Designation: D 6682 – 01

Standard Test Method for
Measuring the Shear Stresses of Powders Using the Peschl Rotational Split Level Shear Tester[1]

This standard is issued under the fixed designation D 6682; 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 test method is applied to the measurement of the mechanical properties of powders as a function of normal stress.

1.2 This apparatus is suitable measuring the properties of powders and other bulk solids, up to a particle size of 5000 micron.

1.3 This method comprises four different test procedures for the determination of powder mechanical properties.

Test A—Measurement of INTERNAL FRICTION as a function of normal stress.
Test B—Measurement of WALL FRICTION as a function of normal stress.
Test C—Measurement of BULK DENSITY as a function of normal stress and time.
Test D—Measurement of DEGRADATION as a function of normal stress.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rocks, and Contained Fluids[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *adhesion*—shear stress between the wall sample and powder at a normal stress of zero.

3.1.2 *consolidation normal stress*—the maximal normal stress applied to the specimen for executing an yield locus.

3.1.3 *consolidation step*—shearing repeated under the consolidation normal stress until the shear stress reaches a maximum τ_m value followed by a steady state value τ_s. This step is performed before each shear step.

3.1.4 *degradation*—change of particle size as result of shearing.

3.1.5 *dynamic wall friction*—calculated from the measured normal stress and the steady state shear stress after certain shearing.

3.1.6 *dynamic yield locus*—line calculated from measured values of normal stress and steady values of the shear stress.

3.1.7 *peak shear stress* (τ_m)—maximum shear stress at the beginning of yield - at the transition between elastic and plastic deformation.

3.1.8 *pre-consolidation normal stress* (σ_{np})—normal stress applied during the first part of the test in order to densify the specimen.

3.1.9 *shear step*—shear after the consolidation step, performed under the normal stress which is equal to or lower than the consolidation normal stress, until the shear stress reaches the peak value followed by a steady state value τ_s.

3.1.10 *split level*—level between the bottom and top cover of the shear cell defined by the transition of the cell base and ring where in the specimen the shear plane occurs.

3.1.11 *static wall friction*—calculated from the measured normal stress and the maximum shear stress at the beginning of yield.

3.1.12 *static yield locus*—line calculated from measured values of normal stress and peak values of the shear stress.

3.1.13 *steady shear stress* (τ_s)—steady state shear stress during the steady state (plastic) deformation.

4. Summary of Test Method

4.1 *Measurement of Internal Friction as a Function of Normal Stress*:

NOTE 1—Sequence of a standard shear test (Fig. 3):
(*a*) The upper graph shows the change of normal stress as a function of time. Before each shear step, a consolidation normal stress σ_n is applied to the specimen, to reestablish the consolidation condition.
(*b*) The next graph shows the change of shear stress during the consolidation step and the shear step.
(*c*) The next graphic shows the expansion and contraction of the specimen during various test stages.
(*d*) The lowest graph shows the change of rotational movement of the shear tester as function of time.

[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 for Handling of Bulk Solids.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

F-normal consolidation force
R-radius for shear stress calculation
r - radius at a point of the cross section of the shear cell
L-radius on which the measured force is acting
T-measured force
τ-shear stress
M-moment of acting shear stresses

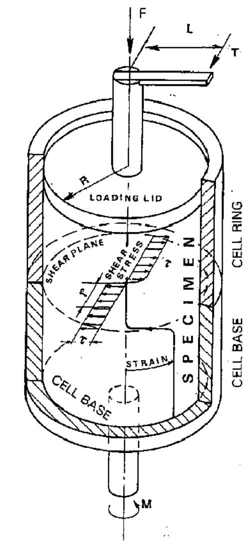


FIG. 1 Schematic View of a Rotational Split Level Shear Cell

4.1.1 For each individual test, the powder is compacted with the pre-consolidation normal stress. It is then pretreated by applying a shear stress until steady state is achieved. The shear stress is repeatedly applied and removed until consistent results are obtained. Next, the normal stress is reduced in steps. Before each shear step, the consolidation normal stress is reapplied. The measurements provide a measure of the instantaneous static and dynamic yield loci.

4.1.1.1 During the entire shear test the height of the specimen is measured simultaneously in order to determine the compaction and expansion of the specimen.

4.1.2 The instantaneous static and dynamic yield loci are determined using the procedure outlined in the above section without any delay between the various stages of the test.

