

Keywords: *Saltstone,
vault, Cured Grout
Sampling*

Retention: *Permanent*

Design and Testing of the Formed-Core Sampling System for Saltstone Facility Vaults

K.R. Hera
E.K. Hansen

March 15, 2010

Savannah River National Laboratory
Savannah River Nuclear Solutions
Aiken, SC 29808

Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



DISCLAIMER

Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy.

Disclaimer:

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied: 1. warranty or assumes any legal liability for the accuracy, completeness, or for the use of results of such use of any information, product, or process disclosed; or 2. representation that such use or results of such use would not infringe privately owned rights; or 3. endorsement or recommendation of any specifically identified commercial product, process, or service. Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

**Prepared For
U.S. Department of Energy**

REVIEWS AND APPROVALS

AUTHOR(S):

K.R. Hera 3/18/2010
K.R. Hera, SRNL, Remote & Specialty Equipment Date

E.K. Hansen 3/23/2010
E.K. Hansen, SRNL, Engineering Process Development Date

TECHNICAL REVIEWER(S):

A.D. Marzolf 3/18/2010
A.D. Marzolf, SRNL, Remote & Specialty Equipment Date

APPROVERS

M.E. Farrar 3/18/2010
M.E. Farrar, Manager, SRNL, Remote & Specialty Equipment Date

S.L. Tibrea 3/29/2010
S.L. Tibrea, Manager, SRNL, Robotics, Remote & Specialty Equipment Date

J.E. Occhipinti 4-5-10
J.E. Occhipinti, Manager, LWO, WS Engineering Date

EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) Remote Systems Engineering (RSE) previously evaluated several Saltstone sampling methods reported in document SRNL-RSE-2008-00029 [1]. One of the proposed methods involved the installation of formed-core samplers within the vaults. In order to reduce the time and effort associated with traditional core sampling techniques, Saltstone Engineering requested RSE to develop a formed-core system to permit removal of core samples from the Saltstone monolith within the vaults.[1] Formed-Core sampling was selected as one of the methods to pursue for the new storage vaults being built at Saltstone. The Formed-Core sampler is a system that consists of sampler tube(s) installed in a pipe. The pipe provides an orifice for filling of the sampler tube, and also a pathway to remove the sampler. The sampler tube is designed to yield cylindrical samples. Technical Task Request (TTR) number HLW-SSF-TTR-2008-0012 [2] initiated the request for SRNL to perform the task and Task Technical and Quality Assurance Plan (TTQAP) number SRNL-L1300-2008-00029 [3] developed the scope and responsibilities for SRNL to perform this task. The goal is to fabricate and test the formed-core sampling system.

Two Formed-Core systems were fabricated, placed into a 35 gallon drum at two different elevations and tested under two different flow conditions. A simulant grout was poured at 1.5 inches per hour to fill one of the sampler tubes, allowed to cure and the second sampler tube was fast poured filling the complete sampler within seven seconds. The grout was allowed to cure for a six week period and then the samplers were removed from the mock-up and cured grout cores were extruded from the sampler tubes. The maximum amount of force to pull the samplers was 1,750 lbs which is the required force to shear the grout around the sampler inlet. A constant force of approximately 300 lbs was needed to extract the samplers from the pipe of the system. To remove the core from the sampler tube, a load cell was used with an arbor press. The initial force to break the core free was approximately 3,000lbs and 800lbs was then required to extrude the core from the sampler tube.

The formed-core system produced viable cured grout samples for chemical or physical analyses. The inlet opening of the sampler easily permitted a complete fill of the sampler cavity of the sampler under different pour conditions equivalent to that observed in the vault. Retrieval of the sampler tubes from the pipe could easily be accomplished with the use of an overhead crane or a winch positioned on top of the vaults. Removal of the sample from the sampler tube is an extrusion process that required more force than removing the sampler tube.



Figures of the Cores, Left (slow pour) and Right (fast Pour)

SRNL recommends: (1) modifying and testing new sampler tube designs that will result in easy extrusion of the cured core sample with minimal breakage of the sample, (2) Fabrication of a core removal tool which will utilize a screw jack to extrude the core from the sample container and that can be utilized in a radiological hood or inside the shielded cells, and (3) the fabrication and installation of the Formed-Core sampling system for use in the new Saltstone vaults.

