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Surrogate Formulations for Thermal Treatment of Low-Level Mixed Waste, Part IV: Wastewater Treatment Sludges

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ABBREVIATIONS AND ACRONYMS

ASTM	American Society for Testing Materials
DE	diatomaceous earth
DOE	U.S. Department of Energy
DOE	Department of Energy
EDS	energy dispersive X-ray spectroscopy
EPA	U.S. Environmental Protection Agency
FFCA	Federal Facility Compliance Act
INEL	Idaho National Engineering Laboratory
LANL	Los Alamos National Laboratory
LDRs	Land Disposal Restrictions
MWIP	Mixed Waste Integrated Program
RCRA	Resource Conservation and Recovery Act
RFP	Rocky Flats Plant
SSQ-EDS	standardless semi-quantitative energy-dispersive X-Ray Spectroscopic
TCLP	Toxicity Characteristic Leaching Procedure
TDS	total dissolved solids
TRU	transuranic
WETF	West End Treatment Facility
XRD	X-ray diffraction

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EXECUTIVE SUMMARY

The category of sludges, filter cakes, and other waste processing residuals represent the largest volume of low-level mixed (hazardous and radioactive) wastes within the U.S. Department of Energy (DOE) complex. Treatment of these wastes to minimize the mobility of contaminants (and thus the risk to personnel and the environment), and to eliminate the presence of free water, is required under the Federal Facility Compliance Act agreements between DOE and the Environmental Protection Agency.

In the text, we summarize the currently available data for several of the high priority mixed-waste sludge inventories within DOE. Los Alamos National Laboratory TA-50 Sludge and Rocky Flats Plant By-Pass Sludge are transuranic (TRU)-contaminated sludges that were isolated with the use of silica-based filter aids; the presence of these filter aids will be beneficial to the treatment of these materials by vitrification (glass formation). The Oak Ridge Y-12 Plant West End Treatment Facility Sludge is predominantly calcium carbonate and biomass; no filter aid is used to isolate this material. The Oak Ridge K-25 Site Pond Waste is a large-volume waste stream, containing clay, silt, and other debris in addition to precipitated metal hydroxides.

We formulate "simulants" for the waste streams described above, using cerium oxide as a surrogate for the uranium or plutonium present in the authentic material. Use of nonradiological surrogates greatly simplifies material handling requirements for initial treatability studies. The use of synthetic mixtures for initial treatability testing will facilitate compositional variation for use in conjunction with statistical design experiments; this approach may help to identify any "operating window" limitations (for the sludge composition, processing additives, or process conditions). The initial treatability testing demonstrations utilizing these "simulants" will be based upon vitrification, although the materials are also amenable to testing grout-based and other stabilization procedures. After the feasibility of treatment and the initial evaluation of treatment performance has been demonstrated, performance must be verified using authentic samples of the candidate waste stream.

1. INTRODUCTION

Most U.S. Department of Energy (DOE) industrial sites operate process wastewater treatment plants that produce large volumes of hazardous or mixed waste sludges. In a recent overview of DOE mixed waste inventories,[1] the subcategory of "sludges, filter cakes, and residues," primarily generated from wastewater treatment, represents the highest volume waste [approximately 12,000 m^3 current inventory (17% of total mixed waste volume), with a generation rate of about 685 m^3/year; these figures exclude about 28,000 m^3 of the partially cemented and raw Pond Sludge from the Oak Ridge K-25 Site awaiting remediation]. These raw sludges do not meet the U.S. Environmental Protection Agency (EPA) requirements for treatment, storage, and disposal; the Land Disposal Restrictions (LDRs)[2] prohibit land disposal and storage of untreated hazardous waste. Until May 1992, a temporary compliance had been granted for mixed waste, based upon insufficient treatment capacity. Under the requirements of the Federal Facility Compliance Act (FFCA) of 1992 (Public Law 102-386), all federal facilities must waive sovereign immunity and come into compliance with applicable federal and state laws and regulations. The FFCA also requires submission of site-specific mixed waste treatment plans to the appropriate regulatory agency, including schedules for developing treatment capability and capacity. This will require installation of new mixed waste processing capacity to meet these regulations, which also specify treatment performance standards for the elimination of hazardous organic constituents and stabilization of hazardous inorganic constituents.

1.1 RATIONALE

The purpose of this document is to present data on some specific examples of high-priority wastewater treatment sludges, and to formulate simulants to allow initial treatability investigations. Preparation of simulants can proceed in several stages, depending upon the maturity of the candidate treatment process and the regulatory permits held by the testing facility. Initially, the investigator may wish to prepare a nonhazardous, nonradioactive simulant, containing only the nonhazardous bulk constituents (i.e., major components, such filter aids and coprecipitating agents such as hydrolyzed salts of Fe, Al, etc.); this may allow facile testing of select physical properties of both the raw waste and the treated product to determine the applicability of the proposed treatment technology. In a second tier of testing, appropriate levels of hazardous constituents, and perhaps radiologic constituents or their surrogates, can be added to the bulk constituents for further performance assessment [including regulatory assessments such as the EPA Toxicity Characteristic Leaching Procedure (TCLP)]. The final qualification challenge, for technologies that have performed satisfactorily on waste simulants, is a graded series of demonstration testing (from bench-scale to production-scale, if justified) using the authentic waste stream.

The initial application of simulated wastewater treatment sludges will be for testing of ex situ vitrification treatment technology on this waste-stream class; this initial work will be performed collaboratively by the Savannah River Technology Center and Clemson University. Under guidance from Mixed Waste Integrated Program (MWIP) Technical Specification Area leaders, three waste streams were selected for initial simulant formulation: (1) Oak Ridge Y-12 Plant West End Treatment Facility (WETF) Sludge (a well-characterized material, containing biological treatment residues and trace oil and grease; no glass-forming filter aid is used to isolate this material); (2) Rocky Flats Plant (RFP) Bypass Sludge (a simulant has already been formulated for microwave drying/sintering technology demonstrations; this material contains diatomaceous earth filter aid); and (3) Los Alamos National Laboratory (LANL) Bypass Sludge (a less well-characterized iron coprecipitation sludge, containing perlite filter aid, a vitreous mineral product). An additional simulant is presented, representing the Oak Ridge K-25 Site Pond Waste (a high-volume mixed waste with significant compliance "drivers").

1.2 SURROGATES FOR RADIOLOGICAL COMPONENTS

The use of actual radioactive material imposes considerable restrictions on material handling, transportation, and performance testing. In addition to obtaining the requisite Nuclear Regulatory Commission licenses to handle the appropriate radioisotopes, there are additional stringent requirements for radiological work, including personnel protective clothing, criticality safety review, emissions monitoring and reporting, etc. There is also a major investment in equipment that may require extensive decontamination after use. For these and other considerations, it may be desirable to use simulated waste formulations that use surrogate materials to represent the expected behavior of the radiological constituents. In selecting a metal surrogate, the object is to mimic as closely as possible, but in a conservative manner, the behavior of the actual metal as incorporated into both the raw waste and the anticipated treatment residues. A companion document[3] reviews the selection of surrogate materials for radiological components for testing in thermal treatment systems. Recommendations are summarized in Table 1; some of the recommendations for nonthermal systems are taken from Bjorklund's *Development and Use of Sintered High-Level Waste.*[4] For example, the lanthanide series of elements are formed by successive filling of the inner 4f electron orbitals, whereas the actinides are formed by filling the 5f orbitals; thus Ce compounds (rare earth lanthanide series) serve as reasonably good chemical and thermal surrogates for both U (actinide series) and Pu (TRU series). However, there are always limitations to the use of surrogates; as Shapiro and coworkers[5] report on the selection of Ce as a surrogate for Pu:

- Ce exhibits a similar chemistry because much of its aqueous solution chemistry is similar to that of Pu;
- Ce exhibits a different oxidation state than Pu so that it is not directly analogous;
- Ce has the potential to form the strongly oxidizing ceric ion, which can enhance corrosion; but Shapiro concludes that
- although some of cerium's atomic characteristics do not perfectly match those of Pu, they are closer than other available surrogates.

Similarly Bates[6] has discussed possible surrogates for U, Pu, and Am, concluding that

> While there are no perfect surrogates for these radionuclides, cerium is generally the most conservative (best simulates their behavior). Other less conservative surrogates of plutonium, in degree of similarity are neodymium > lanthanum > praseodymium > gadolinium > ytterbium > dysprosium > erbium > europium > terbium > lutetium > samarium.

