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.

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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 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.

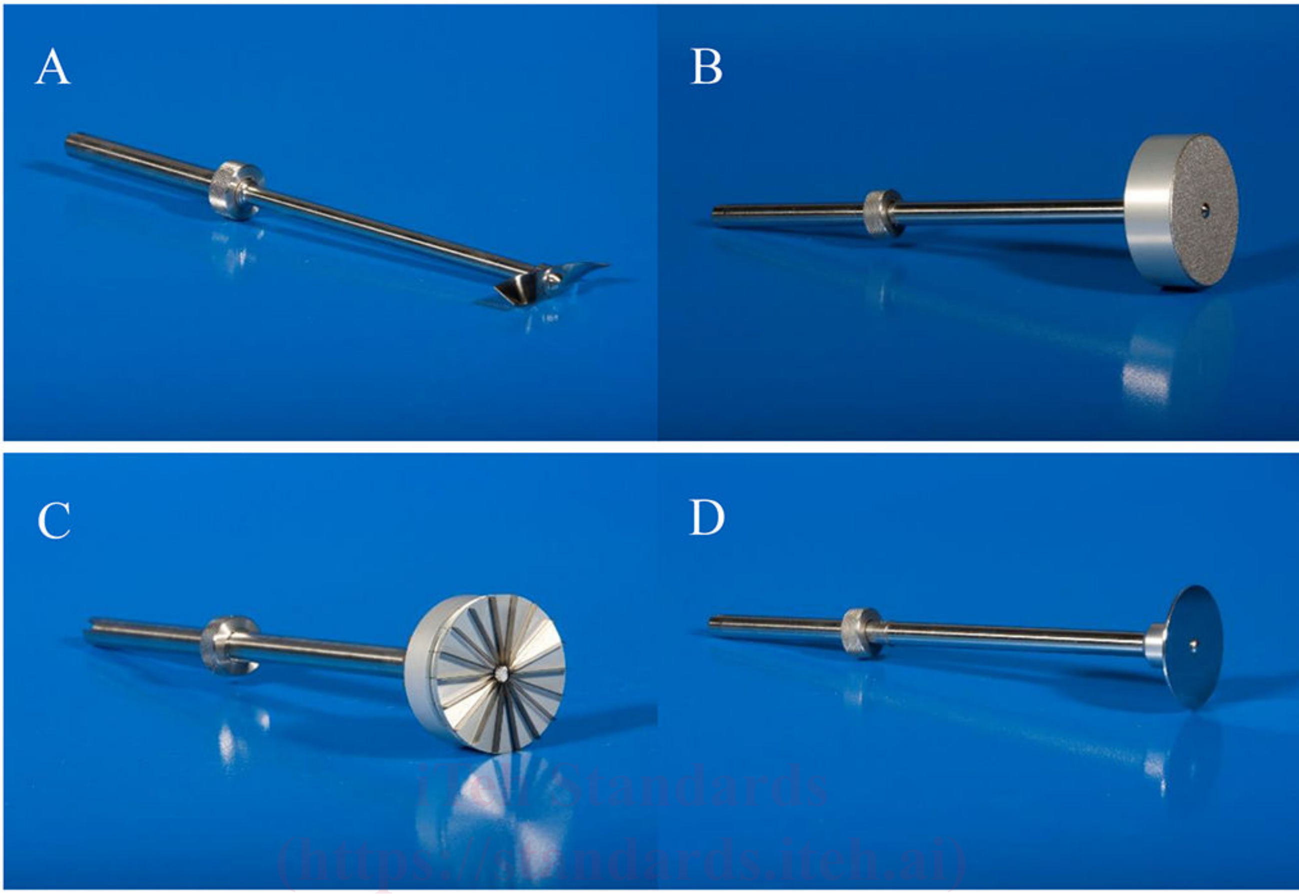

FIG. 3 Spindle-Mounted Attachments Used in Shear and Wall Friction Tests: Blade (A); Vented Piston (B); Shear Head (C); Wall Friction Head (D)