TABLE OF CONTENTS

EXECUTIVE SUMMARY	4
LIST OF FIGURES.....	5
LIST OF TABLES	5
ACRYNOMS	5
1.0 INTRODUCTION.....	6
2.0 EXPERIMENTAL PROCEDURE	6
2.1.1 <i>Design of the System</i>	6
2.1.2 <i>Grout Selection</i>	7
2.1.3 <i>Testing</i>	8
3.0 RESULTS	12
4.0 CONCLUSIONS.....	14
5.0 RECOMMENDATIONS/PATH FORWARD.....	14
6.0 REFERENCES.....	14

LIST OF FIGURES

Figure 1, Formed-Core Sampler Assembly.....	7
Figure 2, Grout Flow During 1 st Test – 1.5 Inches Per Hour Fill Rate.	9
Figure 3, Grout Flow During 2 nd Pour Test.....	10
Figure 4, Formed-Core Sampling Mockup – Hydraulic Jack Used to Extract Samplers.....	11
Figure 5, Formed-Core Force Chart (Slow Pour).....	12
Figure 6, Formed-Core Force Chart (Fast Pour).....	12
Figure 7, Slow Pour Formed-Core Sample. Note: Core Split Most Likely Due to Failure of Screw Jack Adapter.	13
Figure 8, Fast Pour Formed-Core Sample. Note: Arbor Press Used to Remove Core.....	13

LIST OF TABLES

Table 1, Saltstone Cementitious Materials and Premix Blend.....	8
Table 2, ARP/MCU Salt Simulant – One Liter Batch.....	8

ACRYNOMS

ACTL	Aiken County Technical Laboratory
ARP	Actinide Removal Process
CBO	Carbon Burnout
MCU	Modular Caustic Side Solvent Extraction
RSE	Remote Specialty Equipment
SRNL	Savannah River National Laboratory
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
Wt%	Weight Percent

1.0 INTRODUCTION

The Savannah River National Laboratory Remote Systems Engineering previously evaluated several Saltstone sampling methods reported in document SRNL-RSE-2008-00029 [1]. One of the proposed methods was the formed-core samplers, which consisted of placing the sampling system into the vault prior to filling the vault. In order to reduce the time and effort associated with traditional core sampling techniques, Saltstone Engineering requested RSE to develop a formed-core system to permit removal of core samples from the Saltstone monolith within the vaults [2]. The Formed-Core sampling system was selected as the system to pursue for the new storage vaults being built at Saltstone. The Formed-Core sampler system contains a sampler tube and this tube is placed inside a pipe for sampler location. The pipe provides an orifice for filling the sampler tube and the pathway to remove the sampler. The sampler tube is designed to yield cylindrical samples. Technical Task Request (TTR) number HLW-SSF-TTR-2008-0012 [2] initiated the request for SRNL to perform the task and Task Technical and Quality Assurance Plan (TTQAP) number SRNL-L1300-2008-00029 [3] was drafted as a response outline development responsibilities in SRNL. The goal is to perform initial testing to determine the performance of the proposed formed-core sampling system in successfully delivering sample cores for analyses.

2.0 EXPERIMENTAL PROCEDURE

2.1.1 *Design of the System*

The formed-core sampling system is specifically designed for use in the new round vaults presently under construction at Saltstone, though they can easily be modified for use in other vault designs. A 2-½" schedule 10, stainless steel pipe 23 feet long will be used to position the samplers in the vault and the pipe will provide the path to remove the samplers from the vault. Two diametric one inch square openings are cut into the pipe to allow grout to fill the samplers. The Formed-Core samplers are designed to produce a cylindrical 2"x 8" grout core sample which can be cut into multiple pieces and used for several analytical tests. The sampler is constructed of three parts, a main sampler tube, top cap and bottom cap shown in Figure 1. The 1-inch opening into the sampler is also shown to the right of the sampler tube figure. The location and number of samplers in each sample pipe will be dictated by the Saltstone customer.

