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Full-Scale Testing of the Formed-Core Sampler for Saltstone Facility Vaults

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EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) Mechanical Systems & Custom Equipment Development (MS&CED) section previously designed and tested a sampling system to capture a Formed-Core sample from the Saltstone vaults. That testing was proof-of-concept and is documented in SRNL-STI-2010-00167.[1] After the proof-of-concept testing, Saltstone requested MS&CED to fabricate and test a full-scale mock-up of the sampler system to simulate function in the Saltstone vaults. Technical Assistance Request (TAR) number HLW-SSF-TAR-2011-0004 was transmitted to initiate the task.[2] The TAR deliverable was full-scale fabrication and testing of the Formed-Core sampling system.

To accomplish the scope, one full-scale Formed-Core sampling system was fabricated. It was loaded with six sampler vials each spaced approximately four feet apart. In addition, a mock-up of the Saltstone vault was constructed to allow testing the system using grout simulant to ensure the system operated properly. An eight inch diameter vertical tank approximately 25 ft tall acted as the vault. Saltstone grout simulant was poured into the tank in multiple lifts at different rates to test the sampling system. When all the lifts were completed and the grout cured for more than six weeks, the sampler vials were removed. The removal demonstrated sampler vial retrieval capability. The extraction force for each vial was measured and recorded. The magnitude of the initial force required to fracture and dislodge the vial from its grouted position within the sampler pipe ranged from 3000-6000 lbs. Withdrawal forces after dislodging the sampler ranged from under 50 lbs to nearly 5000 lbs.

The mockup demonstrated the functionality of the sampler in a full-scale mockup. The sampler vials were all retrieved; however, smaller withdrawal forces are preferred. For this reason, some modifications to the design are recommended prior to facility use.

With the sampler vials out of the formed-core sampler housing, a second tool is required, the tool must extract the grout core sample out of the sampler vial. A screw-jack style core extraction tool was designed and assembled. It operates by pressing the cured grout cores out of the sample vials. The extractor has been used (external to this test) to prove its function. It removed three good cores (out of three) and is recommended for operation in its current state. The extractor will also be used to remove the six cores associated with this testing per the analysis schedule requirements.

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ACRYNOMS

ACTL	Aiken County Technical Laboratory
ASTM	American Society for Testing & Materials
DI	Deionized
FCS	Formed-Core Sampler
M&TE	Measuring & Test Equipment
MS&CED	Mechanical Systems & Custom Equipment Development
NPS	Nominal Pipe Size
SRNL	Savannah River National Laboratory
TAR	Technical Assistance Request

1.0 INTRODUCTION & BACKGROUND

The Savannah River National Laboratory (SRNL) MS&CED group was tasked to fabricate a full-scale mockup and test the Formed-Core Sampler. The activity was intended to verify the operational feasibility of previously completed proof-of-concept work documented in SRNL-STI-2010-00167.[1]

HLW-SSR-TAR-2011-0004 is the initiating and guiding document for the scope reported herein.[2] This task is defined as: 1) show that the samples can be remotely retrieved and 2) show capability to extract the core from the sample container vials for characterization.

2.0 METHOD

A test was planned that included all the sampling equipment in an environment intended to simulate relevant aspects of the Saltstone vault facility. A vertical tank used to simulate the vault was constructed. A Formed-Core Sampler (FCS) was also fabricated. A total of six sampler vials were loaded and instrumented with temperature data loggers in the FCS assembly. The entire FCS assembly was positioned within the vault tank mockup in preparation for grout fill.

2.1 Mockup and Sampling Equipment Description/Features

2.1.1 Vault Mockup

The vault mockup consists of an 8" diameter tank approximately 25 feet long oriented vertically (Figure 1). The mockup contains the simulant around the sampler in a manner similar to the facility vault. Although not prototypic, viewing windows were included in the mockup tank to enable observation of the local grout flow in the vial inlet port during fill. The FCS assembly (a pipe containing the samplers) is positioned in line with the central axis of the mockup. See drawing RDE-23215-R3-009 for illustration and specification of the mockup vault tank.[3]

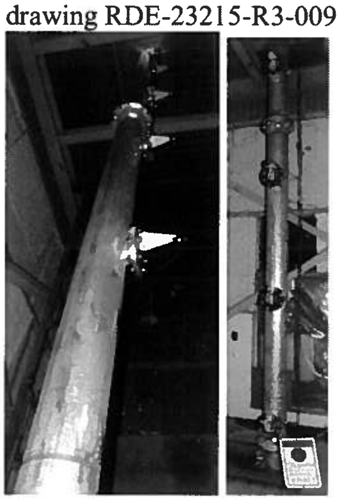

Figure 1: Views of Mockup from Bottom

2.1.2 Formed-Core Sampler Assembly

The FCS assembly consists of a housing sleeve (tube or pipe) which contains the sampler vials. An inlet window is cut in order to allow grout flow into the core chamber of each vial. A total of six sampler vials were loaded into the FCS assembly. The left view of Figure 2 illustrates a close-up of one vial; the right view shows the entire FCS assembly. Not shown, are the cables used to tether each vial to the adjacent vial above and below. The cable is formed into a choker geometry which is looped through eyebolts shown. To limit withdrawal force requirements, excess choker length is included to allow withdrawal of each vial prior to tensioning the choker fastened to the next lower vial. Therefore the choker attached to the bottom vial is the longest when everything in the FCS assembly is positioned.

