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SAMPLING AND ANALYSES OF SRP HIGH-LEVEL WASTE SLUDGES

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BX 199- 60-07-07

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PREPARED FOR THE U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION UNDER CONTRACT AT(07-2) 1

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Printed in the United States of America

Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161

Price: Printed Copy $4.00; Microfiche $2.25

DP-1399
Distribution Category: UC-70

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Publication Date: August 1976

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ABSTRACT

Twelve 3-liter samples of high-heat waste sludges were collected from four Savannah River Plant waste tanks with a hydraulically operated sample collector of unique design. Ten of these samples were processed in Savannah River Laboratory shielded cell facilities, yielding 5.3 kg of washed, dried sludge products for waste solidification studies. After initial drying, each batch was washed by settling and decantation to remove the bulk of soluble salts and then was redried. Additional washes were by filtration, followed by final drying. Conclusions from analyses of samples taken during the processing steps were: (a) the raw sludges contained ∿80 wt % soluble salts, most of which were removed by the washes; (b) ^{90}Sr and $^{238,239}Pu$ remained in the sludges, but most of the ^{137}Cs was removed by washing; (c) small amounts of sodium, sulfate, and ^{137}Cs remained in the sludges after thorough washing; (d) no significant differences were found in sludge samples taken from different risers of one waste tank. Chemical and radiometric compositions of the sludge product from each tank were determined. The sludges had diverse compositions, but iron, manganese, aluminum, and uranium were principal elements in each sludge. ^{90}Sr was the predominant radionuclide in each sludge product.

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SAMPLING AND ANALYSES OF SRP HIGH-LEVEL WASTE SLUDGES

INTRODUCTION

Radioactive wastes from operation of the Savannah River Plant (SRP) are stored in tanks on the plantsite. The waste tanks contain gelatinous sludge layers that occupy about 10% of the total waste volume and have settled from alkaline supernatant waste solutions. Some tanks also contain salt cake, which is water-soluble crystalline material resulting from evaporation of the supernate. The sludge component consists of hydrous oxides of various metal ions encountered in SRP separations processes; most of the radioactive fission products are expected to be in the sludge. The supernate contains various water-soluble salts, such as sodium nitrate, and most of the ^{137}Cs radioactivity.

In 20 years of SRP waste tank operations, the history of each tank has been recorded. Quantities of bulk chemicals and radionuclides added to or transferred from the tanks are known. Experimental determination of supernate compositions was recently completed in an extensive systematic survey of SRP waste tanks.[1] Sludge composition was less well known, although small samples (a few milliliters) occasionally had been taken from the top of a sludge layer for analyses. The composition and characteristics of the waste sludges were expected to be highly variable, not only between tanks, but also at different locations within a single tank.[2]

For long-term storage of SRP waste, a suitable solid waste form is desirable.[3] In one option under investigation, the sludge component[4] would be separated from the supernate, then washed, dried, and incorporated into either concrete[5] or glass[6] for storage in a retrievable surface storage facility for 100 years or more. ^{137}Cs would be removed from supernate by ion exchange and then sorbed on a zeolite.[7] The resultant supernate would then be evaporated to salt cake for bulk storage. Cesium-loaded zeolite from this operation would be blended with the washed, dried sludge before solidification.

Development of a solidification process and evaluation of the solid waste forms required multiliter quantities of SRP waste sludge for studies in Savannah River Laboratory (SRL) shielded cell facilities. Because a sludge sampling program of this magnitude had not been undertaken previously, unique equipment and procedures were designed for sample collection. Criteria for sludge properties included:

- Sludges representative of high-heat waste from both the Purex and the HM processes were to be sampled.

- Sludges were to be as old as possible.

- Sludge samples from a variety of locations were to be obtained.

A survey of information on the status of SRP waste tanks determined which tanks contained sludge best fitting these criteria. Four tanks were selected for sludge sampling: Tanks 5 and 7 in F Area, and Tanks 13 and 15 in H Area. Several 3-liter samples were collected from each of the four waste tanks.

Each sludge sample was washed and dried to yield a powdered product that could be used in solidification studies. The final sludge products from each tank were characterized by chemical and radiometric analyses. The products were complex mixtures of more than 30 elements, principally in the form of hydrous oxides. The principal elements found in the product sludges are summarized in Table 1, and the specific activities of principal radionuclides are given in Table 2.

Sludge collection and processing are described, and results of the analyses are given in this report.

TABLE 1

Chemical Composition of Washed, Dried Sludges, wt % (mole %)[a]

	Tank 5		*Tank 7*		*Tank 13*		*Tank 15*	
Fe	27.5	(39.6)	8.9	(7.4)	27.9	(39.9)	3.1	(3.9)
Mn	10.8	(15.9)	2.2	(1.9)	8.8	(12.8)	2.3	(2.9)
Al	1.5	(4.6)	2.5	(4.3)	7.1	(21.0)	33.5	(86.2)
U	15.4	(5.2)	3.3	(0.7)	4.0	(1.3)	0.9	(0.3)
Na	6.1	(21.6)	2.8	(5.7)	3.1	(10.7)	1.2	(3.6)
Ca	0.6	(1.3)	0.8	(1.0)	2.3	(4.7)	0.2	(0.4)
Hg	0.1	(-)	0.1	(-)	2.1	(0.8)	0.9	(0.3)
Ni	5.1	(7.1)	2.1	(1.6)	0.5	(0.7)	0.5	(0.6)

a. Principal elements only.

TABLE 2

Radionuclides in Washed, Dried Sludges, mCi/g

	Tank 5	*Tank 7*	*Tank 13*	*Tank 15*
^{90}Sr	74.7	27.0	15.5	25.6
^{144}Ce	4.8	0.2	2.0	16.9
^{106}Ru	2.7	1.4	0.4	1.7
^{154}Eu	0.5	-	0.3	1.2
^{137}Cs	1.3	1.3	0.3	0.1
Gross α	0.1	0.1	0.3	0.1

SAMPLING REQUIREMENTS

Criteria for Sludge

Most radioactive waste originates in the two separations plants, designated F and H Areas; some waste is produced in the reactor areas, laboratories, and peripheral operations.[3] The principal processes in the separations plants have been the Purex and the HM processes, but others have been used to process a variety of fuel and target elements. The Purex process recovers and purifies uranium and plutonium from neutron-irradiated natural uranium; wastes from the Purex process are found in both F- and H-Area waste tanks. The HM process recovers enriched uranium from uranium-aluminum alloys used as fuel in SRP reactors; HM process wastes are found only in H-Area waste tanks. Each process produces characteristic wastes. Furthermore, each process produces both high-heat wastes (HHW) and low-heat wastes (LHW) that have been segregated for storage. High-heat wastes originate in process steps that remove most of the fission products; low-heat wastes are produced in other process steps and have fission-product contents less than 0.1% of high-heat wastes.

The following criteria were established for desirable properties of SRP waste sludges to be used in solidification studies:

- Sludges representative of high-heat wastes from both the Purex and HM processes should be obtained.
- Sludges should be as old as possible, preferably aged about 5 years.

- Whenever practical, sludges should be obtained from different tanks and from different locations within a waste tank to obtain samples of varying composition.

Each criterion is related to particular requirements of the waste solidification studies. Most HHW sludges in SRP waste tanks are from the Purex and HM processes, and samples representative of each were to be evaluated. Sludges at least 5 years old were desired, to simulate as closely as possible those to be used in a prospective SRP waste solidification process. Higher radiation fields from sludges less than 5 years old might adversely affect evaluation of solid waste forms. Sludges from several tanks were to be studied because the sludges vary widely from tank to tank. Also, sludges from different locations in the same tank were to be evaluated to determine in-tank variations.

A survey of available information on the status of SRP waste tanks in mid-1974 was made to determine which tanks contained sludge best fitting the above criteria and to permit selection of tanks for sampling.

Selection of Tanks for Sampling

There are 30 waste tanks in the F and H Areas of the Savannah River Plant. F-Area tanks are numbered 1 through 8, 17 through 20, 33, and 34. H-Area tanks are numbered 9 through 16, 21 through 24, and 29 through 32. Tank numbers 25 through 28 are reserved for future construction. Tanks 1 through 16 are double-walled, cooled tanks for storing HHW; Tanks 29 through 34, completed since 1970, are also designed for HHW. The remaining tanks, 17 through 24, are single-walled and uncooled, for wastes other than HHW.

Not all of the waste tanks were satisfactory for sludge sampling. Some tanks contained only salt cake, some contained fresh waste, some had too little sludge, and some were not easily accessible for sludge sampling because of other equipment in tank risers. Table 3 summarizes the status of each tank in mid-1974, with respect to possible sludge sampling.

Many of the tanks could not be selected for sludge sampling because they contained only salt cake or salt solution. Other tanks were not selected because of properties that might interfere with the operation of the sludge-collecting apparatus, including tanks with a salt crust over the sludge layer and tanks with sludge layers less than 12 inches deep. With these additional considerations, the criterion of sludges aged more than 5 years was difficult to meet. In practice, sludges as old as possible were sampled, and all were greater than 3 years old.

Four tanks that best met all criteria were selected for sludge sampling -- Tanks 5 and 7 in F Area and Tanks 13 and 15 in H Area.

TABLE 3

Status of Waste Tanks in Mid-1974

Tank	*Type of Sludge*	*Age, years*	*Depth, inches*	*Remarks*
F-Area Waste Tanks: Purex Waste				
1, 2, 3, 17, 19 20, 33, 34	None	-	-	Salt cake + solution
4	HHW	Mixed	8	Old + fresh HHW
5[a]	HHW	∿6	15	
6	HHW	2.5	12	Some salt cake
7[a]	HHW + Other[b]	>10	82	
8	HHW + LHW	0.5	-	
18	LHW	10	∿60	No riser available
H-Area Waste Tanks: HM Waste and Purex Waste				
9, 10, 22, 24, 29, 30, 31	None	-	-	Salt cake + solution
11	HHW + LHW	0.5	36	
12	HHW	1.5	97	
13[a]	HHW + LHW	6.5	115	
14	HHW	-	8	Sludge cleaned under risers
15[a]	HHW	3	92	
16	HHW	10	19	Some salt cake
21	HHW + LHW	∿6	28	Similar to Tank 13
23	RBOF[c]	-	9	
32	HHW	Fresh	21	

a. Selected for sampling.

b. Reactor-area sand filter backwashes.

c. Receiving basin for offsite fuels.

SAMPLING CAMPAIGN

Sludge Collector

Three-liter batches of sludge were collected in a hydraulically energized apparatus shown schematically in Figure 1. The collector is an open, metal spoolpiece connected to a cylindrical cover that can be closed by a hydraulic actuating system. The collector is submerged in the sludge in the open position. The exposed spoolpiece fills with sludge, then the cover is moved down around the spoolpiece by pumping water into the hydraulic cylinder above the piston. Mechanical latches engage when the collector is fully closed. The closed assembly is approximately 4.5 inches in diameter and 23 inches long. After the water hose is remotely detached from a quick-disconnect fitting, the collector is hoisted by cable into a shielded cask. The collector may be opened with master-slave manipulators. The mechanical latches are first released, the hydraulic seals are opened, and the device is pulled to its open position. Details on the design and construction of the sludge collector are given in Appendix A.

