Designation: D7891 – 24

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

This standard is issued under the fixed designation D7891; 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 incipient failure properties of a powder as a function of the normal stress for a given level of consolidation. The method also allows the further determination of the unconfined yield strength, internal friction angles, cohesion, flow function, major principal stress and wall friction angle (with the appropriate wall coupon fitted to the correct accessory).

1.2 These parameters are most commonly used to assist with the design of storage hoppers and bins using industry standard calculations and procedures. They can also provide relative classification or comparison of the flow behavior of different powders or different batches of the same powder if similar stress and shear regimes are encountered within the processing equipment.

1.3 The apparatus is appropriate for measuring the properties of powders with a maximum particle size of 1 mm. It is practicable to test powders that have a small proportion of particles of 1 mm or greater, 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 reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.5 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement 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:*

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

D6128 Test Method for Shear Testing of Bulk Solids Using the Jenike Shear Tester

D6682 Test Method for Measuring Shear Stresses of Powders Using Peschl Rotational Split Level Shear Tester (Withdrawn 2017)[2]

D6773 Test Method for Bulk Solids Using Schulze Ring Shear Tester

3. Terminology

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

---

[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 March 15, 2024. Published April 2024. Originally approved in 2015. Last previous edition approved in 2015 as D7891 – 15. DOI: 10.1520/D7891-24.

[2] The last approved version of this historical standard is referenced on www.astm.org.

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

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 test specimen by use of a specialized blade attachment.

3.2.2 *wall friction coupon, n—in storing, handling, and processing bulk solids using industrial equipment*, a test piece used in the wall friction test that is manufactured from a material that represents the material of construction of the silo/bin/hopper that stores the powder.

4. Summary of Test Method

4.1 *Selection of the Appropriate Testing Regime*—The particular consolidating stress level or levels used to evaluate the flow properties of the powder 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 Shear Stress*—The instantaneous shear stress is measured by establishing the consolidating stress with the shear head and pre-shearing the test specimen until a steady state condition is reached. The test specimen is then subjected to a reduced normal load and then sheared until the shear force reaches a maximum and then decreases.

4.3 *Measurement of Wall Friction as a Function of Normal Stress*—The kinematic shear stress is measured by establishing the consolidating stress with the wall friction attachment, fitted with a wall friction coupon, representing the material against which the powder is required to flow. A single pre-shearing cycle is completed until a steady state condition is reached. The test specimen is then subjected to a reduced normal load and sheared until the shear force reaches a maximum and then decreases. The shear is maintained such that the kinematic shear stress can be calculated.

5. Significance and Use

5.1 The test can be used to evaluate the following:

5.1.1 *Classification or Comparison of Powders*—There are several parameters that can be used to classify powders relative to each other, the most useful being the measured shear stresses, cohesion, flow function and angle of internal friction.

5.1.2 *Sensitivity Analysis*—The shear cell 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, water content and temperature.

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

5.3 *Storage Vessel Design*—Mathematical models exist for the determination of storage vessel design parameters which are based on the flow properties of powders as generated by shear cell testing, requiring shear testing at a range of consolidating stresses as well as the measurement of the wall friction angle with respect to the material of construction of the storage vessel. The methods are detailed in Refs. (**1-3**).[3]

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 (**4**).

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 which simultaneously measures the force and torque required to mobilize a powder contained in a range of vessel types 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.

6.2 The shear cell vessel is shown in Fig. 2 (assembly described in 7.2). It consists of a serrated base, made from a suitable engineering plastic such as polyoxymethylene (POM), onto which are mounted two borosilicate glass cylinders (50-mm × 85-mL vessel) connected by a POM leveling assembly. 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 glass cylinders are defined 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 attachments to facilitate the various test procedures are shown in Fig. 3. They consist of a twisted blade (Fig. 3(A)) to condition the test specimen, a compaction piston (Fig. 3(B)) to compress the test specimen to achieve the desired consolidating normal stress, a shear head (Fig. 3(C)) to generate shearing within the powder and a wall friction head with interchangeable wall friction coupon (Fig. 3(D)).

NOTE 3—The construction material of the attachments (Fig. 3) is

[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.

FIG. 1 FT4 Powder Rheometer (The left hand image shows the instrument with the shear head fitted; the right hand image shows the shear head and test vessel.)

FIG. 2 Shear Cell Test Vessel

stainless steel, or a combination of stainless steel and anodized aluminum.

NOTE 4—It is practicable to employ test vessels with 1 and 10 mL capacities in conjunction with the FT4 Powder Rheometer. The mode of operation of the 10 mL test vessels is identical to that described herein for the 85 mL test vessel but using a smaller test vessel and range of attachments. The limit on the maximum particle size is commensurately reduced to a maximum of 0.5 mm for the 10 mL test vessel. Shear testing with 1 mL of test specimen requires a different cell design and attachments, which is beyond the scope of this standard.

6.4 A thermometric device and hygrometer are advised to measure temperature and humidity as referenced in 10.3.

7. Preparation of Apparatus

7.1 Make sure that the shear test vessel components and attachments are undamaged, clean, and free from grease and other contaminants (**5**).

