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.

INTERNATIONAL

Designation: D8327 – 24

Standard Test Method for Measuring the Permeability of Powders as a Function of Consolidation Using the Freeman Technology FT4 Powder Rheometer[1]

This standard is issued under the fixed designation D8327; 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 pressure drop and deriving the gas permeability of a powder bed with respect to consolidation stress or superficial gas velocity, or both, using the FT4 Powder Rheometer.

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 can be used to measure the properties of powders and other bulk solids with a maximum particle size of 6 mm. It is practicable to test powders that have a small proportion of particles of 6-10 mm, but it is recommended they represent no more than 5 % of the total mass in samples 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 *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.

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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.

Current edition approved Jan. 1, 2024. Published March 2024. DOI: 10.1520/D8327-24.

[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.

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3.2.2 *gas permeability, n—in storing, handling and processing bulk solids using industrial equipment*, the relationship between superficial gas velocity and pressure drop in a packed powder bed under laminar flow conditions.

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

3.2.3.1 *Discussion*—Calculation for PD_q described in Section 11.

4. Summary of Test Method

4.1 *Selection of the Appropriate Testing Regime*—The particular consolidating stress level or levels used to evaluate the PD_q and gas permeability of the powder 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 PD_q and Calculation of Gas Permeability*—In order to measure PD_q and calculate the gas permeability of the test specimen, a regulated superficial flow of gas is maintained at the base of the test vessel by means of a computer-controlled Aeration Control Unit (ACU), which is supplied with a compressed gas feed of between 300 and 900 kPa. The gas traverses the powder bed and passes through a vented piston. The powder's gas permeability is determined by measuring the gas pressure differential across the powder bed at different consolidating stress levels or superficial gas velocities, or both.

5. Significance and Use

5.1 The test method can be used to evaluate the following:

5.1.1 *Classification or Comparison of Powders*—Both the measured PD_q and calculated gas permeability can be used to classify powders relative to each other.

5.1.2 *Sensitivity Analysis*—The test 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.

5.1.3 *Quality Control*—The test can be used to assess the PD_q and gas permeability of a feedstock, intermediate or product against pre-determined acceptance criteria.

5.1.4 *Process Design and Operation*—The determined parameters can be used to quantify powder behavior in numerous processing environments. The ability of a powder to discharge consistently from a storage vessel, through pipes or down chutes is primarily dependent on its flow properties, interaction with the material of construction and the geometry of the equipment (**1 and 2**)[3]. However, design methodologies assume that there is a gas interchange between the top and bottom of the stored powder that allows consistent flow through the outlet. Gas permeability is a controlling factor in discharge rates (**1 and 2**). Powders with low gas permeability may exhibit reduced or intermittent/pulsatile flow, or both, that cannot be predicted from only considering shear and dynamic flow properties. This could lead to poor filling or discharge of cavities/bags/vessels and thus result in poor weight uniformity or slow discharge. Furthermore, a powder with low gas permeability may be more likely to have compromised compression properties in tableting operations due to increased entrained gas after the filling stage and a reduced ability to release this gas during compression.

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 (**3**).

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[4]

6.1 The FT4 Powder Rheometer is shown in Fig. 1. It is a computer-controlled instrument that simultaneously measures the torque and force required to mobilize a powder contained in a range of vessel types. This allows for the quantification of powder characteristics such as dynamic flow properties, including aeration behavior, shear, wall friction, permeability and compressibility properties 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.

6.1.3 The vertical position of the carriage is measured by a linear potentiometer.

6.1.4 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 × 85 mL split vessel) is shown in Fig. 3. It consists of a stainless steel aeration base (with a stainless steel, permeable, multi-layer sintered mesh disk with

