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Standard Test Method for Dynamic Testing of Powders Using the Freeman Technology FT4 Powder Rheometer¹

This standard is issued under the fixed designation D8328; 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 method covers the apparatus and procedures for quantifying the dynamic flow properties of a fixed volume of powder or other bulk solid as a function of its resistance to being moved by a specially designed blade within a cylindrical test vessel, where the test specimen has an unconstrained upper surface. This allows for the resistance to motion to be assessed as ‘work done’ or energy measurement.

1.2 The parameters generated during this test are most commonly used to assist with the design and operation of powder processing and transport operations. They can also provide relative classification or comparison of the flow behavior of different powders, or different batches of the same powder, that are subjected to similar stress and flow regimes within their processing equipment.

1.3 The 50 mm apparatus described in this standard is suitable for measuring the properties of powders and other bulk solids with a maximum particle size of 6 mm. It is practicable to test powders which have a small proportion of particles between 6-10 mm, but it is recommended they represent no more than 5 % of the total mass with a normal (Gaussian) size distribution.

1.4 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice [D6026](#).

1.4.1 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 decrease 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 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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1.5 *Units*—The values stated in SI units are to be regarded as standard. No other units of measure are included in this standard. Reporting of test results in units other than SI shall not be regarded as nonconformance with this standard.

1.6 *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.*

1.7 *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

[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

[D6026](#) Practice for Using Significant Digits and Data Records in Geotechnical Data

3. Terminology

3.1 *Definitions*—For definitions of common technical terms used in this standard, refer to Terminology [D653](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Aerated Energy, AE_v , n*—in storing, handling and processing bulk solids using industrial equipment, the flow energy of a powder at a controlled superficial gas velocity of v mm/s traversing upwards through the powder bed.

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.

3.2.1.1 *Discussion*—Calculation for AE_v described in 11.4.2.

3.2.2 *Aeration Ratio, AR_v , n —in storing, handling and processing bulk solids using industrial equipment*, the ratio of flow energy of a powder with zero gas flow to that of the same powder when gas is passed upwards through the powder bed at a controlled superficial gas velocity of v mm/s.

3.2.2.1 *Discussion*—Calculation for AR_v described in 11.4.3.

3.2.3 *Basic Flowability Energy, BFE, n —in storing, handling and processing bulk solids using industrial equipment*, the flow energy of a powder measured during the seventh test cycle of a dynamic test.

3.2.3.1 *Discussion*—Calculation for BFE described in 11.3.2.

3.2.4 *Conditioned Bulk Density, CBD, n —in storing, handling and processing bulk solids using industrial equipment*, the bulk density of a test specimen of powder after conditioning.

3.2.4.1 *Discussion*—Calculation for CBD described in 11.3.3.

3.2.5 *conditioning, v —in storing, handling and processing bulk solids using industrial equipment*, the process of homogenizing the state of consolidation of a powder test specimen by use of a specialized blade attachment.

3.2.6 *Consolidated Energy, CE, n —in storing, handling and processing bulk solids using industrial equipment*, the flow energy of a powder consolidated by controlled tapping.

3.2.6.1 *Discussion*—Calculation for CE described in 11.4.6.

3.2.7 *Consolidation Index, CI, n —in storing, handling and processing bulk solids using industrial equipment*, the ratio of flow energy of a consolidated powder to that of the same powder prior to consolidation.

3.2.7.1 *Discussion*—Calculation for CI described in 11.4.7.

3.2.8 *dynamic test, n —in storing, handling and processing bulk solids using industrial equipment*, a test used to quantify the flow properties of a fixed volume of powder or other bulk solid in motion.

3.2.8.1 *Discussion*—The measurement consists of multiple test cycles during which the powder's resistance to being moved by a specially designed blade is measured as a function of external variables such as introduced gas flow and consolidation.

3.2.9 *flow energy, n —in storing, handling and processing bulk solids using industrial equipment*, the resistance to motion of a specialized blade as it traverses downwards through a precise volume of confined, conditioned powder following a prescribed pathway and represented as an energy.

