Experimental Results of Calcine Dissolution Studies Performed During FY-94,95.

**K. N. Brewer
A. L. Olson
W. S. Roesener
J. L. Tonso**

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**Idaho National Engineering Laboratory
Waste Management Technologies
Lockheed Martin Idaho Technologies Company
Idaho Falls, Idaho 83415**

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ABSTRACT

Calcine dissolution studies were performed in FY-94,95 in order to extend the knowledge of dissolution and to obtain information necessary for scale-up design and operation.

Experiments reported in this document were performed with non-radioactive and actual calcines to generate qualitative data regarding: a) calcine dissolution rates, b) undissolved solids settling characteristics, c) undissolved solids heel formation, and d) chemical treatments for undissolved solids heel dissolution.

The goal of this work was to achieve complete calcine dissolution, or to determine conditions that would result in the maximum calcine dissolution. Small scale laboratory experiments (test-tube dissolutions) and a bench scale dissolver set-up were used in the effort.

Results from this work show the bulk of the undissolved solids to settle at a rate of > 9 inches per second when the baseline dissolution parameters are used. Baseline dissolution parameters were 100 grams of calcine being dissolved in 1 L of 5 M HNO_3 at $> 90^\circ \text{C}$ while the solution is being vigorously and constantly mixed. This work also verified that dissolution is most complete when performed with aggressive mixing. Sequential dissolutions performed with non-radioactive and actual calcine indicate that little undissolved solids heel build-up is expected, and this small heel can be further dissolved by increasing the dissolution time or by adding fresh nitric acid.

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Experimental Results of Calcine Dissolution Studies Performed During FY-94,95

INTRODUCTION

Experiments were performed in FY-94 and 95 to identify alternative processing methods for ICPP radioactive wastes. Currently, investigations of several separation schemes are in progress to examine the potential of different methods for partitioning dissolved calcine into high- and low-activity fractions. The proposed separation techniques require that solid calcine be dissolved in an aqueous nitrate solution prior to processing.

Two calcine types make up the vast majority of the ICPP inventory. These are classified as zirconium and aluminum type calcines. Both calcine types will require dissolution before any radionuclide partitioning can occur. Approximately 99 wt% of both calcine types is non-radioactive material such as metal oxides, calcium fluoride, and nitrate compounds [1]; therefore, significant high-activity volume reductions can be achieved by separating the radioactive elements from the non-radioactive constituents.

Herbst et al., performed a dissolution study using simulated calcine to determine the important parameters that affect calcine dissolution [2]. Simulated calcine is non-radioactive calcine generated in one of the pilot-plants located at the ICPP. The calcine is physically and chemically similar to the actual high-activity waste calcine. The effects of acid concentration, volume of acid to calcine mass ratio, temperature, mixing, and time on the dissolution rate and completeness were studied. A baseline dissolution process developed for zirconium calcine types was characterized by the use of hot ($>90^{\circ}\text{C}$) 5 M HNO_3 at 1L per 100 grams of calcine under constant and vigorous mixing. Initial results of the dissolution experiment showed that greater than 90 wt% of the simulated calcines tested will dissolve after 30 minutes under these conditions [2].

This report documents results from several dissolution experiments performed in test tubes and a bench-scale dissolver using simulated and actual calcine. The purpose of this report is to summarize the results of these experiments. The experiments were intended to provide an understanding of a) dissolution rates, b) settling characteristics, c) heel formation, and d) effects of chemical treatment of heels.

The bench-scale process will be used to extend the knowledge of dissolution and to assist in scale-up to pilot plant operation. To properly choose equipment for the bench-scale process, it is desirable to know the settling characteristics of the undissolved calcine solids. The settling characteristics will also be used in the design of the solid/liquid separation units that are needed to produce a feed stream for downstream separations processes.

EXPERIMENTAL

Sedimentation Studies

Dissolved, simulated zirconium calcine from Pilot Plant Run 74 [2] was used to determine the sedimentation rate of undissolved solids resulting from the dissolution of the calcine material. A 100 gram calcine sample, comprised of 80 grams product and 20 grams of fines, was dissolved in 1 L of hot 5 M HNO_3 for 30 minutes to provide feed material. Dissolution took place in a beaker that was placed on a hot plate. A stir bar was used to mix the solution.

The experimental apparatus shown in Figure 1 was used to measure sedimentation rates. It consisted of a 0.5-inch ID glass column on a base. The column was fitted with a drain and glass stopcock 3 inches from the bottom. The top of the column is marked approximately 9.3 inches above the stopcock and drain so that 30-mL samples could be collected from each experiment. A sample was obtained after a specified time by draining the column via the stopcock.

The undissolved solids concentration in dissolved calcine product vs. settling time was measured by filling several sedimentation columns to the 30 mL mark with well mixed, and assumed to be homogeneous, solution at the completion of dissolution of the feed material. Solution in the columns was drained into beakers after the specified sedimentation times were met. These times ranged from 1 to 8,640 minutes. These solutions were filtered through 542 Whatman ashless filter paper. Each beaker was rinsed with water several times and the solution poured over the appropriate filter to ensure quantitative transfer and collection of solids. The mass of solids was determined by heating the filter paper and retained solids at 500°C for two hours in a tared ceramic petri dish. The dish was then cooled to room temperature, re-weighed, and the net solids weight obtained. Two other sedimentation tests were performed in the same manner, but with 30 grams of Run 74 product plus 20 grams of Run 74 fines for feed material to dissolve.

Agitation Studies

Five different levels of agitation were tested using 1 gram of zirconium type calcine (Run 74 pilot plant calcine). The 1 gram of calcine was prepared with 3 parts calcine product plus 1 part calcine fines. All experiments were performed in 25-mL test tubes at 90°C (water bath) in 5 M HNO_3 for 30 minutes.

Figure 1. Calcine sedimentation experimental apparatus.

Agitation studies were performed to determine how mixing intensity affects the rate of calcine dissolution. The specific mixing modes that this study focused on were gas sparge rings and fluidic pumps. Sparge rings have been widely used to mildly agitate liquid systems but are only effective in suspending particles with very low settling velocities. Fluidic pumps have been used to gently circulate liquid as well as move particles through a solution [3]. The mixing conditions that would result from these types of agitators were simulated in the lab and both qualitative and quantitative observations were made to determine their effectiveness regarding the calcine dissolution process. Fluidic pump agitation was simulated with a glass Pasteur pipette, while a stir bar was used to simulate agitation from a sparge ring, propeller, or gentle action fluidic pump, depending on the stir bar speed setting.

The techniques used to simulate the five levels of agitation tested are described below.

1. No agitation during dissolution (control).
2. Vigorous fluidic pump agitation was simulated by filling a Pasteur pipette with dissolved calcine solution from the upper fluid level in the test tube. The filled pipette was then lower into the test tube to approximately 1 inch from the bottom. Solution was forcefully expelled from the test tube in order to suspend the solids.
3. Gentle sparge ring or fluidic pump agitation was simulated using a slow stir bar speed. This slight particle agitation caused solution to swirl above the particles but did not cause significant particle suspension.
4. Combined fluidic pump and sparge ring agitation was simulated by using the Pasteur pipet and stir bar as described above. The pipet was used every 15 seconds to suspend the particles. This fluidic pump/sparge ring coupling allows for gentle agitation of the solution and vigorous agitation of the solids.
5. Aggressive agitation was simulated using a stir bar operated at a high speed. This resulted in constant particle movement in solution. This level of mixing simulated a propeller or perhaps very vigorous fluidic pump mixing.

All dissolution experiments were performed in triplicate which are designated by A, B, and C runs. Percent dissolutions were determined by filtering the solutions through Whatman 542 ashless filter paper. The filters were rinsed 5 times with water, placed in a tared ceramic petri dish, and burned at 500° C for two hours. The petri was allowed to cool to room temperature and re-weighed to obtain a net solids weight. Percent calcine dissolution was obtained by:

$$\left[1 - \frac{UDS\ wt}{Initial\ calcine\ wt}\right] \times 100 = \% \text{ calcine dissolution}$$

Sequential Batch Dissolutions

Sequential Batch Dissolutions with Simulated Calcine

Sequential batch dissolutions were performed with non-radioactive calcine simulant to determine the extent calcine dissolution, heel build-up, and sedimentation characteristics. One to two kg batches of calcine were dissolved in 5 to 25 liters of 5 M nitric acid at 90 to 95 °C for 30 to 60 minutes. The "acid volume-to-calcine mass" ratio was adjusted to obtain a final acid concentration in the range of 1 to 3 M. After 30 to 60 minutes, the dissolver product was transferred to a settling tank where it was allowed to settle for 1 to 6 hours. The supernatant was drawn off as the final product, while the bottoms were flushed back to the dissolver tank and combined with the next batch. The supernatant from each batch was combined and retained for future testing. The bottoms from the final batch were flushed to a small plastic bottle and used in

additional tests performed to determine the appropriate heel dissolution chemistry (discussed later).

Figure 2 is a schematic describing the bench-scale dissolver set-up. A specified volume of distilled water was added to the dissolver vessel (depending on the calcine mass being dissolved). A specified calcine mass was then added via the hopper to the dissolver. The recirculation pump was started, followed by acid addition. Enough concentrated acid was added to result in an initial acid concentration of 5.0 M HNO_3 . The immersed electric heater was started as well as the impeller. Dissolution took place by operating the impeller for a specified time at a set temperature, then the impeller and heater were shut-off. Contents of the dissolver vessel were drained to the settling tank and a specified volume of water was sprayed into the dissolver vessel for rinsing which was also drained into the settling vessel. Water was then used to flush the four side ports of the settling tank, these ports are located at 2", 1.5", 1", and 0.5" above the bottom of the settling tank. The solids were allowed to settle in the settling tank for a given time. Calcine was added to the dissolver vessel via the hopper for the next batch, while the solids were settling. The product pump was used to transfer solution from the 2" port after a specific settling time had elapsed. Dissolver product was progressively taken over a period of time from each of the ports by collecting it into bottle and visually examining the solution for solids. The settling tank bottoms were pumped back to the dissolver vessel when a cloudy sample was observed or when the last port (0.5") was sampled, unless this was the last batch. The bottoms were collected by pumping them to a bottle, the vessel sprayed down with water, and the water rinse collected in the same bottle if this was the last batch.

