

ANALYTICAL CHARACTERIZATION OF WEST VALLEY HIGH-LEVEL WASTE SLUDGE

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ABSTRACT

The West Valley site was the location of the first (and only) operating commercial nuclear fuel reprocessing center in the United States. Nuclear Fuel Services (NFS) operated this facility from 1966 to 1972, processing 640 metric tons of commercial and defense fuels using the PUREX process. The "West Valley Demonstration Project Act" mandates that the resulting high-level nuclear waste be solidified into a form suitable for eventual storage in a federal repository.

The major portion of this waste is stored in a 2.7 million-litre capacity underground steel storage tank. Neutralization of the PUREX process effluent resulted in precipitation of an insoluble sludge which settled to the bottom of the tank. The radioactivity is split approximately 50/50 between the alkaline supernatant (primarily Cs-137) and the sludge solids (primarily Sr-90). The supernatant will be processed by an ion exchange process to remove the cesium, producing a low-level liquid waste which will be evaporated down and solidified in concrete. The sludge solids will be washed in-tank, blended with the cesium loaded ion exchange media, glass formers, and a small volume of acidic (THOREX process) waste stored separately, and fed to a Slurry-Fed Ceramic Melter (SFCM) for melting into a borosilicate glass form.

A core sample of the sludge solids was taken in the fall of 1983 as part of an overall Waste Characterization Program. Small quantities (0.1 - 0.8 gms) of samples from eight vertical horizons in the ~0.4 metre sludge layer were sent to the Babcock & Wilcox Lynchburg Research Center for extensive analytical characterization. Inferences from these results are compared with previous estimates.

The biggest "surprise" was the presence of water-soluble sodium sulfate in the three samples which represented the bottom 75mm in the tank. With the exception of the sodium sulphate, the composition was relatively uniform as a function of vertical direction. In addition, when the total tank curie content for several radionuclides was determined by multiplying the Ci/g insoluble solid composition by the estimated quantity (~100,000 kg), agreement was very close for all the radioisotopes except those of curium, americium, and neptunium. This disagreement (factor of 3-6) was not surprising because of the wide variety of fuels processed and the number of assumptions necessary to obtain the original estimates. The same agreement with original estimates was also true for several major metals. This indicates that the sludge composition is relatively uniform throughout the tank since the probability of taking a sample representing an "average concentration" if there were substantial variation would be extremely low.

Unique analytical techniques used to obtain the data, as well as implications regarding sludge washing, mobilization, and vitrification are presented.

BACKGROUND AND HISTORICAL OVERVIEW

The Department of Energy facility at West Valley, New York was operated by NFS as a commercial nuclear fuel reprocessing plant. West Valley Nuclear Services Co., a subsidiary of Westinghouse Electric Corporation, is now the prime contractor for site operations. The major task of this project involves solidification of 2.1 million litres of fuel reprocessing wastes.

The major portion (~98 percent) of these wastes are stored in a single vaulted underground storage tank. The bulk of its contents was formed by adding excess caustic (NaOH) to a nitric acid-based stream originating from the operation of the NFS reprocessing plant to reprocess uranium fuel using the PUREX process, although other additions of decontamination and cleanup wastes have been made. Concentration of the neutralized solution has resulted in the formation of a sludge layer at the bottom of the waste tank.

The current reference process consists of separate processing of the supernatant and sludge portions of the tank contents. Initially, the supernatant is to be withdrawn from the major waste tank (8D-2) and processed through an ion exchange system for removal of the major activity. The resulting decontaminated supernatant is to be solidified in cement and disposed of as a low-level waste form.

The loaded ion exchanger, now considered to be a zeolite, will substitute as part of the glass-former chemicals (primarily silica (SiO₂), alumina (Al₂O₃), and sodium oxide (Na₂O) in the formulation of a borosilicate glass, which will solubilize the waste. In addition to the cations (primarily cesium isotopes) loaded on the zeolite, the waste will consist of the sludge solids which have been washed in-tank, plus approximately 35,000 litres of a THOREX process waste, which is an acidic waste resulting from the processing of a small batch of thorium-uranium fuel from Indian Point-1 Reactor.

