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Uranium Solubility and Supernate Enrichment Testing for Accelerated Basin De-Inventory Discards to Concentration, Storage, and Transfer Facilities

C. J. Martino

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EXECUTIVE SUMMARY

After future Accelerated Basin De-inventory (ABD) enriched uranium discards into sludge batches (SB), portions of supernate decants during SB preparation will be blended into salt batches (StB) and the resulting feeds must meet the nuclear safety requirements for the Salt Waste Processing Facility (SWPF).

During previous SB10 sampling and testing, which involved H-Canyon material containing enriched uranium being mixed with Tank 51 sludge shortly after the H-Canyon stream was neutralized, it was identified that the uranium isotopic enrichment in the supernate deviated from the uranium isotopic enrichment in the slurry. The higher uranium isotopic enrichment in the supernate introduced the risk of challenging the feed requirements of SWPF. The ABD material added to SB11 was isotopically diluted with depleted uranium, mitigating any downstream impacts. However, H-Canyon desires to eliminate or minimize future depleted uranium additions in order to meet the mission schedule.

Testing showed that the uranium concentration in the supernate of the recently precipitated ABD material being added to the SB is approximately 40 to 50 mg/L, which is higher than the typical uranium concentration in the supernate of the SB preparation tank. Thus, the enrichment of the recently precipitated stream may contribute to the enrichment in the supernate phase diverging from the enrichment of the slurry. Evidence from non-prototypic depleted uranium (DU) addition testing suggests that relatively concentrated DU solutions (approximately 200 to 400 g/L uranium), when neutralized separately, may not lower the uranium enrichment of the supernate phase as efficiently as they lower the uranium enrichment of the slurry.

Testing confirmed the previous laboratory observations of supernate enrichment deviation from slurry enrichment, but now under a more controlled and parametric set of test conditions. In all cases, the supernate resulting from mixing the pH adjusted ABD material and the existing sludge material had a ^{235}U isotopic enrichment that deviated above the slurry mixture ^{235}U isotopic enrichment of 4.0 wt%. The initial supernate decant from a SB had a ^{235}U isotopic enrichment of typically 45 wt% to 47 wt% with a maximum of 60 wt% and a minimum of 38 wt%. After decanting the initial supernate and contacting the material with wash water for 20 days, the supernate ^{235}U isotopic enrichment ranged from 22 wt% to 27 wt%. The supernate ^{235}U isotopic enrichment appears to decrease (in the direction converging toward the slurry enrichment) at cases with longer hold times, regardless of whether the hold time is during the neutralization step, the initial sludge mix step, or the subsequent inhibited-water wash step.

For the same sludge and salt feeds used in this testing, it is estimated that depleted uranium additions targeting 24 wt% and 9.7 wt% enrichment in the pre-neutralized H-Canyon stream should result in StB blends at or below the 8 wt% and 4 wt% enrichment limits, respectively, in the minimum assumed time frame.

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LIST OF ABBREVIATIONS

ABD	Accelerated Basin De-inventory
CSTF	Concentration, Storage, and Transfer Facilities
DMA	Direct Mercury Analyzer
DU	Depleted Uranium
DWPF	Defense Waste Processing Facility
HFIR	High Flux Isotope Reactor
HLW	High-Level Waste
IC	Ion Chromatography
ICP-ES	Inductively Coupled Plasma—Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma—Mass Spectrometry
IW	Inhibited Water
LIMS	Laboratory Information Management System
LTAD	Low Temperature Aluminum Dissolution
LW	Liquid Waste
M&TE	Measurement and Test Equipment
MS&E	Measurement Systems and Equipment
MST	monosodium titanate
MTR	Materials Test Reactor
PES	polyethylene sulfone
RSD	Relative Standard Deviation
SB	Sludge Batch
SNF	Spent Nuclear Fuel
SRE	Sodium Reactor Experiment
SRMC	Savannah River Mission Completion
SRNL	Savannah River National Laboratory
SRNS	Savannah River Nuclear Solutions
SRS	Savannah River Site
StB	Salt Batch
SWPF	Salt Waste Processing Facility
TIC/TOC	Total Inorganic Carbon/Total Organic Carbon
TS	Total Solids
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
WAC	Waste Acceptance Criteria
wt%	weight percent (g/100 g)

1.0 Introduction

1.1 Accelerated Basin De-inventory and Supernate Enrichment Testing Request

The Accelerated Basin De-inventory (ABD) program at the Savannah River Site (SRS) is designed to accelerate the de-inventory of L-Basin and accelerate the Spent Nuclear Fuel (SNF) disposition mission. Spent fuel will be dissolved in H-Canyon without recovery of uranium. The dissolver solutions will be temporarily stored, adjusted to meet downstream requirements, then transferred to the Concentration, Storage, and Transfer Facilities (CSTF) and subsequently immobilized in the Defense Waste Processing Facility (DWPF) during planned Sludge Batch (SB) campaigns.

This work is in response to a Technical Task Request (TTR) that was issued by Savannah River Mission Completion (SRMC).[1] The current Liquid Waste (LW) system plan forecasts that future SBs will contain ABD streams with equivalent uranium-235 ($^{235}U_{eq}$) enrichments[a] as high as 60.1 wt% starting with SB12.[2] As discussed in Section 1.2, addition of recently precipitated enriched uranium may lead to a higher uranium-235 (^{235}U) isotopic enrichment[b] in the supernate versus the sludge slurry. With the planned use of material decanted from the SB preparation tank in salt batches (StB), a higher ^{235}U isotopic enrichment in the supernate may challenge the maximum 8 wt% $^{235}U_{eq}$ enrichment limit for incoming feed to the Salt Waste Processing Facility (SWPF).[c,3] Higher enrichment in StB could have additional downstream impacts as well, such as acceptance of SWPF material into DWPF. To manage the use and processing of sludge supernate generated with higher ^{235}U isotopic enrichments, SRMC and Savannah River Nuclear Solutions (SRNS) requested that Savannah River National Laboratory (SRNL) study the enrichment phenomena between the supernate and sludge slurry in order to identify factors (such as time related to equilibrium conditions, whether or not depleted uranium is needed for the ABD streams, etc.) to lower the risk of exceeding the $^{235}U_{eq}$ enrichment for a StB and maximize the amount of fissile material processed at DWPF. SRNL issued a Task Technical and Quality Assurance Plan (TTQAP) to outline the testing and controls.[4]

1.2 Observations of Supernate Uranium Enrichment in Previous Testing and SRS Tank Farm Sampling

Previously, SRNL performed a radioactive waste study of gadolinium poisoning at SRS Tank Farm and DWPF conditions.[5] Tests involved adding a neutralized portion of ABD material with a slurry ^{235}U isotopic enrichment) of 60.9 wt% to a Tank 51 sample with a slurry ^{235}U isotopic enrichment of 0.58%. This was followed by a single washing and supernate decant step, and the settled slurry was subsequently used in DWPF-related testing. The supernate decanted during this testing showed a higher ^{235}U isotopic enrichment than the settled slurry, although the settled slurry had by far the majority of the total quantity of U. The decanted supernate had a ^{235}U isotopic enrichment of 47.5 wt%, compared with the slurry ^{235}U isotopic enrichment of 6.7 wt%.

The phenomenon of soluble phase ^{235}U isotopic enrichment exceeding the insoluble and slurry phase ^{235}U isotopic enrichment was also noted in SRNL sludge washing and field samples retrieved from Tank 51 for SB10. Freshly precipitated material from H-Canyon with >30% ^{235}U isotopic enrichment was added to Tank 51 during SB preparation and washing activities. Uranium enrichment of the lab-washed and two field-washed slurry supernates (9.0 wt%, 2.9 wt%, and 2.2 wt%, respectively) were greater than bulk slurry

[a] $^{235}U_{eq}$ mass is defined as ^{235}U mass plus 1.4 times ^{233}U mass; $^{235}U_{eq}$ enrichment is defined as $^{235}U_{eq}$ mass divided by total U mass.
[b] ^{235}U enrichment is defined as ^{235}U mass divided by total U mass.
[c] As communicated during test planning, the feed to the SWPF was limited to a $^{235}U_{eq}$ of 8 wt%. However, expected changes to the facility operation to allow the addition of lower quantities of monosodium titanate (MST) and more batches to be filtered during SWPF processing, the feed to SWPF may be limited to a $^{235}U_{eq}$ of 5.5 wt% or 4 wt% (see Reference 3).

^{235}U isotopic enrichment of 1.2 wt%.[6-8] In the field-washed material, both the supernate and sludge slurry were in compliance with the DWPF Waste Acceptance Criteria (WAC) for $^{235}U_{eq}$ enrichment.

A hypothesis for this observed uranium enrichment phenomenon is that the supernate concentration of uranium in the recently precipitated canyon stream is higher than the supernate concentration of uranium in the waste tank sludge. The supernate enrichment in the mixture will initially be weighted toward that of the enrichment of the stream with the higher supernate uranium concentration, which is that of the recently precipitated canyon slurry. At very short times where the supernate uranium has little time to exchange atoms with the solid-phase uranium, the supernate enrichment of the mixture can then be calculated as a mass balance of uranium concentrations and enrichments in the supernates of the two materials. With sufficient time, the supernate uranium concentration and enrichment will approach equilibrium with the solid phase due to interactions and exchange with prolonged contact with the slurry. More complicated hypotheses involve chemistry changes upon combining the neutralized ABD material and sludge slurry in the sludge preparation tank.

Historically, it has been relatively rare for liquid phase measurements of supernate ^{235}U enrichment to be reported for sludge slurry samples or for supernate samples from sludge slurry tanks. Table 1-1 and Table 1-2 reflect recent sampling results for which information is available on supernate ^{235}U enrichment. These recent analyses of active tanks give some indication that deviation of the supernate ^{235}U enrichment from the slurry ^{235}U enrichment is common.

Table 1-1 focuses on the observations in Tank 51 during the late stages of SB10 washing and during SB11 compilation. The results of two samples from SB10 (HTF-51-22-36/37 and HTF-51-22-87/88) were mentioned previously in this section, where the supernate ^{235}U enrichment results of 2.9 wt% and 2.2 wt% deviated from the slurry ^{235}U enrichment result of 1.2 wt%.[7,8] It is suspected that the transfer of recently precipitated Sodium Reactor Experiment (SRE) material from H-Canyon with >30 wt% ^{235}U enrichment contributed to the deviation of the supernate and slurry ^{235}U enrichment. Subsequently, a portion of the slurry material from Tank 51 was moved forward to Tank 40 for SB10 and the compilation of SB11 was initiated. The next applicable Tank 51 sample (HTF-51-23-30/31) was taken after a slurry transfer from Tank 22, multiple transfers of ABD material with slurry ^{235}U enrichment of less than 5 wt%, and a second slurry transfer from Tank 22. The results from HTF-51-23-30/31 show a higher supernate uranium concentration in the unwashed material, but also an unusually low supernate ^{235}U enrichment. The low supernate ^{235}U enrichment is counter to the expectation after an ABD material transfer of freshly precipitated uranium with a higher supernate ^{235}U enrichment (near 5 wt%). A potential explanation for that result is that some of the transfers between tanks involve multiple steps that transfer supernate between tanks. The Tank 51 samples from October 2023 (HTF-51-23-80/81), taken after a slurry transfer from Tank 35, show that the supernate ^{235}U enrichment of 1.8 wt% is indeed lower than the slurry ^{235}U enrichment of 4.3 wt%.[9]

Table 1-1. Uranium Content and Enrichment in Tank 51 Samples from SB10 Washing Through SB11 Compilation.

Tank 51 Samples			Slurry		Supernate	
Sample	Date	Details	U concentration	U-235 enrichment	U concentration	U-235 enrichment
HTF-51-22-36/37	3/29/2022	SB10 washing progress	1.75 wt% of TS	n.d.	0.647 mg/L	2.88 wt%
HTF-51-22-87/88	8/31/2022	SB10 confirmation	2.48 wt% of TS	1.2 wt%	3.92 mg/L	2.2 wt%
HTF-51-23-30/31	3/24/2023	SB11 post Tk22 transfer	n.d.	n.d.	22.9 mg/L*	0.91 wt%*
HTF-51-23-75	9/14/2023	SB11 supernate sample	n.d.	n.d.	16.8 mg/L	2.3 wt%
HTF-51-23-80/81	10/4/2023	SB11 post Tk35 transfer	0.870 wt% of TS	4.33 wt%	11.1 mg/L	1.78 wt%
HTF-51-24-4/5	2/8/2024	SB11 post Tk13 transfer	0.993 wt% of TS	3.86 wt%	5.07 mg/L	2.7 wt%

n.d. = not determined (not requested and not measured), U-235 enrichment = U-235/total U (mass)
* analysis performed much later than sample receipt

Tanks 15, 35, and 26 were also sampled during SB11 compilation, and some of the applicable results are shown in Table 1-2.[10-12] Supernate ^{235}U enrichment results were not included in the previous characterization references for Tanks 15, 35, and 26. The ^{235}U enrichment in the supernate of samples from Tank 26 was measured at the time of the previous characterization, while the ^{235}U enrichment in the supernate of Tanks 15 and 35 were measured as part of this study in order to determine their potential suitability in this study.

Tanks 15 and 35 had relatively low slurry uranium concentrations but high ^{235}U enrichments. Tank 15 slurry and supernate ^{235}U enrichments were in near agreement at 10.9 wt% and 9.6 wt%, respectively. Tank 35 slurry and supernate ^{235}U enrichments, however, showed a larger deviation at 10.5 wt% and 6.5 wt%, respectively. Tank 26 contained a larger uranium concentration at a depleted ^{235}U enrichment and showed a small but significant deviation between slurry and supernate ^{235}U enrichments of 0.25 wt% and 0.38 wt%, respectively. One hypothesis is that the transfer of supernate between tanks for use in sludge retrieval causes blending of the supernates in different tanks and contributes to the observation of deviations between slurry and supernate ^{235}U enrichments.

Table 1-2. Uranium Content and Enrichment in Other Slurry Samples Collected During SB11 Compilation.

Other Applicable SB11 Compilation Samples			Slurry		Supernate	
Sample	Date	Details	U concentration	U-235 enrichment	U concentration	U-235 enrichment
HTF-15-22-113/114	12/12/2022	Tank 15	0.003 wt% of TS	10.9 wt%	n.d.	n.d.
HTF-15-23-70/71	8/9/2023	Tank 15	n.d.	n.d.	1.53 mg/L*	9.6 wt%*
HTF-35-23-34/35	4/11/2023	Tank 35	0.008 wt% of TS	10.5 wt%	3.79 mg/L*	6.50 wt%*
FTF-26-23-17/18	9/1/2023	Tank 26	1.86 wt% of TS	0.25 wt%	1.43 mg/L	0.38 wt%

* analysis performed much later than sample receipt

1.3 Previous Uranium Solubility Research

Previous neutralization flowsheet studies gave an indication of the time for the uranium concentration of ABD material to reach equilibrium. The H-Canyon hold time is expected to be so short after pH adjustment that there will be a greater supernate concentration of uranium in the ABD material at the time of transfer to the Tank Farm than the equilibrium value of the mixture. The neutralization flowsheet studies performed for the aluminum-clad ABD material (and similar materials) used a measurement method that was not sensitive enough to quantify the uranium concentration in the supernate phase after neutralization.[13,14] From the more recent of the two studies, the supernate uranium concentration was <14 mg/L in the neutralized ABD material, corresponding to >99.3% of the uranium being insoluble. These studies did not track the soluble uranium concentration as a function of time after neutralization.

There is evidence that the soluble uranium concentration takes time to get to equilibrium in the CSTF. Solubility testing in uranium-spiked CSTF waste samples showed solubility in most cases resulted in uranium concentrations of 30 to 50 mg/L within the first 100 days but decreased to about 10 mg/L after 200 to 250 days.[15] In testing related to the 2H evaporator feed and drop tanks, mixing and contact with High-Level Waste (HLW) sludge caused precipitation of uranium and plutonium.[16] This was presumedly due to supersaturated Tank 38 and 43 supernate material having prolonged intimate contact with sludge as a nucleation medium. Uranium concentrations started around 55 mg/L initially and attained 26 mg/L and 13 mg/L after 7 days and 35 days, respectively. Plutonium also had reduced soluble concentrations after

contact with the Tank 51 sludge, but appeared to approach equilibrium faster, with not much additional change after about 14 days.

2.0 Experimental

2.1 Testing Summary

There are five test series as defined by the TTR and TTQAP:

- Series A involves the pH adjustment of ABD material.
- Series B involves the pH adjustment of Depleted Uranium (DU).
- Series C involves the addition of pH adjusted ABD material to a CSTF sludge slurry sample during SB preparation.
- Series D involves the interaction of the ABD material and sludge slurry mixture with other streams that change chemistry during SB preparation.
- Series E involves the interaction of decanted SB preparation tank material with other StB feeds during StB preparation.