Sequential Dissolutions with Actual Calcine

A series of sequential batch dissolutions were performed with actual aluminum and zirconium calcine types. These calcines were generated during the New Waste Calcination Facility (NWCF) campaign H3. Since the NWCF calcines are intensely radioactive, characterizing them by direct chemical analysis is very difficult (if not impossible). A. L. Olson has developed an Excel spread sheet which predicts calcine composition [4]. Blend compositions, which include actual waste composition, as well as boric acid and calcium nitrate additions, were entered into this spread sheet so that compositions of both calcine types could be obtained. Calculated compositions from this spread sheet are shown in Table 1.

Dissolution experiments were conducted in a shielded hot cell located at the Remote Analytical Laboratory (RAL). One gram of each calcine type was placed in a 25 mL glass test tube. The test tube contained a small stir bar to facilitate mixing. A known amount of calcine was added to the test tube by placing approximately 1 gram of calcine in a tared weighing boat. A gross weight of the boat was obtained after calcine addition. The calcine was then poured into the tared test tube. The test tube and boat were weighed again after calcine transfer. Agreement between weight loss in the weighing boat and weight gain in the test tube assured complete calcine transfer.

Figure 2. Bench-scale calcine dissolution set-up.

Table 1. Calculated aluminum and zirconium calcine compositions prior to dissolution.

Compound	Aluminum Calcine	Zirconium Calcine
	wt%	wt%
NaNO ₃	0.14	7.96
KNO ₃	0.13	1.31
CaF ₂	0.04	33.8
CaO	26.0	17.9
K ₂ SO ₄	0.06	0.58
Na ₂ SO ₄	0.01	1.96
NaCl	0.01	0.20
NaAlO ₂	0.02	0.70
NaBO ₂	0.02	0.56
Al ₂ O ₃	71.4	12.1
B ₂ O ₃	1.15	3.22
Fe ₂ O ₃	0.54	0.39
ZrO ₂	0.05	15.0
CdO	0.01	2.87
Feed Blend UDS	0.49	1.38

Ten mLs of 5.0 M HNO₃ were added to the test tube after the calcine weight was obtained. The calcine/acid mixture was then placed in a simmering water bath (>90° C) heated by a hot plate which was also used to stir the solution. Calcine dissolution occurred for 45 minutes under the heating and mixing conditions after which the test tube was removed from the water bath and set aside to allow the undissolved solids (UDS) to settle. These UDS settled quickly; therefore, settling times of approximately 30 minutes were used.

Solution was drawn from the test tube after the solids had settled using a plastic dispenser. Care was taken not to re-suspend the solids during this step. The drawn solution was dispensed into a syringe having a 0.45 micron acro disc filter on the end. The weight of this acro disc filter was known. Approximately 9 mLs of the dissolved calcine solution was drawn from the test tube without re-suspending or drawing up the UDS. The dissolved calcine solution was forced through the filter into a collection vial using the syringe. Calcine and test tube weighing, calcine transfer, acid addition, and dissolution steps mentioned above were repeated 10 times for both calcine types, resulting in 10 sequential batch dissolutions. These sequential batch dissolutions were performed in triplicate with the zirconium calcine while only one experiment was performed with the aluminum calcine. These experiments were performed independently, but simultaneously.

Isopropyl alcohol was pushed through each acro disc filter to remove water after the dissolver product was filtered. The acro discs were air dried for 24 hours and weighed again. Each time, the acro disc tare weight matched its weight after filtration, indicating UDS were not lost from the test tube during solution transfer.

Each filtered dissolver product was sampled for analysis. Typically, 0.1 mL of this solution was diluted with 20 mL of 3% HNO₃ and brought out of the RAL hot cell for analysis. This dilution was required because of the intense radioactivity of the dissolved calcine solutions. The remaining dissolver product was transferred to a bottle which contained a composite of each dissolution series. The composite solutions, which contained the 10 fractions from each sequential dissolution experiment, were sampled after the 10th addition. The solids remaining after 10 sequential dissolutions were also weighed and sampled for analysis.

Heel Dissolutions

Bottoms from the batch dissolution study, where 10 sequential batch dissolutions of Run 74 were performed, were filtered through 542 Whatman filter papers. The filtrates were discarded and the solids on the filter were rinsed thoroughly with distilled water. The rinsed solids were placed in a tared crucible and placed in an oven at 500° C for two hours. The crucible was allowed to cool after burning the filter paper and re-weighed. The mass of undissolved solids was recorded, then divided into specified portions. One of the portions was analyzed by X-ray diffraction spectroscopy, other portions were re-dissolved in beakers using: specific acids, acid-to-solids ratios, and specific times.

Heel dissolutions were performed by placing a measured heel weight in a beaker and attempting dissolution by one of several reagents using a stir bar to provide constant and vigorous mixing. All heel dissolutions were performed at $>90^{\circ}\text{C}$ and heating was applied by hot plate. The dissolution time was not started until the solution reached 90°C ; therefore an undetermined lag time was systematically incorporated into these dissolutions. The beaker was removed from the hot plate after the specified dissolution time was met and allowed to cool to room temperature before filtering. The solution was filtered through Whatman 542 ashless filter paper after cooling, and the filter was rinsed 5 times before ashing to remove any entrained residue. The filter paper was placed in a pre-weighed crucible, put in a oven for two hours at 500°C , cooled to room temperature, and weighed again to obtained solids weight.

RESULTS

Sedimentation

Data and results from three sedimentation experiments using Run 74 zirconium type calcine are shown in Tables 2-4. Figure 3 graphically shows the solids concentration in the 30 mL samples from each of the three experiments. The bulk of the calcine particles settle at a high rate of >9 inches per hour, as the results shown in Figure 3 indicate very little solids remain unsettled after 60 minutes.

Table 2. First sedimentation experiment performed with Run 74 calcine (80 g product plus 20 g fines dissolved in 1000 mL).

Sedimentation Time (Min)	Crucible Tare Wt. (grams)	Crucible Gross Wt. (grams)	Total Solids Wt. in 30 mLs (grams)	Solids Concentration (g/L)
1	23.26668	23.55970	0.29302	9.767
5	26.75108	27.04289	0.29181	9.727
10	23.78657	24.19864	0.41207	13.74
15	23.16785	23.60055	0.43270	14.42
20	23.12673	23.50872	0.38199	12.73
30	23.82895	24.18213	0.35318	11.77
45	25.83797	26.13663	0.29866	9.955
60	23.97688	24.20435	0.22747	7.582
120	24.31109	24.52810	0.21701	7.234
300	25.40591	25.62916	0.22325	7.442
600	22.49459	22.70679	0.21220	7.073
1452	22.81317	23.00214	0.18897	6.299
>7200	22.86765	23.30162	0.43397	14.47
>7200	20.83960	21.12326	0.28366	9.455
>7200	23.99203	24.25617	0.26414	8.805
>8640	25.83807	26.92634	1.08827	36.28

Solid weights obtained in the first sedimentation experiment appeared to be coated with filter paper; therefore these weights and subsequent solid concentrations may be erroneously high. The lack of a trend in the data shown in Table 2 would indicate incomplete filter ashing may have occurred. Inadequate rinsing of the filtered solids may have also resulted in abnormally large solids recovery. Solid concentrations for the >7200 and >8640 minute tests, listed in Table 2, are not shown in Figure 3 because of the lack of confidence in these data.

Table 3. Second sedimentation experiment performed with Run 74 calcine (30 grams of product plus 20 grams of fines dissolved in 500 mL of 5 M HNO₃).

Sedimentation Time (Min)	Crucible Tare Wt. (grams)	Crucible Gross Wt. (grams)	Total Solids Wt. in 30 mLs (grams)	Solids Concentration (g/L)
1	26.75006	26.85040	0.10034	3.345
5	23.26420	23.35007	0.08587	2.862
10	23.78050	23.86911	0.08861	2.954
15	22.49611	22.56688	0.07077	2.359
20	23.98261	24.00208	0.01947	0.649
30	23.81764	23.86774	0.05010	1.670
45	25.40840	25.43305	0.02465	0.822
60	25.83890	25.85000	0.01110	0.370
120	23.16275	23.16907	0.00632	0.211
300	23.98243	23.99384	0.01141	0.380
540	24.07058	24.07728	0.00670	0.223
1440	22.86500	22.86915	0.00415	0.138

Table 4. Third sedimentation experiment performed with Run 74 calcine (30 grams product plus 20.1 grams fines dissolved in 500 mLs of 5 M HNO₃).

Sedimentation Time (Min)	Crucible Tare Wt. (grams)	Crucible Gross Wt. (grams)	Total Solids Wt. in 30 mLs (grams)	Solids Concentration (g/L)
1	24.31317	24.44105	0.12788	4.263
5	23.98060	24.10241	0.12181	4.060
10	26.74908	26.85254	0.10346	3.449
15	22.81437	22.90286	0.08849	2.950
20	25.40839	25.48658	0.07822	2.607
30	23.98455	24.03375	0.04920	1.640
45	24.07013	24.10543	0.03530	1.177
60	23.16037	23.17115	0.01078	0.359
120	23.81595	23.82215	0.00620	0.207
300	23.26028	23.26497	0.00469	0.156
600	26.74987	26.75416	0.00429	0.143
1440	25.40898	25.41257	0.00359	0.120

Figure 3. Solids concentration in each sample from the three sedimentation experiments.

Particle sizes were not measured in these tests; therefore, settling rate as a function of particle size cannot be reported quantitatively. However, settling rates can be qualitatively determined by assuming particle sizes and using the equation:

$$U_t = \frac{g \cdot D_p^2 \cdot (\rho_p - \rho)}{18 \cdot \mu}$$

where g is the gravitational force (32.2 ft/sec), D_p is the particle size (ft), ρ_p is the particle density which can be between 168 – 187 lbs/ft³, ρ is the final liquid density (75 lbs/ft³), and μ is the viscosity which is 7.41×10^{-4} lb ft⁻¹ sec⁻¹ for 5 M HNO₃ at 20 °C.

Several settling rates were calculated by assuming a particle size, D_p . These calculated settling rates are listed in Table 5. The settling rate for the 10 micron particle, shown in Table 5,

is 11 in/hr. Data shown in Figure 3 indicates the sedimentation to level off or become extremely slow after 60 minutes; therefore, an assumption can be made that most of the particles that settled in the actual experiments were ≥ 10 microns since they settled at a rate of ≥ 9 in/hr. Very little change in the mass of UDS collected was observed between 60 and 1440 minutes (Tables 3 and 4), which indicates that very slow setting rates can be expected for the smaller particles.

