Designation: D 5891 – 02

# Standard Test Method for Fluid Loss of Clay Component of Geosynthetic Clay Liners[1]

This standard is issued under the fixed designation D 5891; 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 covers an index method that enables the evaluation of fluid loss properties of a clay mineral film deposited on a filter paper from a 6 % solids slurry of clay mineral at 100-psi (-kPa) pressure as a measure of its usefulness for permeability or hydraulic conductivity reduction in geosynthetic clay liners (GCL).

1.2 This test method is adapted from American Petroleum Institute drilling fluid specifications for bentonite.

1.3 Powdered clay mineral is tested as produced; granular clay mineral should be ground to 100 % passing a 100 mesh U.S. Standard Sieve with a minimum of 65 % passing a 200 mesh U.S. Standard Sieve with the whole ground product used for testing.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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]

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[3]

E 1 Specification for Thermometers[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

E 725 Test Method of Sampling Granular Carriers and Granular Pesticides[6]

2.2 *API Standards:*

API RP 131, Recommended Practice for Laboratory Testing of Drilling Fluids[7]

API Specification 13A, 4, 14th ed. for Drilling Fluid Materials[7]

## 3. Terminology

3.1 *Definitions*— For definitions of terms used in this test method, refer to API Standards and ASTM definitions for GCL products.

## 4. Significance and Use

4.1 Clay mineral is the functional component of GCL that reduces the hydraulic conductivity of industrial waste or ground water through the liner.

4.2 Clay mineral quality can vary significantly and effect the hydraulic conductivity of the GCL composite. This test method evaluates a significant property of clay mineral that relates to performance.

## 5. Apparatus

5.1 *Laboratory Balance*, 100 g capacity, ±0.01-g accuracy and precision.

5.2 *Weighing Paper*, or small weighing dish.

5.3 *Graduated Cylinder*, 500 ± 5-mL graduated TD (to deliver) with 10-mL subdivisions, Class A volumetrically calibrated; 10 ± 0.1-mL graduated cylinder, graduated TC (to contain) with 0.1-mL subdivisions.

5.4 *U.S. Standard Sieve*, 100 mesh, 200 mesh, and automated sieve shaker.

5.5 *Mortar and Pestle or Laboratory Hammer Mill*, for grinding clay mineral to required particle sizing.

5.6 *ASTM Calibration Immersion Thermometer*, 0 to 105 ± 0.5°C (see Specification E 1).

5.7 *Mixer*—11 000 ± 300 rpm under load with single sine-wave impeller approximately 25 mm (1.0 in.) in diameter[8] (mounted flash side up). The impeller shall be replaced when it weighs a minimum of 5.1 g, from an original weight of about 5.5 g. New blades will be weighed prior to installation in order to ensure conformance to manufacturing criteria. Mixer speed

[1] This test method is under the jurisdiction of ASTM Committee D35 on Geosynthetics and is the direct responsibility of Subcommittee D35.04 on Geosynthetic Clay Liners.

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[2] When bentonite is removed from a GCL product for testing, it may include adhesives that can influence test results.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] *Annual Book of ASTM Standards*, Vol 04.07.

[7] Available from American Petroleum Institute, 1801 K St. N.W., Washington, DC 20226.

[8] For example, Sterling Multimixer Model 9B with 9B29X impeller blades available from Fann Instrument Co., P.O. Box 4350, Houston, TX 77210, has been found suitable for this purpose.

under sample loading shall be determined and documented once every 90 days unless the manufacturer has documented objective evidence to extend calibration time.

NOTE 1—Sterling Multimixer Model 9B with 9B29X impeller blades or equivalent may be obtained from the suppliers given in Footnote 9.

5.8 *Mixing Container*—Approximate dimensions are 180 mm (7 in.) deep, 97-mm (3 13⁄16-in.) inner diameter at top, and 70-mm (2¾-in.) inner diameter at bottom.[9]

NOTE 2—Mixing containers or equivalent may be obtained from the suppliers given in Footnote 8.

5.9 *Timers*, 30 min, two interval, mechanical or electrical, precision ±0.1 min.

5.10 *Spatula*, flat blade, to dislodge clay mineral clumps adhering to the mixing container walls.

5.11 *Covered or Sealed Container*, of 400 to 600-mL capacity.

5.12 *Ambient Temperature/Low-Pressure Filter Press*, conforming to API RP 13B-1, Section 3.2. This filter press consists mainly of a cylindrical cell having an inside diameter of 76.2 mm (3 in.) and a height of at least 64.0 mm (2.5 in.). This chamber is made of materials resistant to strongly alkaline solutions, and is so fitted that a pressure medium can be conveniently admitted into and bled from the top. Arrangement is also such that a sheet of 90-mm filter paper can be placed in the bottom of the chamber just above a suitable support. The filtration area is 4580 ± 60 $mm^2$ (7.1 ± 0.1 $in.^2$). Below the support is a drain tube for discharging the filtrate into a graduated cylinder. Sealing is accomplished with gaskets, and the entire assembly supported by a stand. A mini-press or half-area press does not directly correlate with the results obtained when using the above described standard-sized press. Pressure can be applied with any nonhazardous fluid medium, either gas or liquid. Presses are equipped with pressure regulators and can be obtained with portable pressure cylinders, midget pressure cartridges, or means of utilizing hydraulic pressure.

NOTE 3—Ambient temperature/low-pressure filter press conforming to API RP 13B-1, Section 3.2, or equivalent, may be obtained from the suppliers given in Footnote 9.