7.2 *Assembly of Shear Test Vessel*—The following items are required to assemble the test vessel: two 50-mm × 85-mL glass cylinders, a 50-mm diameter POM serrated base fitted with an O-ring; a 50-mm diameter clamp ring; a 50-mm diameter leveling assembly; and 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. 4.

NOTE 5—A detailed assembly procedure is also available (**6**).

7.2.1 To assemble the shear test vessel, position the clamp ring approximately 1 mm from the end of one of the glass cylinders and loosely fit the clamp ring onto the glass cylinder (Fig. 5). 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.

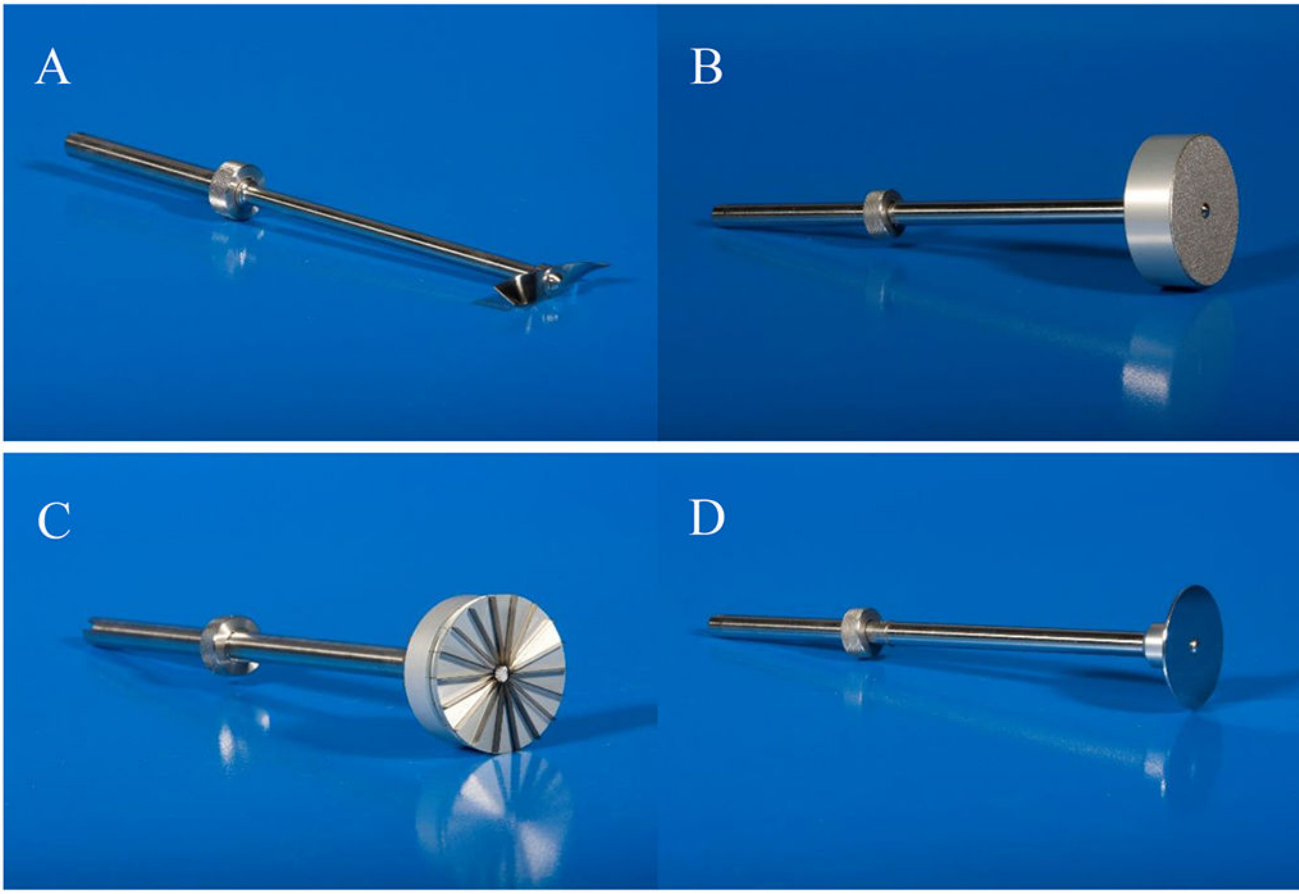

FIG. 3 Spindle-Mounted Attachments Used in Shear and Wall Friction Tests: Blade (A); Vented Piston (B); Shear Head (C); Wall Friction Head (D)

7.2.2 Locate the serrated 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. 6).

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 serrated base. The pivot pin of the leveling assembly should be approximately aligned with the gap in the clamp ring.

7.2.4 Carefully invert the glass cylinder, clamp ring, serrated base and leveling assembly and place on the edge of a flat surface (Fig. 7) 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.

7.2.5 Push down gently on 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 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. 8).

7.2.11 Place a funnel on top of assembled test vessel and locate on the FT4 Powder Rheometer (Fig. 9).

8. Calibration

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

NOTE 6—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. (**7**). Force should be calibrated within the FT4 Powder Rheometer's performance limits of ±50 N, and torque should be calibrated within the performance limits of ±900 mN·m both to tolerances better than 1.0 %.