This sedimentation data is very difficult to interpret because it is purely qualitative. Particle sizes were not measured in the filtrate, nor the collected solids, and viscosity measurements were not performed. The particle sizes were probably changing during the sedimentation experiments which adds to the difficulty of determining settling rates as a function of particle size. The smaller particles (< 1 micron) may take several hours, if not days, to settle. Therefore, a more rigorous evaluation of the UDS settling rates must be performed before recommended dissolver and/or settling tank designs are made.

Table 5. Calculated settling rates for various calcine particle sizes in 5 M HNO₃ at 20 °C.

D_p (microns)	D_p (ft)	U_t (in/hr)	U_t (ft/hr)
1	3.28×10^{-6}	1.0×10^{-1}	9.4×10^{-3}
10	3.28×10^{-5}	1.1×10^1	9.4×10^{-1}
100	3.28×10^{-4}	1.1×10^3	9.4×10^1
200	6.56×10^{-4}	4.4×10^3	3.7×10^2
500	1.64×10^{-3}	2.8×10^4	2.3×10^3
1000	3.28×10^{-3}	1.2×10^5	9.7×10^3

Agitation

Results from the agitation experiments are shown in Table 5 while Figure 4 graphically depicts the average dissolution of each agitation level. These results are also qualitative because there was the agitation level was not quantified in order to relate it to mass transfer.

Calcine particles in the level 4 experiment settled quickly to the bottom of the test tube at the start of dissolution. However, towards the end of the dissolution, the particles became smaller and lighter which allowed for longer suspension times.

Table 6. Initial calcine weight, UDS weight, percent calcine dissolution, and average dissolution for the 5 agitation levels tested.

Agitation Level	Initial Calcine Wt. (grams)	UDS Wt. (grams)	Percent Dissolution	Average Percent Dissolution
1A	1.0091	0.5558	44.92	45.95 $\pm$ 3.73
1B	1.0116	0.5782	42.84	
1C	1.0213	0.5098	50.08	
2A	1.0119	0.3564	64.78	57.46 $\pm$ 11.1
2B	1.0123	0.5601	44.67	
2C	1.0766	0.3991	62.93	
3A	1.0237	0.3410	66.69	66.89 $\pm$ 3.35
3B	1.0315	0.3750	63.65	
3C	1.0023	0.2954	70.53	
4A	1.0035	0.0846	91.57	94.32 $\pm$ 2.53
4B	1.0341	0.0357	96.55	
4C	1.0112	0.0520	94.86	
5A	1.0133	0.0452	95.54	94.67 $\pm$ 0.82
5B	1.0168	0.0619	93.91	
5C	1.0144	0.0552	94.56

Figure 4. Percent dissolution as a function of agitation level.

Sequential Batch Dissolutions with Bench-scale Set-up

Nine sequential batch dissolutions were performed in the bench-scale dissolution set-up. The undissolved solids were sluiced back into the dissolver after settling for 2 hours in the settling tank. Table 6 contains data taken during this experiment. A total of 187 L of dissolver product were generated from this experiment having a final acid concentration of 3.48 M. A total of 20 kg of calcine were used in this test and 98% overall dissolution of the 20 kg was achieved. Approximately 89% of the 3.7 kg used in the last batch was dissolved and typically 85% to 90% dissolution was achieved for each batch. These percent dissolutions were estimated from the difference between the initial and final acid molarities (moles of acid consumed), then assuming stoichiometric amounts of calcine dissolved based on the composition shown in Table 1.

Table 7. Data from sequential batch dissolution experiment.

Batch #	Wt. Calcine (kg)	Initial Acid Volume (L)	Initial Acid Molarity	Final Acid Volume (L)	Final Acid Molarity
1	2.3	38	3	38	2.1
2	2	17	5.7	17	2.95
3	2	22	5.3	21.5	2.8
4	2	19	6.1	17	4.8
5	1.7	17	5.3	14	4.4
6	2	15	3.7	13	2.3
7	2.2	16	6.9	15	4.5
8	2.1	20	7.3	21	5.9
9	3.7	30	5.1	30	3.2

Sequential Batch Dissolutions with Actual Calcine

Zirconium Calcine

Data obtained from samples taken during zirconium calcine dissolutions are listed in Tables 7 - 15. The test tube was accidentally dropped after the 4th dissolution of the 3rd series. Some UDS were lost at this point of the experiment; therefore, final UDS weight is not accurate. Percent dissolutions for the 1st and 2nd series are 98.7% and 97.9%, respectively, resulting in 1.3% and 2.1% overall UDS in these two dissolution series. Percent dissolution of the last batch for the 1st and 2nd dissolution series were 86.2% and 79.1% respectively (determined by: $\{1 - \text{final UDS wt} / \text{final batch wt}\} \times 100$).

X-ray diffraction analysis of the final UDS from each dissolution series indicate the solids to be primarily calcium stabilized zirconia, $\text{Ca}_{0.15}\text{Zr}_{0.85}\text{O}_{1.85}$.

Table 8. First zirconium calcine dissolution series: aqueous analysis after each dissolution.

Batch	Calcine wt	Gross alpha ¹	Sr-90 ²	Eu-154 ³	Eu-155 ⁴	Cs-134 ³	Cs-137 ³	Co-60 ³
	Grams	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL	Dps/mL	Dps/mL
1	1.125	4.37E+04	1.18E+07	7.93E+04	ND	5.63E+04	7.34E+06	3.18E+03
2	0.903	4.83E+04	1.06E+07	7.09E+04	ND	4.05E+04	5.64E+06	4.08E+03
3	0.904	7.97E+04	9.81E+06	9.05E+04	3.15E+04	5.00E+04	6.83E+06	2.98E+03
4	1.031	6.94E+04	9.89E+06	8.57E+04	2.69E+04	5.70E+04	7.65E+06	4.64E+03
5	0.97	5.01E+04	1.15E+07	7.77E+04	2.84E+04	4.65E+04	6.26E+06	2.96E+03
6	0.963	6.33E+04	1.21E+07	8.80E+04	1.70E+04	5.40E+04	7.00E+06	ND
7	1.098	6.89E+04	1.18E+07	1.00E+05	2.83E+04	7.46E+04	9.22E+06	3.92E+03
8	1.041	2.25E+05	1.09E+07	1.91E+05	5.63E+04	2.42E+05	9.86E+06	5.13E+04
9	1.031	9.66E+04	1.15E+07	1.11E+05	2.89E+04	6.81E+04	8.90E+06	8.53E+03
10	0.954	5.94E+04	7.16E+06	6.39E+04	1.95E+04	4.64E+04	5.31E+06	3.55E+03
Final	0.132							
UDS								

1. Gross alpha analytical error = +/- 7%

2. Sr-90 analytical error = +/- 0.7%

3. Eu-154, Cs-134, Cs-137, & Co-60 analytical error = 5%

4. Eu-155 analytical error = 10%

ND = No Data

Table 9. Second zirconium calcine dissolution series: aqueous analysis after each dissolution.

Batch	Calcine wt	Gross alpha ¹	Sr-90 ²	Eu-154 ³	Eu-155 ⁴	Cs-134 ³	Cs-137 ³	Co-60 ³
	Grams	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL	Dps/mL
1	0.895	3.76E+04	9.40E+06	7.12E+04	ND	5.10E+04	5.85E+06	3.79E+03
2	1.116	5.77E+04	1.14E+07	7.78E+04	ND	6.14E+04	7.83E+06	3.60E+03
3	0.978	8.04E+04	9.20E+06	1.47E+05	ND	5.50E+04	8.76E+06	ND
4	1.024	5.63E+04	1.15E+07	8.27E+04	3.07E+04	5.08E+04	7.17E+06	2.91E+03
5	0.946	5.70E+04	1.14E+07	9.39E+04	ND	5.98E+04	7.15E+06	ND
6	0.989	6.70E+04	1.09E+07	8.46E+04	2.56E+04	5.58E+04	7.60E+06	3.02E+03
7	1.011	5.89E+04	1.05E+07	7.87E+04	1.96E+04	5.85E+04	7.65E+06	ND
8	0.96	1.33E+05	1.09E+07	ND	ND	1.92E+05	9.25E+06	5.83E+04
9	1.075	1.81E+05	8.80E+06	1.44E+05	4.94E+04	1.99E+05	7.79E+06	5.11E+04
10	0.999	1.48E+05	7.36E+06	1.15E+05	4.29E+04	1.70E+05	6.40E+06	4.58E+04
Final	0.209							
UDS								

1. Gross alpha analytical error = +/- 7%

2. Sr-90 analytical error = +/- 0.7%

3. Eu-154, Cs-134, Cs-137, & Co-60 analytical error = 5%

4. Eu-155 analytical error = 10%

ND = No Data

Table 10. Third zirconium calcine dissolution series: aqueous analysis after each dissolution.

Batch	Calcine wt	Gross alpha ¹	Sr-90 ²	Eu-154 ³	Eu-155 ⁴	Cs-134 ³	Cs-137 ³	Co-60 ³
	Grams	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL
1	0.934	4.34E+04	1.05E+07	7.62E+04	2.95E+04	4.58E+04	6.33E+06	4.30E+03
2	1.041	5.63E+04	1.08E+07	8.53E+04	ND	5.92E+04	7.71E+06	3.26E+03
3	1.063	6.09E+04	1.12E+07	8.86E+04	ND	5.74E+04	7.74E+06	4.07E+03
4	0.914	3.89E+04	8.68E+06	2.84E+02	1.08E+02	1.87E+02	2.44E+04	1.31E+01
5	1.064	6.28E+04	9.47E+06	8.83E+04	3.25E+04	2.76E+04	3.44E+06	3.15E+03
6	0.928	6.37E+04	1.10E+07	8.49E+04	2.89E+04	5.78E+04	7.40E+06	2.70E+03
7	0.963	6.91E+04	1.06E+07	9.35E+04	ND	6.03E+04	7.93E+06	3.73E+03
8	1.046	6.46E+04	1.59E+07	ND	ND	8.39E+04	9.89E+06	ND
9	1.047	1.61E+05	7.89E+06	1.35E+05	4.68E+04	1.60E+05	6.75E+06	4.59E+04
10	0.966	1.51E+05	7.55E+06	1.19E+05	3.15E+04	1.51E+05	6.17E+06	4.28E+04
Final	0.031							
UDS								

1. Gross alpha analytical error = +/- 7%

2. Sr-90 analytical error = +/- 0.7%

3. Eu-154, Cs-134, Cs-137, & Co-60 analytical error = 5%

4. Eu-155 analytical error = 10%

ND = No Data

Bold = data cannot be explained, test tube was dropped and approximately 9 mL of acid and some solids were lost

Table 11. First zirconium series (FS-1 Sample Code).

