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Contract No. NRC–HQ–12–C–02–0089
Account No. 20.17860.04

U.S. Nuclear Regulatory Commission
ATTN: Mr. Maurice Heath
Office of Nuclear Material Safety and Safeguards
Mail Stop TWFN 8F8
Washington, DC 20555

Subject: Intermediate Milestone 17860.04.005.510: Saltstone Leaching Experiments—Status Report

Dear Mr. Heath:

This letter transmits the subject intermediate milestone, prepared by the Center for Nuclear Waste Regulatory Analyses (CNWRA®) staff as part of the task order titled Technical Assistance for the Review of the U.S. Department of Energy's Non-High-Level Waste Determinations. This report documents fiscal year 2015 activities related to experiments on hydrological properties of and radionuclide release from simulated saltstone (Subtask 3-2). The CNWRA staff has developed protocols, made significant progress building the experimental apparatus, and produced radioactive and nonradioactive saltstone samples. Progress has been slowed by a prolonged vendor response to problems with the pump system that is designed to support flow-through release experiments on intact saltstone columns. We are now positioned to make significant experimental advances during the next fiscal year.

The report is attached in Microsoft® Word and PDF versions. We also will make the report available to the U.S. Nuclear Regulatory Commission (NRC) staff in a convenient location on the CNWRA-hosted shared drive.

If you have any questions regarding this report, please contact Dr. Robert Lenhard at (210) 522-6418 or me at (210) 522-5582.

Sincerely,

David Pickett, Ph.D., P.G.
Senior Program Manager
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SALTSTONE LEACHING EXPERIMENTS
STATUS REPORT

Prepared for

U.S. Nuclear Regulatory Commission
Contract NRC–HQ–12–C–02–0089

Prepared by

Robert Lenhard and Donald Hooper

Center for Nuclear Waste Regulatory Analyses
San Antonio, Texas

September 2015

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This report was prepared to document work performed by the Center for Nuclear Waste Regulatory Analyses (CNWRA®) for the U.S. Nuclear Regulatory Commission (NRC) under Contract No. NRC–HQ–12–C–02–0089. The activities reported here were performed on behalf of the NRC Office of Nuclear Material Safety and Safeguards, Division of Decommissioning, Uranium Recovery, and Waste Programs. The report is an independent product of CNWRA and does not necessarily reflect the views or regulatory position of NRC.

Assistance from Sitakanta Mohanty in testing the pumping system and interacting with the manufacturer to correct problems with it is appreciated. The authors thank David Pickett for technical review, and Wesley Patrick for programmatic and editorial reviews. The authors also thank Mark Hickinbotham and Arturo Ramos, who provided word processing support in preparation of this document.

QUALITY OF DATA, ANALYSES, AND CODE DEVELOPMENT

DATA: All CNWRA-generated data contained in this report meet the quality assurance requirements described in the Geosciences and Engineering Division Quality Assurance Manual. Sources for other data should be consulted for determining the level of quality for those data. Hooper (2015) and Lenhard (2015) were used to document the experimental conditions and activities.

ANALYSES AND CODES: None.

REFERENCES:

Hooper, D. "Saltstone and Tank Grout Batching and Specimen Preparation." Scientific Notebook No. 1242E. San Antonio, Texas: Center for Nuclear Waste Regulatory Analyses. 2015.

Lenhard, R. "Saltstone Breakthrough Experiments to Investigate Potential Technetium Mobility." Scientific Notebook No. 1258E. San Antonio, Texas: Center for Nuclear Waste Regulatory Analyses. 2015.

1 INTRODUCTION

The Center for Nuclear Waste Regulatory Analyses (CNWRA®) is providing technical assistance to the U.S. Nuclear Regulatory Commission (NRC) for meeting its statutory responsibilities under the Ronald W. Reagan National Defense Authorization Act of Fiscal Year 2005 (NDAA). The technical assistance encompasses a range of support related to NRC reviews of the non-high-level waste (non-HLW) determinations that the U.S. Department of Energy (DOE) prepared for the Savannah River Site (SRS), including independent analyses that can be used in NRC reviews of DOE performance assessments under the NDAA.

This report summarizes work conducted by CNWRA for the NRC project entitled Technical Assistance for the Review of the U.S. Department of Energy's Non-High-Level Waste Determinations (Contract No. NRC–HQ–12–C–02–0089, Task Order 004) during Fiscal Year 2015. Specifically, it documents work conducted under Task 3 of the project related to saltstone leaching experiments, which are laboratory studies to evaluate potential radionuclide release rates from saltstone, a waste grout formulation used by DOE at the SRS.

