Evaluation and Interpretation of Properties for Saltstone Cores Extracted from an SDU – 17344

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ABSTRACT

Extraction and subsequent analysis of emplaced saltstone samples from a Savannah River Site (SRS) Saltstone Disposal Unit (SDU) were performed in order to realize the following key objectives: (1) to support the use of historical and future laboratory-derived sample data to predict the behavior of field-emplaced samples, and (2) to substantiate the saltstone properties currently assumed in Saltstone Disposal Facility (SDF) transport modeling, i.e., Performance Assessment (PA) and Special Analyses (SA) models. Multiple cores were extracted via a wet core drilling process in April-May 2015 approximately 20 months after the saltstone of interest was processed in the Saltstone Production Facility (SPF) and subsequently emplaced in SDU Cell 2A (SDU2A). The retrieved samples were stored in an inert N_2 environment prior to, and during, the property testing that took place between October 2015 and March 2016. Sample analyses comprised evaluation of physical properties (density, porosity, and moisture content), saturated hydraulic conductivity (K_{sat}), and determination of distribution coefficients and solubilities for key radionuclides. In general the SDU2A samples indicated comparative properties to laboratory-prepared samples and additionally verified many of the inputs currently assumed in the SDF PA. The principal exception was related to the leaching characteristics of I-129, which demonstrated a lower than anticipated affinity for retention within the saltstone matrix.

OVERVIEW

Saltstone is a cementitious, low-level waste (LLW) material processed and stored at the Savannah River Site (SRS). It consists of fly ash (FA), blast furnace slag (BFS), and Portland cement that are blended with LLW salt solution. The dry feed materials and the salt solution are mixed at the Saltstone Production Facility (SPF), and the fresh grout subsequently pumped into a Saltstone Disposal Unit (SDU) in the Saltstone Disposal Facility (SDF) where it cures to form a rigid, cementitious monolith. The long-term (hundreds to thousands of years) integrity of saltstone and its ability to retain radionuclides and prevent their transport to the surrounding environment is modeled in the SDF Performance Assessment (PA) [1]. The PA has historically relied on assumptions with respect to saltstone properties that have been derived from laboratory-prepared samples. However, it is not known if laboratory-prepared samples are truly representative of field-emplaced saltstone material. As such a strategy was developed in which actual saltstone would be extracted from an SDU (specifically SDU2A) and compared with simulated laboratory samples that were processed and cured in a manner that attempted to mimic the field-emplaced saltstone material; details regarding the overall strategy, the extraction of the emplaced saltstone samples, and the preparation and curing of the laboratory samples are detailed in References 2-5. Properties of interest for comparison of the SDU2A samples and the laboratory-prepared samples are as follows (details regarding the analyses conducted with respect to these properties are provided in References 6-8):

Physical Properties – physical properties, such as density, total and permeable porosity, and water content, are a relatively simple means of evaluating potential disparity between field- and laboratory-prepared samples that may result from differences in processing. For example, the laboratory sample preparation (including accurate measurement of components, homogeneous mixing, and controlled transfer to curing molds) may yield different sample densities and pore volumes to samples that are bulk processed in the SPF, pumped through transfer lines, and ultimately emplaced by free-fall into the SDU. Physical properties, in particular porosity, may also provide insight with respect to, for example, observed differences in the saturated hydraulic conductivity (K_{sat}) values for the SDU2A and the laboratory-prepared samples.

Saturated Hydraulic Conductivity (K_{sat}) – K_{sat} is a measure of the ease by which fluid can be transported through permeable pore spaces, and is a key SDF PA parameter for modeling liquid transport through saltstone and the subsequent leaching of contaminants to the surrounding environment. K_{sat} is not a function of porosity alone but depends on total porosity, pore size and distribution, and the connectivity and tortuosity of pore channels in the material. As mean pore size decreases so the K_{sat} decreases. The saltstone K_{sat} currently used for SDF transport modeling is 6.4E-09 cm/sec. [9]

Radionuclide Solubility and Distribution Coefficient (K_d) – the ability for radionuclides to remain immobilized by the saltstone matrix as groundwater infiltrates the SDU interior is a key determinant with respect to the rate of radionuclide transport to the surrounding environment. Radionuclides of primary concern include I-129, Se-79, Sr-90, Ra-226, and Tc-99. Of these I-129, Se-79, Sr-90, and Tc-99 are designated as radionuclides contained within saltstone that may contribute most significantly to future radiological risks to workers, the public, and the environment. [10] In addition, I-129, Ra-226, and Tc-99 are the primary contributors with respect to the peak dose to chronic intruders after closure. [9] While the initial intent of the leaching analysis was to evaluate the five aforementioned radionuclides, Se-79 and Ra-226 could not be analyzed since their concentrations were below detection limits. [8] Note that the results of the distribution coefficient analyses were reported as distribution ratios (R_d) since it was not confirmed that equilibrium was reached during the extraction test. [8] However, the results will be discussed in this paper as K_d for comparison to the values in the PA. Immobilization of radionuclides by the cementitious saltstone matrix can occur via a number of mechanisms; these include:

