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Reduction Capacity of Saltstone and Saltstone Components

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EXECUTIVE SUMMARY

The duration that saltstone retains its ability to immobilize some key radionuclides, such as technetium (Tc), plutonium (Pu), and neptunium (Np), depends on its capacity to maintain a low redox status (or low oxidation state). The reduction capacity is a measure of the mass of reductants present in the saltstone; the reductants are the active ingredients that immobilize Tc, Pu, and Np. Once reductants are exhausted, the saltstone loses its ability to immobilize these radionuclides. The reduction capacity values reported here are based on the Ce(IV)/Fe(II) system. The Portland cement (198 μeq/g) and especially the fly ash (299 μeq/g) had a measurable amount of reduction capacity, but the blast furnace slag (820 μeq/g) not surprisingly accounted for most of the reduction capacity. The blast furnace slag contains ferrous iron and sulfides which are strong reducing and precipitating species for a large number of solids. Three saltstone samples containing 45% slag or one sample containing 90% slag had essentially the same reduction capacity as pure slag. There appears to be some critical concentration between 10% and 45% slag in the Saltstone formulation that is needed to create the maximum reduction capacity. Values from this work supported those previously reported, namely that the reduction capacity of SRS saltstone is about 820 μeq/g; this value is recommended for estimating the longevity that the Saltstone Disposal Facility will retain its ability to immobilize radionuclides.

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LIST OF ABBREVIATIONS

BFS	Blast furnace slag
DDA	Deliquification, Dissolution, and Adjustment
FA	Fly ash
FAS	Ferrous Ammonium Sulfate
µeq	microequivalents
meq	milliequivalent
PA	Performance Assessment
PC	Portland Cement
R and D	Research and Development
SRNL	Savannah River National Laboratory
SRS	Savannah River Site

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1.0 Introduction

Savannah River Site (SRS) uses saltstone as a low level waste form in which low level liquid waste is mixed with cement, fly ash, and blast furnace slag resulting in a solid cementitious waste form. Studies on different mixtures of these components resulting in saltstone simulants are currently ongoing. The addition of blast furnace slag to these formulations is to create a reducing environment to immobilize redox sensitive radionuclides, such as Tc (technetium), Pu (plutonium), and Np (neptunium). These different formulations or simulants are being studied for their physical properties to determine efficacy in containing low level waste. The **objective** of this study was to measure the reduction capacity of different formulations. Additionally, reduction capacity measurements were made of saltstone components: cement, flyash and pure slag.

Angus and Glasser (1985) define the determination of reduction capacity as largely operational. They adopted the use of redox pairs Ce(IV)/Ce(III) and Fe(II)/Fe(III) to quantify reduction capacity (or poising capacity as they refer to it) for cement systems. An adaptation of this methodology is used here and thus, the reduction capacity, reported in units of microequivalents per gram of solid, refers to the ability of a simulant to reduce a given mass of Ce(IV) per gram of solid (the solid in this case is saltstone). Reduction capacity is one of the most important parameters for understanding risk associated with saltstone because it provides a measure for how long the Saltstone Disposal Facility will immobilize, by creating a reducing environment, some of the key risk drivers for the performance assessment (PA). The slag includes both a chemical reductant (iron(II)) and precipitating agent (sulfide) that promotes reducing conditions, lending to redox sensitive contaminants to be effectively retained in this waste form.

Previous work on the reduction capacity of saltstone simulants[1] (Kaplan et al., 2008), reported the unexpected finding that DDA simulant saltstone[2] which contains 23% blast furnace slag, had the same reduction capacity as 100% blast furnace slag. Several possible mechanisms were proposed including: 1) a higher pH in saltstone (pH 11.8) than blast furnace slag (pH 8.1), resulting in the dissolution of reducing phases and increasing the saltstone reduction capacity; 2) semi-conductor behavior of saltstone; 3) increased reduction capacity of the saltstone simulant due to fly ash contributing other reducing mineral phases; and 4) saltstone may have a higher porosity than pure blast furnace slag as a result of dissolution, creating a greater surface area. Because of the many possible causes of the unexpected finding of a saltstone simulant having the same reduction capacity as blast furnace slag alone, this study evaluated the reduction capacity of four saltstone simulant formulations (TR437, TR451, TR545, TR547), a concrete sample and the three main components of saltstone (Portland cement, fly ash, and blast furnace slag). The compositions of these samples are listed in Table 1.

