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Evaluation of the ACT*DE*CONSM Process for Treating Gunitite Tank Sludge

Barry B. Spencer
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MANAGED AND OPERATED BY
LOCKHEED MARTIN ENERGY RESEARCH CORPORATION
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DEPARTMENT OF ENERGY

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Chemical Technology Division

EVALUATION OF THE ACT*DE*CONSM PROCESS FOR TREATING
GUNITE TANK SLUDGE

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LIST OF ACRONYMS

AES	Atomic Emission Spectroscopy
CASD	Chemical and Analytical Services Division
DOE	Department of Energy
EPA	Environmental Protection Agency
GAAT	Gunite and Associated Tanks
HLW	High-Level Radioactive Waste
ICP	Inductively Coupled Plasma
LLW	Low-Level Radioactive Waste
MS	Mass Spectrometry
MVST	Melton Valley Storage Tanks
ORNL	Oak Ridge National Laboratory
Selentec	Selective Environmental Technologies, Inc.
TRU	Transuranium
TRUEX	Transuranium Extraction



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ABSTRACT

As part of the sludge processing flow sheet development effort, a test to evaluate the ACT*DE*CONSM process for selectively removing actinides from Gunite tank sludge was initiated and completed. Mixed waste sludge from Gunite tank W-6 was subjected to the ACT*DE*CONSM selective leaching process. Nearly all of the transuranium (TRU)-waste content of this sludge was attributed to plutonium. The sludge sample was first washed with a mild caustic solution (~0.01 M NaOH) to remove excess sodium and nitrate associated with the interstitial liquid supernatant. The washed wet solids were treated with the ACT*DE*CONSM solvent using a ratio of ~20 mL of solvent per gram of wet solids. The sludge and solvent were separated by centrifugation, and the ACT*DE*CONSM treatment was repeated twice. On a wet basis the overall solvent-to-sludge ratio was ~60 mL/g, but on a dry basis the ratio was ~178 mL/g.

Samples of the spent solvents (leachates) and residual sludge solids were analyzed. The results indicated that ~71% of the solids in the sludge were dissolved, while ~80% of the TRU-waste components dissolved. A low separation of the TRU-waste components from other components of the sludge mixture is indicated. Almost all the uranium and calcium were removed from the sludge. On a dry solids basis, the total TRU-waste content of the washed sludge was 376 nCi/g and that of the treated sludge was 250 nCi/g. The process did not render the sludge a non-TRU waste.

For sludges wherein most of the TRU content is plutonium, the ACT*DE*CONSM process, as tested, is not effective in rendering the sludge a non-TRU waste. It is recommended that ACT*DE*CONSM be optimized for this specific application and that other processes utilizing different chelating and oxidizing agents be tested. Additionally, the ACT*DE*CONSM process should be tested on TRU mixed waste in which the bulk of the TRU components are elements other than plutonium.



1. INTRODUCTION

Both low-level radioactive wastes (LLW) and high-level radioactive wastes (HLW) in the charge of the U.S. Department of Energy (DOE) are located and stored at sites throughout the United States. Vitrification of the HLW in their present form, followed by deep geologic burial, results in enormous disposal costs (estimated at >\$100 billion). However, the radioactive elements comprise <1% of the total mass of the wastes. Pretreatment of the wastes to remove and isolate specific radionuclides is being considered as a means to reduce the volume of waste that must be treated by more costly methods. A pretreatment process that can remove transuranium (TRU) elements from the waste to produce a waste containing TRU-waste elements at concentrations <100 nCi/g will render the waste non-transuranic and eligible for less expensive, near-surface disposal options.

The ACT*DE*CONSM process, a proprietary process of Selective Environmental Technologies, Inc. (Selentec), is designed to remove actinides from soils or soil-like media. The process is proposed as an alternative for removal of actinides from tank-stored high-level-waste sludges. The ACT*DE*CONSM solvent is an aqueous carbonate solution containing a chelating agent and an oxidant. This combination of chemicals is rather benign and is intended to leach specific metal species from a soil matrix without destroying the bulk of the soil. The process has been tested as a means to decontaminate uranium- and plutonium-contaminated soils (Johnson et al., 1993), to remove actinides from a Hanford tank sludge (Lumetta and Geeting, 1995), and to remove actinides from a Savannah River waste sludge (Hay and Bibler, 1995). ACT*DE*CONSM pretreatment of waste sludges has met with limited success, possibly because the solvent formulation was not optimized for these waste forms.

The objective of the work reported here was to evaluate the ACT*DE*CONSM process for pretreating Gunite and Associated Tanks (GAAT) sludge as part of the sludge processing flow sheet development effort. Sludge samples from Gunite tanks W-3, W-6, and W-10 were collected as part of a characterization study (Bechtel National, 1995) and were available for the test. However, funding and schedule constraints permitted testing on sludge from only one of the tanks. The tests were carried out in consultation with representatives from Selentec, who participated in the selection of the sample to be tested, provided the formulation of the ACT*DE*CONSM solvent, and reviewed the test procedure.

The ACT*DE*CONSM process must be capable of removing insoluble radionuclides to be a viable pretreatment process. Because the sludges contain interstitial liquids having high concentrations of soluble metal nitrates (e.g., sodium nitrate), the sludge was washed with a mild sodium hydroxide (0.01 M) solution to remove these components. Evaluation of the results was based on the selectivity of the leaching, that is, whether the process leaches a greater fraction of the actinides than it does the bulk sludge solids.

2. DESCRIPTION OF EXPERIMENT

The purpose of the experiment was to provide data to evaluate the ACT*DE*CONSM process. Primarily this means that the tests and measurements must support identification of species insoluble in high pH media and permit estimations of the quantities of these species that are leached from the sludge and those that are not leached by the ACT*DE*CONSM solvent. When the sludge is centrifuged, it separates into a solids-rich phase and a liquid supernatant. (Centrifugation was the only practical method of liquid-solid separation using the equipment available, at the time, in the hot cell. The main advantage over filtration is the capability of keeping the solids in one container without losses to the filter media.) The supernatant can be decanted, but liquid remains in the void spaces between solid particles. This interstitial liquid contains soluble metal nitrates which can easily be reduced in concentration by washing the sludge with mild caustic solution. Wash solution containing 0.01 M sodium hydroxide was prepared with reagent-grade sodium hydroxide and distilled, demineralized water.

The ACT*DE*CONSM solvent is an aqueous solution of sodium carbonate, a chelating agent, and a mild oxidizing agent. It was prepared, using reagent-grade chemicals, according to a formula provided by Selentec. The proprietary information agreement with Selentec permitted representatives from Selentec to participate in the selection of the sludge sample to be tested, develop the test procedure as a joint effort, and be present during the test.

