DOE-FIU SCIENCE & TECHNOLOGY WORKFORCE DEVELOPMENT PROGRAM

STUDENT SUMMER INTERNSHIP TECHNICAL REPORT

June 6, 2011 to August 12, 2011

Saltstone Liquid Permeability and Formed Core Grout Sampler

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ABSTRACT

The Saltstone facilities at Savannah River Site dispose of low level radioactivity liquid salt waste. The salt waste is immobilized within a grout poured into vaults. As part of the SRNL support of the Z-Area Saltstone facility, a program is under way to evaluate different vault pouring strategies (curing conditions) on various properties of the grout. One of the tasks is to measure the hydraulic conductivity of Saltstone samples made with simulated waste salt solution using ASTM D 6527. ASTM D 6527 is the standard test method for determining unsaturated and saturated hydraulic conductivity in porous media by steady-state centrifugation. Also, in order to verify that the grout performs as expected in the vault, samples must be acquired for performance assessment.

In the past, samples have been acquired using a core drill. This, however, compromised the integrity of the sample, resulting in cracking and breaking in the sample. The in-place formed core sampler has been developed to obtain better core samples without the need for drilling. Sampler vials will be filled as the Saltstone vaults are filled. These vials are then pulled out of the vault from above by crane. The sampler underwent full scale testing with Saltstone grout made with simulated waste salt solution.

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1. INTRODUCTION

At the Saltstone Production Facility (SPF) at the Savannah River Site (SRS), a decontaminated salt solution (DSS) is combined with premix [a mixture of Portland cement (PC), blast furnace slag (BFS) and Class F fly ash (FA)] in a Readco mixer to produce fresh (uncured) Saltstone. After transfer to the Saltstone Disposal Facility (SDF), the hydration reactions initiated during the mixing of the premix and salt solution continue during the curing period to produce the hardened waste form product. The saturated hydraulic conductivity of the cured waste form is a key property input into the performance assessment (PA) models used to predict radionuclide release. Historically, the values of saturated hydraulic conductivity used for Saltstone mixes were determined for samples that had been cured at room temperature (20 °C to 25 °C). However, a number of studies have shown that the solid grout properties (e.g., dynamic Young's modulus and compressive strength) depended significantly on curing temperature. A preliminary study showed that the hydraulic conductivity increased by 700 times for a sample cured at 60 °C vs. 20 °C. The curing temperature of Saltstone is relevant since the temperature of Saltstone within the vault can reach values greater than 80 °C for extended periods of time during the curing process. Therefore, the current study was performed to measure the dependence of saturated hydraulic conductivity on curing temperature of Saltstone mixes and to correlate these results with measurements of Young's moduli. A parallel study is being performed with the goal of ensuring that values of this property introduced as input to the PA are representative of the Saltstone in the vaults.

The measurement of hydraulic conductivity is inherently difficult. There are (1) sample-to sample, (2) batch-to-batch, (3) lab-to-lab, and (4) technique-to-technique variations that are typically on the order of 0.5 to 1 order of magnitude but often higher. The ASTM method (D5084-03) using a permeameter indicates that no round robin testing has been performed to quantify the variation in results between laboratories for the same technique. In addition, no standard exists from which to measure accuracy. The measurements become even more difficult and show greater variance as the hydraulic conductivity decreases. The history of measurement of hydraulic conductivity for Saltstone mixes reveals the wide range of measured values. The latest measurements for Saltstone, cured at room temperature, show values in the range of 10^{-9} cm/sec and it is this value that is currently used as input to the PA.

Due to the difficulties in measuring permeability, an independent technique was investigated for measuring hydraulic conductivity using the ASTM based unsaturated flow apparatus (UFA) for comparison to the conventional falling head permeameter. The UFA method, if successful, could then be used for in-house measurement of hydraulic conductivity for Saltstone samples. If the hydraulic conductivity can be correlated with Young's modulus (E), then measurement of E values could also be done in-house to evaluate hydraulic conductivity. Measurement of E is much simpler than measuring hydraulic conductivity and can be readily performed as a function of time. It also has the potential for use with radioactive samples.