- *Phase Incorporation* – radionuclide ions may be chemically substituted into the complex Ca-Si-(Al)-H gel structure during cementitious material hydration. Chemical matrix incorporation is probably the most stable form of radionuclide immobilization and limits exposure of the radionuclides to infiltrating groundwater.

- *Solubility Limitations* – radionuclides (or rather the compound forms in which they are in) that exhibit low solubility in groundwater infiltrating the saltstone monolith will remain immobilized longer than those radionuclide compounds that are readily soluble. Solubility is pH dependent but the presence of oxygen in the groundwater may also influence the solubility of radionuclides; this is the case for redox sensitive Tc-99. In reducing, high pH cementitious environments Tc exists in its reduced +4 (IV) oxidation state in the form of sparingly soluble TcS_x or $TcO_2.xH_2O$. [11] It is believed that sulfide (S^{2-}) and ferrous iron (Fe^{2+}), both present in the BFS component of saltstone, serve as Tc reductants. [12] However, in the presence of O_2 the +4 oxidation state can be oxidized to the +7 state (i.e., pertechnetate (TcO_4^-)) which is highly soluble, and as such can be readily leached from the monolith via infiltrating groundwater. [13]
- *Adsorption* – dissolved radionuclide ions may adsorb to the surface of the cementitious particles. In the high pH pore solutions of saltstone, anions, such as I^- and TcO_4^-, are poorly adsorbed to the cementitious solids because: (1) the radionuclide anions in solution are competing with significantly higher concentrations of hydroxyl anions (OH^-) for coordination sites on the solid surfaces, and (2) hydroxyl anions that adsorb to the solid surfaces electrostatically repel the I^- and TcO_4^- anions. Dissolved cations, like Sr^{2+}, are expected to be better adsorbed to the cementitious solids at high pH.

SAMPLE DATA

The SDU2A cores that were analyzed by Savannah River National Laboratory (SRNL) as part of this study are indicated in Figure 1. Inset text indicates the analysis type that was conducted on specific sub-cores.