[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[4] The sole source of supply of the apparatus known to the committee at this time is Freeman Technology Ltd, 1 Miller Court, Severn Drive, Tewkesbury, Gloucestershire, GL20 8DN, United Kingdom. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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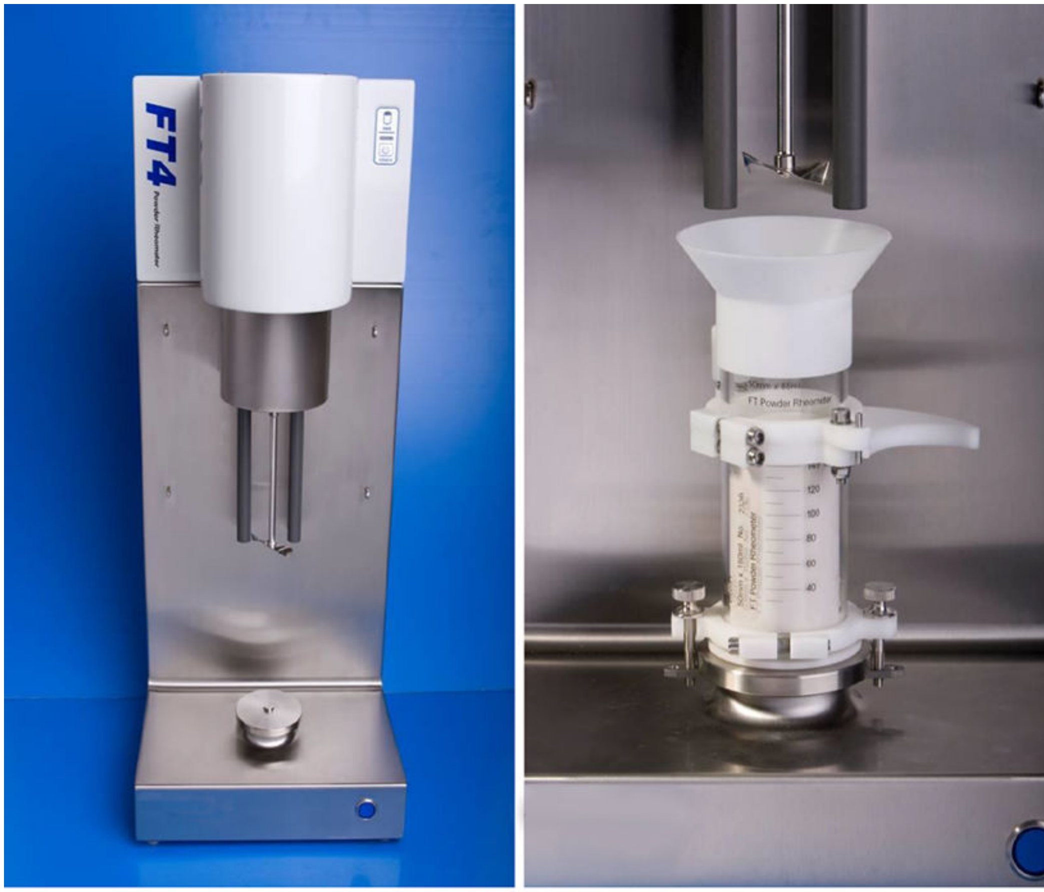

FIG. 1 FT4 Powder Rheometer (The left hand image shows the instrument with the vented piston attachment fitted; the right hand image shows the test vessel with the specialized blade attachment.)

a filtration grade of 60 µm and gas output port), onto which are mounted two borosilicate glass cylinders (both 50 mm × 85 mL) connected by a polyoxymethylene (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 attached 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 specialized blade attachment is shown in Fig. 4. It consists of a stainless steel, twisted blade that is used to condition the test specimen thus generating a repeatable stress condition within the powder as it traverses the test specimen through a prescribed path at a pre-determined speed.

6.3.1 During a conditioning cycle, the blade is moved at a tip speed of –60 mm/s and a helix angle of +5 degree (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. 5.

6.4 The vented piston attachment, shown in Fig. 4, consists of a cylindrical, hollow aluminum shell body and a stainless steel, multi-layer sintered mesh disk with a filtration grade of 60 µm that is presented to the surface of a test specimen and can simultaneously be used to compress the specimen while allowing gas to pass through its woven metal face with minimal resistance. The vented piston compresses the test specimen to achieve the desired consolidating normal stress.

NOTE 3—It is practicable to employ test vessels with 10 mL capacity in conjunction with the FT4 Powder Rheometer if the quantity of available test specimen is less than 85 mL. The mode of operation of the 10 mL test vessels is identical to that described herein for the 50 mm × 85 mL split vessel but using a smaller test vessel with a diameter of 25 mm, a 23.5 mm diameter blade and 24 mm diameter vented piston. The limit on the maximum particle size is commensurately reduced to a maximum particle size of 3 mm.