The sampler tube was fabricated from 300 series stainless steel and the threaded caps were made from Nitronic 60 which reduces the possibility of galling the threads during assembly and disassembly. The top cap has a one inch square thru inlet hole to allow grout to flow into and fill the sampler tube, as shown in the cross-sectional view of Figure 1. Silicone o-rings were used to seal the sampler's inlet hole to the main pipe. Another silicon o-ring was used at the bottom of the sampler and a viton o-ring (not shown) seals the grout material inside the sampler. Viton and urethane o-rings were tested in addition to the silicone o-ring, however the silicone o-ring provided the best grout sealing and the least frictional force during sample removal. The interior walls of the sampler were electro-polished to minimize the friction when extruding the sample cores. A vent hole was located in the sampler chamber to allow air to escape if the sampler is filled quickly. A plastic fastener was used to lock the sampler into position (Figure 1) and is designed to shear off when the sampler is removed.

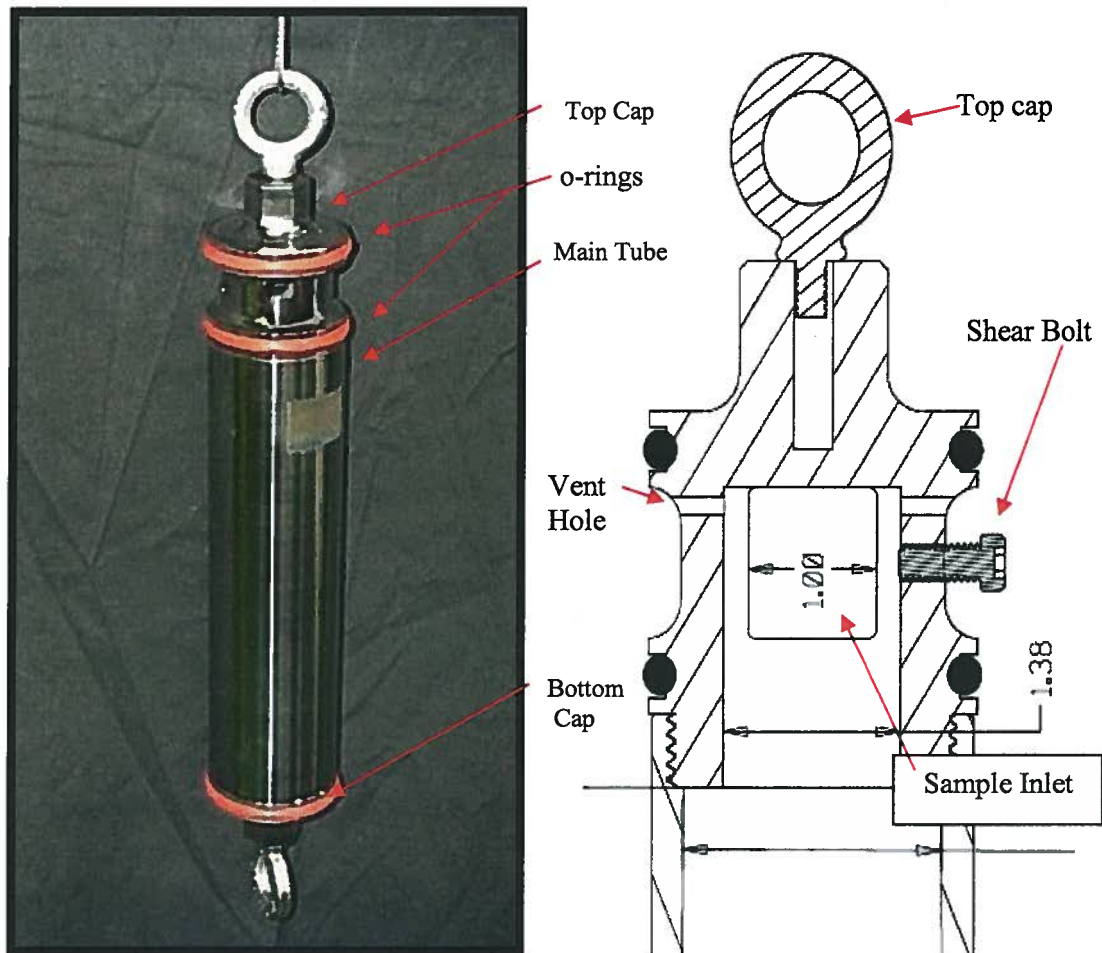


Figure 1, Formed-Core Sampler Assembly.