2.1 SELECTION OF SLUDGE SAMPLE

As part of a characterization study performed by the Chemical and Analytical Services Division (CASD), sludge samples were available that had been previously retrieved from the Gunite tanks (Bechtel National, 1995). About 10 g of centrifuged wet solids from each of the three tanks, W-3, W-6, and W-10, were made available for this study. Analyses of sludges

from these three tanks have been previously reported (Bechtel National, 1995), and the results of those analyses are summarized in Table 2.1. The quantities of radionuclides in the sludge are very small, as shown in Table 2.2, and contribute a negligible amount to the total mass of the sludge. Because the ACT*DE*CONSM process was to be tested on only one sludge (due to budget and schedule constraints), it was necessary to select which of the three sludge samples to use in the test. The selection was made in cooperation with Selentec, for whom Tables 2.1 and 2.2 were made available.

The sludge sample from Gunitite tank W-6 was selected for the ACT*DE*CONSM leaching test. Several factors were considered in the relatively qualitative decision process. A sludge with a low-to-moderate TRU content was desired so the solvent-to-sludge ratio would not be high; this had implications as to the volume of the centrifuge bottles that were available for use as process vessels. Sludge from W-6 contained 111 nCi/g TRU waste, but this was on a wet basis and included only the americium and plutonium because regulatory definitions of TRU waste excluded transuranium elements having half-lives less than 20 y (e.g., curium). On a dry basis, W-6 sludge contains 323 nCi/g TRU waste and is therefore well above the definition of TRU waste. For test purposes, removal of the curium was considered important, and W-6 sludge contained a moderate amount of curium. Low concentrations of uranium and thorium were desirable because these elements were thought to be easily leached by ACT*DE*CONSM; a measurable quantity of residue depleted in TRU-waste elements was wanted. For similar reasons, the high concentrations of iron, silicon, and aluminum were desirable to demonstrate the selectivity of the leaching. Lastly, the concentrations of ¹³⁷Cs and ⁹⁰Sr, significant sources of radiation exposure, were low enough to reduce concerns regarding handling the materials.

2.2 EXPERIMENTAL PROCEDURES

A detailed test procedure was prepared, reviewed by Selentec personnel, and modified to include comments. The test procedure is provided in the Appendix for reference. The essential details of the experiment are summarized in this section.

Two methods were used to contact the sludge solids with liquid solutions. In one method, referred to as washing, the solids were mixed with the liquid solution for ~60 s using a Vortex mixer. Solids are not expected to be dissolved into the wash solution; it is only necessary to ensure that the interstitial liquid is well mixed with the wash solution. The other contact method, called leaching, required longer contact times to allow solids to dissolve in

Table 2.1. Data on selected GAAT sludge solids concentrations^a

	Molecular weight	Tank W-3	Tank W-6	Tank W-10
General				
Density, g/mL		1.07	1.19	1.13
pH		10.47	11.14	10.62
TOC, mg/L		5300.	2400.	4900.
TIC, mg/L		<2400.	3300.	5100.
Total solids g/g		0.424	0.344	0.272
Radionuclides				
		(nCi/g)	(nCi/g)	(nCi/g)
⁶⁰ Co	60.	<1.4	<15.1	194.6
⁹⁰ Sr/ ⁹⁰ Y	90.	15660.	5130.	64800.
¹³⁷ Cs/ ^{137m} Ba	137.	1243.	2430.	45900.
¹⁵² Eu	152.	<5.9	<10.	91.9
¹⁵⁴ Eu	154.	<3.5	12.4	124.
¹⁵⁵ Eu	155.	<12.2	<1.6	<48.6
²³⁸ Pu/ ²⁴¹ Am	238./241.	11.	19.	622.
^{239/240} Pu	239./240	182.	92.	201.
²⁴⁴ Cm	244.	5.	186.	1360.
Gross alpha		324.	297.	2190.
Gross beta		27000.	29700.	205400.
Total TRU		193.	111.	823.
Other metals				
		(mg/g)	(mg/g)	(mg/g)
Ag	107.87	<0.0011	0.0086	0.00209
Al	26.98	51.1	10.9	30.2
B	10.81	<0.00252	0.00636	0.00941
Ba	137.33	0.00901	0.210	0.0753
Be	9.01	<0.0002	<0.0001	0.0104
Ca	40.08	7.16	31.6	5.96
Cd	112.41	0.0013	0.00495	0.00468
Co	58.93	<0.00147	0.00489	<0.00174
Cr	52.00	0.468	1.39	0.171
Cu	63.55	0.0175	0.0312	0.0868
Fe	55.85	2.89	14.7	8.40
Hg	200.59	0.00642	0.0402	0.294
K	39.10	0.381	0.595	3.86
Mg	24.31	0.303	0.746	0.592
Mn	54.94	0.127	1.51	0.152
Na	22.99	16.9	43.9	14.7
Ni	58.70	0.0057	0.102	0.0712
Pb	207.2	<0.0714	1.01	0.473
Si	28.09	0.509	3.36	N.M. ^c
Sr	87.62	0.0175	0.0545	0.034
Th	232.04	<0.340	0.679	6.25
Tl	204.37	<0.0252	0.0361	<0.030
U	238.03	128.	8.86	20.5
Zn	65.38	0.0182	0.0899	0.110
Anions				
		(mg/g)	(mg/g)	(mg/g)
Br ⁻	79.90	<0.005	<0.064	<0.046
Cl ⁻	35.45	<0.005	<0.064	0.546
F ⁻	19.00	0.024	11.9	0.437
CO ₃ ²⁻	60.00	<11.2 ^b	13.9 ^b	22.6 ^b
NO ₃ ⁻	62.00	0.087	12.3	6.27
PO ₄ ³⁻	94.97	3.24	7.90	<0.455
SO ₄ ²⁻	96.06	0.318	9.40	2.87

^aData taken from ORNL/ER/Sub/87-99053/74. As, Sb, Se, and V were below the detectable limit.

^bEstimated from TIC.

^cN.M. = not measured.