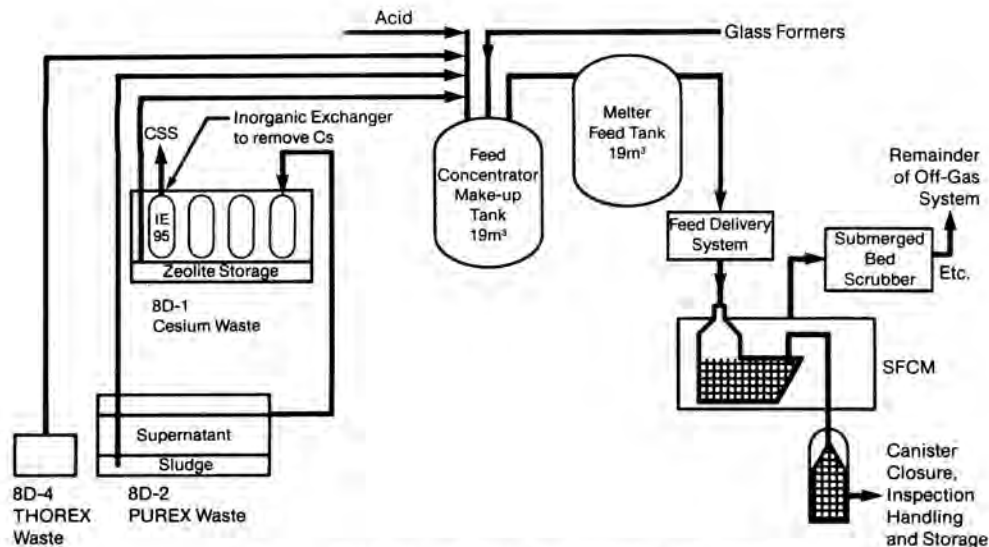


Fig. 1. West Valley HLW Processing Flow Sheet.

The overall process is shown schematically in Fig. 1.

A Waste Characterization Program has been in place since 1982 to better define the chemical, physical, and radiological characteristics of the on-site waste. Previous activities have included sampling and analysis of the 8D-2 tank supernatant¹, sampling and analysis of the THOREX waste tank,² in-cell decontamination tests on the 8D-2 supernatant,^{3,4} in situ physical characterization of the sludge solids,⁵ and core sampling of the 8D-2 sludge solids.⁶ As part of the core sampling project, extensive chemical/radiological analyses of a portion of the sampled solids were performed. This work was done by Babcock and Wilcox (B/W) at their Lynchburg Research Center. This paper reports the methods and results of the analytical work and inferences that can be made when this data is combined with information learned from other aspects of the program.

SAMPLE SOURCE

A 19mm (0.75 inch) diameter core sample of the 8D-2 sludge solids was taken through an accessible tank riser during the fall of 1983. The sampler was designed so as to minimize "smearing" of the sample so that any layering effect (variation of composition with depth) could be determined.⁶

Previous in situ probings⁷ had established that the neutralized sludge existed as two distinct layers, which we have designated the "soft" and the "hard" layer. Figure 2 shows the topography between two of the bottom tank girders which has been developed from this probing. The basic difference between the two layers is in the measured shear strength - the "hard" layer has the shear strength of a medium stiff clay, while the "soft" layer has virtually none.

The sample was transferred to a sample tray, which split the sample into 25.4 mm (one-inch) segments. Overall recovery, calculated by recovered weights from each section is graphically shown in Fig. 3. Part of the top of the hard layer and most of the soft layer were lost by leakage through the split barrel of the sampler. Sufficient soft layer material was saved for an analytical sample, as well as material from the bottom seven one-inch sections. Due to the consistency of the soft layer, it is unlikely that there is significant compositional variation with depth. Thus, approximately 90 percent, by volume, of the sludge layer at that point was able to be sampled. Small portions (0.1 - 0.8 gms) of the first seven one-inch segments and the soft layer were sent to B/W for analytical work. Samples 1 - 7 represent one-inch segments from the bottom of the tank up. Sample 8 is the soft layer.