[1] Simulant refers to Saltstone without radionuclides included.
[2] DDA (Deliquification, Dissolution, and Adjustment) refers to the type of salt solution simulant. The saltstone formulation dry feeds ratio was 45% fly ash, 45% slag, and 10% cement, typical of most Saltstone mixtures and had a 0.6 water:premix ratio.

Table 1. Solid Sample Composition

Sample Number	Blast Furnace Slag	Fly Ash	Portland Cement	W/CM	Simulant	Aluminate	Temp	Admix-tures	Organics	Cast Date	Comment
	(wt-%)	(wt-%)	(wt-%)			(molarity)	°C				
TR437*	45	45	10	0.6	MCU	0.054	22	No	No	3/31/08	
TR451	45	45	10	0.6	SWPF	0.054	22	No	No	4/22/08	
TR547*	45	45	10	0.6	MCU	0.054	22	No	No	2/4/09	#1
TR431	45	45	10	0.6	DDA	0.054	22	No	No	3/18/08	#2
TR545*	90	0	10	0.6	MCU	0.054	22	No	No	1/16/09	#3
Portland Cement	0	0	100								
Class F Flyash	0	100	0								
Blast Furnace Slag	100	0	0								
Vault 2 Concrete	10	6	0								
Aged (30 yr) concrete	0	45	10								

#1 Mix #2 from SRNL-L3100-2009-00019
#2 Results Reported in SRNS-STI-2008-00045
#3 Mix #1 from SRNL-L3100-2009-00019
* MCU Simulants identical except TR437 Nitrate = 3.32M and TR547/TR545 Nitrate = 3.16.

2.0 Experimental Procedure

The method used to determine the reduction capacity of the solids was adapted from Angus and Glasser (1985). As mentioned earlier, these authors define the measurement of reduction capacity as an operational one, and for quantifying reduction capacity in cement and cement blends have used the end point of the reaction $Fe^{2+} + Ce^{4+} \rightarrow Fe^{3+} + Ce^{3+}$. For this reason, such procedural steps as particle size of the sample, and contact time of the cerium with the sample may become important. Conversations with Dr. Lukens (Lawrence Berkeley National Laboratory, Berkeley, CA) who had conducted preliminary experiments evaluating contact time and chemical formulation, was helpful in refining the procedure here (details presented in Appendix A). Briefly, this method is a colorimetric titration in which a Ce(IV) solution of known concentration is made and calibrated using a reducing iron solution (FAS or ferrous ammonium sulfate). The Ce(IV) solution is added to each solid and titrated against the FAS. The difference between the reduction of Ce(IV) without and with the solid is then used to calculate the reduction capacity of each solid.

3.0 Results and Discussion

The results of the Ce(IV)-Fe(II) colorimetric titrations of 0.5 g of solid are listed in Table 2. The blast furnace slag can be used to compare with the work of others since there is no real reduction capacity standard commercially available. Our reduction capacity value of blast furnace slag of 819 μeq/g (Table 2) agrees well with previously reported values:

- 819 μeq/g (this study)
- 832 μeq/g (Kaplan et al., 2008),
- 817 μeq/g (Kaplan et al., 2005), and
- 820 μeq/g (Lukens et al., 2005).

Kaplan et al. (2008 and 2005) were conducted at the SRNL, but Lukens et al. (2005) was conducted with the same material at Lawrence Berkeley National Laboratory. The ingredients and recipes for these three samples were the same, but the samples were prepared at different times by different people. It should be noted that the slag is not porous and as such, it is possible that a greater reduction capacity may exist, if there is some "internal reduction capacity sites" not accessed by the Ce(IV) assay. The role of "internal reduction capacity sites" becomes less important if the system is found to have semiconductor properties, as is being commonly found in iron bearing systems (Cornell and Schwertmann 2003).