From the arguments presented above, it would appear that Ce compounds represent conservative surrogates for both U and Pu (principle alpha-emitting radionuclides in the wastewater treatment sludges discussed in this report). Bates[6] has recommended the use of approximately 0.5 wt% Ce_2O_3 to ensure an adequate level of surrogate for easy detectability in the waste form or residues. A companion document[3] has shown that the thermal behavior of

Table 1. Suggested surrogates for selected radioisotopes (from Ref. 3)

Radioisotope	Suggested surrogate(s)	
	Thermal system	Nonthermal system
U-238/U-235	Ce, RE[a]	Ce, RE, Zr, Mo
Pu-239/TRU[b]	Ce, RE	Ce, RE, Zr
Tc-99, Ru-103	Ru	Ru, Re, Mo, Mn
Cs-137	Natural Cs	Natural Cs
Sr-90	Natural Sr	Natural Sr

[a] RE = rare earth element (lanthanide series)
[b] TRU = transuranium element (e.g., Np, Am, etc.)

CeO_2 and Ce_2O_3 are very similar and that their solubilities are similar, and, hence, due to economic consideration, the use of CeO_2 is being suggested for use in the surrogate formulations. For nonthermal applications, the more soluble $CeCl_3$ salt is recommended (to challenge the leachability testing criterion).

1.3 CAVEATS ON THE APPLICATION OF SIMULATED WASTE

In December 1990, a workshop on radioactive, hazardous and mixed waste sludge management was sponsored by DOE with the primary objective of an exchange of information, experiences, solutions, and future plans for the packaging, grouting, storage, and transport of these materials.[7] The Working Group on Waste Characterization made the following observations:

> Often it is far easier to work with and/or obtain a synthetic surrogate waste as opposed to using or obtaining samples of the actual waste stream. This is especially true for radioactive and mixed wastes in which the radioisotopes impose a more stringent set of handling, safeguards, and transportation regulations. . . . [However] in several cases, the use of a surrogate has yielded dramatically different solidification results when compared to the actual waste stream. Some of these problems occur because an oversimplified surrogate was used.

Several examples were cited wherein trace constituents (e.g., chelates, unexpected set accelerants and retardants) affected the solidification of cement-based waste forms. The authors conclude that

> surrogate wastes are a valuable tool for formulation development but must be used with caution. This is not to say that the use of surrogates be completely abandoned. The problem is that trace components differ between real waste and the surrogate waste and can cause vastly different behavior and interactions with the solidification matrix. . . . Therefore, any work performed using surrogate wastes has to be preliminary, and final formulations must be prepared using the real waste stream.

1.4 NOTES ON WASTE CHARACTERIZATION DATA

Under the aegis of the MWIP, literature reviews are in progress by other investigators to asses the sensitivities and effects of waste constituents upon solidification/stabilization in cement-based matrices and upon waste vitrification. The objectives include identification of constituents that may be problematic in terms of consistently producing an acceptable waste form, and to identify the concentrations at which these problematic species become intolerable for a given technology. For example, numerous anions (such as chloride, fluoride, sulfate, silicate, phosphate, borate, etc.), when present at relatively high concentrations (i.e., >1 wt. %), may affect the quality of cementitious and vitreous waste forms. Certain cationic species may also affect cementation reactions, such as divalent Pb, Zn, or Cu (set inhibitors) or Ca, Ni, Ba, etc. (set accelerants).[8] In addition, certain organic constituents (including chelates) may interfere with cementation reactions even at trace levels (organics are typically destroyed under operating conditions during waste vitrification). Waste constituents of interest to vitrification applications include "glass-formers" (such as oxides of Si, B, Al, Fe, P) as well as some anions that may interfere with the production of durable glass if present at high concentrations (e.g., chloride, sulfate, etc.).[9] In the data summaries reported herein for wastewater treatment sludges, we have attempted to report data (when available) for major constituents as well as for trace constituents that may affect sludge treatability.

From the discussion above, anion concentrations in the aqueous component of the sludge or slurry are important. If these have not been determined in the actual sludge sample, it may be possible to formulate an estimate based upon the concentrations of anions and other soluble constituents in the wastewater influent to the sludge-precipitation process or the supernate from the sludge isolation, using the moisture content of the precipitated sludge material as the basis for estimation of the probable concentration in the wet or dry sludge.

Many sludge waste streams may be inadequately characterized to formulate a completely faithful simulant. In some cases, information may be limited to the minimum required for assessment of regulatory status (e.g., the TCLP test determines what regulated constituents are leached from the raw waste or processed waste form under a defined set of conditions, but provides little or no information on the less mobile constituents). Similarly, many sludge wastes may have been analyzed by EPA SW-846 Method 3050, which is essentially a nitric acid extract from the waste matrix. This methodology, and other wet digestions that do not incorporate HF, may not attack the metals incorporated into aluminosilicate matrices (e.g., soils, sediment, ashes, etc.), and thus may underestimate constituents of potential interest (e.g., Si) to the application of a given treatment technology.[10]

In reviewing analytical data for actual sludges and other "wet" samples, it is imperative that the tabulated results be identified as either "as received" (i.e., including moisture content) or "dry weight" basis. Interconversion of this information requires knowledge of the total solids content or moisture loss upon drying. Note that the results from individual analytical procedures may be reported using different bases.

Another "problematic" analysis in the characterization of wastewater sludges is "oil and grease," operationally defined as a gravimetric determination of the mass of material extracted with use of a nonpolar solvent such as Freon; this parameter does not identify the individual components of the "oil and grease," and typical analytical protocol for determination of organics in waste only identify the hazardous organic constituents.[11] In the wastewater sludges characterized in this report, only the sludge from the Oak Ridge Y-12 Plant WETF contains appreciable "oil and grease," although other wastes stored within the DOE complex may have much higher concentrations.[11] Selection of a suitable surrogate for ''oil and grease'' in a given waste stream may be hampered by a lack of actual component identification. In lieu of actual component identification, a molecular weight profile, using size exclusion chromatography, may facilitate selection of a more realistic surrogate.

In addition to duplicating the essential chemical composition of the sludge, it may be important to mimic certain physiochemical or mechanical properties that may affect processability of the waste stream (such as rheology, etc.); this may require a separate simulant formulation that does not necessarily duplicate the waste-stream chemical composition.[12,13] Also (especially in the case of high-level wastes, in which radiation effects are significant), there can be unexpected changes in physical and chemical properties upon prolonged storage.[14]

2. Y-12 PLANT WEST END TREATMENT FACILITY SLUDGE

2.1 PROCESS DESCRIPTION

The chief purpose of the WETF is to treat nitrate-containing wastes by biodenitrification. Wastes are neutralized with lime and mixed with nutrients (acetate, triethylphosphate, etc.) and transferred to a bioreactor for anoxic denitrification. The denitrified waste slurry is next subjected to bio-oxidation to complete the removal of residual organic compounds; sludge is removed by gravitational sedimentation. The decantate is sent to an effluent polishing unit, in which the solution pH is decreased and the solution sparged (to dissociate any residual uranium from its solubilizing complex with carbonate ion); ferric sulfate is added, and the solution pH is readjusted to a slightly alkaline value, with the addition of flocculating polymer to effect coprecipitation for metals removal.

The sludge bottoms from the WETF clarifier are stored in 500,000-gal. tanks Nos. F-7, F-8, F-9, and F-13 (partially filled); current total inventory (1992) is approximately 7,100 m.[3] The applicable EPA Resource Conservation and Recovery Act (RCRA) waste codes are F001, F002, and F005 (from treatment of solvent residues), and F006 (from treatment of plating waste).

2.2 WASTE CHARACTERIZATION

The WETF sludge is said to consist predominantly of calcium carbonate, biomass, and iron oxyhydroxide.[15] Depleted U (at an average of about 0.42% U-235 isotope, vs 0.71% in natural U) is the primary radioisotope of concern in this waste stream, with very low activity contributions from Tc-99 and TRUs (Np, etc.).[15] Phenolic compounds represent the principal organic hazardous constituents in the sludge. Preliminary characterization data for Tank F-7 raw sludge are compiled by SAIC[15]; a summary of this data, and computed values for simulant material (see Sect. 2.3), are given in Table 2.