Table 2.2. Concentrations of radionuclides in GAAT sludges (wet basis)^a

Species	Molecular weight	Tank W-3 (mg/g)	Tank W-6 (mg/g)	Tank W-10 (mg/g)
⁶⁰ Co	60.	$<1.23 \times 10^{-9}$	$<1.32 \times 10^{-8}$	1.71×10^{-7}
⁹⁰ Sr/ ⁹⁰ Y	90.	1.08×10^{-4}	3.54×10^{-5}	4.47×10^{-4}
¹³⁷ Cs/ ^{137m} Ba	137.	1.44×10^{-5}	2.81×10^{-5}	5.31×10^{-4}
¹⁵² Eu	152.	$<3.22 \times 10^{-8}$	$<5.46 \times 10^{-8}$	5.02×10^{-7}
¹⁵⁴ Eu	154.	$<2.40 \times 10^{-8}$	8.49×10^{-8}	8.49×10^{-7}
¹⁵⁵ Eu	155.	$<8.97 \times 10^{-9}$	$<1.18 \times 10^{-9}$	3.57×10^{-8}
²³⁸ Pu/ ²⁴¹ Am	238.	6.63×10^{-7}	1.14×10^{-6}	3.75×10^{-5}
^{239/240} Pu	239.	2.97×10^{-3}	1.50×10^{-3}	3.29×10^{-3}
²⁴⁴ Cm	244.	6.24×10^{-8}	2.32×10^{-6}	1.70×10^{-5}

^aCalculated from data in Table 2.1 and the specific activity of the listed isotope; specific activity of ⁹⁰Sr used for ⁹⁰Sr/⁹⁰Y, ¹³⁷Cs for ¹³⁷Cs/^{137m}Ba, ²³⁸Pu for ²³⁸Pu/²⁴¹Am, and ²³⁹Pu for ^{239/240}Pu.

the liquid reagent. To ensure good contact, the bottle containing the solids and liquid was placed on a rotator, which kept the materials well mixed through a sloshing action.

An electronic balance was used to weigh the wet solids, the clean liquid solutions, the liquid-solids mixture, the spent liquid solutions, and the remaining wet solids for each contact cycle. These measurements were used to track the total masses of solids dissolved at each step of the process.

The mass of the available, centrifuged, wet Gunite tank W-6 sludge sample was 8.98 g. It had the consistency of a fine silt. The sludge was mixed with 25 mL of 0.01 M sodium hydroxide to aid transfer to a polypropylene centrifuge bottle and to wash the sludge solids. This mixture was centrifuged and the supernatant was decanted. The solids were then washed again with 20 mL of fresh 0.01 M sodium hydroxide solution. Again the mixture was centrifuged and the supernatant was decanted. About 8.71 g of wet solids was recovered. The wet solids were then separated into three portions: a portion for the leaching test, a portion for analysis, and a portion for archive. A 3.82-g portion of the washed sludge solids was leached with ~78 mL of ACT*DE*CONSM solvent (a ratio of ~20 mL of solvent per gram of wet solids) with a contact time of 2 h. The sample was then centrifuged and the solvent was decanted from the remaining solids. The leaching procedure was repeated twice using ~78 mL of solvent each time. The contact time for the second leach was 2 h, and the contact time for the third leach was ~16 h (overnight). Wet solids from the third leach were washed with mild caustic (pH of 10.0) in a ratio of ~10 mL per gram of wet solids to remove most of the ACT*DE*CONSM solvent and associated leachates from the interstitial liquid. Samples of the initial washed solids and the solids remaining after the final washing were submitted for analysis. The ACT*DE*CONSM solvent from the first leach and a combined solvent sample from the second and third ACT*DE*CONSM leach were filtered with Nalgene-nylon-membrane 0.45- μ m-porosity syringe filters prior to submission for analysis as a precaution to ensure that only substances in solution would be analyzed. Plugging of the filters indicated the presence of particulate matter or, possibly, colloidal material. (Beahm et al., 1995, have observed gel formation in sludge leachates.)

2.3 ANALYSIS OF SAMPLES

Density measurements on the aqueous samples were performed in the laboratory. Chemical and radiochemical analyses of samples were performed by the CASD. Analyses included free-acid concentration, cation concentrations, anion concentrations, and

radiochemical measurements. Solids were dissolved in an aqueous medium prior to analysis. The following is a brief summary of the analysis methods.

The water content of the solids samples was determined by the difference in weight between the centrifuged wet solids and the same solids after drying to a constant weight at 105°C.

Potentiometric titration with 0.1 M sodium hydroxide solution was used to measure hydrogen ion concentration. The endpoint of the titration was elucidated by use of a Metrohm pH meter.

Metal analysis was accomplished by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES). A Spectroflame-ICP Model FAO-05 analyzer simultaneously measured up to 22 metals at emission wavelengths between 210 and 800 nm. All samples were fed to the analyzer through a Spectro cross-flow nebulizer.

The common ions, including fluoride, chloride, bromide, nitrate, phosphate, and sulfate, were analyzed using a Dionex series 4500i ion chromatograph. This unit was configured with a pulsed electrochemical detector operating in the conductivity mode.

Gross alpha analysis was accomplished on a Tennelec LB4000 low-level alpha/beta counter. Aqueous samples were prepared by dilution with nitric acid. Samples, in a liquid form, were pipetted onto a stainless steel planchet and evaporated to dryness. The radioactive materials were fixed to the planchet by heating it over a Bunsen burner.

Following the gross alpha counting measurements, the planchets were analyzed using a Tennelec TC256 alpha spectrometer. Resolution of the energy peaks in the spectrograph was used to identify the alpha emitters and their relative abundance. Isotopes measured by this method included the actinides ^{239}Pu , ^{241}Am , and ^{244}Cm .

A method requiring heating of the sample could not be used for analysis of beta emitters because of the volatility of cesium. Gross beta analysis was performed using a Packard 2500 TR liquid scintillation counter. Gamma-emitting nuclides were counted with a high-purity germanium detector and a Canberra-Nuclear Data AccuSpecTM Genie-PC system, which identifies the nuclides based on their associated gamma-ray energies. Strontium was isolated by extraction chromatography, placed on a stainless steel planchet, and counted with a Tennelec LB4000 gas flow proportional counter.

Densities of liquid solutions were measured using volumetric flasks and an electronic balance. A 1-L flask was used to measure the density of the fresh ACT*DE*CONSM solution.

Spent ACT*DE*CONSM solutions containing radioactive leachates were placed in preweighed 10-mL volumetric flasks and reweighed to determine the density.

3. RESULTS AND DISCUSSION

3.1 MACROSCOPIC EFFECTS OF ACT*DE*CONSM LEACHING

As described in Sect. 2.2, washed GAAT W-6 sludge was used in the selective leaching tests. A portion of the washed sludge, ~3.82 g, was subjected to the ACT*DE*CONSM leaching procedure, and a portion was analyzed to establish the composition of the beginning material. The quantity of ACT*DE*CONSM solvent used for each of the three sequential leaches was based on the initial mass of the wet, washed sludge and a solvent-to-wet solids ratio of 20 mL/g. The test specimen was placed in a preweighed 250-mL centrifuge bottle, and the bottle was weighed on an electronic balance immediately following any addition or removal of material from the bottle. This permitted the mass of the remaining wet solids to be tracked at each point along the leaching procedure, as shown in Table 3.1.

