Designation: D8328 – 24

Standard Test Method for Dynamic Testing of Powders Using the Freeman Technology FT4 Powder Rheometer[1]

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.

1.5 *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.

[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.

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*—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 the following:

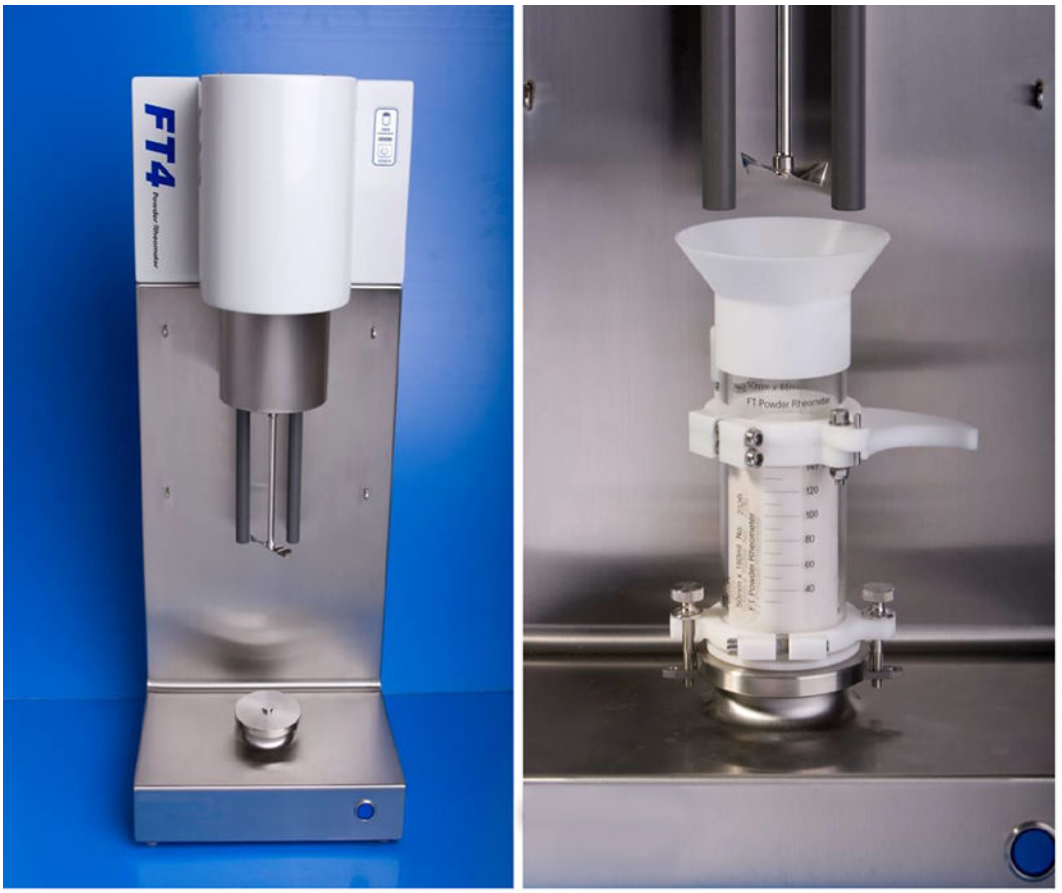


FIG. 1 FT4 Powder Rheometer (The left hand image shows the instrument with the specialized blade attachment fitted; the right hand image shows a test vessel (50 mm × 160 mL split vessel) (containing a test specimen) and the specialized blade.)

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.)

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 × 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 × 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 × 160 mL split vessel): a 50 mm × 85 mL glass cylinder; a 50 mm × 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 × 160 mL split vessel), position the clamp ring approximately 1 mm from the end of the 50 mm × 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 × 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).

FIG. 6 Positioning the Clamp Ring Approximately 1 mm from the End of the 50 mm × 160 mL Glass Cylinder

FIG. 7 Fitting the Clamp Ring to the 50 mm × 160 mL Glass Cylinder with the Ball-Ended Hex Key

7.3 *Assembly of Test Vessel (50 mm × 260 mL vessel) for the determination of AE_v, AR_v, NAS 317 and PD_v*—The following items are required to assemble the test vessel (50 mm × 260 mL vessel): a 50 mm × 260 mL glass cylinder; a 50 mm diameter stainless steel aeration base fitted with an O-ring; a 50 mm diameter clamp ring; 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. 13.

NOTE 6—A fully detailed assembly procedure is also available (**3**).

7.3.1 To assemble the test vessel (50 mm × 260 mL vessel), position the clamp ring approximately 1 mm from the end of the 50 mm × 260 mL glass cylinder (Fig. 14) and loosely fit the clamp ring onto the glass cylinder (Fig. 15). 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

FIG. 8 Fitting the POM Base with O-ring onto the 50 mm × 160 mL Glass Cylinder

ring is approximately aligned with the text printed on the glass cylinder. Secure the clamp ring using the ball-ended hex key.

7.3.2 Locate the stainless steel, aeration 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. 16).

7.3.3 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. 17).

7.3.4 Connect the ACU's gas output hose to the aeration base.

NOTE 7—The assembled test vessel is described as a 50 mm × 260 mL vessel assembly, which indicates the glass cylinder's internal diameter and the precise volume of the test vessel with the base fitted.

8. Calibration

8.1 *Apparatus*—Calibrate and verify the instrument according to the manufacturer's instructions. The manufacturer's recommended verification frequency is 90 days.

NOTE 8—The force and torque transducers located within the instrument are calibrated using proprietary fixtures in conjunction with calibration masses that are supplied with the instrument (**4**). Force should be calibrated within the instrument's performance limits of ±50 N, and torque should be calibrated within the performance limits of ±900 mNm, both to tolerances better than 1.0 %. The Mass Flow Controllers fitted to the ACU and the pressure transducer are calibrated by the manufacturer.

9. Conditioning

9.1 *Preparation of the specimen for the determination of BFE, CBD, FRI, SE and SI*—Add the test specimen to the test vessel and its mass is automatically determined using the instrument's built-in balance. Initiate the automated test program, it runs independently of the operator other than to use a leveling assembly. The test specimen first undergoes conditioning using the specialized blade attachment which reduces variability in packing density introduced during filling of the test vessel or from the material's previous history. Excess powder must then be removed from the test cell by means of a leveling assembly to leave a controlled volume of conditioned powder with a level surface that is ready for dynamic testing. The leveling assembly must then be closed, and the test initiated.

9.2 *Preparation of the specimen for the determination of AE_v, AR_v, NAS and PD_v*—Add the test specimen to the test vessel which is attached to an ACU. Initiate the automated test program, it runs independently of the operator. The test specimen first undergoes two conditioning cycles using the specialized blade attachment to reduce any variability in packing density introduced during filling or from the material's previous history. A controlled gas flow at a superficial velocity of 2 mm/s is supplied by the ACU without operator input, and the test is initiated.

9.3 *Preparation of the specimen for the determination of CE and CI*—Add the test specimen to the test vessel and its mass is automatically determined using the instrument's built-in balance. Initiate the automated test program, it runs independently of the operator other than to use a leveling assembly and to consolidate the test specimen. The test specimen first undergoes conditioning using the specialized blade attachment which reduces any variability in packing density introduced during filling or from the material's previous history. Remove the test vessel from the instrument and consolidate the powder. Reintroduce the test vessel to the instrument and remove excess powder from the test vessel by means of a leveling assembly to leave a controlled volume of consolidated powder with a level surface that is ready for dynamic testing. The leveling assembly must then be closed, and the test initiated.