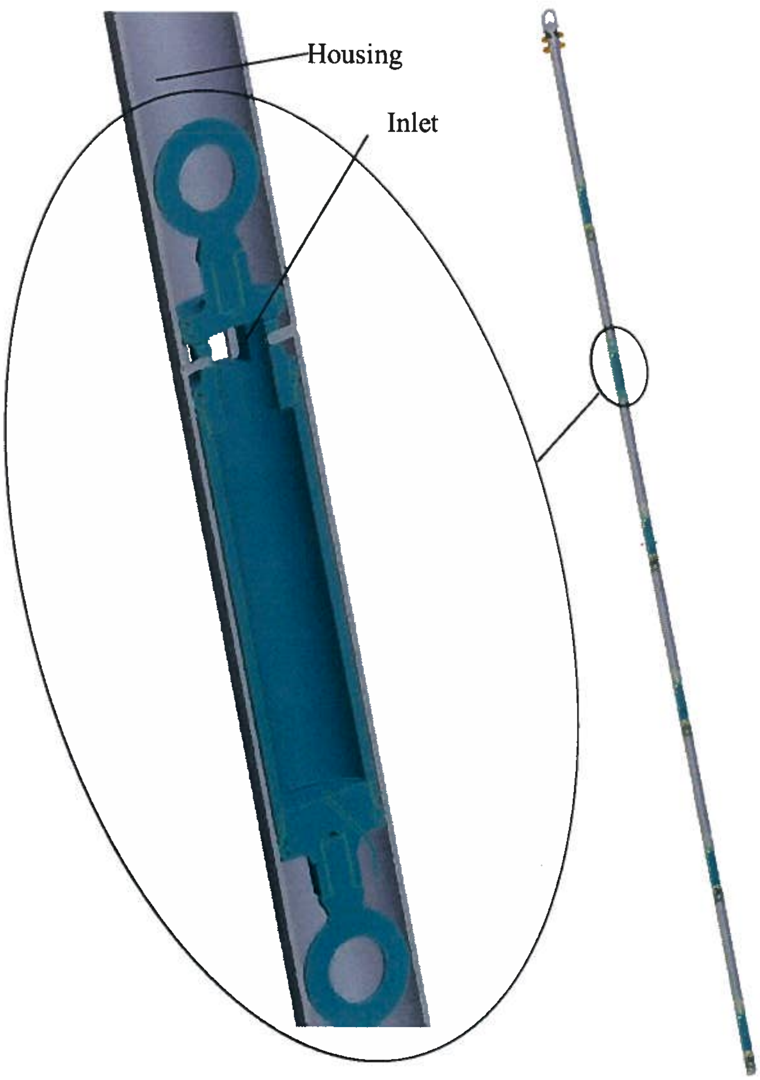

Figure 2: FCS in Section with Detail of One Sampler Vial

2.1.3 Sampler Vial

The design of the individual sampler vial is discussed in SRNL-STI-2010-00167 (Figure 3).[1] The details of construction are not repeated here and the reader is directed to the report for information. Design specification of the sampler vial is located in the latest revision of drawing RDE-23215-R4-001.[4]

