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Characterization of Transuranium-Contaminated Solid Wastes Residues

G. H. Bryan

April 1981

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
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CHARACTERIZATION OF TRANSURANIUM-
CONTAMINATED SOLID WASTES RESIDUES

G. H. Bryan

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Prepared for
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Pacific Northwest Laboratory
Richland, Washington 99352

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SUMMARY

The selection of technologies for the immobilization of transuranic wastes will depend in part upon the chemical and physical nature of the wastes. Characterization of some of the more important defense transuranic wastes is necessary to develop and evaluate alternative immobilization systems that might be applied to selected defense transuranic wastes. Process sludge and incinerator ash were selected for detailed study because of their importance in the transuranic waste inventory and because of proposed waste acceptance criteria that would exclude sludge and ash as-generated. By experimentally determining the variations in the chemical and physical properties of these wastes, it will be possible to develop and characterize immobilization products (and processes) and to examine their sensitivity to variations in the waste.

Results indicate that there can be rather large differences in the composition of the two wastes. In the case of incinerator residues, large variations in the chemical composition may be expected if the feed is not homogenized to some extent. With sludge, analyses indicated variations in some metallic constituents at a given site and significant variations between sites.

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INTRODUCTION

Several Federal agencies [U.S. Department of Energy (DOE), Environmental Protection Agency (EPA), Department of Transportation (DOT) and others] are developing disposal criteria for radioactive wastes contaminated with trans-uranium (TRU) elements. These wastes were contaminated as a result of operations performed in support of government research and defense programs and from certain commercial nuclear-power fuel cycles. For the purposes of waste classification, TRU wastes are defined as those waste materials containing greater than 10 nCi of certain long-lived alpha-emitting radionuclides per gram of waste. These radionuclides include uranium-233 and all transuranium nuclides, except plutonium-238 and plutonium-241.

Regulations require that TRU wastes be packaged and stored in a retrievable manner separate from other wastes. However, future performance characteristics required of waste forms and packages to be used in the final disposal of TRU wastes are currently uncertain. A program sponsored by the U.S. Department of Energy is being conducted by Battelle at the Pacific Northwest Laboratory (PNL) to characterize some of the more important defense TRU wastes and to develop and evaluate alternative immobilization systems that might be applied to selected defense TRU wastes. To establish minimum waste characterization information required to perform this evaluation, some of the more important waste categories were surveyed.

This report summarizes location, type and amount, projected generation rates, and plutonium content of TRU wastes stored at various DOE sites. The report also describes the physical, chemical, and radiological properties, along with their variations, of two wastes representative of TRU wastes that will require processing before final disposal--process sludge and incinerator ash. The properties described may have a significant effect on the immobilization form of these two wastes.

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SURVEY OF TRU WASTE INVENTORIES

TRU wastes, as distinguished from other radioactive wastes, represent a group of liquid and solid materials with chemical and physical properties that vary over a wide range. These wastes arise from several sources, including currently operating experimental and production facilities and from decontamination and decommissioning activities. Table 1 indicates the quantities of TRU wastes buried or retrievably stored at DOE sites through October 1, 1979 (DOE 1980a). These wastes are categorized as contact handled (surface dose rate <200 mR/h) or remote handled (surface dose rate >200 mR/h). Estimated defense TRU waste generation rates shown in Table 2 (DOE 1980b) indicate the magnitude of the TRU waste problem.

Preliminary inventory data representing TRU solid wastes retrievably stored at the Idaho National Engineering Laboratory (INEL) during a 7-yr period are shown in Table 3. These data were provided by staff at the Rocky Flats Plant.