4.1.3 The time dependent static yield locus is measured as a function of time by preconditioning the specimen for various times under consolidation normal stress conditions; the peak shear stress is then measured.

4.2 *Measurement of Wall Friction as a Function of the Normal Stress*—By placing a wall specimen under the cell ring, the shear stresses (wall friction) are measured between the wall specimen and the powder.

4.2.1 The instantaneous static and dynamic friction are determined.

4.3 *Measurement of Degradation as a Function of Normal Stress*—The influence of shearing on particle degradation is measured by particle size analysis after shearing the specimen at a predetermined normal stress. Particle size degradation is measured from the change of particle size distribution before and after test (see 10.4.3).

5. Significance and Use

5.1 The test method is useful for the following:

5.1.1 *Classification of Powders*—The cohesion and angle of internal friction are flowability indicators of powders and can be used to classify the powders.

5.1.2 *Quality Control*—For a number of industrial applications flowability factors are used to compare the material flowability at different times during production. The material produced has to be held within given limits for each application and each powder so as to ensure trouble- free operation.

5.1.3 *Material Engineering*—Powder properties are influenced by particle size, particle size distribution, fat content, humidity and other parameters. By selecting the correct parameters and the correct mixtures of powders, the required mechanical properties of the product are achieved.

5.1.4 *Design of Handling Equipment*—For certain storage and conveyor equipment there are mathematical models exist which require the mechanical properties of powders.

6. Apparatus [3]

6.1 The Rotational Split Level Shear tester is schematically shown in Fig. 1 and the specimen is contained in the following shear cell components.

6.1.1 *Cell Base*, is cylindrical and has a knurled interior bottom surface.

6.1.2 *Cell Ring*, is a ring-formed element to be placed on the cell base.

6.1.3 *Loading Lid*, is a knurled interior cover surface for loading of the specimen, to be placed on the specimen.

6.1.4 *Shear Plane*, shown in Fig. 1, occurs at the transition plane between the cell base and the cell ring.

[3] Available from Dr. I. Peschl, Post Box 399, NL-5600 AJ Eindhoven, The Netherlands.

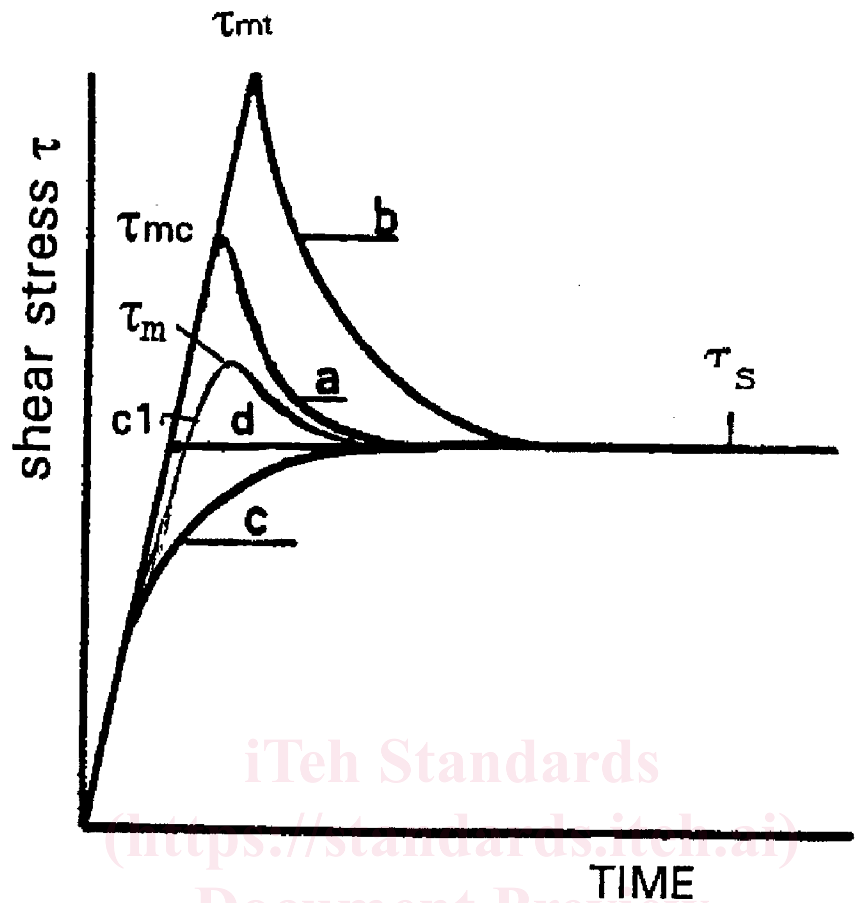


FIG. 2 Shear Resistance as Function of Time (Angular Rotation) in Relation to the Steady Shear Stress