Additional compiled data for Tank F-8 and Tank F-9, courtesy of Y. F. Weaver of the Oak Ridge Y-12 Plant, are given in Table 3. These data show a relatively consistent composition both within a tank and from tank-to-tank.

A grab sample of sludge from Tank F-9 was obtained; this sample had a pH value of 9.1, solids content of 44.4%, and a density of 1.27 g/cm.[3] Subsamples of sludge from Tank F-9 and from the prepared simulant as described in Sect. 2.3 were dried at 110°C and examined by energy dispersive X-ray spectroscopy (EDS), to "fingerprint" elemental composition of major constituents; these spectra are presented in Fig. 1a and 1b, respectively. [Note that with the instrumentation used, analytical sensitivity is limited to $\geq$ 1% relative concentration of elements with atomic numbers greater than about 8 (i.e., elements heavier than O)]. The dried sample from Tank F-9 was examined by X-ray diffraction (XRD) to identify the major crystalline phases; calcite ($CaCO_3$) predominated, with additional contributions from aragonite (another mineral phase of $CaCO_3$).

Table 2. Properties of Mixed Waste Sludge and surrogate from WETF Tank F7

Constituent	Units	Preliminary data (mean ± SD)	WETF Surrogate (nominal value)[a]
Physical			
H_2O Weight loss	%	71.4 ± 0.8	(71.1)
Density	g/m^3		
pH	Std. Unit	8.9 ± 0.2	8.5
Flash Point	deg F	135 ± 35	160[b]
Macro[c]			
Calcium	μg/g	267,000	270,000
Aluminum	μg/g	50,300 ± 2,700	45,000
Sodium	μg/g	21,200	21,300
Iron	μg/g	14,300 ± 1,500	14,300
Oil and grease	μg/g	12,700 ± 2,300	15,000[b]
Micro[c]			
Phenol	μg/g	114 ± 45	200
Total Cyanide	μg/g	3.3 ± 2.3	
Total Organic C	μg/g	592 ± 328	
Arsenic	μg/g	ND[d]	--
Barium	μg/g	540 ± 30	600
Beryllium	μg/g	13 ± 3	--
Cadmium	μg/g	44 ± 5	54
Chromium	μg/g	410 ± 30	470
Copper	μg/g	998 ± 135	1,270
Lead	μg/g	240 ± 20	280
Lithium	μg/g	397 ± 31	--
Magnesium	μg/g	9,400 ± 383	9,400
Nickel	μg/g	1,100 ± 100	1,300
Phosphorous	μg/g	3,000 ± 885	3,000
Silicon	μg/g	660 ± 28	--
Silver	μg/g	23 ± 4	--

Table 2 (continued)

Constituent	Units	Preliminary data (mean ± SD)	WETF Surrogate (nominal value)[a]
Uranium (or surrogate[f])	μg/g	1,370 ± 128	1,600
Zinc	μg/g	250 ± 20	--
Chloride	μg/g	628 ± 24	680

[a] Nominal value does not include elemental contributions from dried yeast (biomass surrogate).

[b] Value reported is for dodecane component.

[c] Value reported on dry weight basis.

[d] ND = Not Determined .

[e] NR = Not Reported.

[f] See Table 1; Cerium oxide is recommended

Table 3. Properties of WETF Mixed Waste Sludge (Preliminary Data)

Determination	Units	Tank F9	Tank F8	Tank F7
Physical[a]				
H_2O Weight loss	%	63.0 ± 7	63.3 ± 5	71.4 ± 0.8
Specific Grav.		1.25	1.22	NR[b]
pH	Std. Unit	NR	8.4	8.9 ± 0.2
Flash Point	deg F	>172	>145	135 ± 35
Macro[b]				
Calcium	μg/g	NR	(87.5±26)E3	267,000
Aluminum	μg/g	40,000	(13.7±3)E3	50,300 ± 2,700
Sodium	μg/g	14,300	(7.4±1.2)E3	21,200
Iron	μg/g	9,200	(5.9±3.8)E3	14,300 ± 1,500
Oil and grease	μg/g	11,200	(7.0±5.9)E3	12,700 ± 2,300
Micro[b]				
Phenol	μg/g	154	114	
Total Cyanide	μg/g	<2.7	3.3 ± 2.3	
Total Organic C	μg/g	NR	592	
Arsenic	μg/g	1.97	<0.9	NR

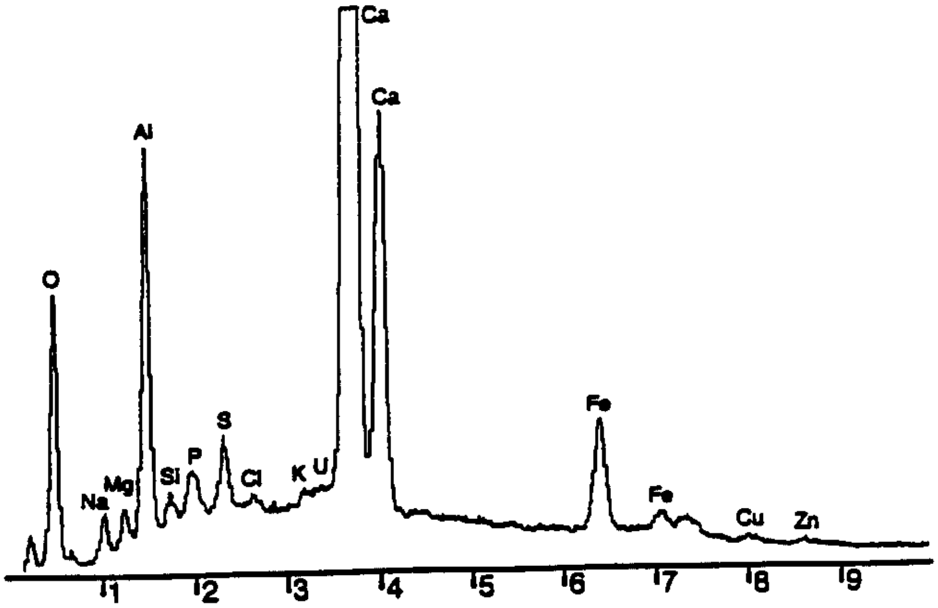
Table 3 (continued)

Determination	Units	Tank F9	Tank F8	Tank F7
Micro[b] (continued)				
Barium	µg/g	210	155	540
Beryllium	µg/g	5.0	2.3	13
Cadmium	µg/g	44	15	44
Chromium	µg/g	405	120	410
Copper	µg/g	530	258±127	1000
Lead	µg/g	1,180	117±75	240
Lithium	µg/g	560	150	400
Magnesium	µg/g	9,700	2700±630	9,400
Mercury	µg/g	5.0	0.29	
Nickel	µg/g	830	315	1,100
Phosphorous	µg/g	2,700	1,100	3,000
Silicon	µg/g	NR	NR	660
Silver	µg/g	175	17	23
Uranium (tot)	µg/g	3,350	4300±2200	1,370
Zinc	µg/g	320	124	250
Alpha	pCi/g	880	774±455	930
Beta	pCi/g	600	980±194	1300
Gamma	pCi/g	230	NR	250
Chloride	mg/L	1,400	780±420	628
Fluoride	mg/L	110	414	320
Nitrate	mg/L	<10	<200	<100
Sulfate	mg/L	2,300	NR	NR

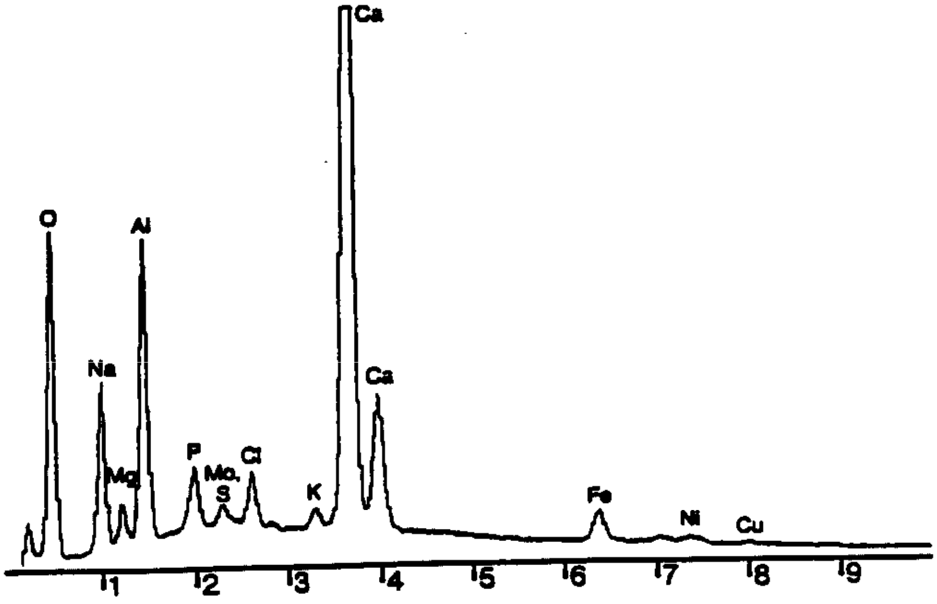
[a] As-Received basis.