It is unclear whether contaminated metal scrap, the major stored-waste category, would be acceptable for final disposal without further processing. The recently developed Waste Isolation Pilot Plant (WIPP) acceptance criteria

TABLE 1. Quantities of Defense TRU Wastes as of October 1, 1979(a)

<u>DOE Burial/Storage Sites</u>	<u>Retrievably Stored, m³</u>	
	<u>Contact Handled</u>	<u>Remote Handled</u>
Idaho Falls, Idaho	39,571	19
Hanford, Washington	8,038	0.8
Savannah River, South Carolina	2,365	0
Los Alamos, New Mexico	3,528	73.5
Oak Ridge, Tennessee	384	789
Nevada Test Site, Nevada	243	0
TOTAL	54,129	882.3

(a) DOE 1980a

TABLE 2. Projected Annual DOE Defense TRU Waste Generation

Site	Average Generation Rate 1980 to 2000, m ³ /yr	
	Contact Handled	Remote Handled
Argonne National Laboratory - East	180	10
Battelle, Columbus Laboratories	40	
Bettis Atomic Power Laboratory	<10	
Hanford Operations	470	<740
Idaho National Engineering Laboratory	110	5
Los Alamos National Laboratory	550	10
Lawrence Livermore National Laboratory	25	
Lovelace Biomedical and Environmental Research Institute	<1	
Mound Facility	560	
Oak Ridge National Laboratory	70	75
Paducah Gaseous Diffusion Plant	1	
Rocky Flats Plant	1,700	
Sandia National Laboratories	5	3
Savannah River Plant and Laboratory	<u>150</u>	<u> </u>
Total Projected Annual TRU Waste Generation:	3,900	800

(Irby 1980) would allow disposal of the waste in its current form. However, alternatives for treatment of metals, such as size reduction, electro-decontamination or decontamination by melting, are being developed through other programs (DOE 1980b). Based upon these data and the WIPP acceptance criteria, which specifically exclude unprocessed sludge and ash, process sludges and incinerator residues were selected for immobilization studies.

TABLE 3. Volumes and Plutonium Contents for Important Waste Forms Stored at Idaho National Engineering Laboratory From September 1971 Through December 1978

Waste Form	Volume		Plutonium Content
	m ³	% of Total	wt% of Total
Metal Scrap	9248	30	13
Paper and Rags - Dry	3023	10	3
Second-Stage Sludge	1562	5	1
Paper and Rags - Moist	1554	5	1
Filters - CWS	1491	5	2
First-Stage Sludge	1262	4	13
Raschig Rings - Unleached	317	1	5
Incinerator Ash - Leached	21	0.1	8

As shown in Table 3, first- and second-stage sludges represent 9% of the TRU waste volume and 14 wt% of the total plutonium inventory received by INEL for storage. Leached incinerator residues in the INEL inventory constitute a relatively minor volume (although the combustible waste volume is large) but do constitute 8 wt% of the plutonium. The Los Alamos National Laboratory reports that similar residues comprise over 45 wt% of the plutonium in waste stored at that site (Kosiewicz, Barraclough and Zerwekh 1980).

Although paper and rags (dry and moist) comprise a large volume (15%) and contain a significant fraction of the plutonium (4 wt%), they were not selected for the immobilization studies because their present form is not amenable to immobilization without some prior treatment, such as incineration. Incinerator ash composition will be discussed later in this report.

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PROCESS SLUDGE CHARACTERISTICS

At the Rocky Flats Plant, aqueous wastes from research and defense-related production activities are decontaminated before discharge by a precipitation/flocculation/filtration process. In this process, waste streams are neutralized by addition of sodium hydroxide and scavenged for plutonium and americium by addition of ferric sulfate to produce a hydrated ferric oxide precipitate. The solution is clarified using a flocculant to remove the finely divided particles that may otherwise remain in the effluent, and the solids are removed from the waste stream with a precoated vacuum-drum filter. The resulting sludge consists of a variety of metallic compounds and diatomaceous earth filter aid. Then, the effluent from the first stage of precipitation and clarification, and other wastes, are treated a second time in a similar manner to produce a second-stage sludge. The new decontamination facility at the Rocky Flats Plant (not yet in operation) will not produce two separate sludges but will, instead, have three stages of precipitation and clarification. The resulting sludges will be combined in a common rotary-drum filter, followed by a sludge drier. The sludge from this plant will contain 10 to 15 wt% water as compared to the sludge from the old plant, which contains 50 to 60 wt% water.

At the Los Alamos National Laboratory, treatment of aqueous waste is similar to the Rocky Flats Plant process except that lime is used rather than sodium hydroxide to adjust the pH and increase the flock blanket by the formation of calcium carbonate. In addition, effluent from the precipitation process is treated with carbon dioxide to convert excess lime to calcium bicarbonate to prevent filter media cementation. This treatment is followed by a high-capacity cation exchange process to remove strontium and/or cesium (Emelity, Buchholz and McGinnis 1977). Sludges produced at the Los Alamos National Laboratory contain ~70 wt% water.