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 use a leveling assembly and for the interchange of the spindle-mounted attachments for different sections of the test. The test specimen first undergoes conditioning using the blade attachment, then the piston attachment is fitted to compress the

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

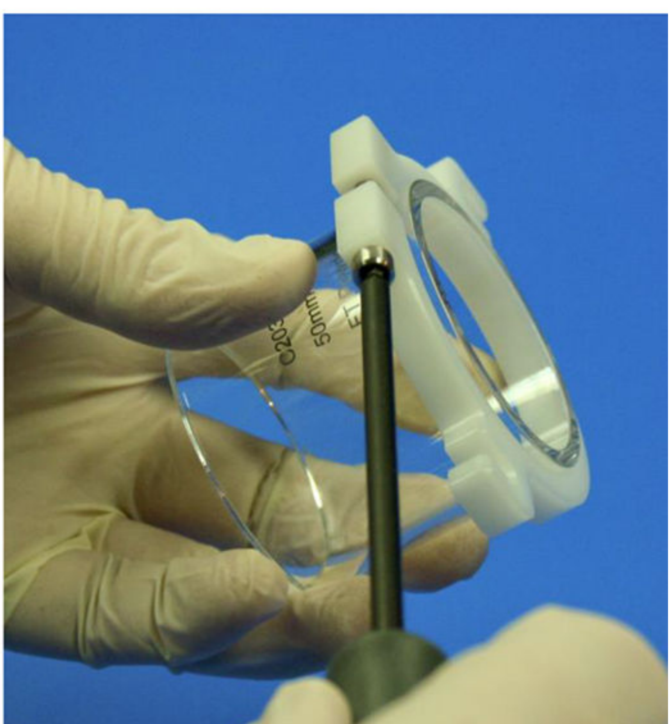

FIG. 5 Fitting the Clamp Ring to the Glass Cylinder Using the Ball-ended Hex Key

powder to the target consolidating normal stress. Excess powder must then be removed from the test cell by means of a leveling assembly to leave a controlled volume of consolidated powder with a level surface that is ready for shear testing. The shear head must then be fitted to the instrument.

10. Procedure

10.1 *Measurement of Shear Stress:*

10.1.1 Select the appropriate test program from the program library. There are four automated test programs which are based on consolidating stresses of 3, 6, 9, and 15 kPa.

NOTE 7—The test method can be modified with respect to consolidating stress, shear rate, and, the number and length of pre-shear cycles. These options are detailed in Annex A1 and Ref. (**8**).

10.1.2 Securely fasten the assembled test vessel (7.2) to the instrument table using the stainless steel clamping assembly (Fig. 9).

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 powder such that, following consolidation, the test specimen does not fall below the split level of the leveling assembly.

NOTE 8—If the level of the powder is below the level of the leveling assembly following the compression phase, the test is classified as a failure and re-run with a greater starting volume.

10.1.5 Select the start button on the computer screen to commence the test.

NOTE 9—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.

FIG. 6 Fitting the Serrated Base with O-ring to the Glass Cylinder

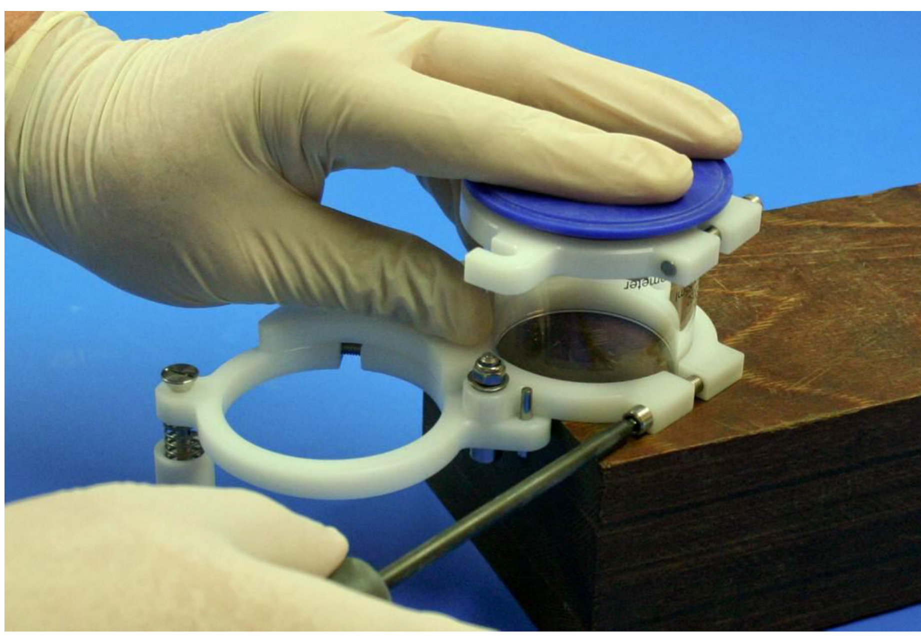

FIG. 7 Fitting the Leveling Assembly onto the Glass Cylinder with the Ball-ended Hex Key

10.1.6 Once conditioning is complete and the test has been automatically paused, exchange the blade attachment for the vented piston attachment.