3.2.9.1 *Discussion*—Calculation for flow energy described in 11.2.

3.2.10 *Flow Rate Index, FRI, n —in storing, handling and processing bulk solids using industrial equipment*, the ratio of flow energy of a powder after a test at one blade tip speed to a test at a different blade tip speed where conditioning is completed in between each test cycle.

3.2.10.1 *Discussion*—Calculation for FRI described in 11.3.4.

3.2.11 *Normalized Aeration Sensitivity, NAS, n —in storing, handling and processing bulk solids using industrial equipment*, the maximum rate of change in flow energy with respect to a controlled, variable, superficial gas flow traversing upwards through the powder bed.

3.2.11.1 *Discussion*—Calculation for NAS described in 11.4.4.

3.2.12 *Pressure Drop, PD_v , n —in storing, handling and processing bulk solids using industrial equipment*, the pressure difference across the powder bed resulting from the passage of a controlled, superficial gas flow traversing the test specimen at v mm/s.

3.2.12.1 *Discussion*—Determination of PD_v described in 11.4.5.

3.2.13 *Specific Energy, SE, n —in storing, handling and processing bulk solids using industrial equipment*, the resistance to motion of a specialized blade traversing upwards through an unconfined powder bed following a prescribed pathway and represented as a flow energy per unit mass (mJ/g).

3.2.13.1 *Discussion*—Calculation for SE described in 11.3.5.

3.2.14 *Stability Index, SI, n —in storing, handling and processing bulk solids using industrial equipment*, the ratio of flow energy of a powder after one test to that after n tests where conditioning is completed between each test cycle.

3.2.14.1 *Discussion*—Calculation for SI described in 11.3.6.

3.2.15 *test cycle, n —in storing, handling and processing bulk solids using industrial equipment*, a section of a dynamic test where the specialized blade completes both a downwards and upwards traverse through a precise volume of conditioned powder following a prescribed pathway.

4. Summary of Test Method

4.1 *Selection of the Appropriate Testing Regime*—The particular stress and flow regimes used to evaluate the flow properties of the test specimen may depend on the reason for generating the data, as outlined in Section 5, and should broadly reflect the stresses that the powder is subjected to in its processing environment.

4.2 *Measurement of Flow Energy*—The specialized blade attachment is used to traverse the test specimen through a prescribed path at a pre-determined speed. The torque and force required to maintain the motion of the blade are recorded with respect to the axial position of the blade at a suitable measurement interval.

4.3 *Measurement of PD_v* —The resistance of the powder bed to the passage of controlled gas flow is recorded by means of a computer-controlled Aeration Control Unit (ACU) as a superficial gas velocity of v mm/s is maintained.

5. Significance and Use

5.1 The test can be used to evaluate flow properties



FIG. 1 FT4 Powder Rheometer (The left hand image shows the instrument with the specialized blade attachment fitted; the right hand image shows a test vessel (50 mm x 160 mL split vessel) (containing a test specimen) and the specialized blade.)

5.1.1 *Classification or comparison of powders*—There are several parameters that can be used to classify powders relative to each other: AE_v , AR_v , BFE, CBD, CE, CI, FRI, NAS, PD_v , SE and SI.

5.1.2 *Sensitivity analysis*—Dynamic tests can be used to evaluate the relative effects of a range of powder properties or environmental parameters, or both, such as (but not limited to) humidity, particle size and size distribution, particle shape and shape distribution, moisture content and temperature.

5.2 *Quality control*—The test can, in some circumstances, be used to assess the flow properties of a feedstock, intermediate or product against pre-determined acceptance criteria.

5.3 *Process design and operation*—The determined parameters can be used to quantify powder behavior in numerous processing environments. The determined parameters may also be used to quantify the effect of changing process parameters on powder flowability, including (but not limited to) shear rate during mixing, water addition during granulation and temperature during drying.