To quantify the compositional variability of the sludges, a sampling and analytical program was initiated at both the Rocky Flats Plant and Los Alamos National Laboratory in cooperation with their research staffs. Sludge samples were taken at 2- to 3-wk intervals for 3 mo during steady-state operations

to minimize transient anomalies. A variety of wet-chemical and instrumental techniques were used to analyze each sample for cations and anions in excess of 1 wt%. In addition, residual water content was determined and Pu, Am and U contents ascertained.

Sludge samples were analyzed by emission spectrographic procedures. The results of these analyses are only semi-quantitative in nature and were only used to identify major metallic constituents. The Pu and Am values of the sludge were determined by counting techniques. Uranium was determined by X-ray fluorescence at the Los Alamos National Laboratory and fluorimetry at the Rocky Flats Plant.

Atomic absorption techniques were used at the Rocky Flats Plant for the quantitative determination of Ca, Ga, Mg, Na, K, Si and Fe, while the Los Alamos National Laboratory used a variety of analytical techniques that included spectrophotometric, gravimetric, and titimetric procedures. Anions were also determined by several different procedures at each site, which included spectrophotometry, ion chromatography, titration and the use of specific-ion electrodes. Sludge samples were dried to constant weight at 110 to 120°C to determine water content.

The results of these determinations from each site are shown in Tables 4 and 5. The Rocky Flats Plant results indicate that the material is 55 to 60 wt% water with Si, Ca, Fe and Na comprising the major metallic constituents. The physical characteristics of the sludge are that of a rather sticky, reddish-brown, mud-like substance with a density that varies from about 0.7 to 1.3 g/cm³. The Los Alamos National Laboratory results indicate that the material is ~70 wt% H₂O with major metallic constituents of Ca, Si and Fe. A carbonate content was also noted and is believed to be due to the absorption of CO₂ by the lime used in the waste-treatment process.

The Na content of the Rocky Flats Plant sludge is higher than that of the Los Alamos National Laboratory sludge since the ion-exchange column effluents are neutralized by sodium hydroxide at the Rocky Flats Plant in the first stage of the Aqueous Waste Treatment Plant producing the sludge. At the Los

TABLE 4. Rocky Flats Plant Sludge Analysis

Constituents Analyzed	Average, wt%	Range, wt%	
		High	Low
Al	0.9	2.0	0.5
Ca	9.9	19.4	3.8
Fe	5.0	5.2	4.0
K	1.7	6.5	0.6
Mg	4.2	10.0	1.0
Na	10.0	14.0	6.6
Si	7.8	10.0	5.0
NO ₃ ⁻	7.4	9.1	4.2
PO ₄ ⁻³	3.2	8.9	0
CO ₃ ⁻²	1.7	5.1	0.4
SO ₄ ⁻²	0.3	0.4	0.2
Cl ⁻	0.6	0.8	0.2
F ⁻	0	0	0
H ₂ O	50.5	61.0	19.4
Pu (ppm)	16	39	4
Am (ppm)	8	32	1
U (ppm)	410	1700	1

Alamos National Laboratory plant, the ion-exchange column effluents are treated by a separate process. The Pu and Am contents of the sludge from both plants are quite low.

TABLE 5. Los Alamos National Laboratory Sludge Analysis

Constituents Analyzed	Average, wt	Range, wt%	
		High	Low
Al	0.4	0.4	0.3
Ca	7.1	7.7	6.0
Fe	1.0	1.2	0.8
K	<0.3	<0.3	<0.3
Mg	0.3	0.3	0.3
Na	<0.3	<0.3	<0.3
Si	2.9	3.9	2.3
NO ₃ ⁻	0.4	0.8	0
PO ₄ ⁻³	0.9	1.5	0.4
CO ₃ ⁻²	10.0	11.5	8.1
SO ₄ ⁻²	0	0.1	0
Cl ⁻	0	0	0
F ⁻	0.1	0.1	0.1
H ₂ O	70.1	71.6	67.5
Pu (ppm)	1	2	1
Am (ppm)	0.1	0.1	0.1
U (ppm)	81	104	69