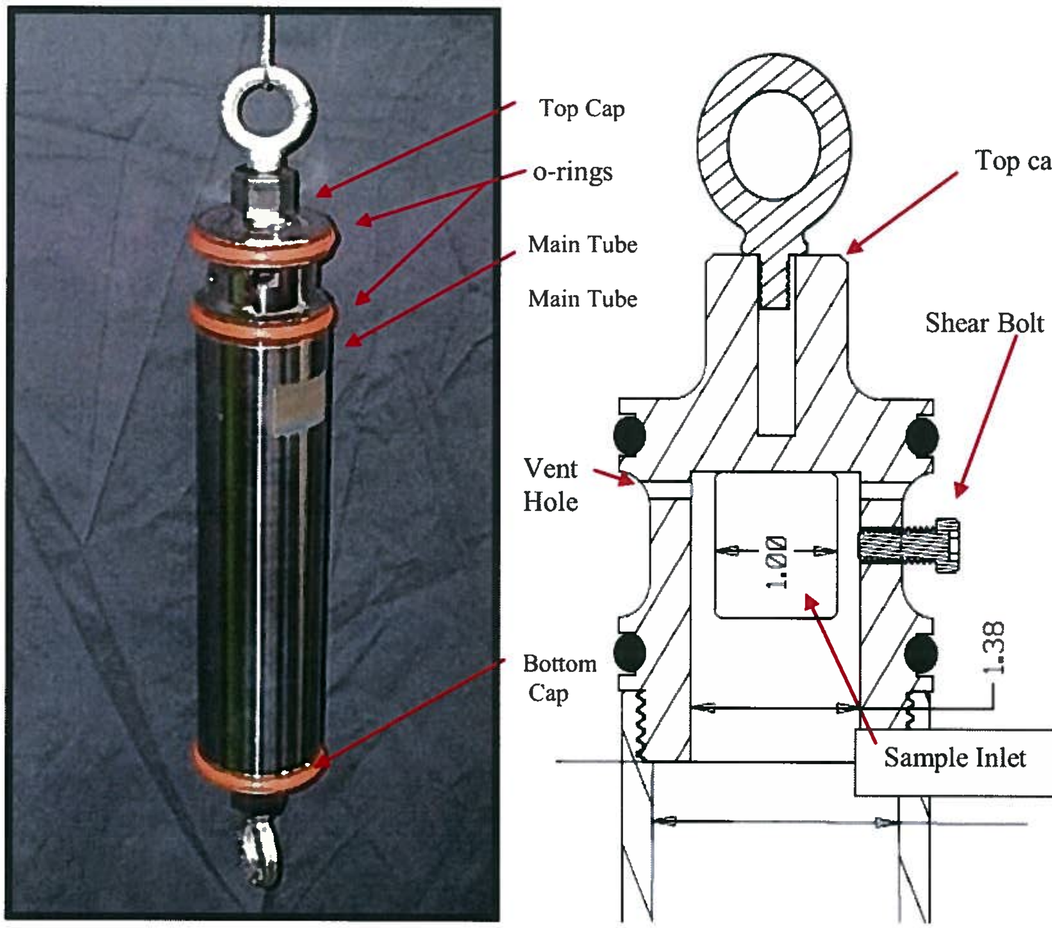

Figure 3: Formed-Core Sampler Vial Assembly.

2.2 Simulant Description

The simulated Saltstone grout was prepared by mixing the low-level salt solution simulant with a premix blend of 45% fly ash, 45% slag and 10 % Portland cement. The grout was mixed just prior to pouring and pumped into the tank at the required delivery rate. Each day's pour was recorded using a camera positioned outside the view port located at each sampler location. A second camera was lowered from the top of the stand along with the pour tube to allow visual observation of the grout flow. Additional information about the simulant and each grout pour is provided in Appendix A.

2.3 Test Method

With the equipment in this position, grout was pumped into the mockup at different predetermined rates and lifts (Appendix A). After the grout cured for at least six weeks, each sampler was extracted from the assembly. Calibrated load measurement equipment monitored the withdrawal force. Due to the difficulty in extracting the samplers a second extraction method with higher load capability was implemented.

2.3.1 Extraction Method 1

This method utilized a facility crane located in the building 723-A high bay. The mockup was positioned directly below the crane. A manually-operated chain fall was connected inline between the crane hook and the vial. This allowed for much better control of the load application rate compared to using the jog button on the crane control pendant. The design allowed loads of 4000 lbs maximum to be applied to withdraw the sampler vials via the crane. The cables used to tether the vials together were of different lengths and arranged such that only one vial was tensioned at a given time. When a vial was withdrawn out of the FCS assembly, it was disconnected from the choker below it and this required moving the chain fall hook to engage the lower choker.

Figure 4: Sampler Vial Removal from FCS

2.3.2 Extraction Method 2

When larger withdrawal forces were required, Method 2 was contrived. It consisted of a hydraulic ram placed on the top of the FCS sampler assembly (Figure 5). The tether cables and eyebolts were unthreaded from the vial and replaced with a threaded rod that acted as a drawbar. The threaded rod was inserted through a hollow center of the ram and a nut threaded on the rod bears against the top surface of the ram actuator. When the hydraulic ram was extended against the nut, the drawbar was tensioned in turn pulling on the vial in the FCS assembly. At full extension, the return valve was opened and the ram retracted. After retraction, the nut was re-adjusted to bear against the ram. The process was repeated every six inches of lift. An alternative approach used with this setup was to continue tightening the nut to lift the vial. This was only advantageous when the extraction forces were low. Occasionally the vial could be lifted by hand a short distance.

Figure 5: Hydraulic Actuator in Position

2.4 Core Extraction

A core extraction tool was designed and fabricated as shown in Figure 6. To remove the core the vial ends were unthreaded and removed in preparation for the extraction as shown in Figure 6. The motor turns a screw jack which extends a ram into the sampler vial sleeve. The core was pushed out of the sleeve into a sample carrier. After the core was extracted, it was stored in a sealed container until analysis was started.

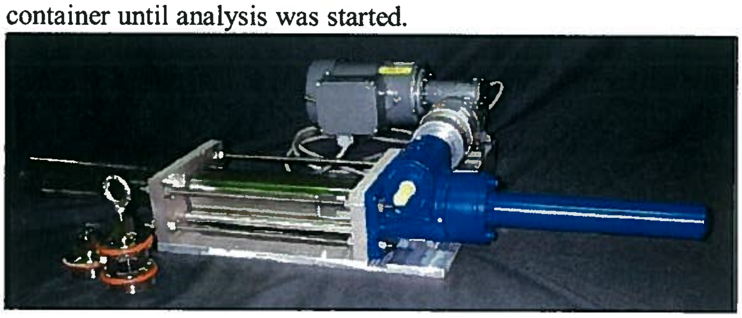

Figure 6: Core Extraction Tool

3.0 RESULTS/DISCUSSION

3.1 Sampler Vial Removal Results

The forces required to remove the sampler vials from the FCS are given in Table 1. Note the sampler numbering scheme is opposite the retrieval order; the bottom sampler is numbered 1 in the table. The grout fracture force was a fairly discrete event. The load would increase slowly until a popping sound was noticed along with a sudden drop in the load. In all cases but one, the post-fracture peak withdrawal forces were always lower than the fracture load. The data for Table 1 was taken from WSRC-NB-2006-00086 which also includes the R&D Direction and M&TE instrumentation used for the data collection.[5]

Table 1: Sampler Vial Extraction Forces (Measured)