[b] NR = Not Recorded.

[c] Dry-weight basis.



(a) X-ray spectra of dried sludge from WETF Tank F-9 (grab sample).



(b) X-ray spectra of dried WETF simulant.

Fig. 1. EDS Spectra of (a) WETF sludge and (b) WETF surrogate.

2.3 FORMULATION OF SIMULATED WEST END TREATMENT FACILITY SLUDGE

Simulant for WETF sludge was formulated in two parts: (1) a dry material blend representing the bulk, nonhazardous constituents and (2) an acidic solution containing RCRA metals and radioactive component or its surrogate. Thus the nonhazardous dry material blend (Table 4) could be used for initial testing using a thermal treatment system. (Note: oxides, such as Fe_2O_3, are acceptable for use in simulants that are to be used in vitrification testing, since they will be formed from the corresponding amorphous oxyhydroxides during processing. However, for nonthermal testing, it is more appropriate to use precipitated amorphous iron oxyhydroxide, rinsed free of soluble impurities.) Note that dried yeast is used in the simulant as a surrogate for the bacterial biomass component in the sludge; yeast is inexpensive, readily available, has comparable elemental composition (notably, trace P, S, Cl, K), and (unlike some bacterial cultures) its use is not restricted by the Food and Drug Administration.

Table 4. Dry mix blend: Recipe to prepare 1 Kg material

Constituent	Surrogate: amount added(g/Kg)	Dry-weight basis Nominal concentration (g/Kg)	
		Surrogate	WETF Sludge
$Al(OH)_3$ FW = 78	130	Al: 45	Al: 44.9 - 55.7
Fe_2O_3 (hematite) FW = 159.7	20.4	Fe: 14.3	Fe: 12.8 - 15.8
$Na_2HPO_4 {*} 7H_2O$ FW =268.1	25.9	P: 3.0 Na: 4.44	P: 2.1 - 3.8 Na: 21
$NaHCO_3$ FW = 84	61	Na: 16.6	Na: 21
$CaCO_3$ FW = 84.3	666	Ca: 267	Ca: 260 - 270
$MgCO_3$ FW = 84.3	32.6	Mg: 9.4	Mg: 9.0 - 9.8
$Ba(OH)_2 {*} 8H_2O$ FW = 315.5	1.38	Ba: 0.60	Ba: 0.51 - 0.60
Dry yeast powder (biomass)	62.7	62.7	Not specified
Total	1000		

Aliquots of the metals spike (Table 5; slightly acidic to facilitate solubilization of metals) may be added for the next tier of performance testing. Note that the uranium surrogates suggested in Table 5 are less readily soluble than the uranyl ion; an acceptable option is to add the relatively insoluble cerium oxide to the dry mix blend for dispersion.

Table 5. Metals Spike Solution: Recipe for 1 Kg Stock (sufficient to prepare 6.7 Kg of wet WETF Sludge)

Constituent	Amount added (g/Kg):
RCRA Metals:	
$Cr(NO_3)*9H_2O$ FW = 400	7.20
$Cd(C_2H_3O_2)_2*2H_2O$ FW = 266.5	0.253
$Cu(C_2H_3O_2)_2*2H_2O$ FW = 199.6	8.00
$Pb(C_2H_3O_2)_2*3H_2O$ FW = 379.4	1.03
$Ni(NO_3)_2*6H_2O$ FW = 290.8	12.9
RAD or Surrogate (to give 1600 μg metal/g-dry weight):	
$UO_2(NO_3)_2*6H_2O$ FW = 502.2	6.73
or	
$Na_3MO_4*2H_2O$ FW = 242	8.07
or	
Ce_2O_3 FW = 328	3.75
or	
CeO_2 FW 172	3.93
0.1 N HCl	balance of 1 Kg
Total	*1000g Metal Spike Solution*

The recommended mixing procedure, to yield a wet sludge comparable in essential composition to the raw WETF material (cf. Table 2), is given in Table 6. Dodecane is selected as the surrogate for the "oil and grease" component on the following bases: (1) no definite composition was determined for the authentic waste "oil and grease," (2) dodecane is a reasonable surrogate for refractory hydrocarbon fragments from biological treatment, and (3) it has a flash point comparable to the values recorded for authentic WETF sludge samples (Table 2).

Table 6. Recipe for preparation of 1 Kg surrogate WETF Sludge

A. To approximately 200g H_2O, add 150g "Metals Spike Solution" (Table 5);

B. Add 0.2 g phenol (C_6H_5OH);

C. Add, alternately, a total of 300 g "Dry Mix Blend" (Table 4) and remainder of water (approx. 500 g) required to yield a final mass of 1000 g. When approximately 1/2 of materials have been added, add 1.3 g dodecane (flash point 160°F) as "oil & grease" surrogate. Add remainder of dry blend and water.

D. Adjust slurry pH value [using HCl or $Ca(OH)_2$] to a value between 8.7–9.1.

This slurry, representing the "as generated" WETF, will give the nominal dry weight composition given in Table 2 (assuming the loss of approximately 70% of mass as water).

Sample calculation: Estimate Cr in 1 Kg sludge slurry prepared as in Steps A–D above.

From Step A: 0.15 Kg of "metals spike" yields

$$[0.15\ \text{Kg ``spike''}] * \frac{7.2\ \text{g}\ Cr(NO_3)_3{*}9H_2O}{\text{Kg ``spike''}} * \frac{52.01\ \text{g Cr/mol}}{400\ \text{g}\ Cr(NO_3)_3{*}9H_2O\text{/mol}} = \text{g Cr Added.}$$

Thus, Cr added to 1 Kg surrogate wet sludge slurry is 0.14 g. The 1 Kg slurry will yield approximately 0.3 Kg solids (from water loss of 70% mass from slurry); thus Cr in dry residue from 1 Kg slurry is

$$\frac{0.14\ \text{g Cr}}{0.3\ \text{Kg dry solids}} = \frac{0.47\ \text{g Cr}}{\text{Kg (dry weight)}} \quad \text{or} \quad Cr = 470\ \mu g/g\ \text{[dry weight (cf. Table 2)]}$$

3. ROCKY FLATS PLANT BY-PASS SLUDGE

3.1 PROCESS DESCRIPTION

An historical perspective on RFP waste streams is presented by Luckett et al.[16] Many sludges were generated, as described in this reference. In general, process sludge slurries were concentrated by passing them through a rotary drum vacuum filter, precoated with diatomaceous earth filter media. A thin layer of filter cake was continuously cut from the drum filter, producing a wet sludge with a water content of up to 60%, and an effective waste loading of about 40% sludge. Operational practice was to add about 0.5 Kg of portland cement/diatomaceous earth (DE) blend (1:1 mix) to each 55-gal drum of sludge (contents, about 600 Kg) to control free water. Some waste packages were solidified by mixing the raw sludge with approximately 30% portland cement.[17]

The sludge considered here originates from treatment (chemical precipitation) of aqueous waste from Pu recovery operations and thus is a TRU waste. The chemicals used for the first stage of this treatment are ferric sulfate, calcium chloride, magnesium sulfate, and coagulates. Sludge solids are concentrated using a filter press coated with diatomite, a diatomaceous earth filter aid. Rocky Flats sludge that was shipped to Idaho National Engineering Laboratory (INEL) was packed into 55-gal drums with cement/DE blend to absorb free liquids.