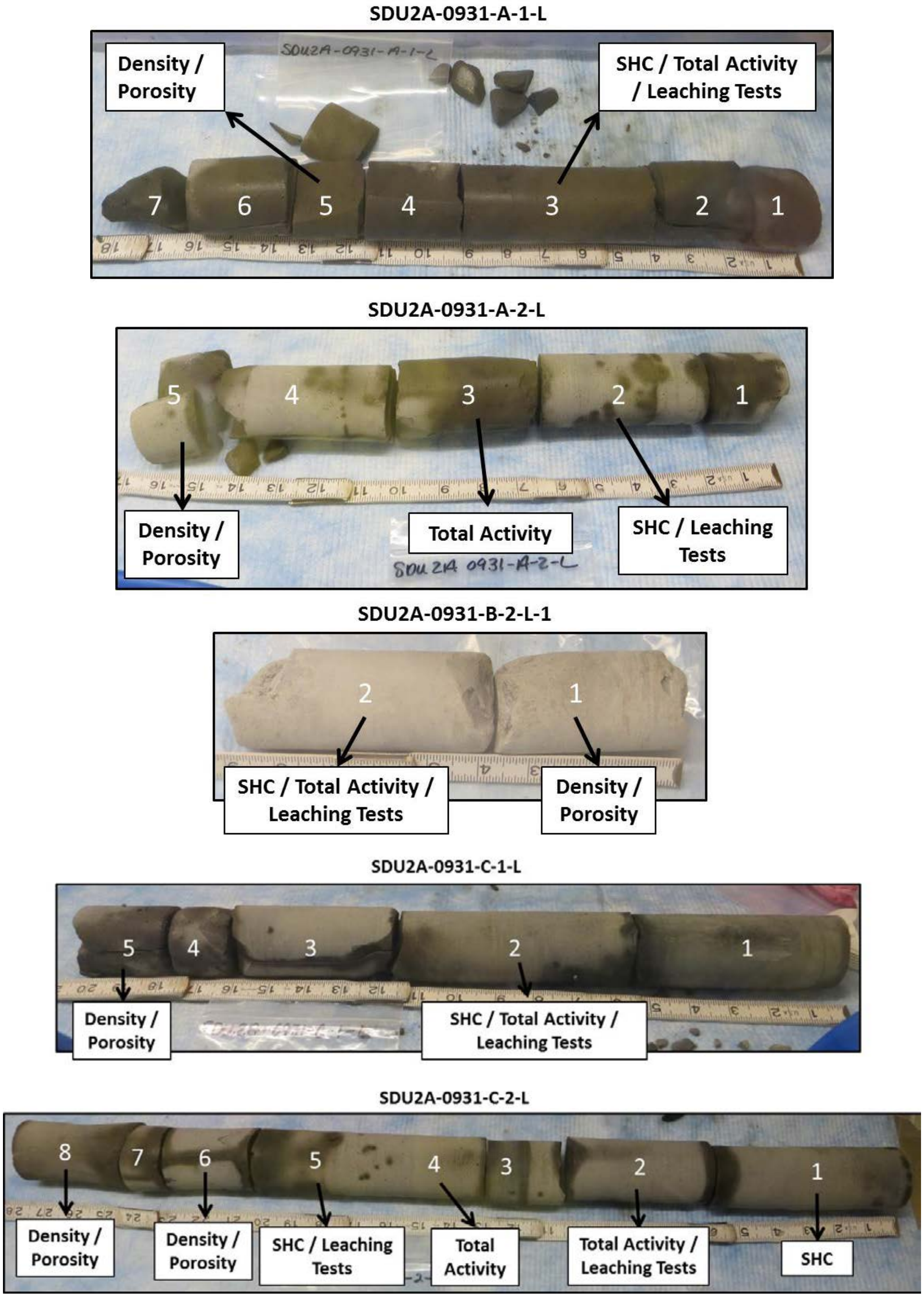

Fig. 1. SDU2A Core Samples Used for Analyses

Data presented in this section for SDU2A samples is derived from Reference 8. For some analysis, replicate measurements were conducted; specifically, density, porosity, moisture content, total radionuclide activity, and radionuclide solubility and K_d were conducted in triplicate. For the purpose of this report in many instances only the data ranges and means are provided rather than each individual data point. Where applicable the results for the SDU2A samples are compared to laboratory-prepared samples. This data is derived from Reference 6. Since the simulant saltstone samples were not spiked with the relevant radionuclides, radiochemical analysis was not conducted.

Physical Properties

Table I indicates SDU2A and laboratory-prepared samples with respect to bulk (saturated) density, total/permeable pore volume, and water content.

Table I. Physical Property Data for SDU2A and Laboratory-Prepared Samples

Sample Type	Bulk Density (g/cm^3)	Total Porosity (vol. %)	Open Porosity (vol. %)	Water Content (wt. %)
SDU2A **(18 discrete sub-samples)**	**1.71 – 1.76** $\bar{x}$ = 1.73	**63.6 – 68.8** $\bar{x}$ = 65.8	**41.9 – 46.6** $\bar{x}$ = 43.7	**29.0 – 32.1** $\bar{x}$ = 30.3
Lab-Prepared Samples **(9 discrete sub-samples)**	**1.75 – 1.77** $\bar{x}$ = 1.76	**58.3 – 60.9** $\bar{x}$ = 59.8	**40.1 – 42.1** $\bar{x}$ = 40.8	**29.9 – 30.1** $\bar{x}$ = 30.4

Discussion with respect to the comparative physical properties (Table I) of SDU2A and laboratory-prepared samples will emphasize the significance of certain differences in the processing, curing, and handling/storage of each sample type. The key differences between controlled laboratory activities and variable field operations with respect to saltstone processing are highlighted in Table II.

Table II. Processing, Curing, and Storage Disparities between SDU2A and Laboratory-Prepared Samples