6.1.5 Several shear cell sizes are available to accommodate a variety of particle sizes. The selected shear cell diameter should be at least 25 times larger than the average particle diameter. The most frequently used shear cell is a nominal 60 mm diameter and would accommodate powders with an average particle diameter smaller than 2400 microns.

6.2 *Rotating Table*, on which the Cell Base is fixed causes the Cell Base to rotate against the Loading Lid.

6.2.1 In Fig. 1, the cross section shows the cell base, ring and loading lid. The cell base rotates. The loading lid is placed on the specimen and loaded with predetermined weights. The shear resistance is measured by measuring the moment on the loading lid.

7. Selection of Test Parameters

7.1 *Sampling*:

7.1.1 Prepare and store the test specimens in accordance with any valid safety and environmental regulations.

7.1.2 Prepare the specimens in accordance with the operating conditions expected during the application; i.e. temperature, humidity and other conditions. Use an adequate climate chamber to condition the specimen as necessary.

7.1.3 If a powder contains large particles which are uniformly distributed in a mixture, which otherwise meets the criteria of 6.1.5, the large particles may be sieved out. It is acceptable to sieve out the large particles until the proportion of large particles does not exceed about 5 % of the test specimen. Beyond this limit a larger diameter of the shear cell should be used according 6.1.5 to retain the large particles in the mixture.

7.2 *Determination of Test Parameters*:

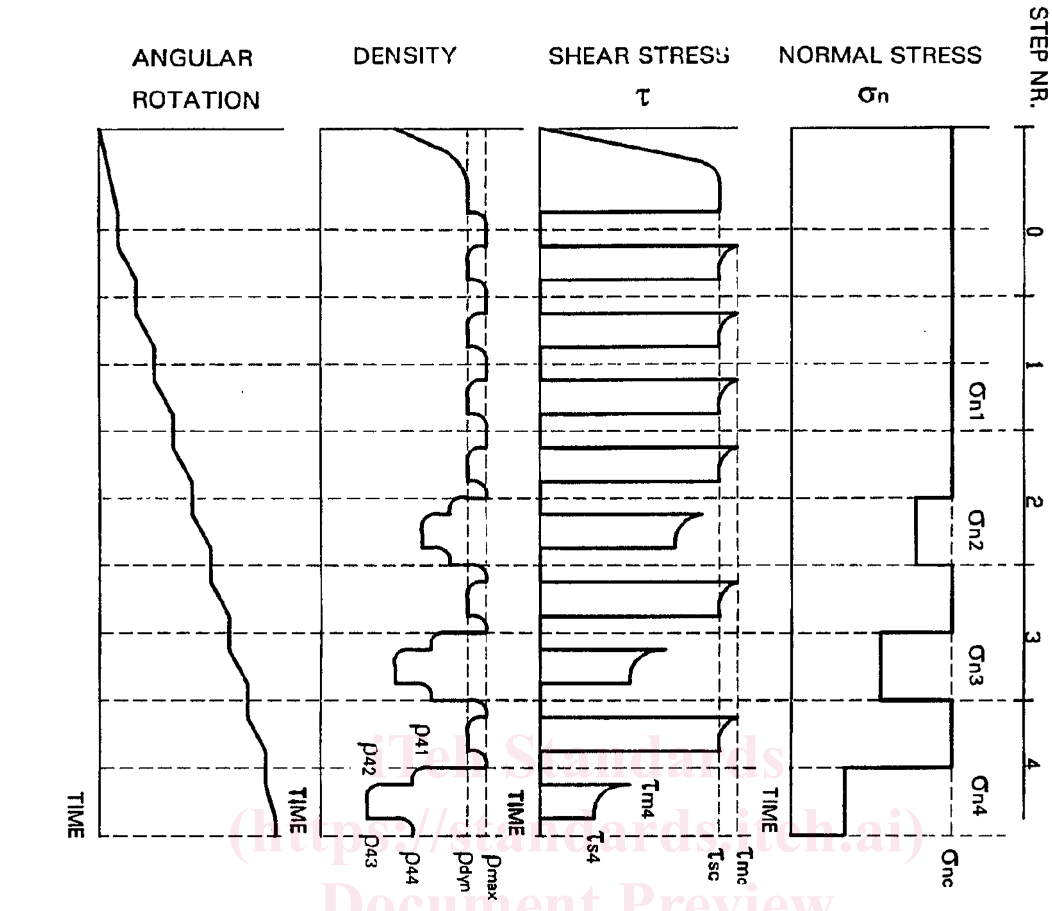
NOTE 2—The selected consolidation normal stress should match the expected stress in the actual process specified by an engineer/scientist having a knowledge of shear testing and a theoretical background.

