Designation: D 6527 – 00

Standard Test Method for Determining Unsaturated and Saturated Hydraulic Conductivity in Porous Media by Steady-State Centrifugation[1]

This standard is issued under the fixed designation D 6527; 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 the determination of the hydraulic conductivity, or the permeability relative to water, of any porous medium in the laboratory, in particular, the hydraulic conductivity for water in subsurface materials, for example, soil, sediment, rock, concrete, and ceramic, either natural or artificial, especially in relatively impermeable materials or materials under highly unsaturated conditions. This test method covers determination of these properties using any form of steady-state centrifugation (SSC) in which fluid can be applied to a specimen with a constant flux or steady flow during centrifugation of the specimen. This test method only measures advective flow on core specimens in the laboratory.

1.2 *This standard may involve hazardous materials, operations, and equipment. 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 420 Guide to Site Characterization for Engineering, Design, and Construction Purposes[2]

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

D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock[2]

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction[2]

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Testing Soil, Rock, and Related Construction Materials[2]

D 5084 Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter[2]

D 5730 Guide for Site Characterization for Environmental Purposes With Emphasis on Soil, Rock, the Vadose Zone, and Ground Water[3]

D 6026 Practice for Using Significant Digits in Calculating and Reporting Geotechnical Test Data[3]

3. Terminology

3.1 *Definitions*—For common definitions of terms in this guide, such as porosity, permeability, hydraulic conductivity, water content, and matric potential (matric suction, water suction, or water potential), refer to Terminology D 653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *hydraulic steady state*—the condition in which the water flux density remains constant along the conducting system. This is diagnosed as the point at which both the mass and volumetric water contents of the material are no longer changing.

3.2.2 *SSCM or SSC-UFA*—Apparatus to achieve steady-state centrifugation. The SSCM (steady-state centrifugation method) uses a self-contained flow delivery-specimen system **(1)**[4]. The SSC-UFA (unsaturated flow apparatus) uses an external pump to deliver flow to the rotating specimen **(2)**. This test method will describe the SSC-UFA application, but other applications are possible. Specific parts for the SSC-UFA are described in Section 6 as an example of a SSC system.

3.2.3 *steady-state centrifugation*—controlled flow of water or other fluid through a specimen while it is rotating in a centrifuge, as distinct from water retention centrifugation methods which measure drainage from a wet specimen by centrifugation with no flow into the specimen.

3.2.4 *water flux density*—the flow rate of water through a cross-sectional area per unit time, for example, 5 $cm^3/cm^2/s$, written as 5 cm/s.

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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 D18.21 on Ground Water and Vadose Zone Investigations.

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

[3] *Annual Book of ASTM Standards*, Vol 04.09.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

3.3 *Symbols:*

K = hydraulic conductivity, cm/s
q = water flux density, $cm^3/cm^2/s$ or cm/s
r = distance from axis of rotation, cm
ρ = dry density, g/cm^3
ω = rotation speed, radians/s

4. Summary of Test Method

4.1 Using a SSC-UFA is effective because it allows the operator to control the independent variables in Darcy's Law. Darcy's Law states that the water flux density equals the hydraulic conductivity times the fluid driving force (See Section 11). The driving force is fixed by imposing an acceleration on the specimen through an adjustable rotation speed. The water flux density is fixed by setting the flow rate into the specimen with an appropriate constant-flow pump and dispersing the flow front evenly over the specimen. Thus, the specimen reaches the steady-state hydraulic conductivity which is dictated by that combined water flux density and driving force. The operator can impose whatever hydraulic conductivity is desired within the operational range of rotation speeds and flow rates, from 10^{-4} cm/s (0.l darcy; 10^{-9} cm^2) to 10^{-11} cm/s (10^{-8} darcy; 10^{-16} cm^2). Higher conductivities are measured using falling head or constant head methods (**3**). These methods are also convenient to saturate the specimen. Following saturation and constant or falling head measurements, the specimen is stepwise desaturated in the SSC-UFA by increasing the speed and decreasing the flow rate, allowing steady state to be reached at each step. Because a relatively large driving force is used, the SSC-UFA can achieve hydraulic steady state in a matter of hours for geologic materials, even at very low water contents. Sample size is up to about 5-cm diameter and 6-cm length cores. This test method is distinct from water retention centrifugation methods which measure simple drainage from a wet specimen by centrifugation with no flow into the specimen. Hydraulic steady state cannot be achieved without flow into the specimen.

