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Dissolution of FB-Line Cabinet Sweepings


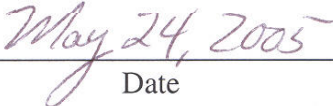
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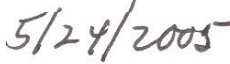
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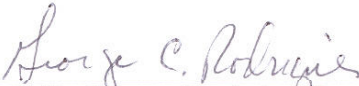
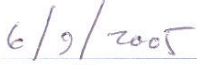
 

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Summary

Three FB-Line samples were received by the Savannah River National Laboratory (SRNL) for characterization and evaluation for suitability for HB-Line dissolution. These samples are part of a larger sampling/evaluation program in support of FB-Line deinventory efforts. The samples studied were identified as MC04-147- HBL, MC04-148-HBL, and FBL-SWP-04-016-HBL (N). The first sample, MC04-147-HBL, is a portion of FB-Line Packaging and Stabilization (P&S) materials. The second sample, MC04-148-HBL, is a sweeping from Cabinet 6-8, which is not representative of the mechanical line. The third sample, FBL-SWP-04-016-HBL (N), is an FB-Line North cabinet sweeping. The samples were described by FB-Line personnel as containing plutonium oxide (PuO_2) which had not been high-fired. This description was generally confirmed by solids analysis and off gas measurements.

All three samples were dissolved in 8 M HNO_3 / 0.1 M KF at 90-100°C leaving minor amounts of solid residue. During dissolution, sample MC04-147 did not generate hydrogen gas. Sample MC04-148 generated modest amounts of gas, which contained 4.0 to 4.7 volume percent (vol %) hydrogen (H_2) at a ratio of up to 8.4×10^{-5} mol H_2 /g sample. Sample FBL-SWP-04-016-HBL (N) was nearly completely soluble in 8 M HNO_3 and produced a very small amount of gas. Apparently, the CaF_2 in that sample dissolves and provides sufficient fluoride to support the dissolution of other components.

Experimental

Samples were handled in a glovebox authorized for beryllium work, since each sample was labeled as having potential beryllium contamination. All three of the samples looked quite similar, dark colored and granular. Sample portions were weighed with a calibrated analytical balance (M&TE #ATD1-196). Portions of each sample were dissolved in a solution of 8 M nitric acid (HNO_3) and 0.1 M potassium fluoride (KF). In addition, a portion of one sample, FBL-SWP-04-016-HBL (N), was dissolved in 8 M HNO_3 . The solutions were initially at or near room temperature and were heated to about 99°C during the dissolution. Details of the dissolutions of each sample are included in the Results section.

Dissolutions were conducted in a four-necked 1-L round-bottomed flask surrounded by a heating mantle. A thermometer was inserted through one neck, and was immersed in the solution. The other flask openings were used for the nitrogen purge gas line, off gas outlet, and sample addition. A condenser, a glass bulb (for gas sampling), and an empty 1 L Tedlar bag (for gas volume measurement) were connected in series (with tubing) to the off gas outlet neck. Prior to use, the sample bulbs were purged with nitrogen (N_2) gas for 5-10 minutes, and Tedlar bags were evacuated with a vacuum pump. When multiple gas samples were taken, the initial glass bulb/Tedlar bag pair was removed and another pair was attached. The volume of gas collected in each bag was determined by water displacement, less the volume of an empty bag. Generally, the extent of dissolution was estimated – portions of the filter papers containing residual solids were cut while still wet

and mounted for analysis. However, for the dissolution of FBL-SWP-04-016-HBL (N) in 8 M HNO_3 , the filter paper containing residual solids was rinsed three times with water to minimize soluble salt content and allowed to dry overnight. The dry mass less the average mass of a dry, new piece of filter paper was used to determine the mass of residual solids.