The mass of fresh solvent added to the sludge is shown in Table 3.1, along with the volume calculated using the density of the fresh solvent. (Densities of fresh and spent solvent and solids content of the sludge are given in Tables 3.2 and 3.4, which will be discussed later.) As shown in Table 3.1, the mass of spent solvent was higher than the fresh solvent due to the dissolution of material from the sludge solids, and the mass of remaining wet solids decreased significantly with successive leaches. Weight gains in the solvent do not precisely correspond to the weight losses of the wet solids because the volume (and density) of interstitial liquid was not constant. The initial washed sludge sample contained ~2.53 mL of water and had a total mass of 3.82 g (i.e., 66.3% water), and the treated sludge sample contained 1.54 mL of water and had a total mass of 1.91 g (i.e., 80.5% water). On a wet basis, about half the sludge appeared to dissolve in the solvent. However, on a dry basis about 71% of the sludge solids was dissolved.

3.2 MEASURED COMPOSITIONS OF SOLIDS AND LEACHATES

Analyses of wet, centrifuged sludge solids are shown in Table 3.2. The table lists the species measured; the molecular weight; the concentration of each species in the original sludge, from the Bechtel National report (1995); an analysis of the sludge after washing with mild (0.01 M) caustic; and an analysis of the sludge residue after three consecutive

Table 3.1. Sludge weight losses for successive ACT*DE*CONSM treatments

Step	Fresh solvent		Spent solvent (g)	Solvent wt gain (g)	Remaining wet solids (g)	Remaining dry solids (g)	Color of spent solvent
	g	mL					
Start					3.82	1.287	
1st leach	85.30	78.8	86.27	0.97	2.85		Light yellow
2nd leach	85.27	78.8	85.70	0.43	2.42		Light yellow- brown
3rd leach	85.65	79.2	85.98	0.33	2.09		Light yellow- brown
pH 10 rinse	20.56		20.74	0.18	1.91	0.372	

Table 3.2. Assay of GAAT W-6 sludge solids on *wet* basis; as received from tank, after mild caustic wash and after selective leaching^a

	Molecular weight	Tank W-6 ^b	Washed solids	Residual solids
General				
Density, g/mL		1.19		
pH		11.14		
TOC, mg/L		2400.		
TIC, mg/L		3300.		
Total solids,		0.344	0.337	0.195
Radionuclides				
		(nCi/g)	(nCi/g)	(nCi/g)
⁶⁰ Co	60.	<15.1	20.8	17.8
⁹⁰ Sr/ ⁹⁰ Y	90.	5130.	9190.	219.
¹³⁷ Cs/ ^{137m} Ba	137.	2430.	2620.	2700.
¹⁵² Eu	152.	<10.	<16.8	<10.5
¹⁵⁴ Eu	154.	12.4	<11.1	<7.57
¹⁵⁵ Eu	155.	<1.6	<17.8	<13.2
²³⁸ Pu/ ²⁴¹ Am	238./241.	19.	10.8 ^c	5.41 ^c
^{239/240} Pu	239./240.	92.	116.	43.2
²⁴¹ Am	241.		<35.1	<26.8
²⁴⁴ Cm	244.	186.		
Gross alpha		297.	405.	151.
Gross beta		29700.	40500.	8380.
Total TRU		111.	126.8	48.6
Other metals				
		(mg/g)	(mg/g)	(mg/g)
Ag	107.87	0.0086	<0.0783	<0.00376
Al	26.98	10.9	12.2	23.2
B	10.81	0.00636		
Ba	137.33	0.210	0.255	0.0328
Be	9.01	<0.0001	<0.00142	<0.00144
Ca	40.08	31.6	38.5	0.0907
Cd	112.41	0.00495	0.00422	<0.00216
Co	58.93	0.00489	0.00500	0.00267
Cr	52.00	1.39	1.80	0.966
Cu	63.55	0.0312	0.0582	0.0267
Fe	55.85	14.7	19.7	14.2
Hg	200.59	0.0402		
K	39.10	0.595	0.277	0.208
Mg	24.31	0.746	1.19	1.25
Mn	54.94	1.51	2.10	0.454
Na	22.99	43.9	24.8	24.3
Ni	58.70	0.102	0.128	0.132
P	30.97		18.4	4.43
Pb	207.2	1.01		
Si	28.09	3.36	3.75	1.61
Sr	87.62	0.0545		
Th	232.04	0.679	1.32	0.666
Tl	204.37	0.0361	<0.0233	<0.0236
U	238.03	8.86	12.5	0.502
Zn	65.38	0.0899	0.114	0.121
Anions				
		(mg/g)	(mg/g)	(mg/g)
Br ⁻	79.90	<0.064	<0.00902	<0.00898
Cl ⁻	35.45	<0.064	0.0207	0.0155
F ⁻	19.00	11.9	5.40	2.85
CO ₃ ²⁻	60.00	13.9 ^d		
NO ₃ ⁻	62.00	12.3	0.698	0.341
PO ₄ ³⁻	94.97	7.90	0.783	0.0398
SO ₄ ²⁻	96.06	9.40	0.575	0.109

^aNo entry implies that no measurement was made for that species. As, Sb, Se, and V were below the detectable limit.

^bData taken from ORNL/ER/Sub/87-99053/74.

^c²³⁸Pu only.

^dEstimated from TIC.

ACT*DE*CONSM leaches followed by a rinse with very mild caustic (pH of 10). Washing the sludge removed the solute mass associated with interstitially trapped liquid, thereby increasing the mass concentration of insoluble species, such as the TRU components. The most meaningful data are in the last two columns of Table 3.2; these data show how selective leaching affected the sludge on a wet basis. The solids fraction shown in the table readily illustrates a variable water content that obscures the interpretation of the concentration data. The concentration per unit mass of any given species is highly dependent on water content, and the concentration can be reduced to as low a value as desired by simply adding water, which is not a valid treatment method. Comparison of the concentrations on a dry basis poignantly reveals the effects of the treatment process.