2 SALTSTONE LEACHING EXPERIMENTS

In support of the saltstone leaching experiments, CNWRA conducted several activities. These included (i) a review of related DOE documents, (ii) design of saltstone curing conditions and leaching experiments, and (iii) preparation of saltstone samples. Each of these activities is briefly discussed in the following sections.

2.1 Review of Related DOE Documents

Several DOE reports on the physical and chemical characteristics of saltstone were reviewed (Cantrell and Williams, 2012; Estes et al., 2012; Seaman and Chang, 2014a; Seaman et al., 2014b). The purposes of this focused literature review were to better understand the scope of previous experimental work and to become sufficiently familiar with the DOE procedures to use them as guidance for designing saltstone curing conditions and leaching experiments. Based on this review, it was determined that DOE cured the saltstone samples and prepared them for testing under oxic conditions. Furthermore, DOE did not conduct any experiments on saltstone samples by flowing an aqueous phase through a cured, consolidated, saltstone sample.

2.2 Design of Saltstone Curing Conditions and Leaching Experiments

Consistent with anticipated conditions at the SRS, NRC provided technical direction that (i) the leaching experiments should be conducted on cured, consolidated saltstone samples; (ii) the saltstone samples with technetium-99 (Tc-99) should be cured under low oxygen conditions; and (iii) Tc-99 release from the saltstone samples would be the primary constituent to be measured. To accommodate these required conditions, which contrast with those reported by DOE, a curing cell and experimental approaches were designed. The approaches and the chemical formulation of the saltstone were discussed with NRC staff in conference calls throughout the year. Following NRC acceptance of the approaches, appropriate materials and equipment were obtained. The chemical formulation of the saltstone samples and the design for the curing and experimental systems are outlined in the following sections.

2.2.1 Chemical Formulation of the Saltstone Samples

The saltstone formulation consists of 45 percent blast-furnace slag, 45 percent fly ash, and 10 percent Portland cement by dry mass, which is mixed with a salt solution. The blast-furnace slag, fly ash, and Portland cement were obtained from the same vendor that supplies SRS. The chemical formulation of the salt solution that was mixed with the blast-furnace slag, fly ash, and Portland cement is listed in Table 2-1. The composition of the salt solution is called the actinide removal process (ARP)/modular caustic side solvent extraction unit (MCU) salt simulant. The same salt solution composition was used in the studies of Seaman and Chang (2014a and Seaman et al. (2014b) and used to make saltstone at the SRS.

The compounds in Table 2-1 were mixed with distilled water to yield 1-liter solutions. During this fiscal year, two 1-liter salt solutions were made: one with and one without Tc-99. The total Tc-99 activity in the 1-liter salt solution was 5.365 MBq [145 µCi], yielding a concentration of 4405 Bq/g [0.119 µCi/g]. The procedures for mixing the Tc-99 in the salt solution are contained in Lenhard (2015). The salt solution was mixed with the blast-furnace slag, fly ash, and Portland cement at a water-to-dry-mass ratio of 0.6. Further details on the mixing of the saltstone samples can be found in Hooper (2015) and Lenhard (2015). The saltstone sample made without Tc-99 will be used for refining experimental protocols for saltstone samples with Tc-99 and assessing hydraulic properties of the saltstone samples. The saltstone sample made with Tc-99 will be used in the leaching experiment.

2.2.2 Saltstone Curing System

Curing was conducted on saltstone samples with and without Tc-99. At the SRS, Tc-99 will be contained in the saltstone mixture, which will yield a reducing chemical environment. The Tc-99 will achieve a chemically reduced state, thus becoming less mobile. DOE also believes that the oxygen content of the saltstone mixture as it cures in vaults at the SRS will be very low. Therefore, oxygen is being limited during curing of the saltstone samples with Tc-99. For the saltstone samples without Tc-99, there is no need to limit exposure to oxygen during curing of the samples, because the samples will be used to test protocols for the leaching experiments with Tc-99 saltstone samples and for determining hydraulic properties of the saltstone mixture, which are not affected by exposure of the mixture to oxygen.

For the saltstone samples without Tc-99, an ESPEC Temperature and Humidity Bench Top Chamber (model BTL–433) was used under ambient (oxic) conditions to cure the samples. The temperature and humidity of the chamber was programmatically controlled. To ensure that the relative humidity was close to 100 percent, two glass beakers with free water were placed in the chamber. The water was replenished, as needed. Ambient conditions were used because the samples will be used only for refining experimental protocols for the Tc-99 leaching experiments and for determining hydraulic properties.