5. Significance and Use

5.1 Recent results have demonstrated that direct measurements of unsaturated transport parameters, for example, hydraulic conductivity, vapor diffusivity, retardation factors, thermal and electrical conductivities, and water potential, on subsurface materials and engineered systems are essential for defensible site characterization needs of performance assessment as well as restoration or disposal strategies. Predictive models require the transport properties of real systems that can be difficult to obtain over reasonable time periods using traditional methods. Using a SSC-UFA greatly decreases the time required to obtain direct measurements of hydraulic conductivity on unsaturated systems and relatively impermeable materials. Traditionally, long times are required to attain steady-state conditions and distributions of water because normal gravity does not provide a large enough driving force relative to the low conductivities that characterize highly unsaturated conditions or highly impermeable saturated systems (Test Method D 5084). Pressure techniques sometimes can not be effective for measuring unsaturated transport properties because they do not provide a body force and cannot act on the entire specimen simultaneously unless the specimen is saturated or near-saturated. A body force is a force that acts on every point within the system independently of other forces or properties of the system. High pressures used on saturated systems often induce fracturing or grain rearrangements and cause compaction as a result of high-point stresses that are generated within the specimen. A SSC-UFA does not produce such high-point stresses.

5.2 There are specific advantages to using centrifugal force as a fluid driving force. It is a body force similar to gravity and, therefore, acts simultaneously over the entire system and independently of other driving forces, for example, gravity or matric potential. Additionally, in a SSC-UFA the acceleration can dominate any matric potential gradients as the Darcy driving force. The use of steady-state centrifugation to measure steady-state hydraulic conductivities has recently been demonstrated on various porous media (**1,2**).

5.3 Several issues involving flow in an acceleration field have been raised and addressed by previous and current research (**1,4**). These studies have shown that compaction from acceleration is negligible for subsurface soils at or near their field densities. Bulk densities in these specimens have remained constant (± 0.1 g/cm^3) because the specimens are already compacted more than the acceleration can affect them. The notable exception is structured soils. Special arrangements must be made to preserve their densities, for example, the use of speeds not exceeding specific equivalent stresses. As an example, for most SSC-UFA specimen geometries, the equivalent pressure in the specimen at a rotation speed of 2500 rpm is about 2 bar. If the specimen significantly compacts under this pressure, a lower speed must be used. Usually, only very fine soils at dry bulk densities less than 1.2 g/cm^3 are a problem. Whole rock, grout, ceramics, or other solids are completely unaffected by these accelerations. Precompaction runs up to the highest speed for that run are performed in the SSC-UFA prior to the run to observe any compaction effects.

5.4 Three-dimensional deviations of the driving force as a function of position in the specimen are less than a factor of two. Theoretically, the situation under which unit gradient conditions are achieved in a SSC-UFA, in which the change in the matric potential with radial distance equals zero ($d\psi/dr = 0$), is best at higher water flux densities, higher speeds, or coarser grain-size, or combination thereof. This is observed in potential gradient measurements in the normal operational range where $d\psi/dr = 0$. The worst case occurs at the lowest water flux densities in the finest-grained materials (**1**).

5.5 There is no sidewall leakage problem in the SSC-UFA for soils. The centrifugal force maintains a good seal between the specimen and the wall. As the specimen desaturates, the increasing matric potential (which still operates in all directions although there is no potential gradient) keeps the water within the specimen, and the acceleration (not being a pressure) does not force water into any larger pore spaces such as along a wall. Therefore, capillary phenomena still hold in the SSC-UFA, a fact which is especially important for fractured or heterogeneous media (**2**). Cores of solid material such as rock or concrete, are cast in epoxy sleeves as their specimen holder, and this also prevents sidewall leakage.

