



Designation: **D7891–15** **D7891 – 24**

## Standard Test Method for Shear Testing of Powders Using the Freeman Technology FT4 Powder Rheometer Shear Cell<sup>1</sup>

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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. ~~Scope~~ Scope\*

1.1 This method covers the apparatus and procedures for ~~measuring~~ 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 ~~for 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 ~~suited~~ appropriate for measuring the properties of powders with a maximum particle size of 1 mm. It is ~~possible~~ practicable to test powders ~~which that~~ have a small proportion of particles of 1 mm or greater, but ~~they should be present in the bulk sample as 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 and health 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.*

<sup>1</sup> 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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\*A Summary of Changes section appears at the end of this standard

## 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)<sup>2</sup>
- 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.

### 3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *wall friction coupon, n—in powders, 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 will depend on the reason for generating the data, as outlined in Section 5, and should broadly reflect the stresses that the powder will be is subjected to in its processing environment.

4.2 *Preparation of the specimen*—The specimen is added to the test vessel and its mass determined using the instrument's built-in balance. The selected test program is then initiated and runs independently of the operator other than the interchange of the spindle-mounted attachments for different sections of the test. The powder first undergoes a conditioning cycle using the blade attachment which removes any variability introduced during filling or from the material's previous history. The piston attachment is then fitted and is used to compress the powder to the required consolidating stress as determined in the selected test program. Excess powder is then removed from the test cell by means of a leveling assembly to leave a specimen of compressed powder with a level surface that is ready for shear testing. The shear head is then fitted to the instrument.

4.2 *Measurement of Shear Stress*—The instantaneous shear stress is then measured by re-establishingestablishing the consolidating stress with the shear head and then pre-shearing the test specimen until a steady state condition is reached. The powder-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 same specimen preparation method is used for this test, but a kinematic shear stress is measured by establishing the consolidating stress with the wall friction attachment, fitted with a coupon-wall friction coupon, representing the material against which the powder is required to flow, is used instead of a shear head: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:

<sup>2</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

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 ~~and/or environmental parameters or environmental parameters, or both,~~ such as (but not limited to) humidity, particle size and size distribution, particle shape and shape distribution, ~~moisturewater~~ content and temperature.

5.1.3 *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.

5.1.4 *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).<sup>3</sup>

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.

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## 6. Apparatus<sup>4</sup>

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

<sup>3</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

<sup>4</sup> 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 shear cell test vessel.)



FIG. 2 Shear Cell Test Vessel

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.

6.2.1 The shear cell vessel is located on the powder rheometer using a POM clamp ring.

NOTE 2—The glass cylinders are defined as ‘x mm × y mL’, which indicates the glass cylinder’s internal diameter, x, ( $50 \pm 0.04$  mm) and the precise volume of the lower section of the test vessel with the base fitted, y.

6.2.2 A POM funnel is also fitted to assist with the filling of the vessel.

6.3 Attachments are fitted—The attachments to facilitate the various test procedures are shown in Fig. 3. They consist of a twisted blade (Fig. 3(A)) to the powder rheometer condition the test specimen, a compaction piston (Fig. 3(B)) to facilitate various test procedures, (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)).

6.3.1 The first is a twisted blade (shown in Fig. 3(A)) that is used to condition the test specimen thus generating a repeatable stress condition within the powder.

NOTE 2—This conditioning process eliminates the effects of the powder’s history and also any operator-induced effects generated during the filling process.

NOTE 3—The construction material of the attachments (Fig. 3) is stainless steel, or a combination of stainless steel and anodized aluminum.

6.3.2 The second (Fig. 3(B)) is a compaction piston that compresses the specimen to achieve the desired consolidating normal stress:

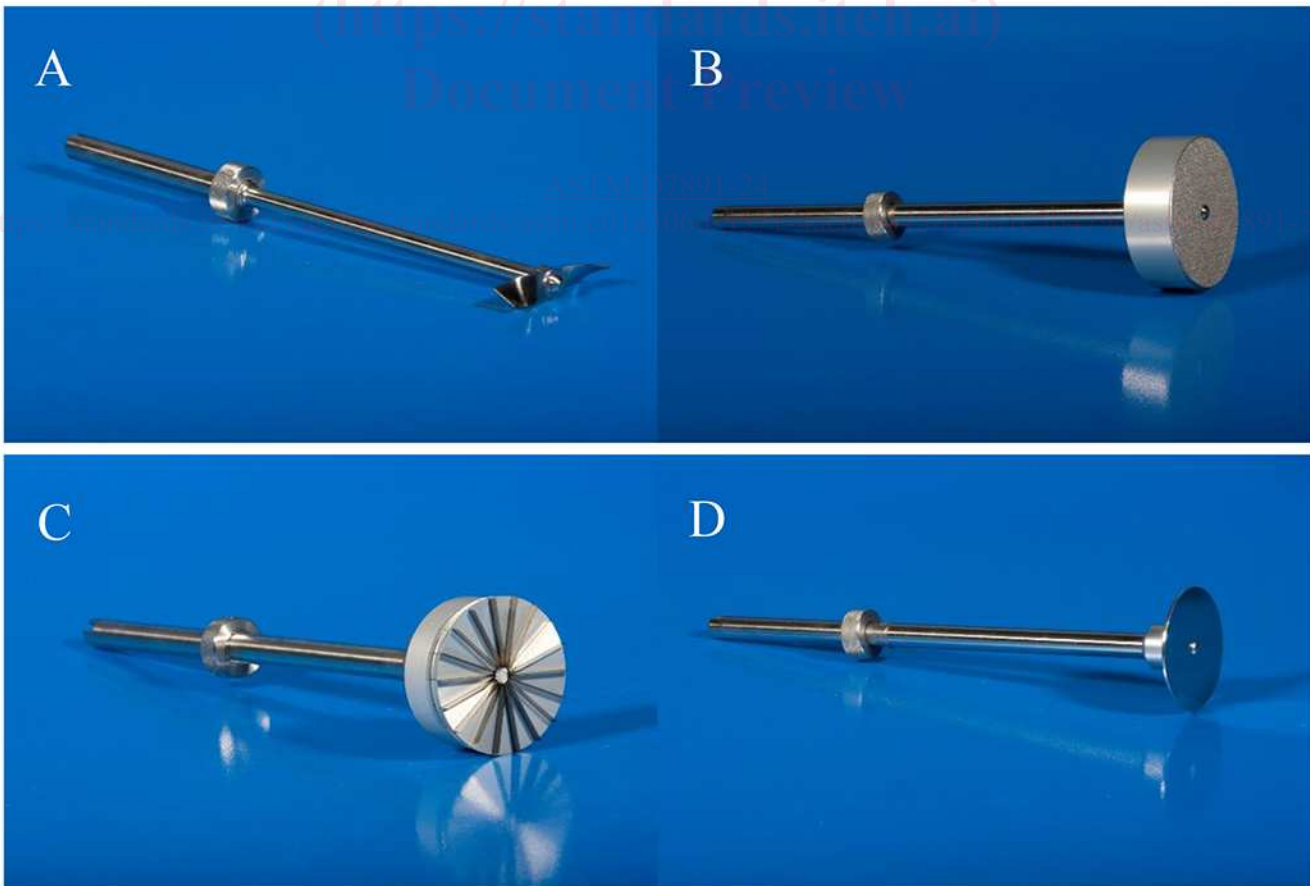


FIG. 3 Spindle-Mounted Attachments Used in Shear and Wall Friction Tests: Blade (A); Vented Piston (B); Shear Head (C); Wall Friction Head (D)

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.

6.3.3 The third (Fig. 3(C)) is a shear head consisting of 18 blades that are used to generate shearing within the powder.

6.3.4 The fourth (Fig. 3(D)) is a wall friction head and an interchangeable coupon representing the material of construction against which the powder will be required to flow.

6.3.5 All of the attachments (Fig. 3) are made from stainless steel and stainless steel+anodized aluminum.

NOTE 3—The blades located in the shear head are thin and thus relatively sharp. Care must be taken when handling the shear head to prevent skin abrasions and cuts.