10. Procedure

10.1 *Measurement procedure for determination of BFE, CBD, FRI, SE and SI parameters:*

10.1.1 Select the appropriate test program from the program library.

NOTE 9—Once selected, the test program displays images of the test vessel and attachment that are advised to commence the test.

NOTE 10—There is an automated test program available which is based on an initial conditioning cycle followed by the removal of the excess powder using the leveling assembly. Through instrument automation this is followed by seven conditioning and test cycles using a blade tip speed of 100 mm/s, then four conditioning and test cycles using blade tip speeds of 100, 70, 40 and 10 mm/s. This program is called a 'stability and variable flow rate' test. The test method can be modified in detail with respect to number of test and conditioning cycles are undertaken.

10.1.2 Securely fasten the assembled 50 mm × 160 mL split vessel (7.2) to the instrument table using the stainless steel clamping assembly (Fig. 12).

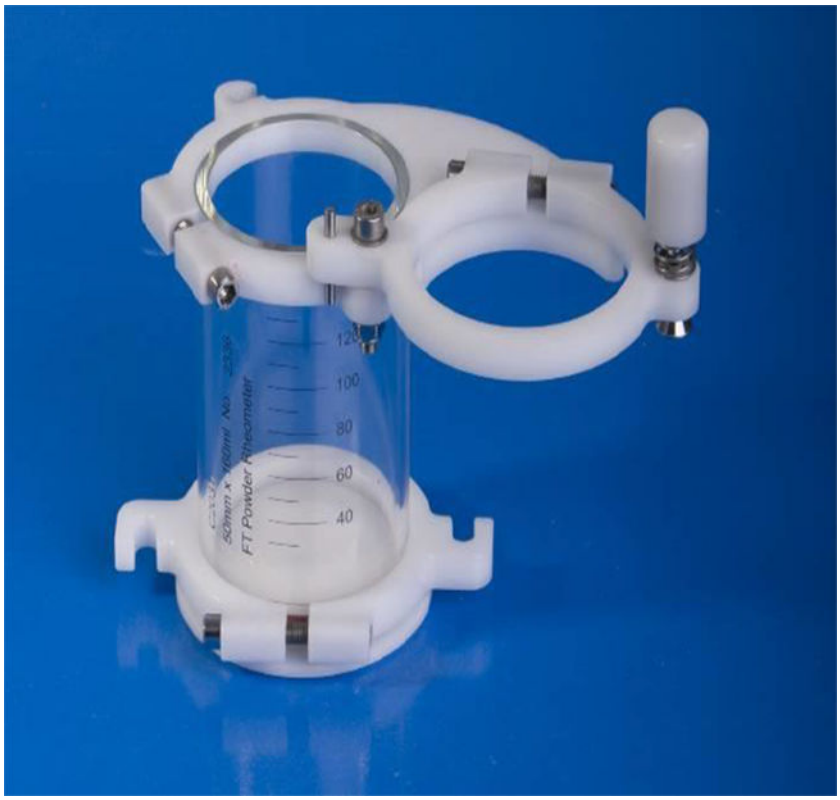


FIG. 9 Positioning the Leveling Assembly with Respect to the 50 mm × 160 mL Glass Cylinder

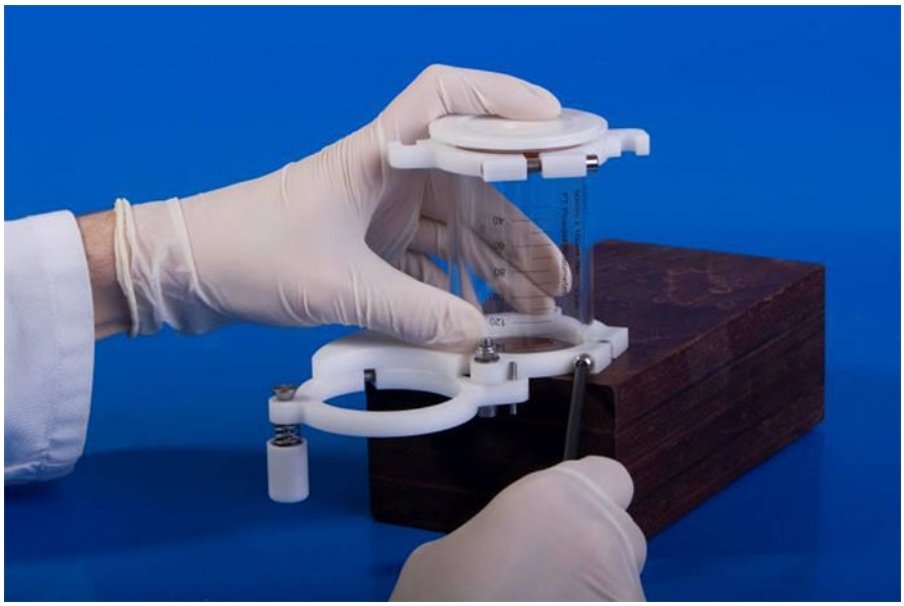


FIG. 10 Fitting the Leveling Assembly onto the 50 mm × 160 mL Glass Cylinder with the Ball-Ended Hex Key

10.1.3 Tare (zero) the mass of the empty test vessel using the built-in balance prior to filling with the test specimen.

10.1.4 Once tared, fill the test vessel with sufficient powder such that, following conditioning, the test specimen does not fall below the split level of the leveling assembly.

NOTE 11—If the level of the powder is below the level of the leveling assembly following conditioning, the test is classified as a failure and re-run with a greater starting volume.

10.1.5 Push the start button on the computer screen to commence the test program.

NOTE 12—This automatically causes the blade to be slowly lowered into the test vessel, after which it performs conditioning by traversing through the powder along a prescribed helical path. When the test program is initiated, the mass of the test specimen is registered within the data file associated with the test.

NOTE 13—Though instrument automation, during conditioning the

FIG. 11 Fitting the Upper Glass Cylinder

blade is moved along a helical pathway at a tip speed of –60 mm/s and a helix angle of +5 degrees (deg) during the downward traverse and at a tip speed of +60 mm/s and a helix angle of –5 deg for the upward traverse. The downward parameters are described schematically in Fig. 18. Tip speed is defined as the velocity of the outer edge of the blade during a specified conditioning or test cycle. The Helix Angle, α, is defined as the angle between the helix of the blade's pathway and the horizontal, Fig. 18. The designation of positive (+) and negative (–) values for the tip speed and helix angle denotes the directionality of the traverse. For the tip speed, positive values denote upward movement and negative values denote downward movement. For the helix angle, positive values denote clockwise rotation and negative values denote anti-clockwise rotation from the perspective of the direction of axial travel.

10.1.6 Once conditioning is complete and the test has been automatically paused, the leveling assembly must be used to separate and remove the excess powder left in the upper section of the test vessel and collect it in a suitable container.

10.1.7 Close the leveling assembly.

NOTE 14—After this has been confirmed, the program continues by first calculating the CBD from the remaining mass of test specimen and known test vessel volume. Then a series of seven pairs of conditioning and test cycles are automatically undertaken to evaluate the BFE, SE and SI of the test specimen.