Analyses of the solids, final dissolver solutions, and off gas samples were performed at SRNL. Samples of as-received solids, as well as residual solids which were collected on filter paper after dissolution, were analyzed by X-ray powder diffraction (XRD) and sometimes by scanning electron microscopy (SEM). Elemental analyses of the dissolver solutions were performed by Inductively Coupled Plasma – Emission Spectroscopy (ICP-ES); Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS) was used to aid in interpretation of the ICP-ES results. The concentrations of Pu-239/240 and Pu-238 were measured by a spiked extraction method (termed Pu-TTA for thenoyl-trifluoroacetone), and concentrations of Pu-241, Am-241, and U-237 were determined by gamma pulse height analysis (PHA). Off gas samples were analyzed by gas chromatography using an HPLC 5890 gas chromatograph with a Carboxen 1000 packed column, a thermal conductivity detector, and argon carrier gas to determine the hydrogen content. Dissolver solutions were also analyzed by atomic absorption (AA) to determine arsenic and selenium, and by cold vapor atomic absorption to determine mercury. The uncertainties in the analytical results were reported at the one-sigma (or one standard deviation) level. For Pu-TTA and gamma PHA results, the uncertainties were generally $\pm 5\%$ or less, for ICP-ES and gas chromatography, uncertainties were $\pm 10\%$, and for ICP-MS and AA, uncertainties were $\pm 20\%$.

Results

Dissolution / Solids Analysis

Sample MC04-147-HBL. Based on the XRD analysis, the as-received sample contained, in decreasing amounts, magnesium oxide (MgO), calcium fluoride (CaF_2), PuO_2 , and plutonium tetrafluoride hydrate ($\text{PuF}_4 \cdot 2.5\text{H}_2\text{O}$). A portion of the sample, 3.18 g, was added to 250 mL of dissolver solution. The mixture was heated to 86 – 99°C for 30 minutes. After dissolution, the predominant residual solid was talc ($\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$), with minor amounts of PuO_2 , a uranium oxyfluoride ($\text{U}_2\text{O}_2\text{F}_7$), and quartz (SiO_2). It is estimated that 95% of the initial solid charge dissolved. SEM analysis of the residual solids showed that particles containing Pu were rare. The predominant elements in the residual solid matrix had low atomic numbers, which may be indicative of talc, and contained significant phosphorus and potassium. Residual particles, believed to be corrosion products, containing iron, chromium, and nickel, were also found.

Sample MC04-148-HBL. Based on the XRD analysis, the as-received sample contained, in decreasing amounts, MgO , PuO_2 , uranium dioxide (UO_2), talc, calcite (CaCO_3), SiO_2 , and $\text{U}_2\text{O}_2\text{F}_7$. Initially, 9.89 g of sample was added to 250 mL of 8 M HNO_3 /0.1 M KF at 30°C. Immediately, modest gas generation was observed for about 30 seconds as bubbles

broke the surface of the liquid, but no significant increase in the gas bag volume was observed. The dissolver solution and sample were then heated to 90-100°C for 30 minutes. After the solution cooled (but later the same day), another 250 mL of dissolver solution was added to the flask, along with another 5.00 g of solid. Again, an initial reaction took place for 20-30 seconds and a few bubbles were observed at the surface of the liquid, but no obvious change in the gas bag volume was observed. The combined solution (with added solid) was heated again and held at 90 – 100°C for 50 minutes.

The remaining solids were separated from the solution by filtration. The sample was quite soluble; it is estimated that 95% of the solids dissolved. Based on XRD results, talc was again identified as the predominant component in the insoluble solids. A significant amount of quartz was also present in the insoluble fraction, along with a trace amount of PuO₂ (qualitatively estimated to be 1-5 wt %). SEM analysis of the residual solids showed that the residual solid matrix was primarily silicon, magnesium, and oxygen, which is indicative of talc. The SEM results also showed scattered particles containing Pu (along with calcium, oxygen, and fluoride). The SEM also identified a particle of nearly pure tungsten or tungsten oxide, a particle containing zirconium (along with aluminum, silicon and oxygen), and another containing tin (along with silicon and oxygen).

Sample FBL-SWP-04-016-HBL (N). Analysis of the as-received solids by XRD showed the following phases (in decreasing amounts), MgO, CaF₂, PuO₂, talc, and CaCO₃. A 10-g portion of the sample was dissolved in 414 mL of 8 M HNO₃/0.1 M KF. The solution was initially at 33°C and was heated to 90°C in 34 minutes, kept between 90 and 92°C for 60 minutes, and subsequently allowed to cool. The sample was very soluble; it is estimated that 98-99% of the solids dissolved based on visual comparison to the filter paper described below. The remaining solids were separated by filtration. Since there were not sufficient solids on the filter paper for scraping off and submitting for analysis, a piece of the filter paper was cut and mounted for analysis. The residual solids on the filter paper consisted of talc, PuO₂, and SiO₂ (in decreasing amounts).