2. EXECUTIVE SUMMARY

This research work has been supported by the DOE-FIU Science & Technology Workforce Initiative, an innovative program developed by the US Department of Energy's Environmental Management (DOE-EM) and Florida International University's Applied Research Center (FIU-ARC). During the summer of 2011, a DOE Fellow intern (Mr. Givens Cherilus) spent 10 weeks doing a summer internship at Savannah River National Laboratory under the supervision and guidance of Mr. Alex Cozzi and Ms. Marissa Reigel. The intern's project was initiated in June 6, 2011, and continued through August 12, 2011.

This report focuses on the impact of curing temperature on the performance properties of simulated Saltstone mixes. The key performance property of interest is saturated liquid permeability (measured as hydraulic conductivity), an input to the Performance Assessment (PA) modeling for the Saltstone Disposal Facility (SDF). Therefore, the current study was performed to measure the dependence of saturated hydraulic conductivity on curing temperature of Saltstone mixes, to correlate these results with measurements of Young's moduli on the same samples. The hydraulic conductivity is measured with the Steady State Centrifugation Unsaturated Flow Apparatus. The SSC UFA is useful because it allows the user to control the independent variable in Darcy's Law equation making it easy to calculate hydraulic conductivity. Using the SSC UFA allows testing for hydraulic conductivity to be done in house at the Aiken County Technology Laboratory, as opposed to having samples sent offsite to Mactech in Atlanta, GA.

3. RESEARCH DESCRIPTIONS

Permeability - Specimen Preparation

Four Saltstone grout samples were measured for liquid permeability. Sample TP001 had 0.60 water to cement ratio(w/cm). It was cured according to the temperature profile shown in Figure 1, called the K cell type temperature profile. Sample TP002 was a repeat of TP001 with an additive Q2, commercially known as Daratard. The purpose of the additive is to delay curing time. Sample TP003 had 0.55 w/cm ratio, and was cured according to the F cell type temperature profile as shown in Figure 2. Sample TP004 had a 0.06 w/cm ratio, and was cured with the F cell type temperature profile.

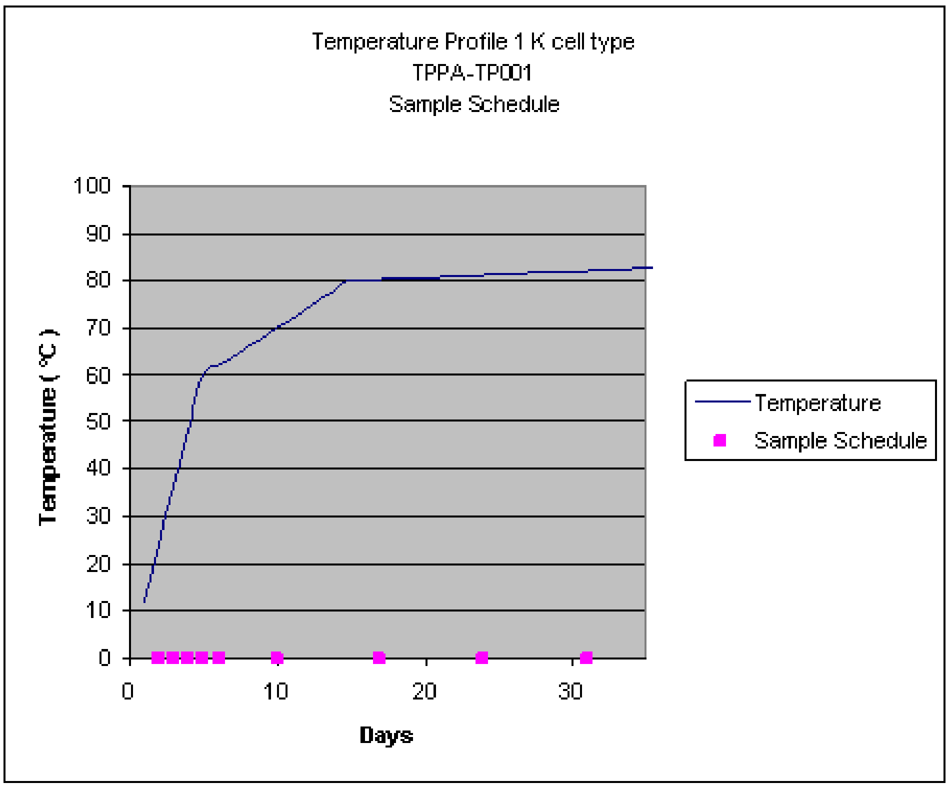


Figure 1. Temperature profile K cell type.

