Purification Testing for HEU Blend Program

by

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Abstract

The Savannah River Site (SRS) is working to dispose of the inventory of enriched uranium (EU) formerly used to make fuel for production reactors. The Tennessee Valley Authority (TVA) has agreed to take the material after blending the EU with either natural or depleted uranium to give a ^{235}U concentration of 4.8 %. The low-enriched uranium will be fabricated by a vendor into reactor fuel for use in TVA reactors. SRS prefers to blend the EU with existing depleted uranium (DU) solutions, however, the impurity concentrations in the DU and EU are so high that the blended material may not meet specifications agreed to with TVA. The principal non-radioactive impurities of concern are carbon, iron, phosphorus and sulfur. Neptunium and plutonium contamination levels are about 40 times greater than the desired specification.

Tests of solvent extraction and fuel preparation with solutions of SRS uranium demonstrate that the UO_2 prepared from these solutions will meet specifications for Fe, P and S, but may not meet the specifications for carbon. The reasons for carbon remaining in the oxide at such high levels is not fully understood, but may be overcome either by treatment of the solutions with activated carbon or heating the UO_3 in air for a longer time during the calcination step of fuel preparation.

Calculations of the expected removal of Np and Pu from the solutions show that the specification cannot be met with a single cycle of solvent extraction. The only way to ensure meeting the specification is dilution with natural U which contains no Np or Pu. Estimations of the decontamination from fission products and daughter products in the decay chains for the U isotopes show that the specification of 110 MEV Bq/g U can be met as long as the activities of the daughters of U-235 and U-238 are excluded from the specification.

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Introduction

The Savannah River Site (SRS) is working to dispose of the inventory of enriched uranium (EU) formerly used to make fuel for production reactors. The Tennessee Valley Authority (TVA) has agreed to take the material after blending the EU with either natural or depleted uranium to give a ^{235}U concentration of 4.8 %. The low-enriched uranium will be fabricated by a vendor into reactor fuel for use in TVA reactors. SRS prefers to blend the EU with existing depleted uranium (DU) solutions, however, the impurity concentrations in the DU and EU are so high that the blended material may not meet specifications agreed to with TVA. The principal non-radioactive impurities of concern are carbon, iron, phosphorus and sulfur. Carbon and phosphorus are from residual tributyl phosphate (TBP) and dibutyl phosphate (DBP) remaining after reprocessing by solvent extraction with TBP based processes. Iron and sulfur are from ferrous sulfamate (FS) used as a reductant for plutonium during reprocessing. The tests reported here were performed to determine the decontamination obtainable from the non-radioactive components during fuel fabrication processing. Testing of the effect of solvent extraction is also included to determine the overall effect of additional processing and the fuel fabrication process. Tests of removal of the radioactive impurities Np and Pu were estimated from past operations.

Discussion

The flow sheet favored by several fuel fabricators in the United States involves precipitation of U by ammonium hydroxide to form ammonium diuranate. The ammonium diuranate is calcined to produce UO_3 and then reduced to UO_2 with hydrogen. The UO_2 produced must meet the TVA specifications for use in their reactors. Tests were done spiking in only those impurities which have been shown to be high in the uranium solutions. Tests were done at the uranium concentration of the blended solution rather than the separate streams, however, the results are expected to be applicable to either stream if purified separately.

Tests for Non-Radioactive Contaminants

Tests of fuel fabrication were made with solutions containing U with DBP and TBP added to give carbon and phosphorus along with ferric nitrate and sulfuric acid to give iron and sulfur. The impurity concentrations were determined based on their concentrations in existing solutions being mixed to produce a final ^{235}U concentration of 4.8 %. The composition of the solution is shown in Table 1. The value for P is shown as a less than number due to U interference in the analytical method.

Table 1. Solution Composition for First Tests

Component	Concentration
Uranium	57 g/L
HNO_3	0.1 M
Carbon	2790 $\mu\text{g/g U}^*$
Iron	1070 $\mu\text{g/g U}^*$
Phosphorus	<400 $\mu\text{g/g U}^*$
Sulfur as sulfate	15663 $\mu\text{g/g U}^*$

* Micrograms per g U

In the first tests, the prepared solution was divided into two equal parts with one portion being washed with n-paraffin and one being untreated. Each portion was then treated to precipitate uranium by adding a 50 % stoichiometric excess of concentrated NH_4OH , then filtered, washed with three portions of 0.5 M NH_4OH , air dried, and calcined at 450°C for one hour. The solids were sampled for analysis after cooling to ambient temperature. All solutions were sampled and submitted for analysis. Problems were encountered with obtaining complete dissolution of DBP and TBP in the solutions and with the measurement of P by emission spectroscopy. The latter problem was a result of U spectral interference resulting in a high detectability limit for P. Table 2 shows the concentrations of the impurities in the oxide and the decontamination factors (DF).

