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Evaluation of Proposed Flowsheet Changes for the Highly Enriched Uranium Blenddown Program

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Abstract: At the Savannah River Site, highly enriched uranium (HEU) is dissolved, purified, and blended with natural uranium to make low enriched uranium solutions sufficiently pure for conversion to power reactor fuel. The process to dissolve and purify aluminum-clad HEU fuel at SRS is well-established. However, for the dissolution and recovery of metal scrap, flowsheet changes were proposed. This study evaluates the proposed changes. Specifically, solvent extraction modeling calculations were performed which indicated that one solvent extraction cycle would be sufficient to purify the metal scrap solution by removing boron, which is added as a neutron poison. In addition, stability constants from the literature and Savannah River National Laboratory corrosion studies were documented to demonstrate that boron complexation of fluoride in nitric acid solutions, at the levels anticipated, is sufficient to prevent excessive corrosion in stainless steel vessels. Downstream from the purification process, limitations on the boron concentration in waste evaporators were recommended to prevent formation of boron-containing solids.

Keywords: Boron; corrosion; fluoride complexation; solvent extraction; uranium

INTRODUCTION

The HEU Blenddown campaign began at the Department of Energy's Savannah River Site (SRS) in 2003 with the dissolution of HEU fuel

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tubes. The resulting HEU solution is purified by two cycles of solvent extraction with tri-butyl phosphate (TBP) as the extractant. The purified HEU solution is blended with natural uranium to achieve a 4.95% enriched product solution that is converted to fuel and burned in a Tennessee Valley Authority (TVA) reactor to produce electricity. A large portion of the HEU designated for blenddown by the Department of Energy (DOE) was aluminum-clad fuel. As the fuel portion of the campaign approached completion, SRS prepared for flowsheet changes to dissolve metal scrap, which is often referred to as plutonium-contaminated scrap (PuCS). This paper describes the evaluation performed prior to implementing the needed flowsheet changes.

PROPOSED FLOWSHEET CHANGE

The flowsheet for uranium fuel involves dissolution in nitric acid with a mercuric nitrate catalyst to promote aluminum dissolution. The resulting solution is centrifuged then purified via two cycles of solvent extraction (i.e., First and Second U Cycle). The flowsheet for metal scrap involves dissolution in nitric acid/potassium fluoride (KF) with B as a neutron poison. Initially, dissolved metal scrap was blended with dissolved uranium fuel and processed in the same way. The proposed flowsheet change involved not blending the dissolved metal, but rather placing it in a U storage tank and purifying it via one cycle of solvent extraction (Second U Cycle). Figure 1 is a schematic of the flowsheet showing the proposed changes. Table 1 lists key characteristics of the dissolved PuCS solution and the current U storage tank solution.

As shown in Table 1, the PuCS solution contains two species, B and F^-, which are currently at trace levels in the U storage tank. If PuCS solution is re-routed to the U storage tank, the concentrations of both B and F^- in the tank would steadily increase throughout the campaign. Periodic removals from the U storage tank during the PuCS campaign would remove relatively pure solution, thus yielding higher B and F^- concentrations in the U tank as the PuCS campaign proceeded.

SCOPE OF STUDY

Prior to implementation of the proposed changes, an evaluation was needed to determine if uranium product purity could be maintained with only one solvent extraction cycle. Specifically, the Second U Cycle alone would need to provide sufficient decontamination of B, F^-, and Pu to meet TVA Blend Grade HEU specifications. Solvent extraction modeling calculations were used to address the decontamination question. Because