A second 9.4 g sample was dissolved in 300 mL of 8 M HNO₃. The solution was initially at 34°C and was heated to 89°C over a 35 minute time interval, kept between 89 and 94°C for 60 minutes, and subsequently allowed to cool. The sample was very soluble. Measurement of the dried filter paper containing residual solids indicated that 97% of the initial solids dissolved.

Discussion of Dissolution / Solids Analysis Results. The extent of dissolution for these three samples ranged from 95-99% after heating to nominally 90°C for 30-80 minutes. For each of the three samples, the predominant phase in the residual solids was talc; PuO₂ was also present in all three residues. It is likely that a four-hour hold time at 90-100°C would further dissolve the PuO₂. When evaluating XRD results, it should be noted that some of the particles may have been non-crystalline, making them nearly invisible to XRD analyses. When SEM was performed on the residual solids, it generally confirmed the XRD results while providing more information about minor constituents. Some

constituents, such as the tungsten particles found in sample MC04-148, are unlikely to dissolve in the nitric acid solution used in these experiments.

The dissolution of sample FBL-SWP-04-016 (N) in 8 M HNO₃ was conducted without added fluoride since characterization of the solids indicated the presence of significant CaF₂ in the sample. Apparently, the CaF₂ in the sample provides sufficient fluoride to promote dissolution of plutonium oxide. This is not surprising, as CaF₂ has been used as a fluoride source instead of KF. Since fluoride used in the HB-Line dissolution process must be complexed with aluminum before downstream processing, dissolution in 8 M HNO₃ is preferred as it reduces the amount of aluminum that must be added after dissolution.

Gas Generation

Tables 1 and 2 contain the measured gas volumes and H₂ concentrations from the dissolution tests. In Tables 1 and 2, the far right column, "H₂ Content of New Gas," contains values calculated assuming ideal mixing of gas in the headspace, condenser, tubing, sample bulb, and sample bag. In other words, these values account for the dilution effect of the gas initially present in the headspace. Note also that temperature effects were neglected in the reported results, since the headspace vapor temperature is generally much lower than the solution temperature during the heating cycle.

Table 1. Gas Generation During Dissolution of Samples MC04-147 and MC04-148.

Sample	Sample Mass, g	Elapsed Time, min	Temp./Heating Profile °C	Gas Volume, mL	Measured H ₂ Content Vol %	H ₂ Content of New Gas, Vol %
147	3.1823	149	29 to 99 to 51	126	<0.1	~ 0
148	9.8872	40	28 to 75	318	1.2	4.7
		32	75 to 107 to 92	205	1.7	4.0
148	4.9962	12	32 to 41	24	<0.1	~ 0
		10.5	41 to 59	28	0.19	4.5
		13.5	59 to 77	32	<0.1*	~ 0
		59	77 to 96 to 92	166	<0.1	~ 0

*Sample may have been compromised. Even if no hydrogen had been produced during this sampling interval, a hydrogen content of 0.17 vol% would be expected based on the composition of the previous sample and the volume of gas produced.

Sample MC04-147-HBL. Dissolution of 3.18 g of this sample was conducted in 250 mL of 8 M HNO₃/0.1 M KF. The mixture was heated to 86-99°C for 30 minutes. The headspace of the vessel was not purged prior to dissolution. One gas sample was taken over the entire dissolution process. Since hydrogen was not found (see Table 1), an additional test was not conducted. The gas in the headspace, gas sample bulb, and Tedlar bag was colorless, making it highly unlikely that appreciable NO₂(g) was present.

Sample MC04-148-HBL. Initially, a nominally 10 g sample was dissolved in 250 mL of 8 M HNO₃/0.1 M KF and heated at 90-107°C for 30 minutes. After the solution cooled (but later the same day), another 250 mL of 8 M HNO₃/0.1 M KF were added to the flask. The headspace was flushed with N₂, and more sample (5 g) was added. The combined solution (with added solid) was heated again to 90-96°C for 50 minutes. Details of gas generation results, as well as temperature profile, are included in Table 1. The presence of brown gas (most likely NO₂) was observed in the headspace and in gas sample bulbs and bags.

Table 2. Gas Generation During Dissolution of Sample FBL-SWP-04-016-HBL (N)

Sample Mass, g	Elapsed Time, min	Temp./Heating Profile °C	Generated Gas Volume, mL	Measured H ₂ Content vol %	H ₂ Content of New Gas, vol %
10.0016	16.5	33 to 66	0	<0.1	~0
	14.5	66 to 87	19	<0.1	~0
	21	87 to 92	30	2.2	58
	19	92	26	Compromised sample	
	20	92 to 91	10	0.34	0
9.4309 *	35	34 to 89	15	NM	NM
	60	89 to 94			
	15	94 to 60			

NM = Not Measured

* Dissolution in 8 M HNO₃. Only one Tedlar bag was used for gas volume measurement.