5.6 The SSC-UFA can be used in conjunction with other methods that require precise fixing of the water content of a porous material. The SSC-UFA is used to achieve the steady-state water content in the specimen and other test methods are applied to investigate particular problems as a function of water content. This has been successful in determining diffusion coefficients, vapor diffusivity, electrical conductivity, monitoring the breakthrough of chemical species (retardation factor), pore water extraction, solids characterization, and other physical or chemical properties as functions of the water content **(2,5)**.

5.7 Hydraulic conductivity can be very sensitive to the solution chemistry, especially when specimens contain expandable, or swelling, clay minerals. Water should be used that is appropriate to the situation, for example, ground water from the site from which the specimen was obtained, or rainwater if an experiment is being performed to investigate infiltration of precipitation into a disposal site. Appropriate antimicrobial agents should be used to prevent microbial effects within the specimen, for example, clogging, but should be chosen with consideration of any important chemical issues in the system. A standard synthetic pore water solution, similar to the solution expected in the field, is useful when it is difficult to obtain field water. Distilled or deionized water is generally not useful unless the results are to be compared to other tests using similar water or is specified in pertinent test plans, ASTM test methods, or EPA procedures. Distilled water can dramatically affect the conductivity of soil and rock specimens that contain clay minerals, and can induce dissolution/precipitation within the specimen.

5.8 This test method establishes a dynamic system, and, as such, the steady-state water content is usually higher than that which is attained during a pressure plate or other equilibrium method that does not have flow into the specimen during operation. This is critical when using either type of data for modeling purposes. This test method does not measure water vapor transport or molecular diffusion of water, both of which become very significant at low conductivities, and may actually dominate when hydraulic conductivities drop much below 10^{-10} cm/s.

5.9 The quality of the result produced by this test method depends upon the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing and sampling. Users of this test method are cautioned that compliance with Practice D 3740 does not in itself ensure reliable results. Reliable results depend on many factors; Practice D 3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 A SSC-UFA instrument consists of an ultracentrifuge with a constant, ultralow flow pump that provides water to the specimen surface through a rotating seal assembly and micro-dispersal system. An example of a rotor and seal assembly is shown in Fig. 1. Fig. 2 shows an actual SSC-UFA apparatus. This commercially available SSC-UFA can reach accelerations of up to 20 000 g (soils are generally run only up to 1 000 g), temperatures can be adjusted from –20 to 150°C. Infusion and syringe pumps can provide constant flow rates as low as 0.001 mL/h. Effluent from the specimen is collected in a transparent, volumetrically calibrated chamber at the bottom of the specimen assembly. Using a strobe light, an observer can check the chamber while the specimen is being centrifuged. Two specimens are run at the same time in a SSC-UFA with water flowing into each by means of two feedlines, the central feed or inlet path, and the annular feed. Specific parts are defined as follows (see Fig. 1):

6.1.1 *Specimen Holder*—The metal, polysulfone, fiberglass, or epoxy shell that contains the soil, rock, cement, or aggregate specimen to be tested.

6.1.2 *Specimen Cup*—The metal canister that contains the specimen holder. It has a dispersion cap that disperses flow evenly across the top of the specimen. O-ring seals prevent water flow around the sides of the specimen holder. The bottom of the specimen cup has a cone-shaped spacer that holds the bottom of the holder horizontal and allows effluent to drain out of the specimen cup.

6.1.3 *Bucket*—The metal shell that holds the specimen cup and screws into the rotor.

6.1.4 *Effluent Collection Chamber*—The plastic graduated vessel at the end of the specimen cup that collects the effluent as it exits the specimen cup.

6.1.5 *Rotor*—The central aluminum fixture that holds the specimen and bucket and spins on the rotating shaft. Most SSC-UFAs have rotors that hold three specimen sizes: a 3.33-cm diameter specimen, a 4.44-cm diameter specimen, and a 20-in. Shelby Tube-sized specimen.