NOTE 1—The quality of the result produced by this test method is dependent on the competence of the 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 test method 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. Additional

guidance on sampling of powders is given in Reference (1).

Practice D3740 was developed for agencies engaged in the testing and/or inspection of soil and rock. As such it is not totally applicable to agencies performing this test method. However, users of this test method should recognize that the framework of Practice D3740 is appropriate for evaluating the quality of an agency performing this practice. Currently there is no known qualifying national authority that inspects agencies that perform this test method.

6. Apparatus

6.1 The FT4 Powder Rheometer is shown in Fig. 1. It is a computer-controlled instrument which simultaneously measures the torque and force required to mobilize a powder contained in a range of vessel types. This allows for the generation of powder characteristics such as dynamic flow properties, including aeration behavior, shear properties, wall friction, permeability and compressibility of test specimens, using a series of spindle-mounted attachments driven by an electric motor located on a carriage, driven by another electric motor, which moves the attachments in the vertical direction.

6.1.1 The force is measured by a force transducer located beneath and fixed to the table that supports the test vessel during the measurement process.

6.1.2 The torque (shear resistance) is evaluated by measuring the moment on the attachment using a torque transducer. Both torque and force are measured with respect to the axial position of the blade within the test vessel.



FIG. 2 Connection of ACU's Gas Output Hose to an Aeration Base

6.1.3 A dry, compressed gas supply is moderated using a computer-controlled ACU (Fig. 2) which also monitors the delivery pressure of the supply. The gas flow rate is controlled using mass flow controllers and the pressure is measured using a pressure transducer.

6.2 The test vessel (50 mm × 160 mL split vessel) to generate parameters BFE, CBD, CE, CI, FRI, SE and SI, is shown in Fig. 3 (left hand image) (assembly described in 7.2). It consists of a base, made from a suitable engineering plastic such as polyoxymethylene (POM), onto which are mounted two borosilicate glass cylinders (50 mm × 85 mL glass cylinder and 50 mm × 160 mL glass cylinder) connected by a POM leveling assembly which allows a precise volume of powder to be obtained for testing. The test vessel is located on the powder rheometer using a POM clamp ring which attaches to a stainless steel clamping device. A POM funnel is also fitted to assist with the filling of the test vessel.

NOTE 2—The assembled test vessel is described as 'x mm × y mL', which indicates the glass cylinder's internal diameter, x, (50 ± 0.04 mm) and the precise volume of the lower section of the test vessel with the base fitted, y.

6.3 The test vessel (50 mm × 260 mL vessel) to generate parameters AE_v, AR_v, NAS and PD_v, is also shown in Fig. 3

(right hand image) (assembly described in 7.3). It consists of a stainless steel aeration base (with a stainless steel, permeable, woven disk and gas input port) onto which is mounted a borosilicate glass cylinder (50 mm × 260 mL glass cylinder). The test vessel is located on the powder rheometer using a POM clamp ring which attaches to a stainless steel clamping device. A POM funnel is also fitted to assist with the filling of the test vessel.

6.4 The specialized blade attachment is shown in Fig. 4. It consists of a stainless steel, twisted blade with a diameter of 48 mm. This attachment is used to condition the test specimen thus generating a repeatable stress condition within the powder and is used to measure flow energy as it traverses the test specimen through a prescribed path at a pre-determined speed. The specialized blade attachment and test vessel are nominally concentric with a blade tip clearance of 1 mm to the test vessel wall. The blade is pitched as a propeller so that different flow regimes are achieved depending on the speed and direction of its rotation and translation upwards or downwards through the powder bed.