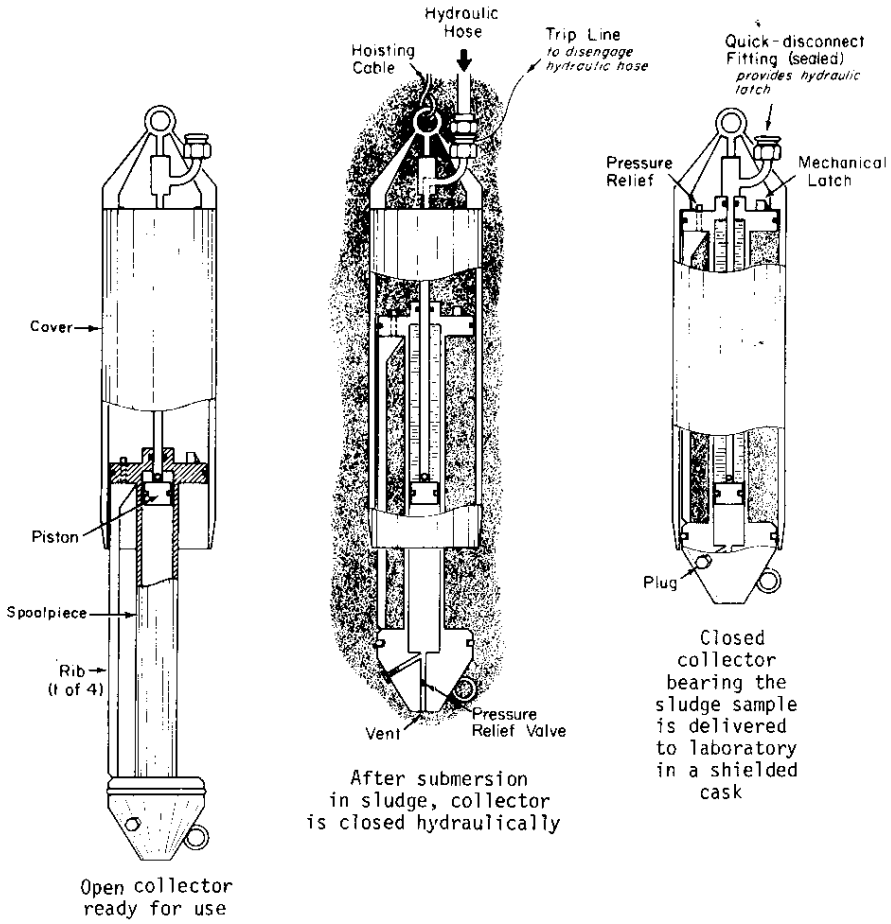

FIGURE 1. Sludge Collector

Sampling Procedure

Use of the sludge collector in a waste tank is shown schematically in Figure 2. The open collector, hydraulic hose, and trip line were lowered by a cable through a riser, into the tank. The assembly was lowered through the supernate into the sludge layer as far as it would go. The collector was hydraulically closed; a successful closure was indicated when the collector accepted a predetermined volume of water and when continued pumping merely increased the pressure. The hydraulic hose then was disconnected with the trip line, and both were removed from the riser.

Removal of the filled and closed sludge collector from the waste tank is shown in Figure 3. The open-bottomed, lead-shielded transport cask through which the cable passes was placed with a crane directly over the riser. The collector was then raised, sprayed with water, and reeled up into the cask. The cask and collector were lifted on to the baseplate and liner, sealed, and shipped to SRL.

In a shielded laboratory cell, the collector was opened and sludge was removed. Figure 4 shows residual sludge before the emptied collector was washed. After unloading, the collector was decontaminated and returned to SRP for another sample.

Five 3-liter sludge samples from Tank 13, one from Tank 7, three from Tank 5, and three from Tank 15 were collected. The chronology of the sampling campaign is given in Table 4.

TABLE 4

Sludge Samples Collected

Date, 1974	*Waste Tank*	*Sample Designation*	*Remarks*
August 5	13	13-1	
August 13	13	-	Sample lost in cell
August 22	13	13-2	
August 27	13	13-3	
August 30	13	13-4	
September 10	7	7-1	Sand and coal in sludge
September 18	7	-	Sampling attempt aborted
October 4	5	5-1	
October 10	5	5-2	
October 14	5	5-3	
October 17	15	15-1	
October 23	15	15-2	
October 29	15	15-3	

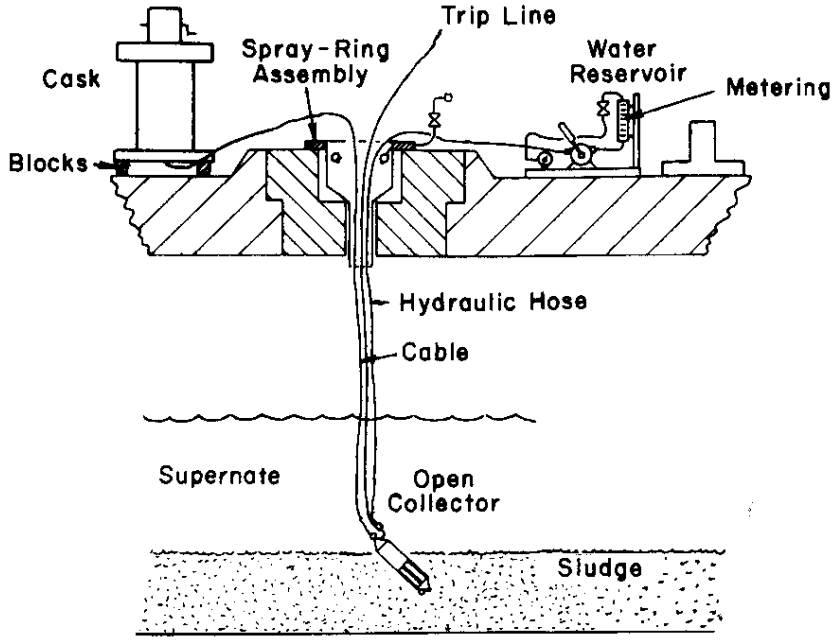

FIGURE 2. Sludge Sampling

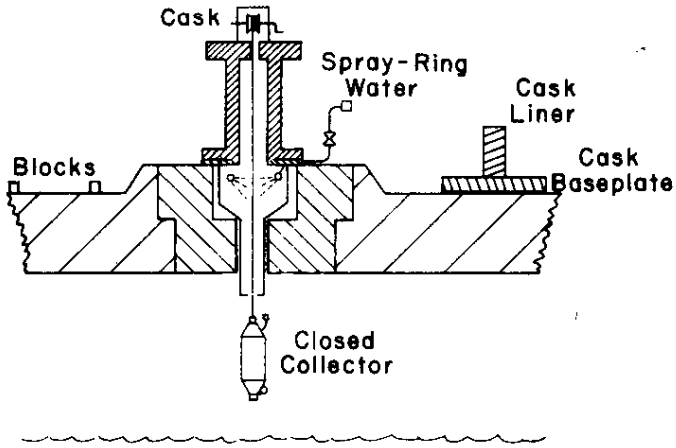

FIGURE 3. Sludge Retrieval

Only minor problems were encountered while sampling from two risers in Tank 13. One sample was lost in the laboratory cell because the sample collector opened prematurely. Mechanical and procedural modifications prevented this from recurring. Figure 4 shows Tank 13 sludge from the successful first attempt of the campaign. Tank 13 was the only waste tank for which more than one riser was used for sampling; here the two risers were widely separated.

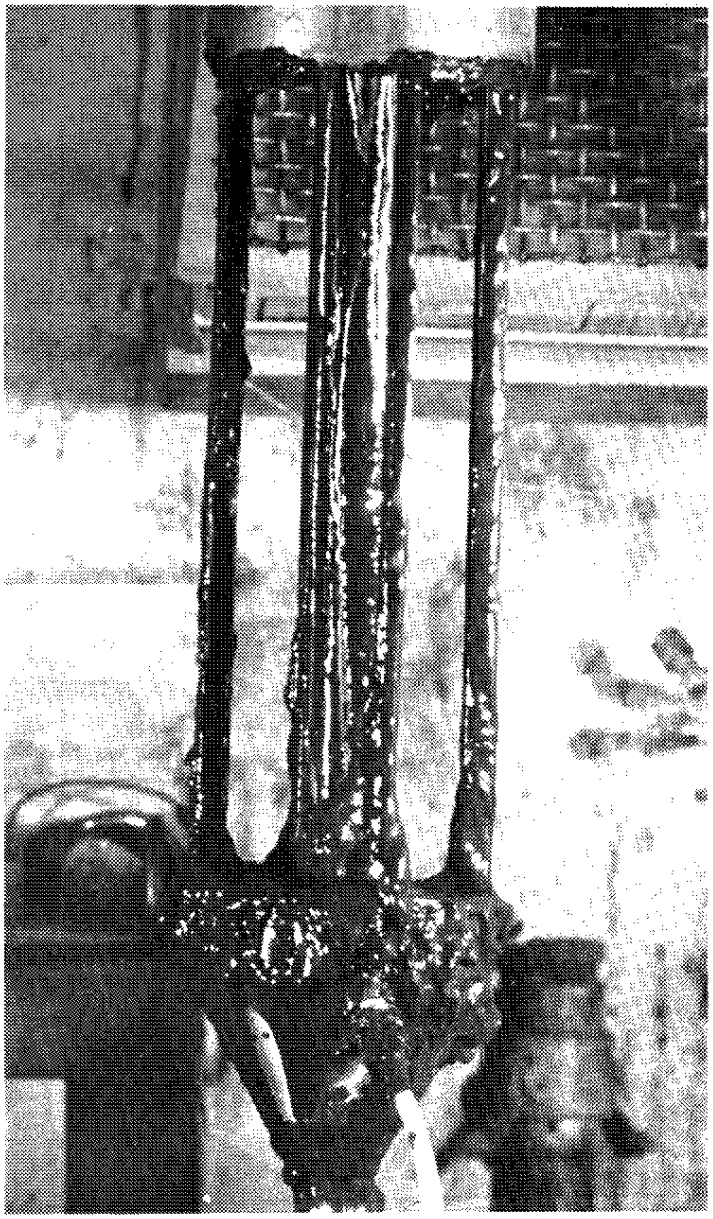

FIGURE 4. Residual Sludge on Collector after Emptying the First Tank 13 Sample

From Tank 7, only one sample was collected because of sand in the tank. The sand caused the sample collector to bind and prevented complete closing. The Tank 7 sludge sample is shown in Figure 5. A second attempt to obtain sludge from Tank 7 was unsuccessful because sand prevented the sample collector from closing sufficiently to fit into the cask. The sample collector was flushed and retrieved from the tank; no further attempts were made to sample Tank 7. Analysis of the sample obtained indicated approximately equal amounts of sand and coal in the sludge. This material is believed to have originated from backwashes of reactor area sand filters.

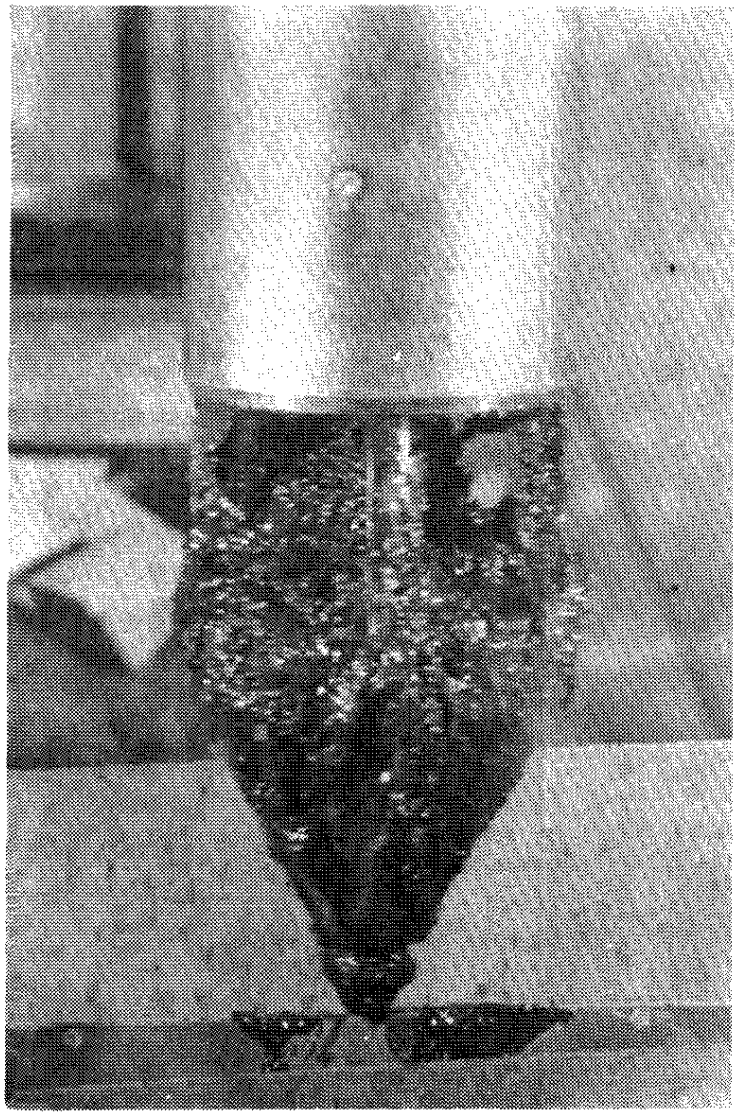

FIGURE 5. Tank 7 Sludge Sample

Three sludge samples then were collected from Tank 5. Before the first sample was collected, a bundle of wire was removed from the riser opening to permit access to the sludge. Finally, three samples were collected without difficulty from Tank 15. Sludge from Tank 15 was particularly viscous, as shown in Figure 6.