Sampler Number	Grout Fracture Force (lb)	Post-Grout Fracture Peak Force (lb)	Comments
1	6048	3456	1728 lb - 3456 lb (after first 4 feet, forces were much lower)
2	2289	1296	<50 lb - 1296 lb (much of withdrawal was by hand)
3	3888	1728	<50 lb - 1728 lb
4	3456	2160	<50 lb - 2160 lb
5	3126	4752	432 lb - 3888 lb (typical post-fracture was in this range)
6	2148	1018	150 lb - 500 lb (typical post-fracture was in this range)

3.2 Sampler Vial Removal – Potential Causes for Higher Withdrawal Load

Extraction forces listed in Table 1 were notably higher than measured in the previous work (see SRNL-STI-2010-00167). A facility upper limit of 5000 lb had been discussed -- however it was understood lower extraction forces were the goal with the equipment. Table 1 illustrates not only is the fracture load higher than the previous work (compare to 1750 lb), but that a similar situation exists for the post-fracture load as well (compare to 300 lb). The similar trend in both pre- and post-fracture indicates the cause of the higher loads is probably due to housing bore geometry and related mechanical interference. The following paragraphs discuss this in more detail.

3.2.1 FCS Housing Bore Geometry

The dimensional tolerance applicable to the internal geometry of American Society for Testing & Materials -- ASTM A312 pipe (used for the housing) is wide relative to the needs for this application (+/-0.070).[6] Dimensional characterization of the FCS housing pipe bore was not performed prior to either test. However, in both cases the bore near the top end of the housing pipe was available for post-test measurement. Using calipers, the proof-of-concept work documented in Ref. 1 scaled between 2.644-2.647 in. The same measurement made in the full-scale mockup ranged from 2.621–2.678 in. The measurements indicate considerably more ovality is present in the full-scale test mockup. Vial assembly in the FCS housing bore for the full-scale mockup required noticeably more force than in the proof-of-concept work. Also the proof-of-concept work used seamless pipe for the housing, whereas the full-scale work utilized welded pipe. The presence of the extra ovality and weld seam in the latter likely contributes to the higher withdrawal forces. The larger insertion force requirement is consistent with such a conclusion for the full-scale work.

Further discussion related to the geometry and formed-core process is necessary to reveal other potential contributors to increased extraction load requirements. Consider the assembly with the sample vial positioned for operation within the housing pipe. Between the top two vial seals, a grout plug is cast as a result of the fill (Figure 8). The plug fills the annular region circumferentially at the grout inlet elevation. On the outside of the housing pipe, grout is also cured. After fracturing the grout, the withdrawal force then depends on a summation of seal and grout drag. The seals are elastic, so initial assembly fit is important as it creates the compressive load component of the seal drag force. (Seal drag is a function of radial compression and the prevailing friction.) It follows that the magnitude of seal drag force is then dependent on any geometric bore shape change encountered during withdrawal. Some examples of "bore shape change" would be hourglass-, barrel-, or curved-shape (Figure 7). Other examples could be dents created by rough handling or chucking tightly in a mill or a lathe.

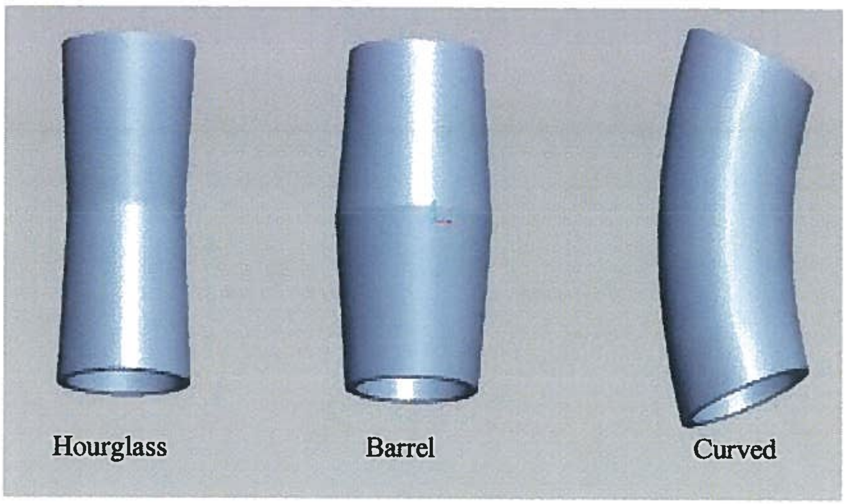

Figure 7: Illustration of Geometric Deviations

Compare the seal to the grout plug (Figure 8). The cast plug establishes a near zero-clearance in the bore since the bore acts as a form for the poured grout. The plug extraction load is independent of the assembled dimensional condition of the vial in the housing unlike the seals. Rather, the grout plug extraction load at any point in time after fracture is dependent on bore shape *change.* That is, if the change involves any reduction in plug clearance compared to the cast location, the grout will experience compression from the housing and the load increases – probably steeply. The obvious difference in stiffness between the seals and the grout plug makes the grout plug clearance a potentially dominant factor in the total extraction load. The issue is not confined to just the cross-sectional geometry at the plug location. That is, if curvature exists in the longitudinal axis of the housing pipe; additional plug interference may occur. That is in part due to the length of the vial and the proximity of the plug to one vial end. This means a simple diameter check may not be sufficient. Such interference can many times be relieved by slight flexure of the housing pipe; however, a housing grouted in the vault is constrained against flexure. Therefore controlling all relevant aspects of the bore geometry is important to ensuring lower and more consistent vial extraction force requirements.