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)

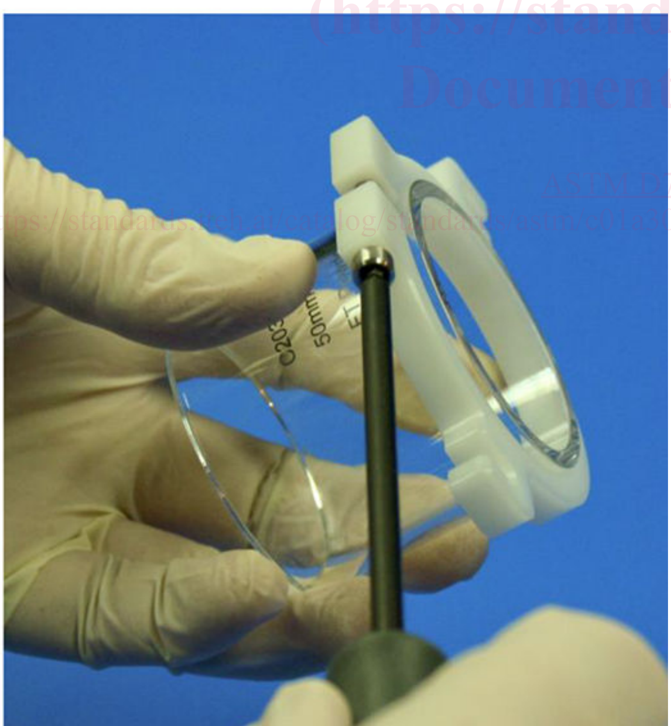

FIG. 5 Fitting the Clamp Ring to the Glass Cylinder Using the Ball-ended Hex Key

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

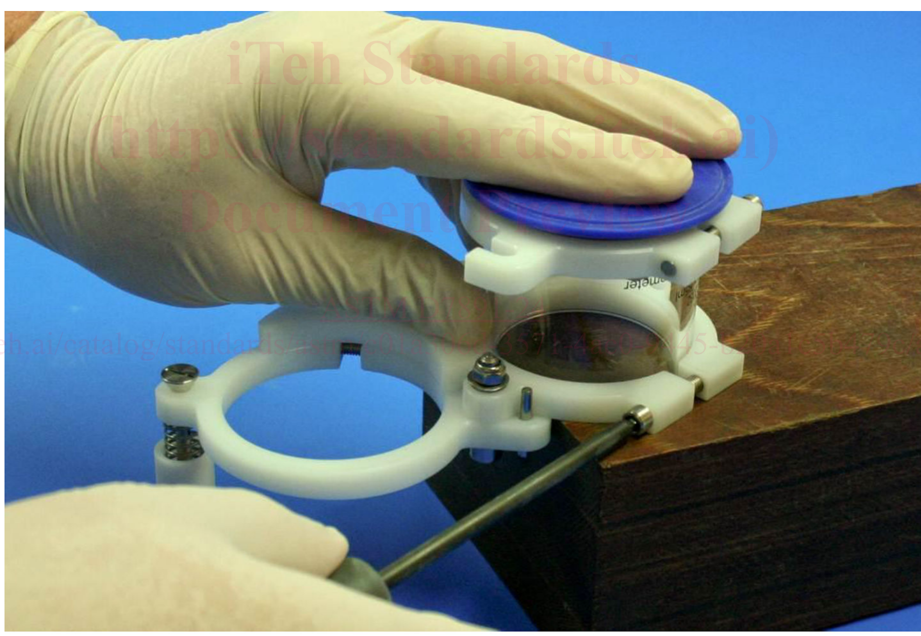

FIG. 7 Fitting the Leveling Assembly onto the Glass Cylinder with the Ball-ended Hex Key

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