NOTE 10—The vented piston attachment automatically moves into the test vessel to compress the test specimen until the target consolidating stress has been achieved and held for 60 seconds.

10.1.7 Once the compression cycle is complete and the test has automatically paused, remove the funnel.

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

10.1.9 Leaving the leveling assembly in an open position, replace the vented piston with a shear head.

NOTE 11—The shear head automatically moves to the surface of the test specimen at a speed of 0.5 mm/s, then moves down at a maximum speed of 0.08 mm/s until the target consolidating stress is re-established and held for 60 seconds.

10.1.10 Pre-shearing cycles are completed until a steady state stress has been achieved.

NOTE 12—During a pre-shearing cycle, the shear head is automatically rotated within the powder at a fixed rate of 18 degrees/min while maintaining the target consolidating stress until steady state shear stress has been achieved. The shear head direction of rotation is then reversed to

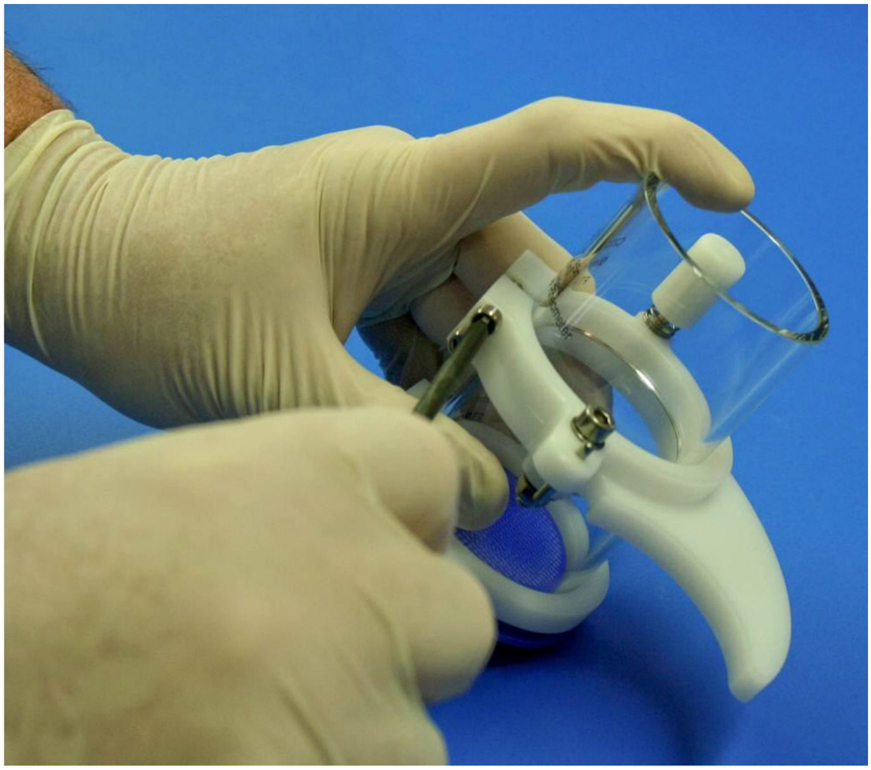

FIG. 8 Fitting the Upper Glass Cylinder

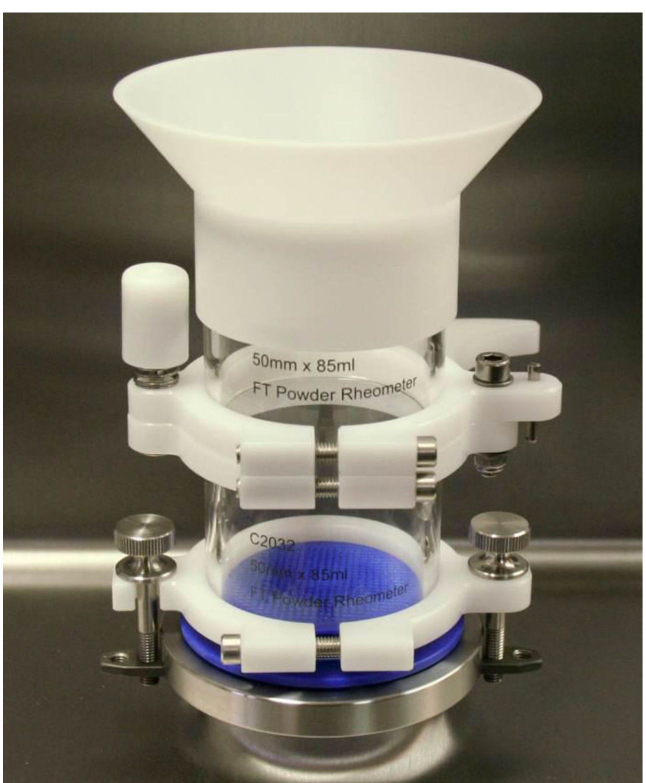

FIG. 9 Shear Test Vessel Located on Instrument Table Prior to Taring (Zeroing) of the Empty Test Vessel Mass

establish a zero torque level. Pre-shearing cycles are repeated for a maximum of 10 times or until two consecutive steady state shear stress values are within 1 % of each other.