For the saltstone samples with Tc-99, oxygen needs to be limited from the curing system because DOE believes that the oxygen content will be very low in the saltstone vaults at SRS. Therefore, a low-oxygen curing system was designed and discussed with NRC staff. Key elements of the design were to (i) provide inert gas to keep the oxygen content low, (ii) maintain a 100 percent relative humidity environment, (iii) control temperatures between 25–65 °C [77–149 °F], and (iv) monitor the oxygen content in the gas phase at the parts per million (ppm) level. The objective was to maintain the oxygen concentration below 1 ppm in the gas phase. The design is shown in Figure 2-1.

Table 2-1. Composition of ARP/MCU* Salt Simulant		
Compound	**Molarity (moles/L)**	**Mass for 1 L (g/L)**
Sodium Hydroxide 50 weight percent NaOH	1.594	127.52
Sodium Nitrate $NaNO_3$	3.159	268.52
Sodium Nitrite $NaNO_2$	0.368	25.39
Sodium Carbonate $NaCO_3$	0.176	18.66
Sodium Sulfate $NaSO_4$	0.059	8.38
Aluminum Nitrate $Al(NO_3)_3 \cdot 9H_2O$	0.054	20.25
Sodium Phosphate $Na_3PO_4 \cdot 12H_2O$	0.012	4.56

*Actinide Removal Process/Modular Caustic Side Solvent Extraction Unit
Seaman, J.C. and H. Chang. “Dynamic Leaching Characterization of Saltstone.” SREL Doc: R–14–0007. Version 1.0. Aiken, South Carolina: Savannah River Ecology Laboratory, University of Georgia. 2014.
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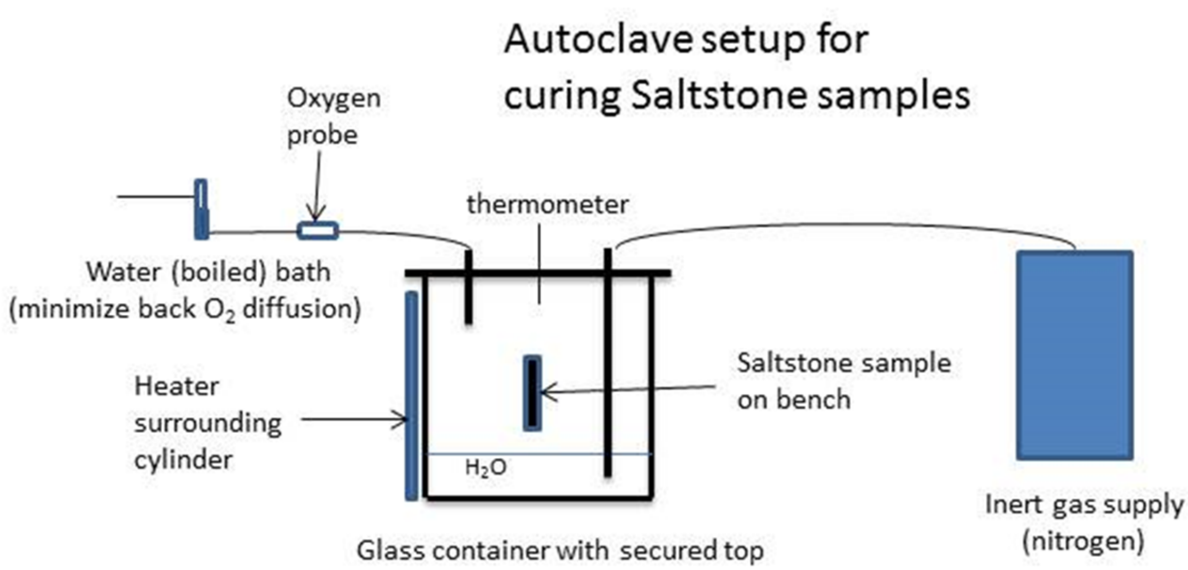