2.2 Testing Details

Series A and B are meant to approximate the chemistry of the neutralization process that takes place within H-Canyon. Series C and D are meant to represent the SB preparation tank from the time that ABD material is added to a SB through subsequent processing steps in the preparation tank (sludge washing, additional sludge transfers, and Low Temperature Aluminum Dissolution (LTAD)). Series E is meant to represent blending of a stream decanted from the SB preparation tank into a StB blend tank.

Figure 2-1, Figure 2-2, and Figure 2-3 provide general overviews for the testing of series A and B, series C and D, and series E, respectively. The green vertical arrows show addition to or removal from the 15 mL centrifuge tubes used for testing. The light blue horizontal arrows represent the progression of time during the test using the same slurry or solution in a centrifuge tube (not the transfer of material to another centrifuge tube).

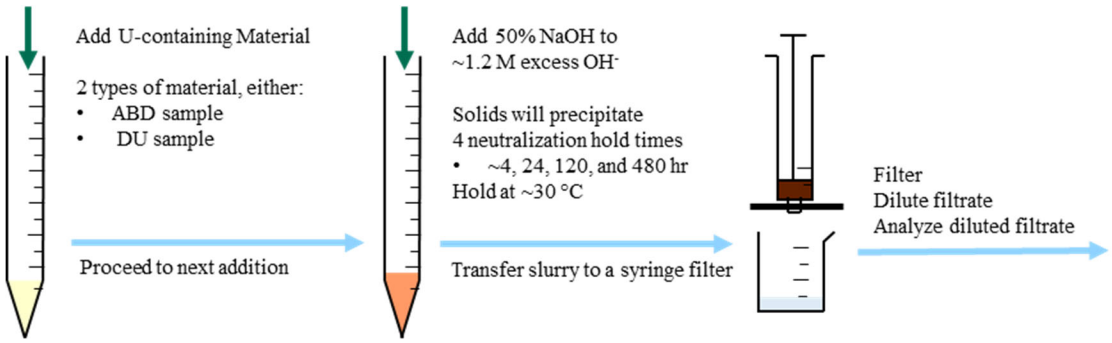

Figure 2-1. Test Series A and B.

Figure 2-1 represents test series A and B. First, the uranium-containing material (either the composite of ABD sample material or the DU sample material) was weighed into a 15-mL centrifuge tube. Next, the targeted weight of 50 wt% NaOH was added to the centrifuge tube over the course of approximately 5 minutes. The tube was capped and inverted multiple times to induce mixing. Visually noticeable precipitation was occurring during the NaOH addition and may have continued during mixing. The material

was held for a period of time before proceeding to the next step (nominally 4, 24, 120, or 480 hours). For these tests, the slurry was filtered with a cartridge syringe filter.

Nominally, the solutions were held at 30 °C on a temperature controlled shaker table. The 4-hour series A and B tests were not held at a specific temperature due to the short duration of the tests. Due to the time of the year, the ambient temperature in the cells was relatively cold at the time of these tests (~15 °C). However, addition of 50 wt% NaOH to the highly acidic uranium-containing materials likely caused a temperature increase in the mixture above the 30 °C target.

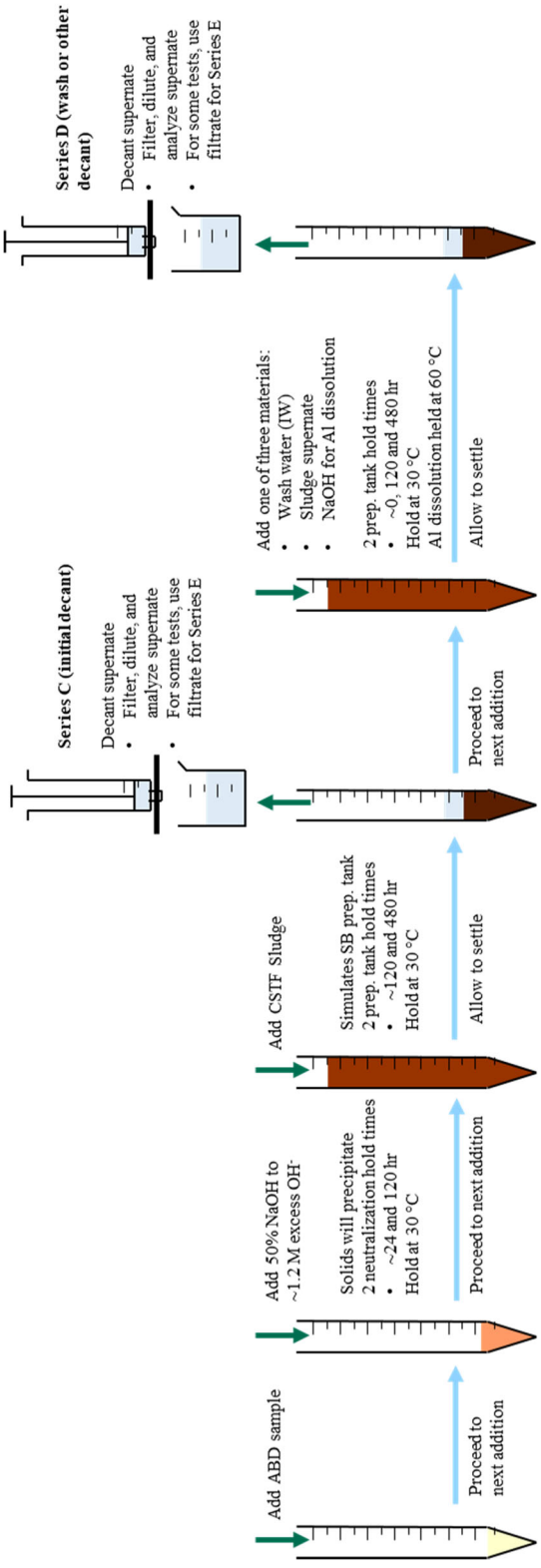

Figure 2-2. Test Series C and D.

Figure 2-2 represents test series C and D, which were performed sequentially. For series C, the ABD material was added, the pH was adjusted as in test series A, and the material was held at 30 °C for the targeted time period. The CSTF sludge material was added to the centrifuge tube neutralized ABD material over the course of approximately 5 minutes at a mass ratio of 5:1 sludge to neutralized ABD material. The tube was capped and inverted multiple times to induce mixing. The combined material was held at 30 °C on a heated shaker table for the targeted time period (representing the SB preparation tank) and was allowed to settle. The supernate was decanted and filtered. A portion of the supernate was diluted for chemical analysis and the remaining supernate material was used in series E testing or other analysis.

Series D involved contacting the slurry remaining from the series C test (after decanting the initial supernate) with one of three liquids: Inhibited Water (IW) for sludge washing, a mixture of 50 wt% NaOH and IW for LTAD, and supernate from a Tank 35 sludge slurry sample. The tube was capped and inverted multiple times to induce mixing. For the IW and the sludge supernate tests, tubes were held at 30 °C on a heated shaker table. For the LTAD tests, the tubes were held at 60 °C in a drying oven and solids were resuspended once per working day. For series D, typically one or more intermediate time samples were taken during the hold time. Intermediate samples were of the slurry, which were subsequently filtered prior to dilution and analysis of the liquid phase. At the final hold time, the supernate was decanted and filtered. A portion of the supernate was diluted for chemical analysis and the remaining supernate material was used in series E testing or other analysis.

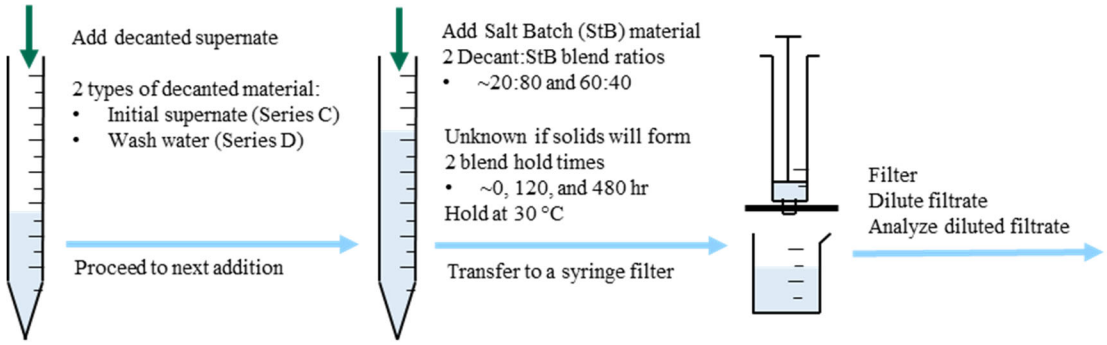

Figure 2-3. Test Series E.

Table 2-1. General Testing Matrix.

Test Series	Variable	Levels	Variable Levels Description
A + B	neut. hold time	4	4 hr, 24 hr, 120 hr (5 days), 480 hr (20 days)
	material	2+	ABD only; DU only; post adjustment mixture
C	neut. hold time	2	24 hr, 120 hr
	prep. tank blend order	3	ABD only, DU then ABD, ABD then DU
	prep. tank hold time	3	0 hr, 120 hr, 480 hr
D	neut. hold time	2	24 hr, 120 hr
	prep. tank interactions	3	wash water, transferred supernate, Al dissolution
	prep. tank hold time	3	0 hr, 120 hr, 480 hr
E	decant source	2	supernate from Series C, wash water from Series D
	blend ratio	2	decant:salt approximate mass ratios of 20:80 and 60:40
	blend hold time	3	0 hr, 120 hr, 480 hr

Figure 2-3 represents test series E, where decanted supernate from series C or D testing is added to a salt solution at fixed ratios and held for specified periods of time. Intermediately and at the end of testing, a portion of the supernate was filtered and diluted for chemical analysis.

Table 2-1 was included in the TTQAP and displays the testing parameters that were varied during each series of testing. The test design was not full factorial. Rather, priority was given to the conditions that were expected to provide the most useful information. Deviating from the TTQAP, no series C testing was performed to investigate the preparation tank blending order. The H-Canyon uranium source for the series C, D, and E tests was only the ABD material.

2.3 Equipment Details

Tests were generally performed on a very small scale using 1 to 1.5 grams of the uranium-containing sample per test (either ABD material or DU material). Tests were conducted in 15 mL polypropylene centrifuge tubes. When filtration was necessary, 0.45-µm or 0.2-µm polyethylene sulfone (PES) syringe filter cartridges (25 mm disks) were used with 5 mL or 10 mL syringes.

Test mixing was not prototypic, as mixing during holding periods was more mild than likely would be encountered for H-Canyon pH adjustment and for LTAD mixing, while mixing during holding periods was more vigorous than likely would be encountered for the majority of the time in the SB preparation tank. Mixing may increase the rate at which the uranium in the supernate phase comes to equilibrium with the uranium in the solid phase by removing some concentration gradients and diffusion limitations. For the conditions that were heated to 30 °C, the mixing and heating was performed on a small shaker table oscillating at 300 rpm (see Figure 2-4). The objective of temperature control was to remove temperature fluctuation as a variable, and 30 °C is within the range of temperatures typical in CSTF. For many samples, the mild mixing of the shaker table allowed for settling of the solids. In those cases, the solids were resuspended by shaking manually several times during the holding period. For the LTAD test at 60 °C, testing was performed in a drying oven. Samples in the drying oven were resuspended daily on working days, but were not continuously mixed. Temperatures were confirmed with Measurement and Test Equipment (M&TE) Type K thermocouples and M&TE thermocouple readers.

Figure 2-4. Heated Shaker Table Used in Cell 2.

2.4 Analytical Details

The primary analytical method used for this testing was Inductively Coupled Plasma—Mass Spectrometry (ICP-MS). For liquid phase analysis, supernates or slurries were filtered by 0.45-µm or 0.2-µm PES syringe filter cartridges. Samples for submission to ICP-MS analysis were diluted approximately 10-times with 2 M nitric acid. Select acid diluted samples were also analyzed by Inductively Coupled Plasma—Optical Emission Spectroscopy (ICP-ES) and Direct Mercury Analysis (DMA). For some tests, similarly prepared dilutions of approximately 10-times with deionized water were analyzed by titration for total base and free hydroxide, by Ion Chromatography (IC) for anions, and by Total Inorganic Carbon/Total Organic Carbon (TIC/TOC) analysis. Analytical methods conform with Measurement Systems and Equipment (MS&E) program requirements.

Slurry analytical results, where performed, used aqua regia digestion preparations followed by ICP-MS, ICP-ES, and DMA measurement.

Density was determined gravimetrically on 2 mL aliquots of supernate and was performed using M&TE balances. For test series A and B supernates, separate representative neutralization tests were performed to produce material for density measurements to represent the testing. For test series C, D, and E, density was measured on individual or combinations of representative samples and applied to the applicable materials. Weight percent solids measurements were not performed because, due to the scale of the tests, it would involve repeating tests from all test series to produce material for measurements.

2.5 Compiled Analysis of Materials Used

This section details the materials that were utilized in the testing.

2.5.1 ABD Material

The ABD material used in this testing is a compiled set of samples from H-Canyon Tanks 8.3 and 7.4. Tank 8.3 samples represent dissolved High Flux Isotope Reactor (HFIR) material and Tank 7.4 samples represent dissolved Materials Test Reactor (MTR) material. The previous sample analysis results for the individual samples and the sum or weighted average values for the combined mixture of the ABD material is shown in Table 2-2. The isotope abundance values are reported in mass fraction. Samples are referenced by their Laboratory Information Management System (LIMS) number.

The initial approximation of the combined mixture of ABD material was that 7 mL each of 20 samples would produce 140 mL of a blend with ^{235}U isotopic enrichment of 66 wt%. As seen in Table 2-2, the actual mixture of ABD material used in this testing was made from 16 samples each containing different amounts of material. This yielded an ABD material blend with a volume of 70 mL, a density of 1.349 g/mL, a uranium concentration of 3.4 g/L, and a ^{235}U isotopic enrichment of 74.2 wt%. Based on previous sample analysis, ^{233}U was not measured on the samples shown in Table 2-2 because it was likely to be below the limit of detection. Thus, the $^{235}U_{eq}$ enrichment is considered to be equal to the ^{235}U enrichment in the ABD material.

The ABD material ^{235}U isotopic enrichment of 74.2 wt% is higher than the enrichment limit in CSTF and would have required down-blending to less than or equal to 66 wt% prior to transfer. Nevertheless, this ABD material was used in this testing without down-blending. For the goals of this testing, the ABD material ^{235}U isotopic enrichment just needed to be very different from the ^{235}U isotopic enrichment of the sludge and salt samples used in order to trace the difference in the behavior of the uranium in neutralized (recently precipitated) ABD material from the CSTF sludge.

Table 2-2. Samples Composited to Create the ABD Material Mixture.

Sample	Tank ID	Mass (g)	Density (g/mL)	Volume (mL)	U (g/L)	U-234 Abundance	U-235 Abundance	U-236 Abundance	U-238 Abundance	Pu-238 (dpm/mL)	Pu-239/240 (dpm/mL)	Free Acid (N)
40017345	8.3	9.404	1.3633	6.898	3.22	0.0115	0.8589	0.0626	0.067	5.99E+06	1.23E+06	1.12
40017346	8.3	9.264	1.3634	6.795	3.24	0.0115	0.8589	0.0625	0.067	6.12E+06	1.01E+06	1.1
40017818	8.3	1.405	1.3750	1.022	3.59	0.0115	0.8587	0.0628	0.067	1.03E+07	1.88E+06	0.84
40017819	8.3	10.121	1.3769	7.351	3.64	0.0115	0.8586	0.0628	0.0671	7.48E+06	1.35E+06	0.9
40017844	7.4	5.509	1.2961	4.250	3.97	0.0061	0.3777	0.0591	0.5571	3.28E+07	4.82E+06	0.92
40017845	7.4	5.204	1.2946	4.020	4.11	0.0061	0.3777	0.0593	0.5567	2.89E+07	4.11E+06	0.74
40018557	8.3	9.076	1.3518	6.714	3.06	0.0117	0.8588	0.0626	0.067	5.70E+06	1.02E+06	1.09
40018558	8.3	6.154	1.3536	4.546	3.06	0.0116	0.8589	0.0625	0.067	5.41E+06	9.87E+05	1.09
40018837	8.3	7.401	1.3590	5.446	3.15	0.0116	0.8681	0.0555	0.0649	1.01E+06	4.44E+06	1.18
40018838	8.3	6.536	1.3586	4.811	3.19	0.0116	0.8681	0.0554	0.0649	4.12E+06	9.42E+05	1.2
40018952	7.4	5.009	1.3158	3.807	4.11	0.0057	0.384	0.0571	0.5532	3.09E+07	5.11E+06	1.08
40018953	7.4	3.172	1.3146	2.413	4.11	0.0057	0.3838	0.0571	0.5534	3.04E+07	4.55E+06	1.16
40019178	8.3	7.015	1.3588	5.163	3.15	0.0116	0.8654	0.058	0.0651	4.40E+06	7.46E+05	0.96
40019179	8.3	4.546	1.3597	3.343	3.17	0.0116	0.8655	0.0579	0.065	4.17E+06	9.63E+05	0.98
40019291	8.3	1.424	1.3574	1.049	2.98	0.0117	0.8546	0.0656	0.0681	6.27E+06	1.13E+06	1.29
40019292	8.3	3.537	1.3562	2.608	3.01	0.0117	0.8547	0.0656	0.068	6.02E+06	9.25E+05	1.47
Total or Average		94.777	1.3494	70.235	3.39	0.0102	0.7423	0.0602	0.1873	1.06E+07	2.07E+06	1.06

2.5.2 DU Material

The DU material used was decanted supernate from H-Canyon Tank 171 sample 40014698. Table 2-3 displays the original analytical results for that sample. The sample contained a very high 395 g/L of uranium and a very low ^{235}U isotopic enrichment of 0.19%. The sample reportedly contained black settled solids, which were not used in this testing. If the solids in the sample contained uranium, the uranium concentration in the material used in this testing may have been lower than the reported sample uranium concentration.