The concentration data in Table 3.2 have been converted to a dry basis using the measured total solids content. Concentrations of species in dry sludge are given in Table 3.3. Now the caustic washed sludge solids may be compared with the solids treated with selective leaching on an equal basis. Comparing the assay of the dry, washed sludge solids with the treated, dry residual solids (Table 3.3) indicates that the selective leaching procedure increased the concentrations of some species and reduced the concentrations of other species. For example, cobalt-60, cesium-137, aluminum, iron, and nickel, among others, increased in concentration, indicating that these species did not dissolve in the ACT*DE*CONSM solvent as well as the bulk average of the solids. Materials, including strontium-90, plutonium, calcium, and uranium decreased in concentration, indicating that those species were more soluble in the ACT*DE*CONSM solvent than the bulk average of the solids.

The TRU-waste content of the GAAT W-6 sludge is essentially all plutonium, as the americium is below measurable concentrations (Table 3.3). The plutonium concentration in the solids decreased during the selective leaching procedure, but not enough to render the treated solids a non-TRU waste.

Samples of the ACT*DE*CONSM leachates were collected and analyzed for the constituents found in the sludge. The results are summarized in Table 3.4, along with calculated concentrations of sodium, carbonate, and organic carbon in the fresh solvent. These data indicate that the first leach step removed a larger amount of the soluble materials than either of the two subsequent leaches, which is in agreement with the total weight losses reported in Table 3.1. The data on the spent solvent must be combined with the analysis of the starting sludge solids (in this case, washed sludge) to calculate the fraction of each species removed from the sludge.

Table 3.3. Assay of GAAT W-6 sludge solids on *dry* basis; as received from tank, after mild caustic wash and after selective leaching^a

	Molecular weight	Tank W-6 ^b	Washed solids	Residual solids
General				
Density, g/mL		1.19		
pH		11.14		
TOC, mg/L		2400.		
TIC, mg/L		3300.		
Total solids g/g		1.0	1.0	1.0
Radionuclides				
		(nCi/g)	(nCi/g)	(nCi/g)
⁶⁰ Co	60.	<43.9	61.7	91.3
⁹⁰ Sr/ ⁹⁰ Y	90.	14900.	27300.	1120.
¹³⁷ Cs/ ^{137m} Ba	137.	7060.	7770.	13800.
¹⁵² Eu	152.	<29.1	<49.9	<53.8
¹⁵⁴ Eu	154.	36.0	<32.9	<38.8
¹⁵⁵ Eu	155.	<4.65	<52.8	<67.7
²³⁸ Pu/ ²⁴¹ Am	238./241.	55.2	32.0 ^c	27.7 ^c
^{239/240} Pu	239./240.	267.	344.	222.
²⁴¹ Am	241.		<104.	<137.
²⁴⁴ Cm	244.	541.		
Gross alpha		863.	1200.	774.
Gross beta		86300.	120000.	43000.
Total TRU		323.	376.	250.
Other metals				
		(mg/g)	(mg/g)	(mg/g)
Ag	107.87	0.025	<0.232	<0.0193
Al	26.98	31.7	36.2	119.
B	10.81	0.0185		
Ba	137.33	0.610	0.668	0.168
Be	9.01	<0.0003	<0.00421	<0.00738
Ca	40.08	91.9	114.	0.465
Cd	112.41	0.0144	0.0125	<0.0111
Co	58.93	0.0142	0.0148	0.0137
Cr	52.00	4.04	5.34	4.95
Cu	63.55	0.0907	0.173	0.137
Fe	55.85	42.7	58.5	72.8
Hg	200.59	0.117		
K	39.10	1.73	0.822	1.07
Mg	24.31	2.17	3.53	6.41
Mn	54.94	4.39	6.23	2.33
Na	22.99	128.	73.6	125.
Ni	58.70	0.297	0.380	0.677
P	30.97		54.6	22.7
Pb	207.2	2.94		
Si	28.09	9.77	11.1	8.26
Sr	87.62	0.158		
Th	232.04	1.97	3.92	3.42
Tl	204.37	0.105	<0.0691	<0.121
U	238.03	25.8	37.1	2.57
Zn	65.38	0.261	0.338	0.621
Anions				
		(mg/g)	(mg/g)	(mg/g)
Br ⁻	79.90	<0.186	<0.0268	<0.0461
Cl ⁻	35.45	<0.186	0.0614	0.0795
F ⁻	19.00	34.6	16.0	14.6
CO ₃ ²⁻	60.00	40.4 ^d		
NO ₃ ⁻	62.00	35.8	2.07	1.75
PO ₄ ³⁻	94.97	23.0	2.32	0.204
SO ₄ ²⁻	96.06	27.3	1.71	0.559

^aNo entry implies that no measurement was made for that species. As, Sb, Se, and V were below the detectable limit.

^bCalculated from data taken from ORNL/ER/Sub/87-99053/74.

^c²³⁸Pu only.

^dEstimated from TIC.

Table 3.4. Analysis of ACT*DE*CONSM leachates^a

	Molecular weight	ACT*DE*CON SM solvent	First leachate	Mixed 2nd & 3rd leachate
General				
Density, g/mL		1.082	1.089	1.091
pH		8.98	9.26	9.22
TOC, mg/L		12100. ^b		
TIC, mg/L				
Total solids, g/g				
Radionuclides			(nCi/L)	(nCi/L)
⁶⁰ Co	60.		200.	100.
⁹⁰ Sr/ ⁹⁰ Y	90.		459000.	170000.
¹³⁷ Cs/ ^{137m} Ba	137.		43200.	11400.
¹⁵² Eu	152.		432.	<91.9
¹⁵⁴ Eu	154.		459.	<81.1
¹⁵⁵ Eu	155.		<324.	<159.
²³⁸ Pu/ ²⁴¹ Am	238./241.		189. ^c	81.1 ^c
^{239/240} Pu	239./240.		1570.	946.
²⁴¹ Am	241.		<622.	<324.
²⁴⁴ Cm	244.			
Gross alpha			9190.	2650.
Gross beta			1000000.	405000.
Total TRU			1759.	1027.
Other metals		(mg/L)	(mg/L)	(mg/L)
Ag	107.87		<0.418	<0.418
Al	26.98		27.9	39.5
B	10.81			
Ba	137.33		7.65	1.99
Be	9.01		<0.160	<0.160
Ca	40.08		1350.	288.
Cd	112.41		<0.240	<0.240
Co	58.93		<0.266	<0.266
Cr	52.00		13.3	16.7
Cu	63.55		1.95	0.715
Fe	55.85		189.	152.
Hg	200.59			
K	39.10		19.3	16.2
Mg	24.31		9.48	6.77
Mn	54.94		37.0	19.6
Na	22.99	35380. ^b	33900.	35200.
Ni	58.70		0.572	0.671
P	30.97		478.	141.
Pb	207.2			
Si	28.09		32.2	22.9
Sr	87.62			
Th	232.04		23.2	12.1
Tl	204.37		<2.62	<2.62
U	238.03		343.	112.
Zn	65.38		1.41	<0.388
Anions		(mg/L)	(mg/L)	(mg/L)
Br ⁻	79.90		<5.00	<5.00
Cl ⁻	35.45		8.65	<5.00
F ⁻	19.00		263.	179.
CO ₃ ²⁻	60.00	60000. ^b		
NO ₃ ⁻	62.00		317.	<100.
PO ₄ ³⁻	94.97		1535.	425.
SO ₄ ²⁻	96.06		40.6	15.7

^aNo entry implies that no measurement was made for that species. As, Sb, Se, and V were below the detectable limit.