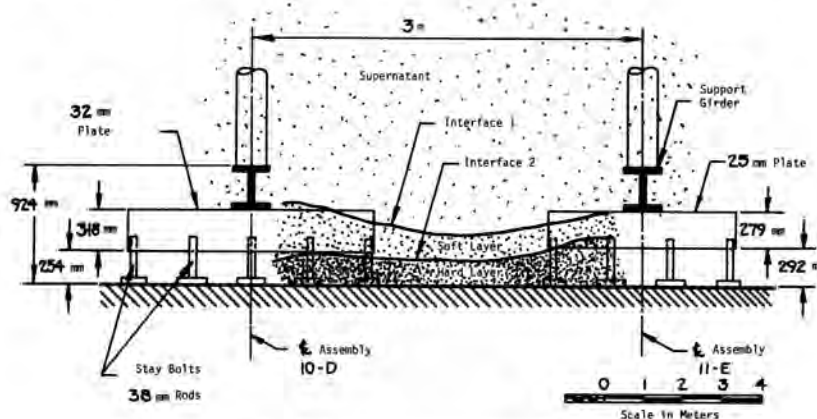


Fig. 2. Tank 8D-2 sludge layering sectional view.

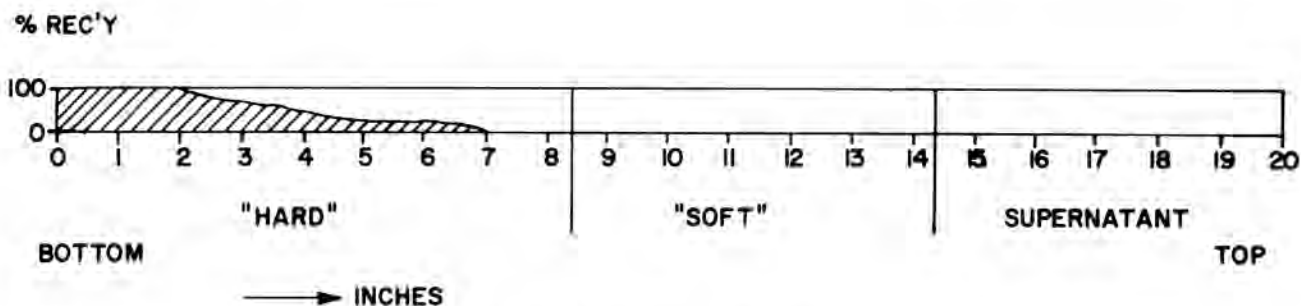


Fig. 3. Core Sample Recovery.

SAMPLE WASHING

The samples were received by B/W in 10 ml "pyramid" vials. A small amount of deionized water was placed in the vials, the vials placed in an ultrasonic apparatus to enhance recovery, and the solids recovered on a 0.45 μ m filter. Further washing was done on the filter. The objective was to remove all the entrainment and solubles (sodium/potassium salts). However, insufficient washing was done on six of the eight samples, and corrections had to be made for remaining supernatant salts, and in the case of Samples 1, 2, and 3, for sodium sulfate solids.

WASH SOLUTION ANALYSIS

The gamma-emitting radionuclides were measured in the wash solution as well as selected cations and all detectable anions. Based on these results (Table I), the following conclusions were reached:

1. Solid phase sodium sulfate was present in Samples 1, 2, and 3. The calculated amounts, expressed as a percentage of supernatant-free solids are:

Sample	Weight Percent Na_2SO_4
1	84.8
2	68.0
3	13.2

2. The low amounts of gamma-emitting radionuclides other than cesium-137/134 indicate negligible solubilization of solid phase components other than the sodium sulfate.
3. Anions detected in the wash solution were Cl^- , NO_2^- , NO_3^- , SO_4^{2-} , CO_3^{2-} , and HCO_3^- , consistent with the major anions in the supernatant. Other than the sulfate, the other anions were reasonably consistent with removal of that amount of supernatant corresponding to the cesium analysis. A check on analytical accuracy can be made by summing the equivalents of anions and cations to see if they "balance". The balance was reasonably close on all samples except No. 1. Trial adjustments were made to the anions and/or the sodium cation to close the balance. The effect on the calculated amount of solid phase sodium sulfate present was negligible.