Sample FBL-SWP-04-016-HBL (N) in 8 M HNO₃/0.1 M KF. Prior to dissolution, glass sample bulbs were purged with nitrogen for 5 minutes each, and Tedlar sample bags were evacuated with a vacuum pump. In addition, the headspace of the flask and the condenser were purged with nitrogen for 6 minutes. However, the flask was opened for about 8 seconds while the sample was poured in. Five sets of sample bulbs and bags were used over the course of the dissolution. Results are shown in Table 2. Small amounts of gas were produced throughout the dissolution (average: 8.5 mL gas/g sample dissolved). Note that the total headspace volume is an order of magnitude greater than the amount of gas present in any sample. As mentioned above, the “H₂ Content of New Gas” values in Table 2 involve an ideal mixing assumption. This assumption provides a conservatively high estimate of the hydrogen concentration for the third sample, which contained significant hydrogen. The fourth sample, reported to have 0.03% hydrogen, was deemed compromised because the small volume of gas produced could not have diluted the headspace to such a low level of hydrogen. All of the gas samples taken during the dissolution of sample FBL-SWP-04-016 (N) were essentially colorless, so it is unlikely that NO₂(g) was present at more than trace concentrations.

Sample FBL-SWP-04-016-HBL (N) in 8 M HNO₃. Prior to dissolution, a glass sample bulb and a Tedlar sample bag were purged with nitrogen for 10 minutes. The Tedlar bag

was then evacuated with a vacuum pump. A very small amount of gas, 15 mL, was generated during the dissolution process and no brown gas was observed in the system.

Dissolver Solutions

Samples from each of the dissolver solutions were submitted for analyses. Table 3 shows the amounts of uranium and the seven most abundant elements detected by ICP-ES. Complete ICP-ES results are tabulated in the Appendix.

Table 3. Content of Dissolver Solutions

Analyte	MC04-147-HBL		MC04-148-HBL		FBL-SWP-04-016 (N)	
	g/L	wt% in sample	g/L	wt% in sample	g/L	wt% in sample
Al	0.0835	0.7	0.164	0.6	0.170	0.8
Ca	0.319	2.5	1.080	3.6	1.390	6.9
Fe	0.130	1.0	0.409	1.4	0.259	1.3
Mg	1.820	14	1.750	5.9	2.580	12.9
Na	0.0842	0.66	0.270	0.9	0.119	0.6
Si	0.675	--	0.639	--	0.786	--
U	0.0181	0.14	4.570	15.4	0.0106	0.053
Pu-239/240	2.71	21.3	4.33	14.5	2.95	14.7
Pu-238	0.00077	6.0E-03	0.0038	0.013	5.71E-04	0.0029
Pu-241	Bdl	---	0.0122	0.041	7.47E-04	0.0037
Am-241	0.0041	3.2E-02	0.0516	0.17	1.84E-03	0.0092
U-237	2.6E-10	2.0E-09	6.7E-10	2.3E-09	Bdl	---

Bdl – Below detection limit.

For radioisotopes, the reported results, provided in units of activity (dpm/mL), were converted to g/L concentrations and are shown in Table 3, along with a conversion to the weight percent of the analyte in the original dry solid sample. Note that the most abundant elements present in the dissolver product solutions were also observed in XRD analyses of the as-received solids. Some of the silicon (and boron, as shown in Table A-1 of the Appendix) is attributed to the effect of fluoride on the laboratory glassware.

Analysis of solutions by ICP-MS (samples MC04-147 and FBL-SWP-04-016 (N)) confirmed the order of magnitude of the concentrations reported for Pu and U, as well as for some minor components (shown in the Appendix) such as molybdenum (Mo) and tin (Sn). Since initial analysis of strontium (Sr) by ICP-ES was suspected to be high due to spectral interferences, ICP-MS was used to measure Sr and showed that the isotopic abundances were indicative of naturally-occurring Sr. In addition, ICP-MS reported that isotopes of mass 90 were present on the order of 0.7 mg/L. These were almost certainly zirconium, based on the relative abundances of masses 90 and 91. Furthermore, the

presence of significant strontium-90 in these sweepings is unlikely based on process history and the absence of other fission products, such as cesium-137. Analysis of MC04-148 solution by ICP-MS indicated that the U in that sample is 21% U-235.