7.2.1 For the measurement of internal friction, the consolidation normal stress is the same during both, the pre-consolidation σ_{np} and the consolidation steps σ_n. See Fig. 3.

7.2.1.1 The normal stress during the shear step is equal to or lower than the consolidation normal stress. See σ_{nx} in Fig. 3.

7.2.1.2 In the absence of specified values of consolidation normal stress the test should be performed with consolidation normal stress of 5.0 kPa, 15.0 kPa and 25.0 kPa.

7.2.1.3 In the absence of specified values of normal stress during the shear steps the test should be performed with normal stress equal to 100 %, 80 %, 60 %, 40 % and 20 % of the consolidation normal stress.



SHEAR STEP	SHEAR STRESS	EXPLANATION
0	σ_{nc}	consolidation normal stress during the consolidation step.
0	τ_{sc}	consolidation normal stress, during the consolidation step, brings the specimen to a constant density and shear stress.
0	τ_{sc}	repeatable peak after reaching constant shear stress during the consolidation step.
1-x	τ_{mc}	maximum shear stress during the consolidation step to be performed before each shear step.
1-x	τ_{sc}	steady state shear stress during the consolidation step to be performed before each shear step.
1-x	σ_{nx}	normal stress, equal or smaller then consolidation normal stress, during the shear step.
1-x	τ_{mx}	maximum shear stress during the shear step, to be performed after the consolidation step, by equal or decreased consolidation stress at begin of flow.
1-x	τ_{sx}	steady state shear stress during the flow by equal or decreased consolidation stress.
0	ρ_0	initial density after pre-consolidation
0-x	ρ_{max}	density after consolidation step
0-x	ρ_{dyn}	density during the shearing under consolidation stress.
1-x	ρ_{x1}	decrease density, during shear step, after change of normal stress for static condition.
1-x	ρ_{x2}	decreased density, during shear step, after change of normal stress for dynamic condition.
1-x	ρ_{x3}	increased density as result of change from dynamic to static condition.
x		Shear step Number

FIG. 3 Sequence of a Shear Test

7.2.2 The measurement of density is performed by applying the predetermined normal stress to the specimen (see 7.2.1). In the absence of specified values for normal stress, the test should be performed at 1 kPa, 5 kPa, 10 kPa, 15 kPa, 25 kPa. 15 kPa, 10 kPa, 5 kPa, and 1 kPa.

7.2.3 The measurement of wall friction is performed by applying the predetermined normal stress to the specimen. In the absence of specified values for normal stress, the test should be performed at 1 kPa, 10 kPa, 15 kPa and 25 kPa.

7.2.4 The degradation test should simulate the normal stress and time during which the shearing takes place. For a good simulation, a number of such steps might be necessary in order to simulate the stresses and the time during which they are acting throughout the whole process. In the absence of specified values for normal stress, the test should be performed at 5 kPa during 10 min.

8. Specimen Preparation for Measurement

8.1 *Preparation for the Measurement of the Internal Friction—Shear Test* (Fig. 5):

8.1.1 Place the shear cell ring on top of the cell base. Center the shear cell ring with the three centering screws.

8.1.2 Determine the mass of the empty shear cell including the shear cell ring. Use a scale with the accuracy of 0.1 gram.

8.1.3 Place the fill ring on top of the shear cell ring.

8.1.4 Fill the shear cell, as uniformly as possible, with powder to be tested. Use a sieve for filling the shear cell in order to remove lumps and agglomerates from the specimen.