6.1.6 *Rotating Seal*—The mechanism which connects the stationary exterior of the system to the rotating interior; the boundary through which the two fluids must pass from the external pumps to the rotating specimens. The components are usually composed of TFE-fluorocarbon/graphite polymers and sintered graphite, traditional bearing assemblies, and heat sinks.

6.2 Parts can be made of different materials, for example, TFE-fluorocarbon, titanium, stainless steel, copolymers, and nylon, to address the many chemical compatibility requirements. Each rotor and seal assembly comes preconfigured and the operator does not need to configure any part of a SSC-UFA as part of this test method.

6.3 Materials can be run in a SSC-UFA as recomposited specimens or as minimally disturbed samples subcored directly into the specimen SSC-UFA holder from trench, outcrops, or drill cores. Whole rock cores and cores of ceramics, grouts, and other solids are cast in an appropriate epoxy sleeve for use in a SSC-UFA (see Section 7).

6.4 In addition to the SSC-UFA instrument, other apparatus are necessary for specimen preparation and handling of soils, rock, aggregate, concrete, and other porous media (see Section 7). However, once the specimen is prepared, all that is needed is a balance accurate to ±0.01 g for determining the mass of the specimen at each steady-state point for water content determination (see Test Method D 2216) and an oven for drying the specimen after the final point to obtain the dry mass.