NOTE 13—If convergence does not occur in 10 pre-shearing cycles, the powder is considered unstable, possibly due to the degradation within the shear zone and may mean that the powder is not suitable for shear testing.

10.1.11 Once the pre-shear requirements are met, the shear testing cycle is initiated.

NOTE 14—During a shear testing cycle, the normal stress applied to the test specimen is automatically reduced to a pre-selected value lower than the consolidating stress and is held for 20 seconds while maintaining zero torque. Shear is then reinitiated until a maximum shear stress is achieved and the shear stress has subsequently reduced. The maximum shear stress recorded is the peak shear point for the selected normal stress.

10.1.12 Once a shear testing cycle is complete, the consolidating stress is re-established, and a single pre-shearing cycle is initiated. The program then selects another lower normal test stress and a shear testing cycle is completed. This process is repeated until the test conditions that were specified have been completed.

10.2 *Measurement of Wall Friction:*

10.2.1 Repeat 10.1.1 to 10.1.8.

10.2.2 Leaving the leveling assembly in the open position, replace the vented piston with the wall friction attachment.

NOTE 15—The wall friction attachment automatically moves to the surface of the test specimen at a speed of 0.5 mm/s, then moves down at a maximum speed of 0.08 mm/s until the target consolidating stress is re-established and held for 60 seconds.

10.2.3 A single pre-shearing cycle is completed.

NOTE 16—During a pre-shearing cycle, the wall friction attachment is automatically rotated within the powder at a fixed rate of 18 degrees/min while maintaining the target consolidating stress until steady state shear stress has been achieved. The wall friction attachment direction of rotation is then reversed to establish a zero torque level. Wall friction programs typically have a single pre-shear cycle.

10.2.4 Once the pre-shear requirements are met, a wall friction testing cycle is initiated.

NOTE 17—During a wall friction testing cycle, the normal stress applied to the test specimen is automatically reduced to a pre-selected value lower than the consolidating stress and is held for 20 seconds while maintaining zero torque. Shear is then reinitiated until a maximum shear stress is achieved and the shear stress has subsequently reduced. The shear is maintained for 45 seconds such that the kinematic shear stress can be calculated. The direction of rotation for the wall friction attachment is reversed to establish a zero torque level.

10.2.5 Once a wall friction testing cycle is complete, the program then selects another lower normal test stress and another testing cycle is completed. This process is repeated until all test conditions that were specified have been completed.

NOTE 18—Automated test routines typically contain five measurement points recorded between approximately 20-80 % of the consolidating stress.

10.3 Record ambient temperature and humidity using appropriate equipment.

11. Calculation and Interpretation of Results

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

11.2 Calculate the normal stress by dividing the normal force measured by the instrument load cell by the cross-sectional area of the shear head.

$$\sigma = \frac{N}{A} \tag{1}$$

where:

σ = normal stress, Pa,
N = measured normal force, N, and
A = area of the shear head, m^2.

11.3 Calculate the shear stress from the measured torque using the following equation:

$$\tau = \frac{3 \cdot T}{2 \cdot \pi \cdot r^3} \tag{2}$$

where:

τ = shear stress, Pa,
T = measured torque, N·m, and
r = radius of the shear head, m.

11.4 Use pro-rating, if necessary, to compensate for slight changes in pre-shear stress during a series of shear measurements. Pro-rating involves recalculating the shear stress for each test point using the following formula:

$$\tau_{s,pro-rated} = \tau_s \times \frac{\tau_{p\ average}}{\tau_p} \tag{3}$$

where:

$\tau_{s,pro-rated}$ = pro-rated shear stress, Pa,
τ_s = measured shear stress, Pa,
$\tau_{p\ average}$ = average of all pre-shear shear stresses, Pa, and
τ_p = measured pre-shear shear stress preceding the shear step, Pa.

11.5 Plot the pre-shear point and all the valid shear test points as normal stress/shear stress data pairs (Fig. 10). Draw a least mean squares, linear regression line through the test points, and extrapolate this line to the normal stress level of the pre-shear point and, in the opposite direction, to meet the y-axis (where the normal stress is zero). This line is known as the yield locus. The angle that the yield locus makes with the horizontal axis is known as the angle of internal friction and is designated as φ. The point at which the yield locus crosses the y-axis is known as the cohesion (kPa), C.

11.6 Check to make sure that the yield locus passes above or through the pre-shear point. If it passes below the pre-shear point, scrutinize the individual test points to make sure that they are valid or re-run the test.

11.7 Draw a Mohr stress circle such that its center is located on the x-axis, it is tangential to the yield locus, and the origin is a point on the circle. The non-zero intersection of this circle with the x-axis defines the unconfined yield strength (kPa), f_c.

11.8 Draw a second Mohr stress circle such that its center is located on the x-axis, it passes through the pre-shear point, and it is tangential to the yield locus. This circle intersects the x-axis at two positions—the greater of which defines the major principal stress (kPa), σ_1, seen in the powder at this level of consolidation during steady state flow, as shown in Fig. 10.