The Vault 2 concrete sample has also been measured previously. Unlike the blast furnace slag samples, the Vault 2 concrete sample reduction capacity values reported here, 178 μeq/g, (Table 2) differed substantially, 25%, from the previously reported value of 239 μeq/g (Kaplan et al. 2008). Given that the blast furnace slag values seem to be very reproducible, it seems reasonable to assume that the variability of the concrete sample may be attributable to sample variability/heterogeneity, and not analytical variability. Another possible explanation for the variability is that of adaptation of the original method. The Angus and Glasser (1985) method calls for determination of the endpoint of the reaction by measuring the potential until 1.057 V is reached. In this adaptation, the determination was made colorimetrically. The latter may allow for more subjective interpretation and thus more variable results. A last possibility is that of sample preparation. The solid samples were pulverized and passed through the same mesh size

and each sample was assumed to be the same size but surface area of each sample was not measured directly.

Unlike the slag, the measurement of the reduction capacity of cementitious materials involves diffusion of the Ce(IV) solution into porous media (the slag is non-porous). For this reason we use a strong acid to facilitate the dissolution of carbonates and grind the samples into fine particles in the analysis.

Based on the chemical composition of the components in saltstone simulants, it is not surprising the Portland cement (a calcium silicate mineral phase along with other phases), had the lowest reduction capacity (198 µeq/g), followed by fly ash (299 µeq/g) and the highest component was the blast furnace slag (819 µeq/g), which contains both Fe^{2+} and various forms of sulfides. These results indicate that Portland cement and fly ash do have chemical reducing agents but the slag is still the main contributing source to the reduction capacity.

Table 2. Reduction capacity (µeq/g) of solids using the Ce(IV)- Fe(II) colorimetric titration method with 0.5 g of solid sample.

Sample	% Blast Furnace Slag	This Study	Kaplan et al., 2008
Saltstone TR437	45 %	849	
Saltstone TR451	45 %	793	
Saltstone TR547	45 %	607	
Saltstone TR431	45 %		821 ± 5
Saltstone TR545	90 %	681	
Portland Cement	0 %	198	
Class F Fly Ash	0 %	299	
Blast Furnace Slag	100 %	819	832.4 ± 5
Vault 2 Concrete	10 %	178	239 ± 31
Aged (30 yr.) Cement	0 %		86 ± 10

Three (TR437, TR451, TR547) of the four saltstone simulants studied had compositions of 45% blast furnace slag, 45% fly ash, and 10% Portland cement, and the fourth simulant (TR545) had 90% blast furnace slag and 10% cement. Based on this composition, it would be expected that the saltstone simulant with the largest blast furnace slag composition (90%, TR545) would have the highest reduction capacity. However, the results in Table 2 do not support this expectation. The three saltstone samples with 45% slag had 849, 793, and 607 µeq/g, whereas the pure slag had 819 µeq/g. Clearly the reduction capacity is not proportional to the amount of slag in the sample. This supports the finding by Kaplan et al. (2008).

All the saltstone reduction capacity data is presented in Figure 1, showing its relation to slag concentration in saltstone. The plot shows some low points, then at 45% slag it plateaus. What is not known is how the points connect between 10 % and 45% and whether it is a gradual or steep curve.

The use of the Ce(IV)-Fe(II) system is useful for comparison with previous work as discussed previously. Because the reduction capacity values are relative to the system being used to

quantify it, the Ce(IV)-Fe(II) is not the only system used in the literature. Kaplan (2003) used the method developed by Lee and Batchelor (2003) based on the Cr(III/VI) couple to assess the reductive capacity of SRS soils and slag. This method, unlike the Ce(IV) method of Angus and Glasser (1985) is designed to probe only the surface reductant. Kaplan (2003; Fig 2) measured a reduction capacity for the SRS slag of 32 µeq/g using the Cr(III/IV) assay, as compared to 820µeq/g (page 3) using the Ce(III/IV) assay. Again, this difference reflects that the Cr system measures only the surface reduction capacity, whereas the Ce system measures all or almost all of the reduction capacity. Serne (2006) also reported that for blast furnace slag the results of these methods are significantly different, with the Angus and Glasser (1985) method yielding results 20 times higher than the Lee and Batchelor (2003) method.