Parameter	Laboratory-Prepared Samples	SDU2A Samples
Dry and Liquid Component Proportions	— Exact mass of each constituent measured in laboratory	— Variable bulk delivery in SPF — Avg. dry feed rate 33.0 ton/hr (± 1%) — Avg. salt soln. feed rate 87.8 GPM (± 2%)
Mixing	— Small scale laboratory batch mixer	— Large scale, continuous mixer
Additional Flush Water Added	— Not applicable to laboratory-prepared samples	— Approx. 50-60 gal. flush water added to clean SPF hopper every 45 min. during processing — Approx. 800-900 gal. flush water added at end of processing; a proportion expected to be incorporated into grout
Delivery	— Poured into cylindrical molds from mixing bowl	— Transferred through pipe from SPF to SDU2A utilizing peristaltic pump — Grout allowed to free-fall into SDU2A and subsequently spread towards extremities
Curing	— All samples cured according to same thermal profile in controlled humidity oven	— Temperature profile of each sample dependent on location in SDU — SDU2A humidity may vary based on ambient humidity
Storage	— Samples remained in controlled environment in capped molds until time of testing	— Samples core drilled, extracted from core holes, and stored in inerted transport containers — Upon receipt at SRNL, samples removed from transport containers (in an inerted, low humidity chamber) and cataloged/photographed prior to placement in sealable containers

With respect to bulk density, the mean density of 1.73 g/cm^3 for SDU2A samples was only slightly lower than the 1.76 g/cm^3 for the laboratory-prepared samples.

This would be expected since the SDU2A samples are assumed to incorporate at least a portion of the flush water added at the end of processing each day. The differences between total and permeable porosity for the two sample types also match the expected trend based on the assumption that the SDU2A samples incorporate flush water compared to no additional water (other than that contained in the accurately measured simulant salt solution) that was added to the laboratory-prepared samples. The water content data for the two sample types is perhaps the most surprising result with respect to the physical properties since both sample types indicate similar water content values (30.3 - 30.8 mass %) despite the measured differences in porosity. For the SDU2A samples it is possible that water evaporation (prior to testing) accounts for the lower than predicted water content measured for these samples. SDU2A samples sat uncovered in a 99.99% N_2 environment (with no source of humidity) while each sample was catalogued and photographed. It is also possible that additional air may have been incorporated into the fresh grout slurry during through-pipe transfer and free-fall into the SDU. Air voids would not be saturated with pore solution and this may account for the increased porosity without a similar increase in water content for the SDU-emplaced samples. However, it is also important to recognize that these same energetic processes (i.e., grout pumping and free-fall) can result in air being expelled from the fresh grout slurry.

Saturated Hydraulic Conductivity

The measured K_{sat} values of six SDU2A samples are provided in Table III**.** The physical appearance of each core utilized for measuring K_{sat} is provided in Figure 2. Four of the six SDU2A samples had K_{sat} values lower than the detection limit (1.0E-09 cm/sec) for the permeameter equipment used [8]. The main anomaly in Table III is the 4.4E-09 cm/sec K_{sat} value. Figure 2 illustrates the physical condition of the sample in question; in addition during sample preparation it was noted that the sample was difficult to prepare for K_{sat} measurement since it cracked or spalled when attempting to cut the sample to the desired 2-inch length with parallel end faces [8]. The higher K_{sat} for this sample is thus assumed related to physical degradation of the sample caused by core-drilling and subsequent sample preparation. It is noteworthy, however, that despite the compromised integrity of this particular SDU2A sample, the K_{sat} is still less than that the value currently assumed in the SDF PA. All laboratory-prepared samples exhibited K_{sat} values below the 1E-09 cm/sec detection limit of the equipment.

Table III. K_{sat} for SDU2A and Laboratory-Prepared Samples and SDF PA Assumptions

Sample ID	**K_{sat} (cm/sec)**	**Comments on Sample Integrity**
SDU2A-0931-A-1-L-3	1.2E-09	Side defects that could not be excluded when sample sectioned to 2” length
SDU2A-0931-A-2-L-2	<1.0E-9	No observable surface defects
SDU2A-0931-B-1-L-2	4.4E-09	Sample was difficult to section and fractured easily resulting in surface defects
SDU2A-0931-C-1-L-2	<1.0E-9	No observable surface defects
SDU2A-0931-C-2-L-1	<1.0E-9	No observable surface defects
SDU2A-0931-C-2-L-5	<1.0E-9	No observable surface defects
SDU2A Sample Mean	**<1.6E-09**	4 of 6 samples with no visible surface defects
Laboratory Sample Mean	**<1.0E-09**	Demolded samples without surface defects
SDF PA Assumption	**6.4E-09**	N/A