Effluent from the first stage treatment was filtered and sent to the second stage batch process where it was mixed with radioactive high-nitrate wastes. The same chemicals used to precipitate first stage wastes are used to treat second stage wastes. Again, the precipitated phase was packed with cement/DE blend to control free liquids. The Pu content of the first stage sludge is approximately 15 times that in the second stage material..

3.2 WASTE CHARACTERIZATION

Limited information presented in Luckett et al.[16] suggests that average Pu content in an historic inventory of RFP first stage sludge is of the order of 340 μg/g, wet weight basis.

Recently, Sprenger and Eschen[18] summarized RCRA metals concentration in the RFP sludge; this information is presented in Table 7.

3.3 FORMULATION OF SIMULATED ROCKY FLATS PLANT SLUDGE

Simulant/surrogate for RFP sludge has been formulated for use in testing in-drum melting/solidification with use of microwave energy technology.[18,19] These simulant recipes (Table 8) are recommended for use with other treatment technologies, since microwave treatment of this material represents a benchmark for treatment performance assessment. The simulant described by Peterson et al.[19] approximates historic sludge originating from RFP Bldg. 774. The more recent simulant formulation presented by Sprenger et al.[18] is recommended for comparative purposes (Table 8); if a wet slurry, representing as-stored material, is desired, water can be added to the dry mix blend at about 60 wt% in final slurry. RCRA metals (as nitrate salts) can be spiked to the levels recommended in Table 7 (i.e., 500 μg/g, as metal); Ce_2O_3, or the less expensive CeO_2, can also be added to give a Ce concentration of 500 μg/g to represent the Pu in the actual sludge (see Sect. 3.2).

Table 7. RCRA metals concentration (μg/g, dry wt basis) in actual versus simulated Rocky Flats Plant Sludges[a]

Metal	Actual sludge	Recommended simulant
Ag	56 to 189	500
Cd	4.6 to 13.0	500
Cr	54 to 481	500
Ni	no data	500
Pb	29 to 250	500

[a] Sprenger et al. 1993.[14] (See Reference List.)

EPA hazardous organic waste codes associated with this sludge are F001 (trichloroethylene), F002 (tetrachloroethylene), and F005A (methyl ethyl ketone).

Table 8. Simulated Rocky Flats Plant Sludges (dry weight basis)

	Weight % in Simulated Sludge (Dry)	
Component	Report RFP-4148 (1987)	Sprenger et al. (1993)[a]
Al_2O_3	6.5	--
NaOH	2.5	--
CaO	5.5	--
$CaSO_4 \cdot 2H_2O$	-	38.6
Na_3PO_4	0.4	--
MgO	5.5	--
$Mg(OH)_2$	--	12.6
K_2CO_3	0.9	--
Fe_2O_3	3.5	--
$Fe(OH)_3$	--	31.0
$NaNO_3$	0.9	9.5
Diatomite	74.3	8.3
Total	100.0	100.0

[a] See Ref. 19 in Reference List.

4. LOS ALAMOS NATIONAL LABORATORY DEWATERED TREATMENT SLUDGE

4.1 PROCESS DESCRIPTION

The LANL Liquid Waste Processing Plant uses influent water containing about 100 mg/L total dissolved solids (TDS) for decontamination, rinse down, etc. The resulting wastewater contains about 1000 mg/L TDS. Wastewater is treated by the addition of about 2000 mg/L ferric sulfate [about 400 mg/L as Fe(III)], and precipitated with about 8000 mg/L lime (calcium hydroxide). The precipitate from two sequential stages of precipitation is stored in a tank, then filtered through a rotary drum filter, precoated with filter aid. Formerly, DE filter aid was used, but its use has been phased out in favor of perlite, a processed amorphous volcanic glass; relative to calcined DE (which is essentially pure silica), perlite contains appreciable K and Al impurities. Annual generation of filter cake is about 250 55-gal drums. Total current inventory is estimated at 270 m,^{3} with about 139 m^3 subject to EPA LDR prohibition (source reduction activities have effectively eliminated the RCRA regulated constituents, so that currently generated material is no longer a mixed waste).[20]

4.2 WASTE CHARACTERIZATION

The LANL sludge is not currently very well characterized; information presented here is based, in part, upon on-site interviews.[21] RCRA hazardous organic waste codes are F001, F002, and F005A, caused by low concentrations of miscellaneous chlorinated hydrocarbon solvents. Of 1800 drums of past production, one occurrence is recorded for Cd exceeding TCLP limits. Estimated concentration of bulk constituents in the sludge is given in Table 8. Concentrations of trace metals in sludge is not well documented, although the radioactive elements in the sludge (in units of μg/g, dry weight basis) are reported to be: U (230–360); Pu (3.3–5.2), and Am (0.2–0.32).

The LANL TA-50 sludge predominantly contains precipitating agents and filter media (approximately 25 vol% perlite). From process knowledge, usage of perlite is estimated at about 50 lb per 55-gal waste package,[21] or (assuming that the wet sludge has about 35 wt% solids and has a specific gravity of about 1.35) about 9–10 % by weight of perlite in the wet sludge (about 29 % on a dry-weight basis). Some waste packages may be partially solidified by the addition of portland or gypsum cement. The sludge is reported to have a pH value of 12–12.4, indicating that it is saturated with lime.[21] The composition of major constituents in LANL sludge, based upon interviews and review of available data,[21] is presented in Table 9.

4.3 FORMULATION OF SIMULATED LOS ALAMOS NATIONAL LABORATORY SLUDGE

Table 10 gives a formulation for simulated LANL sludge. Cerium oxide is added as a surrogate for U and Pu in the sludge. Although Bates[6] has recommended the use of Ce_2O_3, the volatility and solubility of this compound is not greatly different from the less expensive CeO_2, which is an acceptable substitute.[22] No reliable data was found to indicate the concentration of RCRA metals in the authentic sludge; however, the basic formulation in Table 10 for 1 Kg

Table 9. Estimated Los Alamos National Laboratory Sludge composition[a]

Parameter	Units	LANL Sludge composition (Dry Weight Basis)
pH	Standard	12–12.4
Total Solids	wt%	≥30
Ca	wt%	21.2–25.9
Si	wt%	7.0–13
Fe	wt%	2.6–3.8
Al	wt%	1.2–1.4
Mg	wt%	0.76–0.88
Chloride	wt%	0.03–0.1
Sulfate	wt%	0.05–0.22
Nitrate	wt%	0.09–2.0
Carbonate	wt%	28.5–34.8
U	µg/g	230–360
Pu	µg/g	3.3–5.2
Am	µg/g	0.2–0.3

[a] See Ref. 21 in the Reference List.

solids can be fortified with the following compounds to yield ~500 μg/g each of the corresponding metal: $NiSO_4{*}6H_2O$ (2.3 g); $CrCl_3{*}6H_2O$ (2.5 g); $Ba(NO_3)_2$ (1 g); $Pb(NO_3)_2$ (0.8 g); and $Cd(NO_3)_2{*}4H_2O$ (1.4 g). If desired, 10 g/Kg of naphthalene and/or 1,2-dichlorobenzene can be added to determine the probable destruction and removal efficiency for RCRA hazardous organic compounds during vitrification.[22]

Table 10. Formulation of simulated Los Alamos National Laboratory Sludge solids

Constituent	Simulant: Amount Added (g/Kg Solids)	Dry-Weight basis Nominal Concentration (g/Kg)	
		Simulant	LANL Sludge
Perlite(SiO_2)	300	Si: ~140	Si: ~70 - 130
$CaCO_3$	600	Ca: 240	Ca: 210 - 280
Fe_2O_3	50	Fe: 35	Fe: 26 - 38
Al_2O_3	26	Al: 14	Al: 12 - 14
MgO	23	Mg: 14	Mg: 13 - 15
CeO_2	1	Ce: 0.85	--
Total:	1000 g		