The samplers will stay inside the pipe until the vault is completely filled or when the grout has had sufficient time to cure, as dictated by Saltstone Engineering. To remove the samplers from the pipe, a cable is attached to each sampler (Figure 1) and the sampler is removed by pulling them one by one from the pipe. Eyebolts are attached to each end of the samplers and are connected using 1/8th inch thick stainless steel braided cables.

2.1.2 Grout Selection

The cementitious materials specified for Saltstone processing are listed in Table 1 which were obtained from Saltstone. Table 1 also contains the vendors and the weight percent (wt%) contribution of the material in the premix blend used at Saltstone and in this task. The fly ash used at Saltstone must be thermally treated to remove ammonia. The fly ash provided by the vendor is referred to as carbon burnout or CBO fly ash, which satisfies the Class F Fly Ash specification required by Saltstone. The premix was blended in multiple batches using a V-blender to homogenize the blend.

Table 1, Saltstone Cementitious Materials and Premix Blend.

Material	Vendor	Wt% of Blend
Type II Portland Cement	Holcim	10
Grade I or II Blast Furnace Slag	Holcim	45
Class F Fly Ash	SEFA	45

A simulant Actinide Removal Process (ARP) - Modular Caustic Side Solvent Extraction (MCU) salt solution provided in Table 2 was utilized in this task. The sodium and aluminum concentration of aluminum in this simulant are 5.44 M and 0.22 M, respectively and are based on the composition of the remaining three batches of ARP/MCU waste [4]. This simulant was approved by Saltstone Engineering. The density and wt% total solids for this simulant are 1.247 g/mL and 29.4 wt%, respectively.

Table 2, ARP/MCU Salt Simulant – One Liter Batch.

Waste Stream		Chemicals Used	
Compound	Molarity	Compound	Grams
NaOH	2.680	50% wt% NaOH solution	214.40
NaNO ₃	1.700	NaNO ₃	144.48
NaNO ₂	0.550	NaNO ₂	37.94
Na ₂ CO ₃	0.176	Na ₂ CO ₃	18.65
Na ₂ SO ₄	0.059	Na ₂ SO ₄	8.37
Al(NO ₃) ₃	0.220	Al(NO ₃) ₃ + 9 H ₂ O	82.53
Na ₃ PO ₄	0.012	Na ₃ PO ₄ + 12 H ₂ O	4.67
H ₂ O	-	DI H ₂ O	735.26

The ARP/MCU salt solution and premix were blended at water to premix ratio of 0.60, resulting in a grout containing 45.9 wt% salt solution and 54.1 wt% premix. The physical properties of this grout have been analyzed and reported in reference [5].

2.1.3 Testing

Testing of the formed-core system consisted of filling a container with a Saltstone grout simulant at two different pour rates and then determining the amount of force required to remove the samplers from the pipe after the grout cured and extruding the cured cores from the sampler tubes. A mockup was fabricated and delivered to Aiken County Technical Laboratory (ACTL) for grout pouring activities. A 35 gallon drum was used as the fill container for the grout simulant and contained two 2-1/2" pipes each containing one sampler assembly. The samplers were positioned at different elevations approximately six inches apart. This permitted a slow pour test with the first sampler and a fast pour test with the second sampler.

Prior to commencing the first test, grout was poured into the drum to a position just below the sampler opening and the grout was allowed to cure for three days. The drum was covered to minimize evaporative losses. The first test was the slow pour test, targeting the nominal vault fill rate of 1.5 inches per hour or 110 ml/min considering the cross-section of the free area of the 30 gallon drum. The grout was prepared in quantities that provided feed for 10 minutes. The grout was poured 110 ml/min until the grout was above the sampler opening and then left to cure for three more days. Conditions of grout pour/flow during the first test are shown in Figure 2. During grout flow, it was observed that the cured grout was absorbing liquid from the fresh grout, darkening the cured grout. This condition can cause the leading edge of the grout flow to thicken due to

absorption of free liquid from the flowing grout. Grout flow on the surface was not uniform, but ran in fingers. The black particles observed floating on top of the grout is carbon from the fly ash. The grout was pumped to approximately one inch above the opening and allowed to cure for three days prior to the second test.



Figure 2, Grout Flow During 1st Test – 1.5 Inches Per Hour Fill Rate.