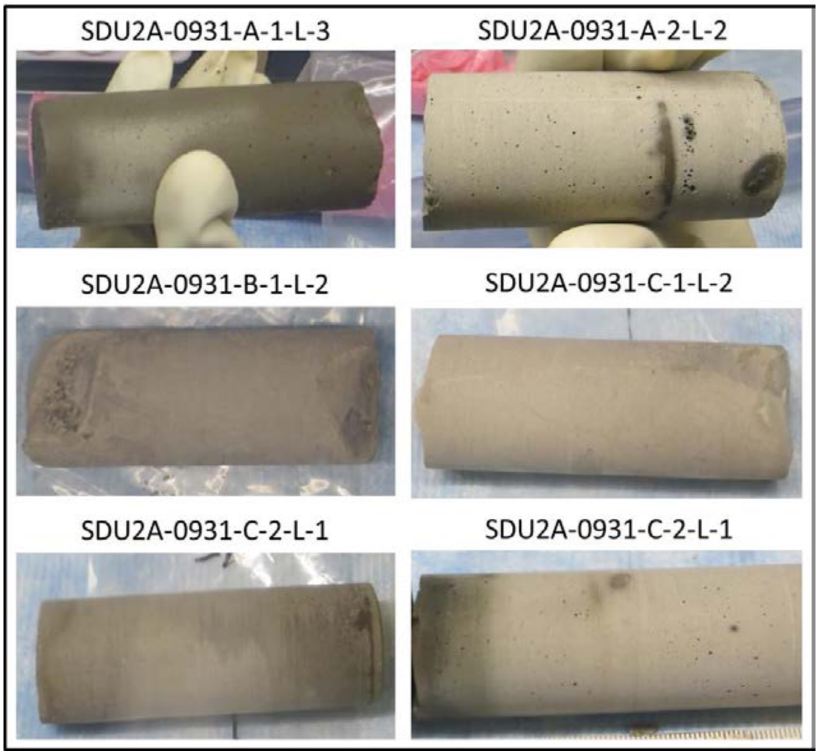

Fig. 2. SDU2A Samples used for K_{sat} Measurements

Radionuclide Solubility and Distribution Ratio (K_d)

Measurement of radionuclide solubility and liquid-solid partitioning provides key data for confirmation of input assumptions used in the SDF PA transport models. Radionuclides that are in the form of insoluble compounds, and/or have a high affinity to solid surface adsorption, exhibit lower transport rates from saltstone into the surrounding environment. The mobility of Tc-99 under anoxic/reducing conditions is modeled as a solubility-limited mechanism due to the redox-sensitivity of Tc-99 and formation of poorly soluble Tc(IV) phases; in contrast, for Tc-99 (in the form of highly soluble Tc(VII) phases) under oxic conditions, and Sr-90 and I-129 under both oxic and anoxic conditions, adsorption is assumed the predominant mechanism controlling radionuclide transport rates. The distribution coefficient (K_d in mL/g) is used to define the partitioning of a radionuclide between solid and liquid phases.

Measurement of leachate reduction potential (E_h) and pH are also required to faciltate an improved understanding of solubility and/or adsorption of the radionuclides; ranges and means of these properties measured under anoxic and oxic conditions are provided in Table IV, and will be referred to in the subsequent discussion.

Table IV. Leachate pH and E_h under Anoxic and Oxic Conditions, After 7 Days Equilibration with Ground SDU2A Samples

Anoxic		**Oxic**	
pH	**E_h (mV)**	**pH**	**E_h (mV)**
10.4 to 10.6 $\bar{x}$ = 10.5	-186 to -240 $\bar{x}$ = -217	10.6 to 10.8 $\bar{x}$ = 10.7	+144 to +178 $\bar{x}$ = +155

Tc-99 Solubility under Anoxic Conditions

As stated previously in anoxic (reducing) environments the leaching of Tc-99 is assumed to be controlled by the solubility limits of reduced Tc species. The Tc-99 concentrations measured for the anoxic tests are provided in Table V, together with the solubility assumed for SDF PA.

Table V. Tc-99 Solubility under Anoxic Conditions

Sample Type	**Tc-99 Conc. (mol/L)**
SDU2A Cores (6 samples)	7.0E-09 to 3.9E-08 $\bar{x}$ = 2.2E-08
SDF PA Assumption	1.0E-08

For a given pH-E_h of an aqueous system the pH-E_h diagram (also referred to as a Pourbaix or Predominance Diagram) predicts the most thermodynamically stable species. The pH-E_h diagram for the Tc-O-H system is provided in Figure 3. [14] Note that although the pH-E_h diagram can predict the most stable species for a particular environment it does not account for the transformation kinetics of a given Tc species as the chemical system transitions from one environment to another: for example reducing (negative E_h) to oxidizing (positive E_h).