Table 2. Results of Decontamination in Fuel Fabrication

Impurity	No Diluent Wash Conc'n, $\mu\text{g/g U}$	Diluent Wash Conc'n, $\mu\text{g/g U}$	No Diluent Wash DF	Diluent Wash DF
Carbon	597	1058	4.7	2.6
Phosphorus	<400	<400	NC	NC
Iron	775	664	1.4	2.8
Sulfur	1625	221	9.6	70.7

NC = Not Calculated

The carbon content of the washed material was higher than for the unwashed material. The n-paraffin wash should not increase the carbon. Since n-paraffin does not remove DBP from aqueous solutions, the difference is either due to a non-homogeneous sample or problems in sample analysis. Dibutyl phosphoric acid tends to adhere to plastic surfaces so it is possible that the difference was a result of contacting plastic surfaces during solution handling. Overall the DF is expected to be low because addition of base results in precipitation of uranyl dibutyl phosphate complexes carrying the carbon and phosphorus into the oxide product. Little decontamination from iron was expected because ferric hydroxide is highly insoluble in base. The decontamination from sulfate is in the expected range. Additional decontamination could be obtained by washing more. However, a DF of 70 is adequate to meet the specification of $< 15 \mu\text{g/g U}$, if the initial sulfur concentration is $< 1000 \mu\text{g/g U}$.

All solutions will be processed through solvent extraction to remove impurities especially the Pu, Np, and Fe which precipitate along with U in ammonium hydroxide solution. Thus, a second set of tests included batch solvent extraction to determine the overall purification obtained with processing existing solutions through a cycle of solvent extraction prior to fuel fabrication. Solution was prepared by dissolving 45 g of uranyl nitrate hexahydrate in 100 mL of 0.1 M HNO_3 , adding 0.0271 g of 96 wt% H_2SO_4 , and 0.181 g of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$. Solution analyses showed 190 g/L U with 1179 $\mu\text{g SO}_4^{2-}/\text{g U}$ and 1170 $\mu\text{g Fe/g U}$. The solution was made 0.012 M ferrous sulfamate (FS) by adding 1.8 M FS solution (FS is the reductant used to reduce Pu and improve decontamination). Then 59 mL of 1.5 M HNO_3 was added to increase acidity and aid extraction. After mixing the aqueous solutions in a separatory funnel, 450 mL of 30 vol.% TBP n-paraffin was added and the solutions were mixed in the separatory funnel. The aqueous was drained out for disposal. Twenty-nine mL of 1.5 M HNO_3 was added and the organic and aqueous shaken together to scrub the solution. The aqueous was drained and 30 mL of water was added to reduce the acid concentration prior to stripping the U into two 225 mL portions of 0.08 M HNO_3 . The strip solutions were combined, sampled, and divided into two equal portions. One portion was washed with a small portion of fresh plant diluent (n-paraffin) prior to further processing. Samples were taken from both the washed and unwashed aqueous solutions for U and impurity analyses. Both solutions were then treated by the following steps representative of preparation of UO_2 powder for fuel fabrication.

Uranium was precipitated as ammonium diuranate by addition of a 50 % stoichiometric excess of concentrated NH_4OH , filtered through a stainless steel filter frit, and washed 3 times with 0.5 M NH_4OH . After air drying with vacuum, the solids were transferred to a quartz crucible and heated at 450 C in an air atmosphere for 1 hr to convert to UO_3 . After cooling the UO_3 solids were stirred and sampled for analyses. The remaining UO_3 was placed in a quartz boat in a tube furnace and heated for 3-4 hours in a stream of 4 vol% hydrogen in argon at 700°C for conversion to UO_2 . The UO_2 was cooled, stirred, and sampled for analyses. Table 3 summarizes the impurity concentrations obtained in the test.