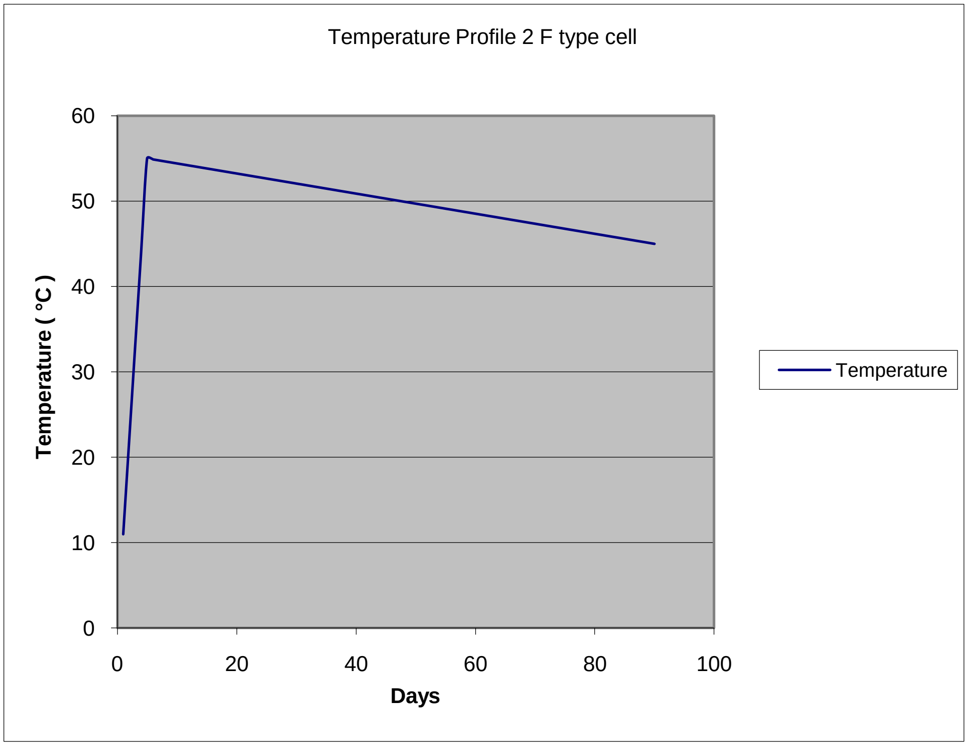


Figure 2. Temperature profile F cell type.

Specimens were prepared to be consistent with ASTM 6527, Section 7.2, *Whole Rock Cores, Concrete, or Ceramics*. Solid coherent materials can be cored using a coring bit, usually diamond, which produces cylinders that can be potted in a mold with the correct interior diameter.

However, the samples in this case were received having already been poured into a holder. Samples had to be prepared to be run in the centrifuge. This involved removing the caps using a vice and a mallet or pliers. Both ends of the holder should be flat, so any grout that extended out of the holder was filed down to be flush with the holder.

Measurements of hydraulic conductivity were made using the UFA steady state centrifuge method (ASTM D6527) located at Aiken County Technology Laboratory (ACTL). The UFA system is shown schematically in Figure 33. This system maintains permeant flow during centrifugation. The hydraulic conductivity can be calculated from the flow of liquid through the porous medium over time. Figure 4 and Figure 5 are photographs of the UFA system with the centrifuge top down and open. For the permeability measurements performed as part of this task, the permeant (water) was fed gravitationally to the sample which promotes and maintains saturation of the samples.

The samples are run in the SSC-UFA and operational parameters are recorded.