8.1.5 Scrape off the surplus material in small amounts by scraping off with a blade as shown in Fig. 10. The blade should be scraped across the ring with a zigzag motion. Prevent downward forces from acting on the specimen.

8.1.6 Center the consolidation lid on top of the material in the shear cell.

8.1.7 Load the specimen uniaxially by placing weights on the consolidation lid so as to achieve a pre-consolidation normal stress corresponding to one of the predetermined consolidation normal stresses specified in 7.2.

8.1.8 Consolidate the powder with the predetermined pre-consolidation normal stress until the consolidation is completed. The time required to consolidate the specimen will vary with the material. Take 10 min for the first trial.

τ - shear stress

σ_1 - major principal stress

σ_3 - minor principal stress

σ_n - normal stress

σ_d - unconfined compressive strength for instantaneous yield locus

σ_{dt} - unconfined compressive strength for time yield locus

τ_{mt} - top shear stress for time consolidation

τ_{mx} - peak shear stress for instantaneous measurement

τ_{sx} - steady state shear stress during the steady state (plastic) deformation

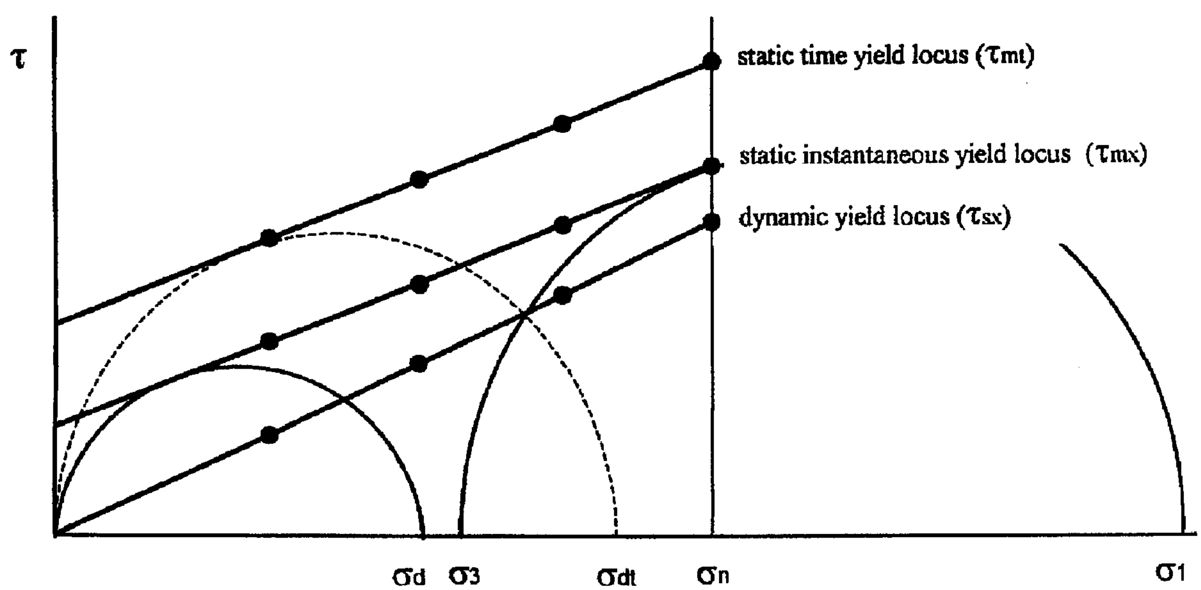


FIG. 4 Instantaneous and Time Yield Loci

1 Cell base
2 Shear cell ring
3 Fill ring
4 Centering screws
5 Consolidation lid
6 Weights

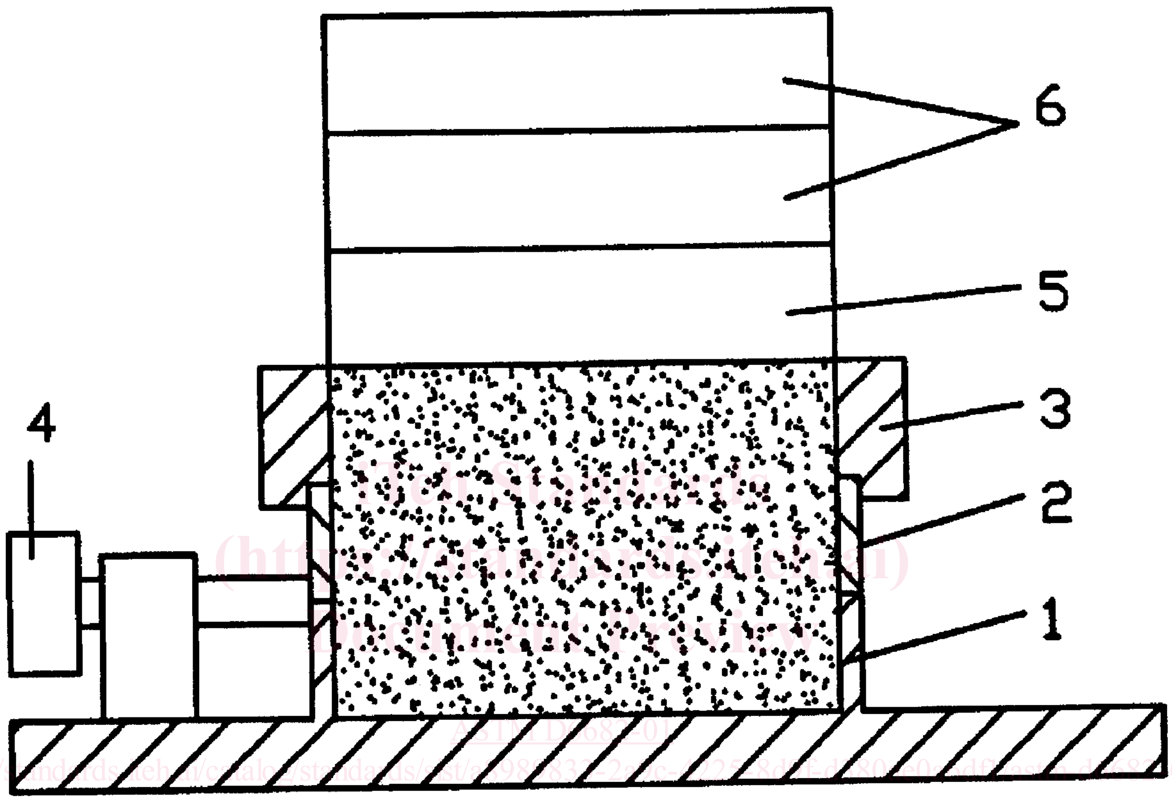


FIG. 5 Shear Cell Assembly for Filling

8.1.9 Remove the weights, consolidation lid and the fill ring.

8.1.10 Perform 8.1.5.

8.1.11 Determine the mass of the shear cell filled with powder.