NOTE 15—Through instrument automation, during a test cycle the blade is moved along a helical pathway at a tip speed of –100 mm/s and a helix angle of –5 deg during the downward traverse of the blade and a tip speed of +60 mm/s and a helix angle of –5 deg for the upward traverse. The downward parameters are described schematically in Fig. 19.

NOTE 16—Once the initial seven test cycles have been completed the program automatically undertakes four additional conditioning and test cycles to evaluate the FRI of the test specimen. For the evaluation of FRI, the specialized blade is moved at a tip speed of –100 mm/s, –70 mm/s, –40 mm/s and –10 mm/s and a helix angle of 5 deg during the downward traverses of the blade, and at a tip speed of 60 mm/s and a helix angle of –5 deg for the upward traverses.

FIG. 12 Test Vessel (50 mm × 160 mL split vessel) Located on Instrument Table Prior to Taring (zeroing) of the Empty Vessel Mass

NOTE 17—Measurements of torque and force are usually automatically taken approximately every 20 ms during the vertical movement of the blade and these measurements are stored in a test file.

10.2 *Measurement Procedure for determination of AE_v, AR_v, NAS and PD_v parameters:*

10.2.1 Select the appropriate test program from the program library.

NOTE 18—Once selected, the test program displays images of the correct test vessel and attachment that are advised to commence the test.

NOTE 19—There is an automated test program available which is based on two initial conditioning cycles followed by a test cycle with no gas input. A superficial gas flow of 2 mm/s is then applied while the blade completes a conditioning cycle to promote uniform distribution of the gas within the test specimen. This is followed by five pairs of conditioning and test cycles using superficial gas velocities (v mm/s) of 2 mm/s, 4 mm/s, 6 mm/s, 8 mm/s and 10 mm/s respectively. This program is called an 'aeration' test. The program can be modified if other aspects of the test specimen's behavior need to be investigated in greater detail.

10.2.2 Securely fasten the assembled 50 mm × 260 mL vessel (7.3) to the instrument table using the stainless steel clamping assembly (Fig. 17).

FIG. 13 Components to Assemble the 50 mm × 260 mL Vessel (4mm ball-ended hex key not shown)

10.2.3 Connect the ACU gas output hose to the aeration base.

NOTE 20—Typically the gas used is air under ambient conditions; however other gases may be used. If other gases are used, the ACU would likely require re-calibration.

10.2.4 Tare (zero) the mass of the empty test vessel using the built-in balance prior to filling with the test specimen.

10.2.5 Once tared, fill the test vessel with 160 mL of powder.

NOTE 21—The equivalent mass for 160 mL can be calculated from the split mass or CBD values obtained in the stability and variable flow rate test.

10.2.6 Push the start button on the computer screen to commence the test program.

NOTE 22—This automatically causes the blade to be slowly lowered into the test vessel, after which it performs two conditioning cycles by traversing through the powder along a prescribed helical path. When the test program is initiated, the mass of the test specimen is registered within the data file associated with the test. During conditioning, the blade is moved along a helical pathway at a tip speed of –60 mm/s and a helix angle of +5 degrees (deg) during the downward traverse of the blade and at a tip speed of +60 mm/s and a helix angle of –5 deg for the upward traverse. The downward parameters are described schematically in Fig. 18. Once conditioning is complete, the program automatically continues to conduct a test cycle to measure the flow energy of the test specimen with no gas input. During a test cycle, the blade is moved along a helical pathway at a tip speed of –100 mm/s and a helix angle of –5 deg during the downward traverse of the blade and a tip speed of +60 mm/s and a helix angle of –5 deg for the upward traverse. The downward parameters are described schematically in Fig. 19.

NOTE 23—The program then automatically continues to conduct a series of five pairs of conditioning and test cycles to evaluate the AE_v, AR_v, NAS and PD_v of the test specimen, these measurements are stored in a test file. During each conditioning and test cycle pair, gas input is present. Increasing superficial gas flow is automatically applied to traverse the powder bed during each pair, the gas flow is increased by an increment of 2 mm/s after each completed pair, reaching a maximum of 10 mm/s during the automated test program.

10.3 *Measurement Procedure for determination of CE and CI parameters:*

10.3.1 Select the appropriate test program from the program library.

NOTE 24—Once selected, the test program displays images of the correct test vessel and attachment that are advised to commence the test.

NOTE 25—There is an automated test program available which is based on an initial conditioning cycle followed by consolidation of the test specimen. Excess powder is removed using the leveling assembly, then a single test cycle is conducted. This program is called a 'consolidation' test. The test method can be modified in detail with respect to number of test and conditioning cycles undertaken.

10.3.2 Securely fasten the assembled 50 mm × 160 mL split vessel (7.2) to the instrument table using the stainless steel clamping assembly (Fig. 12).

10.3.3 Tare (zero) the mass of the empty test vessel using the built-in balance prior to filling with the test specimen.

10.3.4 Once tared, fill the test vessel with sufficient powder such that, following conditioning and consolidation stages, the test specimen does not fall below the split level of the leveling assembly.

NOTE 26—If the level of the powder is below the level of the leveling assembly following conditioning or consolidation stages, or both, the test is classified as a failure and re-run with a greater starting volume.

10.3.5 Push the start button on the computer screen to commence the test program.

NOTE 27—This automatically causes the blade to be slowly lowered into the test vessel after which it performs conditioning by traversing through the test specimen along a prescribed helical path. During conditioning, the blade is moved along a helical pathway at a tip speed of –60 mm/s and a helix angle of +5 degrees (deg) during the downward traverse of the blade and at a tip speed of +60 mm/s and a helix angle of –5 deg for the upward traverse. The downward parameters are described schematically in Fig. 18.

NOTE 28—When the test program is initiated, the mass of the test specimen is registered within the data file associated with the test.

10.3.6 Once conditioning is complete and when prompted, remove the filled test vessel (50 mm × 160 mL split vessel) from the powder rheometer by loosening the clamping assembly.

10.3.7 Consolidate the test specimen within the test vessel by applying 50 taps using an automated tapping device.

NOTE 29—Alternative methods of consolidation, by vibration or application of a load, may also be used. The test specimen's response would likely be dependent on the method of consolidation used, as such the consolidation method should be kept consistent when different specimens are being compared.

10.3.8 Return the filled test vessel to the powder rheometer and securely fasten the test vessel containing the test specimen to the instrument table using the clamping assembly.

FIG. 14 Positioning the Clamp Ring Approximately 1 mm from the End of the 50 mm × 260 mL Glass Cylinder

FIG. 15 Fitting the Clamp Ring to the 50 mm × 260 mL Glass Cylinder with the Ball-Ended Hex Key

FIG. 16 Fitting the Aeration Base with O-ring onto the 50 mm × 260 mL Glass Cylinder

10.3.9 Use the leveling assembly to separate and remove the excess powder left in the upper section of the test vessel and collect it in a suitable container.

10.3.10 Close the leveling assembly.

NOTE 30—After this has been confirmed, the program automatically continues by undertaking one test cycle to evaluate flow energy of the consolidated powder, represented as CE. During a test cycle, the blade is moved along a helical pathway at a tip speed of –100 mm/s and a helix angle of –5 deg during the downward traverse of the blade and a tip speed of +60 mm/s and a helix angle of –5 deg for the upward traverse. The downward parameters are described schematically in Fig. 19. Measurements of torque and force are usually taken approximately every 20 ms during the vertical movement of the blade and these measurements are stored in a test file.