The second test was a fast pour test. Grout was filled to the point just below the opening of the second sampler. Two gallons of grout were poured into the drum all at once, quickly submerging the sampler opening to a point approximately one inch above the opening. The conditions of the grout surface prior to adding grout and during the fast pour cycle are provided in Figure 3. It is observed that the cured surface, prior to fast pour test has cracks. During this fast pour, the grout quickly covered the sampler fill opening and air escaped the sampler via its vents, allowing the sampler to fill with grout within 7 seconds. The level of the grout after the fast pour was approximately one inch above the opening. After the quick pour was complete and allowed to cure for five days, additional grout was added to completely fill the drum. The contents in the drum were then allowed to cure for six weeks before the removal of the samplers.

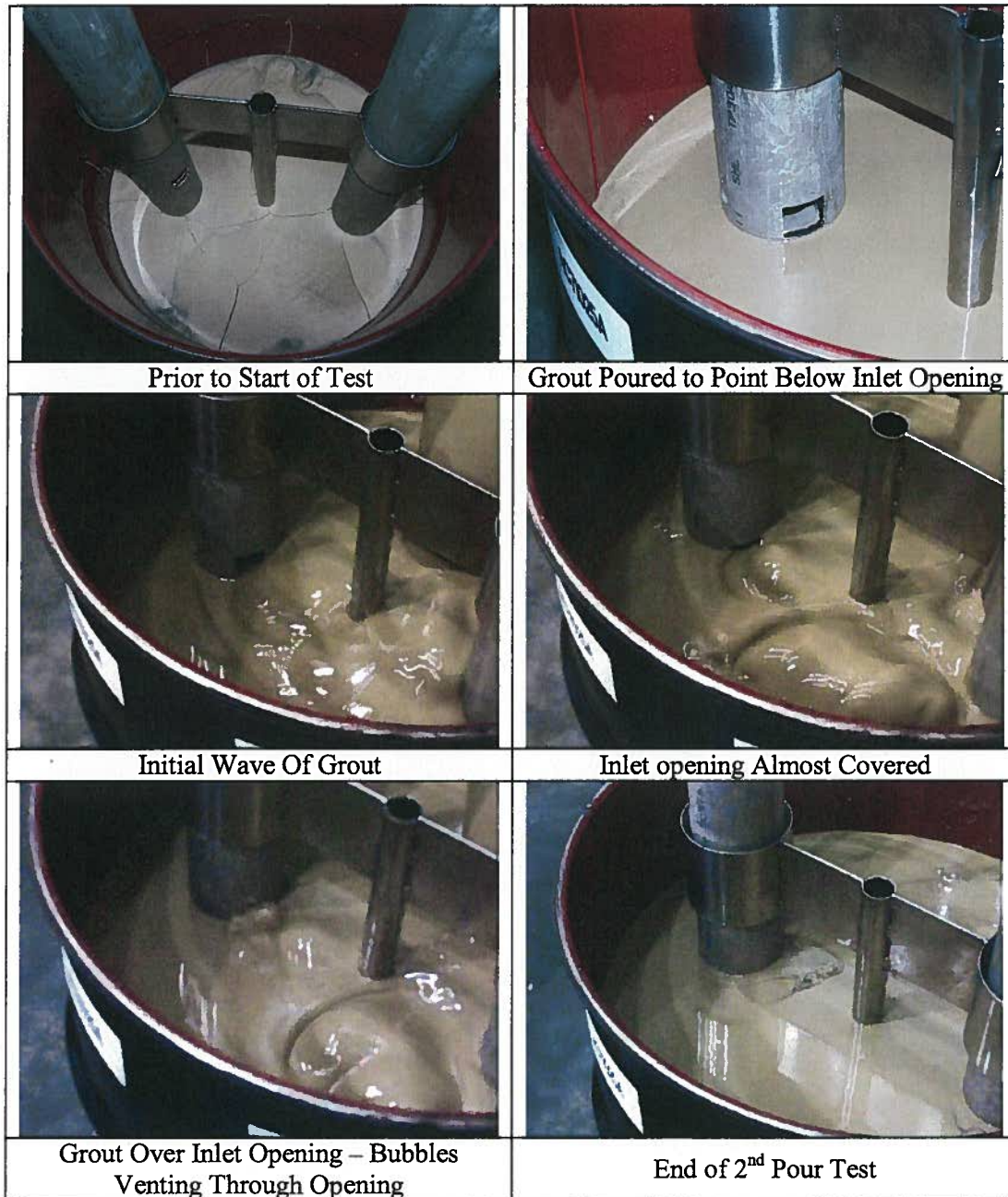


Figure 3, Grout Flow During 2nd Pour Test.