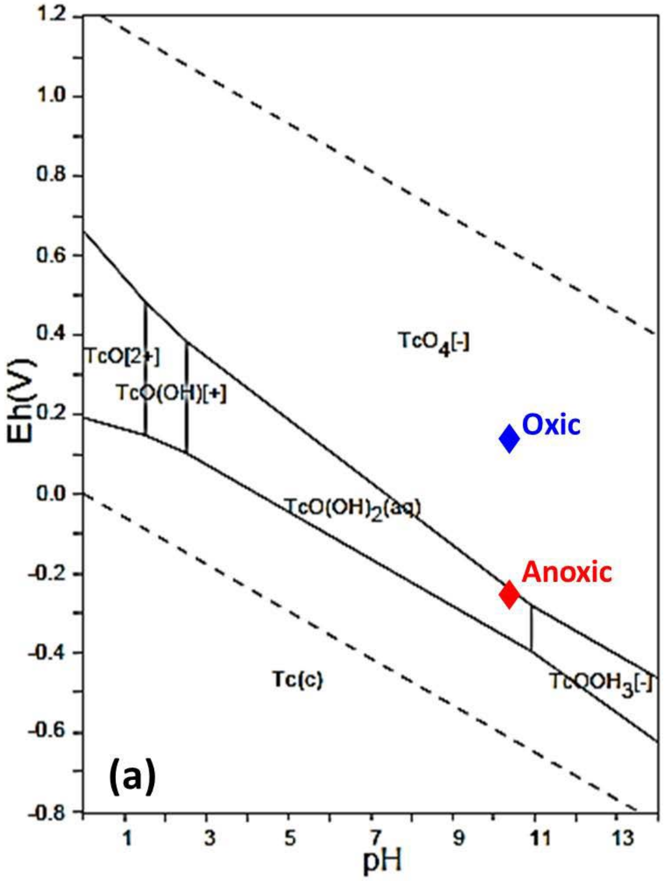

Fig. 3. pH-E_h Diagram for Tc-O-H [14]

For anoxic conditions, E_h -217 mV and pH 10.5 (anoxic – designated by a blue diamond in Figure 3) the predicted phase is a reduced hydrated Tc-oxide (often written as $TcO_2.xH_2O$). As expected under oxic conditions, E_h +155 mV and pH 10.7, the oxidized pertechnetate anion (TcO_4^-) is predicted. Figure 4 indicates the pH-dependent solubility of various Tc species (calculated using inputs from a Lawrence Livermore National Laboratory (LLNL) thermodynamic database – see note below Figure 4) relevant to this study. It indicates increasing solubility as the Tc species transition from sulfides to hydrated oxides to oxidized pertechnetate compounds. At the anoxic pH 10.5, $TcO_2.xH_2O$ phases predicted by the pH-E_h diagram have solubility limits on the order of E-08 mol/L. The mean Tc-99 concentration measured for the ground SDU2A cores after exposure to a leachate solution for 7 days was 2.2E-08 mol/L (Table V) which suggests that for anoxic conditions the Tc-99 concentration is indeed controlled by the solubility of hydrated Tc-oxides as modeled in the SDF transport simulations.