Figure 2-1. Conceptual Design of Curing System for Low-Oxygen Conditions

For the inert gas, ultra high purity nitrogen gas (NI UHP300) from Airgas, in San Antonio, Texas, is used. The reported oxygen content in the nitrogen gas is less than one part per billion (<1 ppb). Tubing connects the gas cylinder to the curing cell. The gas is delivered to the cell through a reservoir of deoxygenated, distilled water on the bottom of the curing cell. As the nitrogen gas bubbles through the water reservoir, the gas becomes saturated with respect to water, enabling a 100 percent relative humidity environment within the curing cell. As needed, the water reservoir can be replenished through tubing, which is not shown in Figure 2-1. The saltstone mold is elevated above the water reservoir so that curing will not be conducted under water-saturated conditions. Figure 2-2 shows the curing cell before the nitrogen gas line was connected. The nitrogen gas exits the curing cell through tubing near the top of the cell. The tubing goes to a Y-type glass connector where one leg of the Y-section is connected to a tube that goes to an oxygen probe to monitor the oxygen content of the gas leaving the curing cell. The other leg connects to a tube that goes to a water bath, which facilitates visually monitoring the nitrogen gas flow rate through the curing cell and limits backward diffusion of oxygen into the curing cell.

A low oxygen concentration can be maintained by the continuous flow of nitrogen gas through the curing cell. A cylindrical autoclave heater is placed around the curing cell and a thermocouple provides monitoring to support temperature control. A thermometer serves as a check on the thermocouple and monitors temperature within the curing cell. Figure 2-3 shows the curing system after all connections are made. Two curing cells were developed so that two saltstone samples can be cured simultaneously, each with a different curing time protocol (i.e., 90 days and one year).

2.2.3 Saltstone Leaching Experiments

To generate liquid flow through low-permeability-consolidated samples in a reasonable time period, a relatively large injection pressure needs to be applied. Because tests similar to these are common in the petroleum industry, Coretest Systems, Inc. (a company in Morgan Hill, California, that supplies measurement systems to the petroleum industry) was contacted to assist CNWRA in designing a required low-dead-volume, high-pressure permeameter. The primary elements of the test system are a cell to hold the saltstone samples under high pressures and a high-pressure, liquid pump. Another important design feature is for the cell to prevent bypassing of pumped liquid around the saltstone sample.

The custom permeameter purchased from Coretest, Inc. for conducting the saltstone leaching experiments is shown in Figure 2-4. The permeameter cell is designed to test cylindrical samples 3.81 cm [1.5 in] in diameter. Internal to the permeameter cell is a Buna confining sleeve (Figure 2-5) that surrounds the cylindrical saltstone samples. Between the sleeve and the metal permeameter cell, hydraulic pressure can be applied uniformly to the longitudinal surface of the sample to form a tight seal.

Bypassing of pumped liquid around the saltstone samples is prevented by applying a higher hydraulic pressure around the cylindrical saltstone samples than the liquid pressure applied to the front end of the sample. Spacers and end caps are placed on the front and back end of the saltstone sample to minimize dead volume and secure the sample in the permeameter cell. Stainless steel tubing 0.159-cm [0.0625-in] in diameter connects the high-pressure pump through the front end cap to the saltstone sample. Stainless steel tubing also is used to pass liquid flow through the saltstone sample through the back end cap where fluid samples can be extracted. There is little dead volume between the back end of the saltstone sample and the