The mockup was delivered to the 723-A Engineering lab to complete the testing. A 30 ton hydraulic jack with a hollow ram was positioned on top of the sampler pipe and a thread rod was used to connect to the top of the sampler as shown in Figure 4. The jack was operated with a manual hydraulic pump with a pressure transducer installed in the hose. The calibrated pressure transducer (M&TE #Q207257) provided a direct correlation to the amount of force required to extract the formed-core sampler from the installed piping. The pressure was recorded on a data logger at a 10 millisecond sampling rate. This process was repeated in the removal of both of the samplers from the mockup.

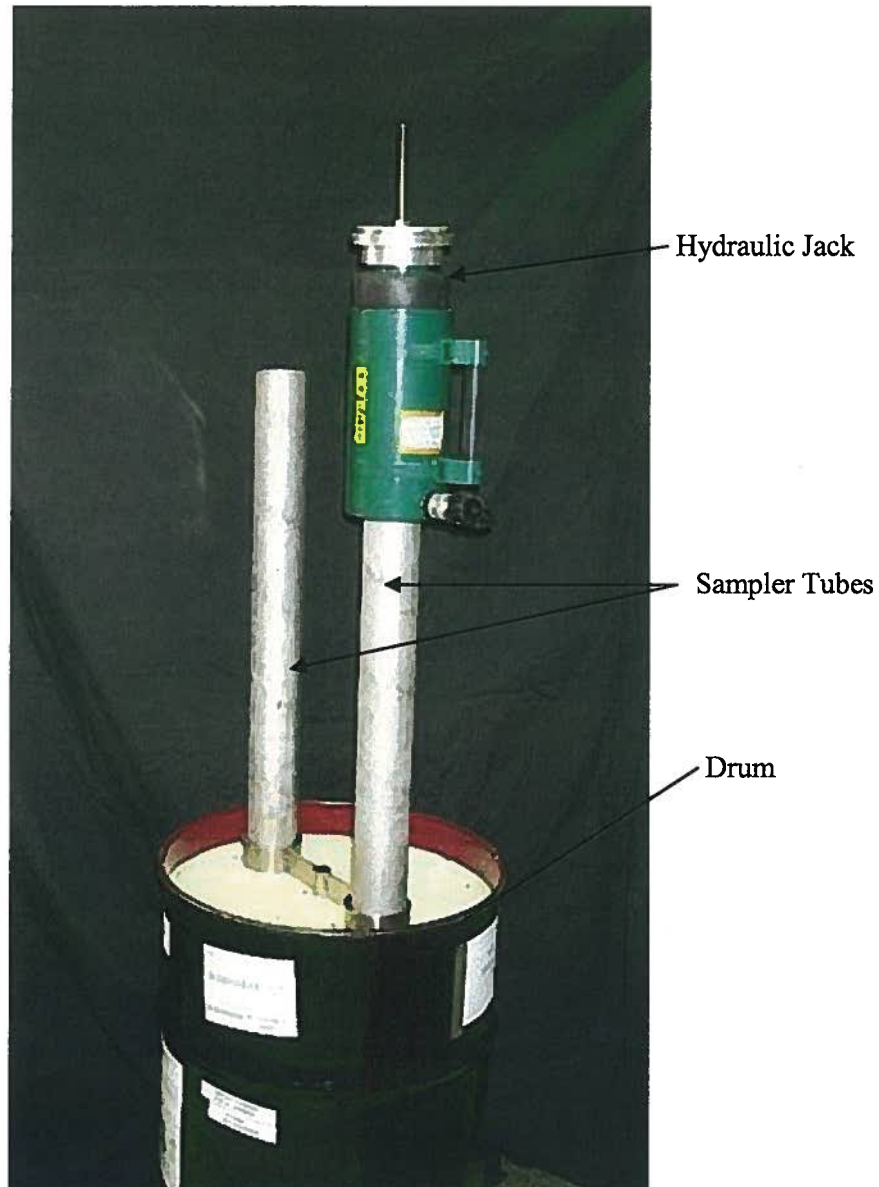


Figure 4, Formed-Core Sampling Mockup – Hydraulic Jack Used to Extract Samplers.