6.5 A thermometric device and hygrometer are advised to measure temperature and humidity as referenced in 10.1.12.

7. Preparation of Apparatus

7.1 Make sure that the test vessels and the spindle-mounted attachments are undamaged, clean and free from grease and other contaminants (**4**).

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

7.2 *Assembly of Test Vessel (50 mm × 85 mL split vessel)*—The following items are required to assemble the test vessel: two 50 mm × 85 mL glass cylinders; a 50 mm diameter

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FIG. 2 – Computer-controlled ACU Connected to an Aeration Base via a Gas Output Hose

aeration 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. 6.

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

7.2.1 To assemble the test vessel, position the clamp ring approximately 1 mm from the end of one of the 50 mm × 85 mL glass cylinders (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 aligned with the printing on the glass cylinder. Secure the clamp ring using the ball-ended hex key (Fig. 8).

7.2.2 Locate the 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. 9).

NOTE 6—The 50 mm diameter aeration base consists of the base; permeable, woven, metal disk; m3 × 6 countersunk screw and O-ring.

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. 10). Make sure that the gap in the leveling assembly is approximately in line with the gap in the clamp ring.

7.2.4 Carefully invert the glass cylinder, clamp ring, aeration base and leveling assembly and place on the edge of a flat surface (Fig. 11) 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 (**5**).

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 second 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. 12).

7.2.11 Place the funnel on top of the assembled test vessel and locate on the powder rheometer, ensuring that the platform

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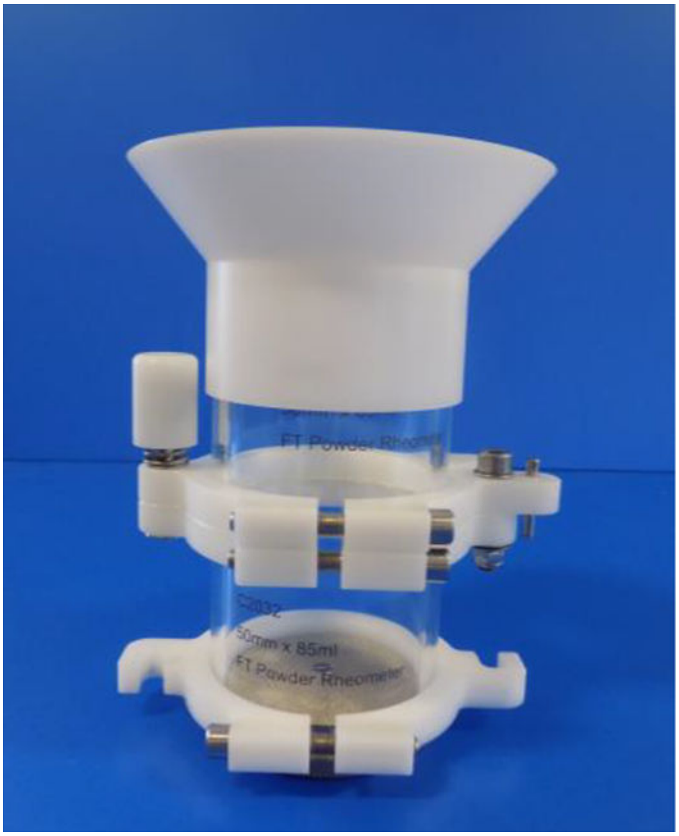

FIG. 3 Test Vessel (50 mm × 85 mL split vessel with stainless steel aeration base)

is free from obstruction and the test vessel is level (Fig. 13). Connect the ACU gas output hose to the aeration base.

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 7—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 (**6**). 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 height/carriage position is calibrated using two height gauges. 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*—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 interchange spindle-mounted attachments for different sections of the test and use a leveling assembly. The test specimen first undergoes three conditioning cycles 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 the test specimen of conditioned powder with a level surface that is ready for testing. The specialized blade must then be interchanged for the vented piston attachment, which is used to compress the test specimen to the target consolidating stress, or range of stresses, as determined in the selected test program.

10. Procedure

10.1 Measurement procedure for the determination of PD_q and gas permeability parameters.

10.1.1 Select the appropriate test program from the program library.

NOTE 8—There is an automated test program available, which is based on three initial conditioning cycles followed by the removal of excess powder using the leveling assembly. This is followed by applying a consolidating, normal stress of 1 kPa to the top of the powder bed. A fixed superficial gas velocity of 2 mm/s then traverses the powder bed and the PD_q is recorded consecutively at applied normal stresses of 1, 2, 4, 6, 8, 10, 12 and 15 kPa. This program is called a 'permeability' test, additional information is given in Reference (**7**). The program can be modified if other aspects of the test specimen's behavior need to be investigated in greater detail.