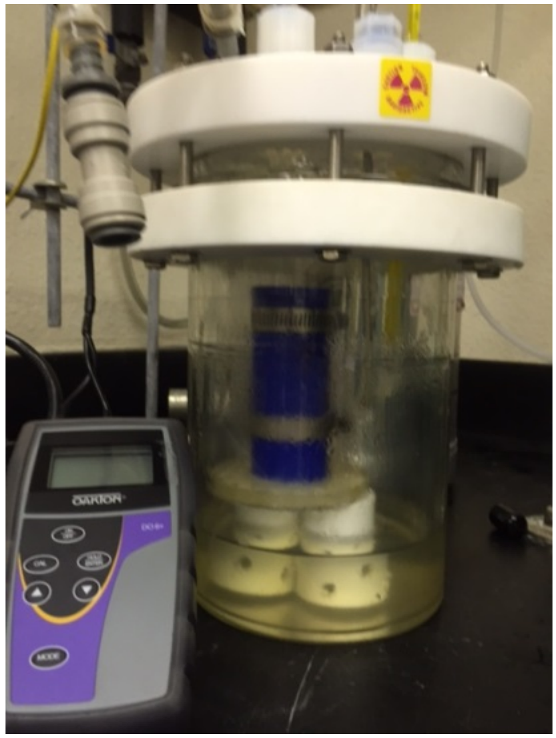

Figure 2-2. Curing Cell with Saltstone Sample and Oxygen Meter

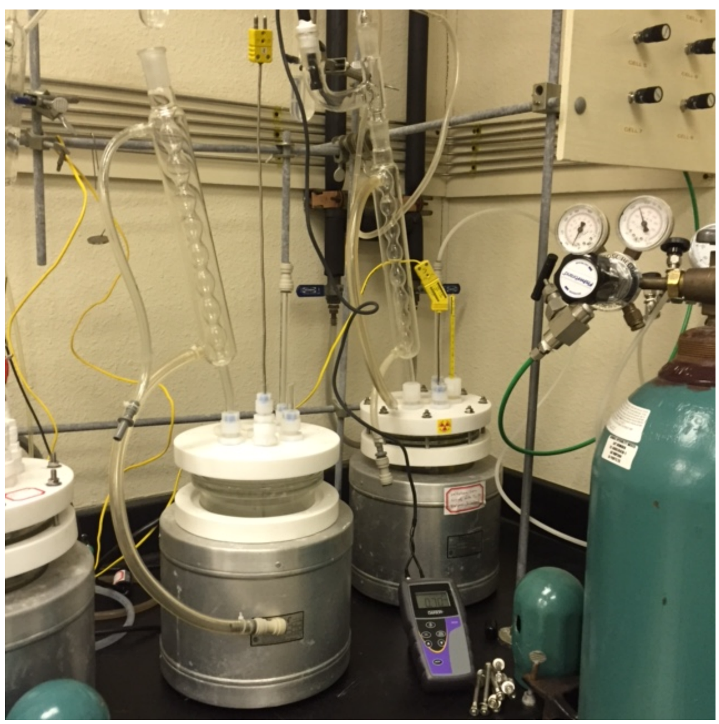

Figure 2-3. Setup of Two Curing Cells with Autoclave Heaters

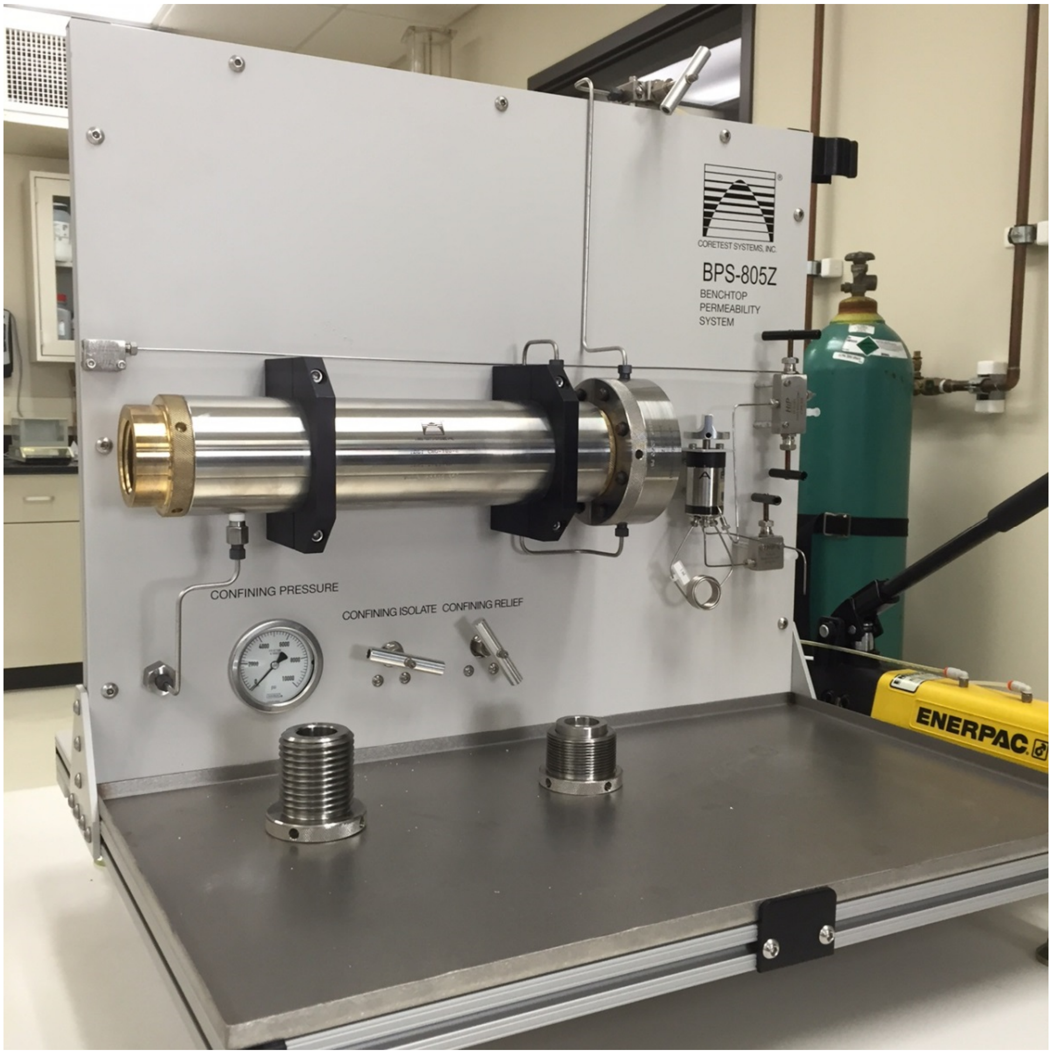

Figure 2-4. Custom Permeameter Purchased from Coretest, Inc.

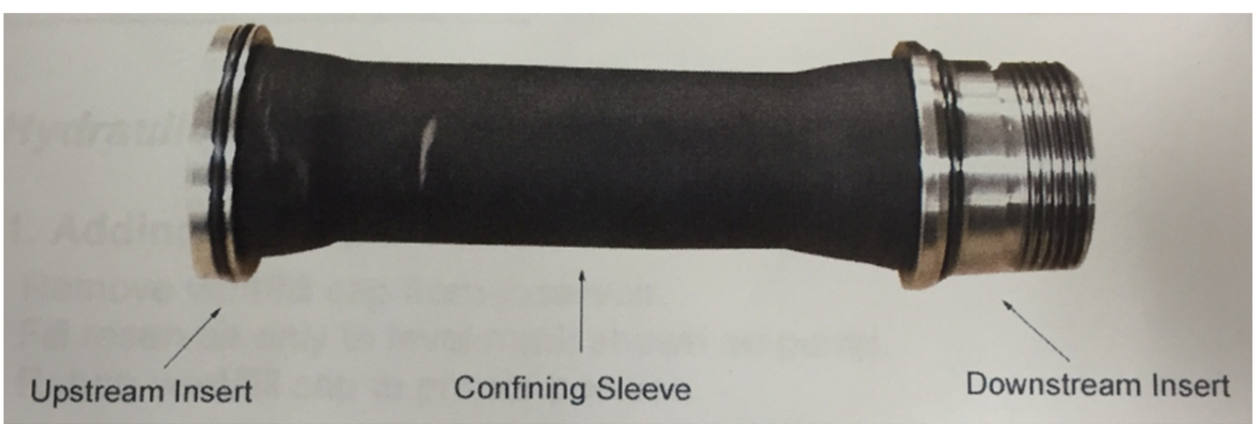

Figure 2-5. Buna Confining Sleeve That Holds the Saltstone Cylindrical Samples

sampling location, so that chemical measurements will better reflect the compositions of solutions exiting the samples.

The primary high-pressure pump is a Quizix Model SP-5200, which was reconditioned and upgraded by the manufacturer to a capability of up to approximately 340 atm [5,000 psi] pressure. Extensive testing of the reconditioned pump showed that the pump was not performing to specifications, requiring further work by the manufacturer.

A combination of these performance problems with the pump and project funding constraints has delayed initiating saltstone leaching experiments until next year. To mitigate further technical risk, a backup to the primary high-pressure pump is being procured from another manufacturer. The backup pump will be capable of conducting flow experiments at approximately 340 atm [5,000 psi] of pressure under either constant pressure or constant flow modes.

2.3 Preparation of Saltstone Samples

In parallel with design, development, and testing of the experimental apparatus, saltstone samples were prepared. Three saltstone samples, each 3.81 cm [1.5 in] in diameter and 10.16 cm [4 in] long, were made: two without Tc-99 and one with Tc-99 spiking.

2.3.1 Saltstone Samples Without Tc-99

The saltstone samples without Tc-99 are to be used for refining experimental protocols for the samples with Tc-99 and for measuring hydraulic properties. Two saltstone samples without Tc-99 were mixed and poured into silicone molds on April 30, 2015. Figure 2-6 shows a silicone mold on a Petri dish. Prior to pouring the saltstone mixture into the molds, hot glue (Arrow® All Purpose Clear Mini Glue Sticks) was placed along the bottom of the mold to prevent free liquid from draining from the saltstone paste while the hydration process is underway, helping to preserve the water-to-dry-mass ratio of the saltstone mixture. The Petri dish captures any liquid if drainage does occur.