Table 2-3. Sample Used for DU Material.

analyte	units	value
density	g/mL	1.611
U	g/L	394.82
U-234	g/g U	0.00001
U-235	g/g U	0.00191
U-236	g/g U	0.00005
U-238	g/g U	0.99804
Free Acid	N	2.98

2.5.3 CSTF Sludge Material

The CSTF sludge material representing the SB preparation tank prior to additional neutralized ABD material was a mixture of Tank 51 and Tank 26 samples. The Tank 51 material was formed from an approximately equal volume blend of three sets of Tank 51 samples that were taken near the end of SB10 preparation: HTF-51-22-36/37, HTF-51-22-49/50, and HTF-51-22-87/88.[7,8] The Tank 51 samples were collected late in the sludge washing process and after SRE material was added to SB10. As seen in Table 1-1, two of the three sets of Tank 51 samples have both slurry and supernate uranium isotopic enrichment measurements. The Tank 51 material had a hydroxide concentration of 0.6 M and a slurry density of 1.08 g/mL. Because these samples were collected near the end of the SB10 washing process, the salt concentration was relatively low. Tank 26 sample FTF-26-23-17/18,[12] which was taken to support SB11 compilation, was blended with the Tank 51 material. As seen in Table 1-2, the Tank 26 material had a depleted ^{235}U isotopic enrichment (0.25 wt% in the slurry and 0.38 wt% in the supernate). Tank 26 material had a sodium concentration of 9.5 M, a hydroxide concentration of 2.8 M and a slurry density of 1.5 g/mL.

In creating 1009.3 grams of CSTF sludge slurry blend, 718.4 grams of washed Tank 51 material and 290.9 grams of unwashed Tank 26 material were used. Total solids, insoluble solids, slurry density, and supernate density was not confirmed for the Tank 51 and Tank 26 sludge material mixture. Table 2-4 contains the analytical results for the blended sludge sample slurry utilized in this testing. As measured by ICP-MS, the blended sludge sample had a slurry uranium concentration of 5.39 g/kg and a ^{235}U isotopic enrichment of 0.69 wt%. Table 2-5, Table 2-6, and Table 2-7 contain the anion, ICP-ES, and ICP-MS analytical results, respectively, for the supernate of the blended sludge sample utilized in this testing. The supernate of the blended sludge mixture had a uranium concentration of 11.6 mg/L and a ^{235}U isotopic enrichment of 0.34 wt%.

Table 2-4. CSTF Sludge Sample Blend from Tank 51 and Tank 26, Slurry Analysis.

Analyte	Average (mg/kg)	RSD	Analyte	Average (mg/kg)	RSD
Ag	< 4.8E+01	--	Si	4.60E+02	0.4%
Al	1.34E+04	1.5%	Sn	< 2.5E+02	--
B	< 2.4E+01	--	Sr	2.29E+01	1.4%
Ba	5.91E+01	1.7%	Th	1.91E+03	2.6%
Be	< 5.7E-01	--	Ti	< 3.9E+01	--
Ca	1.14E+03	3.8%	U	5.20E+03	2.5%
Cd	< 1.1E+01	--	V	< 6.9E+00	--
Ce	1.18E+02	1.3%	Zn	< 5.9E+01	--
Co	< 1.3E+01	--	Zr	1.18E+02	2.0%
Cr	4.86E+02	2.9%	Th-230	< 4.2E-02	--
Cu	< 1.1E+02	--	Th-232	1.80E+03	1.6%
Fe	1.37E+04	2.2%	U-233	6.07E-01	0.3%
Gd	6.20E+01	3.6%	U-234	7.50E-01	2.1%
K	< 3.5E+02	--	U-235	3.72E+01	1.6%
La	3.92E+01	5.0%	U-236	2.99E+00	1.5%
Li	< 4.0E+01	--	Np-237	1.54E+00	1.9%
Mg	4.32E+02	1.5%	U-238	5.35E+03	1.3%
Mn	3.25E+03	2.2%	Pu-239	2.30E+01	1.5%
Mo	< 2.6E+01	--	Pu-240	1.75E+00	1.4%
Na	6.37E+04	2.5%	mass 241	5.03E-01	2.3%
Ni	3.67E+02	2.2%	mass 242	1.18E-01	2.1%
P	< 2.4E+02	--	mass 243	< 4.2E-02	--
Pb	< 6.7E+01	--	mass 244	< 4.2E-02	--
S	3.39E+03	2.1%	Total U	5.39E+03	1.3%
Sb	< 5.3E+01	--	U-235/U	0.69%	0.5%

Table 2-5. CSTF Sludge Sample Blend from Tank 51 and Tank 26, Supernate Anion Analysis.

Analyte	Average	RSD
F^- (M)	7.21E-02	1.1%
CHO_2^- (M)	< 3.9E-03	--
Cl^- (M)	< 4.9E-03	--
NO_2^- (M)	3.69E-01	0.1%
NO_3^- (M)	1.06E+00	0.7%
PO_4^{3-} (M)	< 1.8E-03	--
SO_4^{2-} (M)	1.20E-01	0.0%
$C_2O_4^{2-}$ (M)	1.56E-02	0.2%
Br^- (M)	< 1.1E-02	--
CO_3^{2-} (M)	2.20E-01	0.1%
TOC (mg C/L)	6.63E+02	1.5%
Total Base (M)	1.49E+00	2.3%
OH^- (M)	9.81E-01	7.0%

Table 2-6. CSTF Sludge Sample Blend from Tank 51 and Tank 26, Supernate ICP-ES Analysis.

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	< 2.8E-01	Mn	< 4.5E-01
Al	3.18E+03	Mo	9.09E+00
B	2.35E+01	Na	6.35E+04
Ba	< 1.7E+00	Ni	< 8.3E-01
Be	< 8.0E-02	P	4.83E+01
Ca	< 3.3E+00	Pb	< 5.7E+00
Cd	< 3.3E-01	S	4.06E+03
Ce	< 3.0E+00	Sb	< 3.0E+00
Co	< 5.5E-01	Si	< 8.6E+00
Cr	1.91E+02	Sn	< 1.2E+01
Cu	< 4.6E+00	Sr	< 1.3E-01
Fe	< 4.0E+00	Th	< 3.1E+00
Gd	< 2.8E-01	Ti	< 8.5E-01
K	3.68E+02	U	< 2.9E+01
La	< 2.7E-01	V	< 6.7E+00
Li	< 1.3E+01	Zn	< 1.1E+00
Mg	< 1.6E+00	Zr	< 7.4E-01

Table 2-7. CSTF Sludge Sample Blend from Tank 51 and Tank 26, Supernate ICP-MS Analysis.

Analyte	Average	RSD
Th-232 (mg/L)	9.93E-02	28.2%
U-233 (mg/L)	< 9.2E-03	--
U-234 (mg/L)	< 9.2E-03	--
U-235 (mg/L)	4.02E-02	1.2%
U-236 (mg/L)	< 9.2E-03	--
Np-237(mg/L)	< 9.2E-03	--
U-238 (mg/L)	1.16E+01	1.4%
Pu-239 (mg/L)	< 9.2E-03	--
Pu-240 (mg/L)	< 9.2E-03	--
Total U (mg/L)	1.16E+01	1.4%
U-235/U (wt%)	0.35%	2.6%

2.5.4 Salt Batch Material

Series E tests used a StB9 qualification sample from Tank 21 (HTF-21-23-36/37/38/39).[17] The sample as utilized was free of insoluble solids. The Tank 21 StB sample had a uranium concentration of 6.2 mg/L and a ^{235}U isotopic enrichment of 1.8 wt%.

2.5.5 Other Sludge Supernate

For the other sludge supernate, the liquid phase material from Tank 35 samples (HTF-35-23-34/35) was used.[11] The Tank 35 supernate had a uranium concentration of 3.8 mg/L and a ^{235}U isotopic enrichment of 6.5 wt%.

2.6 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60.[18] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[19] Data are recorded in the electronic laboratory notebook system as experiment A6583-00142-36 and associated experiments. Except where noted in Section 3.3, all work, analysis, and documentation were performed with quality assurance methods commensurate with the Safety Class data requirements.

3.0 Results and Discussion

This section presents results of the testing, followed by interpretation and a discussion of the impacts of the results on processing.

3.1 Neutralization of ABD and DU Samples

3.1.1 ABD Neutralization (Series A)

Series A tests involved the addition of 50 wt% NaOH to the ABD material, holding that mixture for a pre-determined amount of time, and measuring the uranium concentration and enrichment in the supernate by ICP-MS (see Figure 2-1). Table 3-1 contains the test conditions and results for the eight series A tests.

As seen in Figure 3-1 and Table 3-2, the liquid phase uranium concentration shows a clear downward trend with time over the course of this test. Initial liquid phase uranium concentration was in the range of 50 mg/L after 4 hours, 40 mg/L after one day, 25 mg/L after five days, and 20 mg/L after 20 days.

Table 3-3 contains data for liquid phase anions for neutralization testing representative of different portions of this work. While testing was in progress, it was realized that the target free hydroxide concentration of the post-neutralized material for the initial tests was much greater than the target of >1.2 M. The left-most series A column and the series B column represent the neutralization tests as performed in the initial tests (including most of section 3.1 and tubes 001 through 008 of section 3.2). The amount of 50 wt% NaOH used in subsequent testing was changed to achieve a free hydroxide closer to the target of >1.2 M. The right-most column of series A data in Table 3-3 represents the subsequent testing with the adjusted NaOH addition that was used for tubes 009 through 012, 019, and 020 in Section 3.2.

The ^{235}U isotopic enrichment of the liquid phase averaged 73.3 wt%. This matches the ABD material enrichment of 74.2 wt% within the experimental uncertainty. Because ^{233}U was below the limit of detection, the $^{235}U_{eq}$ enrichment for the supernate is equal to the ^{235}U enrichment.

Plutonium supernate concentration results were generally low, with the highest Pu concentration in the freshly neutralized ABD material supernate being 0.07 mg/L. All subsequent test series had lower supernate Pu concentrations than this 0.07 mg/L result.

Table 3-4 contains ICP-ES data for supernates from the pH-adjusted Series A and Series B tests. Sodium was the only component in the neutralized DU material supernate present at above the ICP-ES detection limit. For the ABD material supernate, elements detected by ICP-ES included aluminum, chromium, copper, iron, molybdenum, sodium, silicon, and zinc.

Table 3-1. Series A - Uranium and Plutonium in the Liquid Phase of the Neutralized ABD Slurry.

Series	A	A	A	A	A	A	A	A
ABD sample (g)	1.013	1.037	0.494	0.524	0.550	0.496	0.492	0.492
50 wt% NaOH (g)	0.989	1.029	0.477	0.544	0.492	0.501	0.470	0.565
Neut. hold (hr)	4	4	26	26	121	122	483	483
LIMS #	34441	34442	34445	34446	34449	34450	34453	34454
U-233 (mg/L)	< 6.6E-03	< 5.7E-03	< 1.7E-01	< 1.6E-02	< 1.7E-02	< 1.0E-01	< 2.1E-02	< 4.1E-02
U-234 (mg/L)	5.30E-01	5.71E-01	4.21E-01	4.44E-01	2.66E-01	2.77E-01	2.31E-01	1.75E-01
U-235 (mg/L)	3.66E+01	3.82E+01	2.85E+01	2.96E+01	1.85E+01	1.87E+01	1.56E+01	1.25E+01
U-236 (mg/L)	3.11E+00	3.32E+00	2.40E+00	2.59E+00	1.57E+00	1.59E+00	1.34E+00	1.06E+00
Np-237(mg/L)	2.20E-02	2.36E-02	< 1.7E-01	1.72E-02	< 1.7E-02	< 1.0E-01	< 2.1E-02	< 4.1E-02
U-238 (mg/L)	9.60E+00	1.00E+01	7.44E+00	7.84E+00	4.87E+00	4.91E+00	4.22E+00	3.30E+00
Pu-239 (mg/L)	3.39E-02	4.13E-02	< 1.7E-01	6.64E-02	3.28E-02	< 1.0E-01	4.19E-02	< 4.15E-02
Pu-240 (mg/L)	< 6.6E-03	6.51E-03	< 1.7E-01	< 1.6E-02	< 1.7E-02	< 1.0E-01	< 2.1E-02	< 4.1E-02
Total U (mg/L)	4.99E+01	5.21E+01	3.87E+01	4.05E+01	2.52E+01	2.55E+01	2.14E+01	1.70E+01
U-235/U (wt%)	73.4%	73.3%	73.5%	73.2%	73.4%	73.4%	73.0%	73.3%

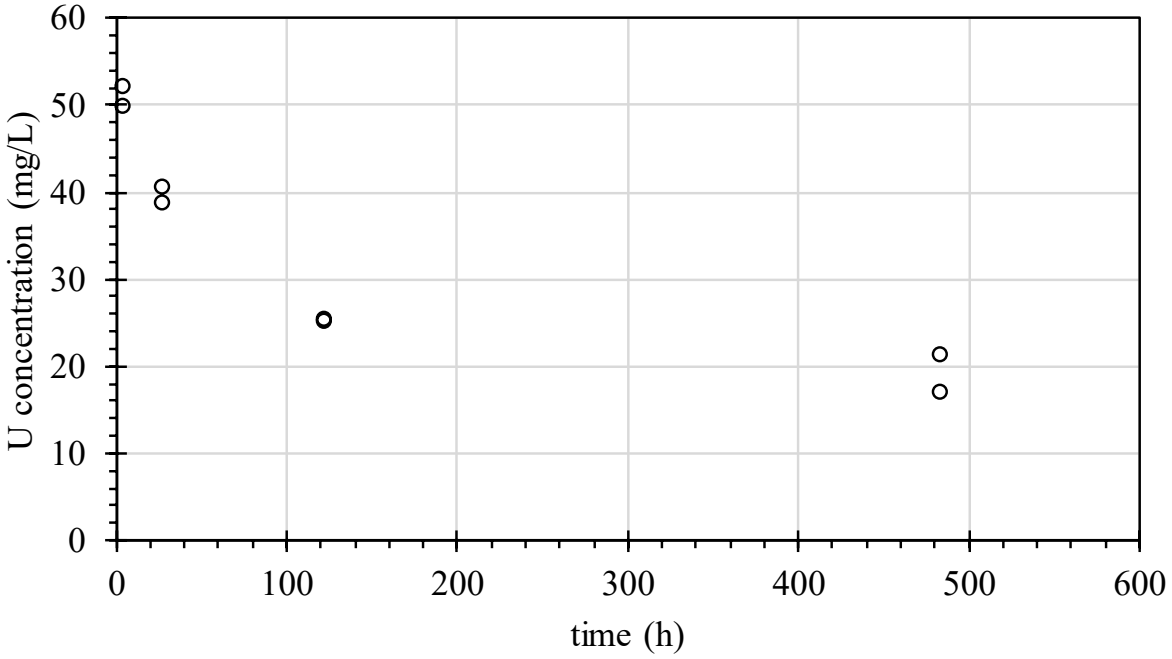

Figure 3-1. Series A - Liquid Phase Concentration of Uranium as a Function of Time after Neutralization of ABD Material.

Table 3-2. Series A - Summary of Liquid Phase Uranium Concentration in the Neutralized ABD Slurry.

time (h)	U (mg/L)	RSD
4	5.10E+01	3.1%
26	3.96E+01	3.2%
121	2.54E+01	0.7%
483	1.92E+01	16%

Table 3-3. Series A and B - Anion Analysis of Neutralized ABD and DU Materials.