6.4 Additionally, it is possible to employ shear cells with 10 mL and 1 mL capacity. A thermometric device and hygrometer are advised to measure temperature and humidity as referenced in 10.3 in conjunction with the FT4 Powder Rheometer if the quantity of available test specimen is less than 85 mL (at the chosen consolidation stress). The mode of operation of the 10-mL shear cell is identical to that described herein for the 85-mL shear cell but using a smaller shear cell and range of attachments. The limit on the maximum particle size is commensurately reduced to a maximum particle size of 0.5 mm. The 1-mL shear cell uses a significantly different cell design and attachments, which is beyond the scope of this standard.

## 7. Preparation of Apparatus

7.1 Since the integrity of the blades within the shear cell head is critical to generating accurate and reliable data, handle the shear head with care, store it in the case provided and inspect it for damage at regular intervals.

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

7.3 The following items are required to assemble the shear cell vessel: two 50-mm × 85-mL glass cylinders, a 50-mm serrated base, a 50-mm clamp ring, a 50-mm leveling assembly, and a 50-mm funnel. These items are shown in Fig. 4. A fully detailed assembly procedure is also available (6).

7.2 Assembly of Shear Test Vessel—To assemble the shear cell 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 (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; 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 hex driver ensuring that the screw is 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 not over tightened.

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.

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.



FIG. 4 Components Required to Assemble the 50-mm x 85-mL Shear Cell Vessel (4-mm ball-ended hex key not shown)

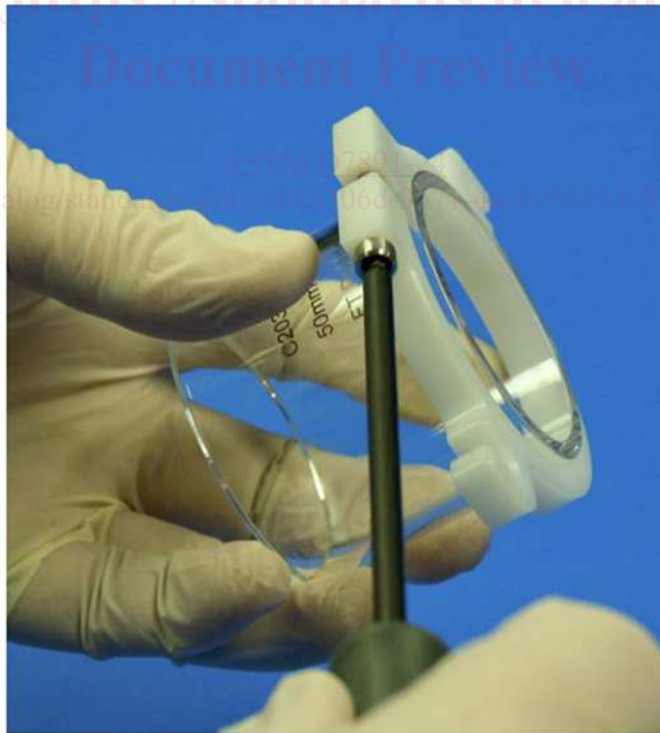
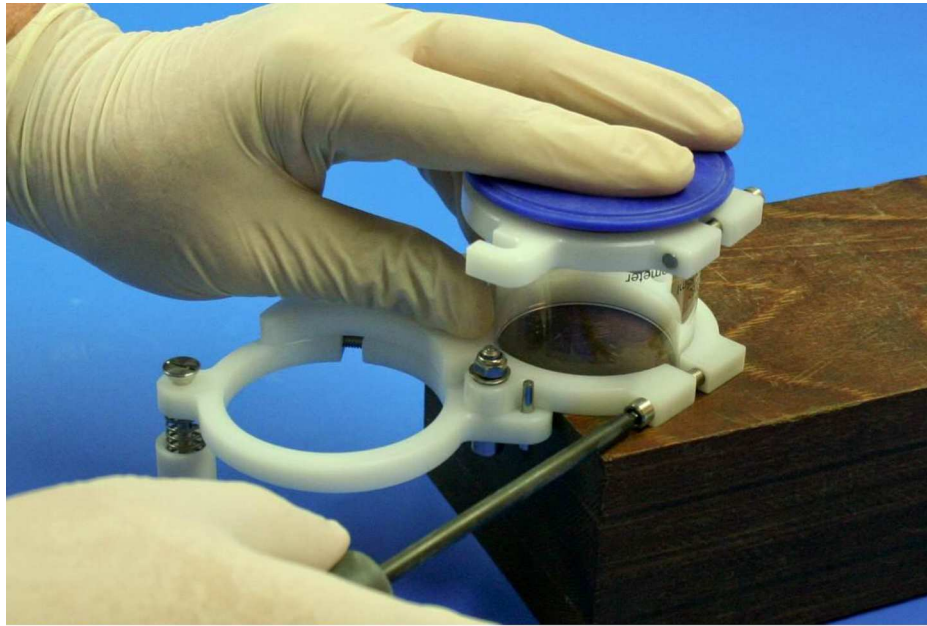