10.1.2 Securely fasten the assembled 50 mm × 85 mL split vessel (7.2) to the instrument table using a stainless steel clamping assembly (Fig. 13) and connect the ACU gas output hose to the aeration base.

NOTE 9—Typically the gas used is air, however other gases may be used. If other gases are used, the ACU may require re-calibration.

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 test specimen such that, following conditioning, the upper surface of the test specimen does not fall below the split level of the leveling assembly.

NOTE 10—If the level of the powder is below the level of the leveling assembly following the conditioning phase, the test is classified as a failure and re-run with a larger starting volume.

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

NOTE 11—This automatically causes the blade to be slowly lowered into the test vessel, after which it performs three conditioning cycles by traversing through the test specimen along a prescribed helical path (6.3.1).

NOTE 12—When the test program is initiated, the mass of the test specimen is registered within the data file associated with the test.

10.1.6 Once conditioning is complete and the test has been automatically paused, remove the funnel.

10.1.7 After confirming this step, use the leveling assembly to separate and remove excess powder left in the upper section of the test vessel, collect it in an appropriate container.

10.1.8 Leaving the leveling assembly in the open position, exchange the specialized blade for the vented piston attachment.

NOTE 13—After this has been confirmed, the program automatically continues by moving the vented piston attachment towards the surface of the test specimen in the test vessel at a speed of 0.5 mm⁄s until a pre-consolidation stress is reached. Then the vented piston is moved down at a maximum speed of 0.05 mm⁄s until the initial target consolidating stress is established and held for 60 seconds.

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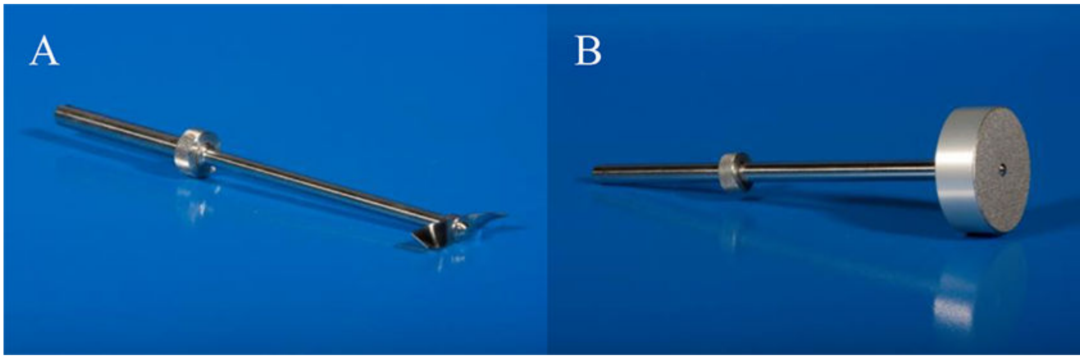

FIG. 4 Spindle-mounted Attachments: Specialized Blade (A); Vented Piston (B)

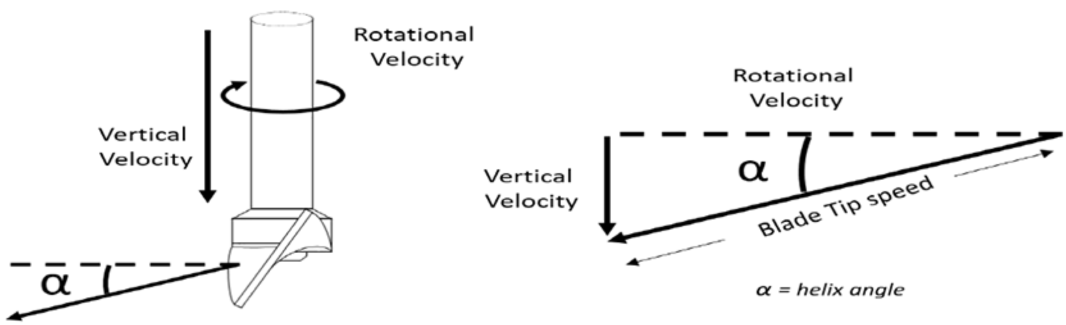

FIG. 5 Motion of the Specialized Blade During the Downward Traverse of a Conditioning Cycle

FIG. 6 Components to Assemble the Test Vessel (4 mm ball-ended hex key not shown)

10.1.9 The program then automatically continues to switch on the compressed gas at the designated velocity defined in the program.