Series		A		A		B	
Sample type		ABD		ABD		DU	
Sample (g)		1.039		0.984		0.993	
DI water (g)		0.000		0.000		0.977	
50 wt% NaOH (g)		1.000		0.669		0.724	
Neutralization hold (hr)		64		48		64	
Supernate Density (g/mL)		1.347		1.333		1.173	
Analyte	Units	Average	RSD	Average	RSD	Average	RSD
F^-	M	<7.1E-02	--	<7.0E-02	--	<1.6E-01	--
CHO_2^-	M	<3.0E-02	--	<3.0E-02	--	<6.8E-02	--
Cl^-	M	<3.8E-02	--	<3.8E-02	--	<8.6E-02	--
NO_2^-	M	<2.9E-02	--	<2.9E-02	--	<6.6E-02	--
NO_3^-	M	4.43E+00	1.7%	4.93E+00	0.3%	2.14E+00	4.9%
PO_4^{3-}	M	<1.4E-02	--	<1.4E-02	--	<3.2E-02	--
SO_4^{2-}	M	<1.4E-02	--	<1.4E-02	--	<3.2E-02	--
$C_2O_4^{2-}$	M	<1.5E-02	--	<1.5E-02	--	<3.5E-02	--
Br^-	M	<4.2E-02	--	<8.3E-02	--	<9.6E-02	--
OH^-	M	3.70E+00	8.3%	1.50E+00	8.5%	1.87E+00	26%

Table 3-4. Series A and B – ICP-ES Analysis of Neutralized ABD and DU Materials.

Analyte	Series A (mg/L)	Series B (mg/L)	Analyte	Series A (mg/L)	Series B (mg/L)
Ag	< 5.5E-01	< 3.8E-01	Mn	< 9.0E-01	< 6.2E-01
Al	2.49E+04	< 1.7E+01	Mo	4.87E+01	< 3.8E-01
B	< 2.6E+00	< 1.8E+00	Na	1.79E+05	8.59E+04
Ba	< 7.7E-01	< 5.3E-01	Ni	< 1.8E+00	< 1.2E+00
Be	< 1.6E-01	< 1.1E-01	P	< 4.9E+00	< 3.4E+00
Ca	< 1.3E+00	< 9.0E-01	Pb	< 1.5E+01	< 1.0E+01
Cd	< 2.3E-01	< 1.6E-01	S	< 9.7E+00	< 6.6E+00
Ce	< 6.0E+00	< 4.1E+00	Sb	< 6.0E+00	< 4.1E+00
Co	< 1.1E+00	< 7.5E-01	Si	8.98E+01	< 5.4E+00
Cr	3.80E+01	< 5.2E+00	Sn	< 6.9E+00	< 4.7E+00
Cu	2.34E+01	< 7.8E-01	Sr	< 2.7E-01	< 1.8E-01
Fe	1.72E+01	< 2.2E+00	Th	< 9.3E+00	< 6.4E+00
Gd	< 5.5E-01	< 3.8E-01	Ti	< 3.6E+00	< 2.5E+00
K	< 2.1E+02	< 1.4E+02	U	< 3.7E+01	< 2.6E+01
La	< 5.4E-01	< 3.7E-01	V	< 6.5E+00	< 4.5E+00
Li	< 2.7E+01	< 1.8E+01	Zn	1.21E+01	< 1.9E+00
Mg	< 3.1E+00	< 2.1E+00	Zr	< 2.3E+00	< 1.6E+00

3.1.2 DU Neutralization (Series B)

Series B tests involved the addition of 50 wt% NaOH to the concentrated DU material, holding that mixture for a pre-determined amount of time, and measuring the uranium concentration and enrichment in the supernate by ICP-MS (see Figure 2-1). For all but the 4-hour tests, deionized water was added to the DU material at a mass ratio of 0.98:1 water to DU to represent how H-Canyon would dilute a DU material that was this concentrated. Table 3-5 contains the test conditions and results for the eight series B tests.

As seen in Figure 3-2, there is a general downward trend in the supernate uranium concentration with time for the DU neutralization tests. There is more scatter in the supernate uranium concentration data for the DU neutralization than was observed for the ABD neutralization tests. The soluble uranium concentrations also trended lower for the DU tests than for the ABD tests. The 4-hour uranium concentration averaged just above 20 mg/L and after 20 days the uranium concentration averaged just below 10 mg/L.

The exact reason why the post-neutralization uranium concentration differed between the ABD and DU tests is unknown, but it is expected to be a relative concentration effect. The acidic DU material has a much higher uranium concentration than the ABD material (395 g/L vs 3.3 g/L). The neutralized ABD material has far more other metals precipitating and in solution than the DU material. This is because the ABD material is dissolved spent nuclear fuel with fission products and cladding components while the DU material is primarily dissolved uranium oxide. While both the ABD material and DU material precipitated solids immediately upon pH adjustment, the solids were visibly different in color, particle/agglomerate size, and flow behavior. It is likely that a combination of chemical and physical differences in the two materials contributed to their differing time dependencies of post-neutralization liquid phase uranium concentration. See Figure 3-3 for photographs of the neutralized ABD material and DU material.

Table 3-5. Uranium and Plutonium in the Liquid Phase of the Neutralized DU Slurry.

Series	B	B	B	B	B	B	B	B
DU sample (g)	1.047	1.014	1.009	1.198	0.991	1.035	0.999	1.007
water (g)	0.000	0.000	0.985	1.125	0.934	0.981	0.964	0.945
50 wt% NaOH (g)	0.734	0.737	0.696	0.847	0.734	0.712	0.690	0.709
Neut. hold (hr)	4	4	26	26	121	121	482	482
LIMS #	34443	34444	34447	34448	34451	34452	34455	34456
U-233 (mg/L)	< 3.4E-02	< 2.2E-02	< 1.7E-02	< 1.8E-02	< 2.3E-02	< 1.2E-02	< 9.5E-03	< 1.3E-02
U-234 (mg/L)	< 3.4E-02	< 2.2E-02	< 1.7E-02	< 1.8E-02	< 2.3E-02	< 1.2E-02	< 9.5E-03	< 1.3E-02
U-235 (mg/L)	3.79E-02	3.85E-02	1.89E-02	1.99E-02	2.57E-02	3.67E-02	1.37E-02	1.87E-02
U-236 (mg/L)	< 3.4E-02	< 2.2E-02	< 1.7E-02	< 1.8E-02	< 2.3E-02	< 1.2E-02	< 9.5E-03	< 1.3E-02
Np-237(mg/L)	< 3.4E-02	< 2.2E-02	< 1.7E-02	< 1.8E-02	< 2.3E-02	< 1.2E-02	< 9.5E-03	< 1.3E-02
U-238 (mg/L)	2.00E+01	2.17E+01	1.05E+01	1.08E+01	1.39E+01	2.09E+01	7.71E+00	1.08E+01
Pu-239 (mg/L)	< 3.4E-02	< 2.2E-02	< 1.7E-02	< 1.8E-02	< 2.3E-02	< 1.2E-02	< 9.5E-03	< 1.3E-02
Pu-240 (mg/L)	< 3.4E-02	< 2.2E-02	< 1.7E-02	< 1.8E-02	< 2.3E-02	< 1.2E-02	< 9.5E-03	< 1.3E-02
Total U (mg/L)	2.00E+01	2.17E+01	1.05E+01	1.09E+01	1.39E+01	2.09E+01	7.72E+00	1.08E+01
U-235/U (wt%)	0.189%	0.177%	0.179%	0.183%	0.185%	0.176%	0.178%	0.173%

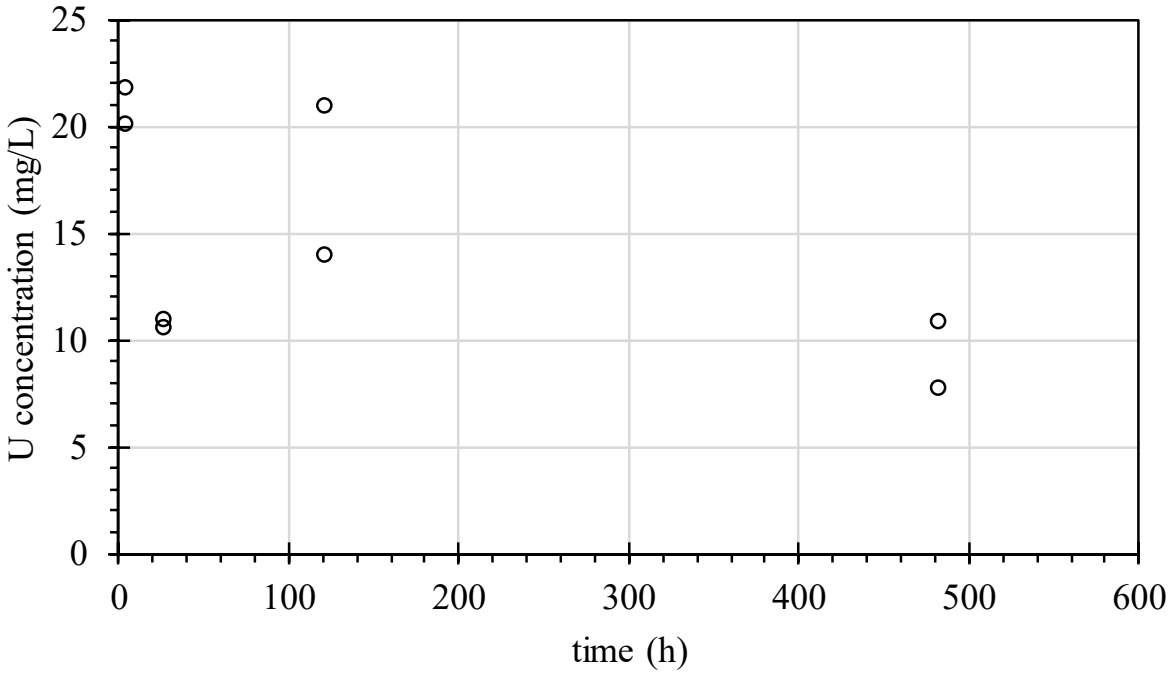

Figure 3-2. Series B - Liquid Phase Concentration of Uranium as a Function of Time after Neutralization of DU Material.

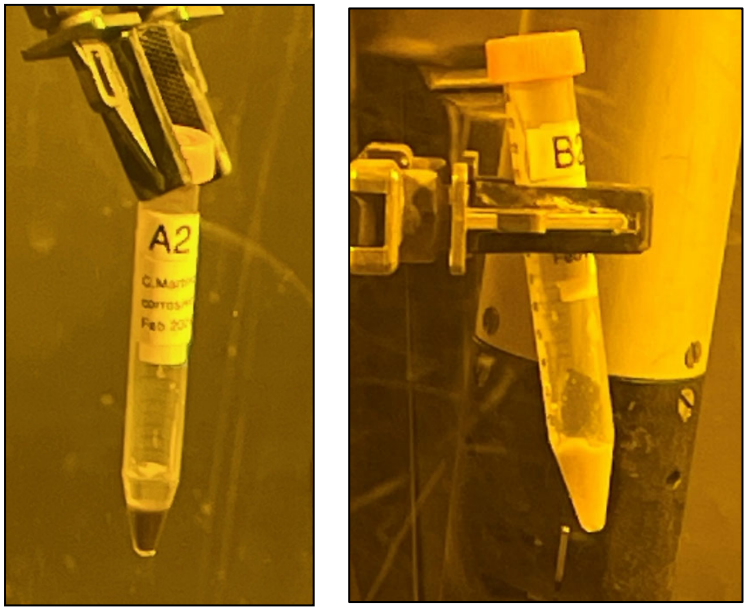

Figure 3-3. Series A and B - Appearance of the pH-Adjusted ABD Material (left) and pH-Adjusted DU (right).

3.1.3 Observations from ABD and DU Mixtures

Two tests were performed in duplicate to test a scheme to potentially reduce the amount of DU added to ABD material in H-Canyon. This does not represent the way that ABD and DU are processed together or separately in H-Canyon. The parameters and results from that testing are shown in Table 3-6. First, ABD material is added to the centrifuge tube. Next, the amount of 50 wt% NaOH required to adjust both the ABD and DU material is added to the centrifuge tube. This slurry is mixed and held for a day. Finally, the DU is added to the centrifuge tube. This slurry is mixed and held for a day, followed by decanting, filtering, diluting, and analyzing the supernate. One test targeted the amount of DU required to adjust the slurry ^{235}U isotopic enrichment to 5.0 wt% and the other test targeted approximately 1/10 of that DU addition (which would adjust the slurry isotopic enrichment to approximately 38 wt%).

The results from this testing were not as expected. Due to the DU being added second, the thought was that the supernate enrichment after DU addition would be below the enrichment of the slurry. However, while the test targeting 5 wt% slurry enrichment hit the target slurry enrichment, the supernate enrichment was 15 wt%, which is considerably higher than expected. One hypothesis is that the very high concentration of uranium in the DU fluid led to a relatively small volume of that stream being added, and this low volume stream was not ideal to target isotopic dilution of the supernate phase. Another hypothesis is that the mixing at the time of DU addition was not adequate to focus the subsequently added DU to isotopically dilute the supernate phase.

As seen in Figure 3-4, the yellow DU solids and the brown ABD material solids separated after gravity settling. The pH adjustment of the very concentrated DU stream apparently led to much larger and/or denser particles to allow for this separation. In contrast to a conclusion of the ABD analysis for inclusion in SB11,[20] the DU material would ideally not be added separately to a SB due to the risk that the DU may subsequently not be homogeneously distributed throughout the settled sludge. However, these tests may not precisely represent how ABD and DU materials can be pH adjusted separately in H-Canyon. Also, such separation behavior would not be expected when ABD and DU materials are blended prior to pH adjustment.

Table 3-6. Uranium and Plutonium in the Liquid Phase of a Test Performed for Sequential Addition of ABD and DU Material

Series	AB		AB	
ABD sample (g)	3.988		4.972	
50 wt% NaOH (g)	3.600		3.878	
Neut. hold (hr)	18		18	
DU sample (g)	0.602		0.055	
DU hold (hr)	~24		~24	
LIMS #	34457	34458	34459	34460
U-233 (mg/L)	< 5.7E-03	< 5.5E-03	< 5.4E-03	< 5.7E-03
U-234 (mg/L)	6.49E-02	6.49E-02	1.92E-01	1.89E-01
U-235 (mg/L)	3.98E+00	4.17E+00	1.22E+01	1.22E+01
U-236 (mg/L)	3.74E-01	3.75E-01	1.12E+00	1.12E+00
Np-237(mg/L)	< 5.7E-03	< 5.5E-03	6.61E-03	6.78E-03
U-238 (mg/L)	2.24E+01	2.30E+01	1.36E+01	1.36E+01
Pu-239 (mg/L)	1.36E-02	1.27E-02	2.33E-02	2.24E-02
Pu-240 (mg/L)	< 5.7E-03	< 5.5E-03	< 5.4E-03	< 5.7E-03
Total U (mg/L)	2.68E+01	2.76E+01	2.70E+01	2.71E+01
U-235/U (wt%)	14.9%	15.1%	45.0%	45.0%

Figure 3-4. Sequentially pH Adjusted ABD Material and Depleted Uranium Material, After Settling.

3.2 Mixtures in the Tank Farm After ABD Addition to Sludge Receipt Material

3.2.1 Overall Summary of Tank Farm ABD Blending

Series C and D tests simulate interactions of the ABD material within the SB preparation tank (see Figure 2-2). Series C and D tests were performed in series within seven sets of two centrifuge tubes. Table 3-7 contains the overall summary of the test conditions and key results for the series C and D tests. The source of the H-Canyon uranium in the testing was ABD material only, DU was not used. The uranium concentration and ^{235}U isotopic enrichment results in Table 3-7 are averaged results from the two tests, and the hold times listed are nominal. Data for the individual tests with detailed masses combined and more precise hold times are reported in subsequent sections. The three types of series D testing involve contact with IW, heated aluminum dissolution (LTAD), and contact with Tank 35 supernate.

The rows in Table 3-7 are organized into four groups. The top section provides information on the hold times and type of fluid used for the series D test contacts. The next two sections contain the liquid phase results for the uranium concentration and ^{235}U isotopic enrichment, respectively. The bottom section contains the results for slurry ^{235}U isotopic enrichment. The first four columns of data are the tests that examine the impact of varying the hold time of the pH adjusted ABD stream prior to mixing with the CSTF sludge slurry, and the hold time of the ABD and sludge slurry mixture prior to decanting the supernate. The last three columns of data are the tests that examine the three different contact fluids for series D. As noted in Section 3.1.1, the pH adjustment for the first four columns (tubes 001 through 008) used more sodium hydroxide than was used in for the last three columns (tubes 009 through 012 and 019 through 020). The last three columns were closer to the target sodium hydroxide addition of >1.2 M.