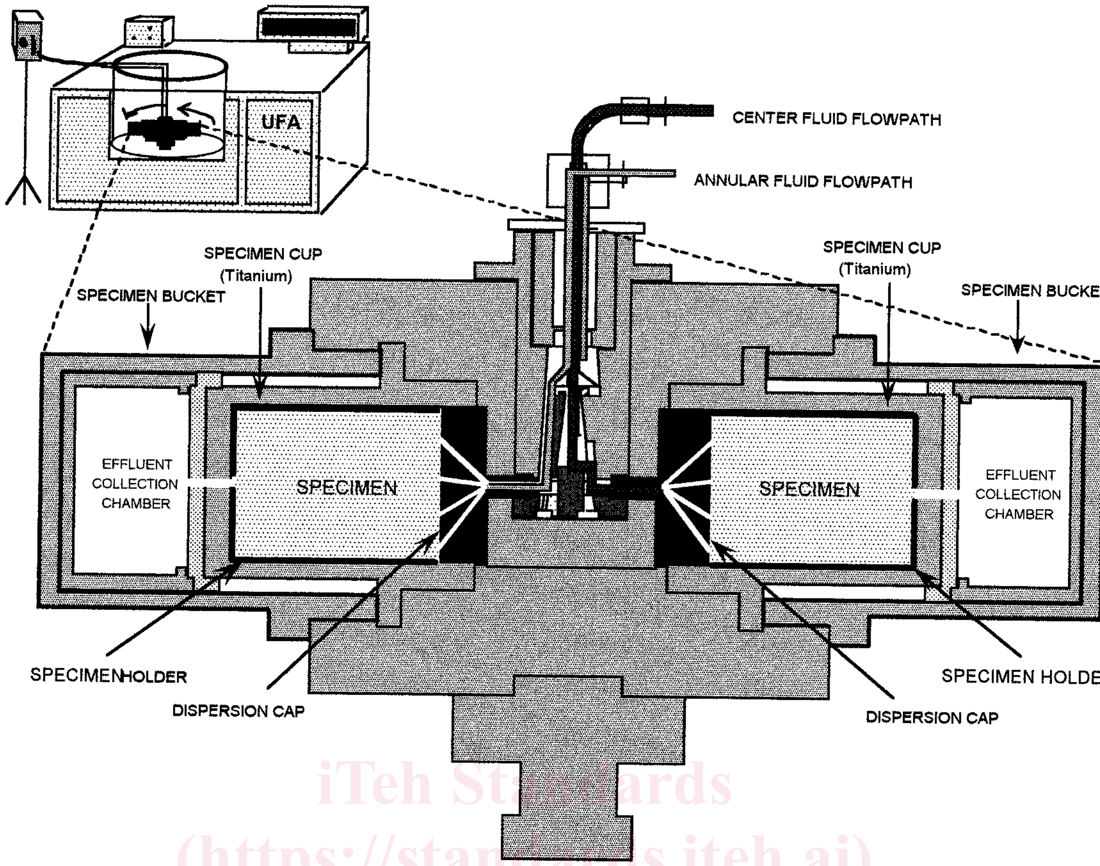

FIG. 1 SSC-UFA Rotor with Seal Assembly

Some kind of dust-free wipes, clean brushes for cleaning threads, various spoons and spatulas, squeeze bottles, distilled water for cleaning, and other basic laboratory implements are essential for smooth operation. As with any precision instrument, it is important to keep the area clean and dirt-free because grit can wear or destroy certain moving parts in a SSC-UFA. The SSC-UFA comes with the specific tools necessary for operation, for example, spanner wrenches of the correct dimensions.

7. Specimen Preparation

7.1 *Soil and Disaggregated Materials*—Depending upon the specific investigation, specimens are obtained in many ways (Guides D 420 and D 5730). The best possible sampling is to subcore the outcrop, trench, or undisturbed specimen directly into a SSC-UFA specimen holder using a subcoring device that holds a SSC-UFA specimen holder. Often, however, undisturbed samples are not available and the specimen must be recomposited or reassembled into a form that is representative of the field conditions. Soil scientists have developed numerous methods for preparing recomposited soils for flow tests (**3**). Two useful methods for use in a SSC-UFA are; (*1*) fill and tamp, which works best damp with fine to medium soils and with expandable clays, and (*2*) slurry, which works best wet with silts and non-expandable clays. These methods usually result in dry bulk densities between 1.4 and 1.6 g/cm^3 for most soils and sediments, and between 1.0 and 1.4 g/cm^3 for clay-rich soils. For higher densities, an hydraulic press can be used with an appropriately sized piston and confining cylinder for the specimen holder. Centrifugation in the SSC-UFA will generally not affect the dry density for specimens that are already within 0.2 g/cm^3 of their field dry density, or about 1.4 g/cm^3 and above for most soils. If compaction is a problem, lower rotation speeds must be used. The maximum speed can be decided by running the specimen at progressively higher speeds until compaction becomes unacceptable. However, the specimen must be at the desired dry density before running. Alternatively, the SSC-UFA can be used to compact the specimen to the desired dry density by running at progressively higher speeds until the target dry density is achieved which will also define the maximum speed. Because each run is fast, iteration to determine dry density and maximum speeds is rapid.

NOTE 1—The dispersion cap does not rest on the specimen top nor does it follow the specimen down if it compacts.

7.1.1 Other established soil sampling and recompositing methods can also be used. The following recompositing methods are just two simple methods that have been used to achieve specimen densities and form similar to the field for many types of specimens. While this test method does not include a detailed method for specimen recompositing, recently the ASTM/ISR Reference Soils and Testing Program has developed a detailed test protocol to prepare fill and tamp specimens.