11.9 Draw a line from the origin that is tangential to the major Mohr circle. This line is the effective yield locus, and the angle that this line subtends to the x-axis is the effective angle of friction (°), δ.

11.10 Construct a flow function, FF, by drawing a smooth curve through a series of data points, each of which represents a pair of values of major principal stress and unconfined yield strength.

11.11 *Calculation of Wall Friction Angle:*

11.11.1 Plot the pre-shear point and all valid shear test points as normal stress/shear stress data pairs (Fig. 11). Draw a least mean squares regression line through all the data points. This is known as the wall yield locus (WYL), and the angle that is subtends to the x-axis is known as the angle of wall friction (°), φ'.

11.11.2 Calculate the wall friction angle by:

$$\varphi' = \tan^{-1}\left(\frac{\tau_w}{\sigma_w}\right) \tag{4}$$

where:

φ' = angle of wall friction, °,
σ_w = normal stress at steady state flow, kPa, and
τ_w = shear stress at steady state flow, kPa.

NOTE 19—In some cases, the WYL is not linear or may not pass through (or close to) the origin, or both. In these circumstances, the alternative analysis presented in Test Method D6128 may be employed.

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):

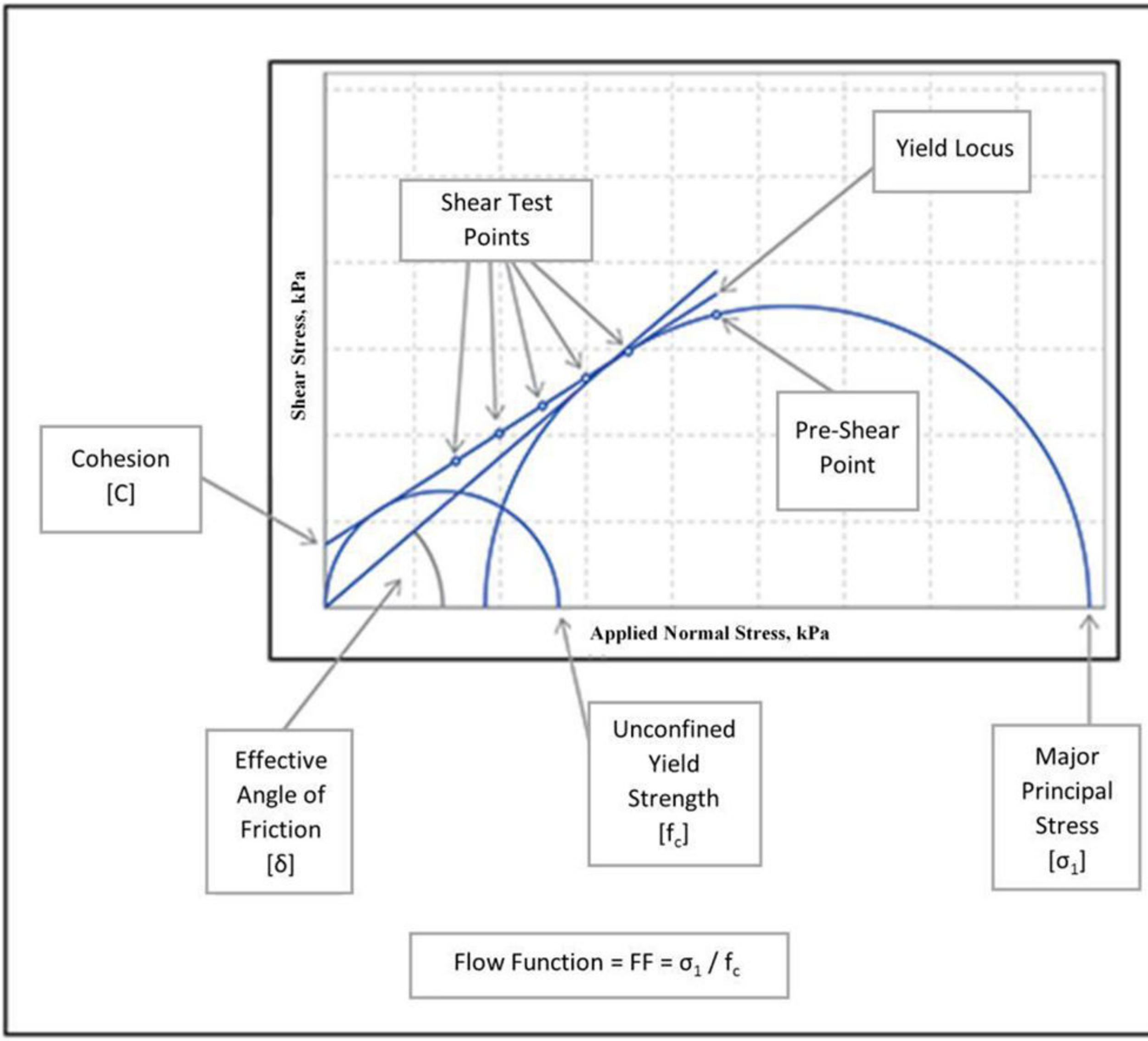

FIG. 10 Graphical Representation of the Shear Points, Mohr Circle Constructions and the Derived Parameters

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 water content 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 two significant digits.