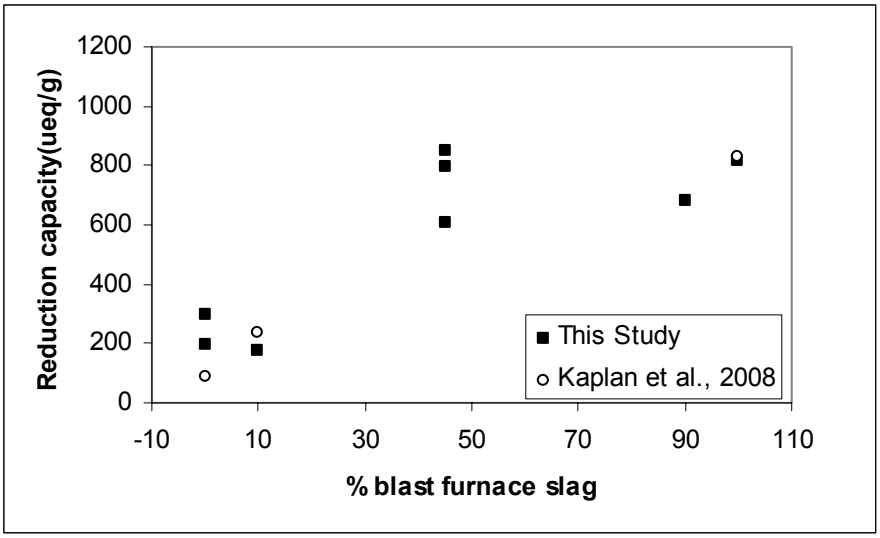

Figure 1. Reduction capacity (µeq/g) versus amount of blast furnace slag (%) in solid.

One question raised while following the Angus and Glasser method was how they came to the solid to liquid ratio used in their study. To address this, this study also investigated the reduction capacity as a function of solid concentration. This was tested as a part of methods development and part of our quality control. It confirmed that our sample size of 0.5 g was adequate to provide a consistent result for all types of samples. Using sample weights <0.1 g, results varied greatly due to different chemical reactions; at samples weights greater ≥0.1 g, the results were more consistent (Appendix C). One experimental condition not addressed in this study is the role of kinetics to reduction capacity measurements. These experiments were conducted on approximately an hour of mixing the Ce solution with the solid sample and it is undetermined as to whether that is sufficient time to allow the Ce to diffuse completely (i.e. in the pore solution to be reduced and back out in time to be measured).

4.0 Conclusions

The reduction capacity measurements made here were based on the Ce(IV)-Fe(II) system as described by Angus and Glasser (1985). The reduction capacities of four saltstone simulants were measured to be similar to that of pure blast furnace slag. The reduction capacity was also measured of the three major solid components of saltstone, blast furnace slag, fly ash, and

Portland cement. The Portland cement (198 μeq/g) and especially the fly ash (299 μeq/g) had a measurable amount of reduction capacity, but the blast furnace slag (819 μeq/g) accounted for most of the reduction capacity. The blast furnace slag contains ferrous iron and sulfides which are strong reducing and precipitating species for a large number of solids. Values from this work supported those previously reported, namely that the reduction capacity of SRS saltstone is about 820 μeq/g; this value is recommended for estimating the longevity that the Saltstone Disposal Facility will retain its ability to immobilize radionuclides.

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Appendix A: R&D Directions for Reduction Capacity of a Solid

R&D Directions:
Reduction Capacity of Solid

Kim Roberts & Dan Kaplan
6/1/09

HAP: SRNL-ECP-2007-00009

Hazards: Acids

Hazards Mitigations: Acids: Wear appropriate PPE (gloves, safety glasses)

Materials:
Ferrous ammonium sulfate (FAS)
Ceric ammonium sulfate
Phenanthroline (ferroin)
Sulfuric acid
Deionized water
10mL volumetric flasks
250 mL volumetric flasks
250mL Erlenmeyer flasks
25mL titrating burette
25mL volumetric pipette
pipette bulb
Saltstone simulants and components

Methods:

Reduction Capacity of Solid

(Angus and Glasser, 1985, Mat. Res. Soc. Symp. Proc. 50:547–556.)