Analyses	Units	Final UDS	Uncertainty
Al	mg/g	<1.02E+3	NA
B	mg/g	ND	ND
Cd	mg/g	<1.40E+2	NA
Cr	mg/g	<1.74E+1	NA
Fe	mg/g	1.05E+00	3.71E+00
Zr	mg/g	1.53E+02	2.23E+01
Ca	mg/g	3.90E+01	4.12E+00
K	wt%	0.24	NA
Na	wt%	0.4	NA
Cs _{Total}	wt%	<0.02	NA
Sr _{Total}	wt%	7.12E-02	NA
NO ₃ ⁻	g/g	ND	ND
SO ₄ ⁻²	g/g	<4.07E-2	NA
F ⁻	g/g	ND	ND
H ⁺	NA	NA	NA
Eu-154	dps/g	6.51E+08	6.39E+07
Eu-155	dps/g	1.07E+09	1.49E+08
Co-60	pds/g	7.95E+07	1.39E+07
Sr-90	dps/g	1.40E+10	2.86E+08
Cs-134	dps/g	ND	ND
Cs-137	dps/g	4.02E+09	4.12E+08
Pu-238	dps/g	2.28E+06	NA
Pu-239	dps/g	9.45E+04	NA
Am-241	dps/g	1.52E+05	NA
Np-237	dps/g	ND	ND
U _{Total}	mg/g	<1.00E+1	ND
Gross alpha	dps/g	7.10E+08	4.98E+07

NA = Not Available

ND = Not Determined

Table 12. First zirconium series (FL-1 Sample Code).

Analyses	Units	Final Dissolver Product	Uncertainty
Al	g/L	2.10E+01	7.81E-01
B	g/L	1.07E+00	1.41E-01
Cd	g/L	1.87E+00	1.41E-01
Cr	g/L	1.04E-01	6.80E-03
Fe	g/L	4.43E-01	1.08E-02
Zr	g/L	7.03E+00	4.55E-01
Ca	g/L	2.24E+01	5.44E-01
K	ug/mL	3.30E+02	NA
Na	ug/mL	1.90E+03	NA
Cs _{Total}	ug/mL	<4.00E+0	NA
Sr _{Total}	ug/mL	1.16E+02	NA
NO ₃ ⁻	M	4.10E+00	2.32E-01
SO ₄ ⁻²	M	1.21E-02	1.29E-03
F ⁻	M	<1.43E+0	NA
H ⁺	N	1.88E+00	4.16E-02
Eu-154	dps/mL	ND	ND
Eu-155	dps/mL	ND	ND
Co-60	dps/mL	ND	ND
Sr-90	dps/mL	2.17E+09	3.31E+07
Cs-134	dps/mL	ND	ND
Cs-137	dps/mL	5.81E+06	2.35E+05
Pu-238	dps/mL	4.47E+04	NA
Pu-239	dps/mL	4.43E+03	NA
Am-241	dps/mL	1.65E+04	NA
Np-237	dps/mL	ND	ND
U _{Total}	g/L	<1.09E+0	ND
Gross alpha	dps/mL	4.59E+04	4.62E+03

NA = Not Available

Table 13. Second zirconium series (FS-2 Sample Code).

Analyses	Units	Final UDS	Uncertainty
Al	mg/g	<1.76E+3	NA
B	mg/g	ND	ND
Cd	mg/g	<2.41E+2	NA
Cr	mg/g	<2.99E+1	NA
Fe	mg/g	1.34E+01	5.82E+00
Zr	mg/g	7.59E+01	1.27E+01
Ca	mg/g	<5.98E+1	NA
K	wt%	0.21	NA
Na	wt%	0.29	NA
Cs _{Total}	wt%	<0.04	NA
Sr _{Total}	wt%	5.59E-02	NA
NO ₃ ⁻	g/g	ND	ND
SO ₄ ⁻²	g/g	<6.98E-2	NA
F ⁻	g/g	ND	ND
H ⁺	ND	ND	ND
Eu-154	dps/g	1.97E+10	2.83E+09
Eu-155	dps/g	ND	ND
Co-60	dps/g	ND	ND
Sr-90	dps/g	1.19E+10	3.84E+08
Cs-134	dps/g	1.04E+10	7.15E+08
Cs-137	dps/g	5.48E+11	2.14E+10
Pu-238	dps/g	1.25E+06	NA
Pu-239	dps/g	6.22E+04	NA
Am-241	dps/g	8.74E+04	NA
Np-237	dps/g	ND	ND
U _{Total}	mg/g	<1.72E+1	ND
Gross alpha	dps/g	2.72E+08	2.64E+07

NA = Not Available

ND = Not Determined

Table 14. Second zirconium series (FL-2 Sample Code).

Analyses	Units	Final Dissolver Product	Uncertainty
Al	g/L	1.86E+01	7.41E-01
B	g/L	1.01E+00	1.41E-01
Cd	g/L	2.44E+00	1.77E-01
Cr	g/L	9.23E-02	5.99E-03
Fe	g/L	4.07E-01	1.04E-02
Zr	g/L	7.17E+00	4.59E-01
Ca	g/L	2.38E+01	5.62E-01
K	ug/mL	5.00E+02	NA
Na	ug/mL	2.60E+03	NA
Cs _{Total}	ug/mL	4.40E+00	NA
Sr _{Total}	ug/mL	1.34E+02	NA
NO ₃ ⁻	M	3.99E+00	2.31E-01
SO ₄ ⁻²	M	1.66E-02	1.35E-03
F ⁻	M	ND	ND
H ⁺	N	1.93E+00	4.16E-02
Eu-154	Dps/mL	8.43E+04	1.16E+04
Eu-155	Dps/mL	ND	ND
Co-60	Dps/mL	1.73E+04	2.59E+03
Sr-90	Dps/mL	2.28E+09	3.50E+07
Cs-134	Dps/mL	9.58E+04	6.17E+03
Cs-137	Dps/mL	7.67E+06	2.50E+05
Pu-238	Dps/mL	7.92E+05	NA
Pu-239	Dps/mL	2.38E+03	NA
Am-241	Dps/mL	4.30E+03	NA
Np-237	Dps/mL	ND	ND
U _{Total}	g/L	ND	ND
Gross alpha	Dps/mL	1.02E+05	7.34E+03

NA = Not Available

Table 15. Third zirconium series (FS-3 Sample Code).

Analyses	Units	Final UDS	Uncertainty
Al	mg/g	<3.95E+2	NA
B	mg/g	ND	ND
Cd	mg/g	<5.42E+1	NA
Cr	mg/g	<6.72E+0	NA
Fe	mg/g	3.71E+00	1.39E+00
Zr	mg/g	2.78E+01	5.01E+00
Ca	mg/g	<1.34E+1	NA
K	wt%	0.047	NA
Na	wt%	0.094	NA
Cs _{Total}	wt%	<0.007	NA
Sr _{Total}	wt%	1.08E-02	NA
NO ₃ ⁻	g/g	ND	ND
SO ₄ ⁻²	g/g	<1.57E-2	NA
F ⁻	g/g	ND	ND
H ⁺	ND	ND	ND
Eu-154	dps/g	1.20E+09	1.30E+08
Eu-155	dps/g	ND	ND
Co-60	dps/g	ND	ND
Sr-90	dps/g	2.43E+09	7.47E+07
Cs-134	dps/g	ND	ND
Cs-137	dps/g	1.06E+10	5.09E+08
Pu-238	dps/g	4.56E+05	NA
Pu-239	dps/g	2.09E+04	NA
Am-241	dps/g	2.42E+04	NA
Np-237	dps/g	ND	ND
U _{Total}	mg/g	<3.88E+0	ND
Gross alpha	dps/g	9.72E+07	7.35E+06

NA = Not Available
ND = Not Determined

Table 16. Third zirconium series (FL-3 Sample Code).

Analyses	Units	Final Dissolver Product	Uncertainty
Al	g/L	2.53E+01	8.50E-01
B	g/L	1.29E+00	1.44E-01
Cd	g/L	2.75E+00	1.95E-01
Cr	g/L	9.71E-02	6.33E-03
Fe	g/L	4.33E-01	1.07E-02
Zr	g/L	8.59E+00	5.04E-01
Ca	g/L	2.98E+01	6.32E-01
K	ug/mL	3.40E+02	NA
Na	ug/mL	1.80E+03	NA
Cs _{Total}	ug/mL	<4	NA
Sr _{Total}	ug/mL	1.12E+02	NA
NO ₃ ⁻	M	4.95E+00	2.48E-01
SO ₄ ⁻²	M	1.37E-02	1.31E-03
F ⁻	M	<1.43E+0	NA
H ⁺	N	2.29E+00	4.16E-02
Eu-154	dps/mL	ND	ND
Eu-155	dps/mL	ND	ND
Co-60	dps/mL	ND	ND
Sr-90	dps/mL	2.87E+09	3.98E+07
Cs-134	dps/mL	ND	ND
Cs-137	dps/mL	5.55E+06	3.03E+05
Pu-238	dps/mL	4.83E+04	NA
Pu-239	dps/mL	4.05E+03	NA
Am-241	dps/mL	1.44E+04	NA
Np-237	dps/mL	ND	ND
U _{Total}	g/L	<1.09E+0	ND
Gross alpha	dps/mL	6.96E+04	4.88E+03

NA = Not Available

Aluminum Calcine

Data from the aluminum calcine dissolution series are shown in Tables 16 - 18. A series of 5 sequential dissolutions, rather than 10, was performed with this calcine because of the limited quantity available for use. 98.8% of the calcine dissolved after 5 sequential dissolutions, resulting in 1.2% UDS. Analysis of the final UDS were not performed, and only limited analysis of the final composite were performed.

Table 17. Aluminum calcine dissolution series: aqueous analysis after each dissolution.