After pouring the saltstone paste into the molds, the molds were placed in the temperature- and humidity-controlled chamber (ESPEC Temperature and Humidity Bench Top Chamber; model BTL–433), where exposure to oxygen was not restricted. In consultation with NRC, a 30-day temperature-controlled curing protocol was devised that, based on information from DOE, reasonably reflects thermal conditions during saltstone curing in the SRS vaults. Although not a precise match to field conditions, the 30-day temperature protocol allows for a relatively short curing duration to facilitate timely testing of protocols and hydraulic properties. The temperature in the chamber was raised from 25 °C [77 °F] on April 30, 2015 to 65 °C [149 °F] by daily 5 °C [9 °F] increments. Afterwards, the temperature in the chamber was held constant at 65 °C [149 °F] for 14 days and then reduced by 5 °C [9 °F] increments every day until the temperature was 25 °C [77 °F] on May 29, 2015. Thereafter, the temperature in the chamber was held constant at 25 °C [77 °F] and 100 percent relative humidity.

On July 7, 2015, one saltstone mold was removed from the temperature- and humidity-controlled chamber, after which the cylindrical saltstone sample was extracted from the mold and subjected to acoustical testing. The dual purposes of the testing were to determine the severity of ultrasonic attenuation in saltstone and to determine whether the ultrasonic properties of saltstone are stable during later stages of hydration. The acoustical testing of the saltstone samples is described in Puchot et al. (2015). The second saltstone mold remains in the

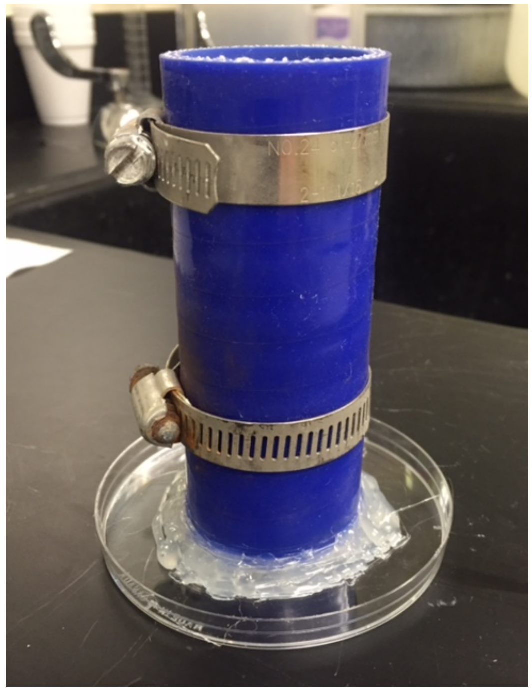

Figure 2-6. Silicone Mold Used To Form Saltstone Samples

temperature- and humidity-controlled chamber, and will remain there until project funds are available next fiscal year for refining experimental procedures and performing hydraulic measurements.

2.3.2 Saltstone Sample With Tc-99

A saltstone sample with Tc-99 was mixed and poured into a silicone mold on September 3, 2015. Procedures for mixing the saltstone materials and pouring the paste in a mold are recorded in Hooper (2015; Lenhard, 2015). Immediately after pouring the paste into the mold, the mold and Petri dish were placed on white posts inside the low-oxygen curing cell (see Figure 2-2). The posts serve to elevate the Petri dish and curing mold so that they are above the water reservoir on the bottom of the curing cell. Thereafter, connections were made to supply nitrogen gas to the curing cell (see Figure 2-3). The following day, the temperature within the curing cell was raised 5 °C [9 °F] to 30 °C [86 °F]. Every day afterwards, the temperature was raised another 5 °C [9 °F] until the temperature within the curing cell was 65 °C [149 °F], at which time the temperature in the curing cell was held constant. The plan is to hold the temperature at 65 °C [149 °F] for 29 days and then lower the temperature 5 °C [9 °F] every day until the temperature in the curing cell reaches 40 °C [104 °F]. The tentative plan is to keep the temperature at 40 °C [104 °F] until December 2, 2015, which will provide a curing time of 90 days under controlled conditions. The 90-day temperature curing protocol was devised in consultation with NRC based on information from DOE concerning thermal conditions during saltstone curing in the SRS vaults.

After sealing the curing cell and beginning nitrogen gas flow, the oxygen content of the gas exiting the curing cell was monitored with the oxygen probe. After the first day of curing, when the oxygen concentration was relatively high, the oxygen content has not exceeded one part per million (1 ppm) (Table 2-2). Monitoring will continue during the full curing cycle, but at a reduced frequency.