4. Another analytical check was to determine if the cesium-137/cesium-134 ratio was the same as that measured by Westinghouse Labs on the supernatant¹, after correcting for 1.6 years decay. This was found to be the case within a few percent:

Westinghouse Lab Data (mCi/gm) (corrected to 1984)		
Cs-137	Cs-134	Cs-137/Cs-134
2.73	1.37×10^{-2}	199

B/W Data

Sample	Cs-137/Cs-134
1	198
2	190
3	195
4	198
5	193
6	195
7	193
8	209

WASHED SOLIDS ANALYSIS

A dissolved solids stock solution was prepared for each of the eight sections. The solutions were prepared by dissolving from 3 to 40 mg of accurately weighed dried solids in 2 to 3 ml of 12M HCl. The stock solutions were diluted to 25.0 ml with 6M HCl.

The scheme for the radiochemical analysis is shown in Fig. 4. In addition to the radiochemical analysis, the solutions were analyzed for metals by emission spectroscopy. Based on these results, additional metals were analyzed more precisely using other analytical techniques. Most of the metals could be analyzed with the atomic absorption spectrometer using the flame or the graphite furnace, and in the case of mercury, the cold vapor technique.

Zirconium could not be measured with atomic absorption spectrometry, but was expected in concentrations greater than one percent. Therefore, the zirconium was precipitated with cupferron and determined by X-ray fluorescence.

In addition to the metals analyses just described, the washed solids were also analyzed for carbonate concentration. For the carbonate analysis, a sample aliquot from each section was

TABLE I
Wash Solution Composition

Sec No.	[l] ml	pH	mg										mequiv.		μCi					
			Cl ⁻	NO ₂ ⁻	NO ₃ ⁻	SO ₄ ²⁻	CO ₃ ²⁻	HCO ₃ ⁻	Na ⁺	K ⁺	Al ⁺⁺⁺	Cs ⁺ x1000	Sr ⁺ x1000	Anions	Cations	Cs-137	Cs-134	Eu-154	Co-60	Sb-125
1	50	10.4	1.9	14	24	190	< 4.0	< 3.0	173	0.75	0.22	14	3.8	4.9	7.6	397	2.01	1.7 E-1	LD[+]	LD
2	25	10.4	1.4	34	53	100	13	10	105	1.50	0.14	30	0.30	4.3	4.6	847	4.46	9.0 E-2	5.5 E-4	2.5 E-2
3	50	8	1.9	22	32	20	0.7	4	42	0.84	0.33	17	0.22	1.65	1.88	503	2.58	LD	LD	LD
4	50	[+]	3.2[+]	45[+]	98	10.5	[-]	[-]	66	1.97	0.96	41	0.34	—	—	1,222	6.17	3.7 E-2	5.8 E-3	LD
5	25	6	1.9	12.5	19	2.0	[4]	[-]	16	0.49	0.13	10.3	0.15	0.67	0.72	338	1.75	1.2 E-1	9.3 E-3	LD
6	25	6	0.12	5.6	8.6	1.03	[-]	[-]	7.9	0.23	0.016	3.8	0.063	0.29	0.35	125	0.64	5.0 E-2	LD	LD
7	25	7.1	2.8	28	39	5.6	[-]	[-]	31	0.97	0.40	19	0.16	1.43	1.42	600	3.11	8.6 E-2	4.5 E-3	LD
8	50	6	7.8	24	36	3.8	[-]	[-]	32	1.20	0.46	23	0.55	1.40	1.47	714	3.41	LD	9.5 E-3	LD

Notes: [1] Section numbers 1-7 are from the bottom of the tank up in one-inch increments; section 8 is the "soft" layer

[2] HCl inadvertently added to sample

[3] Estimated

[4] pH too low for measurement

[5] LD = Below limit of detection.

accurately weighed into a 6-ml gas vial and 0.5 ml of 18 M sulfuric acid was added. The evolved carbon dioxide gas was removed from the vial with a gas syringe and injected into a gas chromatograph. The concentration of carbon dioxide gas in the vial is proportional to the concentration of carbonate present in the sample.