^bEstimated from solvent recipe.

^c²³⁸Pu only.

3.3 FRACTION OF EACH SPECIES REMOVED FROM THE SLUDGE

The data provided in Tables 3.3 and 3.4 permit two different calculations of the fraction of each species removed from the sludge by the selective leaching. Data on the initial washed sludge and the residual sludge following leaching given in Table 3.3 may be used to calculate the percentage of each species removed by

$$\% i \text{ removed} = \frac{(m_{dry,0})(C_{i,0}) - (m_{dry,residual})(C_{i,residual})}{(m_{dry,0})(C_{i,0})} \times 100, \quad (3.1)$$

where

$m_{dry,0}$ = dry mass of initial sludge, g;

$m_{dry,residual}$ = dry mass of residual sludge, g;

$C_{i,0}$ = concentration of species i in initial sludge, g/g; and

$C_{i,residual}$ = concentration of species i in residual sludge, g/g.

Results of the calculation are given in the last column of Table 3.5.

As an alternative, the amount of each species removed from the sludge by the ACT*DE*CONSM solvent may be calculated from the volumes of the solvent and the measured concentrations of the spent solvent. Because the solids and liquids were separated by centrifugation, a measurable amount of liquid remains interstitially with the solids. The quantities of interstitial liquid for the untreated solids and the remaining solids were provided at the end of Sect. 3.1. Because all the liquid cannot be recovered due to interstitial amounts of liquid, it is necessary to assume that the volume of the solvent does not change as the sludge components are leached. This is probably a good assumption since a large liquid-to-solids ratio was used. In the first leaching contact, 78.8 mL of the solvent was mixed with a sludge, which contained about 2.53 mL of water. Based on the above assumptions, the total volume of the resulting solution would be 81.33 mL and the mass of solute in the solution should be based on this volume. The solvent itself introduces significant quantities of various components, such as sodium, which must be deducted from the amount in the spent solvent. For the first leach, the amount of each species removed is given by

$$R_i = (V_a + V_p)(c_i) - (V_a)(c_{i,0}), \quad (3.2)$$

where

R_i = amount of species i removed, g;

V_a = volume of solvent added, L;

Table 3.5. Percentage of washed GAAT W-6 sludge solids removed by selective leaching (dry basis)^a

	By first leachate	By all leachates	Based on residual solids
General			
Total solids			71.1
Radionuclides			
⁶⁰ Co	20.5	40.1	57.3
⁹⁰ Sr/ ⁹⁰ Y	106.	181.	98.8
¹³⁷ Cs/ ^{137m} Ba	35.1	52.5	48.5
¹⁵² Eu	<i>b</i>	<i>b</i>	<i>b</i>
¹⁵⁴ Eu	<i>b</i>	<i>b</i>	<i>b</i>
¹⁵⁵ Eu	<i>b</i>	<i>b</i>	<i>b</i>
²³⁸ Pu	37.7	67.8	75.0
^{239/240} Pu	28.8	62.3	81.4
²⁴¹ Am	<i>b</i>	<i>b</i>	<i>b</i>
²⁴⁴ Cm			
Gross alpha	48.3	74.5	81.4
Gross beta	52.6	93.2	89.7
Total TRU	29.5	62.7	80.9
Other metals			
Ag	<i>b</i>	<i>b</i>	<i>b</i>
Al	4.87	18.3	5.01
B			
Ba	72.4	108.	92.7
Be	<i>b</i>	<i>b</i>	<i>b</i>
Ca	74.6	104.	99.9
Cd	<121. ^c	<356.	>74.4 ^d
Co	<113.	<333.	73.3
Cr	15.7	54.2	73.2
Cu	71.3	121.	77.1
Fe	20.4	52.2	64.0
Hg			
K	148.	390.	62.5
Mg	17.0	40.4	47.5
Mn	37.5	75.7	89.2
Na	-33.7 ^e	-60.9	51.1
Ni	9.51	31.2	48.5
P	55.3	86.0	88.0
Pb			
Si	18.3	43.4	78.6
Sr			
Th	37.4	74.9	74.8
Tl	<i>b</i>	<i>b</i>	<i>b</i>
U	58.4	94.5	98.0
Zn	26.3	<39.9	47.0
Anions			
Br ⁻	<i>b</i>	<i>b</i>	<i>b</i>
Cl ⁻	890.	>1000.	62.6
F ⁻	104.	240.	73.6
CO ₃ ²⁻			
NO ₃ ⁻	967.	>1000.	75.6
PO ₄ ³⁻	>1000.	>1000.	97.5
SO ₄ ²⁻	150.	261.	90.5

^aPercentages are calculated to three digits, more precision than is justified by the data. No entry implies that no measurement was made for that species.

^bConcentration of species in initial solids was below the detectable limit; percentage removed cannot be calculated.

^cThe less than symbol indicates the percentage removed is less than the given value when the species was measured in the initial solids but was below the detectable limit in the leachates.

^dThe greater than symbol indicates the percentage removed is greater than the given value when the species was measured in the initial solids but was below the detectable limit in the residual solids or when the leachate was measured to contain more of a given species than was in the initial solids.

^eNegative values occur when the required result is the difference of large numbers and when these large numbers contain errors on the order of the difference.

V_p = interstitial (or pore) volume of solution contributed by wet solids, L;

c_i = concentration of species i in spent solvent, g/L; and

$c_{i,0}$ = concentration of species i in fresh solvent, g/L.

The percentage removed can be found by substituting R_i for the numerator in Eq. (3.1). The amounts and percentages removed by all three leaches may be calculated by extending these equations. The algebraic expression is

$$A_i = R_i + (V_{a2} + V_{a3} + V_{p2})(c_{i,c}) - (V_{a2} + V_{a3})(c_{i,0}) - (V_{p2})(c_i) , \quad (3.3)$$

where

A_i = amount of species i removed by all three leaches, g;

V_{a2} = volume of solvent added in second treatment, L;

V_{a3} = volume of solvent added in third treatment, L;

V_{p2} = interstitial volume of leachate left from first treatment, L;

$c_{i,c}$ = concentration of i in combined second and third spent solvent sample, g/L; and

c_i = concentration of i in spent solvent from first treatment, g/L.

