sheet(s)/Form(s)

10.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.3.

10.2 Record as a minimum the following general information (data):

10.2.1 Requesting agency or client

10.2.2 Identifying number for job or project

10.2.3 Technician name or initials

10.2.4 Date test was run

10.3 Record as a minimum the following sample information (data):

10.3.1 Generic name of powder tested

10.3.2 Chemical name of sample, if known

10.3.3 Specimen moisture, if determined. Record value to nearest 0.1 %. Indicate method used to determine moisture if not Test Method D2216

10.3.4 Temperature of specimen to the nearest 1°C.

10.3.5 Specimen particle size to two significant digits

10.4 Record as a minimum the following test data:

10.4.1 Density cup diameter to three significant digits

10.4.2 Density cup height to three significant digits

10.4.3 Mass of density cup empty (tare) to three significant digits

10.4.4 Mass of cover and hanger to three significant digits

10.4.5 Mass of density cup filled with material to three significant digits

10.4.6 Series of dial indicator readings with corresponding applied masses to three significant digits

10.4.7 Time duration for each applied mass to nearest min.

10.4.8 Calculated compressive stress value for each applied mass to three significant digits

10.4.9 Calculated corresponding bulk densities to three significant digits

10.5 A curve is then generated from the data obtained with this test. The heading of the curve is "Bulk density as a function of compressive stress" with the "x" axis being the compressive stress in kPa (1000 N/m^2) and the "y" axis being the bulk density in kg/m^3. The data is then plotted on a linear graph. As an alternate, the data can be plotted on a log-log graph.

11. Precision and Bias

11.1 *Precision*—Test data on precision is not presented due to the nature of the powder and other bulk solids tested by this standard. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. In addition, it is either not feasible or too costly to produce multiple specimens that have uniform physical properties. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.

11.1.1 Subcommittee D18.24 is seeking any data from the users of this standard that might be used to make a limited statement on precision.

11.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

12. Keywords

12.1 bulk density; bulk solids; compressive stress; powder

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (2008) that may impact the use of this standard.

(1) Reference to Test Method D2216 added.
(2) In a few instances, "sample" replaced by "specimen" or vice-versa.
(3) Section 10 text revised.