5.13 *Filter Paper, 90-mm*, very dense, hardened with smooth lint-free surface,[10] must be used. These papers have high wet strength permitting application of high pressure during filtration. They also have good resistance to alkalies and acids.

## 6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type I, II, or III. Such water is best prepared by distillation or the passage of tap water through an ion-exchange resin.

6.2 Specification D 1193 for reagent water, Type I, II, or III.

## 7. Hazards

7.1 *Safety Precautions*—Establish appropriate safety and health practices for high-pressure equipment prior to use.

## 8. Sampling and Selection

8.1 Conduct the sampling in accordance with Test Method E 725.

## 9. Procedure

9.1 Grind the clay mineral sample to greater than 100 % passing a 100 mesh U.S. Standard Sieve, and a minimum of 65 % passing a 200-mesh U.S. Standard Sieve with a mortar and pestle or laboratory hammer mill as required.

9.2 Weigh 22.50 ± 0.01 g of the whole composite of finely ground clay mineral with "as received" moisture, typically 5 to 10 %, onto a weighing paper. If bentonite is removed from a GCL product, the bentonite would be dried to less than 10 % moisture prior to weighing.

9.3 Measure 350 ± 5 mL of reagent water with the 500- mL graduated cylinder and added to the mixing cup. Place the cup on the mixer, and add the clay mineral slowly over approximately 30 s.

9.4 After stirring for 5 ± 0.5 min, remove the container from mixer, and scrape its sides with the spatula to dislodge any clay clinging to the container wall. Ensure that all of the dislodged clay mineral clinging to the spatula is incorporated into the suspension.

9.5 Replace the container on the mixer, and continue to stir for a cumulative total stirring time of 20 ± 0.1 min. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 or 10 min of stirring.

9.6 Age the clay mineral suspension for a minimum of 16 h in a sealed or covered container at ambient temperature. Record the initial temperature, final temperature, and actual hydration aging time.

9.7 After aging the clay mineral suspension, shake vigorously to break its gel strength, and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 ± 0.5 min to completely disperse the clay mineral slurry.

9.8 Assemble the dry filter cell with filter paper and gaskets, and immediately after remixing the clay mineral slurry, pour it into the filter cell and complete assembly of the filter cell. Place the filter cell in the filter frame and close the relief valve. Place a 10 mL graduated cylinder under the filter cell drain tube.

9.9 Set one timer for 7.5 ± 0.1 min and the second timer for 30 ± 0.1 min. Start both timers and adjust pressure on the fluid loss cell to 100 ± 2 psi. Starting the timers and adding 100 psi pressure should be completed in less than 15 s. Supply pressure by compressed air, nitrogen, helium, or carbon dioxide.

9.10 At 7.5 ± 0.1 min on the first timer, remove the graduated cylinder and any adhering liquid on the drain tube, and discard. Immediately place a clean dry 10-mL graduated cylinder under the drain tube, and collect the fluid for 22.5 ± 0.1 min to the end of the second timer. This corrects the fluid loss value for any initial unpredictable spurt loss from the fluid loss cell. Remove the graduated cylinder after the second time

[9] For example, Hamilton Beach Mixer Cup No. M110-D, or equivalent, has been found suitable for this purpose. Mixing containers supplied by Fann Instrument Co., P.O. Box 4350, Houston, TX 77210.

[10] For example, Whatman No. 50, S & S No. 576, or equivalent, have been found suitable for this purpose.

interval and record the volume of fluid collected.

## 10. Calculation

10.1 Calculate the fluid loss in millilitres using Eq 1:

$$\textit{Fluid loss volume} = 2(\text{mL filtrate volume for last 22.5 min. interval}) \text{ mL} \quad (1)$$

## 11. Report

11.1 Report the following information:

11.1.1 Source of clay mineral, including sample identification or lot number,

11.1.2 Method of sampling used,

11.1.3 ASTM Test Method number used to perform the test,

11.1.4 Any modifications to the test method or unusual observations which may effect the test results,

11.1.5 Calculated fluid loss as millilitres to the nearest 0.1 mL, and

11.1.6 Temperature of the slurry at the start and completion of the test to the nearest 0.5°C.

## 12. Precision and Bias

12.1 *Interlaboratory Test Program*—An interlaboratory study of the test method was run in 1999. The design of the experiment, similar to that of Practice E 691. Seven different clay mineral samples were distributed to seven laboratories. Three sets of test results were generated for each sample by each of the laboratories.

12.2 *Test Results*—The precision information is given in Table 1. The average fluid loss values ranged from 9 to 22 for the seven clay mineral samples tested. However, since the statistics were not related to the magnitude of the test result, the precision values have been presented in terms of coefficients of varaiation, CV %.

**TABLE 1 Test Results**

| Statistic | ILS Range |
|---|---|
| Within laboratory repeatability limit, CV %$^r$ | 1.8 to 4.7 % |
| Between laboratory reproducibility limit, CV %$^R$ | 6 to 18 % |
| 95 % confidence limit | 5 to 13 % |
| Within laboratory repeatability, 2.8 CV %$^r$ | |
| 95 % confidence limit | 11.8 to 51 % |
| Between laboratory reproducibility, 2.8 CV %$^R$ | |

12.3 *Bias*—The procedure in Test Method D 5891 for measuring the fluid loss value of the clay mineral component of geosynthetic clay liners has no bias because the values of swell index can be defined only in terms of this test method.