NOTE 14—The selection of the fixed superficial gas velocity depends on the relative gas permeability of the powder. A velocity that is too low could result in minimal PD_q values being recorded close to the lower limits of the instrument's resolution. Velocities that are too high may result in an out-of-range issue with the pressure transducer, non-laminar flow may occur, or material may be ejected from the test vessel, or combinations thereof. Most powders provide meaningful results when tested using a superficial gas velocity of 2 mm/s; for very impermeable powders, a lower superficial gas velocity is recommended; for very permeable powders a higher superficial gas velocity is recommended.

10.1.10 Once the stress applied to the powder bed and PD_q are both stable, these values and the vertical position of the piston are recorded by the instrument.

10.1.11 Repeat 10.1.10 with increasing applied normal stress levels at a constant superficial gas velocity defined in the

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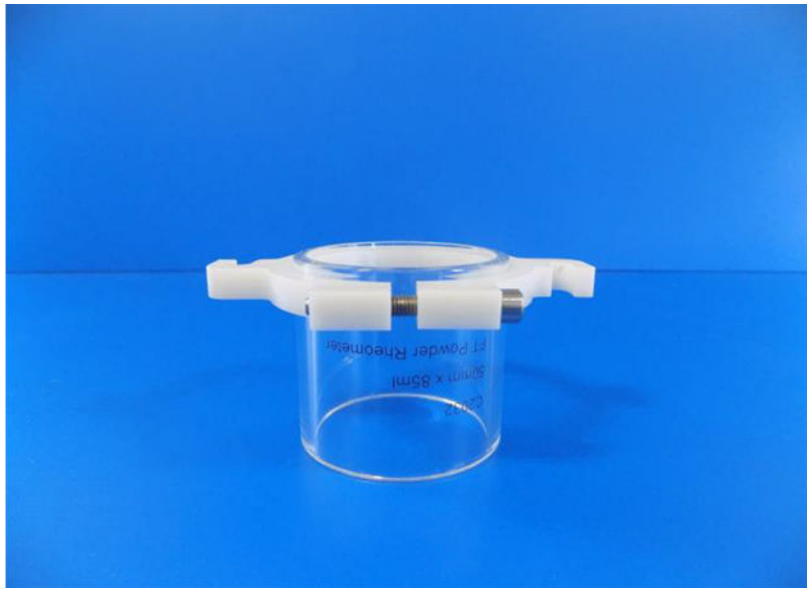

FIG. 7 Positioning the Clamp Ring Approximately 1 mm from the End of the 50 mm × 85 mL Glass Cylinder

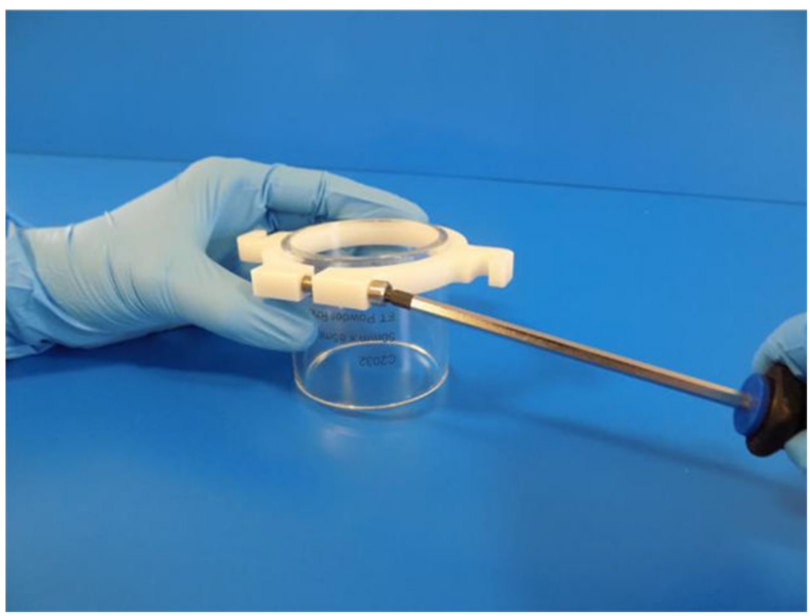

FIG. 8 Securing the Clamp Ring to the 50 mm × 85 mL Glass Cylinder with the Ball-ended Hex Key

program, until the advised number of steps designated in the test program have been completed.