5. OAK RIDGE K-25 SITE POND WASTE SLUDGE

5.1 PROCESS DESCRIPTION

Prior to their closure, the K-1407-B and K-1407-C ponds at the Oak Ridge K-25 Site (formerly the Oak Ridge Gaseous Diffusion Plant) were used as holding and settling ponds for various wastewater treatment streams, originating from coal pile runoff, steam plant boiler blowdown and ash products, raffinate from various U recovery and equipment decontamination operations, plating wastes, purge cascade blowdown, and miscellaneous laboratory wastes and chemicals.[23,24] K-1407-B pond was operated as a flow-through settling and holding pond, whereas C pond was operated as a total containment basin, receiving dredged sludge from K-1407-B. The primary difference between the sludges in the two ponds is that off-gas scrubber blowdown, ion-exchange resin, chlorides and fluorides were added to C pond, whereas coal pile runoff and fly ash were added to B pond. As part of the remediation of these ponds, a portion of the sludge material was stabilized in a cement-based matrix. In order to comply with negotiated closure milestones (October 1988 for C-pond and June 1989 for B-pond), stabilization efforts were discontinued in 1988, and raw sludge (intermixed with dredged clay pond liner) was placed directly into steel drums (ASTM A-366), resulting in the production of approximately 32,000 drums (89- and 96-gal capacities) of raw sludge and about 46,000 drums of stabilized sludge. Current inventory is about 26,500 m.[3]

Drums containing both grouted and unprocessed sludges were stored outdoors, and many developed both internal and external corrosion, causing leakage which resulted in RCRA noncompliance.[7] Currently, the 45,000 drums of grouted/compliant sludge are stored in process buildings, stacked 2 to 3 drums high. Average grouted drum weight is 1350 lb. Many drums containing the raw/noncompliant sludge have now been overpacked.

Subsequent attempts at remediation of the raw sludge have included sludge drying and repackaging to alleviate the free liquid and corrosion problems.

5.2 WASTE CHARACTERIZATION

Analytical data summaries for B & C pond raw sludges are presented in Tables 11 and 12, respectively; these data are compiled from several sources. Shoemaker[23] and Shoemaker et al.[24] summarize data from an extensive statistical sampling program performed in May 1985, prior to pond closure. More recent data,[25] from a limited sampling of readily accessible drummed waste in Spring 1991, provides some analyses of anions that were not analyzed for in the earlier sampling. This resampling was much less extensive than the earlier work, and some significant differences are noted for some analytes (especially phosphorous). Note that the historical data are based upon the EPA Method 3050 extraction procedure and do not necessarily represent total constituent analysis.

The raw sludges are not characteristically hazardous but contain EPA-listed F006 waste (derived from plating activities). The raw wastes fail the LDR limits for nickel and silver (i.e., leachate concentrations > 0.32 mg/L and > 0.072 mg/L, respectively), whereas the grout stabilized wastes pass these criteria.[25]

Table 11. Summary of data for K-1407-B Pond Sludge samples

Parameter	Unit	K/QT-428 (1991): Sludge phase Mean (Min-Max)	K/TCD-1019 (1992): Sludge phase Mean (Min - Max)
Major Constituents[a]			
Al	µg/g	36,200 (19E3 - 49E3)	11,500 (5.9E3 - 21E3)
Ca	µg/g	58,000 (29E3 - 200E3)	34,700 (6.7E3 - 92E3)
Fe	µg/g	75,500 (20E3 - 200E3)	NR[b]
Phosphate (T)	µg/g	38,400 (18E3 - 63E3)	7.6 (5.0 - 12)
P (T)	µg/g	12,800 (6200 - 21E3)	NR
Metals[a]			
Ag	µg/g		2.2 (0.57 - 7.5)
As	µg/g	162 (5- 250)	159 (20 - 311)
Cd	µg/g	2.0 (0.3 - 5.6)	2.4 (1.0 - 3.8)
Cr	µg/g	815 (290 - 2400)	193 (86 - 330)
Cu	µg/g	1030 (420 - 1600)	197 (100 - 290)
K	µg/g	4100 (2000 - 7300)	1720 (1100 - 3700)
Mg	µg/g	6790 (4.7E3 -16E3)	2120 (1.6E3 - 3E3)
Mn	µg/g	642 (460 - 830)	519 (160 - 2100)
Na	µg/g	1151(390 - 3100)	489 (310 - 950)
Ni	µg/g	4130 (34 - 7100)	1020 (590 - 1400)
Pb	µg/g	121 (66 - 180)	104 (49 - 142)
U (T)	µg/g	516 (69 - 1044)	1620 (420 - 2700)
Zn	µg/g	288 (29 - 1300)	127 (50 - 180)
Radiochem[c]			
U-235	pCi/g	NR	20.8 (3.6 - 34.5)
Tc-99	pCi/g	3640 (1130 - 6760)	2860 (14 - 12300)
Cs-137	pCi/g	6.7 (6.7)	7.3 (2 - 11.5)

Table 11. (continued)

Parameter	Unit	K/QT-428 (1991): Sludge phase Mean (Min-Max)	K/TCD-1019 (1992): Sludge phase Mean (Min - Max)
Radiochem[c] (continued)			
Np237	pCi/g	3.2 (0.6 - 7.7)	10.9 (1.4- 23)
Pu239	pCi/g	3.2 (0.9 - 9)	10.3 (3.2 - 22)
Anions			
Chloride	μg/g	NR	191 (28 - 310)
Fluoride	μg/g	NR	50 (19 - 300)
Nitrate	μg/g	NR	<23
Sulfur	μg/g	NR	3360 (612 - 4900)
Organics:	μg/g	None Detected	NR
Physical[c]			
Density	g/cm^3	1.1 (1.1 - 1.2)	1.35 (1.11 - 1.58)
pH	Std.	7.0 (6.7 - 7.4)	7.9 (7.0 - 11.6)
Total Solids	wt. %	35.2 (14.2 - 51.6)	

[a] dry weight basis
[b] NR = Not Reported; T = total
[c] as-received basis (wet)

Table 12. Summary of data for K-1407-C Pond Sludge samples

Parameter	Unit	K/QT-428 (1991): Sludge phase Mean (Min-Max)	K/TCD-1019 (1992): Sludge phase Mean (Min - Max)
Major Constituents[a]			
Al	μg/g	25,400 (8500 - 42E3)	16,800 (7.6E3 - 26E3)
Ca	μg/g	35,000 (30E3 - 90E3)	74,300 (3.5E3 - 180E3)
Fe	μg/g	25,200 (2500 - 73E3)	NR[b]
Phosphate (T)	μg/g	16,700 (1100 - 60E3)	30 (1.5 - 130)
P (T)	μg/g	5,000 (320 - 18,000)	NR

Table 12. (continued)

Parameter	Unit	K/QT-428 (1991): Sludge phase Mean (Min-Max)	K/TCD-1019 (1992): Sludge phase Mean (Min - Max)
Metals[a]			
Ag	μg/g		3.3 (0.52 - 7.6)
As	μg/g	20 (5 - 97)	58 (18 - 380)
Cd	μg/g	0.7 (0.3 - 1.8)	2.2 (1.0 - 3.8)
Cr	μg/g	600 (30 - 2400)	149 (48 - 510)
Cu	μg/g	580 (120 - 2000)	132 (16 - 270)
K	μg/g	9500 (2600 - 15000)	3150 (490 - 5200)
Mg	μg/g	7900 (5500 - 11000)	2500 (1000 - 4100)
Mn	μg/g	383 (73 - 1000)	518 (120 - 1100)
Na	μg/g	7400 (740 - 15000)	910 (230 - 2000)
Ni	μg/g	5670 (240 - 21000)	980 (410 - 2100)
Pb	μg/g	42 (6 - 140)	51 (33 - 180)
U (T)	μg/g	515 (58 - 1840)	910 (230 - 2800)
Zn	μg/g	220 (68 - 660)	76 (44 - 120)
Radiochem[c]			
U-235	pCi/g	NR	1.4 (1.2 - 2.7)
Tc-99	pCi/g	1570 (132 - 6130)	1110 (330 - 3230)
Cs-137	pCi/g	54 (6.8 - 230)	19 (4.6 - 50)
Np237	pCi/g	20 (0.7 - 82)	24 (6.6 - 63)
Pu239	pCi/g	28 (0.4 - 108)	24 (7.5 - 62)
Anions			
Chloride	μg/g	NR	106 (<50 - 245)
Fluoride	μg/g	NR	121 (15 - 320)
Nitrate	μg/g	NR	<23
Sulfur	μg/g	NR	1430 (480 - 4000)

Table 12. (continued)

Parameter	Unit	K/QT-428 (1991): Sludge phase Mean (Min-Max)	K/TCD-1019 (1992): Sludge phase Mean (Min - Max)
Organics[c]			
Acetone	μg/g	0.3 (0.1 - 1)	NR
Freon-113	μg/g	0.11 (0.1 - 0.27)	NR
Toluene	μg/g	0.062 (0.06 - 0.09)	NR
Physical[c]			
Density	g/cm^3	1.1 (1.1 - 1.2)	1.35 (1.11 - 1.58)
pH	Std.	10 (8.1 - 11)	10.6 (7.2 - 11.6)
Total Solids	wt. %		55.8 (26.3 - 83.8)

[a] dry weight basis
[b] NR = Not Reported; T = total
[c] as-received basis (wet)

Examination of the raw sludge data indicates that these materials contain no significant organic detects (few "detects" above analytical detection limits).