The cumulative percentage of each species removed by all three treatments is found by substituting A_i for the numerator in Eq. (3.1).

It is appropriate to make two qualifying remarks about Eq. (3.3). First, analysis of the sample containing equal portions of the second and third leachate is only useful if equal volumes of fresh solvent are used for the second and third treatments. Experimentally this condition is closely approximated. Second, the interstitial volume of spent solvent was not measured following the first treatment (due to the planned sequence of execution of the test). However, it was estimated to be 2.0 mL based on the assumption that the interstitial volume varied linearly with the mass of wet solids and using the known values for the initial washed sludge and the residual sludge.

Results of the calculations regarding the leachates are listed in the second and third columns of Table 3.5. For convenience, the constituents of the sludge are placed in two groups in Table 3.6; one group represents species that were less soluble than the average bulk solids, and the other represents more soluble species. Calculations of the percentage of each species removed, based on either the cumulative leachate analyses or the residual solids, generally show fair agreement on the important sludge components. For instance, the amount of plutonium removed ranges from ~60 to ~80%. Uranium and calcium are largely removed, while aluminum is barely affected. Thorium, chromium, and silicon are leached to about the

Table 3.6. Percentage of washed GAAT W-6 sludge solids removed by selective leaching (dry basis) compared with total solids leached^a

	By first leachate	By all leachates	Based on residual solids
Total solids leached			71.1
Species less soluble than average of the bulk			
⁶⁰ Co	20.5	40.1	57.3
¹³⁷ Cs/ ^{137m} Ba	35.1	52.5	48.5
Al	4.87	18.3	5.01
Fe	20.4	52.2	64.0
K	148.	390.	62.5
Mg	17.0	40.4	47.5
Na	-33.7 ^b	-60.9	51.1
Ni	9.51	31.2	48.5
Zn	26.3	<39.9 ^c	47.0
Cl ⁻	890.	>1000. ^d	62.6
Species more soluble than average of the bulk			
⁹⁰ Sr/ ⁹⁰ Y	106.	181.	98.8
²³⁸ Pu	37.7	67.8	75.0
^{239/240} Pu	28.8	62.3	81.4
Gross alpha	48.3	74.5	81.4
Gross beta	52.6	93.2	89.7
Total TRU	29.5	62.7	80.9
Ba	72.4	108.	92.7
Ca	74.6	104.	99.9
Cd	<121.	<356.	>74.4
Co	<113.	<333.	73.3
Cr	15.7	54.2	73.2
Cu	71.3	121.	77.1
Mn	37.5	75.7	89.2
P	55.3	86.0	88.0
Si	18.3	43.4	78.6
Th	37.4	74.9	74.8
U	58.4	94.5	98.0
F ⁻	104.	240.	73.6
NO ₃ ⁻	967.	>1000.	75.6
PO ₄ ³⁻	>1000.	>1000.	97.5
SO ₄ ²⁻	150.	261.	90.5

^aBased on the analysis of solids residue.

^bNegative values occur when the required result is the difference of large numbers and when these large numbers contain errors on the order of the difference.

^cThe less than symbol indicates the percentage removed is less than the given value when the species was measured in the initial solids but was below the detectable limit in the leachates.

^dThe greater than symbol indicates the percentage removed is greater than the given value when the species was measured in the initial solids but was below the detectable limit in the residual solids or when the leachate was measured to contain more of a given species than was in the initial solids.

same extent as the bulk average of the solids. The data in this form support the remarks made in Sect. 3.2 concerning the dissolution of specific species in relation to the dissolution of the bulk sludge solids. Dissolution of ~71% of the bulk solids and ~80% of the plutonium indicates a poor separation factor, particularly in view of the large liquid-to-solids ratio used.

4. CONCLUSIONS AND RECOMMENDATIONS

Mixed waste sludge from Gunite tank W-6 was subjected to the ACT*DE*CONSM selective leaching process. Nearly all of the TRU-waste content of this sludge was attributed to plutonium. The sludge sample was first washed with a mild caustic solution (~0.01 M NaOH) to remove excess sodium and nitrate associated with the interstitial liquid supernatant. The washed, wet solids were then treated with the ACT*DE*CONSM solvent using a ratio of ~20 mL of solvent per gram of wet solids. The sludge and solvent were separated by centrifugation, and the ACT*DE*CONSM treatment was repeated twice. On a wet basis the overall solvent-to-sludge ratio was ~60 mL/g, but on a dry basis the ratio was ~178 mL/g.

Samples of the spent solvents (leachates) and residual sludge solids were analyzed. The results indicated that ~71% of the solids in the sludge were dissolved, while ~80% of the TRU-waste components dissolved. A low separation of the TRU-waste components from other components of the sludge mixture is indicated. Almost all of the uranium and calcium were removed from the sludge. On a dry solids basis, the total TRU-waste content of the washed sludge was 376 nCi/g and that of the treated sludge was 250 nCi/g. The process did not render the sludge a non-TRU waste.

For this sludge, wherein most of the TRU-waste content is plutonium, the ACT*DE*CONSM process, using the specified formulation, was not effective in rendering the sludge a non-TRU waste. It is recommended that ACT*DE*CONSM be optimized for this specific application and that other formulations or processes utilizing different chelating and oxidizing agents be tested. Additionally, the ACT*DE*CONSM process should be tested on TRU mixed waste in which the bulk of the TRU components are elements other than plutonium.

5. REFERENCES

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APPENDIX: TEST PROCEDURE FOR SELECTIVE LEACHING OF TRANSURANICS FROM GUNITE TANK SLUDGE

A procedure for the selective leaching test was prepared to serve as a guide in the laboratory. Four modifications were made as the test was being performed: (1) the washed, centrifuged sludge sample was separated into three portions using a spatula rather than suspending the solids as a slurry in caustic and pipetting the slurry; (2) two sample numbers were changed; (3) the third leach, rather than the second, was permitted to mix overnight because more steps of the procedure were done the first day than was anticipated; and (4) larger samples were submitted for analysis than originally planned. Otherwise, the test procedure was followed exactly. The test procedure given below has been modified to reflect the steps that were actually performed in the laboratory. (Note that parts of Sect. 3 and all of Sect. 4 were performed by the Chemical and Analytical Services Division.)