1. Preparation of Solutions and Samples:

1.1 Preparation of 0.0500 M $(NH_4)_2Fe(SO_4)\bullet 6H_2O$ (FAS) (primary standard) in 0.71 M H_2SO_4

- 4.902 g of FAS weighed into a 250 mL volumetric flask
- 100 mL H_2O and 10 mL H_2SO_4 (17.8 M) added in quick succession
- Filled with H_2O
- Allowed to cool to RT (~2 hours)
- Added H_2O to make 250.0 mL

1.2 Preparation of 0.0608 M Ce(IV) solution

- 9.615 g of $(NH_4)_4Ce(SO_4)_4\bullet 2H_2O$ weighed into a 250 mL volumetric flask
- Added 150 mL H_2O and 25 mL H_2SO_4 in quick succession
- Filled flask to 250 mL and added a stirbar
- Magnetically stirred for 2 hours ($(NH_4)_4Ce(SO_4)_4\bullet 2H_2O$ dissolves slowly)
- Stirbar removed and water added to make 250 mL
- Titrated with FAS to determine the actual Ce(IV) concentration (0.059 M, in this case)

1.3 Preparation of 0.025M Fe(o-phenanthroline)$_3^{2+}$ (aka Ferroin)

- 0.069 g of $FeSO_4 \cdot 7H_2O$ and 0.131 of 1,10-phenanthroline weighed into a 10 mL volumetric flask
- H_2O added to make 10.0 mL
- Dark red solution

1.4 Preparation of Solid Samples

- ~0.5 g of sample was added to a 250 mL Erlenmeyer flask equipped with a stirbar
- 25.0 mL of ~0.05 M Ce(IV) solution was added by volumetric pipette
- The contents of the flask were stirred magnetically for 1 hour at room temp.
- Added 0.100 mL 0.025M Fe(*o*-phenanthroline)$_3^{2}$ (solution acquires a greenish tint)

<u>2. Measurements:</u>

2.1 Calibration of Ce(IV) Concentration

- 25.0 mL Ce (IV) solution added to a 250 mL Erlenmeyer flask
- Added 0.100 mL Ferrion indicator (solution acquires a greenish tint)
- Titrated with 0.050 M $(NH_4)_2Fe(SO_4) \cdot 6H_2O$ until solution remained lilac colored.

2.2 Ce(IV) Titration of Samples

- Titrated with 0.050 M $(NH_4)_2Fe(SO_4) \cdot 6H_2O$ until solution remained lilac colored
- During titration, solution goes from green to pale blue to lilac to pink

<u>3 Calculations:</u>

3.1 Ce(IV) Calibration

- Ce(IV) concentration is determined from the volume of FAS added to reach endpoint

Example: 25.0 ml Ce(IV) solution was titrated with 0.03021 L of 0.050 M FAS

(0.03021 L) x (0.05 M FAS) x 1mol Fe(II)/1mol Ce(IV) = 1.5×10^{-3} mol Fe(II)

1.5×10^{-3} mol Fe(II) x 1mol Ce(IV)/1mol Fe(II) / 0.025 L = 0.060 M Ce(IV)

3.2 Determining the Total Number of Oxidizing Equivalents in Ce(IV) Solutions

- The total number of oxidizing equivalents in the Ce(IV) solution is determined from the Ce(IV) concentration established in 3.1

Example: 0.025 L of Ce(IV) solution titrated

0.025 L Ce(IV) x 0.060 M Ce(IV) = 1.5105×10^{-3} mole e^- Ce(IV)

3.3 The Number of Reducing Equivalents of Fe(II) Needed to Neutralize Ce(IV) after it Reacts with Sample

Example: Unknown Ce(IV) solution was titrated with 0.02202 L of FAS

0.02202 L FAS x 0.050 M Fe(II) x (1 mol Fe(II) / 1 mol FAS) = 1.101×10^{-3} Mole e^- Fe(II)

3.4 Determining the Reducing Equivalents in the Solid Sample

- The reducing equivalents in the solid sample are determined from the difference between the total oxidizing equivalents in the Ce(IV) solution (3.2) and the number of reducing equivalents needed to neutralize the Ce(IV) solution after it reacted with the sample (3.3)

Example:

$1.310 \times 10^{-3} - 1.101 \times 10^{-3} = 4.095 \times 10^{-4}$ mole e^-

3.5 Reducing Equivalents per Gram

- The reducing equivalents per gram are calculated by dividing the reducing equivalents in the solid sample (3.4) by the mass of the sample.