In all cases, the supernate from the ABD and sludge mixture had a ^{235}U isotopic enrichment that deviated above the slurry mixture ^{235}U isotopic enrichment of 4.0 wt%. This included the initial supernate decanted from the ABD and sludge mixture (series C), as well as subsequent washes with inhibited water, sodium hydroxide addition for heated aluminum dissolution, and contact with other CSTF sludge supernate (series D).

From the tests looking at neutralization and sludge mixture hold times (the first four data columns), the following observations can be made.

- Uranium concentration in the initial decant (series C) appears to be a function of hold time in the SB preparation tank.
- Uranium concentration in the wash water decant (series D) is not a strong function of hold time of the IW wash in the SB preparation tank.
- The supernate ^{235}U isotopic enrichment appears to decrease (in the direction of the slurry enrichment) at cases with longer hold times, regardless of whether the hold time is during the neutralization step, the initial sludge mix step, or the subsequent IW wash step.
- The initial decant supernate ^{235}U isotopic enrichment was highest (60 wt%) for the shortest hold times lowest (38 wt%) for the longest hold times.
- After decanting the initial supernate and contacting the material with wash water for 20 days, the supernate ^{235}U isotopic enrichment ranged from 22 wt% to 27 wt%.

For the tests looking at contact with different fluids (the last three data columns), the following observations can be made:

- The initial supernates from the three tests at this more typical hydroxide addition had lower ^{235}U isotopic enrichment (45 wt% to 47 wt%) than the analogous test with a higher hydroxide addition and should be considered more representative.
- LTAD testing resulted in the closest approach to the slurry ^{235}U isotopic enrichment (supernate of 18 wt% vs. slurry of 4.0 wt%), but further approach to the slurry enrichment did not continue between the 5 day and 20-day LTAD samples.
- Mixing of the Tank 35 sludge supernatant with the ABD material and sludge slurry gave unexpected ^{235}U isotopic enrichment results, remaining at a relatively high supernate enrichment (45 wt% after 20 days).

From the entire set of data, the following observations can be made:

- Uranium concentration in the initial decant (series C) appears to be a function of hold time in the SB preparation tank.
- Uranium concentration in the wash water decant (series D) is not a strong function of hold time in the SB preparation tank during washing.

Table 3-7. Series C and D Test Summary - Mixtures of pH Adjusted ABD and CSTF Sludge.

Tube number	001/003	002/004	005/006	007/008	009/012	010/011	019/020
Neutralization hold	1 day	1 day	5 days	5 days	1 day	1 day	1 day
Sludge mix hold	5 days	20 days	5 days	20 days	5 days	5 days	5 days
Series D case	IW	IW	IW	IW	LTAD	IW	Tk35 sup.
Total uranium concentration in liquid (mg/L)							
Series C	14.9	11.2	15.0	11.5	15.1	15.2	16.9
Series D, 0 days	10.2	n.m.	10.3*	n.m.	10.8	n.m.	n.m.
Series D, ~5 days	12.1	9.2	10.1	8.9	9.0	10.4	n.m.
Series D, ~20 days	10.0	9.8	8.9	8.7	8.1	12.0	9.9
U-235/Total U in liquid (wt%)							
Series C	60%	48%	48%	38%	45%	45%	47%
Series D, 0 days	53%	n.m.	39%*	n.m.	47%	n.m.	n.m.
Series D, ~5 days	34%	25%	32%	26%	18%	34%	n.m.
Series D, ~20 days	27%	22%	27%	22%	19%	23%	45%
U-235/Total U in slurry (wt%)							
Series D, ~20 days	4.0%	n.m.	4.0%	n.m.	n.m.	4.0%	n.m.

n.m. = not measured, * indicates time is ~24 h rather than 0 days

Series E tests simulate the material decanted from the SB preparation tank into a StB blend tank. Table 3-8 contains the overall summary of the test conditions and key results for the series E tests. There were two types of fluids mixed with the StB material, the initial decanted material during series C and the IW wash decanted during series D. The neutralization blend times were nominally 1 day, the initial sludge mix hold times were nominally 5 days, and the IW contact hold time was nominally 20 days. Data for the individual tests with detailed masses combined and more precise hold times are reported in subsequent sections.

For the tests contacting initial SB decants and StB material, supernate uranium concentrations and ^{235}U isotopic enrichment did not change significantly during testing, indicating that not much precipitation occurred with time. Additionally, there was no visual indication of precipitation. Thus, the IW wash and StB contact tests were performed only at the longest hold time.

The supernate ^{235}U isotopic enrichment was consistent with the mass balance, where cases with more of the ABD material had higher enrichment and cases with more StB material had lower enrichment.

Table 3-8. Mixtures of Decants with Salt Batch Material: Series E Test Summary.

Tube number	013/016	014/015	017	018
Decant source	C (initial)	C (initial)	D (IW)	D (IW)
Decant:Salt ratio	20:80	60:40	20:80	60:40
Total uranium concentration in liquid (mg/L)				
Series E, 0 days	8.0	12.9	n.m.	n.m.
Series E, ~5 days	7.6	11.4	n.m.	n.m.
Series E, ~20 days	8.6	12.6	6.8	8.8
U-235/Total U in liquid (wt%)				
Series E, 0 days	21%	36%	n.m.	n.m.
Series E, ~5 days	21%	36%	n.m.	n.m.
Series E, ~20 days	23%	38%	11%	21%

n.m. = not measured

3.2.2 Initial Decants (Series C)

Table 3-9 and Table 3-10 contain the test conditions and actinide results from the initial decants of the pH adjusted ABD material and CSTF sludge mixtures (series C). Results are grouped in sets of two for the tests that were approximately duplicated. Concentrations and ^{235}U isotopic enrichment results were relatively consistent between duplicated tests.

Pu-239 was measured at low levels in some of the tests. U-233 and Pu-240 were not measured in the tests, but a detection limit is provided. Other actinide masses not shown (230 and 241 through 244) have the same detection limit as U-233 and Pu-240.

Additional cation, anion, and density analysis of select samples from this test series are included in Table 3-11, Table 3-12, and Table 3-13. Hydroxide was near the detection limit.

Figure 3-5 contains a photograph of a mixture of pH adjusted ABD material and SB material representative of the series C tests. As seen at the end of a test, there is a layer of settled dark solids below a layer of relatively clear supernatant liquid.

Table 3-9. Test Series C Decanted Supernate Results, Part 1 of 2.

Series	C	C	C	C	C	C	C	C
Tube	001	003	002	004	005	006	007	008
ABD sample (g)	1.002	0.998	0.980	0.992	1.028	0.982	1.010	0.992
50 wt% NaOH (g)	0.980	0.959	0.991	0.965	0.998	0.942	0.956	0.956
Neut. hold (h)	28	28	28	28	120	120	120	120
Sludge (g)	9.889	9.905	9.913	9.922	9.901	9.894	9.927	9.902
Sludge hold (h)	116	116	480	480	119	119	480	480
Supernate decant (g)	2.566	2.242	3.409	3.029	3.000	2.820	3.480	2.842
LIMS #	LW34500	LW34501	LW34849	LW34850	LW34497	LW34496	LW34863	LW34864
Th-232 (mg/L)	2.05E-02	2.71E-02	3.01E-02	2.79E-02	2.34E-02	3.36E-02	2.03E-02	1.66E-02
U-233 (mg/L)	< 8.0E-03	< 5.4E-03	< 1.0E-02	< 1.2E-02	< 6.0E-03	< 5.8E-03	< 6.4E-03	< 6.2E-03
U-234 (mg/L)	1.31E-01	1.27E-01	7.89E-02	7.29E-02	1.04E-01	1.05E-01	6.79E-02	6.35E-02
U-235 (mg/L)	8.95E+00	8.82E+00	5.63E+00	5.10E+00	7.23E+00	6.78E+00	4.51E+00	4.20E+00
U-236 (mg/L)	7.68E-01	7.42E-01	4.67E-01	4.27E-01	6.04E-01	6.13E-01	3.98E-01	3.71E-01
Np-237(mg/L)	< 8.0E-03	< 5.4E-03	< 1.0E-02	< 1.2E-02	< 6.0E-03	< 5.8E-03	< 6.4E-03	< 6.2E-03
U-238 (mg/L)	5.25E+00	5.03E+00	5.19E+00	5.49E+00	7.08E+00	6.55E+00	6.75E+00	6.68E+00
Pu-239 (mg/L)	8.81E-03	7.72E-03	< 1.0E-02	< 1.2E-02	7.96E-03	9.53E-03	1.06E-02	1.60E-02
Pu-240 (mg/L)	< 8.0E-03	< 5.4E-03	< 1.0E-02	< 1.2E-02	< 6.0E-03	< 5.8E-03	< 6.4E-03	< 6.2E-03
Total U (mg/L)	1.51E+01	1.47E+01	1.14E+01	1.11E+01	1.50E+01	1.40E+01	1.17E+01	1.13E+01
U-235/U (wt%)	59.3%	59.9%	49.5%	46.0%	48.2%	48.3%	38.4%	37.1%

Table 3-10. Test Series C Results, Part 2 of 2.

Series	C	C	C	C	C	C
Tube	009	012	010	011	019	020
ABD sample (g)	1.517	1.529	1.601	1.514	1.586	1.493
50 wt% NaOH (g)	1.151	1.178	1.263	1.213	1.232	1.180
Neut. hold (h)	23	23	23	23	24	24
Sludge (g)	13.432	13.463	14.291	13.665	14.117	13.370
Sludge hold (h)	148	148	168	168	120	120
Supernate decant (g)	4.012	4.921	4.818	3.861	5.769	3.634
LIMS #	LW34511	LW34512	LW34510	LW34515	LW35160	LW35161
Th-232 (mg/L)	1.81E-02	2.59E-02	2.09E-02	4.12E-02	< 6.1E-03	2.60E-02
U-233 (mg/L)	< 5.7E-03	< 1.0E-02	< 4.9E-03	< 5.4E-03	< 6.1E-03	< 5.3E-03
U-234 (mg/L)	1.18E-01	9.67E-02	1.03E-01	1.11E-01	1.13E-01	1.01E-01
U-235 (mg/L)	7.40E+00	6.20E+00	6.56E+00	7.08E+00	8.26E+00	7.49E+00
U-236 (mg/L)	6.73E-01	5.65E-01	5.99E-01	6.54E-01	6.68E-01	6.09E-01
Np-237(mg/L)	< 5.7E-03	< 1.0E-02	< 4.9E-03	< 5.4E-03	< 6.1E-03	< 5.3E-03
U-238 (mg/L)	7.72E+00	7.45E+00	7.85E+00	7.39E+00	7.98E+00	8.57E+00
Pu-239 (mg/L)	8.22E-03	< 1.0E-02	7.92E-03	8.90E-03	9.23E-03	8.60E-03
Pu-240 (mg/L)	< 5.7E-03	< 1.0E-02	< 4.9E-03	< 5.4E-03	< 6.1E-03	< 5.3E-03
Total U (mg/L)	1.59E+01	1.43E+01	1.51E+01	1.52E+01	1.70E+01	1.68E+01
U-235/U (wt%)	46.5%	43.3%	43.4%	46.5%	48.5%	44.7%

Table 3-11. Test Series C ICP-ES and Hg Results (Tubes 005/006).

Analyte	Average (mg/L)	%RSD	Analyte	Average (mg/L)	%RSD
Ag	< 1.5E-01	--	Mn	< 1.5E-01	--
Al	6.43E+03	0.5%	Mo	1.53E+01	0.4%
B	2.15E+01	0.3%	Na	8.55E+04	1.3%
Ba	< 9.5E-02	--	Ni	< 8.4E-01	--
Be	< 8.3E-02	--	P	< 5.9E+01	--
Ca	< 2.3E+00	--	Pb	< 9.4E+00	--
Cd	< 3.5E-01	--	S	3.43E+03	1.4%
Ce	< 2.3E+00	--	Sb	< 7.4E+00	--
Co	< 3.2E-01	--	Si	< 1.2E+01	--
Cr	1.70E+02	0.4%	Sn	< 2.4E+01	--
Cu	< 2.6E+00	--	Sr	< 4.7E-02	--
Fe	< 1.2E+00	--	Th	< 1.4E+01	--
Gd	< 3.1E-01	--	Ti	< 5.2E-01	--
Hg	7.42E+01	9.7%	U	< 1.8E+01	--
K	2.80E+02	0.9%	V	< 9.7E-01	--
La	< 2.0E-01	--	Zn	< 2.9E+00	--
Li	< 2.3E+00	--	Zr	< 1.3E-01	--
Mg	< 7.1E-02	--			

Table 3-12. Test Series C ICP-ES Results (Tubes 009/011).

Analyte	Average (mg/L)	%RSD	Analyte	Average (mg/L)	%RSD
Ag	< 1.8E-01	--	Mn	< 3.0E-01	--
Al	6.47E+03	1.2%	Mo	1.48E+01	1.9%
B	1.96E+01	1.1%	Na	8.46E+04	1.6%
Ba	< 2.5E-01	--	Ni	< 5.8E-01	--
Be	< 5.2E-02	--	P	4.04E+01	1.6%
Ca	< 4.3E-01	--	Pb	< 4.9E+00	--
Cd	< 7.7E-02	--	S	3.27E+03	0.2%
Ce	< 2.0E+00	--	Sb	< 2.0E+00	--
Co	< 3.6E-01	--	Si	< 8.0E+00	--
Cr	1.66E+02	0.2%	Sn	< 2.3E+00	--
Cu	< 3.8E-01	--	Sr	< 8.7E-02	--
Fe	< 1.0E+00	--	Th	< 3.1E+00	--
Gd	< 1.8E-01	--	Ti	< 1.2E+00	--
K	3.16E+02	0.7%	U	< 1.7E+01	--
La	< 1.8E-01	--	V	< 2.1E+00	--
Li	< 8.8E+00	--	Zn	< 2.2E+00	--
Mg	< 1.0E+00	--	Zr	< 7.6E-01	--

Table 3-13. Test Series C Anion Results.

Series	C	C		
Tube	009	011		
ABD sample (g)	1.517	1.514		
50 wt% NaOH (g)	1.151	1.213		
Neut. hold (h)	23	23		
Sludge (g)	13.432	13.665		
Sludge hold (h)	148	168		
Supernate decant (g)	4.012	3.861		
Supernate density (mg/L)	1.189	1.189		
LIMS #	LW34520	LW34521	Average	RSD
F^- (M)	<6.6E-02	<6.4E-02	<6.5E-02	
CHO_2^- (M)	<2.8E-02	<2.7E-02	<2.8E-02	--
Cl^- (M)	<3.5E-02	<3.4E-02	<3.5E-02	--
NO_2^- (M)	2.49E-01	2.50E-01	2.50E-01	0.5%
NO_3^- (M)	1.27E+00	1.25E+00	1.26E+00	1.3%
PO_4^{3-} (M)	<1.3E-02	<1.3E-02	<1.3E-02	--
SO_4^{2-} (M)	8.63E-02	8.70E-02	8.66E-02	--
$C_2O_4^{2-}$ (M)	<1.4E-02	<1.4E-02	<1.4E-02	--
Br^- (M)	<7.9E-02	<7.6E-02	<7.8E-02	--
CO_3^{2-} (M)	1.77E-01	1.77E-01	1.77E-01	0.1%
TOC (mg C/L)	1.28E+03	3.85E+02	8.33E+02	76%
Total Base (M)	1.56E+00	1.59E+00	1.57E+00	1.4%
OH^- (M)	<1.0E+00	9.53E-01	9.53E-01	--

Figure 3-5. Settled Mixture of ABD Material and CSTF Sludge at Completion of a Series C Test.

3.2.3 Sludge Washing Decants (Series D)

Table 3-14, Table 3-15, and Table 3-16 contain the tabulated data for the test conditions and actinide concentration results for the single contact washing of the sludge with IW. Results are grouped in sets of two for the tests that were approximately duplicated. Concentrations and ^{235}U isotopic enrichment results were relatively consistent between duplicated tests. Other actinide masses not shown (230 and 241 through 244) have the same detection limit as U-233 and Pu-240.

Additional cation, anion, and density analysis of select samples from this test series are included in Table 3-17 and Table 3-18. Hydroxide was below the detection limit.

Table 3-14. Test Series D Results for a Single Inhibited Water Wash, Part 1 of 3.