Batch	Calcine wt	Gross alpha ¹	Sr-90 ²	Eu-154 ³	Eu-155 ⁴	Cs-134 ³	Cs-137 ³	Co-60 ³
	grams	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL	dps/mL
1 to 5	0	No Data for dissolutions 1 through 5; something must have occurred during testing						
6	1.073	2.07E+05	1.08E+07	1.47E+05	5.59E+04	2.23E+05	1.11E+07	7.24E+04
7	0.854	1.75E+05	1.02E+07	1.32E+05	4.55E+04	2.20E+05	9.72E+06	5.63E+04
8	0.962	1.67E+05	9.59E+06	1.41E+05	4.43E+04	2.45E+05	9.26E+06	5.91E+04
9	1.19	1.54E+05	8.59E+06	1.23E+05	4.12E+04	1.66E+05	7.98E+06	5.39E+04
10	1.003	9.93E+04	7.14E+06	9.95E+04	3.79E+04	1.51E+05	6.83E+06	4.27E+04
Final	0.062							
UDS								

1. Gross alpha analytical error = +/- 7%
 2. Sr-90 analytical error = +/- 0.7%
 3. Eu-154, Cs-134, Cs-137, & Co-60 analytical error = 5%
 4. Eu-155 analytical error = 10%
- ND = No Data

Table 18. Aluminum series (FS-4 Sample Code).

Analyses	Units	Final UDS	Uncertainty
Al	mg/g	ND	ND
B	mg/g	ND	ND
Cd	mg/g	<5.86E+1	NA
Cr	mg/g	<7.26E+0	NA
Fe	mg/g	5.79E+00	1.69E+00
Zr	mg/g	<1.72e1	NA
Ca	mg/g	1.62E+01	1.72E+00
K	wt%	0.25	NA
Na	wt%	0.14	NA
Cs _{Total}	wt%	0.008	NA
Sr _{Total}	wt%	ND	ND
NO ₃ ⁻	g/g	ND	ND
SO ₄ ⁻²	g/g	ND	ND
F ⁻	g/g	ND	ND
H ⁺	ND	ND	ND
Eu-154	dps/g	2.17E+09	1.95E+08
Eu-155	dps/g	ND	ND
Co-60	dps/g	1.36E+09	8.24E+07
Sr-90	dps/g	ND	ND
Cs-134	dps/g	1.20E+10	8.68E+08
Cs-137	dps/g	5.68E+11	2.03E+10
Pu-238	dps/g	2.92E+05	NA
Pu-239	dps/g	1.93E+04	NA
Am-241	dps/g	2.82E+04	NA
Np-237	dps/g	ND	ND
U _{Total}	mg/g	ND	ND
Gross alpha	dps/g	ND	ND

NA = Not Available

ND = Not Determined

NOTE: 23.4 mg solids dissolved in 100 mL after fusion

Table 19. Aluminum series (FL-4 Sample Code).

Analyses	Units	Final Dissolver Product	Uncertainty
Al	g/L	ND	ND
B	g/L	ND	ND
Cd	g/L	ND	ND
Cr	g/L	ND	ND
Fe	g/L	ND	ND
Zr	g/L	ND	ND
Ca	g/L	ND	ND
K	ug/mL	4.10E+00	NA
Na	ug/mL	2.00E+03	NA
Cs _{Total}	ug/mL	<4	NA
Sr _{Total}	ug/mL	4.79E+01	NA
NO ₃ ⁻	M	ND	ND
SO ₄ ⁻²	M	ND	ND
F ⁻	M	ND	ND
H ⁺	M	ND	ND
Eu-154	dps/mL	ND	ND
Eu-155	dps/mL	ND	ND
Co-60	dps/mL	ND	ND
Sr-90	dps/mL	7.82E+06	6.52E+04
Cs-134	dps/mL	8.23E-01	5.11E-02
Cs-137	dps/mL	3.55E+01	1.53E+00
Pu-238	dps/mL	1.01E+05	NA
Pu-239	dps/mL	3.29E+03	NA
Am-241	dps/mL	3.46E+04	NA
Np-237	dps/mL	ND	ND
U _{Total}	g/L	ND	ND
Gross alpha	dps/mL	1.35E+05	9.33E+03

NA = Not Available

Heel Dissolution Studies

The composition of the undissolved solids resulting from the 10 sequential batch dissolution study performed with surrogate calcine in the bench scale dissolver set-up is shown in Table 19.

Table 20. Composition of Run 74 undissolved solids.

Compound	Wt% UDS Composition
CaF ₂	19.8
ZrO ₂	25.6
Ca _{0.15} Zr _{0.85} O _{1.85}	35.8
Other oxides and silicates	18.8

Re-dissolving the UDS was performed in pure solutions of HNO₃ using different dissolution times, UDS weight to acid volume ratios, different acids, and acid concentrations. The specific conditions tested and subsequent percent UDS dissolution are shown in Table 20.

A portion of UDS was taken and dissolved sequentially with a combination of sodium fluoride (NaF) in nitric acid, followed with aluminum nitrate in nitric acid. Another portion of the UDS heel was taken and sequentially dissolved in nitric acid, followed by an aluminum nitrate in nitric acid, sodium fluoride in nitric acid, and ammonium fluoride in nitric acid dissolutions. Results from these sequential heel dissolutions are shown in Tables 21 and 22.

Several other heel dissolution experiments were conducted using various reagents under different experimental conditions. Results from these tests are shown in Table 23.

Table 21. UDS heel dissolutions using nitric acid, sulfuric acid, and hydrofluoric acid.

Experiment Identification	Acid Tested	UDS Wt. to Acid Volume Ratio grams:mL	Dissolution Time (hours)	Percent Dissolution
1	5.03 M HNO ₃	1:10	2	53.7
6	5.10 M HNO ₃	1:40	8	78.9
8	Two sequential dissolutions with 5.03 M HNO ₃	1:10	4 (total)	79.4
12	10.1 M HNO ₃	1:10	2	73.4
15	15.8 M HNO ₃	1:40	4	74.6
16	9.6 M H ₂ SO ₄	1:10	2	14.9
18	~4 M HF	1:10	2	21.7

Table 22. Sequential UDS heel dissolutions using NaF in HNO₃ and Al(NO₃)₃ in HNO₃.

Experiment Identification	Reagents Tested	UDS Wt. to Acid Volume Ratio grams:mL	Dissolution Time (hours)	Percent Dissolution
7	0.2081 g NaF in 0.2082 10 mL of 5.1 M HNO ₃	1.006 g/10 mL (1:10)	2	69.5
7	0.0712 g NaF in 3 mL of 5.1 M HNO ₃	0.3066 g/3 mL (1:10)	2	25.6
11	4.4 mL of 2.2 M Al(NO ₃) ₃ and 4.8 M HNO ₃	0.2282 g/4.4 mL (1:20)	2	31.9
13	4.65 mL of 0.2 M Al(NO ₃) ₃ and 9.6 M HNO ₃	0.1555 g/4.65 mL (1:30)	2	14.9
Total percent dissolution				86.8

Table 23. Sequential UDS heel dissolutions using HNO₃ only, Al(NO₃)₃ in HNO₃, NaF in HNO₃, and NH₄F in water.

Experiment Identification	Reagents Tested	UDS Wt. to Acid Volume Ratio grams:mL	Dissolution Time (hours)	Percent Dissolution
8	5.1 M HNO ₃	2.01 g/20 mL (1:10)	2	77
8	5.1 M HNO ₃	0.463 g/4.6 mL (1:10)	2	10.4
9	4.0 mL of 0.2 M Al(NO ₃) ₃ and 9.6 M HNO ₃	0.415 g/4.0 mL (1:10)	2	8.2
14	0.306 g NaF in 3.8 mL of 5.1 M HNO ₃	0.3809 g/3.8 mL (1:10)	2	37.9
17	5 mL of 0.1 M NH ₄ F	0.2366 g/5 mL (1:20)	2	14.7
Total percent dissolution				89.9

Table 24. Other UDS heel dissolution tests.

Experiment Identification	Reagents Tested	UDS Wt. to Acid Volume Ratio grams:mL	Dissolution Time (hours)	Percent Dissolution
2a	2.54 g of CaF ₂ in 20 mL of 5.03 M HNO ₃	2.05631 g/20 mL (1:10)	2	-33.5 ^b
3	1.32 M Al(NO ₃) ₃ in H ₂ O	0.5017 g/20 mL (1:40)	2	59.5
4	1.32 M Al(NO ₃) ₃ in 5.1 M HNO ₃	0.50427 g/17 mL (1:34)	2	56.1
10	0.2 M Al(NO ₃) ₃ in 9.6 M HNO ₃	1.01864 g/10 mL (1:10)	2	64.0
10	0.66 M Al(NO ₃) ₃ in 4.8 M HNO ₃	1.02976 g/10 mL (1:10)	2	45.1
19	0.27 g NaF in 10 mL of 4.9 M HNO ₃	1.0141 g/10 mL (1:10)	22	64.0
20	1.08 g of NaF in 10 mL of 4.9 M HNO ₃	1.0246 g/10 mL (1:10)	4	1.1
20	1.08 g of NaF in 10 mL of 4.9 M HNO ₃	1.0246 g/10 mL (1:10)	4	1.3
21	Two steps: 10 mL of 4.9 M HNO ₃ followed by 0.27 g NaF in 10 mL 4.9 M HNO ₃	1.0111g/10 mL (1:10)	8 hours total (4 hours each)	89.4
22	10 mL of 0.6 M HF and 5.0 M HNO ₃	1.003 g/10 mL (1:10)	4	77.2
23	Two steps: 10 mL 4.9 M HNO ₃ followed with 10 mL of 2.4 M HF	1.0173 g/10 mL (1:10)	8 hours total (4 hours each)	97.1
24	Two steps: 10 mL of 4.9 M HNO ₃ followed by 10 mLs 0.6 M HF in 4.9 M HNO ₃	1.0076 g/10 mL (1:10)	2	88.9
25	Two steps: 10 mL of 4.9 M HNO ₃ followed by 1.1 g NaF in 10 mLs of 4.9 M HNO ₃	1.0408 g/10 mL (1:10)	2	59.3

^a UDS from experiment identified as 1 used in this test.

^b Weight increase may be due to incomplete CaF₂ dissolution in HNO₃.

Heel dissolutions were performed using the dissolver product solution in an attempt to further dissolve the undissolved solids. A 1 liter sample of the dissolver product containing the undissolved solids was split into two 500 mL fractions. An assumption was made that the 1 liter sample was homogeneous; therefore, both fractions were representative of the whole. One 500 mL fraction was filtered and the solids weighed in order to obtain percent dissolution (control). The other fraction was heated for an additional two hours then filtered. These experiments were performed in triplicate and provided insight as to the effects of increased dissolution time on reducing the UDS heel. Table 24 shows the results of this experiment.