FIG. 5 Fitting the Clamp Ring to the Glass Cylinder Using the Ball-ended Hex Key

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.



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

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).

7.5 Locate the serrated base into the glass cylinder adjacent to the clamp ring. Carefully rotate the serrated base to make sure that the entire circumference is in contact with the glass cylinder (Fig. 6).

7.6 Open the leveling assembly and place it on top of the glass cylinder at the opposite end to the clamp ring and serrated base. Make sure that the gap in leveling assembly is approximately in line with the gap in the clamp ring.

7.7 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.

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

7.9 Tighten the leveling assembly with the hex driver such that the leveling assembly and the glass cylinder are securely located.

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





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

7.11 Close the leveling assembly.

7.12 Place the other 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.

7.13 Tighten the leveling assembly with the hex driver such that the leveling assembly and the upper glass cylinder are securely located (Fig. 8).

7.14 Place the funnel on top of assembled shear cell vessel (Fig. 9) and locate on the FT4 Powder Rheometer.

NOTE 4—The assembled shear cell vessel is described as a 50-mm × 85-mL split vessel assembly, which indicates the glass cylinders' internal diameter and the precise volume of the lower section of vessel with the base fitted.

## 8. Calibration and Standardization

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  $\pm 50$  N, and torque should be calibrated within the performance limits of  $\pm 900$  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 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.

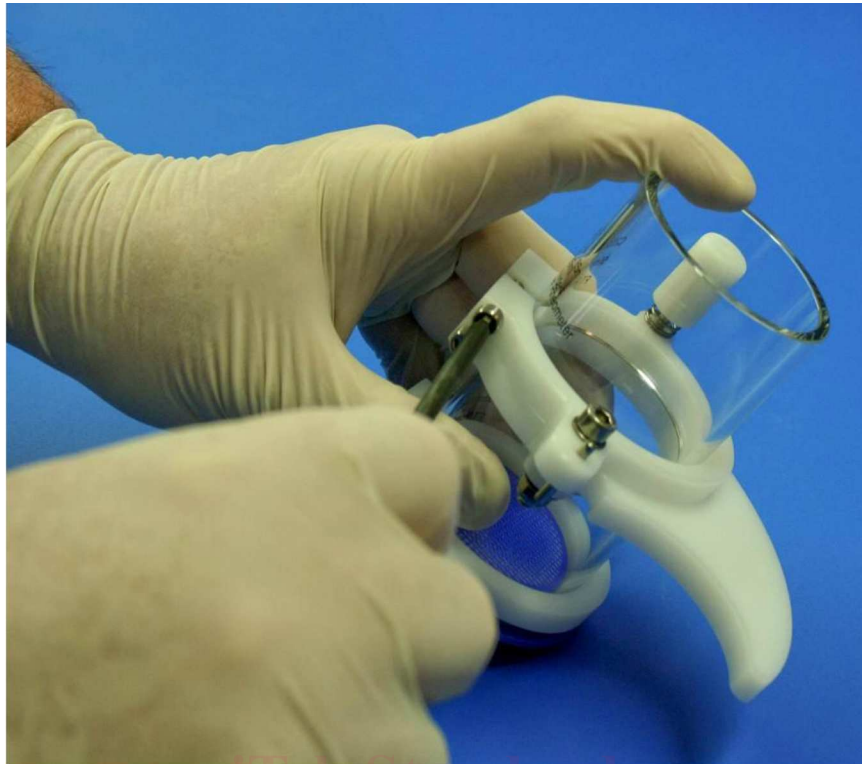


FIG. 8 Fitting the Upper Glass Cylinder



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