Series	D	D	D	D	D	D	D	D	D	D
Tube	001	003	001	003	001	003	002	004	002	004
ABD sample (g)	1.002	0.998	1.002	0.998	1.002	0.998	0.98	0.992	0.980	0.992
50 wt% NaOH (g)	0.980	0.959	0.980	0.959	0.980	0.959	0.991	0.965	0.991	0.965
Neut. hold (h)	28	28	28	28	28	28	28	28	28	28
Sludge (g)	9.889	9.905	9.889	9.905	9.889	9.905	9.913	9.922	9.913	9.922
Sludge hold (h)	116	116	116	116	116	116	480	480	480	480
Supernate decant (g)	2.566	2.242	2.566	2.242	2.566	2.242	3.409	3.029	3.409	3.029
Contact fluid	IW	IW	IW	IW	IW	IW	IW	IW	IW	IW
IW (g)	7.523	7.229	7.523	7.229	7.523	7.229	7.411	7.478	7.411	7.478
Contact hold (h)	0	0	120	120	480	480	120	120	480	480
LIMS #	LW34498	LW34499	LW34502	LW34503	LW34865	LW34866	LW34859	LW34860	LW35164	LW35165
Th-232 (mg/L)	3.57E-02	3.28E-02	2.92E-02	2.43E-02	6.27E-03	1.10E-02	3.76E-02	2.22E-02	< 7.1E-03	6.08E-03
U-233 (mg/L)	< 4.6E-03	< 4.3E-03	< 3.4E-03	< 3.0E-03	< 5.2E-03	< 6.2E-03	< 3.5E-03	< 3.3E-03	< 7.1E-03	< 5.1E-03
U-234 (mg/L)	7.74E-02	7.91E-02	6.07E-02	5.91E-02	4.19E-02	4.07E-02	3.68E-02	3.39E-02	3.03E-02	2.74E-02
U-235 (mg/L)	5.38E+00	5.44E+00	4.13E+00	4.02E+00	2.80E+00	2.69E+00	2.45E+00	2.20E+00	2.25E+00	2.03E+00
U-236 (mg/L)	4.52E-01	4.56E-01	3.57E-01	3.41E-01	2.43E-01	2.30E-01	2.16E-01	1.93E-01	1.89E-01	1.67E-01
Np-237(mg/L)	< 4.6E-03	< 4.3E-03	< 3.4E-03	< 3.0E-03	< 5.2E-03	< 6.2E-03	< 3.5E-03	< 3.3E-03	< 7.1E-03	< 5.1E-03
U-238 (mg/L)	4.20E+00	4.23E+00	7.68E+00	7.46E+00	6.92E+00	7.07E+00	6.60E+00	6.57E+00	7.48E+00	7.44E+00
Pu-239 (mg/L)	< 4.6E-03	< 4.3E-03	< 3.4E-03	< 3.0E-03	1.43E-02	1.20E-02	< 3.5E-03	< 3.3E-03	5.25E-02	4.79E-02
Pu-240 (mg/L)	< 4.6E-03	< 4.3E-03	< 3.4E-03	< 3.0E-03	< 5.2E-03	< 6.2E-03	< 3.5E-03	< 3.3E-03	< 7.1E-03	< 5.1E-03
Total U (mg/L)	1.01E+01	1.02E+01	1.22E+01	1.19E+01	1.00E+01	1.00E+01	9.31E+00	9.00E+00	9.96E+00	9.66E+00
U-235/U (wt%)	53.2%	53.3%	33.8%	33.8%	27.9%	26.8%	26.4%	24.4%	22.6%	21.0%

Table 3-15. Test Series D Results for a Single Inhibited Water Wash, Part 2 of 3.

Series	D	D	D	D	D	D	D	D	D	D
Tube	005	006	005	006	005	006	007	008	007	008
ABD sample (g)	1.028	0.982	1.028	0.982	1.028	0.982	1.010	0.992	1.010	0.992
50 wt% NaOH (g)	0.998	0.942	0.998	0.942	0.998	0.942	0.956	0.956	0.956	0.956
Neut. hold (h)	120	120	120	120	120	120	120	120	120	120
Sludge (g)	9.901	9.894	9.901	9.894	9.901	9.894	9.927	9.902	9.927	9.902
Sludge hold (h)	119	119	119	119	119	119	480	480	480	480
Supernate decant (g)	3.000	2.820	3.000	2.820	3.000	2.820	3.480	2.842	3.480	2.842
Contact fluid	IW	IW	IW	IW	IW	IW	IW	IW	IW	IW
IW (g)	7.533	7.487	7.533	7.487	7.533	7.487	7.523	7.515	7.523	7.515
Contact hold (h)	24	24	120	120	480	480	120	120	480	480
LIMS #	LW34504	LW34505	LW34513	LW34514	LW34868	LW34867	LW35152	LW35153	LW35166	LW35167
Th-232 (mg/L)	4.91E-02	4.42E-02	2.10E-02	5.67E-02	< 6.5E-03	< 6.6E-03	4.74E-02	5.16E-02	< 5.2E-03	< 4.9E-03
U-233 (mg/L)	< 3.8E-03	< 3.5E-03	< 3.8E-03	< 4.4E-03	< 6.5E-03	< 6.6E-03	< 6.6E-03	< 5.5E-03	< 5.2E-03	< 4.9E-03
U-234 (mg/L)	5.86E-02	5.69E-02	5.10E-02	4.87E-02	3.73E-02	3.58E-02	3.23E-02	3.03E-02	2.87E-02	2.41E-02
U-235 (mg/L)	4.05E+00	3.91E+00	3.28E+00	3.05E+00	2.51E+00	2.35E+00	2.44E+00	2.22E+00	2.02E+00	1.75E+00
U-236 (mg/L)	3.41E-01	3.34E-01	2.97E-01	2.78E-01	2.14E-01	2.02E-01	1.98E-01	1.79E-01	1.71E-01	1.47E-01
Np-237(mg/L)	< 3.8E-03	< 3.5E-03	< 3.8E-03	< 4.4E-03	< 6.5E-03	< 6.6E-03	< 6.6E-03	< 5.5E-03	< 5.2E-03	< 4.9E-03
U-238 (mg/L)	6.03E+00	5.85E+00	6.46E+00	6.64E+00	6.14E+00	6.30E+00	5.86E+00	6.89E+00	5.93E+00	7.28E+00
Pu-239 (mg/L)	< 3.8E-03	< 3.5E-03	< 3.8E-03	< 4.4E-03	8.51E-03	1.36E-02	< 6.6E-03	< 5.5E-03	3.62E-02	3.59E-02
Pu-240 (mg/L)	< 3.8E-03	< 3.5E-03	< 3.8E-03	< 4.4E-03	< 6.5E-03	< 6.6E-03	< 6.6E-03	< 5.5E-03	< 5.2E-03	< 4.9E-03
Total U (mg/L)	1.05E+01	1.01E+01	1.01E+01	1.00E+01	8.90E+00	8.89E+00	8.54E+00	9.32E+00	8.15E+00	9.20E+00
U-235/U (wt%)	38.7%	38.5%	32.5%	30.5%	28.2%	26.4%	28.6%	23.8%	24.8%	19.0%

Table 3-16. Test Series D Results for a Single Inhibited Water Wash, Part 3 of 3.

Series	D	D	D	D
Tube	010	011	010	011
ABD sample (g)	1.601	1.514	1.601	1.514
50 wt% NaOH (g)	1.263	1.213	1.263	1.213
Neut. hold (h)	23	23	23	23
Sludge (g)	14.291	13.665	14.291	13.665
Sludge hold (h)	168	168	168	168
Supernate decant (g)	4.818	3.861	4.818	3.861
Contact fluid	IW	IW	IW	IW
IW (g)	4.902	4.817	4.902	4.817
Contact hold (h)	120	120	480	480
LIMS #	LW34861	LW34862	LW35158	LW35159
Th-232 (mg/L)	5.15E-02	3.35E-02	1.25E-02	< 6.9E-03
U-233 (mg/L)	< 4.9E-03	< 4.9E-03	< 6.3E-03	< 6.9E-03
U-234 (mg/L)	5.05E-02	5.62E-02	3.72E-02	3.98E-02
U-235 (mg/L)	3.33E+00	3.79E+00	2.72E+00	2.92E+00
U-236 (mg/L)	2.92E-01	3.30E-01	2.20E-01	2.34E-01
Np-237(mg/L)	< 4.9E-03	< 4.9E-03	< 6.3E-03	< 6.9E-03
U-238 (mg/L)	6.22E+00	6.66E+00	8.92E+00	8.97E+00
Pu-239 (mg/L)	< 4.9E-03	< 4.9E-03	< 6.3E-03	< 6.9E-03
Pu-240 (mg/L)	< 4.9E-03	< 4.9E-03	< 6.3E-03	< 6.9E-03
Total U (mg/L)	9.89E+00	1.08E+01	1.19E+01	1.22E+01
U-235/U (wt%)	33.7%	35.0%	22.9%	24.0%

Table 3-17. ICP-ES Results for Series D Test with Inhibited Water at a 120 Hour Hold Time (Tubes 007/008).

Analyte	Average (mg/L)	%RSD	Analyte	Average (mg/L)	%RSD
Ag	< 1.6E-01	--	Mn	< 1.6E-01	--
Al	2.90E+03	3.9%	Mo	7.14E+00	0.3%
B	9.79E+00	4.7%	Na	4.22E+04	2.1%
Ba	< 2.0E-01	--	Ni	< 1.4E+00	--
Be	< 8.4E-02	--	P	< 3.2E+01	--
Ca	< 2.3E+00	--	Pb	< 9.5E+00	--
Cd	< 3.6E-01	--	S	1.57E+03	3.4%
Ce	< 2.3E+00	--	Sb	< 7.5E+00	--
Co	< 3.2E-01	--	Si	< 3.2E+00	--
Cr	1.00E+02	3.8%	Sn	< 2.5E+01	--
Cu	< 3.2E-01	--	Sr	< 4.8E-02	--
Fe	< 1.2E+00	--	Th	< 1.4E+01	--
Gd	< 3.1E-01	--	Ti	< 5.3E-01	--
K	1.32E+02	3.6%	U	< 1.8E+01	--
La	< 2.0E-01	--	V	< 7.5E-01	--
Li	< 2.4E+00	--	Zn	< 8.2E-01	--
Mg	< 7.2E-02	--	Zr	< 1.3E-01	--

Table 3-18. Anion Results for Series D Test with Inhibited Water.

Series	D	D		
Tube	010	011		
ABD sample (g)	1.601	1.514		
50 wt% NaOH (g)	1.263	1.213		
Neut. hold (h)	23	23		
Sludge (g)	14.291	13.665		
Sludge hold (h)	168	168		
Supernate decant (g)	4.818	3.861		
Contact fluid	IW	IW		
IW (g)	4.902	4.817		
Contact hold (h)	120	120		
Supernate density (g/mL)	1.136	1.136		
LIMS #	LW34524	LW34525	Average	RSD
F^- (M)	<4.4E-02	<4.1E-02	<4.2E-02	
CHO_2^- (M)	<1.8E-02	<1.7E-02	<1.8E-02	--
Cl^- (M)	<2.3E-02	<2.2E-02	<2.3E-02	--
NO_2^- (M)	1.70E-01	1.76E-01	1.73E-01	2.4%
NO_3^- (M)	8.24E-01	8.52E-01	8.38E-01	2.3%
PO_4^{3-} (M)	<8.8E-03	<8.1E-03	<8.4E-03	--
SO_4^{2-} (M)	5.45E-02	5.76E-02	5.60E-02	--
$C_2O_4^{2-}$ (M)	<9.5E-03	<8.7E-03	<9.1E-03	--
Br^- (M)	<5.2E-02	<4.8E-02	<5.0E-02	--
CO_3^{2-} (M)	1.13E-01	1.23E-01	1.18E-01	6.1%
TOC (mg C/L)	9.40E+02	7.27E+02	8.33E+02	18%
Total Base (M)	8.98E-01	9.93E-01	9.46E-01	7.1%
OH^- (M)	<6.7E-01	<6.2E-01	<6.4E-01	--

3.2.4 LTAD Decants (Series D)

Table 3-19 contains the tabulated data for the test conditions and actinide concentration results for the LTAD process performed on the sludge. During series D, a mixture of 50 wt% NaOH and IW was added to the sludge remaining from series C testing, and the mixture was heated to 60 °C for the duration of the test. Results are grouped in sets of two for the tests that were approximately duplicated. Concentrations and ^{235}U isotopic enrichment results were relatively consistent between duplicated tests. Other actinide masses not shown (230 and 241 through 244) have the same detection limit as U-233 and Pu-240.

Table 3-20 contains ICP-ES results for the LTAD samples taken at 120 and 480 hours. The soluble aluminum concentration for the LTAD samples were higher than the IW samples, and the LTAD trended higher with longer LTAD times.

Additional results from anion analysis are included in Table 3-21.

Table 3-19. Test Series D Results for LTAD, Heated Sodium Hydroxide Contact.

Series	D	D	D	D	D	D
Tube	009	012	009	012	009	012
ABD sample (g)	1.517	1.529	1.517	1.529	1.517	1.529
50 wt% NaOH (g)	1.151	1.178	1.151	1.178	1.151	1.178
Neut. hold (h)	23	23	23	23	23	23
Sludge (g)	13.432	13.463	13.432	13.463	13.432	13.463
Sludge hold (h)	148	148	148	148	148	148
Supernate decant (g)	4.012	4.921	4.012	4.921	4.012	4.921
Contact fluid	LTAD	LTAD	LTAD	LTAD	LTAD	LTAD
50 wt% NaOH (g)	2.013	1.800	2.013	1.800	2.013	1.800
IW (g)	3.719	4.466	3.719	4.466	3.719	4.466
Contact hold (h)	0	0	120	120	480	480
LIMS #	LW34506	LW34507	LW34851	LW34852	LW35156	LW35157
Th-232 (mg/L)	5.02E-02	3.47E-02	2.28E-02	6.66E-02	2.49E-02	3.01E-02
U-233 (mg/L)	< 4.2E-03	< 4.3E-03	< 7.1E-03	< 6.1E-03	< 6.5E-03	< 5.3E-03
U-234 (mg/L)	9.88E-02	6.30E-02	2.67E-02	2.09E-02	2.17E-02	2.10E-02
U-235 (mg/L)	6.25E+00	4.02E+00	1.80E+00	1.47E+00	1.58E+00	1.50E+00
U-236 (mg/L)	5.71E-01	3.65E-01	1.53E-01	1.23E-01	1.28E-01	1.22E-01
Np-237(mg/L)	< 4.2E-03	< 4.3E-03	< 7.1E-03	< 6.1E-03	< 6.5E-03	< 5.3E-03
U-238 (mg/L)	5.62E+00	4.68E+00	6.74E+00	7.65E+00	6.59E+00	6.26E+00
Pu-239 (mg/L)	2.34E-02	2.13E-02	< 7.1E-03	< 6.1E-03	1.06E-02	7.35E-03
Pu-240 (mg/L)	< 4.2E-03	< 4.3E-03	< 7.1E-03	< 6.1E-03	< 6.5E-03	< 5.3E-03
Total U (mg/L)	1.25E+01	9.12E+00	8.72E+00	9.27E+00	8.32E+00	7.91E+00
U-235/U (wt%)	49.9%	44.0%	20.7%	15.9%	19.0%	19.0%

Table 3-20. Test Series D ICP-ES Results for LTAD, Heated Sodium Hydroxide Contact.

Analyte	120 h LTAD (mg/L)	%RSD	480 h LTAD (mg/L)	%RSD
Ag	< 2.2E-01	--	< 1.92E-01	--
Al	6.71E+03	0.7%	8.14E+03	3.7%
B	1.42E+01	0.2%	1.40E+01	4.2%
Ba	< 1.3E+00	--	< 1.2E+00	--
Be	< 6.2E-02	--	< 5.5E-02	--
Ca	< 2.6E+00	--	< 2.3E+00	--
Cd	< 2.6E-01	--	< 2.3E-01	--
Ce	< 2.4E+00	--	< 2.1E+00	--
Co	< 4.3E-01	--	< 3.8E-01	--
Cr	2.45E+02	1.8%	2.66E+02	0.3%
Cu	< 3.6E+00	--	< 3.2E+00	--
Fe	< 3.1E+00	--	< 2.7E+00	--
Gd	< 2.2E-01	--	< 1.9E-01	--
K	2.37E+02	1.4%	2.37E+02	4.0%
La	< 2.1E-01	--	< 1.9E-01	--
Li	< 1.1E+01	--	< 9.3E+00	--
Mg	< 1.2E+00	--	< 1.1E+00	--
Mn	< 3.5E-01	--	< 3.1E-01	--
Mo	1.10E+01	3.5%	1.07E+01	0.3%
Na	1.02E+05	0.2%	9.80E+04	4.8%
Ni	< 6.5E-01	--	< 5.8E-01	--
P	< 2.0E+01	--	< 1.7E+01	--
Pb	< 4.5E+00	--	< 4.0E+00	--
S	2.35E+03	0.4%	2.31E+03	5.2%
Sb	< 2.4E+00	--	< 2.1E+00	--
Si	< 6.7E+00	--	< 5.9E+00	--
Sn	< 9.2E+00	--	< 8.1E+00	--
Sr	< 1.0E-01	--	< 9.3E-02	--
Th	< 2.4E+00	--	< 2.1E+00	--
Ti	< 6.7E-01	--	< 5.9E-01	--
U	< 2.3E+01	--	< 2.0E+01	--
V	< 5.2E+00	--	< 4.6E+00	--
Zn	2.25E+00	16.9%	1.70E+00	16.2%
Zr	< 5.8E-01	--	< 5.2E-01	--

Table 3-21. Test Series D Anion Results for LTAD, Heated Sodium Hydroxide Contact.