Figure 8: Grout Plug Visible on Vial Shortly after Removal from the FCS

3.2.2 Seal Leakage

Another potential cause of increased extraction loads worth investigating is grout leakage past the inlet seals. It was noted that fluid was found on the top of most samplers – especially the vial closest to the bottom of the FCS assembly. Each sampler was examined on removal and no evidence of significant cured grout was found. The upper surface of the bottom sampler (above the top seal) was covered with approximately one-inch of liquid. After removal it appeared some grout might be present in this area; however, the higher withdrawal forces could not be attributed solely to this material. Upon withdrawal significant seal damage was witnessed. This damage appeared to be mechanical due to the tattered nature of the removed seals. Video of the bore revealed significant portions of the seal embedded in the grout at several of the fracture locations in the inlets.

3.2.3 Cable Tangling, Bunching & Bending

The tether cable connecting the vials may affect removal load requirements. Earlier it was noted the design requires sufficient slack in each cable to ensure only one vial is pulled at a time. To accomplish this, approximately 20' of steel cable has to be nested within approximately 3' of pipe bore between the bottom two vials. During assembly there was concern that the nested cable might entangle itself and create a situation where multiple vials were simultaneously loaded. One additional related concern is significant load might be required to straighten nested and jammed cable within the confined space of the housing.

3.2.4 Interface with other Sampler Vial Inlet Regions

During the test, it was observed that sometimes when pulling a vial through a region that had previously served as a grout inlet; the extraction force requirement would increase. This is expected as a small portion of the grout plug may remain and interact with the next few samplers as they pass through the region.

3.3 Core Extraction from the Sampler Vial

In the previous proof of concept work it was stated that a better core extraction tool than the arbor press was needed. [1] The improved core extraction tool was fabricated in the interim between that report and the present. In addition, the bore of the vial was electro-polished to minimize "grip". The revised tool is capable of producing 4000 lbs of extraction force on the core. To verify gains,

three vials of revised design were filled, cured and delivered to the new extraction tool. (This occurred between the earlier proof-of-concept testing and this testing.) All three cores were successfully extruded without any breakage. At present, the six cores cast and removed from the full-scale FCS assembly are still contained within the sampler vials awaiting analysis. A similar core extraction result is anticipated with these six.

4.0 CONCLUSIONS

4.1 Sampler Vial Extraction from the Assembly

The mockup and related data does not allow discrimination of the total removal force into each individual contributor – however, the following conclusions are stated based on observation and measurement.

- It was not concluded that grout leakage past the seals significantly affected removal force. While video showed standing liquid on the surface of several vials prior to extraction, only the bottom vial showed evidence of grout on the vial top surface during post-removal examination.
- There was no way to view the tether cable below a particular vial prior to or during its removal. However, tangled tether cable seems unlikely to be the cause of the higher removal forces. This statement is based on the observation that higher vial removal loads were noted prior to sufficient vial displacement to have tensioned the next nested cable.
- It is concluded that seal and/or grout drag were the main contributors to the larger magnitude removal load documented in this work. This is primarily attributed to dimensional issues associated with the housing bore shape cited above.
- In addition to housing bore geometry, removal load requirement increase may be caused by interference at grout inlet regions located at higher elevations than the vial fill position. This would be due to residual grout clinging to the bore. Portions of the o-ring were observed to be left behind in the fractured grout entrance region – these may also contribute to increased removal load in a similar fashion.

4.2 Core Extraction from the Sampler Vial

The cores created in this testing are still located within the sampler vials. Previous testing indicates the improvements related to the core extraction tool and the finish of the vial bore have solved any previous design issues. Similar acceptable results are expected during the extraction of the cores from the existing six filled vials.

5.0 RECOMMENDATIONS/PATH FORWARD

MS&CED does not recommended insertion of the current FCS assembly design into the Saltstone vault without better control of withdrawal load. The following alternative design modifications are listed for consideration prior to issuance of final design.

The recommendations detailed below are largely based on a mechanical perspective of the testing results. The first three modifications (related to reduction of sampler withdrawal load) may create non-mechanical impacts on the sampling process (materials, human factors, cost). Each is labeled "alternative" since one, or a combination of the three may be picked for implementation. Selection of a path forward should reflect input from both SRNL and Saltstone subject matter experts. Some effort may still be required to finalize details and logistics related to each particular

recommendation prior to final endorsement (e.g. material compatibility). The last two recommendations are more straightforward and the "alternative" modifier is dropped.

Alternative Modification 1:
This modification is best visualized as a duplicate of the current design except that the FCS housing is fabricated from thicker-wall pipe. In the current design a 2-1/2" Nominal Pipe Size (NPS) 10S pipe became the housing (as procured) except for the added grout inlet windows. This modification would require starting with a 2-1/2" NPS 40S pipe (or geometrically-similar tube) and honing the bore to a final dimension close to the nominal diameter of the current design. Honing would be the final manufacturing step to the housing (after welding and the grout inlet windows are finished). Post-hone housing bore tolerances of +/-0.005 inch[a] are reportedly achievable according to potential off-site sources. In the axial direction, interference effects of pipe curvature could be minimized if the honing tool length is close to that of the sampler vial. Post-hone surface finish should approach 32 micro-inches.