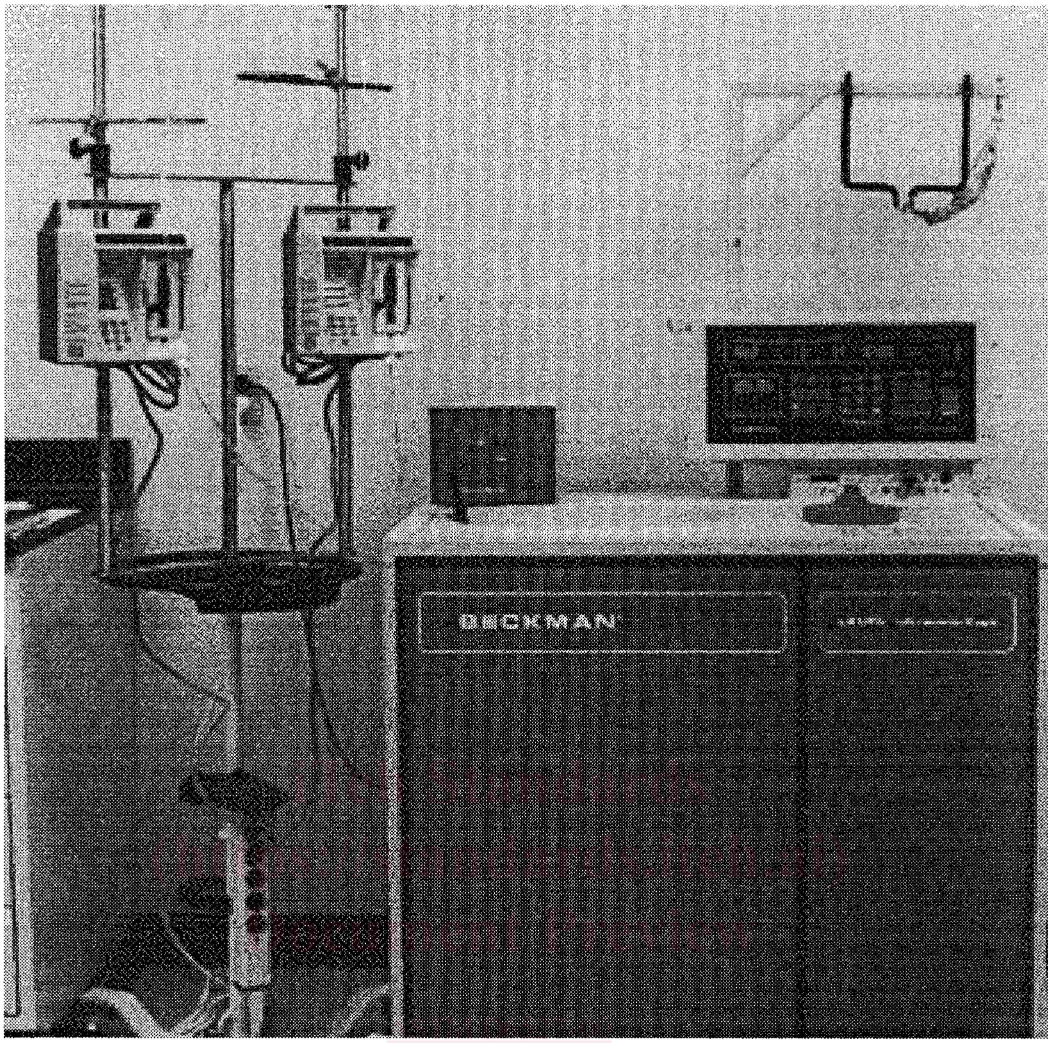

FIG. 2 SSC-UFA with Infusion Pumps

7.1.2 *Fill and Tamp*—Clean the specimen holder and rinse with distilled water and dry. Place filter paper in bottom of specimen holder. Determine the mass of the specimen holder and filter paper and record on the data sheet. Check the soil specimen number against the data sheet. Place a folded paper towel or wipe under the specimen holder to absorb excess water. Carefully spoon small amounts of the dry soil into the specimen holder, tamp down the soil firmly, by hand with a 1-kg piston, and add enough water to dampen but not saturate. Continue this process until the specimen holder is full with damp, well-compacted soil. Wipe off the top and sides carefully to clear away any grit that might damage the holder O-ring or cap threads.

7.1.3 *Slurry*—Rinse the specimen holder with distilled water and dry. Place filter paper in the bottom of the specimen holder. Determine the mass of the specimen holder and filter paper and record on the data sheet. Check the soil specimen number against the data sheet. Spoon an appropriate volume of dry specimen into a jar. Add enough water to just saturate the specimen and stir thoroughly. Place a folded paper towel or wipe under the specimen holder to absorb excess water and carefully mix and spoon soil mixture into the specimen holder, constantly mixing to ensure homogeneity and reduce layering, and periodically tamping mixture down firmly. When the specimen holder is full, let the specimen settle as it loses water slowly. Add additional wet soil to the specimen holder as necessary to top it off. Carefully wipe the top and sides of the specimen holder to clean off any grit that might damage the holder O-ring or cap threads. Determine the maximum speed or compaction density by spinning the specimen up to various speeds and observing what, if any, compaction occurs. Recording the amount of dry soil added to the specimen holder, and knowing the holder volume, allows tracking of the density. If any changes occur. The final bulk dry density is determined after the run by drying the specimen to determine the dry mass, and dividing this mass by the specimen volume.