12.4 Provide in plot form the following properties as a function of major principal stress, σ_1. Record all stresses to three significant digits and all angles to nearest 1°:

12.4.1 Unconfined yield strength, σ_c

12.4.2 Angle of internal friction, φ.

12.4.3 Effective angle of friction, δ.

12.4.4 Cohesion, C.

12.4.5 Wall friction angle, φ'.

13. Precision and Bias

13.1 *Precision*—It is not possible to specify the precision of the procedure in Test Method for Shear Testing of Powders using the Freeman Technology FT4 Powder Rheometer Shear Cell 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

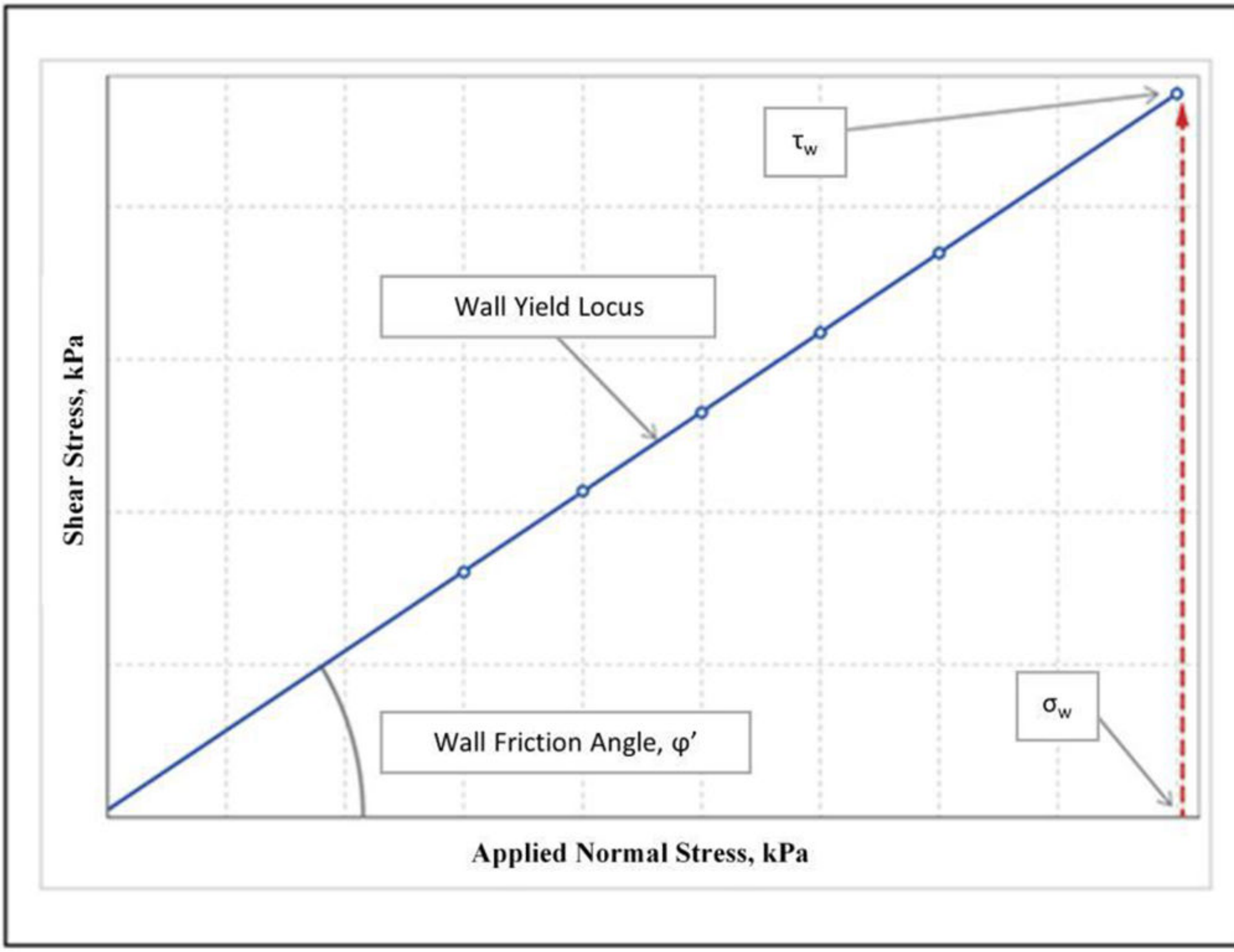

FIG. 11 Graphical Representation of the Wall Yield Locus (WYL)

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 Shear Testing of Powders using the Freeman Technology FT4 Powder Rheometer Shear Cell because no material having an accepted reference value is available.