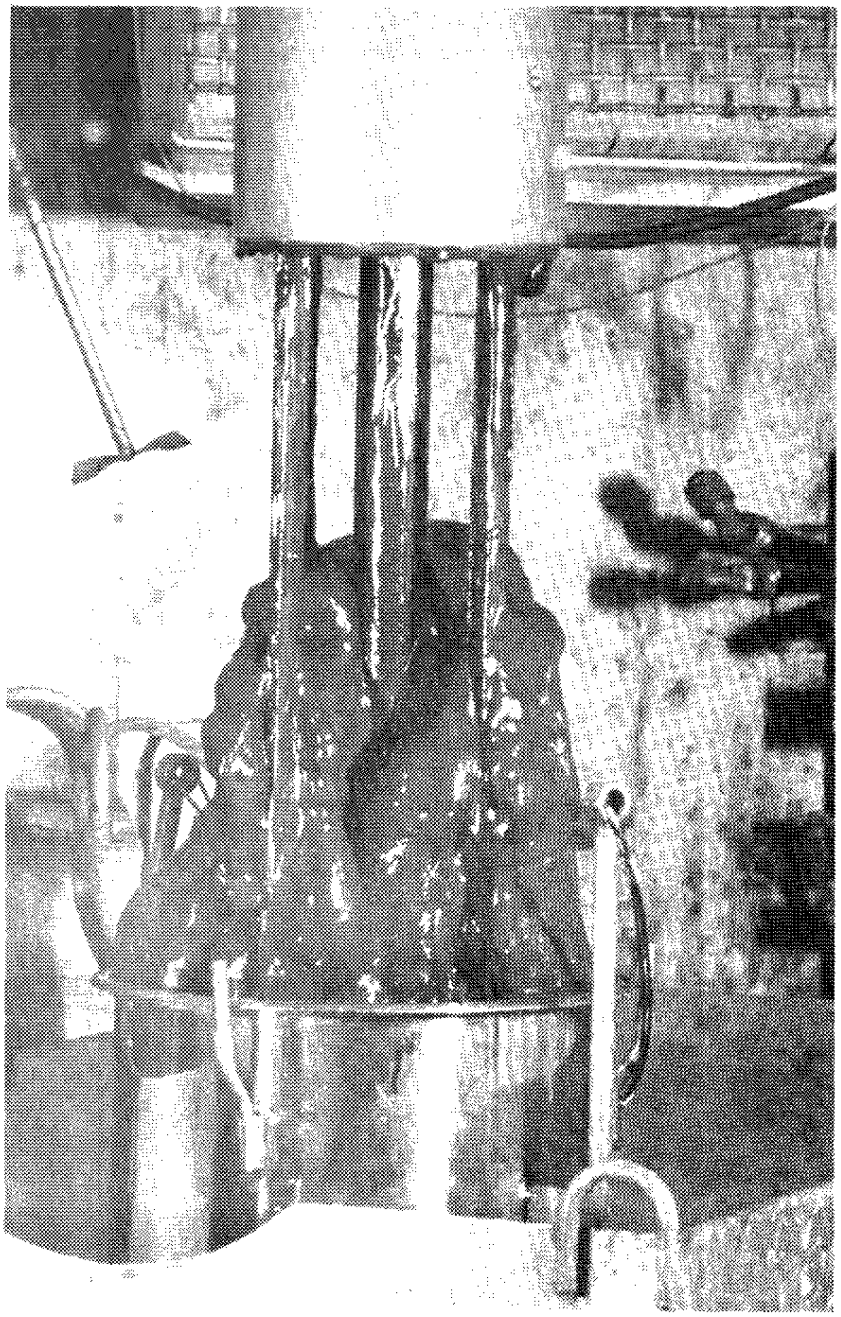

FIGURE 6. Tank 15 Sludge Sample

SLUDGE PROCESSING

Processing Procedure

In SRL shielded cell facilities, each sludge sample was individually washed to remove soluble salts and dried to yield free-flowing powders. Subsequently, all batches from a given tank were blended for additional washing and drying, to yield a product sludge powder. Ten batches of sludge from Tanks 5, 7, 13, and 15 were processed to produce 5.3 kg of washed, dried sludges. One batch of unprocessed sludge from Tank 15 was stored for future studies.

For simplicity, the sludges were washed by settling and decantation, or by filtration; in a large-scale process, more-efficient methods such as centrifugation probably would be used for the washing steps. Analyses were performed at various stages to determine processing behavior. The following procedure was used for processing Tank 13 sludge; operations were similar for sludges from the other tanks.

The initial processing steps are shown in Figure 7. Each of four batches of Tank 13 sludge was allowed to settle, the supernate was decanted, and the raw sludge was dried in an oven at ∿140°C. The dried sludge was washed twice by contacting with 3 to 4 liters of water, settling, and decanting; each batch was dried a second time. Analyses of the dried materials showed large quantities of sodium, nitrate, and sulfate remaining in the sludge. As shown in Figure 8, for further washing the four batches were blended, then washed on a stainless steel filter frit with ∿12 liters of water. Filtration was not practical for earlier washes because of the gelatinous nature of sludge before removal of the bulk of soluble salts. A final drying yielded the sludge product, which was pulverized to powder form with a laboratory blender.

Figure 9 shows the drying oven and a batch of sludge after the initial drying. This step breaks up the gelatinous structure of the sludge and renders it more easily washable. Figure 10 shows the final sludge product.