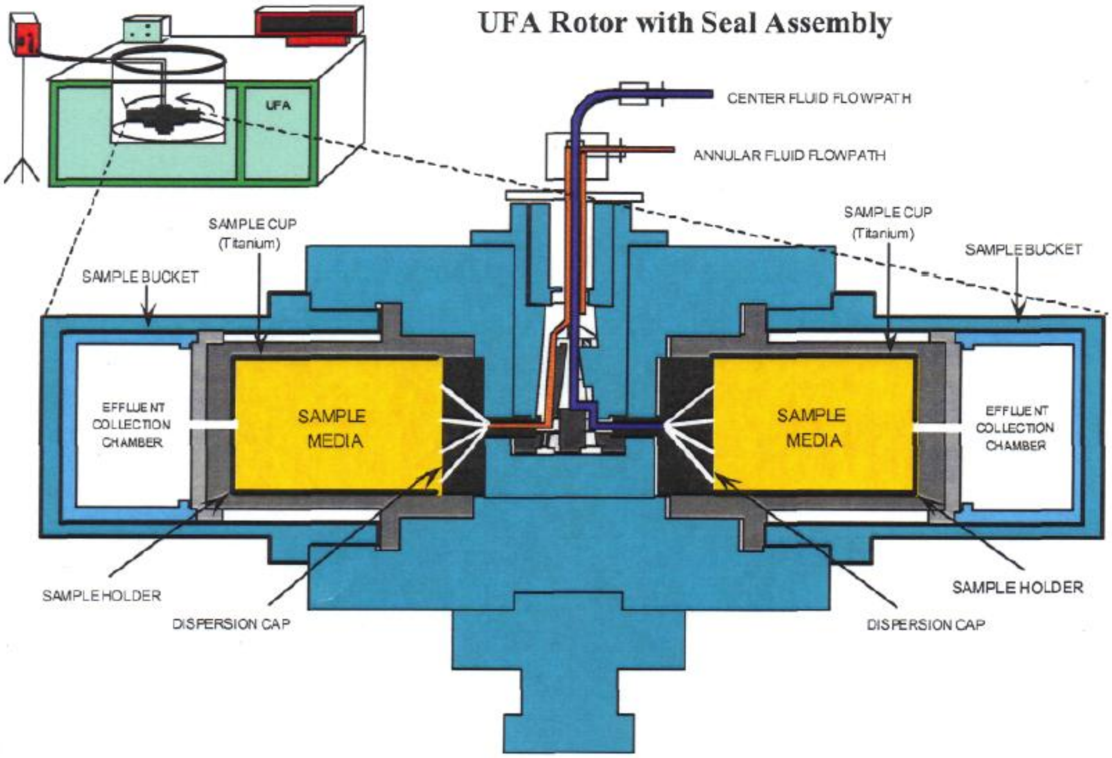


Figure 3. UFA system rotor assembly.

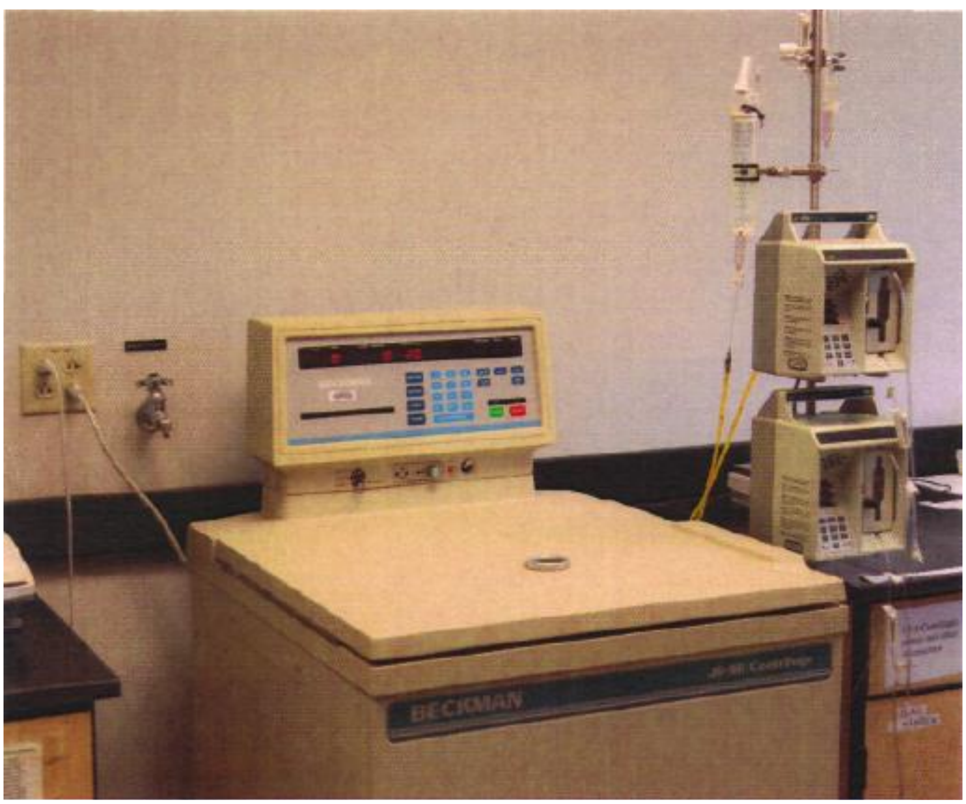


Figure 4. Photo of UFA system.