*Exampl*e: The reducing equivalents in a 0.5015 g sample were determined to be 4.095×10^{-4} mole e^-

4.095×10^{-4} / 0.5015 g sample = 8.16×10^{-4} mole e^-/g or 0.816 meq/g or 816 μeq/g

Appendix B:

SRNL Correspondence Document Regarding Saltstone Property Testing

SRNL L3100-2009-00019, Rev. 0

December 16, 2008

Keywords: Performance
Permeability
Modulus

L. B. Romanowski
Waste Determinations

From: K. L. Dixon, M. A. Phifer and J. R. Harbour

FY09 PA/CA Maintenance Program: Additional Saltstone Property Testing

BACKGROUND

Additional tests have been identified for measurement of important hydraulic and physical properties of Saltstone. The initial phase of this work [1] was completed last year and the results were detailed in an internal report [2]. The proposed testing for FY09 includes measurement of saturated hydraulic conductivity, porosity, bulk density, particle density, water retention and Young's modulus of simulated Saltstone grouts. For completeness, the bleed volumes and gel times for each mix will also be measured.

The testing will be based on a projected salt solution composition for the ARP/MCU stream that will be fed to the Saltstone Production Facility over the next few years. The scope for FY09 will include testing to determine the impact of (1) admixtures, (2) organics, (3) w/cm ratio, (4) aluminate concentration, and
(5) temperature of curing on the hydraulic properties of Saltstone mixes Samples of selected batches prepared as part of this task will be provided to Dan Kaplan for measurement of K_d through leaching tests. The eleven mixes that will be batched and tested are detailed in Table 1.

Table 1. The Eleven Mixes That will be Batched and Tested

Mix #	Simulant Type	Descriptor	w/cm ratio	Aluminate molarity	BFS* wt %	FA* wt %	PC* wt %
1	ARP/MCU	Control - BFS/PC	0.60	0.054	90	0	10
2	ARP/MCU	Baseline	0.60	0.054	45	45	10
3	ARP/MCU	Baseline with Admixtures	0.60	0.054	45	45	10
4	ARP/MCU	Baseline with Organics	0.60	0.054	45	45	10
5	ARP/MCU	Baseline Combo -Organics and Admixtures	0.60	0.054	45	45	10
6	ARP/MCU	w/cm ratio impact	0.55	0.054	45	45	10
7	ARP/MCU	w/cm ratio impact	0.65	0.054	45	45	10
8	ARP/MCU	Impact of Aluminate	0.55	0.280	45	45	10
9	ARP/MCU	Impact of Aluminate	0.65	0.280	45	45	10
10	ARP/MCU	Baseline Combo and Aluminate	0.60	0.280	45	45	10
11	ARP/MCU	Baseline Combo at 60 oC Cure Temp.	0.60	0.054	45	45	10

*** BFS is Blast Furnace Slag, FA is Fly Ash and PC is Portland Cement**

TEST DETAILS

Test 1 Control (Mix 1)

A control mix will be based on the baseline mix modified by exclusion of the Class F fly ash. Consequently, the cementitious materials premix will be a mixture of 90 % blast furnace slag and 10 % portland cement. The degree of reaction will be much greater than with the normal premix and therefore should result in a lower porosity and a lower permeability. This bounding test at 0.60 w/cm ratio is expected to yield a hydraulic conductivity at or below the detection limit for the Mactec permeameter measurement system. Therefore, this test should demonstrate the lowest level of detection of the Mactec system as well as show a resolvable difference between measurements of the control mix and the control mix with the normal premix composition.

Test 2 –Impact of Admixtures (Mixes 2 and 3)

Recent Saltstone batches have required both a set retarder (Daratard 17) and an antifoam agent (Q2) for processing of the Saltstone. Therefore, the baseline mix will be prepared with and without nominal levels of these two admixtures to determine whether these admixtures appreciably affect the hydraulic and physical properties of Saltstone at these nominal concentrations.

Test 3–Impact of Organics (Mixes 2 and 4)

The solvent extraction process is expected to result in some carryover of organics [3].

Consequently, a test will be performed on the impact of Caustic Side Solvent Extraction (CSSX) organics at 100 microliters per 1600 gram batch. The CSSX solvent consists of 0.75 M 1-(2,2,3,3-tetrafluoropropoxy)-3-(4-sec-butylphenoxy)-2-propanol (Cs-7SB) and 0.003 M tri-n-octylamine (TOA) in an Isopar® L diluent.

Test 4–Impact of Combination of Admixtures and Organics (Mixes 2, 5, 10 and 11)

This test will determine the impact of a combination of admixtures (Test 2) and organics (Test 3) together in the mix vs. the baseline case without admixtures and organics.