NOTE 3—It is practicable to employ test vessels with 25 mL capacity in conjunction with the FT4 Powder Rheometer if the quantity of available test specimen is less than 160 mL. The mode of operation of the 25 mL



FIG. 3 Test Vessels (The left hand image shows the test vessel (50 mm × 160 mL split vessel) for generating parameters BFE, CBD, CE, CI, FRI, SE and SI; the right hand image shows the test vessel (50 mm × 260 mL vessel) for generating parameters AE_v , AR_v , NAS and $PD_{v,r}$)



FIG. 4 Specialized Blade Attachment for Conditioning the Test Specimen and Measuring flow Energy as It Traverses the Test Specimen through a Prescribed Path at a Pre-determined Speed

test vessels is identical to that described herein for the 50 mm × 160 mL split vessel and 50 mm × mL vessel but using a smaller test vessel with a diameter of 25 mm and a 23.5 mm diameter blade. The limit on the

maximum particle size is commensurately reduced to a maximum particle size of 3 mm.



FIG. 5 Components to Assemble the 50 mm x 160 mL Split Vessel (4mm ball-ended hex key not shown)

6.5 A thermometric device and hygrometer are advised to measure temperature and humidity as referenced in 10.4.

6.6 An automated tapping device shall be used as a source of consolidation for the determination of CE and CI.

7. Preparation of Apparatus

7.1 Make sure that the test vessels and specialized blade are undamaged, clean and free from grease and other contaminants (2).

NOTE 4—Since the integrity of the specialized blade attachment is critical to generating accurate and reliable data, it should be handled with care and studied for damage at regular intervals.

7.2 *Assembly of Test Vessel (50 mm x 160 mL split vessel) for the determination of BFE, CBD, CE, CI, FRI, SE and SI*—The following items are required to assemble the test vessel (50 mm x 160 mL split vessel): a 50 mm x 85 mL glass cylinder; a 50 mm x 160 mL glass cylinder; a 50 mm diameter POM base fitted with an O-ring; a 50 mm diameter clamp ring; a 50 mm diameter leveling assembly; a 50 mm diameter funnel and a 4 mm ball-ended hex key. With the exception of the 4 mm ball-ended hex key, these items are shown in Fig. 5.

NOTE 5—A fully detailed assembly procedure is also available (3).

7.2.1 To assemble the test vessel (50 mm x 160 mL split vessel), position the clamp ring approximately 1 mm from the end of the 50 mm x 160 mL glass cylinder (Fig. 6) and loosely fit the clamp ring onto the glass cylinder (Fig. 7). The clamp ring must not project past the end of the glass cylinder; otherwise misalignment may occur. Make sure that the gap in the clamp ring is approximately centralized with the printing on the glass cylinder. Secure the clamp ring using the ball-ended hex key, ensuring that the screw is not over tightened.

7.2.2 Locate the POM base fitted with an O-ring into the glass cylinder adjacent to the clamp ring. Carefully rotate the

base to make sure that the entire circumference is in contact with the glass cylinder (Fig. 8).

7.2.3 Open the leveling assembly and place it on top of the glass cylinder at the opposite end to the clamp ring and base (orientation shown in Fig. 9). Make sure that the gap in the leveling assembly is approximately aligned with the text printed on the glass cylinder.

7.2.4 Carefully invert the glass cylinder, clamp ring, base and leveling assembly and place on the edge of a flat surface (Fig. 10) so that the glass cylinder can be fitted flush with the inner face of the leveling assembly without impediment from the upper part of the leveling assembly and in the correct orientation (3).

7.2.5 Push down gently on both the glass cylinder and the leveling assembly so that they are both flush with the flat surface.

7.2.6 Tighten the leveling assembly with the ball-ended hex key such that the leveling assembly and the glass cylinder are securely located.

7.2.7 Confirm that the glass cylinder and leveling assembly are flush, and check that the leveling assembly operates smoothly.

7.2.8 Close the leveling assembly.

7.2.9 Place the 50 mm x 85 mL glass cylinder into the top half of the leveling assembly and gently rotate the upper glass cylinder to make sure that it is in contact with the glass cylinder below.

7.2.10 Tighten the leveling assembly with the ball-ended hex key such that the leveling assembly and the upper glass cylinder are securely located (Fig. 11).

7.2.11 Place the funnel on top of the assembled test vessel and locate on the FT4 Powder Rheometer ensuring that the platform is free from obstruction and the test vessel is level (Fig. 12).