1.0 Preparation of Sludge Sample

- 1.1 The sludge sample from Tank W-6 will be used in this test.
- 1.2 Transfer the sludge sample into a preweighed centrifuge bottle labeled GW6-0. (If necessary, use supernatant from centrifuging to rinse all the solid particles from the sludge transport container into the centrifuge bottle.) Weigh the sample and bottle.
- 1.3 Centrifuge the sludge at 4500 rpm (equivalent to $4140 \times g$) for 20 min.
- 1.4 Decant the supernatant into a preweighed bottle labeled GW6-0-L. Weigh the bottle containing the supernatant and the centrifuge bottle containing the sludge. Save the supernatant solution.
- 1.5 Add 20 mL of 0.01 M NaOH to the sludge (GW6-0) and mix thoroughly for 3 min on the vortex mixer. (This step will wash some of the soluble cesium associated with the interstitial liquid from the sludge.) Weigh centrifuge bottle GW6-0 and its contents.
- 1.6 Centrifuge the sludge at 4500 rpm for 20 min.
- 1.7 Decant the wash solution into a preweighed bottle labeled GW6-0-W and save it. Weigh the centrifuge bottle (GW6-0) containing the sludge and the bottle (GW6-0-W) containing the decanted wash solution.

- 1.8 Add 20 ml of 0.01 M NaOH to the sludge (GW6-0) and mix thoroughly for 3 min on the vortex mixer. (This is a second washing step.) Weigh centrifuge bottle GW6-0 and its contents.
- 1.9 Centrifuge the sludge at 4500 rpm for 20 min.
- 1.10 Decant the wash solution into GW6-0-W. Weigh the centrifuge bottle (GW6-0) containing the sludge and the bottle (GW6-0-W) containing the decanted wash solution.
- 1.11 Using a spatula, transfer sludge samples into bottles as follows: (a) Transfer $\sim\frac{1}{4}$ of the sludge to a preweighed centrifuge bottle labeled GW6-A1-S. (b) Transfer $\sim\frac{1}{4}$ of the sludge to a preweighed centrifuge bottle labeled GW6-A1. (c) Leave the remaining sludge, $\sim\frac{1}{2}$ the initial quantity, in the original centrifuge bottle which should be labeled GW6-0. Weigh all three bottles.
- 1.12 Centrifuge GW6-0, GW6-A1, and GW6-A1-S at 4500 rpm for 20 min.
- 1.13 Decant the liquid from each of the three bottles (GW6-0, GW6-A1, and GW6-A1-S), transferring the liquid to bottle GW6-0-W. Weigh all four bottles.

2.0 ACT*DE*CONSM Washing Procedure

- 2.1 Prepare 1 L of stock ACT*DE*CONSM solution according to the procedure provided by Selentec.
- 2.2 To bottle GW6-0 add ACT*DE*CONSM solution in the ratio of 20 mL for each gram of wet sludge contained in the bottle. Add 0.45 mL of 3% hydrogen peroxide for each 20 mL of ACT*DE*CONSM solution added. Mix thoroughly for 2 h. Weigh bottle GW6-0.
- 2.3 Centrifuge bottle GW6-0 at 4500 rpm for 20 min.
- 2.4 Decant the supernatant from GW6-0 into a preweighed sample bottle labeled GW6-A1-1. Weigh the bottle containing the remaining sludge (GW6-0) and the bottle containing the first ACT*DE*CONSM wash (GW6-A1-1).
- 2.5 To centrifuge bottle GW6-0 add the same quantities of ACT*DE*CONSM solution and 3% hydrogen peroxide, as was done in step 2.2. Weigh the bottle. Mix thoroughly for 2 h.
- 2.6 Centrifuge bottle GW6-0 at 4500 rpm for 20 min.

- 2.7 Decant the supernatant from GW6-0 into a preweighed sample bottle labeled GW6-A1-2. Weigh the bottle containing the remaining sludge (GW6-0) and the bottle containing the second ACT*DE*CONSM wash (GW6-A1-2).
- 2.8 To centrifuge bottle GW6-0 add the same quantities of ACT*DE*CONSM solution and 3% hydrogen peroxide, as was done in step 2.2. Weigh the bottle. Mix thoroughly overnight.
- 2.9 Centrifuge bottle GW6-0 at 4500 rpm for 20 min.
- 2.10 Decant the supernatant from GW6-0 into a preweighed sample bottle labeled GW6-A1-3. Weigh the bottle containing the remaining sludge (GW6-0) and the bottle containing the third ACT*DE*CONSM wash (GW6-A1-3).
- 2.11 To centrifuge bottle GW6-0, add 20 mL of dilute sodium hydroxide solution having a pH of 10. Weigh the bottle. Mix thoroughly for 2 min on the vortex mixer.
- 2.12 Centrifuge bottle GW6-0 at 4500 rpm for 20 min.
- 2.13 Decant the supernatant from GW6-0 into a preweighed sample bottle labeled GW6-A1-R. Weigh both bottles GW6-0 and GW6-A1-R.

3.0 Analysis of Samples

- 3.1 Dry both the washed sludge solids (sample GW6-A1) and the remaining "treated" sludge solids (sample GW6-0) at 105°C until a constant weight is obtained. Record the beginning and ending weights.
- 3.2 Analyze the dried sludge solids in samples GW6-A1 and GW6-0 (reported in Bq/g or µg/g, as appropriate) for the constituents given in the analyte list in Sect. 4.0.
- 3.3 Analyze the supernatant from the first ACT*DE*CONSM wash, sample GW6-A1-1, for the constituents given in the analyte list in Sect. 4.0. (Results should be reported in Bq/mL or mg/L, as appropriate.)
- 3.4 Prepare a sample labeled GW6-A1-C by combining 15 mL of the second ACT*DE*CONSM wash solution (GW6-A1-2) with 15 mL of the third ACT*DE*CONSM wash solution (GW6-A1-3). Analyze this sample for the constituents given in the analyte list in Sect. 4.0 below. (Results should be reported in Bq/mL or mg/L, as appropriate.)
- 3.5 All remaining samples or portions thereof should be saved as archive.

4.0 Analyte List

General

Percent moisture in solids samples

pH of liquid samples

Density of liquid samples

Radionuclides

^{90}Sr

^{137}Cs

^{152}Eu

^{154}Eu

^{155}Eu

$^{238}\text{Pu}/^{241}\text{Am}$

$^{239}/^{240}\text{Pu}$

^{244}Cm

Gross alpha

Gross beta

Other Metals

Al

Ca

Cr

Fe

Mg

Na

P

Si

Th

U

Anions

F⁻

NO₃⁻

PO₄⁻³

SO₄⁻²

CO₃⁻²

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