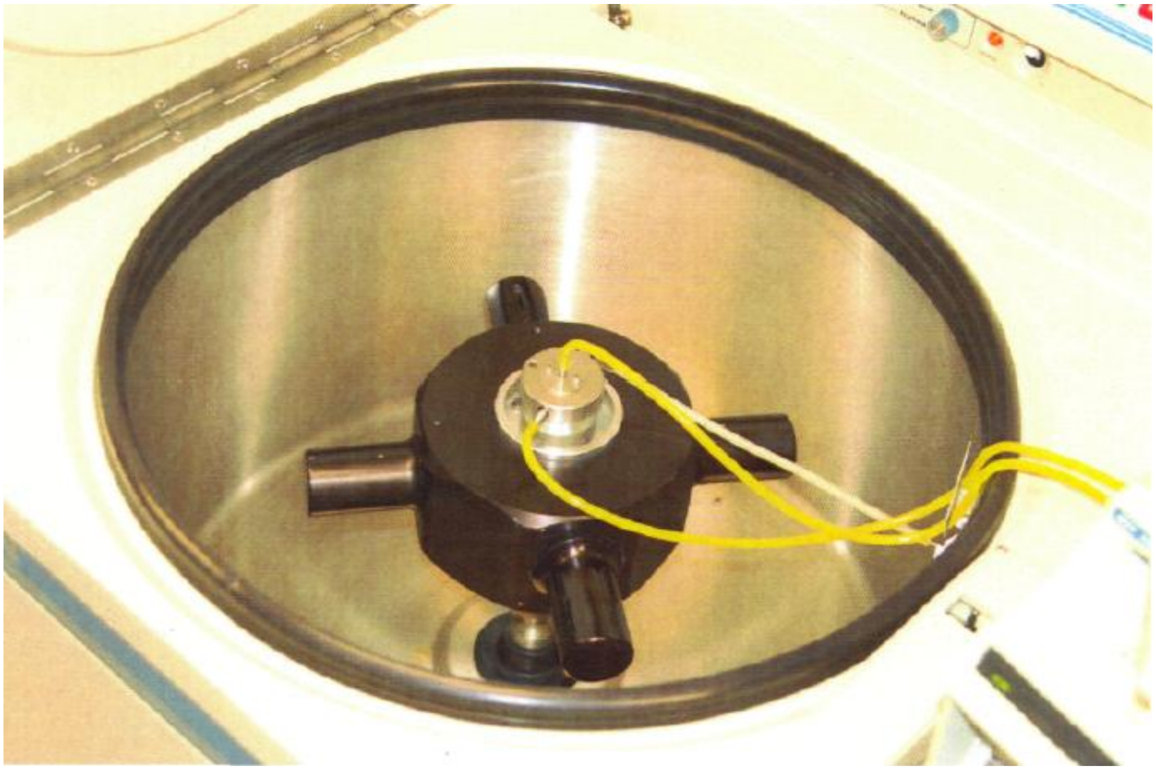


Figure 5. Photo of UFA System with top open.

Formed Core Grout Sampler Testing

For full scale testing, the formed core sampler (FCS) system was 23’ high, the height of the Saltstone vaults, and consisted of six sampler vials at various heights. The system was placed inside another larger pipe; the larger pipe simulates the Saltstone vault and will be filled with grout. The outer pipe has viewports at the location of each sampler with cameras looking into the viewports so that the filling of the samplers can be monitored and recorded. The outer pipe was filled with grout using peristaltic pumps at a rate to simulate the rate at which the Saltstone vaults are filled, nominally 1.5 inches per hour. A large pump was used to perform fast pour tests, while the smaller pump was used to perform slow pour tests at a slower rate. After each sampler was filled, the grout was left to dry for at least 48 hours before poring again to fill the next sampler. After completely filling the full scale sampler, the grout was left to cure at least 28 days. The samplers are due to be pulled September 15, 2011.