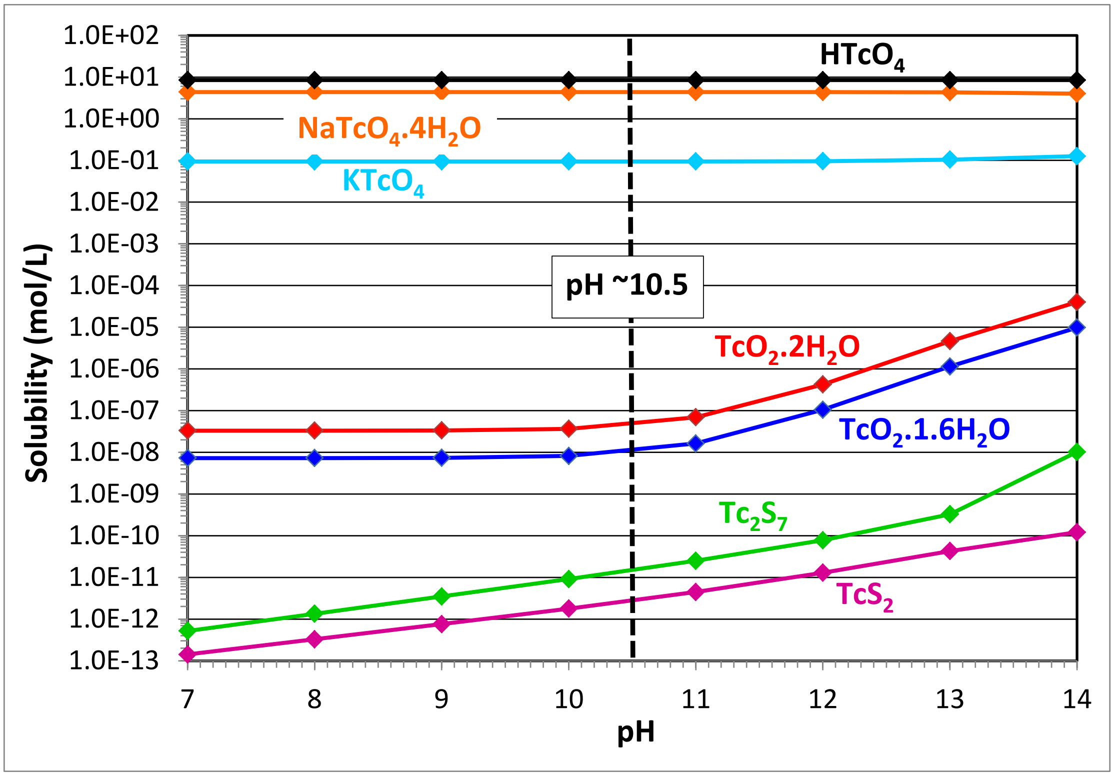

Fig. 4. pH-Dependent Solubility of Reduced Tc Species

Figure 4 was generated at the Savannah River Ecology Laboratory (SREL) using the geochemical computer code PHREEQC-2 (USGS-99-4259) and the Lawrence Livermore National Laboratory (LLNL) thermodynamic data base (thermo.com.V8.R6.230). This LLNL database was prepared by Jim Johnson (LLNL) in Geochemist's Workbench format, and subsequently converted to the Phreeqc format by Greg Anderson (LLNL) with the assistance of David Parkhurst (US Geological Survey [USGS]).

With respect to the SDF transport model, a Tc-99 solubility of 1.0E-08 mol/L (at a pH of 10.5) is assumed [9], which is just slightly less than that observed for the SDU2A cores at the same pH. In support of the SDF transport model, sensitivity models were developed to evaluate the impact of changing the Tc-99 solubility an order of magnitude above (1.0E-07 mol/L) and below (1.0E-09 mol/L) the assumed solubility of 1.0E-08 mol/L. As discussed in the FY2014 SDF Special Analysis (SA) [9], increasing the solubility limit for Tc by an order of magnitude increases the flux by an order of magnitude. Similarly, decreasing the solubility limit for Tc by an order of magnitude decreases the flux by an order of magnitude, except where releases are controlled by oxidation. Therefore, increasing the solubility limit by a factor of two (to more closely match the results from the SDU2A core analysis) would be expected to increase the Tc-99 flux release by a factor of two. Because Tc-99 is not the predominant dose driver until the bottom of the saltstone monolith becomes oxidized (more than 30,000 years after SDF closure), this change is unlikely to have a significant impact on the conclusions of the PA. [1,9]

Tc-99, Sr-90, I-129 Distribution Coefficients (K_d)

Table VI provides the K_d data for Tc-99 under oxic conditions, and Sr-90 and I-129 under both oxic and anoxic environments; K_d values currently used in the SDF transport models are also included. [9] It is important to note that the K_d data presented in Table VI was derived from a desorption measurement. Contaminant K_d data can be determined by (1) adding a solid phase to a liquid containing the contaminant and determining the proportion of contaminant partitioned from the liquid to the solid by adsorption, or (2) adding a solid containing the contaminant to a liquid and determining the proportion of contaminant partitioned from the solid to the liquid by desorption. The latter is referred to as reverse K_d and is representative of the data presented in Table VI.

Table VI. Tc-99, Sr-90, and I-129 Distribution Coefficients (K_d) for SDU2A Cores

Sample ID	**Reverse K_d (mL/g)**				
	Tc-99 (Oxic)	**Sr-90 (Anoxic)**	**Sr-90 (Oxic)**	**I-129 (Anoxic)**	**I-129 (Oxic)**
SDU2A Samples	25 to 32 $\bar{x}$ = 28	36 to 70 $\bar{x}$ = 55	79 to 176 $\bar{x}$ = 112	-5 to +4 $\bar{x}$ = 0.3	-5 to +2 $\bar{x}$ = -2
SDF PA Assumption	0.5	15	15	9	15