NOTE 15—The automated test routine contains eight (8) measurement points between normal stress of 1 and 15 kPa, however the operator can modify the program to utilize additional or alternative measurement points, as well as different superficial gas velocities, if required.

10.1.12 Record ambient temperature and humidity using appropriate equipment.

11. Calculation and Interpretation of Results

11.1 Record the applied normal stress, carriage position, superficial gas velocity and PD_q values generated during the test program.

NOTE 16—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.

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FIG. 9 Fitting the Aeration Base with O-ring onto the 50 mm × 85 mL Glass Cylinder

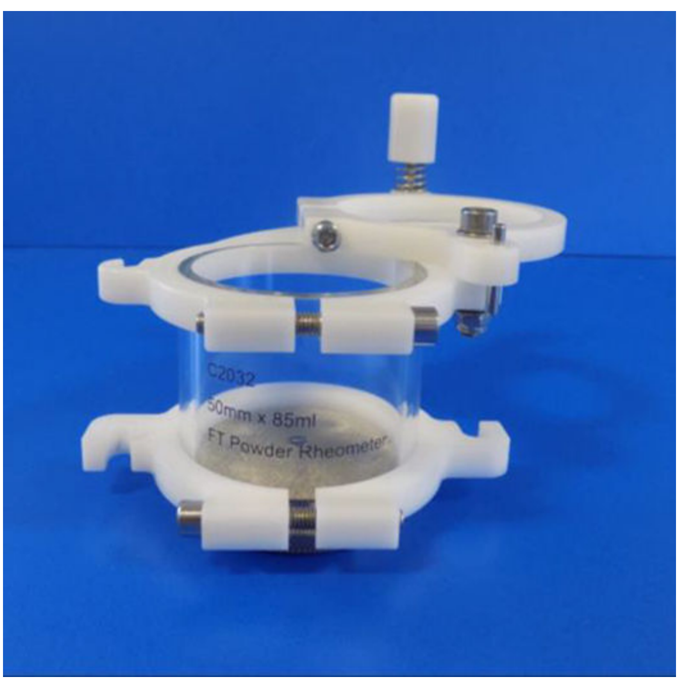

FIG. 10 Positioning the Leveling Assembly with Respect to the 50 mm × 85 mL Glass Cylinder

11.2 Calculate gas permeability from Darcy's Law:

$$Q = \frac{kA}{\mu}\frac{\Delta P}{H} \tag{1}$$

where:

Q = the volumetric gas flow rate, m^3/s,
k = the gas permeability, m^2,
A = the cross-sectional area of the powder bed, m^2,
ΔP = the pressure drop across the powder bed, $N{\cdot}m^2$ (equivalent to Pa),
μ = the gas viscosity, $N{\cdot}m^2{\cdot}s$ (equivalent to Pa·s), and
H = the height of the powder bed, m.

NOTE 17—For most situations $\mu = 1.74 \times 10^{-5}$ $N{\cdot}m^2{\cdot}s$ (equivalent to Pa·s) for air at sea level.

11.3 Rearranging Darcy's Law and dividing by area gives:

$$k = \frac{q\mu H}{\Delta P} \tag{2}$$

where:

q = the superficial gas velocity, m/s.

11.4 The typical method for graphical presentation of the results from a permeability test is shown in Fig. 14, with some examples of PD_q plots for different degrees of gas permeability. Plot the pressure drop or the permeability (based on the above calculation) against the applied normal stress, or both, drawing a smooth line between the data points.

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 nearest 1 % RH.
12.3.6 Superficial gas velocity to the nearest 0.01 mm/s.
12.3.7 Test vessel capacity to the nearest 0.5 mL.