10.4 Record ambient temperature and humidity using appropriate equipment.

FIG. 17 Test Vessel (50 mm × 260 mL vessel) Located on Instrument Table Prior to Taring (zeroing) of the Empty Test Vessel Mass (Gas output hose connected to the aeration base.)

11. Calculation and Interpretation of Results

11.1 Record the force, torque and position values generated during the test program.

NOTE 31—This data, which is stored in a computer file, can be evaluated using the software program Data Analysis, which is provided with the FT4 Powder Rheometer.

11.2 Calculate the energy required for each traverse of the powder bed, moving the specialized blade from the top of the powder bed (H1) to the bottom (H2) along the prescribed pathway.

NOTE 32—These parameters are described schematically in Fig. 20.

NOTE 33—The force data recorded in 11.1 is represented in Fig. 21 in relation to:

Blade radius, R. R = 0.024 m for the specialized blade attachment,

Vertical distance moved by the blade during one complete revolution, L,

The helical path angle, α, along which the blade moves.

11.2.1 Rotational work done is:

$$W_R = 2.\pi.T \tag{1}$$

where:

T = torque acting on blade, N·m,
W_R = rotational work done per revolution, N·m, and
'.' = denotes multiplication.

11.2.2 Axial distance moved per revolution:

$$L = 2.\pi.R.\tan\alpha \tag{2}$$

where:

L = axial distance moved per revolution, m,
α = helical path angle, radians, and
R = blade radius, m.

11.2.3 Axial work done = axial force × axial distance moved is:

$$W_A = F.L = F.2.\pi.R.\tan\alpha \tag{3}$$

where:

W_A = axial work done, N·m and
F = axial force on blade, N.

11.2.4 Total work done per blade revolution is:

$$W_T = W_R + W_A = (2.\ \pi\ .\ T) + (F\ .2.\ \pi\ .\ R\ .\ \tan\ \alpha) \tag{4}$$

where:

W_T = total work done per blade revolution, N·m.

11.2.5 Therefore, work or energy gradient is:

$$\frac{dE}{dH} = \frac{T}{R.\tan\alpha} + F \tag{5}$$

where:

dE/dH = work or energy gradient, N.

11.2.6 If, for each increment or decrement of vertical displacement, dH, the energy consumed is dE, then:

$$\text{Energy Consumed}, dE = \text{Energy Gradient} \times dH \tag{6}$$

11.2.7 Therefore:

$$dE = \left[\left(\frac{T}{r.\tan\alpha}\right) + F\right].dH \tag{7}$$

where:

dE = energy consumed per increment or decrement of vertical displacement, N·m and
dH = increment or decrement of vertical displacement, m

11.2.8 The flow energy consumed during the traverse is the sum of all the dE values, that is, the area under the energy gradient curve as demonstrated in Fig. 22. It is therefore the integral of the energy gradient versus height plot, that is, the sum of all the energies calculated between each height decrement.

$$FE = \Sigma\left[\left(\frac{T}{R.\tan\alpha} + F\right).dH\right] \tag{8}$$

where:

FE = flow energy, N·m.

11.3 *Derivation of BFE, CBD, FRI, SE and SI parameters and interpretation of data:*

11.3.1 The typical method for graphical presentation of the flow energy results from this analysis is shown in Fig. 23.

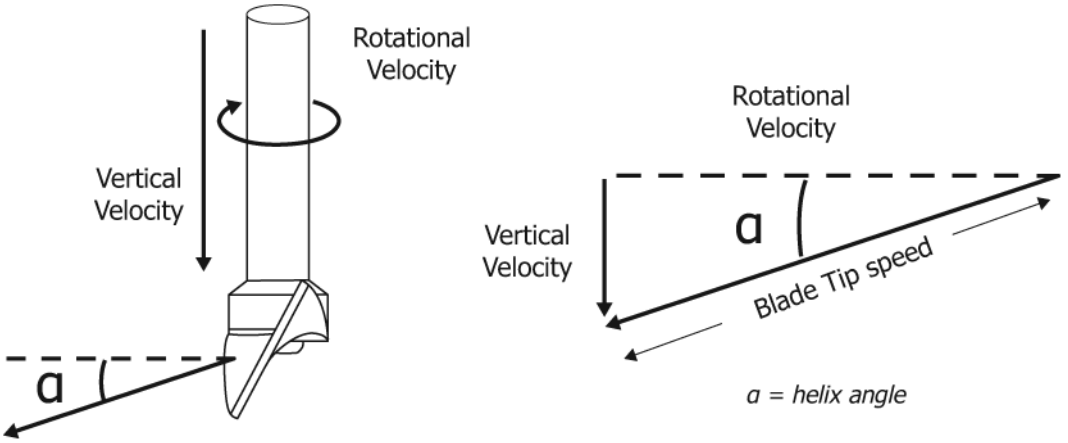


FIG. 18 Motion of the Specialized Blade During the Downward Traverse of a Conditioning Cycle

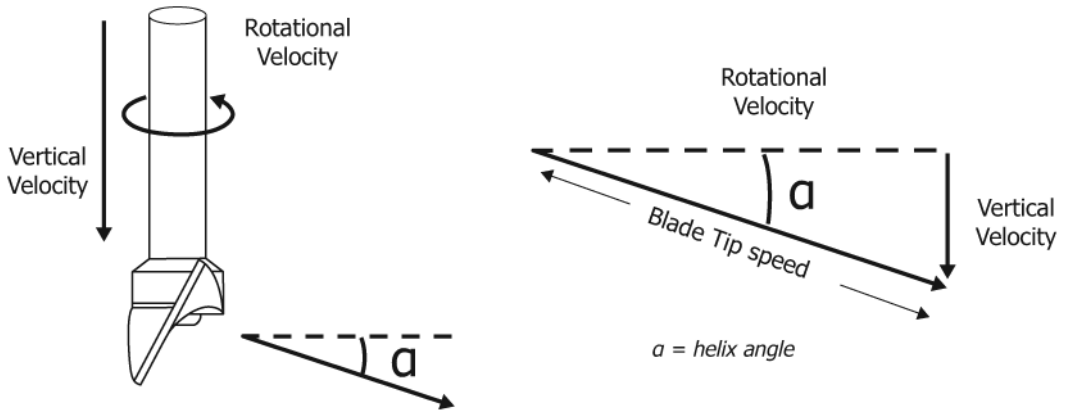


FIG. 19 Motion of the Specialized Blade During the Downward Traverse of a Test Cycle

11.3.2 *Calculation of Basic Flowability Energy, BFE*—Calculate BFE (defined in 3.2.3) using the following equation:

$$BFE = FE_7 \tag{9}$$

where:

BFE = Basic Flowability Energy, mJ and
FE_7 = flow energy from the seventh test cycle, mJ.

NOTE 34—Additional information is given in Reference (**5**).

11.3.3 *Calculation of Conditioned Bulk Density, CBD*—Calculate CBD (defined in 3.2.4) using the following equation:

$$CBD = \left(\frac{m_{Conditioned}}{v} \right) \tag{10}$$

where:

CBD = Conditioned Bulk Density, g/mL,
$m_{Conditioned}$ = mass of conditioned powder specimen, g, and
v = specimen volume, mL.