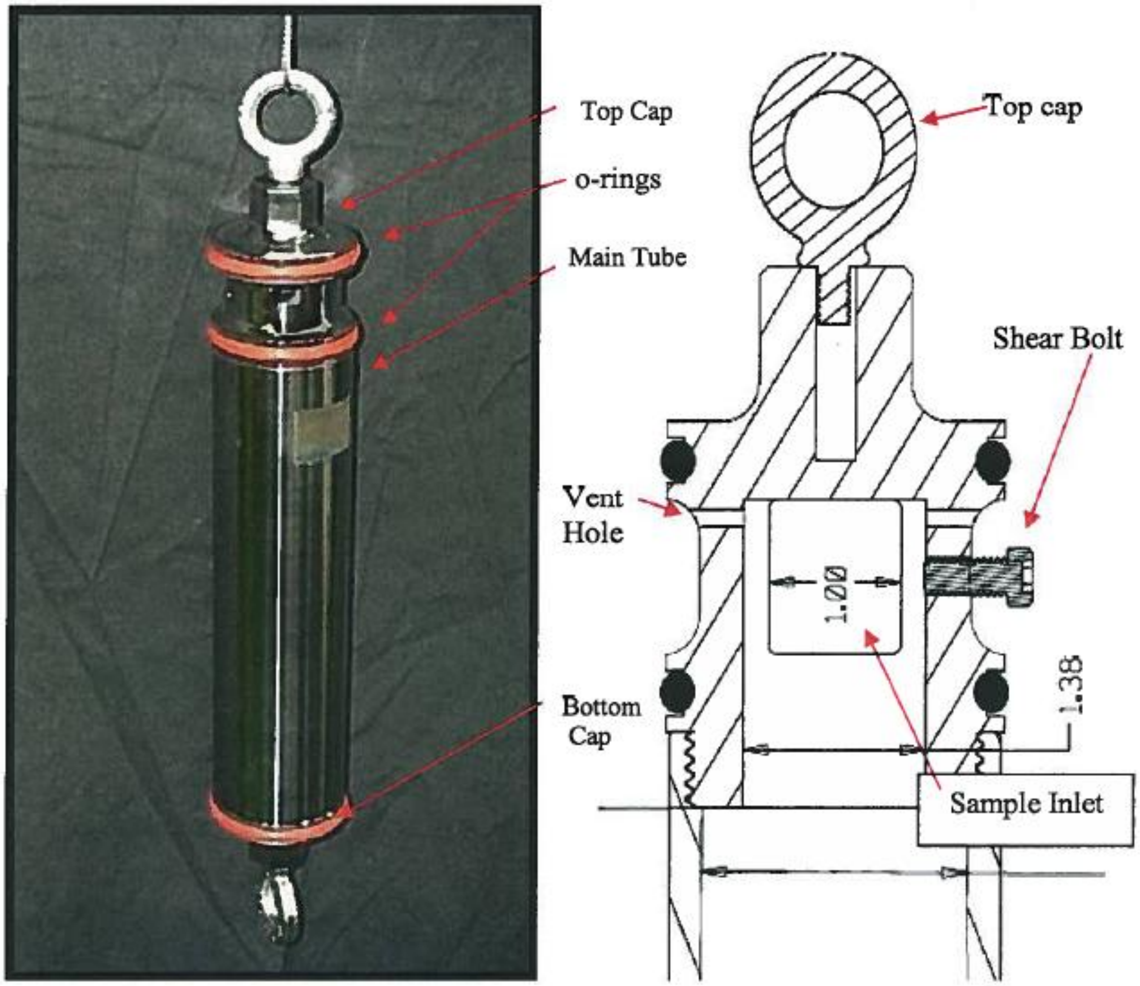


Figure 6. Formed core sampler assembly.