Pros vs. Tested Assembly

- Improved surface finish should reduce abrasion to o-ring during insertion.
- Improved FCS housing bore dimensional control should reduce average withdrawal load.
- Improved FCS housing bore dimensional control should reduce variation in vial insertion/extraction load and also enhance seal integrity.
- If the hone is of equal length or longer than the vial; the binding effect of FCS housing curvature should be nullified.

Cons vs. Tested Assembly

- No reduction in vial insertion length compared to current FCS Assembly relative to seal integrity.
- Ample lubrication is required along the entire length of the FCS Housing to increase likelihood of proper seal function after insertion.
- Significant shipment after final machining – potential transit damage (~25 ft long tubes).
- Axial curvature in the starting tube/pipe material could cause the stated bore tolerance to be exceeded.

Alternative Modification 2:
In this concept, the housing pipe used in the FCS is not continuous but rather segmented. A segmented housing allows access to the housing bore with lathe tools. This creates the possibility to customize the internal geometry of the housing to better suit our purposes. For example, a progressive step increase in the bore diameter at each seal location is possible (from bottom to top of the FCS Assembly). While the feasible bore size increase at each vial elevation is quite small; it may be adequate to reduce or eliminate some grout plug interference upon vial removal. At all other locations (away from the sealed grout inlet chamber) the housing bore could be enlarged to minimize the withdrawal load.

In Modification 1, the seal must be inserted a significant distance within the housing to arrive at the sampling elevation. With a segmented housing pipe, the primary grout seals may have to slide only a few inches during the assembly procedure. This enhances the likelihood of successful seal function by minimizing wear. Most seal manufacturers specify a well-lubricated 16 micro-inch finish for any reciprocating seal application. While a 32 micro-inch finish is achievable in modification 1 and is a significant improvement over the current, it is still considered slightly

[a] This tolerance requires an axially straight bore to achieve. Curvature in the bore to be honed will tend to have a widening effect on the tolerance band.

rough[b] to a seal manufacturer. If the primary grout seal's insertion path is accessible to a lathe the necessary finish can be customized in this area as needed. Not only can the insertion distance be minimized, but the surface finish can be optimized where important.

In order to use the same vial design and core size in conjunction with this modification, a slightly enlarged outer diameter should be considered for the FCS housing. The enlargement allows for the necessary wall thickness increase for threads or similar to connect the segments. The new outer diameter would be 3-inch. This should fit through the 3.068-inch nominal inner diameter inspection port located on the vault roof. Measured verification of assembly clearance will be a prerequisite. The clearance inspection consists of two gauges; the first, a right circular cylinder (plug) which must pass through the vault port; and a second, a hollow tube (same length as the vault port) must pass over the FCS housing. This type of inspection captures all three dimensions, which is required to guarantee assembly clearance.

The specifics of joining each section would be relegated to the detailed design of this modification. Welding is not anticipated to be an option unless provision for post-weld machining is made or a joint style design that promises insignificant bore deformation. Since the bore must remain open and the OD is also constrained, fastening must occur within the wall thickness. This indicates threads or interference fit via shrink or press means is the likely technique. An example of NPS 2-1/2" 10S pipe joined using a custom truncated thread is illustrated in Figure 9. Multiple connection techniques may be feasible if maximum loading on the joints is limited to the FCS assembly in the vault. That requires the housing outer surface to be given the proper machining treatment (grooves, etc.). The grooves are intended to improve housing/grout embedment. This should isolate the housing joints from the sampler vial extraction load path. The individual section lengths would likely be four feet or less.

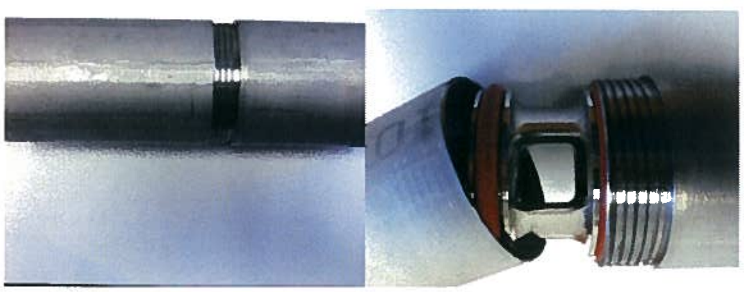

Figure 9: Segmented Housing shown Threaded at an Intermediate Position (left) and Fully Unthreaded (right)

Pros vs. Tested Assembly

- Best surface finish.
- Shortest o-ring insertion distance.
- Less o-ring lubrication required than other designs.
- Narrowest FCS housing bore dimensional tolerance should reduce removal loads and enhance seal integrity.
- Less final transit risk if manufactured onsite.

Cons vs. Tested Assembly

- More complex assembly.
- Have to seal joints – each represents a potential leak path.

[b] Reciprocating operation is more wear-adverse than the FCS insertion requirement – so strict compliance to manufacturer recommendation is likely conservative.

- Tighter assembly fit into vault – however pre-assembly dimensional inspection nullifies the risk.
- Internal surface at joint transition may create problems in practice if a catch point exists – requires more attention to detail in order to avoid.