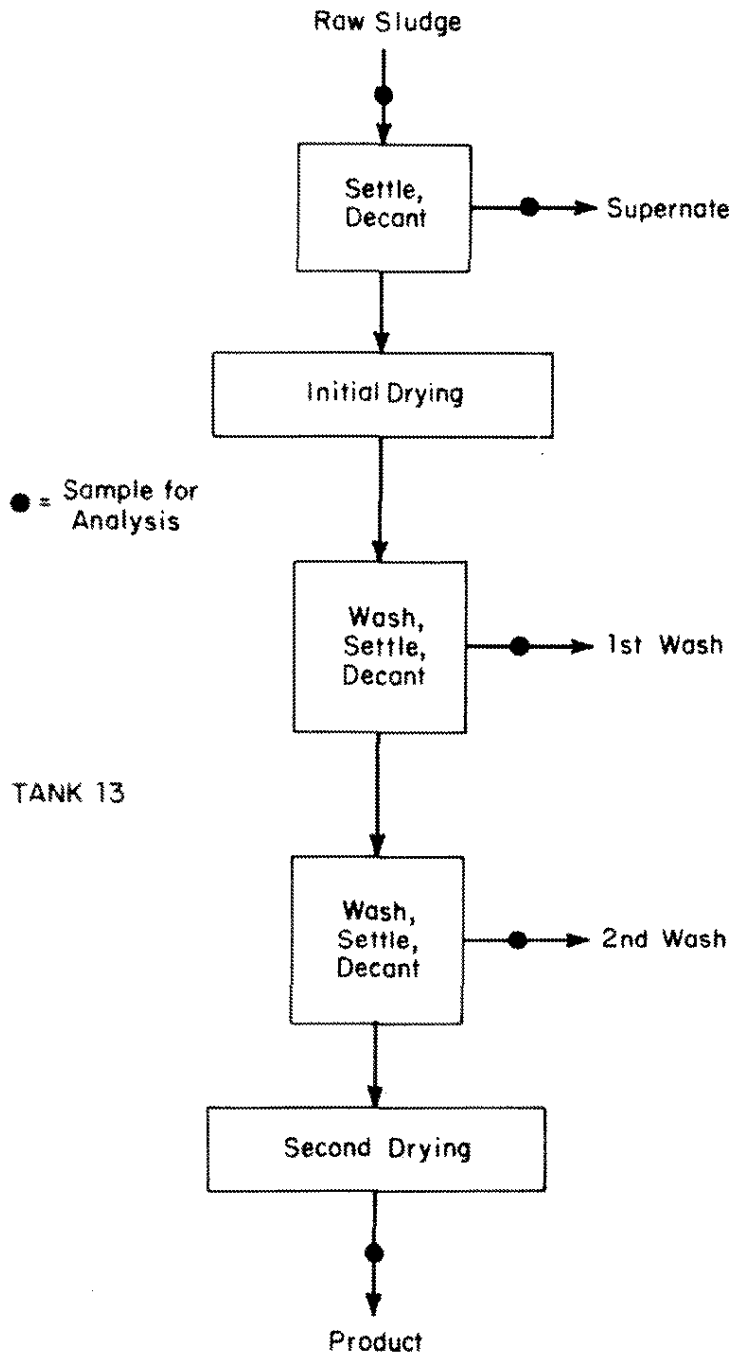

FIGURE 7. Initial Processing of Individual Sludge Samples

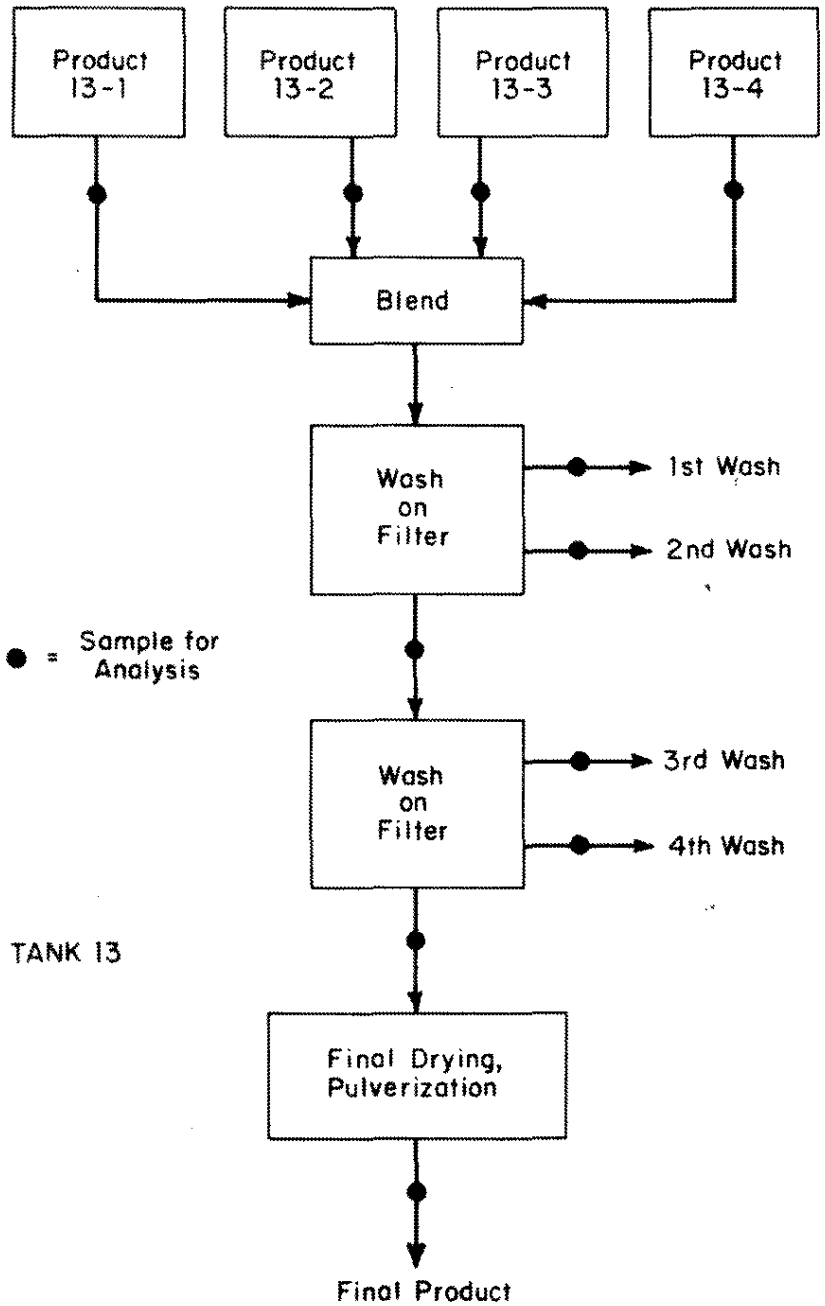

FIGURE 8. Final Processing of Blended Sludge Samples

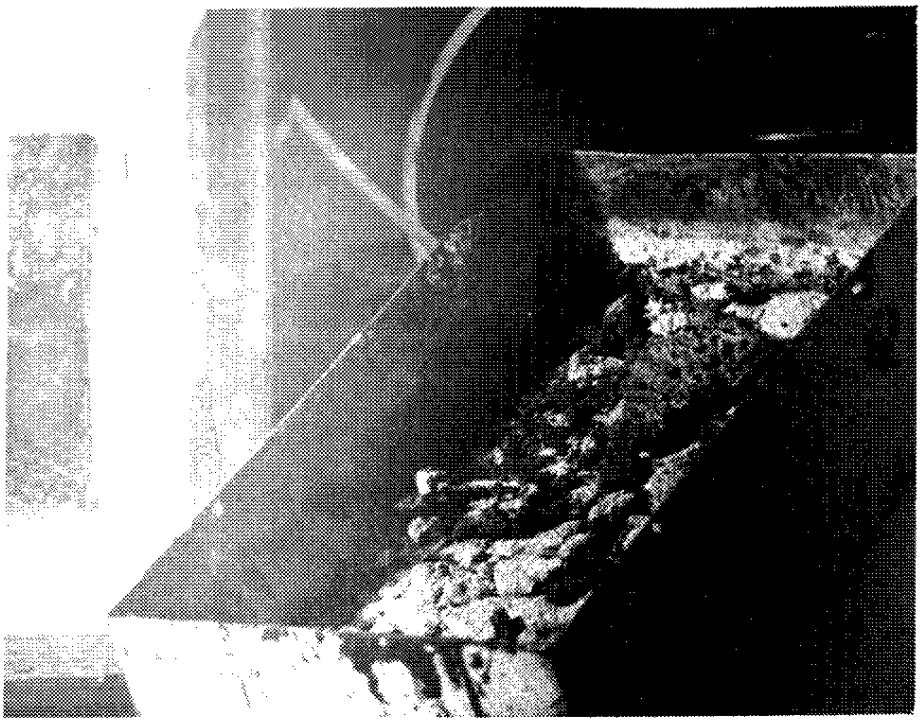

FIGURE 9. Sludge Drying

FIGURE 10. Powdered Sludge Product

The final sludge products from each waste tank were thoroughly characterized by analyses for the major chemical constituents and for radionuclides. These results are given in the following section.

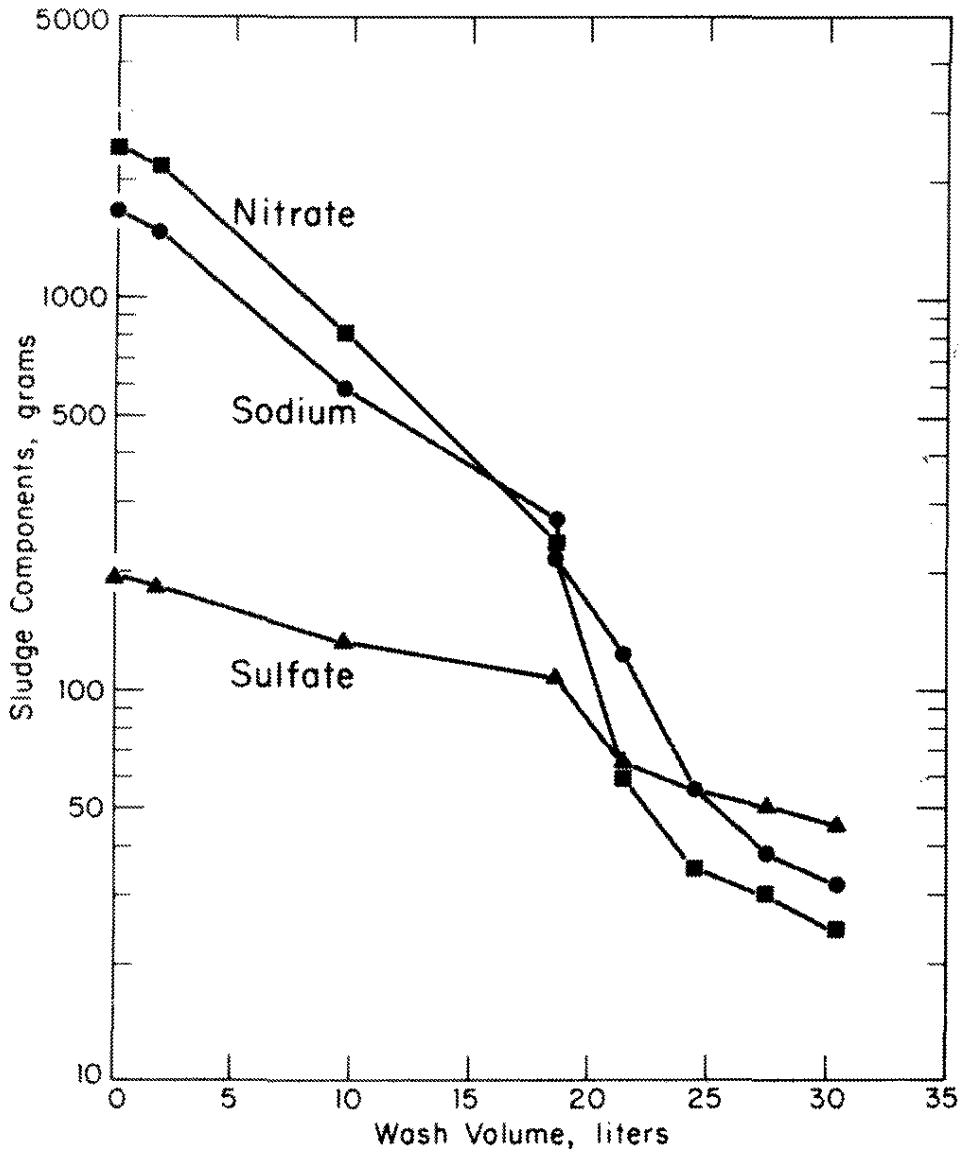

FIGURE 11. Washing Behavior of Tank 13 Sludge

SLUDGE ANALYSES

Chemical Composition

The washed, dried sludge products were characterized chemically by elemental analyses and spark-source mass spectrometry. The chemical composition of sludge from each waste tank is given in Part A of Tables 6 through 9. Measurements on residual anions and Na^+ in the sludges are given in Part B of these tables. Details of the analytical methods are given in References 1, 8, and 9.

The composition of Tank 5 sludge (Table 6) is believed to be representative of sludges from Purex process wastes. As expected, iron and manganese were principal chemical components. The uranium content was higher than for the other sludges but was consistent with the known uranium inventory in Tank 5. This sludge was more difficult to wash than the others, as shown by the high sodium content and the large amounts of residual anions. However, these usually soluble components appeared to be fixed in the sludge, because very little of each component could be removed by vigorous leaching.

The sludge from Tank 7 was predominantly coal and sand (Table 7). Because this sludge was very nonrepresentative of Purex waste, it was not reserved for waste solidification studies. Consequently, fewer analyses were performed on material from Tank 7 than for the other tanks.

Of the H-Area tanks, Tank 13 sludge contained iron, aluminum, and manganese as principal components (Table 8), as expected for Purex and HM process wastes. However, Tank 15 sludge (Table 9) was anomalously high in aluminum; the samples obtained may not be representative of material deeper in the sludge layer. Mercury was expected in larger quantities than was found in either the Tank 13 or the Tank 15 sludge samples. Thus, there could be significant variations in sludge composition at different depths in the sludge layers; such variations would have been undetected with the samples discussed here.

The insoluble chemical compounds in sludges are presumed to be hydrous oxides of the metal ions, as inferred from the method of preparation. Nitric acid waste streams are neutralized with excess NaOH before transfer to the waste tanks. To identify compounds in the washed sludges that had been dried at 140°C, the products were analyzed by x-ray diffraction. Unfortunately, x-ray diffraction was insensitive to compounds of many of the elements known to be in the sludges. However, positive identification of some of the major compounds was made. In both the Tank 5 and the Tank 13 product, α-Fe_2O_3 and $Na_4U_5O_{17}$ were

found. In the Tank 7 product were reported: an Fe_3O_4-type phase, probably containing nickel and manganese as well as iron; a single peak from carbon (graphite) and/or SiO_2; $Na_2U_2O_7$; and possibly $3NaAlSiO_4 \cdot NaOH$. In the Tank 15 product, γ-AlOOH was identified.

Radionuclide Content

The specific activities of radionuclides in the washed, dried sludge products were determined by high-resolution gamma-ray spectroscopy and gross-alpha counting. The radionuclide content of sludge from each waste tank is given in Part C of Tables 6 through 9. All the major fission products were sought except ^{147}Pm, which was not determined. Details of the radiometric methods used are given in References 1, 8, and 9.

Total radioactivity measured in the washed, dried sludges ranged from approximately 20 mCi/g for Tank 13 sludge to 85 mCi/g for Tank 5 sludge. As expected, ^{90}Sr was the principal radioactivity in each of the washed sludges. Other radioisotopes present in significant quantities were the fission products ^{144}Ce, ^{106}Ru, and ^{154}Eu. Most of the ^{137}Cs in the raw sludges was washed out, but a residual quantity (about 3 to 12% of the original) appeared to be fixed in the product sludges. The highest ^{137}Cs content was found in washed Tank 5 sludge, which also had high residual Na^+.

Pulse-height analysis of the alpha radioactivity showed only ^{238}Pu, ^{239}Pu, and ^{244}Cm. Tank 5 sludge contained 56% ^{238}Pu, 33% ^{239}Pu, and 11% ^{244}Cm, expressed as percentage of total alpha activity. In Tank 7 sludge, 56% ^{238}Pu and 44% ^{244}Cm were detected. Tank 13 sludge showed 95% ^{238}Pu and 5% ^{244}Cm. For Tank 15 sludge, 94% $^{238,239}Pu$ and 6% ^{244}Cm were found. The alpha radioactivity was small in every case.

TABLE 6

Washed, Dried Tank 5 Sludge

A. Chemical Composition

Element	*Wt %*	*Mole %*[a]	*Element*	*Wt %*	*Mole %*[a]
Fe	27.5	39.6	P[b]	0.07	0.2
U	15.4	5.2	Zn[b]	0.07	0.09
Mn	10.8	15.9	Th	0.07	0.02
Na	6.1	21.6	Mg	0.06	0.21
Ni	5.1	7.1	Cu[b]	0.06	0.07
Al	1.5	4.6	Nb[b]	0.05	0.05
Ca	0.6	1.3	Y[b]	0.04	0.03
Nd[b]	0.5	0.3	Ag[b]	0.04	0.03
Si	<0.4	<1.2	Pm[b]	0.04	0.02
Zr[b]	0.4	0.4	Pb[b]	0.04	0.02
Cr[b]	0.3	0.5	Ti[b]	0.04	0.06
Ru[b]	0.3	0.2	Sm[b]	<0.04	<0.02
Ba	0.25	0.15	Eu[b]	0.02	0.01
Ce[b]	0.2	0.1	V[b]	0.01	0.02
K	0.14	0.3	Rh[b]	0.01	0.01
Cl[b]	0.1	0.2	Mo[b]	<0.01	<0.01
Sr[b]	0.1	0.09	Pu[b]	<0.01	<0.005
Hg	0.10	0.04	Np[b]	<0.008	<0.002
La[b]	0.09	0.05	F[b]	0.007	0.03
Pr[b]	0.09	0.05	B[b]	<0.003	<0.02
S[b]	0.08	0.2			

a. Calculated from sum of the elements shown, without O, N, or C.

b. From spark-source mass spectrometry (factor of 2 accuracy); all other values from elemental analyses.