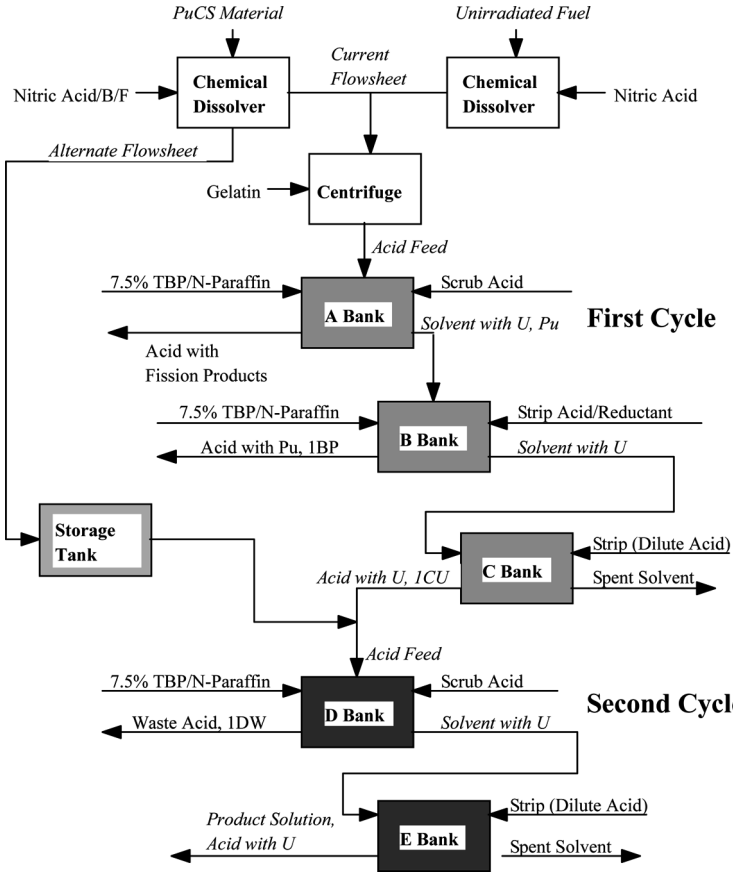

Figure 1. Flowsheet for U purification (1) with proposed changes.

Table 1. Key Characteristics of PuCS Solution and Current U Storage Tank Solution

PuCS Solution	U Storage Tank Contents
1,800 gallons per dissolution	80,000 gallons
2.5 g U/L	8.4 g U/L
3.5 M nitric acid	0.7 M nitric acid
0.03 M F^-	trace
2.5 g B/L	trace
1.0×10^4 Pu dpm/mL	1.0×10^4 Pu dpm/mL

Al, which complexes F^-, was not in the PuCS feed material, and B was planned for use as a neutron poison, this study evaluated the extent to which B complexes F^-, using both literature sources and empirical work. In addition, measured corrosion rates are reported for nitric acid/F^- solutions with different levels of Al and/or B. Finally, this study evaluated general processing concerns that may arise due to the proposed flowsheet changes, including the presence of insoluble solids in the feed material, and downstream B solubility issues.

DECONTAMINATION

Boron Decontamination

Initial processing experience at SRS with blended PuCS/fuel solution demonstrated that most B was rejected by the A Bank in First U Cycle and that the resulting B concentration in the 1BP waste stream was equivalent to 60μg/g U, which is two times the TVA Blend Grade specification. The First Cycle product stream, 1CU, contained <7μg B/g U, which was well below the 30μg/g U specification for B. Stated another way, the decontamination factor[i] (DF) for B in A Bank was slightly more than 1000 and the combined First Cycle DF was over 20,000 and may have been higher, because the B level in the product was below detection limits. These DF values represent the combined effects of the distribution coefficient, number of stages, relative organic/aqueous flow ratio, stage efficiency, entrainment, and interactions with other chemicals in the process. Since so many factors affect DF, it is difficult to predict if the B DF in Second U Cycle would be better or worse than that of First U Cycle.

Distribution coefficients[ii] for hydrogen fluoride (HF) and boric acid (H_3BO_3) between nitric acid solutions and TBP are shown in Table 2. The distribution coefficients for B (and HF) in 7.5 vol% TBP, as used for HEU Blenddown, would be significantly less than in 30 vol% TBP or pure TBP because the diluent material, n-paraffin, which makes up the remaining 92.5 vol% of the solvent, does not extract either B or HF. Hence, a high degree of B decontamination would be expected in First or Second U Cycle. Based on the distribution coefficients in Table 2, it appears that stage efficiency and/or entrainment are the main factors limiting the B DF in A Bank to 1000.