Alternative Modification 3:
This modification involves introducing compliant material to fill some of the void space between the two top vial o-rings prior to the grout pour (Figure 10). This will prevent grout from filling the circumferential space occupied by the fill material. To visualize the concept, consider the vial positioned for grout sampling. In this position, insert a "plug" through each of the four square windows. (The two windows in the vial are annotated by the red "x" area in Figure 10. The housing not shown will have two square windows aligned with those in the vial.) The plug will have two access holes drilled at an angle such that expanding foam (or other acceptable fill material) can be delivered to occupy the void between the seals and the cork (see white-filled ellipse areas). Once the material has cured, the cork would be removed. This eliminates the grout plug formation with the exception of the portion bridging all four windows. The fill material prevents circumferential grout flow left and right of the sampler vial inlet window since that space is now occupied by compliant fill. This modification is expected to have beneficial effect on both pre- and post-fracture removal forces. It may be used in conjunction with either Modification 1 or 2.

Pros vs. Tested Assembly

- Minimizes issues with grout plug by nearly eliminating its formation.
- Mechanically simple to employ.
- May eliminate potential grout leak paths by preventing grout to access a majority of the seal perimeter.

Cons vs. Tested Assembly

- May create a technical issue related to material compatibility with the grout.

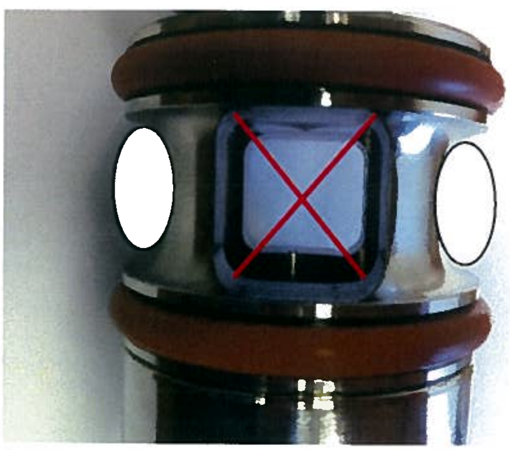

Figure 10: Vial Inlet & Upper Seals Region

<u>Modification 4</u>
This addresses the connections between the sampler vials. Instead of specifying the use of steel cable chokers – a chain is recommended. It is thought it will nest during assembly better than the cables that were used in the full-scale mockup.

<u>Modification 5</u>
As previously mentioned in several locations of this report, two pairs of dimensional inspection gauges should be fabricated. They will provide a pass/fail function. One pair should be designed relative to the insertion of the FCS housing through the Saltstone vault nozzle. The second pair would apply to the insertion of the sampler vials into the bore of the FCS housing.

6.0 REFERENCES

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[5] Coughlin J.T., "Laboratory Notebook: Mechanical Testing", WSRC-NB-2006-00086, p.126 -128

[6] ASTM A 312/A 312M – 11, Standard Specification for Seamless, Welded, and Heavily Cold Worked Austenitic Stainless Steel Pipe

APPENDIX A: Grout Preparation and Pouring

The grout sampler was designed and built to allow verification of the sample technique prior to installation in the vault. Core sampling had been conducted on test samples previously to serve as a baseline for evaluation of the sample vials after their removal.

Pump Testing

Testing was conducted in order to identify the proper pumps and tubing to supply the desired flow rate. Based on the diameters of the tank and sampler pipes, it was determined that a flow rate of approximately 18 ml/min in the test chamber was equivalent to the 1.5 inch/hour rate expected in the vault. Pump tests were carried out initially with water and then repeated with grout to verify that the indicated pump speed reflected actual conditions.

Two pumps were chosen to deliver the grout during the test pours. A Masterflex® model 7523-70 was selected for the nominal fill rate. This was used once the grout reached a level approximately 1 inch below the sample port to ensure the sampler was filled at the nominal vault fill rate. The initial filling of the test stand (up to the level of the port) was completed with a Randolf™ model 610 pump using 0.5" diameter tubing. The rate of fill could be controlled over a range with approximately 4 inch/min being the most common setting used. This pump was used to expedite the filling of the space between samplers.

The mixers selected were Caframo™ Stirrers model BDC 3030. These provide sufficient torque to thoroughly mix the dry powders with the salt solution. They are operated at a speed that produces sufficient mixing without a large vortex that would pull excess air into the mixture. As the level drops during the pouring operation, agitator speed may be reduced to limit splashing and vortex formation.

Material Preparation

The salt solution simulant is prepared according to the batch sheet in Table #1 below. It is based on a simulant Actinide Removal Process (ARP) – Modular Side Solvent Extraction (MCU) salt solution as described in the Grout Selection section of document SRNL-STI-2010-00167 (Ref #1). The sodium and aluminum concentration in this simulant are 5.44 M and 0.22 M respectively and were based on the remaining three batches of ARP/MCU waste. After the solution is thoroughly mixed, a sample is taken and the total solids and density are measured. The results are compared to known values as another check to verify proper batching.

The three components of the premix are individually weighed as directed on the batch sheet. The materials are placed in sealed plastic bags prior to mixing of the components which is accomplished by alternately pouring a portion of the individual materials into a larger plastic bag. This bag is then folded and shaken to mix the components. A uniform color indicates good mixing, but the final blending occurs when the premix is combined with the salt solution. A blend of 45% fly ash, 45% slag and 10 % Portland cement was used for the formed core testing.