Anion measurements are also going to be done by ion chromatography. Of particular interest are water insoluble sulfates, phosphates, and fluorides. Results are not, as yet, available.

Fission/Activation Product Content

Radionuclides in the washed solids were detected based on gamma, beta decay or X-ray spectra. The original data was converted to mCi/gm insolubles by correcting for the supernatant (cesium tracer) and sodium sulfate solids (washed solids sodium and

wash solution sodium and sulfate analyses). A weighted average was then done by separating the sludge layer into three distinct layers:

1. High sulfate "hard" layer (0 - 76 mm)
2. Low sulfate "hard" layer (76 - 267 mm)
3. "Soft" layer (267 - 470 mm)

The high and low sulfate refer to the presence or absence of solid phase sodium sulfate. The depths are averages (approximate) from topographic mapping between the two center girders.

Using previously obtained data/estimates of the specific gravities of the supernatant and insoluble sludge solids and the percent supernatant entrainment in each of the three layers, together with the amount of sulfate in layer one, the following estimates were made:

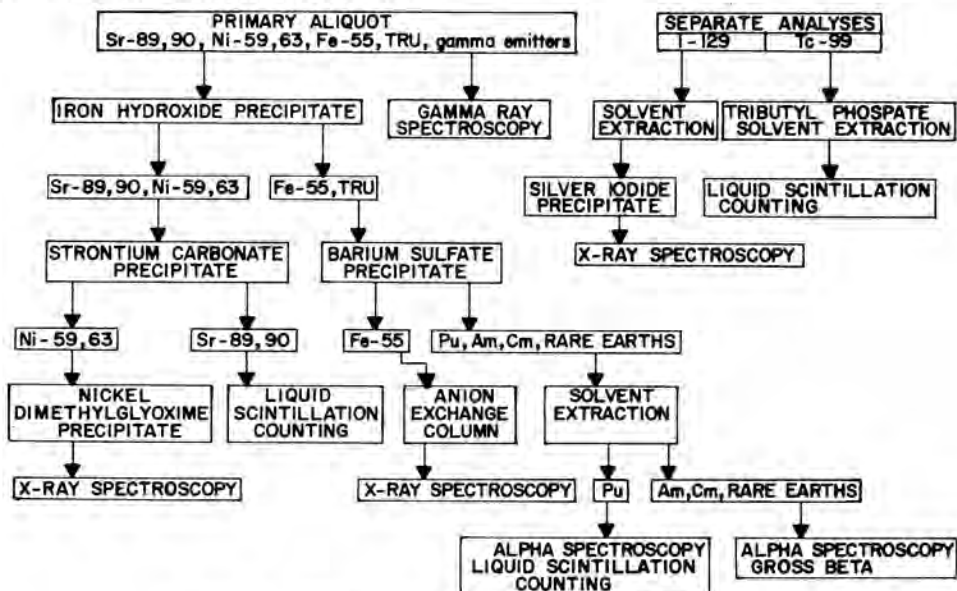


Fig. 4. Washed Solids Radiochemical Analysis.

Component Distribution in Waste Tank

Depth From Tank Bottom (mm)

Component	0-76	76-267	267-470	Total (kg)
Supernatant	23,288	67,987	90,883	182,100 -180,000
Na ₂ SO ₄	15,479	0	0	15,479 -15,000
Insolubles	12,908	74,564	10,092	97,564 -100,000

The -100,000 kg of insoluble solids agrees closely with previous estimates¹ compiled from a number of sources including NFS material balance and analytical data, NFS warehouse records on reagent usage, NFS fuel processing (recovery) records, inferences from the supernatant analytical work, as well as theoretical fission yield and nuclear fuel burn-up calculations (for fission products and transuranics respectively). The -15,000 kg of Na₂SO₄ agrees with the calculated amount using NFS data on total sulfate input and deducting that found analytically in the supernatant.