The carbon concentration in the initial solution was not measured because the TBP solvent would add far more carbon than was present initially. The very high C concentration in the unwashed solution after solvent extraction is due to dissolved TBP as shown by the high removal during washing with n-paraffin. The subsequent increase after precipitation and calcination is unknown. However, the differences could be due to the first two analyses being made on solutions while the latter four analyses were made directly on the solid oxides. There are other questions about the C data in that the UO_2 from n-paraffin washed solution had higher C than UO_2 from unwashed solution. This may indicate contamination during handling of the samples rather than actual removal efficiencies. The earlier test shown in Table 2 indicate a DF of 2.6-4.6 should be obtained during the fuel manufacturing steps. Assuming that to be the case, the C content of material after solvent washing to remove TBP would be less than the specification of 200 $\mu\text{g/g}$ U. Alternatively, diluting with natural U instead of the DU would bring the C within the desired specification.

Table 3. Results from Solvent Extraction Test

<u>Test Step</u>	<u>C, $\mu\text{g/g}$ U</u>	<u>Fe, $\mu\text{g/g}$ U</u>	<u>P, $\mu\text{g/g}$ U</u>	<u>S, $\mu\text{g/g}$ U</u>
Initial Solution	NA	19300	NA	4400
Solvent Extraction Product - Unwashed	48200	<160	NA	88
Solvent Extraction Product - n-paraffin Washed	420	<160	NA	70
UO_3 from Unwashed Solution	2300	NA	<510	210
UO_3 from n-paraffin Washed Solution	1800	NA	160	170
UO_2 from Unwashed Solution	1020	NA	140	140
UO_2 from n-paraffin Washed Solution	1400	NA	250	120
<i>Proposed TVA Specification</i>	200	250	250	100

NA = Not Analyzed

The concentrations of Fe and S in the initial solution are the total of that added initially plus that present from the FS. Iron concentrations in the solvent extraction product solutions were below the detectability limits for the analytical method. Unless Fe contamination from corrosion or added chemicals increased the Fe concentration, the value obtained would be within the proposed specifications for acceptance by TVA (250 $\mu\text{g/g}$ U). Consequently Fe was not analyzed in the solid products.

Phosphorus was not analyzed in the initial solutions because the interference from U would make the results meaningless. Phosphate ion was analyzed by ion chromatography in the UO_3 and UO_2 products after dissolution in HNO_3 . Ion chromatography has a detection limit for PO_4^{3-} which is very close to the values obtained for these solutions. In fact, the UO_3 made from unwashed solution was given as a less than value because the value was below the detection limit. The phosphorus concentration was calculated from the measured U and PO_4^{3-} concentrations. The data show that the UO_2 product will be at or within the proposed specification of 250 $\mu\text{g/g}$ U. It is interesting to note that the C and P analyses for the washed and unwashed UO_2 products correspond in that both analyses are higher for the washed sample than for the unwashed sample.

The analyses are the opposite of what is expected from the chemistry, the solution analyses, and the analyses of the UO_3 .

The data indicates that the specification for sulfur was not met in this test. However, this test used only one stage of scrubbing to remove impurities. At least 3 scrub stages will be present between the addition of FS and exit from the mixer settler during plant operations. The decontamination factor shown here for one stage of scrubbing is about 35 (4400/126). Each scrub stage can be expected to give a decontamination factor of at least 5-10. Thus, the minimum decontamination factor for 3 scrub stages would be $5 \times 5 \times 5$, which is 125. Decontamination by a factor of 125 should give a product with 35 $\mu\text{g/g}$ U, which is well within the specification of 100 $\mu\text{g/g}$ U. An overall decontamination factor of only 50 would bring the S concentration below 100 $\mu\text{g/g}$ U. Thus, solvent extraction can be expected to result in a product that meets the specifications for S.

All analyses were performed by the Analytical Development Section of the Savannah River Technology Center using standard analytical methods with NIST traceable standards. Uranium was analyzed by a Chem Check^{TM} instrument using U phosphorescence. Acid was determined by titration with NaOH after complexation of metal ions. Carbon was determined using a LECO^{TM} instrument in which the sample is oxidized in a stream of oxygen with CO_2 being measured by infrared spectroscopy. Iron and phosphorus were determined by ion-coupled plasma emission spectroscopy (ICP-ES). Sulfate was determined by ion chromatography and sulfur was calculated from sulfate. High concentrations of nitrate as were present in these solutions reduces the detection limit for sulfate ion. Ion chromatography cannot be used to analyze for concentrations lower than about 75 $\mu\text{g/g}$ U unless the nitrate ion is removed by a pretreatment step. Such a pretreatment step is not presently used in our analytical laboratories. Analyses for phosphate and sulfate ions in uranium oxide solids was done by first dissolving the solids in dilute HNO_3 solution, evaporating to dryness at ambient temperature with an air flow across the top of the beaker and then re-dissolving in deionized water. This procedure was followed to minimize interference from nitrate ion during analyses.