12.4 Provide (in plot form) the following properties as a function of normal stress, σ:

12.4.1 PD_q across the powder bed.
12.4.2 Gas permeability of the powder bed.

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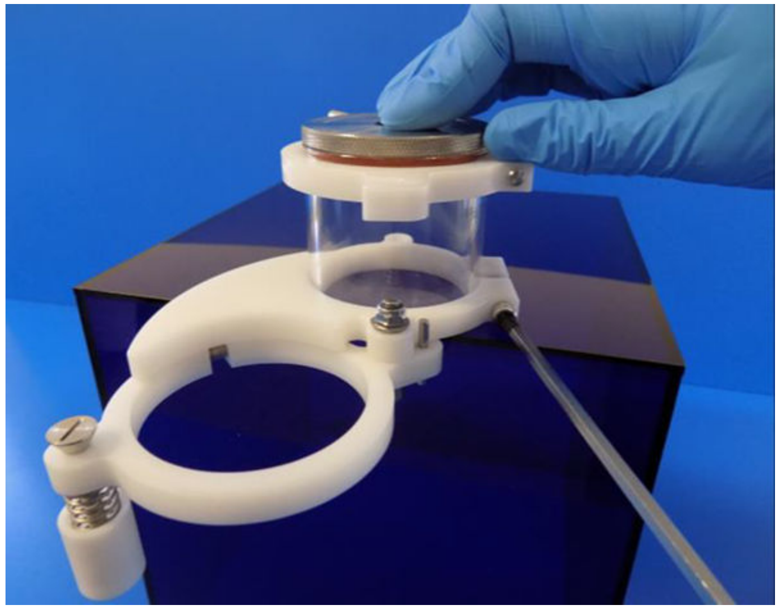

FIG. 11 Fitting the Leveling Assembly onto the 50 mm × 85 mL Glass Cylinder with the Ball-ended Hex Key

FIG. 12 Fitting the Upper Glass Cylinder

12.5 Record stresses and calculated parameters to three significant digits, velocities to 0.01 mm/s and all PD_q values to 0.01 kPa.

13. Precision and Bias

13.1 *Precision*—It is not possible to specify the precision of the procedure in Test Method for Measuring the Permeability of Powders as a Function of Consolidation 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

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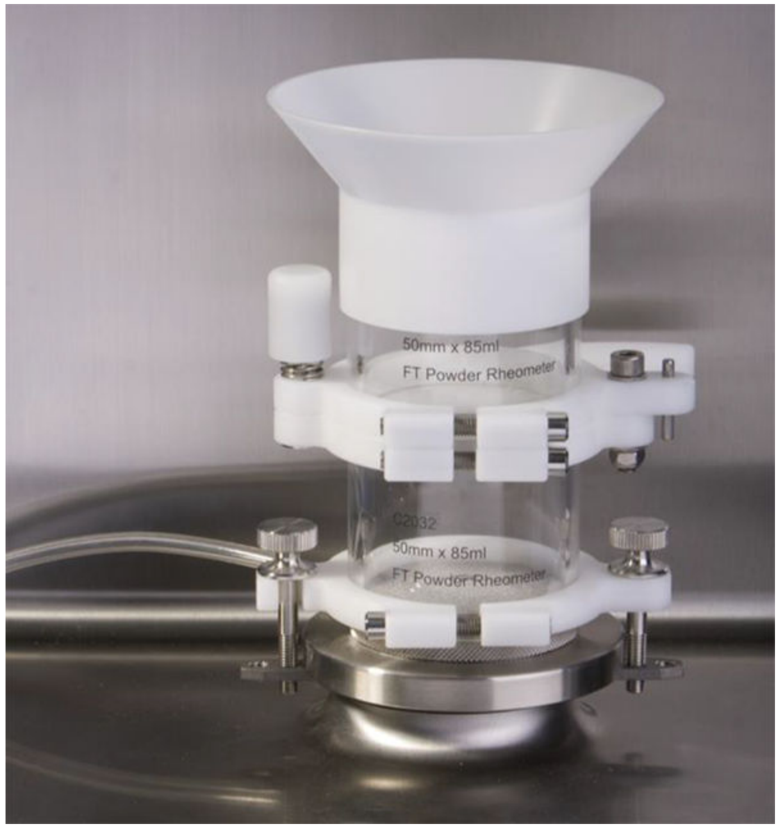

FIG. 13 Test Vessel (50 mm × 85 mL split vessel) Located on Instrument Table Prior to Taring (zeroing) of the Empty Vessel Mass (Gas output hose connected to the aeration base.)

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 Measuring the Permeability of Powders as a Function of Consolidation using the Freeman Technology FT4 Powder Rheometer because no material having an accepted reference value is available.