14. Keywords

14.1 effective angle of friction; effective yield locus; flowability; flow function; Freeman Technology; FT4 Powder Rheometer; internal friction angle; powder; rotational shear cell; unconfined yield strength; wall friction; wall friction angle

ANNEX

(Mandatory Information)

A1. PRE-SHEAR PROTOCOLS AND SHEAR PEAKS

A1.1 *Adjusting Pre-Shear Protocols*—There is still some debate as to the appropriate degree of pre-shearing required to achieve critical consolidation prior to undertaking a shear test. Within the test program it is practicable to select and adjust the pre-shearing protocols such that the length and number of pre-shears is flexible. An automated routine is used to identify a pre-shear peak and move the program onto the next step. This can be disabled, and a defined time period/shear distance can be specified. The number of pre-shears can also be specified from a minimum of one, to between two and 100. A criterion to exit a multiple pre-shearing routine based on the convergence of the observed peak shear stresses of the last two pre-shear steps (to a specifiable limit) is also available. Therfore, it is practicable to replicate the methodologies specified in Test Methods D6682 and D6773.

A1.2 *Identifying Shear Peaks*—The recommended procedure is to undertake multiple pre-shears such that the peak stress of the last two pre-shears are within 1 % of each other or a maximum of ten pre-shear steps have been completed before shear testing is initiated. The shear peaks are automatically identified, and the program steps forward to the next section 20 seconds after a peak has been detected.

REFERENCES

(1) Jenike, A. W., "Storage and Flow of Solids," University of Utah, Bulletin 123, Vol. 53, 1964.
(2) Schulze, D., Powders and bulk solids. Behavior, Characterization, Storage and Flow, 2nd ed., Springer, Berlin Heidelberg, NY, 2007.
(3) Roberts, A. W., Basic Principles of Bulk Solids Storage, Flow & Handling, TUNRA Bulk Solids Research Associates, 1993.
(4) "INF92, Sampling," Freeman Technology Support Document, Issue B, 2013.
(5) "W7004, Cleaning," Freeman Technology Support Document, Issue D, 2017.
(6) "W7020, 50 mm Vessel Assemblies," Freeman Technology Support Document, Issue G, 2017.
(7) "W6010, FT4 Calibration Manual," Freeman Technology Support Document, Issue C, 2019.
(8) "W7107, Pre-Shearing Protocols for Shear Cell Testing," Freeman Technology Support Document, Issue B, 2013.

NOTE 1—Copies of referenced support documents may be obtained from Freeman Technology Ltd (via support@freemantech.co.uk).

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the 2015 edition that may impact the use of this standard. (March 15, 2024)

(1) Amended 1.3.
(2) Revised Units statement in 1.5.
(3) Revised Safety Hazards Caveat in 1.6.
(4) Addited International Caveat in 1.7.
(5) Removed Judgement Caveat.
(6) Added Footnotes 2 and 3.
(7) Revised 3.1, delimiting phases and formatting of Definitions in Section 3.
(8) Minor changes to 3.2.1, definition of 'conditioning'.
(9) Section 3 definitions have been checked against D653 to confirm that this information has not been duplicated.
(10) Removed capitalization of terms that are not proper nouns or acronyms.
(11) Revised text of "Preparation of Specimen" and moved from "Summary of Method" (Section 4) to "Conditioning" (Section 9).
(12) Revised text of 4.3 ('Measurement of Wall Friction as a Function of Normal Stress').
(13) Revised wording from 'moisture content' to 'water content'.
(14) Revised footer sub-note to include 'Sources of Supplier' statement.
(15) Revised text of 6.2 and 6.3 to generate a more precise description of accessories.
(16) Non-mandatory apparatus information moved into NOTEs 2, 3 and 4.
(17) Removed details pertaining to accessory design details and number of blades on the shear head.
(18) Revised formatting of figure numbers and descriptions.
(19) Added 6.4.
(20) Revised text of 7.2 and 7.2.3 with details separated out into NOTEs.
(21) Revised Fig. 5 for greater image quality.
(22) Revised text in figure descriptions for Fig. 5-7.
(23) "Hex driver" has been replaced by "ball-ended hex key".
(24) Revised Section 8, including NOTE 6.

(25) Preparation of the Specimen information moved into new Section 9 and revised to be in the 'imperative mood'.
(26) Significantly revised text in Section 10, non-mandatory text moved into NOTEs (NOTEs 7, 9, 10, 11, 12, 16, 17, 18 and 19).
(27) Removed previous NOTE 14 relating to the use of FT4 Powder Rheometer Data Analysis software.
(28) Added 10.3 to reflect 'Report' section parameters.
(29) Added equation parameter descriptions, equation numbering and regeneration of equations in Section 11 to improve image quality.
(30) Revised 11.2, 11.3, 11.4 and 11.11.2, non-mandatory text moved into NOTE 20.
(31) Improved image resolution for Fig. 10 and 11.
(32) Added reference to D6026 in 12.1.
(33) Revised 12.3.4.
(34) Revised Precision and Bias Statement, Section 13.
(35) Revised Annex information to reduce commercial content, A1.1.
(36) Revised References formatting and addition of NOTE 21.
(37) Revised Reference (4) and (7) issue numbers.
(38) Revised mandatory language.
(39) Revised wording from 'sample' to 'test specimen'.