TABLE 6 (Continued)

B. Residual Leachable Components

Component	*Total Wt %*	*Leachable Wt %*
Na^+	6.1	0.8
NO_3^-	3.0	0.6
NO_2^-	0.06	0.004
SO_4^{2-}	5.5[c]	0.8

C. Specific Activity of Radionuclides

Isotope	*mCi/g*	*Isotope*	*mCi/g*
^{90}Sr	74.68	^{95}Nb	<0.03
^{144}Ce	4.77	^{95}Zr	<0.03
^{106}Ru	2.73	^{134}Cs	<0.02
^{137}Cs	1.29	^{103}Ru	<0.02
^{154}Eu	0.47		
^{125}Sb	0.43		
Gross α	0.10		

c. Total sulfate values may be high by a factor of 5 to 10; results of analyses by three different methods gave poor agreement.

TABLE 7

Washed, Dried Tank 7 Sludge

A. Chemical Composition

Element	*Wt %*	*Mole %*[a]	*Element*	*Wt %*	*Mole %*[a]
C	16.8	65.3	Cu[b]	0.15	0.11
Fe	8.9	7.4	Sr[b]	0.15	0.08
U	3.3	0.7	La[b]	0.15	0.05
Si	3.2	5.3	Pr[b]	0.15	0.05
Na	2.8	5.7	Ba	0.13	0.04
Al	2.5	4.3	Nb[b]	<0.1	<0.05
Mn	2.2	1.9	Mg	0.08	0.15
Ni	2.1	1.6	Cs[b]	0.06	0.02
Cl[b]	2.0	2.6	Hg[b]	0.06	0.01
Nd[b]	1.0	0.3	Y[b]	0.05	0.03
Ca	0.83	1.0	Sm[b]	0.05	0.02
S[b]	0.5	0.7	Rh[b]	0.05	0.02
Zn[b]	0.5	0.4	B[b]	<0.05	<0.21
Zr[b]	0.5	0.3	Cd[b]	<0.05	<0.02
Ru[b]	0.5	0.2	P[b]	0.03	0.05
Cr[b]	0.45	0.4	Pd[b]	0.03	0.01
K[b]	0.3	0.4	Mo[b]	<0.014	<0.007
Ag[b]	0.3	0.1	Eu[b]	0.013	0.004
Ce[b]	0.25	0.08	Co[b]	<0.013	<0.010
Pb[b]	0.25	0.06	Th	0.007	0.001
F[b]	0.15	0.37	V[b]	<0.005	<0.005

a. Calculated from sum of the elements shown, without O or N.

b. From spark-source mass spectrometry (factor of 2 accuracy); all other values from elemental analyses.

TABLE 7 (Continued)

B. Residual Leachable Components

Component	*Total Wt %*	*Leachable Wt %*
Na^+	2.8	1.9
NO_3^-	1.2	1.2
NO_2^-	0.09	0.03
SO_4^{2-}	19.5[c]	3.7

C. Specific Activity of Radionuclides

Isotope	*mCi/g*	*Isotope*	*mCi/g*
^{90}Sr	27.03	^{134}Cs	0.01
^{106}Ru	1.41	^{103}Ru	<0.01
^{137}Cs	1.30	^{154}Eu	<0.01
^{144}Ce	0.21	^{95}Nb	<0.01
Gross α	0.06	^{95}Zr	<0.01

c. Total sulfate values may be high by a factor of 5 to 10; results of analyses by three different methods gave poor agreement.

TABLE 8

Washed, Dried Tank 13 Sludge

A. Chemical Composition

Element	*Wt %*	*Mole %*[a]	*Element*	*Wt %*	*Mole %*[a]
Fe	27.9	39.9	Zn[b]	<0.2	<0.3
Mn	8.8	12.8	Pr[b]	0.2	0.1
Al	7.1	21.0	Ba	0.15	0.09
U	4.0	1.3	Th	0.11	0.04
Na	3.1	10.7	Ti[b]	<0.1	<0.2
Ca	2.3	4.7	Nb[b]	<0.1	<0.09
Hg	2.1	0.8	Ag[b]	<0.1	<0.07
Cl[b]	1.0	2.3	Cr	0.09	0.13
Ce[b]	1.0	0.6	K[b]	0.08	0.2
Nd	<0.6	<0.3	Cs[b]	<0.06	<0.04
Ni	0.5	0.7	Y[b]	0.05	0.05
Pb[b]	0.5	0.2	S[b]	<0.04	<0.1
Si	<0.4	<1.1	Cu[b]	0.03	0.04
Zr[b]	0.4	0.4	V[b]	<0.007	<0.01
Mg[b]	0.3	1.0	B[b]	0.005	0.04
P[b]	0.3	0.8	F[b]	<0.005	<0.02
La[b]	0.3	0.2			

a. Calculated from sum of elements shown, without O, N, or C.

b. From spark-source mass spectrometry (factor of 3 accuracy); all other values from elemental analyses.

TABLE 8 (Continued)

B. Residual Leachable Components

Component	*Total Wt %*	*Leachable Wt %*
Na^+	3.1	1.0
NO_3^-	0.5	0.3
NO_2^-	0.03	0.02
SO_4^{2-}	2.7[c]	0.8

C. Specific Activity of Radionuclides

Isotope	*mCi/g*	*Isotope*	*mCi/g*
^{90}Sr	15.49	^{95}Nb	0.02
^{144}Ce	2.01	^{95}Zr	<0.01
^{106}Ru	0.40	^{134}Cs	<0.01
^{154}Eu	0.30	^{103}Ru	<0.01
^{137}Cs	0.30	^{60}Co	0.01
Gross α	0.28		
^{125}Sb	0.12		

c. Total sulfate values may be high by a factor of 5 to 10; results of analyses by three different methods gave poor agreement.

TABLE 9

Washed, Dried Tank 15 Sludge

A. Chemical Composition

Element	*Wt %*	*Mole %*[a]	*Element*	*Wt %*	*Mole %*[a]
Al	33.5	86.2	Pr[b]	0.04	0.02
Fe	3.1	3.9	La[b]	0.04	0.02
Mn	2.3	2.9	B[b]	0.03	0.20
Na[b]	1.2	3.6	P[b]	0.03	0.07
Hg	0.92	0.32	Ru[b]	0.03	0.02
U	0.91	0.26	Ti[b]	<0.02	<0.03
Ni	0.51	0.60	Nb[b]	<0.02	<0.01
Nd[b]	0.30	0.15	S[b]	<0.02	<0.05
Si[b]	0.22	0.55	K	0.015	0.03
Ca	0.21	0.38	Cr[b]	0.01	0.02
Th	0.18	0.06	Y[b]	0.01	0.01
Mg	0.14	0.39	Sm[b]	0.01	0.007
Zr[b]	0.11	0.08	Pb[b]	0.01	0.005
Ba	0.10	0.05	Rh[b]	0.007	0.005
Ce[b]	0.05	0.02	Pu[b]	<0.005	<0.001
Cu[b]	0.04	0.05	F[b]	0.002	0.009
Sr[b]	0.04	0.03	V[b]	<0.0007	<0.0007
Zn[b]	<0.04	<0.04			

a. Calculated from sum of elements shown, without O, N, or C.

b. From spark-source mass spectrometry (factor of 3 accuracy); all other values from elemental analyses.

TABLE 9 (Continued)

B. Residual Leachable Components

Component	*Total Wt %*	*Leachable Wt %*
Na^+	1.2	0.4
NO_3^-	5.0	0.3
NO_2^-	0.04	0.04
SO_4^{2-}	5.7[c]	0.9

C. Specific Activity of Radionuclides

Isotope	*mCi/g*	*Isotope*	*mCi/g*
^{90}Sr	25.61	^{137}Cs	0.07
^{144}Ce	16.89	^{134}Cs	0.03
^{106}Ru	1.74	^{95}Nb	<0.02
^{125}Sb	1.27	^{95}Zr	<0.01
^{154}Eu	1.18	^{103}Ru	<0.01
Gross α	0.14		

c. Total sulfate values may be high by a factor of 5 to 10; results of analyses by three different methods gave poor agreement.

ACKNOWLEDGMENT

Many people contributed to the work described in this report. Assistance in planning the sludge sampling campaign was provided by the SRP Separations Technology Department. Sampling operations at the waste tanks were carried out by the SRP Separations Department. Sludge processing was done by the SRL High-Level Caves operating group. All analyses were performed by the SRL Analytical Chemistry Division. The authors are grateful to each of these organizations and to the individuals directly involved for their cooperation in the successful completion of this complex effort.

APPENDIX A – ENGINEERING DESIGN OF SLUDGE COLLECTOR

The sludge collector consists of an open spoolpiece, a cylindrical cover, and a hydraulic system. The spoolpiece and cover can be spread apart, lowered into sludge, and then drawn together (by application of water pressure supplied through a hose to the hydraulic system) to enclose sludge trapped in the previously exposed spoolpiece. Operation of the device is shown in Figures 1 through 6. A more detailed sectional view of the collector is given in Figure A-1. In the following discussion, several unique features of the collector are highlighted, and each of the major components of the device is described. Where pertinent to the final design, details of operating experience with the sludge collector also are given.

Features

Compartment Size

The sample compartment is large relative to the overall size of the collector and as compared to previously designed sludge sampling devices.[8] The collector holds approximately 3 liters of material, but the overall size is approximately that of an earlier sampler that held only 50 ml.

Sample Entrapment

The sludge collector is completely open as it is lowered into a waste tank. Sludge can enter the exposed spoolpiece freely as the collector travels into the sludge layer. Sludge also may be dragged into the sample chamber by the relative motion of the spoolpiece and cover during the closing operation.

Energy Source

Energy to close the device is transmitted hydraulically to the collector from a remote location. The closing force is limited only by the mechanical design of the collector itself. There is no internal power source, such as a spring, that might prove inadequate in service.

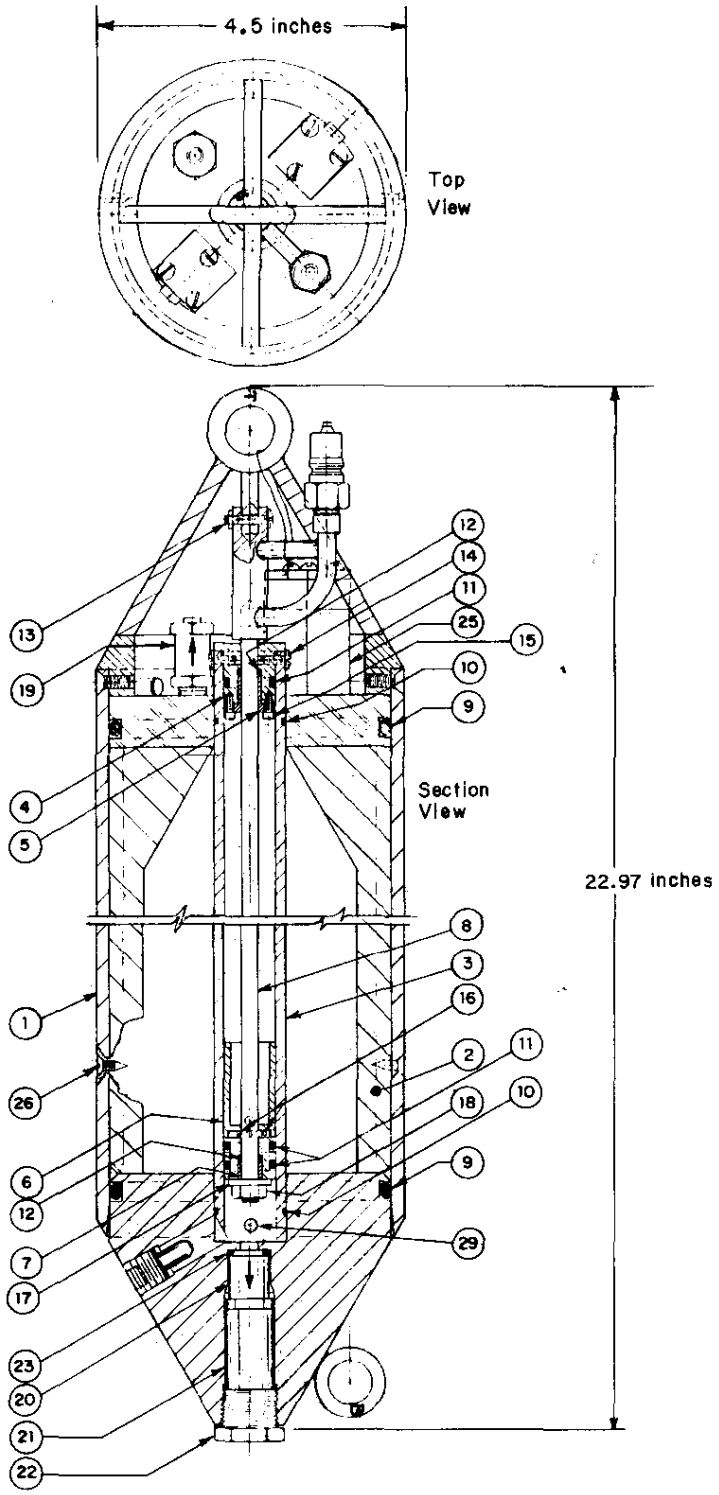

FIGURE A-1. Engineering Details of Sludge Collector

Sample Containment

The device is sealed by cylindrical surfaces sliding past elastomer O-rings. The collector is pressure-tight when closed, and the seals are not affected by pressure. The probability is small that the collected material will be diluted or leak out as the closed collector is raised through the supernate and the water spray jet.