Table 25. Effects of increasing dissolution time on UDS heel.

Experiment Identification	Solids Concentration g/L
Trial #1	
Control	0.0212
2 additional hours	0.0069
Trial #2	
Control	0.0296
2 additional hours	0.0041
Trial #3	
Control	0.0155
2 additional hours	0.0048

REFERENCES

1. J. R. Berreth, "Inventories and Properties of ICPP Calcined High-Level Waste", WINCO-1050, February 1988.
2. R. S. Herbst, D. S. Fryer, K. N. Brewer, C. K. Johnson, and T. A. Todd, "Experimental Results: Pilot Plant Calcine Dissolution and Liquid Feed Stability Study", INEL-95/0097, February 1995.
3. Perry's Chemical Engineer's Handbook, 6th Edition, p. 13-19.
4. K. N. Brewer, R. S. Herbst, T. J. Tranter, A. L. Olson, T. A. Todd, "Dissolution of Two NWCF Calcines: Extent of Dissolution and Characterization of Undissolved Solids", INEL-95/0098, February 1995.

APPENDIX

Analytical Data from Sequential Dissolutions with Actual Zirconium and Aluminum Calcines

Sample Cross Reference Guide:

FL-1 = Final liquid composite sample from 1st Zr dissolution series. Data is in Table 11.
FL-2 = Final liquid composite sample from 2nd Zr dissolution series. Data is in Table 13.
FL-3 = Final liquid composite sample from 3rd Zr dissolution series. Data is in Table 15.
FL-4 = Final liquid composite sample from only Al dissolution series. Data is in Table 18.

B1-1 through B1-10 = 1st Zr dissolutions series batch samples. Data is in Table 7.
B2-1 through B2-10 = 2nd Zr dissolutions series batch samples. Data is in Table 8.
B3-1 through B3-10 = 3rd Zr dissolutions series batch samples. Data is in Table 9.
B4-6 through B4-10 = Only Al dissolutions series batch samples. Data is in Table 16.

FS-1 = Final solids sample from 1st Zr dissolution series. Data is in Table 10.
FS-2 = Final solids sample from 2nd Zr dissolution series. Data is in Table 12.
FS-3 = Final solids sample from 3rd Zr dissolution series. Data is in Table 14.
FS-4 = Final solids sample from only Al dissolution series. Data is in Table 17.

Cautionary notes:

- All boron data for FS samples are meaningless because a lithium tetraborate fusion was performed on these samples as apart of the necessary analytical sample preparation.
- FS-4 fusion preparation (method # 8000) was performed by fusing 23.4 mg of UDS and dissolving the flux in 100 mLs of HNO₃.

- F I N A L R E P O R T for BATCH DISS

Log Type: ** Technical (CPP 637) **

Report for : W S ROESENER
Mailstop : MS 5213

Log Number : 94-08027
Phone Number : 6-1080

Date Received : Aug 02 1994
Time Received : 11:01

Date completed : May 11 1995
Time completed : 11:19

GWA charged : T35370004

Reviewed by JEAN CASTEEL

MSA mR/hr : HOT

Signature Jean Casteel

Hazard Index : >E7

Lab QC/QA reviewed by

Quality Level : III

Signature N/A

Liquids are 1 g of zirconia calcine dissolved in 10 ml of 5M
HNO3. Solids are straight zirconia calcine.

Analysis	Sample	Method	Analyst	Results
AM-241	FL-1	3201		16477.9+-22591.7 D/S/ML
	FL-2	3201		4301.5+-2279.48 D/S/ML
	FL-3	3201		14443.5+-22591.7 D/S/ML
	FL-4	3201		34587.3+-22591.7 D/S/ML
	FS-1	3201		152162+-58613.1 D/S/G
	FS-2	3201		87365.4+-100641 D/S/G
	FS-3	3201		24159.9+-22634.4 D/S/G
	FS-4	3201		28168.7+-24472.2 D/S/G
Acid	DEC-1A	67015		2.26017+-4.16007E-02 NACID
	DEC-2A	67015		1.90046+-0.041595 NACID
	DEC-2A	67015		1.90155+-0.041595 NACID
	FL-1	67015		1.8843+-4.15955E-02 NACID
	FL-2	67015		1.93448+-4.15942E-02 NACID
	FL-2	67015		1.92097+-4.15945E-02 NACID
Aluminum	FL-3	67015		2.29206+-4.16027E-02 NACID
	FL-1	7101		21.0304+-0.780749 G/L
	FL-1	7101		18.9579+-1.12463 G/L
	FL-2	7101		18.6379+-0.741013 G/L
	FL-2	7101		17.9972+-1.09774 G/L
	FL-3	7101		25.3323+-0.849684 G/L
	FL-3	7101		25.7314+-1.29187 G/L
	FS-1	7101		< 1023.92 MG/G
	FS-2	7101		< 1758.12 MG/G
	FS-3	7101		< 395.404 MG/G
Boron	FL-1	7102		1.06903+-0.141464 G/L
	FL-2	7102		1.00881+-0.140913 G/L
	FL-3	7102		1.29469+-0.143599 G/L
	FS-1	7102		10362.8+-754.775 MG/G
	FS-2	7102		15195.7+-1269.8 MG/G
	FS-3	7102		3722.91+-288.656 MG/G
Cadmium	FL-1	7103		1.86745+-0.141263 G/L
	FL-2	7103		2.43609+-0.177457 G/L
	FL-3	7103		2.75099+-0.194699 G/L
	FS-1	7103		< 140.461 MG/G

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Analysis	Sample	Method	Analyst	Results
Calcium	FS-2	7103		< 241.178 MG/G
	FS-3	7103		< 54.2413 MG/G
	FS-4	7103		< 58.6455 MG/G
	FL-1	7111		22.4101+- .544254 G/L
	FL-2	7111		23.7987+- .561538 G/L
	FL-3	7111		29.8336+- .632055 G/L
	FS-1	7111		39.2274+-4.12132 MG/G
	FS-2	7111		< 59.754 MG/G
	FS-3	7111		< 13.4387 MG/G
	FS-4	7111		16.2052+-1.71211 MG/G
Chromium	FL-1	7104		.103689+-6.80298E-03 G/L
	FL-2	7104		9.22665E-02+-5.9889E-03 G/L
	FL-3	7104		9.71206E-02+-6.33468E-03 G/L
	FS-1	7104		< 17.4002 MG/G
	FS-2	7104		< 29.877 MG/G
	FS-3	7104		< 6.71937 MG/G
	FS-4	7104		< 7.26496 MG/G
Co57 Co60	B2-8	73993		58320+-4808 d/s/ml
	B1-1	73993		3184+-242.1 d/s/sple
	B1-10	73993		3549+-214.6 d/s/ml
	B1-2	73993		4077+-464.8 d/s/ml
	B1-3	73993		2983+-175.2 d/s/ml
	B1-4	73993		4651+-317.3 d/s/ml
	B1-5	73993		2956+-171.4 d/s/ml
	B1-7	73993		3917+-403.7 d/s/ml
	B1-8	73993		51300+-4116 d/s/ml
	B1-9	13993		8529+-902.8 d/s/ml
	B2-1	73993		3793+-384.7 d/s/ml
	B2-10	73993		45810+-2939 d/s/ml
	B2-2	73993		3597+-205.4 d/s/ml
	B2-4	73993		2909+-239.6 d/s/ml
	B2-6	73993		3019+-415 d/s/ml
	B2-9	73993		51120+-5294 d/s/ml
	B3-1	73993		4304+-355.3 d/s/ml
	B3-10	73993		42750+-2510 d/s/ml
	B3-2	73993		3260+-218.4 d/s/ml
	B3-3	73993		4070+-371.2 d/s/ml
	B3-4	73993		13.13+-1.651 d/s/sple
	B3-5	73993		3150+-255.2 d/s/ml
	B3-6	73993		2700+-432.2 d/s/ml
	B3-7	73993		3731+-228.8 d/s/ml
	B3-9	73993		45900+-3099 d/s/ml
	B4-10	83993		42740+-2511 d/s/sple
	B4-6	13993		72390+-3598 d/s/sple
	B4-7	23993		56270+-2323 d/s/sple
	B4-8	73993		59110+-3324 d/s/ml
	B4-9	33993		53870+-1983 d/s/sple
Cs134	FL-2	73993		17250+-2589 d/s/ml
	FS-1	73993		7767+-1358 d/s/ml
	FS-4	73993		318500+-19270 d/s/sple
	B1-1	73993		56340+-3005 d/s/sple
	B1-10	73993		46410+-2770 d/s/ml
	B1-2	73993		40480+-2422 d/s/ml
	B1-3	73993		49950+-2591 d/s/ml
	B1-4	73993		57030+-3137 d/s/ml
	B1-5	73993		46500+-2646 d/s/ml