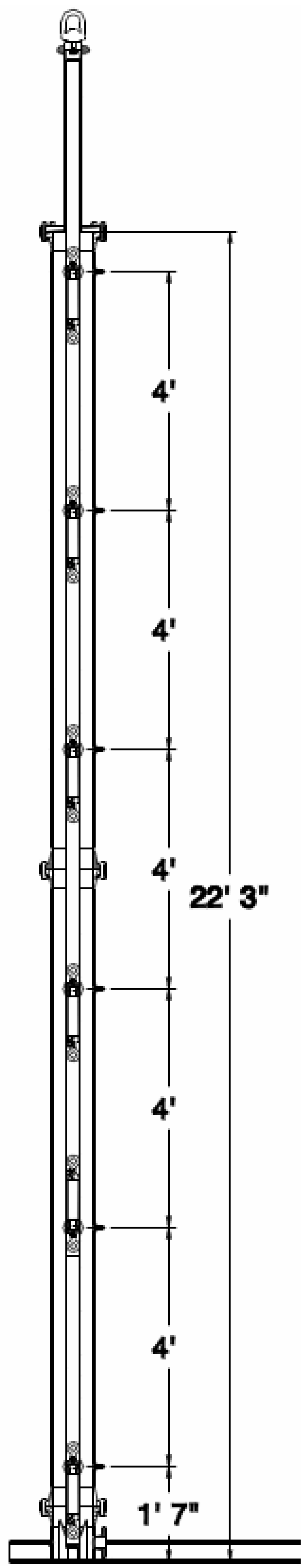


Figure 7. Formed core sampler full-scale mock up.

4. RESULTS AND ANALYSIS

Permeability – Calculation of Hydraulic Conductivity

Water flux density, q, is given by Darcy's Law as the product of the hydraulic conductivity, K, and the fluid driving force. Under a centripetal acceleration in which water is driven by both a matric potential gradient and the centrifugal force per unit volume, Darcy's Law is given as follows:

$$q = -K\,[d\psi/dr - \rho\omega^2 r] \qquad \text{(Equation 1)}$$

where K = hydraulic conductivity (cm/s), r = distance from axis of rotation (cm), ρ = water density (gm/cm3), ω = rotation speed (radians/s), ψ = matric potential, $d\psi/dr$ is the matric potential gradient, and $p\omega^2 r$ is the centrifugal force per unit volume. Hydraulic conductivity is a function of either the matric potential or the volumetric water content. Above speeds of about 300 rpm, provided that sufficient water flux density exists, the matric potential gradient can be much less than the acceleration, $d\psi/dr \ll \rho\omega^2 r$. Therefore, under these conditions, Darcy's Law is given by:

$$q = -K\,[-\rho\omega^2 r] \qquad \text{(Equation 2)}$$

Rearranging, Darcy's Law becomes:

$$K = q/(\rho\omega^2 r) \qquad \text{(Equation 3)}$$

For convenience of calculation and using run parameters from a centrifuge, pump, and specimen geometry used in the SSC-UFA apparatus, the working relationship is:

$$K = (24.8)\ (\text{flow rate in mL/hr})/\ (\text{rotation speed in rpm})^2(\text{X-sectional area in cm}^2)\ (\text{radial distance to center of specimen}) \qquad \text{(Equation 4)}$$

Each sample was run several times in the centrifuge. The run parameters and data have been collected and will be used to calculate K values.

Formed Core Sampler

The results of the formed core sampling will be determined after the samplers are pulled on September 15, 2011. The observations made during the testing through the viewports lead to the preliminary conclusion that the samplers were successfully filled with grout.

5. CONCLUSION

The values will be calculated from the run parameters and data collected. During test runs, it was observed that some samples appeared to inherently have a significantly higher flow rate, and therefore higher permeability. This will be verified once the calculations have been done. Final conclusions for the Formed Core Sampler will be made after the samples are pulled from the full scale mock up test. The samples were originally scheduled to be pulled on September 15, 2011.

6. REFERENCES

ASTM Standard D 6527-00, 2008, "Standard Test Method for Determining Unsaturated and Saturated Hydraulic Conductivity in Porous Media By Steady-State Centrifugation," ASTM International.