[i]Decontamination factor or DF = Volumetric feed concentration/volumetric product concentration.

[ii]Distribution coefficient or Do/a = Concentration in organic phase/concentration in aqueous phase at equilibrium.

Table 2. Distribution coefficients for HF and H_3BO_3 between nitric acid solutions and TBP (2)

	Distribution coefficient, Do/a	
Nitric acid Mol/L	30 vol % TBP HF	100% TBP H_3BO_3
0.5	0.4	
1	0.3	0.15
2	0.2	0.08
3	0.15	0.035
4	0.1	0.007
5	0.05	
6	0.03	

Using the distribution coefficients in Table 2, and nitric acid distribution coefficients from Thompson et al. (3), calculations were performed using a SASSE model (a Spreadsheet Algorithm for Stagewise Solvent Extraction) (4). Decontamination factors were calculated for typical A Bank and D Bank stage separation efficiencies and organic in aqueous and aqueous in organic entrainments. Stage efficiencies of 50% and 70% and entrained volume fractions of 0.02 and 0.05 (for both the organic and aqueous phases) were selected as representative values for A and D Bank operation. SASSE calculation results for selected cases are shown in Table 3. In all cases, the DF's for D Bank exceed those for A Bank. In particular, the DF for B is about three times greater in D Bank at a stage efficiency of 0.5 and entrained volume fractions of 0.05, which are the same parameters that yield an A Bank DF close to the observed value of 1000. Parametric calculations indicate that the improved separation efficiency in D Bank may be attributed to the relatively higher D Bank aqueous scrub flow rate.

Table 3. Calculated DF's for A Bank and D Bank

Stage efficiency	Entrained fraction	A Bank DF		D Bank DF	
		Boron	Fluoride	Boron	Fluoride
0.5	0.02	3400	11	6300	34
0.5	0.05	777	7.5	2200	28
0.7	0.02	58,000	39	60,000	67
0.7	0.05	1600	18	7100	47

Table 3 compares A and D Banks assuming that stage efficiency and entrainment levels are the same in both banks. Though efficiency and entrainment are likely to be similar in the two banks, the actual levels have not been determined. Therefore, it is reasonable to consider a few estimates of the B DF that may be achieved by Second U Cycle. These are shown in Table 4, along with the expected point at which a second solvent extraction pass would be required for the solution in the U storage tank. The obvious consequence of a second pass is an increased processing burden that would slow production significantly. For traditional mixer-settler performance, a stage efficiency of 70% and a volume entrainment fraction of 0.02 are often assumed. If these assumed values are correct, the PuCS solution would never reach a point where a second pass is needed. The PuCS solution starts with a 1 : 1 U : B ratio. A D Bank DF of 60,000 would provide sufficient purification for a feed concentration of 1.8 g B/g U. Therefore, B contamination is not expected to be a problem during the PuCS campaign after flowsheet changes are made.

Fluoride and Plutonium Decontamination

Recent processing experience at SRS has shown that a single pass of the current U storage tank solution through Second U Cycle provides sufficient Pu decontamination to meet the TVA Blend Grade HEU specification for Pu of 400 μg/g U. Since the Pu concentration of PuCS solution is similar to the concentration of the current U storage tank solution, Second U Cycle should provide sufficient Pu decontamination regardless of the amount of PuCS solution sent to the U storage tank. However, purification by Second U Cycle may become more difficult if PuCS solution picked up additional Pu or other isotopes, such as neptunium-237, during transfer. Therefore, flushing of transfer lines prior to use in the proposed PuCS flowsheet change would minimize undesired contamination of U product solution.