Test 5– Impact of w/cm Ratio (Mixes 2, 6 and 7)

It is well known that decreasing the w/cm ratio in a mix will improve permeability in normal portland cement water mixes. This test will measure the variation in permeability for the case of the MCU salt solution at three different w/cm ratios. The initial selection of w/cm ratios is 0.55, 0.60 and 0.65. However, if the mix at an as-batched 0.65 w/cm ratio has significant bleed water and the resulting actual w/cm ratio is close to 0.60, then the three ratios will be adjusted to provide a more evenly spaced set of values. However, the baseline mix at 0.60 will be included as one of the three mixes.

Test 6 – Impact of Aluminate Concentration (Mixes 8, 9 and 10)

The DWPF has modified its process flowsheet to include a caustic washing of HLW sludge to remove some of the aluminum from the HLW prior to vitrification. The resulting aluminate stream will then be blended with tank 50 material and fed to the SPF. This increased aluminate concentration in the salt solution has significant impact on heat of hydration and set times and consequently, it is likely that it will also impact permeability. Therefore a set of three samples will be made at w/cm ratios of 0.55, 0.60 and 0.65 (as in Test 3) with a higher level of aluminate (0.28 M) for testing.

Test 7 – Impact of Increased Curing Temperature (Mix 11)

In an ongoing task, there is evidence that Young's modulus (a performance indicator) [4] is reduced by increasing the curing temperature of the mix. Since the vault temperature increases during curing as a result of the exothermic hydration reactions, one of the baseline mixes with a combination of admixtures and organics will be cured at 60 $^{\circ}$C rather than the normal 22 $^{\circ}$C to determine the impact of curing temperature on the permeability.

SCHEDULE

The schedule for the task of batching and testing of the samples is provided in Table 2. This schedule is based on the fact that the cementitious materials will be available for the testing as needed.

Table 2 Additional Saltstone Hydraulic and Physical Property Tests

Item	Schedule
Start Work	12/1/08
Test Plan Complete	1/5/09
Preparation of 1st set of Samples Complete	1/19/09
90-Day Cure Period for 1st set of Samples Complete	4/20/09

1st set of Samples to Subcontract Laboratory	4/27/09
Testing of 11th set of Samples by Subcontract Laboratory Complete	10/26/09
Draft Report Issued	12/18/09

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[2] *Hydraulic and Physical Properties of Saltstone Grouts and Vault Concretes,* K. L. Dixon, J. R. Harbour and M. A. Phifer SRNL-STI-2008-00421, Rev. 0, 2008

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1/21/09

H. H. Burns, SRNL, Environmental Management and Performance Modeling — Date

1/20/09

L. B. Romanowski, Waste Determinations — Date

Appendix C: Methods Development for Sample Size Needed for the Ce(IV) Reduction Capacity Protocol