Table 2-2. Oxygen Content of the Gas Exiting the Curing Cell	
Day	**Oxygen Content (ppm)**
September 3, 2015	6.64
September 4, 2015	0.72
September 5, 2015	0.70
September 6, 2015	0.66
September 7, 2015	0.71
September 8, 2015	0.67
September 9, 2015	0.66
September 10, 2015	0.65
September 11, 2015	0.64
September 12, 2015	0.65
September 13, 2015	0.62
September 14, 2015	0.63
September 15, 2015	0.63
September 16, 2015	0.62
September 17, 2015	0.65
September 18, 2015	0.60
September 21, 2015	0.59
September 22, 2015	0.59
September 23, 2015	0.60

3 FUTURE WORK

To achieve the objectives stated in Section 1 of this status report, additional work will be conducted as described in the task-order proposal, taking into consideration discussions with NRC staff that will continue throughout the year. It is anticipated that the focus of work next fiscal year will be on (i) using the remaining saltstone sample without Tc-99 to assess hydraulic properties and (ii) using the saltstone sample with Tc-99 currently undergoing curing to conduct a saltstone leaching test. Other activities will involve testing the high-pressure pump, testing the new backup pump, and making one or two additional saltstone samples with Tc-99. If time and funding permit, a second saltstone leaching experiment may be conducted. The saltstone leaching tests may take several months to complete, depending on the number of pore volumes to measure.

Both for determining hydraulic properties and conducting the saltstone leaching experiments, it is important that the pump produce steady-state flow conditions in the saltstone samples with accurately measured and controlled volumetric flow rates. Therefore, the pumps will be extensively tested using long, narrow, metal tubing that will provide sufficient liquid flow resistance so that high pumping pressures are needed to generate reasonable discharge rates from the end of the tubing. Under the constant pressure mode, the pump will be tested to evaluate whether steady-state flow conditions can be established and to assess the stability of the steady-state conditions over time. The volumetric discharge from the tubing will be measured as a function of time to assess steady-state conditions and compare the actual volumetric flow rate with that displayed by the pump software. Similarly, under the constant flow mode, the discharge from the tubing will be compared to readings of the volumetric flow rate of the pump. Pump performance will be evaluated at several constant pressure and constant pumping rates relevant to the anticipated experimental conditions.

Hydraulic properties will be measured using the saltstone samples that were made without Tc-99. The key hydraulic properties are permeability (units of m^2), or saturated hydraulic conductivity (units of ms^{-1}), and porosity. The hydraulic properties are not expected to change based on whether or not Tc-99 is present, especially given the relatively low Tc-99 mass in the saltstone (estimated at less than 3.4×10^{-4} g [1.2×10^{-5} ounces]. The hydraulic properties are a function of the configuration of the pore spaces within the saltstone samples. The presence or absence of Tc-99 salts in the samples is not likely to affect the formation of the pore spaces or block flow through the pore spaces at low Tc-99 concentrations. The saturated hydraulic conductivity will be calculated under steady-state flow conditions, using the measured liquid pressure differential across the saltstone sample. The solution used will be simulated SRS groundwater. The permeability will be estimated based on the measured saturated conductivity and the properties of this water using the relationship between the saturated hydraulic conductivity and permeability shown in Eq. (3-1).

$$K = \frac{k\rho g}{\mu} \tag{3-1}$$

where K = saturated hydraulic conductivity; k = permeability; ρ = fluid mass density; and μ = dynamic fluid viscosity.

The saturated hydraulic conductivity depends on fluid properties, whereas the permeability is independent of fluid properties. Therefore, the saturated hydraulic conductivity is dependent on temperature because fluid properties depend on temperature. Porosity will be estimated based on determining the water volume loss between saturated and oven-dry conditions. It also will be estimated based on the saltstone bulk density.

Leaching experiments will be conducted on the saltstone samples with Tc-99 under steady-state flow conditions in which at least one pore volume of solution can be produced from the samples every 3 days. The liquid discharge will be collected periodically and measured for Tc-99 concentration by liquid scintillation decay counting. Sufficient measurements will be taken so that a well-defined Tc-99 breakthrough curve can be developed as a function of pore volume. Results will be communicated and discussed periodically with NRC staff. From the discussions and available funds, it will be determined when to terminate the saltstone leaching experiments. For the first leaching experiment, the simulated SRS groundwater solution will not be de-oxygenated prior to injection into the saltstone sample. A second leaching experiment may be conducted that uses de-oxygenated, simulated SRS groundwater.

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