Analysis of solutions by AA indicated that levels of arsenic, mercury, and selenium were low in all three samples, as shown in Table A-2 of the Appendix. Note that some of the solutions analyzed by AA had different sample mass-to-volume ratios than solutions analyzed by other methods. These difference are due to additional sample portions being dissolved (by the same method) in preparation for disposition. For clarification, “wt% of sample” values, which represent the amount of analyte in the original dry solid sample, were included in Table A-2.

Discussion of Gas Generation Results. The cumulative gas generation results for each of the dissolution tests are shown in Table 4. These results indicate that the samples represent three different types of material. The first sample, MC04-147, generated a modest amount of gas, but no hydrogen. The second sample, MC04-148, generated a modest amount of gas that included H₂ and NO₂. With sample 148, both a 10 g and a 5 g sample generated about the same volume of gas. However, the 5 g sample generated less hydrogen than the 10 g portion. It is unclear why the two portions produced different amounts of hydrogen. However, for the 5 g sample, the initial presence of dissolved uranium, plutonium and other components may have shifted the dissolution mechanism to one that produces more NO₂. In addition, though the sample appeared homogeneous, the granular material may have had some heterogeneity.

Table 4. Cumulative Gas Generation Results

Sample	Mass g	Gas Generated mL/g	Calculated H ₂ Generation mol H ₂ /g	Presence of NO ₂ ?
MC04-147	3.18	40	~ 0	No
MC04-148	9.89	53	8.4E-5	Yes
MC04-148	5.00	50	2.8E-5	Yes
FBL-SWP-04-016-HBL (N)	10.0	8.5	7.0E-5	Trace
FBL-SWP-04-016-HBL (N) *	9.43	1.6	< 6.4E-5 **	No

* Sample dissolved in 8 M HNO₃. All others dissolved in 8 M HNO₃/0.1 M KF.

** Total moles generated per gram were 6.4×10^{-5} . Hydrogen content was not measured.

In sample MC04-148, the predominant form of uranium (U) in the as-received solids was UO₂. As UO₂ dissolves in nitric acid, the U(IV) in the solid converts to U(VI) in solution, producing uranyl ion, UO₂²⁺, and causing generation of hydrogen and or nitrogen oxides. The oxidation of only one-fourth of the U in sample MC04-148 from U(IV) to U(VI) could account for all of the hydrogen generation observed.

For the portion of sample FBL-SWP-04-016 (N) dissolved in 8 M HNO₃/0.1 M KF, a small amount of hydrogen was generated after the temperature reached 87°C. However,

for the portion of sample FBL-SWP-04-016 (N) dissolved in 8 M HNO_3 without added fluoride, very little gas generation was observed. Since the hydrogen generation occurred at a high temperature in the presence of added fluoride, and Al was present in the resulting solution, a cause of the hydrogen generation appears to be the presence of metallic aluminum impurities in the as-received sample. The total aluminum concentration for this sample was only 170 mg/L after dissolution in 8 M HNO_3 /0.1 M KF. If only 20% of that aluminum was initially metal, that could account for all of the hydrogen generated. It is not likely that metallic calcium or iron were present, since these would have dissolved to produce hydrogen at a lower temperature regardless of the fluoride content. Though magnesium (Mg) was a major component of sample FBL-SWP-04-016 (N), it is unlikely that Mg was present in a metallic state based on process history.

Conclusions

1. Three samples were tested: MC04-147-HBL, MC04-148-HBL, and FBL-SWP-04-016-HBL (N). All three dissolved in 8 M HNO_3 / 0.1 M KF leaving small amounts of solid residue. For all three samples, a four-hour dissolution time at 90-100°C is expected to reduce the plutonium concentrations in the residual solids to trace levels.
2. Sample MC04-147 did not generate hydrogen gas when dissolved in 8 M HNO_3 / 0.1 M KF at 90-100°C.
3. Sample MC04-148 is a true Cabinet 6-8 sweeping and generated modest amounts of hydrogen gas. This sample was similar to sample MC04-147 in terms of crystalline phase composition and elemental content. However, the somewhat higher gas generation and presence of hydrogen and nitrogen dioxide indicate some differences in the samples. The dissolution of UO_2 in this sample can account for all of the hydrogen generated during dissolution.
4. Sample FBL-SWP-04-016-HBL (N) is a true cabinet sweeping and generated a small amount of gas when dissolved in 8 M HNO_3 / 0.1 M KF. Hydrogen was detected in the some of the gas samples. The solids and elemental contents were similar to samples MC04-147 and -148.
5. Dissolution of FBL-SWP-04-016-HBL (N) in 8 M HNO_3 produced a very small amount of gas while maintaining nearly complete dissolution of solids. Apparently, the CaF_2 in the sample dissolves and provides sufficient fluoride to support dissolution of other components.