Decontamination

The collector can be disassembled quickly and completely with only a few hand tools, and thus can be decontaminated very effectively. Reassembly, inspection, and lubrication of the decontaminated collector can be done easily in a hood by two workers in less than an hour.

Spoolpiece

Parts

The stainless-steel spoolpiece is 4.1 inches in diameter and 18.375 inches in length. The top end is a flat disc that has two latches (piece 25) and a small check valve (piece 19) mounted on it. The lower end of the spoolpiece is conical for easy penetration of the sludge. Both ends have O-ring grooves machined around the outside circumference. When the collector is closed, the O-rings (pieces 9) seal the cover over the spoolpiece.

Fabrication

The end pieces are separated and positioned by four guide bars (piece 2). The axis of the spoolpiece is a hydraulic cylinder (piece 3) sealed in place by O-rings (pieces 10) and locked by a bolt (piece 29). During fabrication the cylinder was used to align the partly machined end plates while welding in the guide bars. Final machining of the outside surface and the large O-ring grooves was done after the welding operation.

O-Ring Grooves

The large O-ring grooves are machined to dimensions that permit the cover barely to deform the O-rings. The groove depth was critical and was determined experimentally. If the grooves were too deep, the O-rings would not seal; if too shallow, excessive force would be required to move the cover over the O-rings. The force requirement could be modified to a limited degree

without remachining a groove. For example, if the force was too high, the groove was not deep enough, but some reduction in the force required could be obtained by substituting a 4-inch-OD O-ring for the 4.125-inch-OD O-ring normally used. If the force was too low, the groove was too deep, but could be built up by wrapping the bottom of the groove with thin, adhesive, fiberglass-reinforced, plastic tape. A single batch of O-rings was used throughout the sampling campaign to minimize dimensional variations.

Check Valve

Any internal pressure that might be built up in the closed collector is relieved with a check valve (piece 19). When the collector is closed at the bottom of a waste tank, a bubble of air at perhaps 2 atm pressure could be trapped in the sample compartment. Such internal pressure could increase the possibility of leakage during transport and also create a hazard when opening the collector. The check valve serves as a pressure relief.

Other Features

The possibility that large, hard particles such as sand might cause the collector to jam is minimized by the cross-sectional shape of the four guide bars and by a groove around the bottom circumference just above the large O-ring groove. Support rings (lifting eyes) are provided at both top and bottom of the collector. The bottom support ring is part of the conical spoolpiece end and could be used for lowering the collector horizontally, if desired to obtain a sample from the surface of a sludge layer. The bottom ring also is useful as a grasping point for opening the collector by pulling it apart. The collector was locked in the open position for shipment to a waste tank area by connecting a metal rod between the two support rings.

Cover

Parts

The cover (piece 1) was machined from 4-inch stainless steel pipe. The inside diameter of the cover is smaller near each end, where the O-rings seal, than throughout the central portion. Thus, there is no drag during most of its closing travel. Four 0.25-inch holes are located around the cover near the top, to drain any supernate or wash water that might collect in the cuplike top of the closed collector. Two screws (piece 26) in the lower part of the cover prevent rotation with respect to the spoolpiece and also limit the amount the collector can be opened; the threads were sealed with a coat of heavy grease during assembly to prevent leakage.

Fabrication

The spoolpiece and hydraulic system were assembled with the partly fabricated cover to give proper alignment before welding on the framework to top support ring. To reduce the force needed to close the collector, the bottom edge of the cover was machined with only a 6° taper. This feature gives a large mechanical advantage as the lower O-ring is being compressed.

Latches

Mechanical latches (piece 25) are staggered to provide four successive locks as the cover closes over the spoolpiece. Several features of the hydraulic system also tend to keep the collector closed. Redundant mechanical latches were provided after the collector opened prematurely on one occasion during the sampling campaign. Springs in the mechanical latches were sometimes damaged during decontamination and usually had to be restretched or replaced.

Support Ring and Cable

The top support ring of the cover is the point of attachment for the cable used to lower the collector in a vertical position. During the sampling campaign, the cable occasionally became tangled on the winch reel. This was prevented by keeping the cable under tension throughout the sampling procedure and by hanging a weight on the cable during shipment of the empty cask. Occasionally, the collector would rotate while being lowered into a waste tank, causing the water line and trip wire to wrap around the support cable and preventing the trip wire from functioning properly. At these times the full, closed collector had to be raised to near the top of the waste tank by carefully pulling the cable and water line simultaneously, until the trip wire would operate to disengage the water line. Such spinning could be reduced by keeping the water line, but not the trip wire, taut as the collector was lowered into a waste tank.

Hydraulic System

Parts

The hydraulic cylinder (piece 3) is fabricated from 3/4-inch, schedule 80, stainless steel pipe. The packing gland (piece 4) and piston (piece 6) are made of brass to give reduced friction in the sliding contact with the hydraulic cylinder. The piston "rod" (piece 8) is a 0.25-inch-diameter tube through which water is delivered to the cylinder, discharging from a side opening

in the tube near the piston; this arrangement avoids otherwise cumbersome means of delivering water to the cylinder. The piston (pieces 6, 7, 16, 17, and 18) and packing gland (pieces 4, 5, and 15) have many parts for ease in disassembly and reassembly. The long skirt on the piston reduces the probability for the piston to cock and jam in the cylinder. The piston has two O-rings (pieces 11) to help assure sealing, even if a machining flaw or other rough spot should exist in the cylinder. The O-ring groove nearest the skirt is notched on the skirt side to relieve any pressure that might otherwise build up between the two O-rings.

Quick-Disconnect Fitting

A Hansen quick-disconnect fitting was used to attach the water line to the hydraulic cylinder. This brand of fitting is easy to trigger open with a trip wire, and the part attached to the collector is easy to decontaminate. The seal at the end of the fitting is clearly exposed and can be opened by manually depressing it.

Operating Water Pressures

The pressure nominally required to close the collector is about 40 psig, but may temporarily rise to 80 psig as the bottom and top O-rings are compressed. When the collector is completely closed, the water pressure is increased to about 200 psig; then the quick-disconnect fitting is triggered to free the water line. Pressure is left in the hydraulic cylinder during shipment to assure that the collector will remain closed even if the mechanical latches were to fail.

Pressure Relief Valve

A pressure relief valve (piece 20) for the hydraulic cylinder is installed in the bottom cone. Its purpose is to reduce the tendency for the collector to close prematurely, should it come to rest on its bottom end. The valve permits flow of water from the volume ahead of the piston only when the pressure difference exceeds 10 psi. During assembly, water is preloaded into the cylinder through the opening in the side of the bottom cone. Then the opening is plugged so that water can escape only through the pressure relief valve. Shown in Figure A-1 is a low-pressure check valve that was modified to open at 10-psi pressure differential; alternatively, a commercially available 10-psi valve could be used. Further protection against premature closing while lowering the sampler is obtained by valving off the water

supply at the pump, so that any downward movement of the piston would create a partial vacuum above the piston and thus give an additional restraining force against closing.

Other Features

To open the collector, the 1/4-inch pipe plug is removed from the side opening of the spoolpiece cone and screwed into the bushing (piece 22). Then, if necessary, water can be pumped into the volume beneath the piston, through the side opening of the cone. For easier handling with manipulators, a socket hex-drive plug could be substituted for the pipe plug. The hydraulic system is attached to the cover assembly by a connecting screw (piece 13); during reassembly after decontamination, this screw proved difficult to insert and tighten. Care should be taken during decontamination to avoid bending the 0.25-inch tubular piston rod (piece 8), because even a slight bend would cause it to hang up in the gland (piece 12).

APPENDIX B – ANALYTICAL DATA FOR PROCESSING STEPS FOR TANK 13

		Na^+			NO_3^-		
	Amount[a]	*Conc.*[b]	*grams*	*moles*	*Conc.*[b]	*grams*	*moles*
Sample 13-1							
Supernate	480	3.48	38.4	1.67	2.15	64.0	1.03
Raw sludge (dried)	1800	20.95	377.1	16.40			
1st wash	1000	6.08	139.8	6.08	3.92	243.0	3.92
2nd wash	2000	2.82	129.7	5.64	1.87	231.9	3.74
Total, washes			269.5	11.72		474.9	7.66
Product 13-1	627	16.26	102.0	4.43	16.99[d]	106.5[d]	1.72[d]
Sample 13-2							
Supernate	400	4.90	45.1	1.96	2.29	56.8	0.92
Raw sludge (dried)	1800	20.87	375.7	16.33	19.19[d]	345.4[d]	5.57[d]
1st wash	2000	5.11	235.1	10.22	2.43	301.3	4.86
2nd wash	1500	1.98	68.3	2.97	0.93	86.5	1.40
Total, washes			303.4	13.19		387.8	6.26
Product 13-2	553	9.93	54.9	2.39	10.13[d]	56.0[d]	0.90[d]
Sample 13-3							
Supernate	300	5.22	36.0	1.57	2.97	55.2	0.89
Raw sludge (dried)	1650	23.33	384.9	16.74	24.43[d]	403.1[d]	6.50[d]
1st wash	2500	4.32	248.4	10.80	2.87	444.9	7.18
2nd wash	2800	1.40	90.2	3.92	0.85	147.6	2.38
Total, washes			338.6	14.72		592.5	9.56
Product 13-3	466	8.40	39.1	1.70	7.94[d]	37.0[d]	0.60[d]
Sample 13-4							
Supernate	600	4.90	67.6	2.94	2.36	87.8	1.42
Raw sludge (dried)	1850	18.57	343.5	14.94	23.28[d]	430.7[d]	6.95[d]
1st wash	2500	4.66	268.0	11.65	2.44	378.2	6.10
2nd wash	2600	0.50	30.0	1.30	0.80	129.0	2.08
Total, washes			298.0	12.95		507.2	8.18
Product 13-4	538	8.49	45.7	1.99	6.15[d]	33.1[d]	0.53[d]
Total, Products	2184	11.07	241.7	10.51	10.65[d]	232.6[d]	3.75[d]
Blended Products							
Calculated[e]	2085	10.90	227.3	9.89	10.45[d]	218.0[d]	3.51[d]
1st wash	2800	1.59	102.4	4.45	0.908	157.6	2.54
2nd wash	3100	0.235	16.8	0.73	0.131	25.2	0.41
3rd wash	3000	0.251	17.3	0.75	0.098	18.3	0.30
4th wash	2900	0.103	6.8	0.30	0.032	5.8	0.09
Total, washes			143.3	6.23		206.9	3.34
Final product, Tank 13	1375	3.06	42.1	1.83	0.512	7.0	0.11

a. Volume (ml) for supernates and washes or weight (g) for raw sludges and products.

b. Moles/liter for solutions or wt % for solids.

c. Total sulfate values for solids may be high by a factor of 5 to 10.

d. Leachable amount.

e. From amounts actually blended.