NOTE 35—The term 'density' describes the relationship between mass and volume. For powders, it is an important bulk property as it can influence process performance, with uncontrolled variation in density leading to many process problems, such as mass variability in volumetric feeding systems. Bulk density is dependent on many physical properties, such as, but not limited to, true density, particle size and distribution, particle shape, particle surface texture and cohesive/adhesive forces. The ways in which these properties interact controls the relationship between particle volume and void space.

NOTE 36—External variables such as the level of consolidation can change the gas content within the powder bulk and therefore have a large impact on the test specimen's density. To improve density measurement reproducibility, the measurements should be conducted in relation to a known and controlled packing state. The conditioning used in the dynamic test allows such a controlled packing state to be generated. After conditioning, localized stress and excess gas is reduced. As the conditioning process is automated, it is conducted independently of the operator and results in a more reproducible packing state.

11.3.4 *Calculation of Flow Rate Index, FRI*—Calculate FRI (defined in 3.2.10) using the following equation:

$$FRI = \frac{FE_{11}}{FE_8} \tag{11}$$

where:

FRI = Flow Rate Index,
FE_8 = flow energy from the eighth test cycle, mJ, and
FE_{11} = flow energy from the eleventh test cycle, mJ.

NOTE 37—Additional information is given in Reference (**6**).

NOTE 38—The FRI is a function of shear rate sensitivity and typically correlates well with the process performance of powders that are subjected to variable shear rates, for example during mixing and conveying. Some typical flow energy profiles as a function of flow rate are shown in Fig. 24.

11.3.5 *Calculation of Specific Energy, SE*—Calculate SE (defined in 3.2.13) using the following equation:

$$SE = \frac{\left(FE_{U6} + FE_{U7}\right) \div 2}{m_{Conditioned}} \tag{12}$$

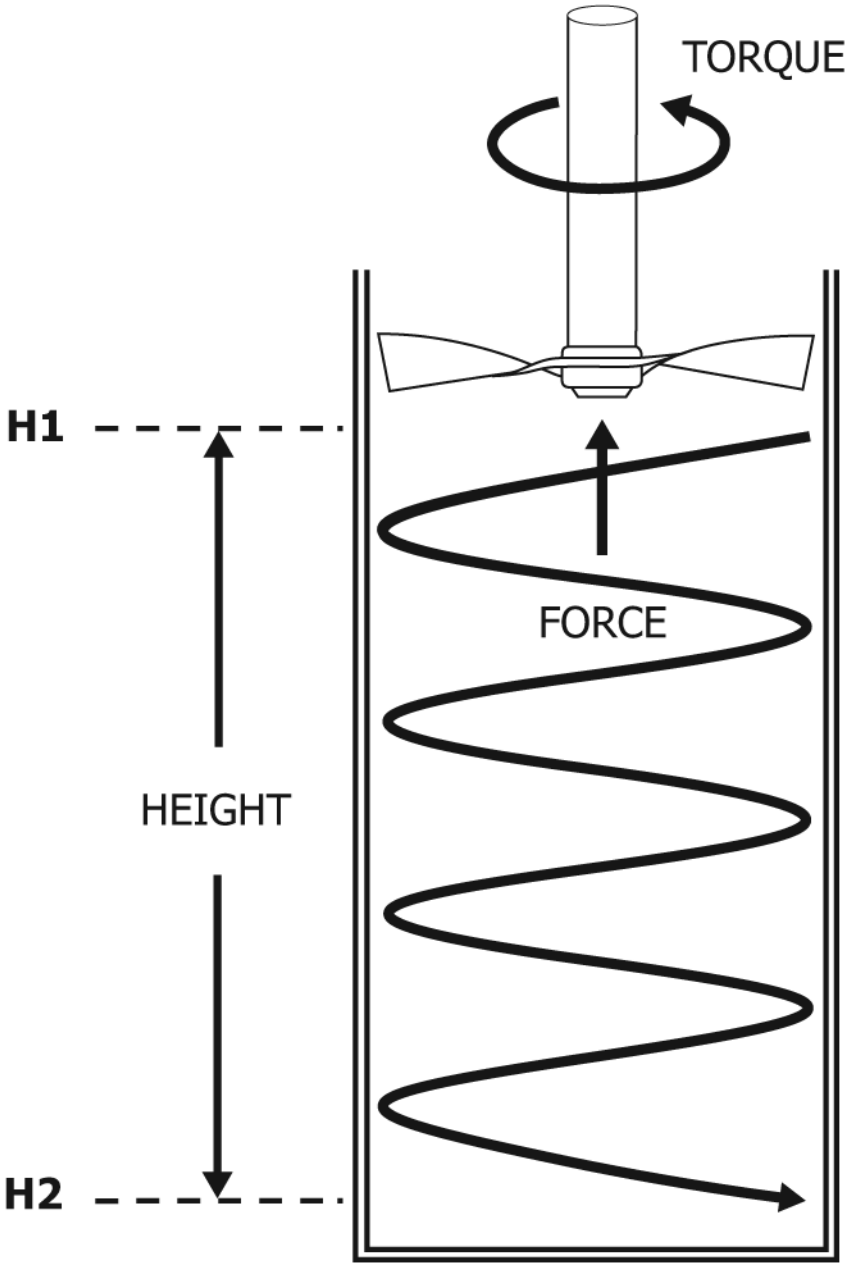


FIG. 20 Schematic of the Motion of the Specialized Blade in a Dynamic Test

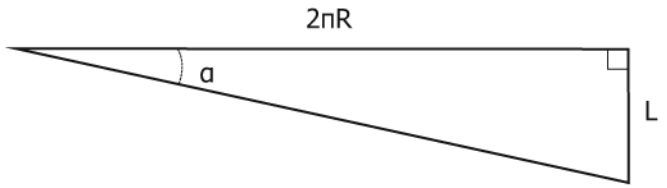


FIG. 21 Definitions Used in the Calculation of Force During a Test

where:

SE = Specific Energy, mJ/g,
FE_{U6} = upwards flow energy from the sixth test cycle, mJ, and
FE_{U7} = upwards flow energy from the seventh test cycle, mJ.

NOTE 39—Additional information is given in Table 1 and Reference (7).

NOTE 40—Unlike the BFE measurement where bulk compressibility/stiffness can significantly contribute to the measured flow energy, the SE mostly relates to cohesion and other physical properties contributing to the mechanical inter-locking of particles. Therefore, SE often correlates well with the flow performance of a powder when in a low stress environment and particularly when being fed gravimetrically, such as in die filling or low shear blending.