After the samplers were extracted from the exterior piping, the two end caps were removed from the sampler assembly which enabled the removal of the grout core. To extract the core, an adapter screw jack with a 5/8" thread was attached to the sampler tube to manually extrude the core from the tube.

Tests were also performed to determine the amount of force required to shear the plastic positioning fastener from the sampler. The plastic fastener is used to secure the sampler when installed in the exterior piping. The data was required to determine the amount of additional force required in extracting the sampler.

3.0 RESULTS

Both samplers were successfully removed from the test mockup and the grout cores were extruded from the sampler tubes. Figures 5 and 6 shows the amount of force required to remove each of the samplers from the exterior pipes. The first figure 5 was recorded during the removal of the sampler used for the slow pour test and the second Figure 6 from the sampler extraction of fast pour test. The maximum amount of force to pull the samplers from the piping was 1,750 lbs which is the force required to shear the plastic fastener and the cured grout that forms around the sampler inlet. An initial shear test to determine the shear force of the plastic fastener resulted in 90 lbs of force. The smaller force of approximately 300 lbs is the constant force required to continually extract the samplers from the pipe of the system.

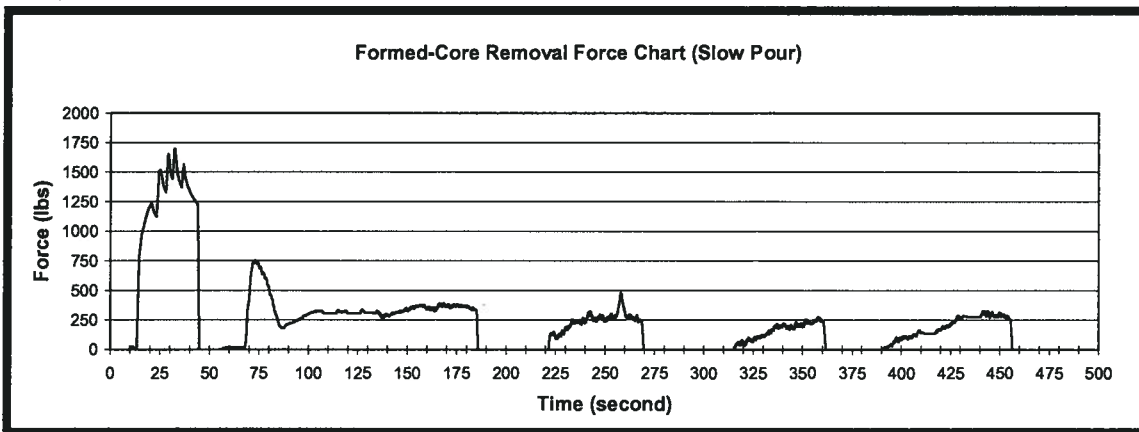


Figure 5, Formed-Core Force Chart (Slow Pour)

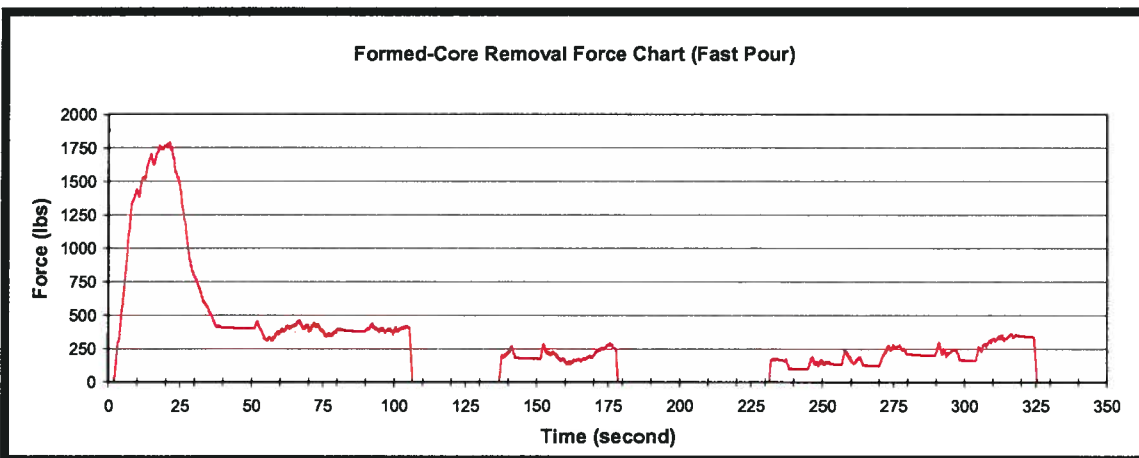


Figure 6, Formed-Core Fore Chart (Fast Pour)