		NO_2^-			SO_4^{2-}		
	Amount[a]	*Conc.*[b]	*grams*	*moles*	*Conc.*[b]	*grams*	*moles*
Sample 13-1							
Supernate	480	0.76	16.8	0.36	0.052	2.4	0.03
Raw sludge (dried)	1800				8.80[c]	158.4	1.65
1st wash	1000	0.98	45.1	0.98	0.11	10.6	0.11
2nd wash	2000	0.46	42.3	0.92	0.049	9.4	0.10
Total, washes			87.4	1.90		20.0	0.21
Product 13-1	627	3.12[d]	19.6[d]	0.43[d]	3.99[c]	25.0	0.26
Sample 13-2							
Supernate	400	1.00	18.4	0.40	0.049	1.9	0.02
Raw sludge (dried)	1800	7.02[d]	126.4[d]	2.75[d]	7.78[c]	140.0	1.46
1st wash	2000	1.25	115.0	2.50	0.059	11.3	0.12
2nd wash	1500	0.48	33.1	0.72	0.025	3.6	0.04
Total, washes			148.1	3.22		14.9	0.16
Product 13-2	553	3.17[d]	17.5[d]	0.38[d]	6.31[c]	34.9	0.36
Sample 13-3							
Supernate	300	0.85	11.7	0.26	0.070	2.0	0.02
Raw sludge (dried)	1650	7.05[d]	116.3[d]	2.53[d]	7.85[c]	129.5	1.35
1st wash	2500	0.91	104.7	2.28	0.072	17.3	0.18
2nd wash	2800	0.33	42.5	0.92	0.022	5.9	0.06
Total, washes			147.2	3.20		23.2	0.24
Product 13-3	466	1.28[d]	6.0[d]	0.13[d]	4.75[c]	22.1	0.23
Sample 13-4							
Supernate	600	0.90	24.8	0.54	0.045	2.6	0.03
Raw sludge (dried)	1850	6.05[d]	111.9[d]	2.43	2.97[c]	54.9	0.57
1st wash	2500	0.94	108.1	2.35	0.055	13.2	0.14
2nd wash	2600	0.37	44.3	0.96	0.019	4.7	0.05
Total, washes			152.4	3.31		17.9	0.19
Product 13-4	538	1.13[d]	6.1[d]	0.13[d]	5.04[c]	27.1	0.28
Total, Products	2184	2.25[d]	49.2[d]	1.07[d]	5.00[c]	109.1	1.13
Blended Products							
Calculated[e]	2085	2.23[d]	46.5[d]	1.01[d]	5.04[c]	105.2	1.10
1st wash	2800				0.150	40.3	0.42
2nd wash	3100				0.032	9.6	0.10
3rd wash	3000				0.035	10.0	0.10
4th wash	2900				0.019	5.2	0.05
Total, washes						65.1	0.67
Final product, Tank 13	1375	0.033	0.5	0.01	2.69[c]	36.9	0.38

	Volume, ml	$Al(OH)_4^-$ moles/liter	grams	moles	OH^- moles/liter	grams	moles
Sample 13-1							
Supernate	480	0.17	7.8	0.08	1.21	9.9	0.58
1st wash	1000	0.31	29.5	0.31	1.21	20.6	1.21
2nd wash	2000	0.36	68.4	0.72	0.42	14.3	0.84
Total, washes			97.9	1.03		34.9	2.05
Sample 13-2							
Supernate	400	0.28	10.6	0.11	1.68	11.4	0.67
1st wash	2000	0.64	121.6	1.28	0.86	29.2	1.72
2nd wash	1500	0.15	21.4	0.23	0.38	9.7	0.57
Total, washes			143.0	1.51		38.9	2.29
Sample 13-3							
Supernate	300	0.26	7.4	0.08	1.57	8.0	0.47
1st wash	2500	0.31	73.6	0.78	0.90	38.3	2.25
2nd wash	2800	0.13	34.6	0.36	0.23	10.9	0.64
Total, washes			108.2	1.14		49.2	2.89
Sample 13-4							
Supernate	600	0.29	16.5	0.17	1.59	16.2	0.95
1st wash	2500	0.024	4.8	0.05	1.20	51.0	3.00
2nd wash	2600	0.20	50.1	0.53	0.30	13.0	0.77
Total, washes			54.9	0.58		64.0	3.77

	Volume, ml	CO_3^{2-} moles/liter	grams	moles
Sample 13-1				
Supernate	480	0.12	3.5	0.06
1st wash	1000	0.20	12.0	0.20
2nd wash	2000	0.16	19.2	0.32
Total, washes			31.2	0.52
Sample 13-2				
Supernate	400	0.087	2.1	0.04
1st wash	2000	0.11	13.2	0.22
2nd wash	1500	0.02	1.8	0.03
Total, washes			15.0	0.25
Sample 13-3				
Supernate	300	0.07	1.3	0.02
1st wash	2500	0.10	15.0	0.25
2nd wash	2800	0.05	8.4	0.14
Total, washes			23.4	0.39
Sample 13-4				
Supernate	600	0.08	2.9	0.05
1st wash	2500	0.12	18.0	0.30
2nd wash	2600	0.049	7.6	0.13
Total, washes			25.6	0.43

	Weight, grams	*Leachable Na+*			*Leachable NO_3^-*		
		Wt %	*grams*	*moles*	*Wt %*	*grams*	*moles*
Blended Products							
Wet cake after 2nd wash	1500	0.956	14.3	0.62	0.936	14.0	0.23
Total component		3.73	56.0	2.43	3.25	48.7	0.79
Wet cake after 4th wash	1400	0.001	0.01	0.0006	0.36	5.0	0.08
Total component		2.66	37.3	1.62	*a*	-	-
Final product, Tank 13	1375	0.96	13.2	0.57	0.339	4.7	0.08
		Fe			*Mn*		
Sample 13-1							
Raw sludge (dried)	1800	3.57	64.3	1.15	1.1	19.8	0.36
Product 13-1	627	11.13	69.8	1.25	4.5	28.2	0.51
Sample 13-2							
Raw sludge (dried)	1800	4.85	87.3	1.56	2.1	37.8	0.69
Product 13-2	553	11.56	63.9	1.14	4.5	24.9	0.45
Sample 13-3							
Raw sludge (dried)	1650	5.72	94.4	1.69	2.5	41.3	0.75
Product 13-3	466	16.67	77.7	1.39	6.3	29.4	0.53
Sample 13-4							
Raw sludge (dried)	1850	8.07	149.3	2.67	3.48	64.4	1.17
Product 13-4	538	18.71	100.7	1.80	9.0	48.4	0.88
Total, Products	2184	14.29	312.1	5.58	5.99	130.9	2.37
Blended Products							
Calculated[b]	2085	14.46	301.6	5.39	6.07	126.6	2.30
Final product, Tank 13	1375	27.89	383.4	6.85	8.80	121.0	2.20

a. None detected.

b. From amounts actually blended, before washes.

	Weight, grams	Leachable NO_2^- Wt %	grams	moles	Leachable SO_4^{2-} Wt %	grams	moles
Blended Products							
Wet cake after 2nd wash	1500				0.84	12.6	0.13
Total component					4.01	60.2	0.63
Wet cake after 4th wash	1400				0.81	11.3	0.12
Total component					3.89	54.5	0.57
Final product, Tank 13	1375	0.018	0.3	0.005	0.841	11.6	0.12
		Al			Hg		
Sample 13-1							
Raw sludge (dried)	1800	7.57	136.3	5.05	1.20	21.5	0.11
Product 13-1	627	3.10	19.4	0.72	0.666	4.2	0.02
Sample 13-2							
Raw sludge (dried)	1800	9.89	178.0	6.59	1.87	33.7	0.17
Product 13-2	553	8.04	44.5	1.65	2.24	12.4	0.06
Sample 13-3							
Raw sludge (dried)	1650	7.2	118.8	4.40	0.990	16.3	0.08
Product 13-3	466	5.9	27.5	1.02	1.60	7.4	0.04
Sample 13-4							
Raw sludge (dried)	1850	5.46	101.0	3.74	0.940	17.4	0.09
Product 13-4	538	3.6	19.4	0.72	1.62	8.7	0.04
Total, Products	2184	5.07	110.8	4.11	1.50	32.7	0.16
Blended Products							
Calculated	2085	5.15	107.5	3.98	1.53	31.9	0.16
Final product, Tank 13	1375	7.07	97.2	3.60	2.06	28.3	0.14

TANK 13 ANALYTICAL RESULTS (Continued)

		Gross α		^{90}Sr	
	Amount[a]	*Activity*[b]	*d/min*	*Activity*[b]	*d/min*
Sample 13-1					
Supernate	480	$<2 \times 10^5$	$<1 \times 10^8$	3.52×10^5	1.69×10^8
Raw sludge (dried)	1800	1.53×10^8	2.75×10^{11}	1.46×10^{10}	2.63×10^{13}
1st wash	1000	$<2 \times 10^5$	$<2.0 \times 10^8$	3.42×10^4	3.42×10^7
2nd wash	2000	$<7 \times 10^4$	$<1.4 \times 10^8$	1.54×10^3	0.31×10^7
Total, washes			$<3.4 \times 10^8$		3.73×10^7
Product 13-1	627	3.71×10^8	2.33×10^{11}	1.56×10^{10}	9.78×10^{12}
Sample 13-2					
Supernate	400	7.08×10^4	2.83×10^7	1.22×10^6	4.88×10^8
Raw sludge (dried)	1800	1.72×10^8	3.10×10^{11}	1.97×10^{10}	3.15×10^{13}
1st wash	2000	1.02×10^4	2.04×10^7	1.91×10^5	3.82×10^8
2nd wash	1500	$<1 \times 10^4$	$<1.5 \times 10^7$	4.29×10^5	6.44×10^8
Total, washes			$<3.5 \times 10^7$		1.03×10^9
Product 13-2	553	3.71×10^8	2.05×10^{11}	1.97×10^{10}	1.09×10^{13}
Sample 13-3					
Supernate	300	6.98×10^4	2.09×10^7	2.12×10^5	6.36×10^7
Raw sludge (dried)	1650	1.63×10^8	2.69×10^{11}	1.48×10^{10}	2.44×10^{13}
1st wash	2500	$<3 \times 10^3$	$<8 \times 10^6$	1.45×10^7	3.63×10^{10}
2nd wash	2800	$<3 \times 10^3$	$<8 \times 10^6$	6.85×10^6	1.92×10^{10}
Total, washes			$<1.6 \times 10^7$		5.55×10^{10}
Product 13-3	466	4.76×10^8	2.22×10^{11}	3.27×10^{10}	1.52×10^{13}
Sample 13-4					
Supernate	600	3×10^4	2×10^7	1.23×10^7	7.38×10^9
Raw sludge (dried)	1850	2.01×10^8	3.72×10^{11}	1.07×10^{10}	1.98×10^{13}
1st wash	2500	4×10^3	1×10^7	9.26×10^6	2.32×10^{10}
2nd wash	2600	$<3 \times 10^3$	$<8 \times 10^6$	7.11×10^6	1.85×10^{10}
Total, washes			$<2 \times 10^7$		4.17×10^{10}
Product 13-4	538	4.62×10^8	2.49×10^{11}	3.58×10^{10}	1.93×10^{13}
Total, Products	2184	4.16×10^8	9.09×10^{11}	2.53×10^{10}	5.52×10^{13}
Blended Products					
Calculated[d]	2085	4.17×10^8	8.70×10^{11}	2.55×10^{10}	5.32×10^{13}
Final Product, Tank 13	1375	6.24×10^8	8.58×10^{11}	3.44×10^{10}	4.73×10^{13}

a. Volume (ml) for supernates and washes or weight (g) for raw sludges and products.

b. d/(min)(ml) for solutions or d/(min)(g) for solids.

c. None detected

d. From amounts actually blended, before washes.