Table 4. Effect of B decontamination factor on need for second pass of solvent extraction

Assumed B DF in Second U Cycle	B concentration in tank that would require a second pass
1000	$3 \times 10^4 \mu$g B/g U
2000	$6 \times 10^4 \mu$g B/g U
5000	$1.5 \times 10^5 \mu$g B/g U
60,000	$1.8 \times 10^6 \mu$g B/g U

The DFs for F^- shown in Table 3 are all less than 70, but are based on the distribution coefficients of pure nitric acid/HF solutions. In solutions containing B and other components, F^- forms complex ionic species that are polar and are not as likely to extract. A recent sample from First U Cycle showed a DF for F^- greater than 40 for A Bank alone. (Since the F^- content in the 1BP sample was below the method detection limit, the actual DF would be greater than 40.) Due to the presence of B and other species, as well as the A Bank DF for F^- of 40, it is reasonable to expect a D Bank DF for F^- of at least 40. The TVA Blend Grade HEU specification for F^- is a relatively high value of 1,150 μg/g U, and the F^- concentration in the U storage tank is expected to approach 32,000 μg/g U at the end of the PuCS campaign. Thus, a DF for F^- of 40 would be sufficient for processing PuCS solutions from the U storage tank.

COMPLEXATION OF FLUORIDE BY BORON

At SRS, the dissolution of metals and refractory materials containing Pu or U is typically performed in nitric acid solutions with the addition of small amounts of F^-. Fluoride is added as a catalyst to increase the rate of dissolution. Once the dissolution is complete, the F^- is complexed with Al to reduce the corrosion of downstream processing equipment. With the addition of Al, evaporation of the waste solutions from purification processes can be performed without unacceptable corrosion in the evaporators. The PuCS material is currently being dissolved using a 3.5 M nitric acid solution containing 0.03 M F^-. When the PuCS solution is blended with the solution from the dissolution of reactor fuel, the Al contained in the fuel is more than sufficient to complex the small amount of F^-. However, if the PuCS solution is transferred directly to the U storage tank instead of blending with the fuel, no Al is added to the solution. One option for the subsequent processing of the PuCS solution is to add Al to complex the F^-. The second option is to take credit for the complexation of F^- by the B which is added to the solution as a nuclear safety poison. However, the adequacy of B for the prevention of excessive corrosion during evaporation of the waste solutions must be assured.

The stability of F^- complexes formed by B and Al was evaluated by Hammer (5) as a function of temperature in 0.96 M and 2.88 M nitric acid. Table 5 provides the data. Inspection of the data shows that the stability constants for the highest order F^- complexes for B are greater than those for Al. This information indicates that the free F^- concentration in the B system will not be more than the concentration in the Al system (assuming equal concentrations of B and Al). During the

Table 5. Stability constants for Al and B fluoride complexes in 0.96 and 2.88 M nitric acid (Reproduced from Hammer (5))

	25°C		35°C		45°C		60°C	
System	0.96 M	2.88 M	0.96 M	2.88 M	0.96 M	2.88 M	0.96 M	2.88 M
$Al^{3+}-HF-HNO_3$								
$Al^{3+} + HF = AlF^{2+} + H^+$	1607	1507	1512	1301	1313	1051	1152	1143
$Al^{3+} + 2HF = AlF_2^+ + 2H^+$	1.96×10^5	2.16×10^5	2.04×10^5	1.83×10^5	1.58×10^5	1.99×10^5	8.61×10^4	1.16×10^5
$Al^{3+} + 3HF = AlF_3 + 3H^+$	1.14×10^6	2.26×10^6	1.36×10^6	2.24×10^6	8.00×10^5	3.65×10^6	5.71×10^5	5.57×10^5
$Al^{3+} + 4HF = AlF_4^- + 4H^+$	1.62×10^6		$\sim1.39\times10^6$				$\sim7\times10^4$	
$H_3BO_3-HF-HNO_3$								
$H_3BO_3 + HF = H_4BO_3F$	15.2	20	10.5	11	11	15	1.7	2.1
$H_3BO_3 + 2HF = H_3BO_2F_2 + H_2O$	460	330	0	235	87	60	114	179
$H_3BO_3 + 3HF = H_2BOF_3 + 2H_2O$	7.03×10^5	9.58×10^5	3.65×10^5	3.41×10^5	1.75×10^5	2.24×10^5	3.01×10^4	6.15×10^4
$H_3BO_3 + 4HF = H^+ + BF_4^- + 3H_2O$	3.80×10^7	1.24×10^8	2.08×10^7	8.06×10^7	1.13×10^7	4.06×10^7	1.52×10^6	2.54×10^6