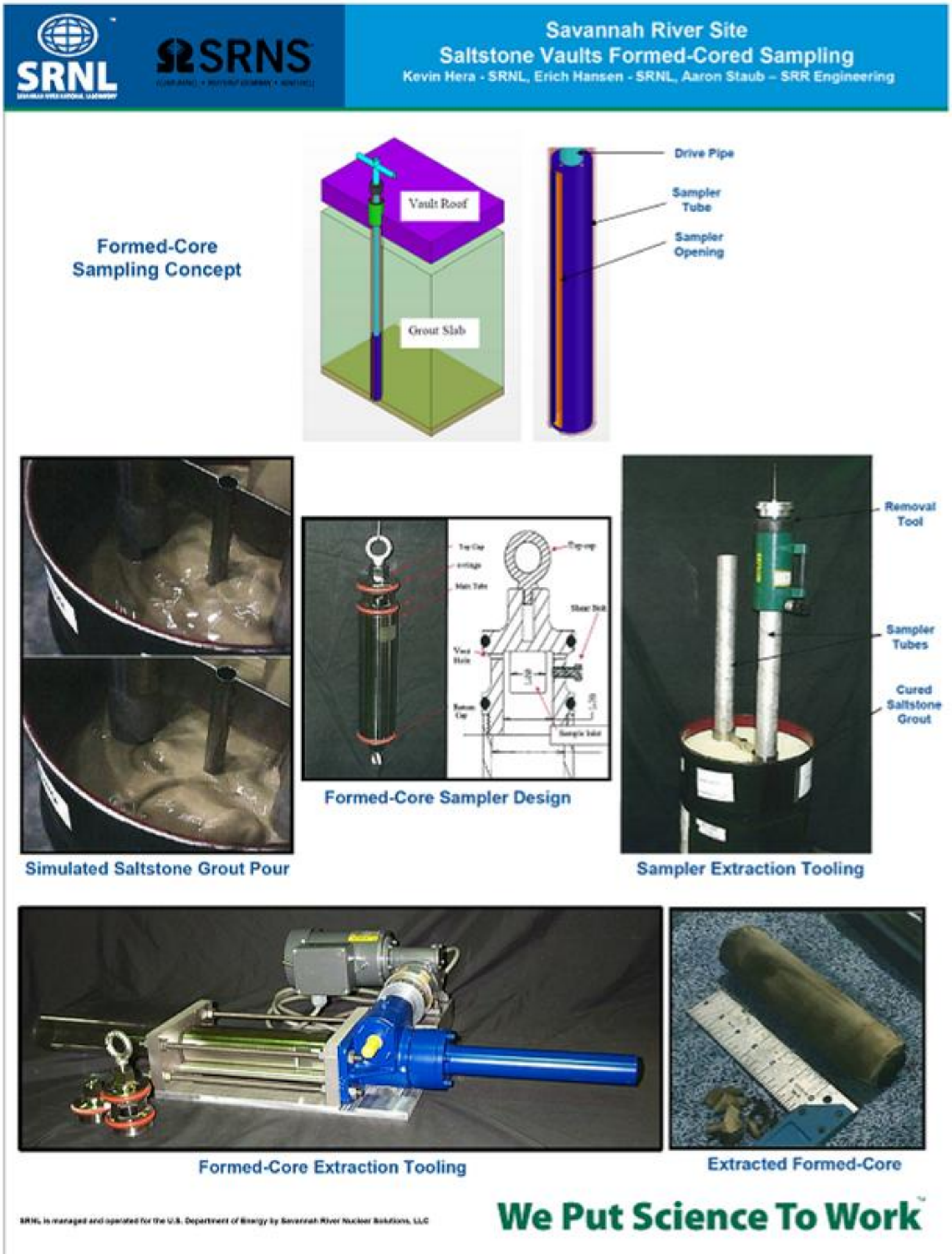
Hansen, E.K., and K.R. Hera "Design and Testing of the Formed-Core Sampling System for Saltstone Facility Vaults Savannah River National Laboratory (2010).

Harbour, J.R., and M.F. Williams "Impact of curing temperature on the saturated liquid permeability of Saltstone" Savannah River National Laboratory (2011).

Test Method For Determining Unsaturated and Saturated Hydraulic Conductivity in porous Media by Steady-State Centrifugation (2000).

UFA Ventures Procedure 03-1 Procedure for the Saturation of core samples and the Measurement of Total porosity

APPENDIX A.


SRNL
SRNS
Savannah River Site
Saltstone Vaults Formed-Cored Sampling
Kevin Hera - SRNL, Erich Hansen - SRNL, Aaron Staub – SRR Engineering
Formed-Core Sampling Concept
Vault Roof
Grout Slab
Drive Pipe
Sampler Tube
Sampler Opening
Simulated Saltstone Grout Pour
Formed-Core Sampler Design
Removal Tool
Sampler Tubes
Cured Saltstone Grout
Sampler Extraction Tooling
Formed-Core Extraction Tooling
Extracted Formed-Core
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