14. Keywords

14.1 Freeman Technology; FT4 Powder Rheometer; normal stress; gas permeability; powder; powder bed; pressure drop; superficial gas velocity

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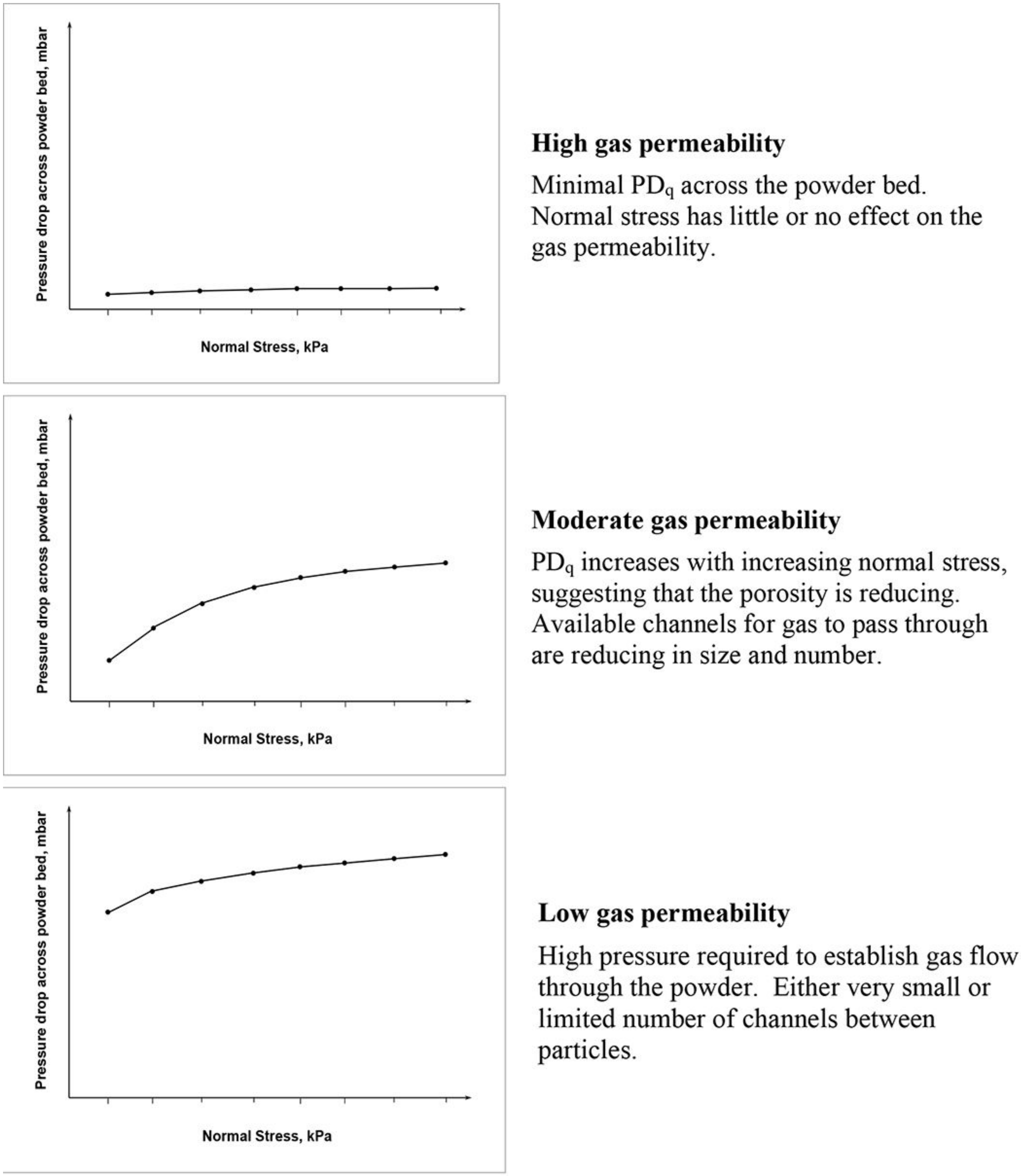

FIG. 14 Schematic Representation of Typical Pressure Drop Profiles for Material Comparison When Analysis is Completed at the Same Superficial Gas Velocity and Range of Applied Normal Stress

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