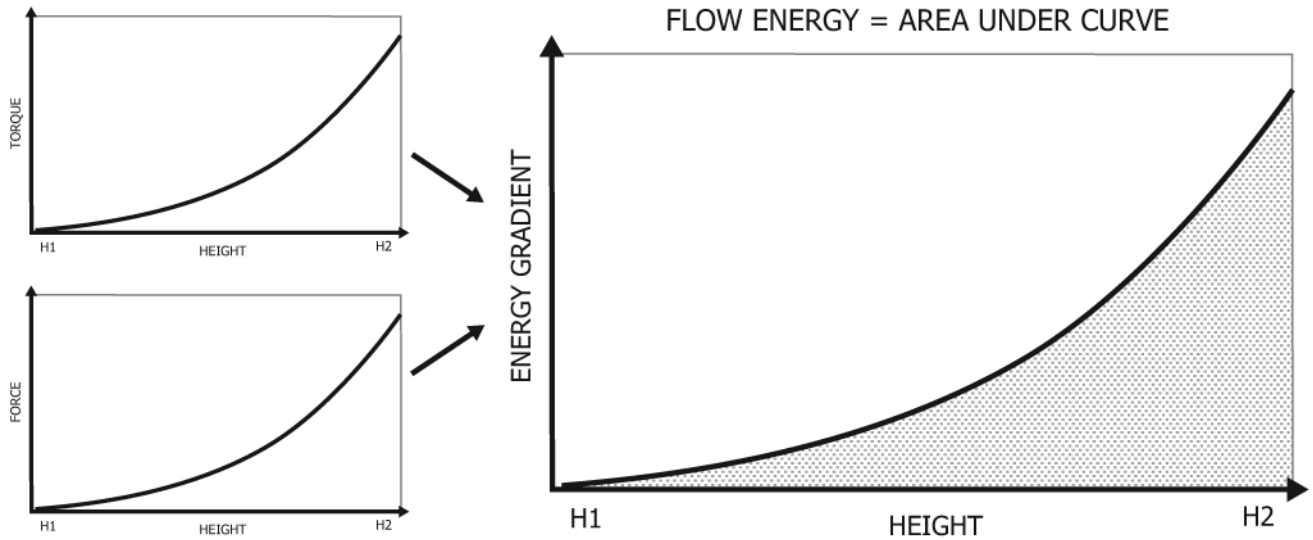


FIG. 22 Energy Gradient is Calculated Directly from the Measurements of Torque and Force

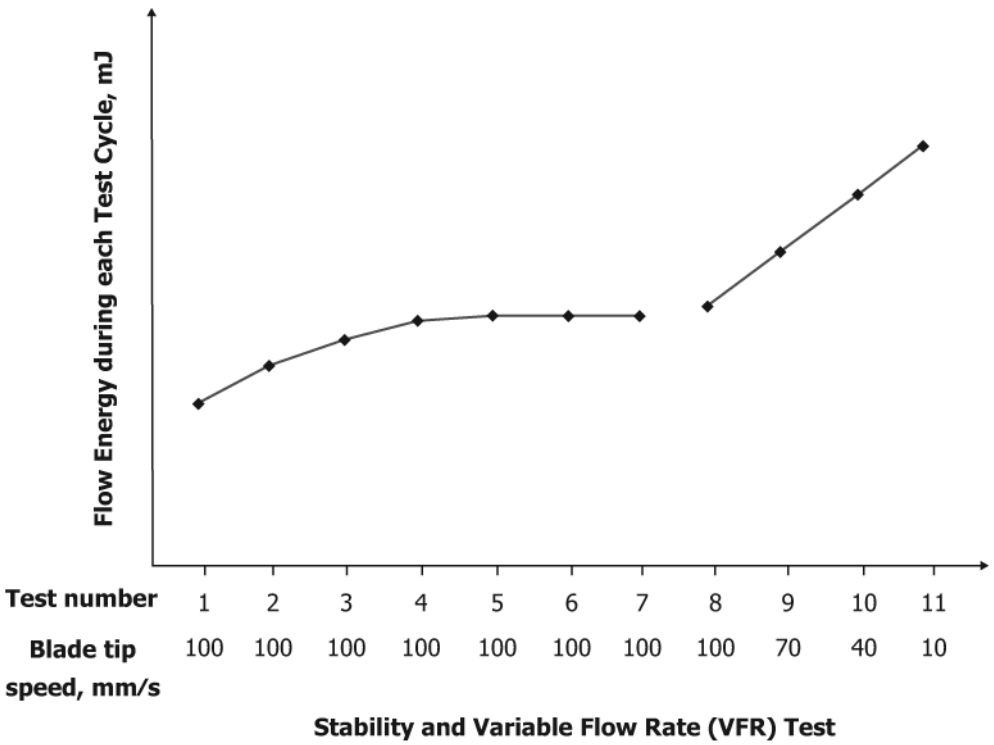


FIG. 23 Typical Method of Presenting Stability and Variable Flow Rate Test Data

11.3.6 *Calculation of Stability Index, SI*—Calculate SI (defined in 3.2.14) using the following equation:

$$SI = \frac{FE_7}{FE_1} \quad (13)$$

where:

SI = stability index,
FE_1 = flow energy from the 1st test cycle, mJ, and
FE_7 = flow energy from the 7th test cycle, mJ.

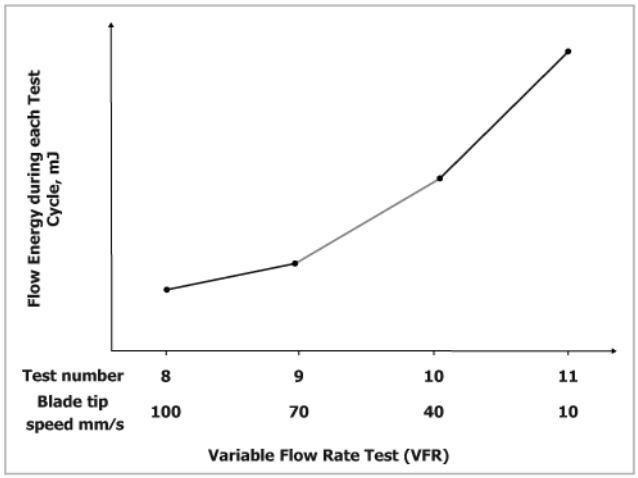
NOTE 41—Additional information is given in Reference (**8**).

NOTE 42—As detailed in Fig. 25, an SI value of approximately 1.0 suggests that the test specimen is physically stable when subjected to repeat testing. Small changes ($0.9 < SI < 1.1$) are normal for most powders and are to be expected. SI values outside of this range indicate that the test specimen may be changed a result of being tested and is likely to be more sensitive to being handled or processed, or both. If the test specimen shows significant instability or does not reach a steady value of flow energy after seven pairs of conditioning and test cycles, or both, it is recommended that the cause of the instability be investigated further.

11.4 *Derivation of AE_v, AR_v, NAS and PD_v parameters and interpretation of data:*

11.4.1 The typical method for graphical presentation of the flow energy results as a function of aeration is shown in Fig. 26.