7.2 *Whole Rock Cores, Concrete, or Ceramics*—Solid coherent materials can be cored using a coring bit, usually diamond, that produces cylinders that can be potted in a mold with the correct interior diameter using an appropriate epoxy for the intended test. The core of the material itself cannot be greater than 3.33 cm for the smaller rotors, or 4.44 cm for the larger rotors, in order to form a thick enough epoxy sheath, and is often smaller, for example, a standard 1-in. diameter core with a diameter usually significantly less than an inch. The potted cores are then machined straight at each end to the correct length. The primary concern is that the epoxy hold a tight bond with the material against flow and repeated accelerations. Be sure that the epoxy has a low enough viscosity to be usable as a potting compound, but has a high enough viscosity that it does not imbibe into the material, fill fractures, or pores and change its hydraulic conductivity.

8. Calibration

8.1 The actual maintenance and calibration of the SSC-UFA instrument is not included in this test method. The SSC-UFA should have a manufacturer's service contract to maintain calibrations, smooth functioning, and long life. Do not attempt to calibrate a SSC-UFA manually.

8.2 The balances used to determine the mass of the specimens and the oven used for drying specimens should be calibrated periodically in accordance with the relevant quality assurance or impact levels for the application (see Specification D 4753).

9. Procedure

9.1 The following discussion refers to a commercially available SSC-UFA and provides guidance to the manufacturer's instructions. At the time of this writing, there are several laboratories in the United States that have SSC-UFAs. However, it is applicable to any centrifuge/infusion pump setup that allows open flow into the specimen during centrifugation. Specific dimensions would have to be adjusted accordingly.

9.2 Before beginning each sequence of unsaturated flow measurements or water content settings the operator will record the setup information for the SSC-UFA on a data sheet. An example of two data sheets are given in Tables 1 and 2, and were developed for the common type of soil experiments.

TABLE 1 Example Data Sheet

LRB________ PAGE________

SINGLE SPECIMEN MULTIPLE RUN DATA SHEET FOR OPERATION OF SSC-UFA

Specimen Description: Lab ID: ________ Client ID:________

Type: Whole Rock?:______ Subcored?:______ Recomposite?:______

Solution Description: Solution Density:______g/cm^3

Initial Water Content Data: Balance: Holder Mass(g)=

Subspecimen Initial Mass w/holder (g)= Subspecimen Dry Mass w/holder (g)=

Instrument No. (SSC-UFA): Rotor: Bucket: Balance: Cal?:Y/N

Temperature (°C): Dry Specimen&Holder Mass (g)= Other Remarks:

Specimen Holder Mass (g)= Wet Specimen&Holder Mass (g)= Orig. Specimen Mass (g)=

Falling Head Data: t_1(s)= t_2(s)= t_3(s)= t_{avg}(s)=

H_1(cm)= H_2(cm)= Mass(g)= K(cm/s)= Date: Tech:

Gravity Feed Data: t_1(s)= t_2(s)= t_3(s)= t_{avg}(s)= mL=

Speed (rpm)= Mass (g)= Flow rate (mL/h)= K(cm/s)= Date: Tech:

Hydraulic Conductivity Run Data ____ stepwise desaturation ____ stepwise resaturation

Date	Flow Rate (mL/h)	Rotation Speed (rpm)	Mass (g)	Comments	Tech

Total Dry Mass(g)= Specimen Dry Mass (g)= Compaction (•l, cm)=

Comments: *This data is input to an appropriate spread sheet program, e.g., EXCEL as in Table 2.*

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