5.2.1 Characterization of Processed Pond Waste

The EPA SW-846 Method 3050 sample preparation procedure has been used for all of the previously compiled sample characterization data for pond waste (as summarized in Tables 11 and 12). This methodology, and others that do not incorporate HF, may not attack the metals incorporated into aluminosilicate matrices (e.g., soils, sediments, ashes, etc.)[10], and thus may underestimate constituents of interest for the application of potential treatment technologies (e.g., silicon and other glass formers, which are significant for vitrification).

We obtained an archive sample of K-25 Site pond waste (original customer ID: PWMP-1; K-25 Site AnaLIS sample identification 920925-005); this material was a grab sample from raw sludge that had been processed by the Chemical Waste Management thermal drying system at a nominal 105°C (the raw sludge had an initial moisture content of 68.7%). The dried sample was submitted to the Analytical Chemistry Laboratory for reanalysis after "total dissolution" by microwave digestion, with HF added ("QWAVE-1000" preparation). In Table 13, these results are compared to previously reported data for the archive sample, using the EPA-3050 methodology (essentially, a digestion using nitric acid). Figure 2 illustrates the apparent representativeness of the selected sample, compared with the historic pond waste sample database, as assessed by the EPA-3050 methodology [also illustrated is the corresponding analysis obtained on a sample of surrogate pond waste; see Table 15 for formulation].

The "total constituent" data confirms the relatively high concentration of silicon in the grab sample of Pond Waste, suggested by earlier standardless semi-quantitative energy-dispersive X-Ray Spectroscopic (SSQ-EDS) estimates (silica, SiO_2, was the major crystalline phase, as identified by X-ray diffraction). Other major constituents of interest to glass formation include iron, aluminum, phosphorous, calcium, and alkali metals. The presence of HF in sample

Table 13. Analysis of dried pond waste sludge sample

Analysis (μg/g)	AnaLIS 920925-005 (EPA-3050 extract)	AnaLIS 940203-099 (total constituent, by microwave digestion, with HF added)	Standardless Semi-Quant. Energy-Dispersive X-Ray Spectroscopy (est.)
Uranium	650	1,000	ND
Silicon	NR	230,000	139,000
Aluminum	37,000	25,000	58,000
Arsenic	100	NR	ND
Barium	120	97	ND
Beryllium	1.8	2.5	ND
Boron	60	84	ND
Cadmium	8.6	14	ND
Calcium	61,000	16,000	150,000
Chromium	220	270	ND
Copper	250	330	ND
Iron	47,000	51,000	220,000
Lead	71	230	ND
Magnesium	4,100	4,300	ND
Manganese	790	4,300	15,000
Nickel	1,500	1,900	ND
Phosphorus	8.0 (as total phosphate)	3,800	1,000
Potassium	5,300	15,000	34,000
Sodium	680	1,600	ND
Strontium	NR	74	ND
Titanium	NR	3,500	15,000
Zinc	200	180	ND

NR = not reported; ND = not detected

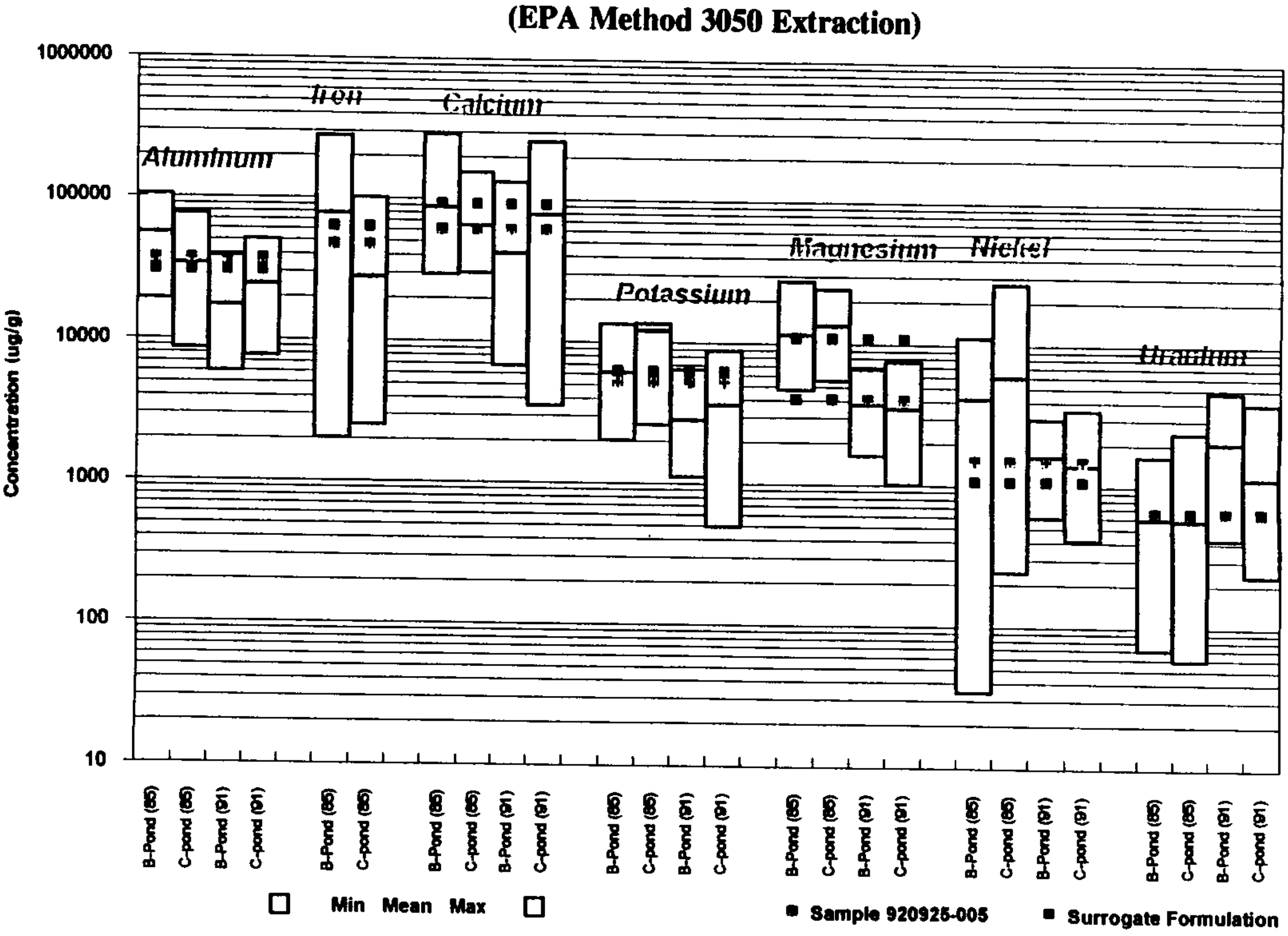


Fig. 2. Analysis of K-25 Site Pond Waste.

preparation QWAVE-1000 appears to somewhat mask the estimates for calcium (forms insoluble compound) and aluminum and (possibly) iron (form strong complexes): spike recoveries are low, and estimates of constituents (especially calcium and iron) are low compared to the SSQ-EDS analysis. The apparent recovery of other soil/sediment major constituents (e.g., potassium, phosphorous) is enhanced by the QWAVE digestion; the recovery of some regulatory constituents (e.g., uranium, cadmium, lead) also appears to be enhanced (relative to the EPA-3050 methodology); more inter-methodology comparisons would be required to determine if this is significant [this is more likely to be significant in the case of (e.g.) incineration ashes, where regulatory metals may be incorporated in the aluminosilicate waste matrix].