Np and Pu Contamination

These tests did not include Np or Pu which are above the desired specifications. The activities of Np and Pu in the EU and DU solutions are shown in Table 4 along with the expected decontamination factors based on past performance of second U cycle in F and H canyons. The decontamination from Pu is better than for Np at

Table 4. Neptunium and Plutonium Contamination of Uranium Solutions

Radionuclide	Existing Solutions		2 nd Cycle DF	Purified Solutions	
	EU	DU		EU	DU
Np	665 Bq/g U	217 Bq/g U	EU = 9, DU = 3	74 Bq/g U	72 Bq/g U
Pu	231 Bq/g U	461 Bq/g U	EU=10, DU=20	23 Bq/g U	23 Bq/g U

comparable concentrations in the feed stream because FS is added to reduce Pu from the extractable Pu(IV) to inextractable Pu(III) (see Equation 1 below). The addition of reductant, however, increases Np extractability by reducing inextractable Np(V) to extractable Np(IV) (see Equation 2 below).

A part of the problem is probably associated with cross contamination from previous solutions. Extensive flushing might increase the DFs slightly; however, the entrainment of aqueous in the organic phase in the mixer settlers also contributes to the low DF and is not readily overcome without extensive equipment changes. The data shows that reduction of both Np and Pu in a single pass through the second uranium cycle is difficult to achieve. The contamination levels which can be achieved by a single cycle of solvent extraction will not produce solution within the desired TVA specification of 25 Bq/g U. It is possible with two cycles of solvent extraction where no reductant is added in one of the cycles could decrease the Np contamination closer to that for Pu, but the total would still be about two times higher than the desired specification of 25 Bq/g U. Dilution with natural U, which contains no Np or Pu, would bring the total Np-Pu within the specification without a second cycle of solvent extraction.

Gamma Contamination

The gamma activity of the solutions is higher than the specification of 110 MEV Bq/g U. The gamma activity is from fission products and daughter products in the decay chains of the various U isotopes. The largest contributor are from the U-236 decay chain which is shown in Figure 1.¹ The efficiency of the second U solvent extraction cycle removal of the gamma activity will vary depending on the element and concentration.

Figure 1. Decays Chains for the Principal Uranium Isotopes

Table 5 shows the principal contributors to the gamma activity in the HEU solutions as reported in a recent analysis from the Y-12 laboratory at Oak Ridge.

Table 5. Gamma Emitting Isotopes in Enriched Uranium Solutions

<u>Element and Isotope</u>	<u>Gamma Activity, MEV Bq/g U</u>	<u>Expected DF</u>	<u>Final Activity, MEV Bq/g U</u>
I-132 - Fission Product	230	1.5-2	115-150
Ra-224 - U-236 Chain	150	150-200	1
Th-231 - U-235 Chain	1200	100-150	8-12
Bi-212 - U-236 Chain	770	90-100	8-9
Pb-212 - U-236 Chain	1900	150-200	10-13
Tl-208 - U-236 Chain	14,000	90-100	140-160
Zr-95 - Fission Product	150	1-2	75-150
All others	210	2-3	70-105
Totals	18,610	31-44	427-600

The expected DFs were estimated on the basis of performance for Pu as a function of activity in the feed (d/m/mL) to the second U cycle in H canyon except for Th which was based on Np performance as a function of activity. Thorium DF should be slightly higher than Np because the distribution coefficient of Th(IV) is lower than that of Np(IV). The Tl-208 has only a 3.1 minute half-life, so it will decay in less than 30 minutes to a level that is in equilibrium with its Bi-212 precursor. Therefore, the DF for Tl-208 was based on the DF for Bi-212. A range is given for the DF and the final activity because actual performance is variable. The HEU solution after solvent extraction will have much lower gamma activity, but will not be within the specification before dilution. It is surprising that fission products such as Zr-95 and I-132 are still present to significant activity because of their short half-lives, 65 day and 2.3 hour, respectively. This may be the level which is maintained in solution due to fissions occurring in solution.