Table A-1: Typical Batch Sheet for Salt Solution

Technician ____________
Batch Date ____________

Use 15 L Carboy
Record Tare Weight of Carboy (including cap) ____________ grams

Full Scale In-Core Test - Lift 6 ARP/MCU Salt Simulant

Label Carboy
Add chemicals in the listed order
DI water in step 9 can be used to flush weighing vessels. .
Inventory water mass if used for flushing
Using magnet mixing bar for mixing.

	Chemical	Target (grams)	Actual (grams)
1	DI Water	4402.6	
2	50% by Weight NaOH	2567.6	
3	NaNO3	1730.3	
4	NaNO2	454.4	
5	Na2CO3	223.4	
6	Na2SO4	100.2	
7	Aluminum Nitrate (9 H2O)	988.3	
8	Sodium Phosphate (12 H2O)	55.9	
9	DI Water	4402.6	
	TOTAL	14925.2	____________

Record Weight of Carboy (including cap) ____________ grams

When solids are completed dissolved - Measure the following:

Total solids #1 ____________ wt%
Total solids #2 ____________ wt%
Average Total Solids ____________ wt%
Density #1 ____________ g/ml
Density #2 ____________ g/ml
Average density ____________ g/ml

General Test Setup

The grout was mixed just prior to pouring. A minimum mix time of three minutes was always completed after the incorporation of the final pre-mix charge into the salt solution. Two mixing stations were used to provide a continuous supply for the pumps. The batch starts were staggered depending on the delivery rate required.

In general, plastic tubing with a 12" piece of stainless steel tubing at the end was used to deliver the product. The tubing was lowered into the test stand and the level was adjusted to keep the metal tubing slightly above the level of grout being poured. Each day's pour was recorded using a camera positioned outside the view port located at each sampler location. A second camera was lowered from the top of the stand along with the pour tube to allow visual observation of the grout flow.

Pour Description

Lift #1, 6/30/11, Partial Nominal Fill,

The pour started with the Roland™ pump and then was switched to the Masterflex® when the level was approximately ½ inch below the vial fill port. The small pump was run at 18 ml/min until it was estimated that the sample vial was ~95% full. Salt solution and premix bags identified and Lift 1A and 1B were used for this pour. The amounts of each component are identified in the attached data sheets. Toward the end of the pour there were problems with the line plugging.

Lift 2, 7/6/11, Nominal Fill

The second pour started with the small pump to complete the filling of the first sampler. Food coloring was added to the first salt solution batch in an effort to identify the interface between the two pour dates when the sample vial was recovered. After the first vial was filled, the large pump was used to bring the level up to just below the fill port of sample #2. The small pump was the used to fill sample vial #2 at a rate of 23 ml/min. Due to the slow pour rate, the grout level did not rise uniformly over the entire surface making an exact estimate of level in the sampler more difficult. The time to fill the second sampler was approximately 46 minutes although it took another 30 more minutes to completely cover the entire volume to the height of the sample port top. After the sampler was completely filled, the pump rate was increased to 170 ml/min to empty the container being used. Plugging problems caused the test to be terminated shortly after the sampler had been filled. Firmer Tygon® tubing was used with the small pump on this pour.

Lift 3, 7/8/11, Fast Pour

The third pour was completed entirely with the large pump using a rate of ~ 4"/min for filling the sampler. The pour continued until the grout level was 16 inches above the third sight port. In addition to the normal fill, three pipes and one valve were filled using grout dipped from one of the stirred buckets. The pipes are being used to study grout removal techniques.

Lift 4, 7/14/11, Fast Pour

The fourth pour was a fast pour using the large pump and a rate of ~4'/min. Grout was poured into the test stand until the level was just below the sampler opening. The pour was stopped until additional grout could be mixed to insure that the sampler would be filled continuously without having to wait for additional feed. At the completion of the pour, the grout level was 23 inches above the fourth sight port.

Lift 5, 7/19/11, Timed Pour

This pour was designed to pour for a specified time once the grout level reached the bottom of the fill port in the sampler. The level was raised using the large pump until the level was just below

the fill port. The small pump was then run for until the grout started entering the sampler and was run for 23 minutes. This time was estimated to partially fill the sampler. Based on the video it appears that the sampler was nearly filled after 23 minutes since the entire grout level began to show an increase.

Lift 6, 7/21/11, Nominal Pour
The large pump was used to complete the filling of the # 5 view port and raise the level up to the bottom of the #6 sampler fill port. The small pump was then used to slowly fill the sampler at a rate of 23 ml/min. Problems with the pump caused the final portion of the sampler to be filled at a faster rate to prevent plugging. The large pump was used to complete the filling of the test stand.

Temperature Measurements
The test unit was instrumented with thermocouples embedded in the samplers and also attached to the tank wall at each sampler location. This was completed in an effort to determine if there were differences between the sampler temperature and the bulk material in the tank. A review of the available data indicates that there is no significant temperature excursions associated with the sample vial thermocouples. The readings appear to fall within the range of ambient temperatures during the period of the tests. A more detailed review can be conducted as part of the sample analysis if deemed necessary.

Distribution

K.R. Hera, 723-A
A.D. Marzolf, 723-A
W.Y. Cheng, 723-A
C.C. Herman, 999-W
J.E. Occhipinti, 704-S
S.L. Tibrea, 730-A
L.T. Reid, 773-A
J.E. Marra, 773-A
J.M. Bricker, 704-27S
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
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J.N. Leita, 704-Z
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