Series	D	D		
Tube	009	012		
ABD sample (g)	1.517	1.529		
50 wt% NaOH (g)	1.151	1.178		
Neut. hold (h)	23	23		
Sludge (g)	13.432	13.463		
Sludge hold (h)	148	148		
Supernate decant (g)	4.012	4.921		
Contact fluid	LTAD	LTAD		
50 wt% NaOH (g)	2.013	1.800		
IW (g)	3.719	4.466		
Contact hold (h)	120	120		
LIMS #	LW34522	LW34523	Average	RSD
F^- (M)	<7.5E-02	<6.3E-02	<6.9E-02	
CHO_2^- (M)	<3.2E-02	<2.6E-02	<2.9E-02	--
Cl^- (M)	<4.0E-02	<3.4E-02	<3.7E-02	--
NO_2^- (M)	1.88E-01	1.79E-01	1.83E-01	3.7%
NO_3^- (M)	8.81E-01	8.94E-01	8.87E-01	1.0%
PO_4^{3-} (M)	<1.5E-02	<1.3E-02	<1.4E-02	--
SO_4^{2-} (M)	6.24E-02	5.91E-02	6.07E-02	--
$C_2O_4^{2-}$ (M)	<1.6E-02	<1.4E-02	<1.5E-02	--
Br^- (M)	<8.9E-02	<7.4E-02	<8.2E-02	--
CO_3^{2-} (M)	1.33E-01	1.29E-01	1.31E-01	2.5%
TOC (mg C/L)	1.02E+03	6.66E+02	8.43E+02	30%
Total Base (M)	2.75E+00	2.41E+00	2.58E+00	9.1%
OH^- (M)	2.16E+00	1.84E+00	2.00E+00	11.1%

3.2.5 Subsequent Sludge Mixture Decants (Series D)

Table 3-22 contains the tabulated data for the test conditions and actinide concentration results for the contact washing of the sludge with Tank 35 sludge supernate. Results are grouped in sets of two for the tests that were approximately duplicated. Concentrations and ^{235}U isotopic enrichment results were relatively consistent between duplicated tests. Other actinide masses not shown (230 and 241 through 244) have the same detection limit as U-233 and Pu-240. Table 3-23 contains supernate ICP-ES results for the contacts with Tank 35 supernate.

Table 3-22. Test Series D Results for Sludge Supernate Contact.

Series	D	D
Tube	019	020
ABD sample (g)	1.586	1.493
50 wt% NaOH (g)	1.232	1.18
Neut. hold (h)	24	24
Sludge (g)	14.117	13.37
Sludge hold (h)	120	120
Supernate decant (g)	5.769	3.634
Contact fluid	Tk35 sup.	T35 sup.
Sludge supernate (g)	7.517	6.312
Contact hold (h)	480	480
LIMS #	LW35170	LW35171
Th-232 (mg/L)	< 7.1E-03	< 8.3E-03
U-233 (mg/L)	< 7.1E-03	< 8.3E-03
U-234 (mg/L)	8.28E-02	7.38E-02
U-235 (mg/L)	4.55E+00	4.43E+00
U-236 (mg/L)	3.97E-01	3.69E-01
Np-237(mg/L)	< 7.1E-03	< 8.3E-03
U-238 (mg/L)	4.79E+00	5.06E+00
Pu-239 (mg/L)	5.75E-02	6.55E-02
Pu-240 (mg/L)	< 7.1E-03	< 8.3E-03
Total U (mg/L)	9.82E+00	9.93E+00
U-235/U (wt%)	46.4%	44.6%

Table 3-23. Test Series D ICP-ES Results for Sludge Supernate Contact.

Analyte	Average (mg/L)	%RSD	Analyte	Average (mg/L)	%RSD
Ag	< 2.5E-01	--	Mn	< 4.1E-01	--
Al	7.30E+03	1.4%	Mo	2.44E+01	1.9%
B	4.92E+01	3.8%	Na	1.18E+05	1.8%
Ba	< 1.6E+00	--	Ni	< 7.6E-01	--
Be	< 7.2E-02	--	P	1.29E+02	4.4%
Ca	< 3.0E+00	--	Pb	< 5.2E+00	--
Cd	< 3.0E-01	--	S	3.02E+03	1.7%
Ce	< 2.7E+00	--	Sb	< 2.7E+00	--
Co	< 5.0E-01	--	Si	< 7.8E+00	--
Cr	1.63E+02	1.4%	Sn	< 1.1E+01	--
Cu	< 4.2E+00	--	Sr	< 1.2E-01	--
Fe	< 3.6E+00	--	Th	< 2.8E+00	--
Gd	< 2.5E-01	--	Ti	< 7.8E-01	--
K	7.62E+02	6.9%	U	< 2.7E+01	--
La	< 2.5E-01	--	V	< 6.1E+00	--
Li	< 1.2E+01	--	Zn	3.98E+00	1.6%
Mg	< 1.4E+00	--	Zr	< 6.8E-01	--

3.2.6 Slurry Analysis (Series D)

Table 3-24 and Table 3-25 contain the results of the slurries remaining after the supernate was decanted after the final hold time of series D IW contact. The results are on a wet slurry basis and the total solids content was not quantified. The majority of the metals remained in the solid phase and are detectable in the final slurry analysis. Most metal components have a consistent Relative Standard Deviation (RSD) from the duplicate analyses because the majority of this RSD is likely introduced by the variance between supernate decanting from the two samples. Samples with more remaining liquid phase would have a consistently lower mass concentration of each analyte on a wet slurry basis. Components such as sodium and sulfur, which are primarily in the liquid phase in the slurry, have much lower RSD values. Isotopic enrichment of ^{235}U was consistently 4.0 wt%, confirming that our blend between ABD material and CSTF sludge material was performed with consistency. The RSD of the isotopic enrichment is also low because it is not a function of the portion of liquid decanted after testing.

Table 3-24. ICP-ES Analyses of Aqua Regia Digestion of Slurry

Analyte	Tube 001/003		Tube 005/006		Tube 010/011	
	Average (mg/kg)	%RSD	Average (mg/kg)	%RSD	Average (mg/kg)	%RSD
Ag	< 4.8E+01	--	< 4.8E+01	--	< 4.8E+01	--
Al	1.51E+04	2.3%	1.73E+04	19%	1.71E+04	9.5%
B	< 2.5E+01	--	< 2.5E+01	--	< 2.5E+01	--
Ba	7.56E+01	9.3%	8.85E+01	23%	7.76E+01	13%
Be	< 5.9E-01	--	< 5.9E-01	--	< 6.0E-01	--
Ca	1.42E+03	16%	1.59E+03	17%	1.64E+03	9.9%
Cd	< 1.1E+01	--	< 1.1E+01	--	< 1.1E+01	--
Ce	1.48E+02	7.2%	1.74E+02	20%	1.51E+02	12%
Co	< 1.3E+01	--	< 1.3E+01	--	< 1.3E+01	--
Cr	5.10E+02	8.2%	5.66E+02	17%	5.51E+02	10%
Cu	< 1.1E+02	--	< 1.1E+02	--	< 1.1E+02	--
Fe	1.65E+04	7.7%	1.95E+04	21%	1.68E+04	13%
Gd	8.10E+01	8.8%	9.46E+01	22%	8.34E+01	12%
K	< 3.5E+02	--	< 3.5E+02	--	< 3.5E+02	--
La	5.16E+01	8.1%	6.05E+01	24%	5.11E+01	8.6%
Li	< 4.0E+01	--	< 4.0E+01	--	< 4.0E+01	--
Mg	5.53E+02	11%	6.40E+02	20%	6.27E+02	13%
Mn	3.90E+03	7.6%	4.55E+03	21%	3.98E+03	13%
Mo	< 2.6E+01	--	< 2.6E+01	--	< 2.6E+01	--
Na	4.23E+04	0.3%	4.22E+04	--	5.27E+04	0.0%
Ni	4.79E+02	8.7%	5.54E+02	22%	4.84E+02	12%
P	< 2.4E+02	--	< 2.4E+02	--	< 2.4E+02	--
Pb	< 7.0E+01	--	< 6.9E+01	--	< 7.0E+01	--
S	1.49E+03	4.7%	1.50E+03	0.5%	2.03E+03	1.0%
Sb	< 5.5E+01	--	< 5.4E+01	--	< 5.5E+01	--
Si	5.92E+02	5.6%	6.76E+02	16%	6.12E+02	11%
Sn	< 2.6E+02	--	< 2.6E+02	--	< 2.6E+02	--
Sr	2.99E+01	11%	3.48E+01	23%	3.13E+01	14%
Th	2.26E+03	5.6%	2.63E+03	22%	2.29E+03	15%
Ti	< 3.9E+01	--	< 3.9E+01	--	< 3.9E+01	--
U	6.54E+03	9.9%	7.57E+03	21%	6.61E+03	13%
V	< 7.2E+00	--	< 7.1E+00	--	< 7.2E+00	--
Zn	< 5.9E+01	--	< 5.9E+01	--	< 5.9E+01	--
Zr	1.50E+02	7.5%	1.75E+02	21%	1.53E+02	13%

Table 3-25. ICP-MS Analyses of Aqua Regia Digestion of Slurry

Analyte	Tube 001/003		Tube 005/006		Tube 010/011	
	Average (mg/kg)	%RSD	Average (mg/kg)	%RSD	Average (mg/kg)	%RSD
Th-230	< 4.4E-02	--	< 4.4E-02	--	< 4.4E-02	--
Th-232	2.09E+03	4.1%	2.44E+03	22%	2.14E+03	15%
U-233	7.07E-01	6.5%	8.09E-01	22%	7.11E-01	13%
U-234	3.79E+00	7.1%	4.36E+00	17%	3.94E+00	9.7%
U-235	2.63E+02	5.9%	3.06E+02	17%	2.73E+02	8.8%
U-236	2.14E+01	7.0%	2.44E+01	17%	2.20E+01	8.7%
Np-237	2.26E+00	6.9%	2.56E+00	20%	2.28E+00	12%
U-238	6.36E+03	7.8%	7.41E+03	21%	6.54E+03	13%
Pu-239	2.75E+01	6.2%	3.15E+01	21%	2.81E+01	13%
Pu-240	2.15E+00	6.6%	2.47E+00	20%	2.17E+00	13%
mass 241	6.39E-01	7.0%	7.26E-01	20%	6.49E-01	13%
mass 242	1.53E-01	4.6%	1.72E-01	25%	1.49E-01	14%
mass 243	≤ 4.5E-02	--	≤ 4.8E-02	--	≤ 4.5E-02	--
mass 244	< 4.4E-02	--	< 4.4E-02	--	< 4.4E-02	--
Total U	6.65E+03	7.7%	7.75E+03	21%	6.83E+03	13%
U-235/U	3.96 wt%	1.8%	3.96 wt%	3.4%	4.01 wt%	4.3%

3.2.7 Decanted Solution Blending in Salt Batches (Series E)

Table 3-26 and Table 3-27 contain the tabulated data for the test conditions and actinide concentration results for the mixtures of materials decanted from the SB preparation tank with the StB material at two different mixture ratios. Tests were not duplicated for this test series, but the lack of change with time in the mixtures represent duplicated or triplicated measurements. Concentrations and ^{235}U isotopic enrichment results were relatively consistent for the samples prepared from a test at different mixture hold times. Other actinide masses not shown (230 and 241 through 244) have the same detection limit as U-233 and Pu-240. Table 3-28 and Table 3-29 contain ICP-ES, anion, and density data for representative mixtures at each of the two ratios.

Table 3-26. Series E Results for 20:80 Mass Ratio of Sludge Batch Preparation Tank Decant to Salt Batch Material.

Series	E	E	E	E	E	E
Tube	013	013	013	016	016	017
Decant:Salt Ratio	20:80	20:80	20:80	20:80	20:80	20:80
Decant Source Series	C	C	C	C	C	D (IW)
Decant Source Tube	009	009	009	011	011	005
Decant (g)	1.609	1.609	1.609	1.593	1.593	2.025
Salt Batch (g)	6.410	6.410	6.410	6.374	6.374	8.081
Mixture hold (hr)	0	120	480	120	480	480
LIMS #	LW34508	LW34855	LW35154	LW34858	LW35163	LW35168
Th-232 (mg/L)	< 8.9E-03	< 5.6E-03	< 4.0E-03	< 5.4E-03	< 4.2E-03	< 5.3E-03
U-233 (mg/L)	< 8.9E-03	< 5.6E-03	< 4.0E-03	< 5.4E-03	< 4.2E-03	< 5.3E-03
U-234 (mg/L)	2.97E-02	2.83E-02	3.00E-02	2.66E-02	2.99E-02	1.35E-02
U-235 (mg/L)	1.70E+00	1.66E+00	1.98E+00	1.59E+00	1.93E+00	7.65E-01
U-236 (mg/L)	1.58E-01	1.49E-01	1.64E-01	1.45E-01	1.63E-01	6.48E-02
Np-237(mg/L)	< 8.9E-03	6.58E-03	7.12E-03	6.50E-03	7.69E-03	6.60E-03
U-238 (mg/L)	6.11E+00	5.81E+00	6.29E+00	5.77E+00	6.53E+00	5.99E+00
Pu-239 (mg/L)	< 8.9E-03	6.35E-03	6.21E-03	5.99E-03	3.30E-02	3.49E-02
Pu-240 (mg/L)	< 8.9E-03	< 5.6E-03	< 4.0E-03	< 5.4E-03	< 4.2E-03	< 5.3E-03
Total U (mg/L)	8.00E+00	7.64E+00	8.47E+00	7.53E+00	8.65E+00	6.84E+00
U-235/U (wt%)	21.2%	21.7%	23.4%	21.1%	22.3%	11.2%

Table 3-27. Series E Results for 60:40 Mass Ratio of Sludge Batch Preparation Tank Decant to Salt Batch Material.

Series	E	E	E	E	E	E
Tube	014	014	014	015	015	018
Decant:Salt Ratio	60:40	60:40	60:40	60:40	60:40	60:40
Decant Source Series	C	C	C	C	C	D (IW)
Decant Source Tube	012	012	012	010	010	006
Decant (g)	4.281	4.281	4.281	3.600	3.600	5.388
Salt Batch (g)	2.839	2.839	2.839	2.401	2.401	3.593
Mixture hold (hr)	0	120	480	120	480	480
LIMS #	LW34509	LW34856	LW35155	LW34857	LW35162	LW35169
Th-232 (mg/L)	< 8.3E-03	< 5.8E-03	< 3.7E-03	< 6.0E-03	< 4.2E-03	< 6.5E-03
U-233 (mg/L)	< 8.3E-03	< 5.8E-03	< 3.7E-03	< 6.0E-03	< 4.2E-03	< 6.5E-03
U-234 (mg/L)	7.30E-02	6.24E-02	6.72E-02	6.45E-02	6.72E-02	2.71E-02
U-235 (mg/L)	4.63E+00	4.05E+00	4.85E+00	4.26E+00	4.74E+00	1.86E+00
U-236 (mg/L)	4.17E-01	3.59E-01	3.95E-01	3.73E-01	3.99E-01	1.58E-01
Np-237(mg/L)	< 8.3E-03	< 5.8E-03	5.04E-03	< 6.0E-03	4.73E-03	< 6.5E-03
U-238 (mg/L)	7.83E+00	6.73E+00	7.36E+00	6.95E+00	7.37E+00	6.79E+00
Pu-239 (mg/L)	8.74E-03	6.35E-03	7.50E-03	6.25E-03	3.56E-02	4.16E-02
Pu-240 (mg/L)	< 8.3E-03	< 5.8E-03	< 3.7E-03	< 6.0E-03	< 4.2E-03	< 6.5E-03
Total U (mg/L)	1.29E+01	1.12E+01	1.27E+01	1.16E+01	1.26E+01	8.84E+00
U-235/U (wt%)	35.7%	36.1%	38.3%	36.6%	37.7%	21.1%