All of the daughter gamma activities from U-235 and U-236 will grow back into the solution after separation as shown in Figure 1. Radioactivity from Th-231, the first daughter in the U-235 chain, grows back into equilibrium in <30 days because of the short half-life of Th-231 (25.6 hours). All radioactivity from decay of U-235 should be excluded from the specification because it will always be in equilibrium corresponding to the U-235 concentration in solution. This means that there will be 95 MEV Bq/g U from Th-231 in any solution containing 4.8 wt% U-235 including virgin material from enrichment of natural U. The daughter products from U-236 are higher than from virgin enriched U. The decay chain for U-236 in Figure 1 shows that the half-lives of Ra-224 and the remainder of the chain are a few days or less, which is the reason the gamma activities are high. The short half lives result in rapid decay to equilibrium with the precursor activity Th-228, which was not determined in recent analyses. However, the half-lives of U-235 and Pa-231 are long enough that it will take years to reach the same activity levels obtained here

The gamma activity in the DU solution is lower because the U-238 has a longer half-life and the decay chain has fewer high energy gamma emitting nuclides. Table 6 shows information similar to Table 5, but for the DU solution. The DFs for Th and Ru were estimated in the same manner as in the case of HEU above. The DF for Pa was made the same as for its precursor Th. The DF for Nb-95 was set to be that expected for Zr-95 its precursor which is very low. As shown in the table, the DFs should be sufficient to bring the DU well within the specification. The reason for this is that high DFs are expected for the two highest activities. As noted above early purification of the solutions would allow the Th and Pa activities to grow back in since they result from decay of U-238. Figure 2 shows the growth of Th-234 into the solution from U-238 decay.

Table 6. Gamma Emitting Isotopes in Depleted Uranium Solutions

<u>Element and Isotope</u>	<u>Gamma Activity, MEV Bq/g U</u>	<u>Expected DF</u>	<u>Final Activity, MEV Bq/g U</u>
Pa-234m - U-238 Chain	220	100-150	1.4-2
Th-234 - U-238 Chain	110	100-150	0.7-1
Nb-95 - Fission Product	40	1-2	20-40
Ru-106 - Fission Product	50	10-15	3-5
All others	50	2-3	17-25
Totals	470	6-11	42-73

The calculations were made using the following equation where N_1 is the atoms of U-238 in a gram of DU, N_2 is the atoms of Th-234, superscript 0 represents the atoms at time = 0, and λ_1 and λ_2 are the respective decay constants.²

$$N_2 = \lambda_1 / \lambda_2 - \lambda_1 * N_1^0 * (\exp(-\lambda_1 t) - \exp(-\lambda_2 t)) + N_2^0 * \exp(-\lambda_2 t)$$

The term N_2^0 was assumed to be zero for the graph, so the second term dropped out. Note that within about 150 days the Th-234 has reached secular equilibrium. In addition, the Pa-234m with a half life of only 1.17 minutes will also be at equilibrium.

Figure 2. Rate of Formation of Th-234 from One Gram of Depleted Uranium

The activities of the daughters of U-238 should be excluded from the specification because they will be there at about the same level in natural U and any enriched U. Mixing of the purified solutions to make 4.8 wt% U-235 would give solution with gamma activity between 61 and 97 MEV Bq/g U assuming the HEU is 60.5 wt% U-235 and the DU is 0.244 wt% U-235 and excluding U-235 and U-238 daughter gamma activities. Thus, it appears feasible that the specification of 110 MEV Bq/g U can be met by purification of both U solutions prior to mixing. However, purification of the HEU should be done shortly before mixing of the solutions to minimize effects of decay of U isotopes increasing the gamma activity out of the desired range.

Conclusions

Tests of solvent extraction and fuel preparation with solutions of SRS uranium demonstrate that the UO_2 prepared from these solutions will meet specifications for Fe, P and S, but may not meet the specifications for carbon. The reasons for carbon remaining in the oxide at such high levels is not fully understood, but may be overcome either by treatment of the solutions with activated carbon or heating the UO_3 in air for a longer time during the calcination step of fuel preparation.

Estimations of the expected removal of Np and Pu from the solutions show that the specification cannot be met with a single cycle of solvent extraction. The only way to ensure meeting the specification is dilution with natural U which contains no Np or Pu. Estimations of the decontamination from fission products and daughter products in the decay chains for the U isotopes show that the specification of 110 MEV Bq/g U can be met as long as the activities of the daughters of U-235 and U-238 are excluded from the specification.

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