If we then assume that Layer 1 is the average of section 1, 2, and 3 compositions based on the insolubles; Layer 2 is the average of section 4, 5, 6, and 7 compositions based on the insolubles; and Layer 3 is the section 8 composition based on the insolubles, the weighted value for any component is

$$\text{equal to } \left(\frac{1+2+3}{3}\right) 0.13 + \left(\frac{4+5+6+7}{4}\right) 0.76 + 8(0.11).$$

Using this method of weighting and assuming 100,000 kg of insoluble solids, the total curies of the fission product and activation product radioisotopes shown in Table II were calculated. For several of these radionuclides, a theoretical curie content had been determined.¹ Generally, there is agreement within ±15 percent except for some short-lived radioisotopes, in which case, there is agreement within -2 years of the assumed effective average age of the reprocessed fuel (16.0 years as of April 15, 1984). This average age was calculated by a cesium tracer method using the supernatant analytical data.¹ Considering the smallness of the sample, agreement this close was very gratifying.

Transuranic Content

Alpha spectroscopy was used to determine the transuranics in all eight samples. Table III summarizes total curies in the 80-2 tank solids, again assuming a total of 100,000 kg insolubles in the tank. Table IV compares the plutonium isotopic breakdown with that previously found in the supernatant.

As can be seen, the plutonium content agrees closely with the previous estimate. The isotopic breakdown (percentage of each isotope) is probably within analytical uncertainty, considering that different methods were used (alpha vs mass spectroscopy). Total curies of plutonium were based on NFS recovery records¹ and, as can be seen, agree closely (within ten percent).

Americium, curium, and neptunium, do not agree closely (higher by a factor of 3-6). These isotopes had been estimated by using a computer program (ORIGEN). A number of simplifying assumptions had

TABLE II

Gamma/Beta Emitters: Calculated Total vs. Previous Estimates (Decay-Corrected to 1984)

Isotope	Total Ci in Solids (This Study)	Total Ci in Solids ⁽¹⁾ (Previous Estimate)
Fe-55	2.4 x 10 ⁴	[*]
Ni-59	8.3 x 10 ⁴	[*]
Co-60	8.5 x 10 ⁴	[*]
Ni-63	5.5 x 10 ⁴	[*]
Sr-90	8.2 x 10 ⁴	7.6 x 10 ⁴
Tc-99	<4.0 x 10 ⁴ ^[2]	(0)[*]
Ru-106	<1.4 x 10 ⁴ ^[2]	1.2 x 10 ⁴ ^[2]
Sb-125	1.6 x 10 ⁴	1.0 x 10 ⁴ ^[2]
I-129	<4.4 x 10 ⁻⁴	0[*]
Eu-154	1.6 x 10 ⁴	1.8 x 10 ⁴
Eu-155	4.3 x 10 ⁴	4.0 x 10 ⁴
Total Rare Earth Beta	6.9 x 10 ⁴	1.1 x 10 ⁴ ^[2]

- NOTES: [1] From Reference 1 - adjusted for 1.6 years decay.
 [2] Due to short half-life, this value is very sensitive to assumed age of fuel.
 [3] Based on one sample only.
 [4] Activation product - could not be theoretically determined.
 [5] High uncertainty due to interference of Sb-125.
 [6] Assumed 100 percent soluble.

TABLE III

Transuranics: Calculated Total vs. Previous Estimates

Isotope	Total Ci in Solids (This Study)	Total Ci in Solids ⁽¹⁾ (Previous Estimate)
Np-237	70	11
Np-239	1,494	238
Pu-238	6,570	9,088
Pu-239/240	3,020	2,759
Pu-241	97,400	100,895
Pu-242	<7.8	1.7
Am-241	73,400	12,315
Am-242	680	212
Am-242m	680	212
Am-243	1,494	238
Cm-242	680	212
Cm-243	86	33
Cm-244	25,414	9,715
Cm-245	4.4	1.7
Cm-246	0.52	0.2

- NOTES: [1] From Reference 1 corrected to 1984.
 [2] Analytical method was such that Cm-243 was included with both Am-243 and Cm-242 determinations. Separate values were calculated by assuming that Cm-243 and Cm-244 were the same percentage over previous estimate.
 [3] Am-242 = Am-242m + Cm-242
 [4] Np-239 = Am-243

to be made because of the wide variety of fuels processed.