development of a flowsheet for the dissolution of sand, slag, and crucible (SS&C) residues in the SRS F Canyon dissolvers, free F^- concentrations were measured in a solution of 9.3 M nitric acid containing 0.025 M total F^- as a function of the B concentration (6). At nominally 0.05 M B (0.5 g B/L), the free F^- concentration was approximately 40 mg/L and dropped to approximately 10 mg/L when the B concentration was increased to 0.23 M (2.5 g/L). For comparison, the free F^- concentration measured in a solution containing 9.3 M nitric acid, 0.05 M F^-, and nominally 0.05 M Al (1.5 g/L) was approximately 80 mg/L. When the Al concentration increased to nominally 0.25 M (7 g/L), the free F^- concentration dropped to approximately 10 mg/L. These data demonstrate that the complexation of F^- in nitric acid solutions by B is similar in effectiveness to Al for equal concentrations of the cations, and B can be credited as a complexant for F^-.

CORROSION

During the development of a related flowsheet, a series of coupon immersion tests was performed to measure corrosion rates for various solutions containing nitric acid, F^-, B, and Al (6). The test coupons were fabricated from 304 L stainless steel; both welded and non-welded coupons were used. The solution temperatures were maintained at 85–90°C. Table 6 contains selected data from these tests.

The B and F^- concentrations used in Solution 2 most nearly correspond to concentrations used in the PuCS dissolving solution. For Solution 2, the free F^- concentration and corrosion rate were 17 mg/L and 24.7 mpy, respectively. It is expected that a slight decrease in the F^- concentration would lower the corrosion rates. The nitric acid concentration in Solution 2 is also nearly three times greater than the concentration (3.5 M) used for the dissolution of PuCS material. A lower nitric acid concentration would be expected to result in a lower corrosion rate.

Table 6. Selected 304 L corrosion rates in nitric acid/fluoride solutions

Solution number	Nitric acid (M)	Potassium fluoride (M)	B (M)	Al (M)	Free fluoride (mg/L)	Corrosion rate (mpy)
1	9.3	0.05	0	0	128	77.5
2	9.3	0.05	0.23	0	17	24.7
3	9.3	0.1	0.23	0	24	26.5
6	9.3	0.2	0.23	0.8	10	3.90

mpy ≡ mils per year.

The corrosion rates of 304 L stainless steels are strongly impacted by both the F^- and nitric acid concentrations resulting in increasing rates for increasing concentrations (7). The corrosion rates given in Table 6 were measured at slightly lower temperatures (85–90°C) than would be experienced in waste evaporators, which operate at boiling temperatures greater than 100°C. Higher corrosion rates would be expected at the higher temperatures. For boiling solutions of 6 M nitric acid with 0.01–0.02 M HF and no Al or B, the corrosion rate for 304 L stainless steel is 50 mpy (8). It should be noted that the small amount of HF in the boiling solution would approach equilibrium with the vapor phase. Therefore, a small amount of HF would collect in evaporator overheads. For process vessels at ambient temperature, the corrosion induced by 0.01 M F^- and 1 M nitric acid is low, well under 10 mpy even without complexation (8).

For the HEU Blendown process, the greatest degree of fluoride-induced corrosion is expected to occur in waste evaporators which will receive the aqueous 1DW waste stream containing B, Fe, and F from processing of the PuCS. The corrosion could impact both tank walls and equipment exposed to the solution. A concentration factor up to 30x can be attained as the evaporator overheads are sent to acid recovery, although a 15x concentration is expected to be more typical. The estimated concentrations of B, Fe, F^- and nitric acid are shown in Table 7 for the waste feed and different evaporator concentration factors. These data are conservative considering that the waste feed figures shown below are prior to water addition to the evaporator feed tank.