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Analysis	Sample	Method Analyst Results
	B1-6	73993 53950+-3318 d/s/ml
	B1-7	73993 74620+-3819 d/s/ml
	B1-8	73993 241900+-13310 d/s/ml
	B1-9	13993 68070+-4546 d/s/ml
	B2-1	73993 50990+-3224 d/s/ml
	B2-10	73993 170300+-7957 d/s/ml
	B2-2	73993 61360+-3191 d/s/ml
	B2-3	73993 55020+-4270 d/s/ml
	B2-4	73993 50840+-5500 d/s/ml
	B2-5	73993 59810+-6765 d/s/ml
	B2-6	73993 55770+-2443 d/s/ml
	B2-7	73993 58530+-3757 d/s/ml
	B2-8	73993 191800+-19730 d/s/ml
	B2-9	73993 199100+-9134 d/s/ml
	B3-1	73993 45820+-3584 d/s/ml
	B3-10	73993 150500+-7591 d/s/ml
	B3-2	73993 59210+-4486 d/s/ml
	B3-3	73993 57430+-4771 d/s/ml
	B3-4	73993 187.4+-14.74 d/s/sple
	B3-5	73993 27570+-2388 d/s/ml
	B3-6	73993 57760+-3368 d/s/ml
	B3-7	73993 60320+-3272 d/s/ml
	B3-8	73993 83920+-7314 d/s/ml
	B3-9	73993 159500+-7024 d/s/ml
	B4-10	83993 150900+-6939 d/s/sple
	B4-6	13993 222900+-10400 d/s/sple
	B4-7	23993 220000+-12600 d/s/sple
	B4-8	73993 245300+-10410 d/s/ml
	B4-9	33993 166000+-7444 d/s/sple
	FL-2	73993 95830+-6167 d/s/ml
	FL-4	73993 .8232+- .05112 d/s/ml
	FS-2	73993 590200+-40710 d/s/ml
	FS-4	73993 2.802E+06+-202900 d/s/sple
Cs137	B1-1	73993 7.342E+06+-327900 d/s/sple
	B1-10	73993 5.309E+06+-198600 d/s/ml
	B1-2	73993 5.642E+06+-232000 d/s/ml
	B1-3	73993 6.832E+06+-260500 d/s/ml
	B1-4	73993 7.654E+06+-291600 d/s/ml
	B1-5	73993 6.26E+06+-244800 d/s/ml
	B1-6	73993 7.002E+06+-257800 d/s/ml
	B1-7	73993 9.225E+06+-407300 d/s/ml
	B1-8	73993 9.862E+06+-439700 d/s/ml
	B1-9	13993 8.901E+06+-440900 d/s/ml
	B2-1	73993 5.845E+06+-252700 d/s/ml
	B2-10	73993 6.402E+06+-287200 d/s/ml
	B2-2	73993 7.829E+06+-360400 d/s/ml
	B2-3	73993 8.758E+06+-297000 d/s/ml
	B2-4	73993 7.169E+06+-314000 d/s/ml
	B2-5	73993 7.154E+06+-259300 d/s/ml
	B2-6	73993 7.599E+06+-269200 d/s/ml
	B2-7	73993 7.652E+06+-309100 d/s/ml
	B2-8	73993 9.247E+06+-355700 d/s/ml
	B2-9	73993 7.793E+06+-316900 d/s/ml
	B3-1	73993 6.327E+06+-255200 d/s/ml
	B3-10	73993 6.167E+06+-287800 d/s/ml
	B3-2	73993 7.709E+06+-329200 d/s/ml

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Sample Method Analyst Results

Eu154

B3-3	73993	7.743E+06+-337000 d/s/ml
B3-4	73993	24410+-1016 d/s/sple
B3-5	73993	3.44E+06+-141100 d/s/ml
B3-6	73993	7.404E+06+-324300 d/s/ml
B3-7	73993	7.934E+06+-362600 d/s/ml
B3-8	73993	9.889E+06+-404600 d/s/ml
B3-9	73993	6.752E+06+-254600 d/s/ml
B4-10	83993	6.832E+06+-271000 d/s/sple
B4-6	13993	1.109E+07+-501800 d/s/sple
B4-7	23993	9.719E+06+-403500 d/s/sple
B4-8	73993	9.259E+06+-397500 d/s/ml
B4-9	33993	7.981E+06+-229800 d/s/sple
FL-1	73993	5.812E+06+-234900 d/s/ml
FL-2	73993	7.674E+06+-249600 d/s/ml
FL-3	73993	5.546E+06+-302700 d/s/ml
FL-4	73993	35.45+-1.534 d/s/ml
FS-1	73993	392600+-40270 d/s/ml
FS-2	73993	3.118E+07+-1.217E+06 d/s/ml
FS-3	73993	2.693E+06+-128700 d/s/ml
FS-4	73993	1.328E+08+-4.755E+06 d/s/sple
B1-1	73993	79250+-7574 d/s/sple
B1-10	73993	63850+-4505 d/s/ml
B1-2	73993	70940+-6008 d/s/ml
B1-3	73993	90520+-6331 d/s/ml
B1-4	73993	85690+-5606 d/s/ml
B1-5	73993	77730+-4828 d/s/ml
B1-6	73993	88030+-6104 d/s/ml
B1-7	73993	100200+-9354 d/s/ml
B1-8	73993	190700+-14250 d/s/ml
B1-9	13993	110500+-10180 d/s/ml
B2-1	73993	71210+-5353 d/s/ml
B2-10	73993	115200+-7629 d/s/ml
B2-2	73993	77810+-5901 d/s/ml
B2-3	73993	147400+-12220 d/s/ml
B2-4	73993	82730+-7372 d/s/ml
B2-5	73993	93860+-8229 d/s/ml
B2-6	73993	84640+-5929 d/s/ml
B2-7	73993	78650+-6791 d/s/ml
B2-9	73993	144100+-11460 d/s/ml
B3-1	73993	76240+-6034 d/s/ml
B3-10	73993	119300+-10620 d/s/ml
B3-2	73993	85280+-6223 d/s/ml
B3-3	73993	88630+-6629 d/s/ml
B3-4	73993	283.8+-40.9 d/s/sple
B3-5	73993	88340+-7076 d/s/ml
B3-6	73993	84850+-7779 d/s/ml
B3-7	73993	93500+-7132 d/s/ml
B3-9	73993	135100+-10090 d/s/ml
B4-10	83993	99460+-7149 d/s/sple
B4-6	13993	147400+-15670 d/s/sple
B4-7	23993	131800+-8042 d/s/sple
B4-8	73993	141400+-9454 d/s/ml
B4-9	33993	123300+-7686 d/s/sple
FL-2	73993	84340+-11590 d/s/ml
FS-1	73993	63630+-6241 d/s/ml
FS-2	73993	1.121E+06+-161100 d/s/ml

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Analysis	Sample	Method	Analyst	Results
Eu155	FS-3	73993		304000+-32780 d/s/ml
	FS-4	73993		508100+-45710 d/s/sple
	B1-10	73993		19530+-2366 d/s/ml
	B1-3	73993		31490+-4004 d/s/ml
	B1-4	73993		26950+-3036 d/s/ml
	B1-5	73993		28440+-3296 d/s/ml
	B1-6	73993		16990+-2405 d/s/ml
	B1-7	73993		28330+-4081 d/s/ml
	B1-8	73993		56330+-6341 d/s/ml
	B1-9	13993		28860+-3721 d/s/ml
	B2-10	73993		42880+-6507 d/s/ml
	B2-4	73993		30680+-3649 d/s/ml
	B2-6	73993		25640+-3404 d/s/ml
	B2-7	73993		19620+-3400 d/s/ml
	B2-9	73993		49380+-5224 d/s/ml
	B3-1	73993		29540+-4454 d/s/ml
	B3-10	73993		31480+-3714 d/s/ml
	B3-4	73993		108+-18.05 d/s/sple
	B3-5	73993		32540+-5727 d/s/ml
	B3-6	73993		28910+-4181 d/s/ml
	B3-9	73993		46760+-5871 d/s/ml
	B4-10	83993		37910+-4606 d/s/sple
	B4-6	13993		55850+-6827 d/s/sple
	B4-7	23993		45510+-5139 d/s/sple
	B4-8	73993		44250+-6538 d/s/ml
	B4-9	33993		41190+-4204 d/s/sple
	FS-1	73993		104900+-14560 d/s/ml
Fluoride	FL-1	97093		< 1.42851 MOLAR
	FL-3	97093		< 1.42851 MOLAR
	FS-4	3450		NO SAMPLE LEFT.
GROSSALPHA Gross Alph	B1-1	3450		43746.2+-3024.25 D/S/ml
	B1-10	3450		59399+-4131.53 D/S/ml
	B1-2	3450		48266.8+-3336.68 D/S/ml
	B1-3	3450		79678.3+-5629.7 D/S/ml
	B1-4	3450		69361.4+-4861.98 D/S/ml
	B1-5	3450		50101.1+-3465.48 D/S/ml
	B1-6	3450		63279.6+-4414.44 D/S/ml
	B1-7	3450		68896.7+-4827.63 D/S/ml
	B1-8	3450		225339+-9325.5 D/S/ml
	B1-9	3450		96631.4+-6906.1 D/S/ml
	B2-1	3450		37558.5+-2611.93 D/S/ml
	B2-10	3450		148322+-9325.5 D/S/ml
	B2-2	3450		57740+-4011.35 D/S/ml
	B2-3	3450		80448.7+-5687.36 D/S/ml
	B2-4	3450		56317.4+-3908.7 D/S/ml
	B2-5	3450		57047+-3961.3 D/S/ml
	B2-6	3450		67009.4+-4688.37 D/S/ml
	B2-7	3450		58901.7+-4095.45 D/S/ml
	B2-8	3450		132580+-9325.5 D/S/ml
	B2-9	3450		180818+-9325.5 D/S/ml
	B3-1	3450		43350.9+-2997.32 D/S/ml
	B3-10	3450		151302+-9325.5 D/S/ml
	B3-2	3450		56284.8+-3906.35 D/S/ml
	B3-3	3450		60878.7+-4239.13 D/S/ml
	B3-4	3450		38879.2+-2698.1 D/S/ml
	B3-5	3450		62794.5+-4378.95 D/S/ml