Table 3-28. Series E ICP-ES Results for 20:80 and 60:40 Blends

Analyte	20:80 (mg/L)	60:40 (mg/L)	Analyte	20:80 (mg/L)	60:40 (mg/L)
Ag	< 2.6E-01	< 2.4E-01	Mn	< 4.3E-01	< 3.9E-01
Al	8.61E+03	7.56E+03	Mo	4.87E+01	3.22E+01
B	7.57E+01	4.94E+01	Na	1.37E+05	1.13E+05
Ba	< 8.1E-01	< 7.4E-01	Ni	< 8.0E-01	< 7.2E-01
Be	< 7.6E-02	< 6.9E-02	P	1.47E+02	9.76E+01
Ca	< 1.6E+00	< 1.5E+00	Pb	< 5.4E+00	< 5.0E+00
Cd	< 3.1E-01	< 2.8E-01	S	1.67E+03	2.73E+03
Ce	< 2.9E+00	< 2.6E+00	Sb	< 2.9E+00	< 2.6E+00
Co	< 5.3E-01	< 4.8E-01	Si	2.10E+01	1.30E+01
Cr	1.21E+02	1.53E+02	Sn	< 1.1E+01	< 1.0E+01
Cu	< 2.2E+00	< 2.0E+00	Sr	< 1.3E-01	< 1.2E-01
Fe	4.49E+00	2.85E+00	Th	< 2.9E+00	< 2.7E+00
Gd	< 2.7E-01	< 2.4E-01	Ti	< 8.1E-01	< 7.4E-01
K	9.98E+02	6.87E+02	U	< 1.4E+01	< 1.3E+01
La	< 2.6E-01	< 2.3E-01	V	< 6.4E+00	< 5.8E+00
Li	< 1.3E+01	< 1.2E+01	Zn	5.22E+00	3.55E+00
Mg	< 1.5E+00	< 1.4E+00	Zr	< 3.5E-01	< 3.2E-01

Table 3-29. Anion, TOC, and Total Base Results for Select Series E Tests

Series	E	E
Tube	013	014
Decant:Salt Ratio	20:80	60:40
Decant Source Series	C	C
Decant Source Tube	009	012
Decant (g)	1.609	4.281
Salt Batch (g)	6.410	2.839
Mixture hold (hr)	480	480
Density (g/mL)	1.291	1.242
LIMS #	LW34526	LW34527
F^- (M)	<5.5E-02	<7.0E-02
CHO_2^- (M)	<2.3E-02	<3.0E-02
Cl^- (M)	<3.0E-02	<3.8E-02
NO_2^- (M)	6.29E-01	4.56E-01
NO_3^- (M)	1.51E+00	1.40E+00
PO_4^{3-} (M)	<1.1E-02	<1.4E-02
SO_4^{2-} (M)	4.25E-02	7.05E-02
$C_2O_4^{2-}$ (M)	<1.2E-02	<1.5E-02
Br^- (M)	<6.6E-02	<8.4E-02
CO_3^{2-} (M)	2.78E-01	2.39E-01
TOC (mg C/L)	9.63E+02	5.36E+02
Total Base (M)	3.52E+00	2.61E+00
OH^- (M)	2.63E+00	1.60E+00

3.3 Discussion of Impacts of ABD on Tank Farm Supernate ^{235}U Enrichment

Decants from the SB preparation tank during SB compilation and washing may be sent to various other points in the tank farm. For example, the decanted material is commonly sent to the 242-25H evaporator system for space management. If the decanted material is dilute enough in sodium content, it could be utilized for saltcake dissolution. The decanted material can also be combined with other salt supernate material as part of a StB feed to SWPF. In any of these cases, the uranium in the supernate decanted during SB preparation has the potential for being included in the feed to SWPF through blending in a StB blend tank. Thus, the uranium in this decanted material has the potential to influence the uranium enrichment in the StB blend tank and the feed to SWPF.

As communicated during test planning, the feed to the SWPF was limited to a $^{235}U_{eq}$ enrichment of 8 wt%.[1] However, changes are expected in SWPF to allow for the addition of lower quantities of monosodium titanate (MST) and allow for more batches to be filtered during SWPF processing. These changes may

result in the feed to SWPF being limited to a $^{235}U_{eq}$ enrichment of 5.5 wt% or 4 wt%, depending on the quantity of MST used.[3]

Based on the testing results, an estimate to determine what enrichment of ABD/DU mixture (pre-neutralization) should be targeted to meet enrichment limits in a StB was developed. Because unverified assumptions were needed in doing this evaluation, it shall not be considered a calculation supporting a safety class functional classification. This evaluation should be used as an estimate for a single set of inputs and a guide to a potential methodology.

1. The evaluation is based on a 20:80 volume ratio of new decanted supernate transferred to the previously existing StB material. Testing also included the 60:40 volume ratio. Increasing the volume ratio of decanted supernate to StB material is not conservative (it will require lower enrichment in the H-Canyon material to meet the same StB enrichment limit).
2. The StB feed used in this evaluation (prior to the ABD contribution) is based on our testing that had a supernate ^{235}U enrichment of 1.8 wt% and supernate U concentration of 6.2 mg/L. Not all StB feeds will have this same ^{235}U enrichment or U concentration. Increasing the supernate ^{235}U enrichment or decreasing the U concentration in the StB material (prior to the ABD contribution) is not conservative.
3. The SB feed material used in this evaluation had a supernate ^{235}U enrichment of 0.34 wt%, a supernate U concentration of 11.6 mg/L, and a slurry ^{235}U enrichment of 0.69 wt%. Not all SBs will have as low of a supernate U enrichment or as high of a supernate U concentration, especially as ABD batches are processed and leave heels in the SB preparation tank. Increasing the ^{235}U enrichment or decreasing the supernate U concentration in the SB material (prior to additional ABD additions to the SB being prepared) is not conservative.
4. The evaluation used testing data from neutralized ABD material that was held for approximately one day. This is not necessarily conservative because time between neutralization and contact with SB material is usually less than 1 day.
5. This methodology assumes that the enriched uranium ABD material and the DU material are mixed as soluble acidic streams prior to neutralization and transfer to the CSTF. If other methods are utilized, such as neutralizing and mixing the streams separately in H-Canyon or sending neutralized streams separately to CSTF, the evaluation would need to consider the relative volumes and sequencing of the streams and would not necessarily follow with the same estimation method.
6. The evaluation assumed that the exchange between the liquid and solid phases of uranium in the SB preparation tank is not a function of the ABD stream enrichment (including the chemistry changes in the ABD stream caused by blending DU with the dissolved SNF).

There are at least two aspects of the evaluation or the data that the evaluation uses that are conservative, which may help to offset some of the non-conservativisms.

1. The initial decant from the SB preparation tank after ABD additions is the most conservative case. Subsequent decants from the SB prep tank (for example, during sludge washing) will have a greater ease of being able to be added to a StB at a 20:80 volume ratio. This trend is expected to continue with further subsequent washes being easier to accommodate for uranium enrichment than the prior SB washes.
2. The evaluation was based on the data that used a 5 day hold time in the SB preparation tank. Longer hold times lead to progressively lower supernate ^{235}U enrichment and U concentration in the decanted material, which in turn would allow for slightly higher enrichments from H-Canyon to meet the StB limit.

The following methodology was used to calculate the slurry enrichment needed in the H-Canyon transfer to CSTF to meet the enrichment limit in the StB. The case considered was transferring the SB preparation tank initial decant material (supernate from series C tests) to the StB at a volume ratio of 20:80 SB decant to StB material. Table 3-30 contains the data used and the results of this estimate. The analytical results

used were based on the average of tubes 009 and 011 from the series C tests shown in Table 3-10 and the average of the five results for tubes 013 and 016 from the series E tests shown in Table 3-26.

Table 3-30. Use of Test Results to Estimate ABD Enrichment Needed to Meet the Target Uranium Enrichment in the Salt Batch Preparation Tank (with 20:80 Volume Ratio of Sludge Batch Decant to Other Salt Batch Material)

supernate mass balance of test	SB Prep Tank		Feeds to StB		20:80 StB Blend	
	ABD	Tk51/26 slurry	initial decant	salt solution	mass balance	test result
U (mg/L)	116*	11.6	15.6	6.2	8.2	8.1
U-235/U (wt%)	73.3%	0.34%	46.5%	1.8%	19.8%	21.9%
estimate of ABD enrichment needed to meet StB target	SB Prep Tank		Feeds to StB		20:80 StB Blend	
	ABD + DU	Tk51/26 slurry	initial decant	salt solution	adjusted target	target
U-235/U (wt%)	23.9%	0.34%	15.2%	1.8%	7.2%	8.0%
U-235/U (wt%)	9.7%	0.34%	6.2%	1.8%	3.6%	4.0%

* 116 mg/L is an effective (non-physical) concentration determined for a supernate mass balance. From the series A tests, the expected actual concentration is ~40 mg/L

The top portion of Table 3-30 contains a mass balance for the series E test that was performed and the bottom portion contains a mass balance for the impact of different starting uranium enrichments of the ABD material based on the same interaction with sludge and salt samples.

For both the supernate mass balance for the test and the estimate of the ABD/DU blend enrichment needed to meet the StB target enrichment, the following four mass balance equations are used:[d]

- The mass of total uranium in the SB initial decant is equal to the mass of total uranium in the supernate of the Tank 51/26 slurry plus the mass of total uranium in the supernate of the ABD (or ABD+DU) mixture.
- The mass of U-235 in the SB initial decant is equal to the mass of U-235 in the supernate of the Tank 51/26 slurry plus the mass of U-235 in the supernate of the ABD (or ABD+DU) mixture.
- The mass of total uranium in the StB blend tank supernate is equal to the mass of total uranium added as the SB initial decant (supernate) plus the mass of total uranium in the supernate of the other salt making up the StB.
- The mass of U-235 in the StB blend tank supernate is equal to the mass of U-235 added as the SB initial decant (supernate) plus the mass of U-235 in the supernate of the other salt making up the StB.

[d] This set of expressions is based on only the supernate (ignoring the interchange of uranium with the solids) and thus is not strictly valid beyond use in this estimate. The supernate mass balance expressions can be used for this estimate with the listed assumption #6 (i.e., the interchange of uranium with the solids for ABD and DU mixtures would be consistent with that of the tests performed with ABD without DU).

For the mass balance for the test based on the SB preparation tank, the input concentration of soluble uranium in the ABD mixture is approximately 40 mg/L based on the 24-hour neutralization testing information in Figure 3-1. However, the measured results for the initial decant supernate do not match the mass balance inputs (the supernate values for the ABD material and the Tank 51 and 26 sludge slurry material). The uranium from the ABD material is continuing to precipitate in the SB preparation tank and the isotopes in both the supernate and slurry would tend to exchange causing the isotopic enrichment in both phases to approach each other with time. For these reasons, a mass balance only looking at the supernate phase would not be expected to close. An effective uranium concentration in the neutralized ABD supernate of 116 mg/L would close the supernate mass balance in the SB preparation tank (holding all the other ABD, Tank 51/26 slurry, and initial decant values constant). While this value for effective supernate uranium concentration of 116 mg/L does not hold physical significance, it is needed to perform the estimate in the bottom half of the table. The mass balance on the feeds to the StB worked much better, with the mass balance result of StB blend supernate uranium of 8.2 mg/L with ^{235}U enrichment 19.8 wt% matching relatively well with the measured series E test result supernate uranium of 8.1 mg/L with ^{235}U enrichment 21.9 wt%. The ^{235}U enrichment mass balance expected result in the StB blend was 10% lower than the measured result.

For the estimation of ABD enrichment to meet the StB limit, it is best to read the bottom portion of Table 3-30 from right to left. The two limits considered for the StB blend were 8 wt% and 4 wt% ^{235}U enrichment. First, considering that the mass balance underpredicted the test results by 10%, the target ^{235}U enrichment for performing the mass balance for the estimate calculation was lowered by 10% (7.2 wt% and 3.6 wt%). For 20:80 mass ratio blend of initial decant to other salt material and using the same uranium concentrations and salt solution enrichment as the test results, the initial decants from the SB would need to be 15.2 wt% and 6.2 wt% to meet the two StB limits of 8 wt% and 4 wt%, respectively. The effective uranium concentration of the neutralized ABD material and the same uranium concentrations and Tank 51/26 slurry enrichment as the test results were assumed. For the same sludge and salt feeds used in this testing, DU additions targeting 24 wt% and 9.7 wt% enrichment in the pre-neutralized H-Canyon stream should result in StB blends at or below the 8 wt% and 4 wt% enrichment limits.

Similar estimates can be formulated for a range of enrichments in the SB and StB materials for which the pH adjusted ABD material will be added. Any of these estimates have their limitations, in that the chemical and physical aspects of the future CSTF conditions would need to match those of the testing. The estimates should not be used as a preventive or mitigative control on the SWPF WAC in lieu of validation sampling of the StB.

3.4 Possible Supernate Enrichment Remediation Flowsheets

The supernate uranium isotopic enrichment results from this study are consistent with the previous results that indicated recently precipitated enriched uranium streams can cause the supernate uranium enrichment to deviate higher than the uranium enrichment in the slurry. For this reason, enrichment limitations for StB processing will likely require DU addition above and beyond what is required to lower ABD material enrichment for the SB to meet DWPF processing requirements. To minimize DU additions and impacts to the various facilities, consideration should be given to the overall flowsheet and the optimal location and method of DU addition.

The test described in Section 3.1.3 was testing a hypothesis that adding acidic DU to an already pH adjusted ABD material slurry would produce a mixture with supernate enrichment lower than the slurry enrichment and thus be an efficient use of the DU. Unexpectedly, that specific test led to supernate enrichments higher than the slurry enrichment, which is even a less efficient use of DU than blending the DU and ABD material prior to pH adjustment. The reason for this observation was likely the much higher uranium concentration in the DU material led to a relatively small volume addition of the DU material. From this observation, it

follows that, if neutralized separately from ABD material, the addition of a diluted DU stream at lower uranium concentration and higher overall volume may be a more efficient use of DU. However, performing lower concentration DU additions within H-Canyon would likely encounter space and volume limitations.

CSTF used a process previously that targeted lowering the uranium isotopic enrichment in the supernate by adding only a fraction of the DU required to lower the enrichment of the overall slurry. During 2H evaporator restart in 2001, 21,000 gallons of depleted uranyl carbonate solution was added to Tank 43 to isotopically dilute the supernate. Although scant, documented information on this process is available from planning calculations,[21,22] laboratory testing results[23], and field results.[24,25] F-Canyon prepared the uranyl carbonate solution with 10 g/L of DU and Flygt mixers were used to mix the Tank 43 supernate without disturbing the settled sludge layer. Although it is not the only option, a similar process could be used in a SB preparation tank or StB blend tank to specifically target reducing the uranium enrichment in the liquid phase while minimizing the use of DU.

4.0 Conclusions

Testing showed that the uranium concentration in the supernate of the recently precipitated ABD material being added to the SB is approximately 40 to 50 mg/L, which is higher than the typical uranium concentration in the supernate of the SB preparation tank. Thus, the enrichment of the recently precipitated stream may contribute to the enrichment in the supernate phase diverging from the enrichment of the slurry. Evidence from non-prototypic DU addition testing suggests that relatively concentrated DU solutions (approximately 200 to 400 g/L uranium), when neutralized separately, may not lower the uranium enrichment of the supernate phase as efficiently as they lower the uranium enrichment of the slurry.

Testing confirmed the previous laboratory observations of supernate enrichment deviation from slurry enrichment, but now under a more controlled and parametric set of test conditions. In all cases, the supernate resulting from mixing the pH adjusted ABD material and the existing sludge material had a ^{235}U isotopic enrichment that deviated above the slurry mixture ^{235}U isotopic enrichment of 4.0 wt%. The initial supernate decant from a SB had a ^{235}U isotopic enrichment of typically 45 wt% to 47 wt% with a maximum of 60 wt% and a minimum of 38 wt%. After decanting the initial supernate and contacting the material with wash water for 20 days, the supernate ^{235}U isotopic enrichment ranged from 22 wt% to 27 wt%. The supernate ^{235}U isotopic enrichment appears to decrease (in the direction converging toward the slurry enrichment) at cases with longer hold times, regardless of whether the hold time is during the neutralization step, the initial sludge mix step, or the subsequent IW wash step.

For the same sludge and salt feeds used in this testing, it is estimated that depleted uranium additions targeting 24 wt% and 9.7 wt% enrichment in the pre-neutralized H-Canyon stream should result in StB blends at or below the 8 wt% and 4 wt% enrichment limits in the minimum assumed time frame.

5.0 Recommendations

The following recommendations follow from this study.

The most efficient addition of DU for control and/or remediation of supernate enrichment should be investigated and optimized. If DU is to be used efficiently, it is difficult to control the uranium enrichment in a StB by placing the control on H-Canyon transfers into a SB. To minimize DU additions and impacts to the various facilities, consideration should be given to the overall flowsheet and the optimal location and method of DU addition. Depending on the DU addition flowsheet, additional testing may be warranted.

Where possible, routine supernate and non-routine slurry samples collected during SB preparation and washing should be analyzed to gather more information on the in-tank soluble uranium behavior with respect to supernate enrichment deviating from slurry enrichment after addition of recently precipitated

enriched uranium material. Due to limitations of the testing described in this report, actual ABD pH adjustment and blending in the CSTF may show a divergence between the supernate and slurry uranium enrichment to a different extent.

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