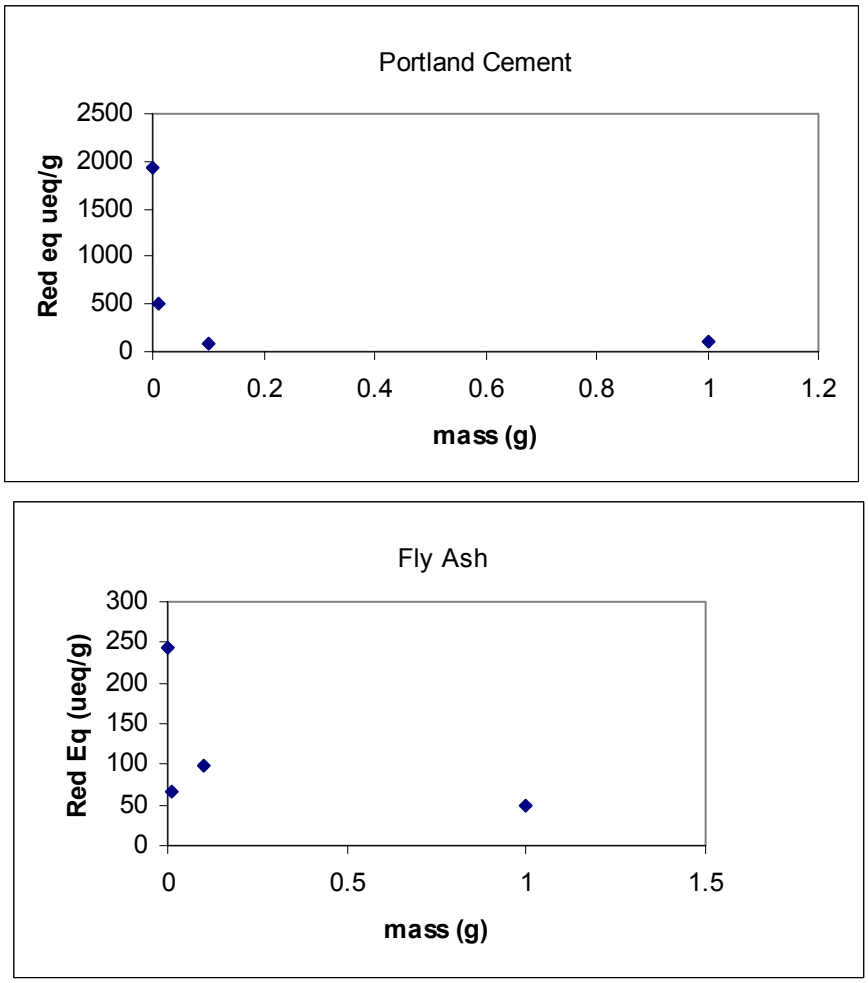
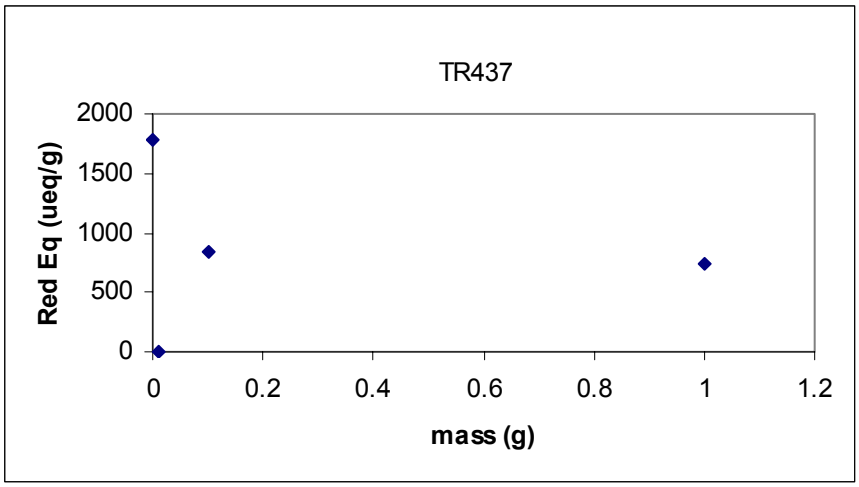


Figure 2. (top) Reduction capacity of Portland cement as a function of solid concentration. (bottom) Reduction capacity of fly ash as a function of solid concentration



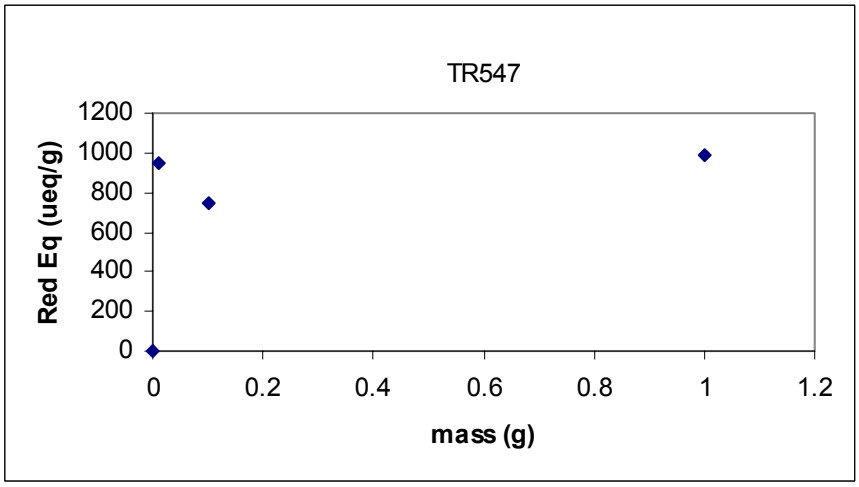


Figure 3. Reduction capacity of saltstone simulant as a function of solids concentration (top) TR 437 (bottom) TR547.

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