Uranium Isotopic Content

Relative amounts of the uranium isotopes were determined by mass spectroscopy. The results are shown in Table V. As can be seen, there is excellent agreement (within analytical error) with the Westinghouse Lab results on the supernatant.

TABLE IV
Relative Plutonium Isotope
Concentrations in the Washed Solids

Section [1]	Relative Weight Percent				
	Pu-238	Pu-239	Pu-240 [2]	Pu-241 [2]	Pu-242 [2]
1	0.97	79.22	15.80	2.90	1.11
2	1.11	79.69	16.25	2.11	0.84
3	0.96	79.52	15.90	2.61	1.01
4	0.92	79.34	15.94	2.75	1.06
5	1.16	79.20	15.91	2.71	1.02
6	1.16	78.91	15.89	2.91	1.13
7	1.14	79.08	15.88	2.81	1.09
8	1.09	79.38	15.84	2.65	1.04
Wt'd Avg	1.08	79.20	15.91	2.75	1.06
Relative Weight Percent in Supernatant (Ref. 1)	1.62	78.11	15.97	3.29	1.29

Notes: [1] Section numbers 1-7 are from the bottom of the tank up in one-inch increments; Section 8 is the "soft" layer.
[2] Pu-240/Pu-241 could not be separated by alpha spectroscopy; assumed to be in same proportions as in supernatant.
[3] A less than number was given; assumed to be in the same proportion (curies) as in supernatant.

TABLE V
Relative Uranium Isotope
Concentrations in the Washed Solids

Section [1]	Weight Percent				
	U-233 [2]	U-234	U-235	U-236	U-238
1	—	0.025	1.603	0.153	98.219
2	—	0.029	1.583	0.165	98.223
3	—	0.013	1.635	0.156	98.196
4	—	0.016	1.727	0.173	98.084
5	—	0.032	1.709	0.183	98.076
6	—	0.019	1.708	0.159	98.114
7	—	0.022	1.709	0.173	98.096
8	—	0.015	1.661	0.154	98.170
Wt'd Avg	—	0.021	1.694	0.170	98.115
[3]	(0.031)	(0.021)	(1.693)	(0.170)	(98.085)
Supernatant Concentrations (Ref. 1)	0.031	0.026	1.727	0.169	98.047

Notes: [1] Section numbers 1-7 are from the bottom of the tank up in one-inch increments; Section 8 is the "soft" layer.
[2] Not determined; U-233 was used for spike.
[3] Values in parenthesis are percentages assuming U-233 is the same as that in supernatant.

Metal Content

A preliminary metals survey was done by emission spectrometry which is a semiquantitative analytical method. This was followed up by atomic absorption spectrometry for selected elements. Uranium will be determined by mass spectroscopy (relative isotopic concentrations are available but not absolute amounts). The rare earths fission products were determined theoretically - good agreement with the radioisotopes determined (Table II) indicate the validity of these calculations. Plutonium, americium and curium were determined directly by alpha spectroscopy. Neptunium was determined by means of the major isotope, Np-237. Table VI sum-

marizes all the metallic elements determined and compares this with the previous estimate¹. With reference to Table VI the following comments are made:

1. Total metal content is higher than previously estimated, and, consequently, there doesn't appear to be "room" for all the anions. The majority of the anions have been assumed to be hydroxyl (OH⁻), which could not be determined analytically. One possibility is the conversion of Fe(OH)₃ to Fe₂O₃ [2 Fe(OH)₃ + Fe₂O₃ + 3 H₂O], either by radiological heating in the tank, or upon drying.
2. From a vitrification standpoint, the key is the ratio of a metal with iron, since the amount of iron is the main determinant of waste solubility. Possible problem areas are a higher ratio of Al:Fe and Mn:Fe (enhances spinel formation). A plus is the lower ratio of Cr:Fe (alleviates spinel formation).
3. Calcium/magnesium (designated M) were not previously known to be present in the tank. Carbonate analyses indicates that they are most likely present as carbonates. They will decompose in the melter (MCO₃ + MO + CO₂). The amount found represents about 0.7 percent (as MO) in the final glass.
4. Silicon was not previously listed as a tank constituent. It is probably in the form of silica (SiO₂) or common sand. This is a component of the glass product and its presence should not have an effect on vitrification.

Washed Solids Scanning Electron Microscope Analysis

For each sample, a small portion of the solids collected from the initial washing procedure was mounted on double-sided tape attached to a SEM standard mount. Spheres with calibrated diameters were also included on the mount to provide a frame of reference to estimate the size of the particles. The mount was coated with a thin layer of gold to prevent static charge buildup on the sample mount.

The SEM pictures showed a combination of crystalline "spikes" and amorphous material. The amorphous material is very friable when dry and appears to be able to be subdivided indefinitely. Ferric hydroxide/oxide is probably the main constituent of the amorphous material. Sodium sulfate is probably a main constituent of the spikes, although uranium was identified in one of the spikes (by qualitative x-ray spectra). Significantly, in-cell work⁶ on settling characteristics indicates that the wet material acts as primary particles between 5 and 60 microns (even after being sheared). The breakup into submicron particles appears to occur only upon drying.

IMPACT OF NEW DATA

The close agreement of the analytical data with previously obtained information argues strongly that a representative sample was obtained. Therefore, any variations from previous estimates must be carefully considered. The main impacts to future processing activities comes from the following:

TABLE VI
Cation (Metal) Summary

Component	This Study		Previous Estimate	
	Wt. % ^[1]	Ratio:Comp/Fe	Wt. % ^[1]	Ratio:Comp/Fe
Fe	43.8	1.0	38.6	1.0
U	TRU ^[2]	—	2.5	0.065
Cr	0.2	0.0005	1.7	0.045
Mn	4.8	0.109	1.5	0.038
Ni	0.96	0.022	1.3	0.033
Rare Earths	1.1	0.025	1.1	0.030
Al	3.0	0.068	1.0	0.026
Fission Prod.	1.2	0.027	1.2	0.032
Hg	0.02	0.0005	0.10	0.003
Pu	0.04	0.001	0.04	0.001
Other TRU	0.13	0.003	0.02	0.0005
Zr ^[3]	0.8	0.002	0.10	0.003
Ca	2.0	0.047	0	0
Si	0.7 ^[4]	0.016	0	0
Cu	0.4	0.008	0	0
Mg	0.3	0.006	0	0
Zn	0.26	0.006	0	0

NOTES: [1] Expressed as a percentage of total insolubles (including anions).
 [2] To be determined.
 [3] Nonfission product zirconium.
 [4] By emission spectroscopy (±50%).
 [5] Co, Mo, Ti, V, Sn, Pb less than detectible.

1. The higher than expected transuranics affects the "worst case accident" safety analyses for vitrification.
2. The unexpected sodium sulfate solids affects sludge washing since the main purpose of this wash step is to reduce the sodium sulfate content of the vitrification feed. The sulfate was not expected because of its relatively high water solubility. However, recent experimentation has shown that the supernatant is close to sulfate saturation because of the common ion effect of the high sodium content. It is theorized that the self-heating of the sludge solids may have caused concentration of the interstitial liquid causing the deposition. The fact that sodium sulfate is inversely soluble helped promote this. This deposition on the bottom was apparently "protected" from later solubilization by the overlying sludge layers.

3. Work is in progress to insure that the higher percentage of manganese and aluminum and the lower percentage of chromium can be tolerated within the envelope of the reference glass composition. The other metallic variations should have no significant impact.

The major impact of this analytical study was confirmatory assurance that the estimates made previously were valid.

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