The dried sludge Pond Waste sample was sieved; median particle size was approximately 70 μm. Material passing through the smallest sieve (25 μm opening) was subjected to microscopic inspection, which suggested a mean grain size of about 0.26 μm.

5.3 FORMULATION OF SIMULATED POND WASTE SLUDGE

Initial attempts to formulate a simulant based upon a representative concentration of bulk constituents, as determined by wet chemical analysis of the authentic sludge using the EPA Method 3050 preparatory procedure (Tables 11 and 12), yielded a soupy slurry containing only about 21% solids, vs a target value of about 35% solids. One possible inference, as suggested above, is that the Pond Waste contains significant soil or clay component, presumably from pond silt and the underlying clay liner, which were augured into the sludge during dredging operations; this aluminum silicate matrix would be incompletely digested by the reference wet chemical procedure. Table 14 compares data for pulverized illitic clay (Indian red pottery clay

Table 14. Standardless energy dispersive X-ray spectroscopy analysis (wt%)

Element	Indian Red Pottery Clay	Dried PW (Table 12)	Simulated PW (Table 14)
Na	0.0	0.0	2.3
Mg	0.2	0.0	1.2
Al	8.8	5.8	7.9
Si	22.3	13.9	6.6
P	0.0	0.1	7.3
S	0.0	0.4	0.0
K	6.0	3.4	3.4
Ca	1.5	15.0	10.3
Ti	2.6	1.5	0.6
Mn	1.5	1.6	0.7
Fe	15.4	22.0	22.2
O*	41.7	36.3	37.4

* Estimated stoichiometry (closure of mass balance)

obtained from American Art Clay Co., Inc., Indianapolis, IN 46222), dried Pond Waste (grab sample), and Pond Waste simulant, containing clay to adjust the matrix solids content (Table 15). The simulant, with added clay, appears to be a good match for the average bulk constituents in Pond Waste (but note that a wide range of concentrations were noted in the authentic raw sludge samples; see Tables 11 and 12). Note that we have included a moderate concentration of phosphate in the simulant, consistent with the data from the 1985 sampling,[23,24] although the data from the 1991 resampling[25] and the data of Table 13 indicate a relatively low concentration of this species.

The simulant is designed to contain levels of "soluble" electrolytes (e.g., chloride, fluoride, sulfate, K, Na, Mg) and insoluble major constituents (e.g., Al, Ca, Fe, P) that are comparable to the authentic sludges. Adjustment of final sludge slurry pH value is made with lime to an intermediate value (i.e., pH 9). No significant concentrations of hazardous organic compounds are present in the authentic raw sludge, so none are added to the simulant.

The regulatory metals of primary concern in Pond Waste are Ag and Ni (Sect. 5.2); for Tier 2 testing, the basic formulation in Table 15 can be supplemented with soluble salts of these metals; substitution of 1.5 g nickel sulfate ($NiSO_4*6H_2O$) for the 1.5 g Na_2SO_4 in Table 14 would yield a nickel content in the dried solids of about 1,100 μg/g (a value consistent with the median values for the sludge as determined from the 1991 sampling). In contrast, the concentration of Ag in the raw sludge is very low; the addition of 0.005 g silver acetate to the formulation in Table 15 would yield a silver content in the dried solids of about 11 μg/g (somewhat greater than the maximum values recorded from the 1991 sampling).

The maximum U content is less than 3,000 μg/g, which could be mimicked by the addition of ~1 g of cerium oxide to the formulation in Table 15. The other radionuclide of concern is Tc-99, a weak beta emitter with a half-life ($t_{1/2}$) of about 212,000 y and a specific activity of 0.017 Ci/g; a typical value for Tc-99 in sludge is 3,000 pCi/g, equivalent to only about 0.2 μg/g. Technetium is a relatively volatile metal, and its control in a thermal treatment system must be demonstrated. As discussed by Stockdale et. al.[3], Re may be the most appropriate nonradiological surrogate for Tc in a temperature range near 2,000 K; however, a relatively large concentration of Re would be required for accurate determination of mass balance in treatment residuals and aqueous leachates. An alternative for trace analysis would be to use a very small quantity of a short half-life (hence, high specific activity) isotope of Tc (preferably a gamma-emitter, to simplify the counting procedure), such as Tc-99m ($t_{1/2}$ of 6.0 h, and specific activity of 5.4E6 Ci/g),[26] or Tc-95m ($t_{1/2}$ of 61 d, and specific activity of 2.2E4 Ci/g).[27,28]

Table 15. Formulation for simulated Pond Waste bulk constituents

Constituent	Amount Added (for 1 kg)	Unit	Simulant	B-Pond (Mean)[a]	C-Pond (Mean)[a]
Total solids		Wt %	32	35.2[b]	55.8
pH	(adjust w/)	std. unit	~9	7.0	10
H_3PO_4 (85%)	52 g(28.4 mL)	P(mg/Kg)	8,000	12,800	5,000
NaCl	0.30 g	Cl (mg/Kg)[b]	180	191	106
NaF	0.2	F (mg/Kg)[b]	110	50	121
$NaHCO_3$	10 g	Na (mg/Kg)	3,300(T)	1,151	7,400
K_2CO_3	5.3 g	K (mg/Kg)	3,000	4,100	9,500
Na_2SO_4	1.5 g	SO4 (mg/Kg)[b]	1,000	3,360	1,430
MgO	6.6 g	Mg (mg/Kg)	4,000	6,790	7,900
Fe_2O_3 or "$Fe(OH)_3$"[c]	29g 38.3 g	Fe (mg/Kg)	20,000	75,500	25,200
"$Al(OH)_3$"[d] or $Al_2O_3 * 2H_2O$	28.9 g 25.6 g	Al (mg/Kg)	10,000	12,700	9,400
$CaCO_3$	75 g	Ca (mg/Kg)	30,000	20,400	41,400
IRPC[e]	100 g	(See Table 13)			
Water	(balance for 1 Kg)				

[a] Based upon 1985 sampling data median values (Ref. 18, 19. See Reference List.)

[b] Based upon 1991 sampling mean data (Ref. 21. See Reference List.)

[c] For 20 g Fe/Kg-slurry, need about 100 g "Ferri-floc" ($Fe_2(SO_4)_3 * 9H_2O$): *dissolve in about 500* mL water; adjust solution pH to a value of about 8.5, using NaOH solution. Decant off excess water, filter slurry (rinse with water plus trace $NaHCO_3$), and add solids to simulant batch.

[d] If $Al(OH)_3$ is not available, can prepare from soluble Al salt by adjusting pH value to 8.5, as illustrated above for iron. For 10 g Al/Kg-slurry, need, e.g., 139 g $Al(NO_3)_3 * 9H_2O$, or 168 g alum $[Al_2(SO_4)_3 * (NH_4)2SO_4 * 24H_2O]$. NOTE: Fe and Al salts can be neutralized and precipitated simultaneously in the same batch.

[d] *Indian* Red Pottery Clay, a pulverized illitic clay. This material contributes to the bulk constituent material balance in the proportions indicated in Table 13.

6. SUMMARY

The wastewater sludge simulants presented in the text are believed to be representative of this waste stream classification and serve to form a basis for evaluation and comparison of applicable treatment technologies. The LANL and RFP sludge materials contain silica-based filter aids that will be favorable to glass formation upon vitrification. The WETF sludge, without filter aid and containing high concentrations of calcium residue, may offer a significant challenge to vitrification technology. The Pond Waste material, containing some aluminosilicates from intermixing with the pond liner, is an intermediate case. The simulated Pond Waste, with added silica and borax frit, has been used to demonstrate glass/ceramic formation by microwave sintering[29] (DelCul[29] also contains a simulant recipe for a similar heavy metals sludge from the Portsmouth Gaseous Diffusion Plant). These simulated sludges can also be used to demonstrate nonthermal treatment technologies, such as grout-based solidification/stabilization; as mentioned in Sect. 2.3 of the text, the corresponding oxyhydroxides should be substituted for Fe_2O_3 and Al_2O_3 in the simulant formulations for these nonthermal applications. Where adequate characterization data is available for the authentic waste, the simulant formulations may be varied to represent the range of concentrations of potentially problematic constituents in the authentic waste; combined with response surface experimental design methodology, this may help to identify any ''operational window'' limitations for the proposed treatment technology.

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