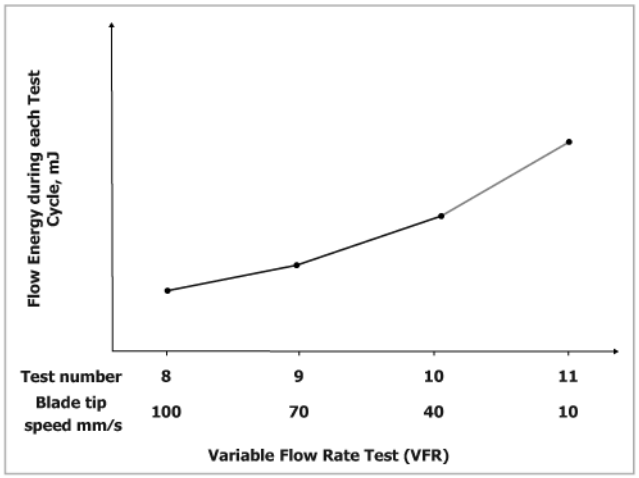
11.4.2 *Calculation of Aerated Energy, AE_v*—Calculate AE_v (defined in 3.2.1) using the following equation:



High flow rate sensitivity

FRI > 3.0

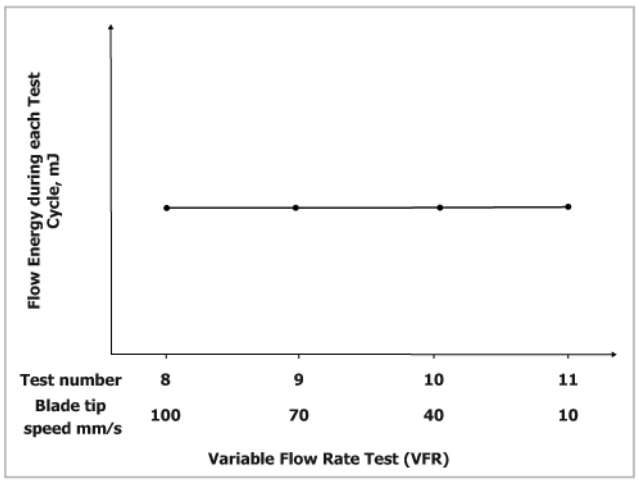
Typically exhibited by highly cohesive powders



Average flow rate sensitivity

1.5 < FRI < 3.0

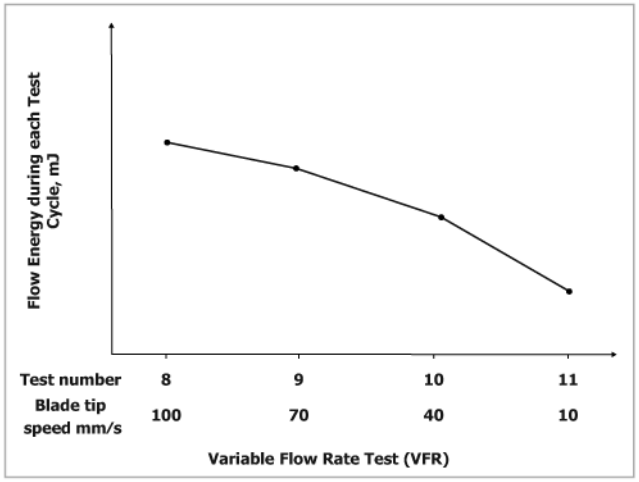
The majority of powders generate values within this range



Flow rate insensitive

FRI ≈ 1.0

Typically powders with large particle size or surface treatments



Pseudoplastic or Newtonian Flow Rate

FRI < 1.0

Typically observed for powders containing flow enhancers

FIG. 24 Typical Flow Energy Profiles as a Function of Flow Rate

TABLE 1 Relationship Between SE and Cohesion

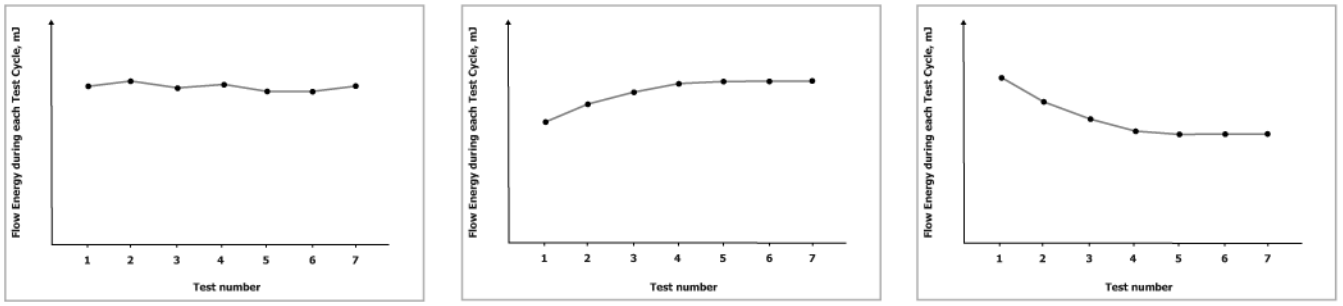
Specific Energy, mJ/g	Flow Properties
SE < 5	Low cohesion
5 < SE < 10	Moderate cohesion
SE > 10	High cohesion

$$AE_v = FE_{v\ mm/s} \tag{14}$$

where:

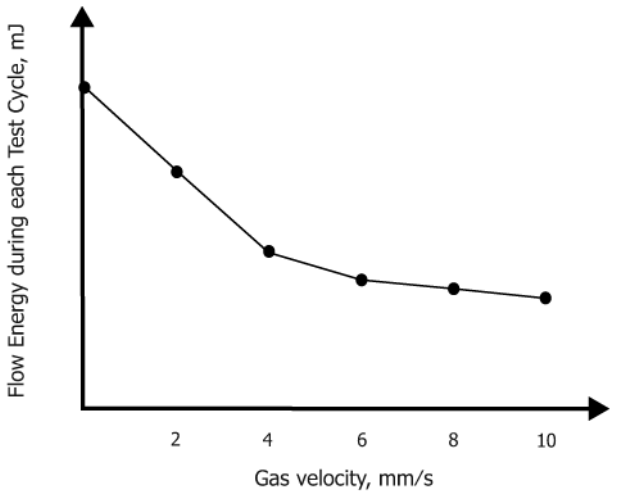
AE_v = Aerated Energy, mJ and
$FE_{v\ mm/s}$ = Flow Energy at gas velocity v, mJ.

NOTE 43—Additional information is given in Reference (**9**).



Stable	Unstable	Unstable
SI ≈ 1.0	SI > 1.0	SI < 1.0
Robust material, not affected by being made to flow.	Powder affected by being made to flow. Possible causes are: - De-aeration, Agglomeration, Segregation, Moisture uptake, Electrostatic charge	Powder affected by being made to flow. Possible causes are: - Attrition, De-agglomeration, Over blending of an additive, Coating of vessel and blade by additive

FIG. 25 Typical Flow Energy Profiles as a Function of Repeat Testing



Aeration Test

FIG. 26 Typical Method of Presenting Flow Energy Test Data as a Function of Aeration

NOTE 44—AE_v is dependent on the powder's response to the introduction of gas, cohesion and other physical properties such as particle size, shape and density. During AE_v measurement, the test specimen is subjected to flow regimes at relatively low stresses. Therefore, the parameters generated, including AE_v typically correlate well with flow performance in low stress or aerated states, for example in fluid bed reactors/dryers or during pneumatic conveyance. Example flow energy profiles as a function of aeration are displayed in Fig. 27.

11.4.3 *Calculation of Aeration Ratio, AR_v*—Calculate AR_v (defined in 3.2.2) using the following equation:

$$AR_v = \frac{AE_0}{AE_v} \quad (15)$$

where:

AR_v = Aeration Ratio and

AE_0 = Aerated Energy at gas velocity 0 mm/s, mJ.

NOTE 45—An interpretation of typical AR_v ranges is given in Table 2 and additional information is given in Reference (**9**).