		^{137}Cs		^{134}Cs		^{144}Ce	
	Amount[a]	*Activity*[b]	*d/min*	*Activity*[b]	*d/min*	*Activity*[b]	*d/min*
Sample 13-1							
Supernate	480	8.30×10^{8}	3.98×10^{11}	3.98×10^{7}	1.91×10^{10}	$<6.48\times10^{4}$	$<3.11\times10^{7}$
Raw sludge (dried)	1800	2.25×10^{9}	4.05×10^{12}	8.63×10^{7}	1.55×10^{11}	1.86×10^{9}	3.35×10^{12}
1st wash	1000	1.75×10^{9}	1.75×10^{12}	7.76×10^{7}	7.76×10^{10}	6.88×10^{3}	6.88×10^{6}
2nd wash	2000	7.74×10^{8}	1.55×10^{12}	3.39×10^{7}	6.78×10^{10}	$<2.68\times10^{4}$	$<5.36\times10^{7}$
Total, washes			3.30×10^{12}		14.54×10^{10}		$<6.05\times10^{7}$
Product 13-1	627	1.54×10^{9}	9.66×10^{11}	4.63×10^{7}	2.90×10^{10}	3.75×10^{9}	2.35×10^{12}
Sample 13-2							
Supernate	400	1.40×10^{9}	5.60×10^{11}	5.46×10^{7}	2.18×10^{10}	3.44×10^{4}	1.38×10^{7}
Raw sludge (dried)	1800	2.52×10^{9}	4.54×10^{12}	8.45×10^{7}	1.52×10^{11}	1.08×10^{9}	1.94×10^{12}
1st wash	2000	1.39×10^{9}	2.78×10^{12}	5.02×10^{7}	1.00×10^{11}	$<3.76\times10^{4}$	$<7.52\times10^{7}$
2nd wash	1500	5.57×10^{8}	0.84×10^{12}	2.01×10^{7}	0.30×10^{11}	$<7.44\times10^{3}$	$<1.12\times10^{7}$
Total, washes			3.62×10^{12}		1.30×10^{11}		$<8.64\times10^{7}$
Product 13-2	553	1.53×10^{9}	8.47×10^{11}	$<3.49\times10^{7}$	$<1.93\times10^{10}$	2.87×10^{9}	1.58×10^{12}
Sample 13-3							
Supernate	300	1.27×10^{9}	3.81×10^{11}	6.30×10^{7}	1.89×10^{10}	$<3.66\times10^{4}$	$<1.10\times10^{7}$
Raw sludge (dried)	1650	2.11×10^{9}	3.48×10^{12}	1.03×10^{8}	1.70×10^{11}	2.04×10^{9}	3.37×10^{12}
1st wash	2500	1.28×10^{9}	3.20×10^{12}	6.07×10^{7}	1.52×10^{11}	c	-
2nd wash	2800	3.39×10^{8}	0.95×10^{12}	1.59×10^{7}	0.45×10^{11}	c	-
Total, washes			4.15×10^{12}		1.97×10^{11}		-
Product 13-3	466	9.17×10^{8}	4.27×10^{11}	$<3.36\times10^{7}$	$<1.57\times10^{10}$	4.72×10^{9}	2.20×10^{12}
Sample 13-4							
Supernate	600	1.34×10^{9}	8.04×10^{11}	5.94×10^{7}	3.56×10^{10}	c	-
Raw sludge (dried)	1850	2.58×10^{9}	4.77×10^{12}	9.35×10^{7}	1.73×10^{11}	1.49×10^{9}	2.76×10^{12}
1st wash	2500	1.29×10^{9}	3.23×10^{12}	5.19×10^{7}	1.30×10^{11}	c	-
2nd wash	2600	3.80×10^{8}	0.99×10^{12}	1.47×10^{7}	0.38×10^{11}	c	-
Total, washes			4.22×10^{12}		1.68×10^{11}		-
Product 13-4	538	1.21×10^{9}	6.51×10^{11}	7.46×10^{7}	4.01×10^{10}	4.59×10^{9}	2.47×10^{12}
Total, Products	2184	1.32×10^{9}	28.91×10^{11}	$<4.77\times10^{7}$	$<10.41\times10^{10}$	3.94×10^{9}	8.60×10^{12}
Blended Products							
Calculated[d]	2085	1.32×10^{9}	27.46×10^{11}	$<4.78\times10^{7}$	$<9.96\times10^{10}$	3.23×10^{9}	6.73×10^{12}
Final Product, Tank 13	1375	6.75×10^{8}	9.28×10^{11}	$<6.06\times10^{6}$	$<0.83\times10^{10}$	4.46×10^{9}	6.13×10^{12}

	Amount[a]	^{95}Zr Activity[b]	^{95}Zr d/min	^{95}Nb Activity[b]	^{95}Nb d/min
Sample 13-1					
Supernate	480	$<2.45\times10^{4}$	$<1.18\times10^{7}$	$<8.34\times10^{3}$	$<4.00\times10^{6}$
Raw sludge (dried)	1800	2.01×10^{7}	3.62×10^{10}	6.55×10^{7}	1.18×10^{11}
1st wash	1000	$<3.27\times10^{3}$	$<3.37\times10^{6}$	$<4.30\times10^{2}$	$<4.30\times10^{5}$
2nd wash	2000	$<3.74\times10^{3}$	$<7.48\times10^{6}$	$<2.69\times10^{3}$	$<5.38\times10^{6}$
Total, washes			$<1.09\times10^{7}$		$<5.81\times10^{6}$
Product 13-1	627	$<1.42\times10^{7}$	$<8.90\times10^{9}$	1.32×10^{8}	8.28×10^{10}
Sample 13-2					
Supernate	400	$<8.08\times10^{3}$	$<3.23\times10^{6}$	$<3.68\times10^{3}$	$<1.47\times10^{6}$
Raw sludge (dried)	1800	$<7.66\times10^{6}$	$<1.38\times10^{10}$	2.42×10^{7}	4.36×10^{10}
1st wash	2000	$<1.69\times10^{4}$	$<3.38\times10^{7}$	$<4.81\times10^{3}$	$<9.62\times10^{6}$
2nd wash	1500	$<2.17\times10^{3}$	$<3.26\times10^{6}$	$<1.87\times10^{3}$	$<2.81\times10^{6}$
Total, washes			$<3.71\times10^{7}$		$<1.24\times10^{7}$
Product 13-2	553	$<9.15\times10^{6}$	$<5.06\times10^{9}$	1.98×10^{7}	1.09×10^{10}
Sample 13-3					
Supernate	300	$<1.17\times10^{4}$	$<3.51\times10^{6}$	$<5.06\times10^{3}$	1.52×10^{6}
Raw sludge (dried)	1650	$<1.48\times10^{7}$	$<2.44\times10^{10}$	5.48×10^{7}	9.04×10^{10}
1st wash	2500	c	-	c	-
2nd wash	2800	c	-	c	-
Total, washes			-		-
Product 13-3	466	$<6.82\times10^{7}$	$<3.18\times10^{10}$	2.05×10^{8}	9.55×10^{10}
Sample 13-4					
Supernate	600	c	-	c	-
Raw sludge (dried)	1850	$<6.11\times10^{6}$	$<1.10\times10^{10}$	$<6.65\times10^{6}$	$<1.23\times10^{10}$
1st wash	2500	c	-	c	-
2nd wash	2600	c	-	c	-
Total, washes			-		-
Product 13-4	538	6.93×10^{7}	3.73×10^{10}	1.13×10^{8}	6.08×10^{10}
Total, Products	2184	$<3.80\times10^{7}$	$<8.31\times10^{10}$	1.14×10^{8}	2.50×10^{11}
Blended Products					
Calculated[d]	2085	$<3.90\times10^{7}$	$<8.14\times10^{10}$	1.15×10^{8}	2.39×10^{11}
Final Product, Tank 13	1375	$<1.39\times10^{7}$	$<1.91\times10^{10}$	4.54×10^{7}	6.24×10^{10}

a. Volume (ml) for supernates and washes or weight (g) for raw sludges and products.

b. d/(min)(ml) for solutions or d/(min)(g) for solids.

c. None detected.

d. From amounts actually blended, before washes.

	Amount[a]	^{103}Ru Activity[b]	^{103}Ru d/min	^{106}Ru Activity[b]	^{106}Ru d/min	^{154}Eu Activity[b]	^{154}Eu d/min
Sample 13-1							
Supernate	480	1.02×10^{5}	4.90×10^{7}	5.57×10^{6}	2.67×10^{9}	$<1.02\times10^{4}$	$<4.90\times10^{6}$
Raw sludge (dried)	1800	$<7.33\times10^{6}$	$<1.32\times10^{10}$	3.77×10^{8}	6.79×10^{11}	2.07×10^{8}	3.73×10^{11}
1st wash	1000	$<1.53\times10^{3}$	$<1.53\times10^{6}$	7.11×10^{4}	7.11×10^{7}	$<1.29\times10^{3}$	$<1.29\times10^{6}$
2nd wash	2000	$<6.09\times10^{3}$	$<1.22\times10^{7}$	2.18×10^{5}	4.36×10^{8}	$<4.19\times10^{3}$	$<8.38\times10^{6}$
Total, washes			$<1.37\times10^{7}$		5.07×10^{8}		$<9.67\times10^{6}$
Product 13-1	627	6.65×10^{6}	$<4.17\times10^{9}$	8.73×10^{8}	5.47×10^{11}	6.77×10^{8}	4.24×10^{11}
Sample 13-2							
Supernate	400	3.49×10^{4}	1.40×10^{7}	3.37×10^{6}	1.35×10^{9}	7.76×10^{3}	3.10×10^{6}
Raw sludge (dried)	1800	$<7.44\times10^{6}$	$<1.34\times10^{10}$	3.37×10^{8}	6.06×10^{11}	1.75×10^{8}	3.14×10^{11}
1st wash	2000	1.74×10^{5}	3.48×10^{8}	5.48×10^{6}	1.10×10^{10}	$<1.46\times10^{4}$	$<2.92\times10^{7}$
2nd wash	1500	1.36×10^{4}	2.04×10^{7}	1.28×10^{6}	0.19×10^{10}	$<1.55\times10^{3}$	$<2.33\times10^{6}$
Total, washes			3.68×10^{8}		1.29×10^{10}		$<3.15\times10^{7}$
Product 13-2	553	$<3.15\times10^{7}$	$<1.74\times10^{10}$	1.23×10^{9}	6.82×10^{11}	3.50×10^{8}	1.94×10^{11}
Sample 13-3							
Supernate	300	2.14×10^{5}	6.42×10^{7}	7.92×10^{6}	2.38×10^{9}	$<1.54\times10^{4}$	$<4.62\times10^{6}$
Raw sludge (dried)	1650	$<6.61\times10^{6}$	$<1.09\times10^{10}$	4.27×10^{8}	7.05×10^{11}	3.41×10^{8}	5.63×10^{11}
1st wash	2500	c	–	4.51×10^{6}	1.13×10^{10}	c	–
2nd wash	2800	1.15×10^{4}	3.22×10^{7}	1.35×10^{6}	3.78×10^{9}	c	–
Total, washes			3.22×10^{7}		1.51×10^{10}		–
Product 13-3	466	7.53×10^{7}	3.51×10^{10}	1.24×10^{9}	5.78×10^{11}	1.55×10^{9}	7.22×10^{11}
Sample 13-4							
Supernate	600	1.29×10^{5}	7.74×10^{7}	6.13×10^{6}	3.68×10^{9}	c	–
Raw sludge (dried)	1850	$<1.96\times10^{7}$	$<3.63\times10^{10}$	2.97×10^{8}	5.49×10^{11}	1.10×10^{11}	2.04×10^{11}
1st wash	2500	1.66×10^{4}	4.15×10^{7}	3.10×10^{6}	7.75×10^{9}	c	–
2nd wash	2600	2.13×10^{4}	5.54×10^{7}	1.19×10^{6}	3.09×10^{9}	c	–
Total, washes			9.69×10^{7}		1.08×10^{10}		–
Product 13-4	538	$<3.34\times10^{7}$	$<1.80\times10^{10}$	1.03×10^{9}	5.54×10^{11}	$<4.91\times10^{7}$	$<2.64\times10^{10}$
Total, Products	2184	$<3.42\times10^{7}$	$<7.47\times10^{10}$	1.08×10^{9}	2.36×10^{12}	$<6.25\times10^{8}$	$<13.66\times10^{11}$
Blended Products							
Calculated[d]	2085	$<3.51\times10^{7}$	$<7.31\times10^{10}$	1.09×10^{9}	2.27×10^{12}	$<6.30\times10^{8}$	$<13.13\times10^{11}$
Final Product, Tank 13	1375	1.66×10^{7}	2.28×10^{10}	8.90×10^{8}	1.22×10^{12}	6.70×10^{8}	9.21×10^{11}

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