After the formed-core samplers are removed from the pipes, the cores were then extruded from the samplers. Initially, a screw jack adapter was attached to the first sampler for extraction of the core, however the amount of force required to extrude the core out of the sampler exceeded the capacity of the jack design. Only half of the core was removed prior to jack failure. An arbor press was then utilized to remove the cores from the samplers. A load cell (M&TE #3-2125) was placed inline with the arbor shaft to determine the amount of force needed to extract the core from the sampler. A force of 3,000 lbs was required to initially break the grout core bond from

the sampler. A continuous force of 800 lbs was then needed to completely remove the core from the sampler. Pictures of the extruded cores are shown in Figures 7 and 8 for the slow and fast pour cores respectively.



Figure 7, Slow Pour Formed-Core Sample. Note: Core Split Most Likely Due to Failure of Screw Jack Adapter.



Figure 8, Fast Pour Formed-Core Sample. Note: Arbor Press Used to Remove Core.

4.0 CONCLUSIONS

SRNL was able to successfully produce quality (in physical appearance) grout cores for the analytical analysis. The wide inlet opening of the sampler will be able to fill the cavity of the sampler under the most severe pour scenarios. Retrieval of the samplers from the pipe will require 1750 lbs of force which can be accomplished with the use of an overhead crane or a winch positioned on the roof of the vault. Removal of the cores will require hands-on to break the ends free and assist in the removal of the cores from the formed-core sampler. The condition of the slow pour Formed-core sample shows it is in multiple sections, Figure 7, where a sub sample may not be available for permeability measurements. The fracture of the first core was due to the failure of the screw jack system initially designed to remove the core from the sampler.

5.0 RECOMMENDATIONS/PATH FORWARD

With the success of the initial Formed-Core testing, SRNL recommends; (1) modifying and testing new sampler tube designs that will result in easy extrusion of the cured core sample with minimal breakage of the sample, (2) Fabrication of a core removal tool which will utilize a screw jack to extrude the core from the sample tube within a radiological hood or the shielded cells, and (3) the fabrication and installation of a Formed-Core sampling system for use in the new Saltstone vaults.

6.0 REFERENCES

- ¹ Gordon, J.R., *Review of Grout Sampling Methods for SaltStone Facility Vaults*, SRNL-RSE-2008-00029, Savannah River Site, Aiken, SC, 29808(2008).
- ² Staub, A. V., *Formed Core Sample Development*, HLW-SSF-TTR-2008-0012, Rev. 0, October 2008
- ³ Milling, R.B., *Task Technical and Quality Assurance Plan for SaltStone Formed Core Sample Development*, SRNL-L1300-2008-00029, Rev.0, 10/1/2008.
- ⁴ Le, T. A., *Future ISDP Batches to Saltstone Processing Facility, Projected Batch and Key Components Compositions*, LWO-LWP-2009-00005, Rev. 1, 2009.
- ⁵ Harbour, J. R., Edwards, T.B., and Williams, V. J., *Key Factors That Influence the Performance Properties of ARP/MCU Saltstone Mixes*, SRNL-STI-2009-00546, Rev. 0, Sept. 2009

Distribution

K.R.Hera, 723-A
A.D.Marzolf, 723-A
C.C.Herman, 999-W
J.E.Occhipinti, 704-S
S.L. Tibrea, 730-A
J.G. Sonnenberg, 773-A
M.E.Farrar, 723-A
E.K.Hansen, 999-W
A.D.Cozzi, 999-W
A.B.Barnes, 999-W
A.V.Staub, 704-Z
T.L.Fellinger, 704-26S
J.R.Gordon, 730-A
M.M.Reigel, 999-W
S.L.Marra, 773-A
D.A.Crowley, 773-43A
F.M.Smith, 705-1C
L.K.Pressley, 705-1C

EES Job Folder No. 23215