11.4.4 *Calculation of Normalized Aeration Sensitivity, NAS*—Calculate NAS (defined in 3.2.11) from the gradient of normalized AE_v plotted against superficial gas velocity. AE_v is normalized against the flow energy of the non-aerated powder (that is, superficial gas velocity of 0 mm/s). Typically, NAS values from sections of the graph with the highest gradient are reported.

11.4.4.1 NAS is calculated using the following equation:

$$NAS = \frac{\delta(AE_v \div AE_0)}{\delta v} \quad (16)$$

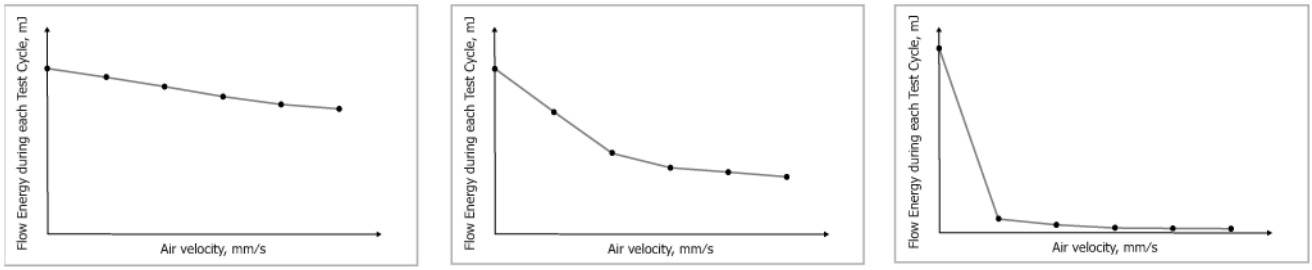


FIG. 27 Typical Flow Energy Profiles as a Function of Aeration, Demonstrating Different Sensitivities to Superficial Gas Velocity

TABLE 2 Relationship between AR_v, Sensitivity to Aeration, and Powder Properties

$AR_v = 1$	$2 < AR_v < 20$	$AR_v >> 20$
Not sensitive to aeration	Average sensitivity to aeration	Very sensitive to aeration and likely to have reached full fluidization
Usually very cohesive powders or those containing high levels of binder	Majority of powders generate values within this range	Usually powders with very low cohesive strength, such as toners, powder coatings

where:

NAS = Normalized Aeration Sensitivity, s/mm,
$\delta(AE_v \div AE_0)$ = change in normalized AE_v (normalized against the flow energy of the non-aerated powder, AE_0), and
δv = change in superficial gas velocity (described in Note 19), mm/s.

NOTE 46—The NAS relates to powder cohesion and other physical properties, such as particle size, shape, density and porosity, and it can be used to improve understanding of how a powder mixes with a gas stream, for example during hopper discharge or mixing applications. Higher NAS values indicate that the powder is more sensitive to the introduction of gas.

11.4.5 *Determination of Pressure Drop (across the powder bed), PD_v*—PD_v (defined in 3.2.12) is directly measured during the aeration test.

NOTE 47—PD_v is typically dependent on the particle size and distribution, particle shape, surface texture and porosity of the powder bed, Fig. 28 displays typical PD_v profiles.

11.4.6 *Determination of Consolidated Energy, CE*—CE (defined in 3.2.6) is described using the following equation demonstrating that CE is flow energy as a function of consolidation.

$$CE = FE_{Consolidated} \quad (17)$$

where:

CE = Consolidated Energy, mJ and
$FE_{Consolidated}$ = Flow Energy of a consolidated powder specimen, mJ.

NOTE 48—Additional information is given in Note 30 and Reference (**10**).

NOTE 49—As CE is the flow energy of consolidated powder, this parameter relates to the ability of powder particles to overcome interparticular forces and rearrange when subjected to external stimuli. Typically, this results in denser particle packing; however in some circumstances, vibrational forces can result in powder bed expansion. In addition to cohesion, CE also relates to physical properties such as particle size, shape and density. This parameter typically correlates well with flow performance in intermediate to high stress environments and is useful for understanding powder behavior following extended periods of transport or storage.

11.4.7 *Calculation of Consolidation Index, CI*—Calculate CI (defined in 3.2.7) using the following equation:

$$CI = \frac{CE}{BFE} \quad (18)$$

where:

CI = Consolidated Index, CI.

NOTE 50—Additional information is given in Reference (**10**).

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

12.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.4 and in Practice D6026.

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

12.2.1 Requesting agency or client or identifying number for job or project, or both.

12.2.2 Technician name or initials.

12.2.3 Date test was run.

12.3 Record the following test specific information (data):

12.3.1 Generic name of test specimen.

12.3.2 Chemical name of test specimen, if known.

12.3.3 Test specimen water content, if determined. Record value to nearest 0.1 %. Indicate method used to determine moisture if not Test Method D2216.

12.3.4 Temperature of environment where the test specimen was tested to the nearest 1 °C.

12.3.5 Humidity of environment where the test specimen was tested to the nearest 1 % RH.

12.4 Provide (in plot form) the flow energy as a function of test number and tip speed. Also provide the BFE, CBD, FRI, SE and SI, results to three significant figures.

12.5 Provide (in plot form) the AE as a function of superficial gas velocity. Also provide AE_v, AR_v, NAS and PD_v results to three significant figures.

12.6 Provide the CE and CI results to three significant figures.

12.6.1 Provide the method and duration of consolidation used to generate CE and CI values.

13. Precision and Bias

13.1 *Precision*—It is not possible to specify the precision of the procedure in Test Method for the Dynamic Testing of

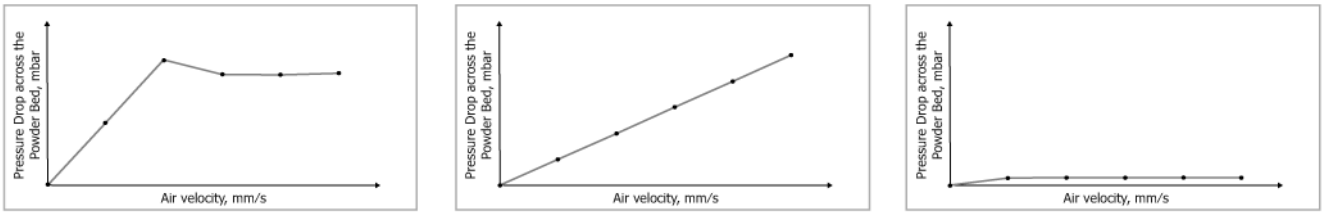


FIG. 28 Examples of Pressure Drop Profiles with Respect to Increasing Superficial Gas Velocity

Powders using the Freeman Technology FT4 Powder Rheometer due to the nature of the granular and powder materials tested by this test method. 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.

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

13.2 *Bias*—No information can be presented on the bias of the procedure in Test Method for the Dynamic Testing of Powders using the Freeman Technology FT4 Powder Rheometer because no material having an accepted reference value is available.

14. Keywords

14.1 aerated energy; aeration ratio; basic flowability energy; conditioned bulk density; consolidated energy; consolidation index; dynamic testing; flow rate index; Freeman Technology; FT4 powder rheometer; normalized aeration sensitivity; powder; pressure drop; specific energy; stability index

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(10) W7023 – Tapped Consolidation, Freeman Technology Support Document, Issue D, 2019.