The corrosion rate for the starting condition in the waste evaporators would be essentially that for 1 M nitric acid since with 0.056 M B most of the F^- would probably be complexed. The corrosion rate would be between 0–5 mpy (9). For the 15x increase in concentration, the maximum corrosion rate will be less than 25 mpy based on data shown in Table 6. For an 8 M nitric acid solution with 0.2 M F^- and 2 g/L B, corrosion rates of approximately 25 mpy were measured. Since the 15x

Table 7. Selected evaporator concentrations

	Concentration M (g/L)		
Constituent	Feed	15x	30x
Fe	0.005 (0.3)	0.081 (4.5)	0.16 (9)
B	0.056 (0.6)	0.83 (9)*	1.67 (18)*
F^-	0.007 (0.14)	0.12 (2.2)	0.22 (4.2)
Nitric acid	1	7	8

* Note that B solubility is 3–4 g/L in 7–8 M HNO_3 at 25°C.

concentration has a lower acidity, lower F^-, and higher B, the free F^- concentration and subsequently the corrosion rate should be lower. For the 30x evaporator concentration, the corrosion rate would be closer to 25 mpy since the F^- is similar to test data. This rate would be expected to be a maximum because the actual B concentration would be higher than the 2 g/L used in corrosion tests. Welded areas would be expected to have slightly higher corrosion rates (2–5 mpy higher) based on available data (7,10). Thus, it is reasonable to use Solutions 2 and 3 in Table 6 to gauge corrosion levels in the waste evaporators for the proposed PuCS campaign.

GENERAL PROCESSING CONCERNS

The first general processing concern was the presence of insoluble solids in the PuCS feed. The PuCS material has no Al which is a normal source of silica. Silica content should be limited to small impurity levels from the carbon steel cans which contain the PuCS material. Historically, First Cycle feed is about 20 ppm silicon (Si). A sample of the PuCS solution was analyzed and found to have less Si content than is normally seen in current First Cycle feed. In addition, the sample was visually inspected and solids were not observed. Therefore, it is unlikely that the PuCS solution has appreciable solids.

The second general processing concern was the effect of increased B in downstream processes. While B would aid the proposed flowsheet changes by complexing F^- and thus limiting corrosion, B presents challenges in that it has limited solubility in concentrated nitric acid solutions. Table 8 lists literature data for solubility (11). Since the waste evaporator

Table 8. Solubility of H_3BO_3 in aqueous nitric acid at 25°C (11)

Nitric acid M	Solubility of H_3BO_3 M	Solubility of B g/L
0.078	0.882	9.54
0.534	0.803	8.68
1.58	0.669	7.23
3.12	0.544	5.89
3.81	0.498	5.38
4.70	0.447	4.83
8.41	0.296	3.20
10.95	0.2305	2.49
15.18	0.197	2.13

(which will eventually receive the B from PuCS solution) has multiple feed sources with different concentrations of nitric acid and other constituents, it is difficult to prescribe operational limits. However, as the proposed PuCS campaign progresses, appropriate limitations on the concentration factor achieved in the waste evaporators would be needed to prevent formation of boron-containing solids.

CONCLUSIONS

The evaluation of the proposed flowsheet change leads to the following conclusions:

1. Modeling calculations for both A and D Banks show that a B DF of $\sim$60,000 should be expected. Therefore, B contamination of the product solution is not a concern.
2. Boron can be credited as a complexant of F^- in nitric acid solutions, since B is similar to aluminum in reducing both free F^- concentration and corrosion of stainless steel.
3. The proposed routing change for Plutonium-Contaminated Scrap solutions will introduce F^- into vessels that do not typically contain F^-. Though B complexes F^-, F^- solutions in evaporators will cause an acceptable amount of corrosion, up to about 25 mils per year.
4. Whenever B is used as a complexant for F^-, the limited solubility of B in concentrated solutions of nitric acid should be recognized. Especially during evaporation operations, adequate controls must be put in place to prevent precipitation of boron-containing solids.
5. The proposed flowsheet changes can be implemented without adversely affecting product quality and without causing unacceptable corrosion.

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