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Appendix A

Table A-1. Dissolver Product Solution Compositions

Analyte	MC04-147-HBL		MC04-148-HBL [†]		FBL-SWP-04-016 (N)	
	mg/L	wt% in sample	mg/L	wt% in sample	mg/L	wt% in sample
Ag	2.11	0.017	4.01	0.013	1.05	5.2E-03
Al	83.5	0.66	164	0.55	170	0.85
B ^{††}	69.3	0.54	46.6	0.16	65.6	0.33
Ba	3.04	0.024	7.33	0.025	1.71	0.009
Be	0.054	4.2E-04	0.229	7.7E-04	0.037	1.8E-04
Ca	319	2.5	1080	3.6	1390	6.9
Cd	1.61	0.013	2.00	0.007	0.893	4.5E-03
Ce	67.7	0.53	64.7	0.22	40.9	0.20
Cr	23.2	0.18	13.8	0.046	7.53	0.038
Cu	20.0	0.16	35.7	0.12	18.0	0.090
Fe	130	1.0	409	1.4	259	1.3
Gd	3.36	0.026	4.92	0.017	1.74	0.009
K ^{***}	3400	--	3290	--	3890	--
La	9.47	0.074	12.6	0.042	6.13	0.031
Li	0.634*	0.005	2.94	0.010	1.05	0.005
Mg	1820	14.3	1750	5.9	2580	12.9
Mn	4.97	0.039	5.98	0.020	3.26	0.016
Mo	25.2	0.20	26.5	0.089	20.9	0.10
Na	84.2**	0.66	270	0.91	119	0.59
Ni	99.6	0.78	20.0	0.067	24.2	0.12
P	9.15*	0.072	15.5	0.052	<7.51	<0.038
Pb	6.68	0.052	20.9	0.070	6.15	0.031
S	31.0	0.24	35.8	0.12	24.7	0.12
Sb	7.76	0.061	11.1	0.037	7.56	0.038
Si ^{††}	675	5.3	639	2.1	786	3.9
Sn	16.1	0.13	41.1	0.14	16.0	0.080
Sr ^{†††}	0.8	0.006	--	--	2.2	0.011
Ti	0.738	0.006	9.81	0.033	5.56	0.028
U	18.1	0.14	4570	15.4	10.6	0.053
V	15.9	0.12	15.5	0.052	34.7	0.17
Zn	8.40	0.066	14.5	0.049	11.5	0.057
Zr	2.24	0.018	29.4	0.099	2.04	0.010

Method accuracy reported as +/- 10% unless otherwise noted. * Quality Control indicates accuracy is +/- 11%. ** Quality Control indicates accuracy is +/- 15%.

*** K levels attributed to 0.1 M KF dissolver solution.

[†] Note: Interelement correction for U applied to all elements (except U).

^{††} Values may be elevated due to effect of fluoride on glassware.

^{†††} Sr levels measured by ICP-MS.

Table A-2. Additional Dissolver Product Solution Contents

Analyte	MC04-147-HBL		MC04-148-HBL [†]		FBL-SWP-04-016 (N)	
	mg/L	wt% in sample	mg/L	wt% in sample	mg/L	wt% in sample
As	0.04*	0.0001	0.04*	0.0001	0.05**	0.0002
Hg	<0.220	<0.0005	<0.110	<0.0003	<0.110	<0.0004
Se	<0.110	<0.0003	<0.055	<0.0002	<0.055	<0.0002

*Prior to the As analysis, samples MC04-147-HBL and MC04-148-HBL were combined at a 3:1 volumetric ratio. Reported values assume identical concentration in each sample.

** Prior to the As analysis, sample FBL-SWP-04-016 (N) was blended with other samples. As such, the reported value represents a maximum – the actual As content in the sample may be less than but is not more than 0.05 mg/L.