8.1.12 Calculate the mass of material in the shear cell by subtracting the net value in 8.1.2 from value in 8.1.11.

8.1.13 Place the loading lid assembly on the cell base and tighten the three clamp screws.

8.2 *Preparation for Measurement of Wall Friction*:

8.2.1 Mount the specimen of wall material on the cell base and secure it with the centering screws as shown in Fig. 7.

8.2.2 Place the cell ring on the wall specimen.

8.2.3 Perform 8.1.4-8.1.10 and 8.1.13.

8.3 *Preparation for Measuring the Density* :

8.3.1 Perform 8.1.1-8.1.4.

8.3.2 Remove the fill ring.

8.3.3 Perform 8.1.5 and 8.1.11-8.1.13.

8.4 *Preparation for Measurement of Degradation*:

8.4.1 Perform a sieve analysis or particle size analysis, before running the degradation test.

8.4.2 Prepare the shear cell in accordance with 7.2.4.

8.4.3 Perform 8.1.1-8.1.13.

9. Procedures for Executing the Test

NOTE 3—The procedures are similar for carrying out manual and automatic testers. Both shear testers can be controlled by hand or by computer, only in the case of the manual shear tester should the weight be changed manually.

9.1 *Mount the Shear Cell on the Turntable of the Shear Tester*:

9.1.1 Place the shear cell assembly on the shear tester as shown in Fig. 6.

9.1.2 Tighten the clamp screws of the turntable.

9.1.3 Loosen the three centering screws which center the cell ring on the cell base.

9.2 *Measurement of the Internal Friction as a Function of Normal Stress and Time*:

NOTE 4—The number and value of the shear steps for one yield locus, should be determined by an engineer in accordance with 7.2.