Tc-99 – as aforementioned the leaching behavior of Tc-99 is assumed to be solubility controlled in anoxic (or reducing) environments and earlier discussion confirms that hydrated Tc-oxide phases govern the solubility. In contrast under oxic conditions, Tc-99 is expected to transition from sparingly soluble Tc(IV) compounds into the highly soluble pertechnetate anion (TcO_4^-) and the leaching behavior is controlled by TcO_4^- adsorption to the saltstone surface. The K_d of approximately 30 mL/g for SDU2A samples is 60 times greater than that assumed in the SDF transport model. However, with prolonged exposure to the oxic environment (i.e., greater than the 7-days utilized for this study) and the potential for continued oxidation of reduced Tc-99-species, it is possible that the K_d may become more aligned with the assumed, and more conservative, value currently utilized in the SDF PA.

Sr-90 – strontium is typically considered a highly mobile contaminant: in the FY2014 SDF SA [9], the K_d value for strontium within saltstone was modeled as 15 mL/g until the saltstone was significantly aged (i.e., oxidized). The assumed SDF transport model value is significantly lower than the K_d for the SDU2A core samples in both oxic and anoxic environments, and therefore does not challenge the current transport model. In addition, sensitivity modeling of the Sr-90 K_d values in the FY2014 SDF SA [9] demonstrated that strontium K_ds are not risk-significant parameters. This is because Sr-90 has a relatively short half-life (less than 30 years) and flow through saltstone remains relatively low until after the Geosynthetic Clay Liner and High Density Polyethylene (GCL-HDPE) liner system of the closure cap becomes degraded; as such, most of the Sr-90 decays prior to any significant release and transport.

I-129 – like Sr-90, I-129 is typically considered a highly mobile contaminant: in the FY2014 SDF SA [9], the K_d value for I-129 within saltstone was modeled as 9 mL/g when the saltstone was reduced, then transitions to 15 mL/g when it oxidizes. In contrast, the K_d values in Table VI indicate that the I-129 K_d in both oxic and anoxic environments is less than 1 mL/g. As Table VI indicates many of the K_ds were calculated as negative values; this is obviously not feasible and implies that more I-129 is being released into the liquid than was originally present in the solid (the minimum possible K_d is zero which indicates that all of a contaminant has been partitioned from the solid to the liquid). The negative values are an artifact of analyzing a constituent that has a low concentration and is known to be poorly adsorbed. Sensitivity modeling of the I-129 K_d values in saltstone shows that releases of I-129 are most sensitive to changes to the K_d values while saltstone is reduced [9]. However, the sensitivity models show that even when assuming a K_d value of 0 mL/g in reducing saltstone (i.e., complete mobilization of I-129), the release of I-129 increases by less than a factor of two, such that the peak dose contribution from I-129 only increased from about 11 mrem/yr to about 17 mrem/yr within 10,000 years of facility closure. This is because with such low I-129 K_d values in saltstone, the release of I-129 is more a function of flow rates, rather than a function of the K_d. Further, since the initial K_{sat} is expected to be lower than currently modeled (see Table 3), the lower K_{sat} is expected to decrease flow rates through saltstone. Since I-129 is sensitive to flow rates, the actual change to the modeled peak dose is expected to be less significant.

SUMMARY

Properties relevant to the long-term performance of saltstone grout were evaluated for actual saltstone samples extracted from an SDU and compared to laboratory-prepared, simulant saltstone samples. The objectives of this endeavor were: (1) to support the use of laboratory-prepared samples to predict the behavior of field-emplaced samples, and (2) to substantiate the saltstone properties currently assumed in the SDF transport model. With respect to these objectives the data derived from this work has demonstrated the following:

1. While minor physical differences (i.e., density and porosity) may exist between field- and laboratory-processed samples, the measured differences were not impactful with respect to key contaminant transport properties, in particular saturated hydraulic conductivity (K_{sat}). As such, there can be greater confidence that studies of laboratory samples can provide sufficient insight into the inherent physical properties of field processed and emplaced saltstone.
2. The assumptions currently made in the SDF transport model with respect to K_{sat}, and radionuclide leaching behavior, have predominantly been affirmed. In particular, the data from this study indicates that: (1) the K_{sat} value of

6.4E-09 cm/sec used in the SDF transport model is conservative, and (2) the use of solubility limited behavior for Tc-99 in a reduced, high pH environment is correct. With respect to the adsorption (or rather desorption) characteristics of I-129 whilst the reverse K_d was less than 1 mL/g sensitivity analysis indicated that even a zero K_d (0 mL/g) would not significantly impact the magnitude or timing of the peak dose in the surrounding environment.

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