FINAL Report for 94-08027 continued

Analysis	Sample	Method	Analyst	Results
	B3-6	3450		63670.9+-4443.1 D/S/ml
	B3-7	3450		69067.9+-4840.29 D/S/ml
	B3-8	3450		64616.6+-4512.43 D/S/ml
	B3-9	3450		161003+-9325.5 D/S/ml
	B4-10	3450		99329.5+-7110.4 D/S/ml
	B4-6	3450		206829+-9325.5 D/S/ml
	B4-7	3450		175128+-9325.5 D/S/ml
	B4-8	3450		167264+-9325.5 D/S/ml
	B4-9	3450		154603+-9325.5 D/S/ml
	FL-1	3450		45888.8+-4622.5 D/S/ml
	FL-2	3450		102306+-7336.04 D/S/ml
	FL-3	3450		69565.2+-4877.05 D/S/ml
	FL-4	3450		134695+-9325.5 D/S/ml
	FS-1	3450		69433.3+-4867.3 D/S/ml
	FS-2	3450		15478.7+-1504.32 D/S/ml
	FS-3	3450		24610.5+-1855.09 D/S/ml
Iron	FL-1	7105		.442671+-1.08189E-02 G/L
	FL-2	7105		.407158+-1.03703E-02 G/L
	FL-3	7105		.432571+-1.06931E-02 G/L
	FS-1	7105		10.5471+-3.70756 MG/G
	FS-2	7105		13.4054+-5.82597 MG/G
	FS-3	7105		3.71188+-1.39147 MG/G
	FS-4	7105		5.79359+-1.69392 MG/G
LIB FUSION	FS-1	8000		0.00977G/100ML
	FS-2	8000		0.00569G/100ML
	FS-3	8000		0.0253G/100ML
Nitrate	DEC-1A	77074		4.838+- .147196 Molar
	DEC-1A	77074		4.82879+- .147055 Molar
	DEC-2A	77074		4.11318+- .135988 Molar
	DEC-2A	77074		4.10608+- .135877 Molar
	FL-1	77074		4.10044+- .232546 Molar
	FL-2	77074		3.99014+- .230513 Molar
	FL-2	77074		4.00667+- .134314 Molar
	FL-3	77074		4.95294+- .247962 Molar
PU-238	FL-1	3202		44693.7+-53052.6 D/S/ML
	FL-2	3202		79203.2+-7888.34 D/S/ML
	FL-3	3202		48296.7+-53052.6 D/S/ML
	FL-4	3202		101329+-53052.6 D/S/ML
	FS-1	3202		2.28422E+06+-210471 D/S/G
	FS-2	3202		1.25172E+06+-268744 D/S/G
	FS-3	3202		456192+-67165.6 D/S/G
	FS-4	3202		291641+-63791.7 D/S/G
PU-239	FL-1	3203		4425.12+-53052.6 D/S/ML
	FL-2	3203		2377.18+-5352.96 D/S/ML
	FL-3	3203		4049.72+-53052.6 D/S/ML
	FL-4	3203		3290.79+-53052.6 D/S/ML
	FS-1	3203		94516.9+-137642 D/S/G
	FS-2	3203		62186.1+-236338 D/S/G
	FS-3	3203		20882.5+-53152.8 D/S/G
	FS-4	3203		19284.7+-56341.8 D/S/G
Potassium	FL-1	2190		=3.3E+02 UG/ML
	FL-2	2190		=5.0E+02 UG/ML
	FL-3	2190		=3.4E+02 UG/ML
	FL-4	2190		=4.1E+02 UG/ML
	FS-1	2190		=0.24 WT%
	FS-2	2190		=0.21 WT%

FINAL Report for 94-08027 continued

Analysis	Sample	Method	Analyst	Results
	FS-3	2190		=0.047 WT%
	FS-4	2190		=0.25 WT%
Sodium	FL-1	2110		=1.9E+03 UG/ML
	FL-2	2110		=2.6E+03 UG/ML
	FL-3	2110		=1.8E+03 UG/ML
	FL-4	2110		=2.0E+03 UG/ML
	FS-1	2110		=0.40 WT%
	FS-2	2110		=0.29 WT%
	FS-3	2110		=0.094 WT%
	FS-4	2110		=0.14 WT%
Sulfate	FL-1	77168		1.21453E-02+-1.29168E-03 Molar
	FL-2	77168		1.65552E-02+-1.35121E-03 Molar
	FL-3	77168		1.37047E-02+-1.31264E-03 Molar
	FS-1	7168		<0.0406731 G/G
	FS-2	7168		<0.0698377 G/G
	FS-3	7168		<0.0157066 G/G
TOTAL SR	B1-1	3381		1.18E+07+-7.95E+04 D/S/ML
	B1-10	3381		7.16E+06+-6.36E+04 D/S/ML
	B1-2	3381		1.06E+07+-7.60E+04 D/S/ML
	B1-3	3381		9.81E+06+-8.04E+04 D/S/ML
	B1-4	3381		9.89E+06+-7.89E+04 D/S/ML
	B1-5	3381		1.15E+07+-7.92E+04 D/S/ML
	B1-6	3381		1.21E+07+-8.12E+04 D/S/ML
	B1-7	3381		1.18E+07+-7.80E+04 D/S/ML
	B1-8	3381		1.09E+07+-7.62E+04 D/S/ML
	B1-9	3381		1.15E+07+-7.74E+04 D/S/ML
	B2-1	3381		9.40E+06+-7.05E+04 D/S/ML
	B2-10	3381		7.36E+06+-6.29E+04 D/S/ML
	B2-2	3381		1.14E+07+-7.85E+04 D/S/ML
	B2-3	3381		9.20E+06+-7.28E+04 D/S/ML
	B2-4	3381		1.15E+07+-8.00E+04 D/S/ML
	B2-5	3381		1.14E+07+-7.88E+04 D/S/ML
	B2-6	3381		1.09E+07+-7.45E+04 D/S/ML
	B2-7	3381		1.05E+07+-7.57E+04 D/S/ML
	B2-8	3381		1.09E+07+-7.67E+04 D/S/ML
	B2-9	3381		8.80E+06+-6.71E+04 D/S/ML
	B3-1	3381		1.05E+07+-7.54E+04 D/S/ML
	B3-10	3381		7.55E+06+-6.44E+04 D/S/ML
	B3-2	3381		1.08E+07+-7.72E+04 D/S/ML
	B3-3	3381		1.12E+07+-8.18E+04 D/S/ML
	B3-4	3381		8.68E+06+-6.85E+04 D/S/ML
	B3-5	3381		9.47E+06+-7.85E+04 D/S/ML
	B3-6	3381		1.10E+07+-7.85E+04 D/S/ML
	B3-7	3381		1.06E+07+-7.51E+04 D/S/ML
	B3-8	3381		1.59E+07+-1.00E+05 D/S/ML
	B3-9	3381		7.89E+06+-6.28E+04 D/S/ML
	B4-10	3381		7.14E+06+-6.19E+04 D/S/ML
	B4-6	3381		1.08E+07+-7.66E+04 D/S/ML
	B4-7	3381		1.02E+07+-7.31E+04 D/S/ML
	B4-8	3381		9.59E+06+-7.92E+04 D/S/ML
	B4-9	3381		8.59E+06+-6.93E+04 D/S/ML
	FL-1	3381		2.17E+09+-3.31E+07 D/S/ML
	FL-2	3381		2.28E+09+-3.50E+07 D/S/ML
	FL-3	3381		2.87E+09+-3.98E+07 D/S/ML
	FL-4	3381		7.82E+06+-6.52E+04 D/S/ML
	FS-1	3381		1.37E+06+-2.79E+04 D/S/ML

FINAL Report for 94-08027 continued

Analysis	Sample	Method	Analyst	Results
	FS-2	3381		6.75E+05+-2.19E+04 D/S/ML
	FS-3	3381		6.16E+05+-1.89E+04 D/S/ML
U235	B4-9	33993		4916+-527.5 d/s/sple
Uranium	FL-1	87920		< 1.08703 G/L
	FL-3	87920		< 1.08703 G/L
	FS-1	87920		< 10.0274 MG/G
	FS-2	87920		< 17.2334 MG/G
	FS-3	87920		< 3.87581 MG/G
XRD	FS-1	2005		=Attached
	FS-2	2005		=Attached
	FS-3	2005		=Attached
Zirconium	FL-1	7110		7.03393+- .454699 G/L
	FL-2	7110		7.16566+- .459103 G/L
	FL-3	7110		8.58731+- .50425 G/L
	FS-1	7110		153.506+-22.3438 MG/G
	FS-2	7110		75.8785+-12.6977 MG/G
	FS-3	7110		27.8574+-5.00952 MG/G
	FS-4	7110		< 17.1848 MG/G
cesium	FL-1	2550		=<4 UG/ML
	FL-2	2550		=4.4 UG/ML
	FL-3	2550		=<4 UG/ML
	FL-4	2550		=<4 UG/ML
	FS-1	2550		=<0.02 WT%
	FS-2	2550		=<0.04 WT%
	FS-3	2550		=<0.007 WT%
	FS-4	2550		=<0.008 WT%
strontium	FL-1	2380		=116. UG/ML
	FL-2	2380		=134. UG/ML
	FL-3	2380		=112. UG/ML
	FL-4	2380		=47.9 UG/ML
	FS-1	2380		= .0712 WT%
	FS-2	2380		= .0559 WT%
	FS-3	2380		= .0108 WT%
End of Report --	403 results.			

Westinghouse Idaho Nuclear Company, Inc.

ANALYTICAL CHEMISTRY

SPECTROCHEMICAL ANALYSIS

Record No.	<u>D-206 thru D-208</u>	Log No.	<u>94-08027</u>
Analyzed By	<u>ESD/BRB</u>	Code	<u>BATCH DISS</u>
Approved By	<u><i>W. Palmer</i></u> Date <u>12/30/88</u>	Charge No.	<u>35300-400-002</u>
Sample Activity (mR/hr)		Requested By	<u>S. Roesener</u>
Method	<u>X-ray diffraction</u>	Determinations	<u>71</u>

<u>Sample code</u>	<u>Ca.15Zr.85O1.85</u>	<u>Ca19(Fe1.8Mg1.7Al1.2)Al8(SiO4)10(Si2O7)4(O3OH)10</u>	<u>Al(OH)3</u>	<u>SiO2</u>	<u>U</u>	<u>A</u>
			Gibbsite	Tridymite-2OH		
FS-1	M	m poss	m poss	m poss		
*RACP	64.1	17.5	13.8	4.6	X	X

		<u>FeAl2SiO5(OH)2</u>	<u>Al2O3</u>	<u>Zr(PO3)4</u>	<u>Mg12.8H2O</u>	<u>Mg3.5Al9Si11.5O20</u>	<u>CaCO3</u>	<u>CaMnSi2O6</u>		
		Chloritoid	Corundum			Sapphirine	Vaterite	Johannsenite		
FS-2	M	m	m	m poss	m poss	m poss	m poss	m poss		
*RACP	43.1	10.5	4.6	8.2	13.4	9.2	3.9	7.2	X	X

		<u>Ca5Si3O11.3H2O</u>	<u>(Li,Na)2(Sr,Ca)Al4(PO4)4(OH)4</u>	<u>Na2Ba4Si10O25</u>	<u>Ca(Fe,Mg)Si2O6</u>	<u>FeTiO3</u>	<u>FeO(OH)</u>		
		Foshagite	Palermoite		Nedenbergite	Ilmenite	Geothite		
FS-3	M	m poss	m poss	m poss	m poss	m poss	m poss		
*RACP	41.1	9.1	14.7	7.8	12.7	5.8	8.9	X	X

*Relative abundance of crystalline phases (RACP) is determined by the attenuation % from the search/match program. RACP is equal to the attenuation % of the crystalline phase divided by the total attenuation % numbers of the sample phases.

M = major, m = minor, poss. = possible, X = present, A = amorphous material, U = unidentified material.