INCINERATOR RESIDUE CHARACTERISTICS

Incineration of contaminated combustible waste is being developed by several government laboratories and is used routinely at the Rocky Flats Plant for plutonium recovery, waste-volume reduction and thermal stabilization. Several reports have described incinerator feed compositions with a representative mixture indicated in Table 6 (Greenhalgh and Devine 1978; Mudge, Schiefelbein, Sullivan and Walter 1975). The resulting residue is dispersible ash of a highly variable composition that depends primarily upon the combustible feed mixture and, to a lesser extent, upon the incinerator design and operating conditions. The ash composition from several incinerators now under development (Mudge et al. 1974; Ziegler et al. 1977) for the volume reduction of radioactive waste is shown in Table 7. Additional information was supplied by D. F. Luthy of The Mound Facility, M. Dukes of Savannah River Laboratory, and F. Hara from PNL.

To establish a better understanding of the chemical variation to be expected in "typical" incinerator residues (and thereby to determine flexibility requirements for proposed host materials), the nonvolatile, inorganic components of several combustible materials were determined by air oxidation in a furnace. The results are shown in Table 8. Estimation of the ash composition

TABLE 6. Typical Incinerator Feed Material

<u>Material</u>	<u>Composition, wt%</u>	<u>Range, wt%</u>
Paper	15	15 to 30
Rags	15	5 to 20
Wood	10	4 to 30
Neoprene	10	10 to 20
Hypalon	10	10 to 20
Latex	5	5 to 15
PVC	20	20 to 50
Polyethylene	<u>15</u>	10 to 20
	100	

TABLE 7. Residue Compositions From Selected Incinerators, wt%

Constituent	Rocky Flats Plant Fluid Bed Incinerator	Mound Cyclone Incinerator	SRL	Los Alamos National Laboratory Controlled-Air Incinerator	Pacific Northwest Laboratory Incinerator
Al ₂ O ₃	35	33	15	28	17
C	5	--	3	6	14
CaCl	--	--	--	2	4
CaO	--	6	24	2	6
Cr ₂ O ₃	10	3	--	--	--
Fe ₂ O ₃	1	5	11	1	--
MgO	--	8	6	--	--
NaCl	5	--	--	--	2
Na ₂ CO ₃	30	--	--	--	--
SiO ₂	10	31	16	35	34
TiO ₂	--	6	18	23	17
ZnO	--	3	6	--	--
TOTAL	96	95	99	97	94

resulting from incineration of any hypothetical combustible mixture was then possible using a simple algorithm. Several mixtures were simulated, and the means, ranges and standard deviations of the ash compositions from these calculations are shown in Table 9. The incineration of lead-containing glove-box gloves results in quite high lead oxide values. Lead gloves may require separate treatment or dilution, depending on the waste-immobilization form of choice. While ash compositions may be encountered that differ greatly from those shown here, these values represent a reasonable range of ash compositions given the information currently available.

TABLE 8. Chemical Composition of Residues in Weight Percent From Oxidation of Combustible Waste Materials(a)

Constituent	Neoprene (1000°C)	Hypalon (1000°C)	Lead- Loaded Neoprene (750°C)	Paper (1000°C)	Cloth (1000°C)	Polyvinyl- Chloride (1000°C)	Polyethelene (1000°C)	Wood (1000°C)	Latex (1000°C)
Al ₂ O ₃	15	24	2	24	6	--	Insufficient	1	--
BaO	--	--	--	--	--	3	Sample After	3	--
CaO	1	3	--	25	12	2	Incineration	13	5
Fe ₂ O ₃	1	--	--	2	3	--	for Analysis.	1	--
K ₂ O	1	1	--	1	--	--		2	10
MgO	29	--	1	2	7	--		2	6
Na ₂ O	2	1	1	2	4	1		11	--
P ₂ O ₅	--	--	--	--	1	8		50	--
PbO	--	--	78	--	--	--		--	--
SiO ₂	39	25	3	31	29	--		3	11
TiO ₂	1	38	3	6	27	--		3	1
ZnO	6	--	1	--	--	--		--	26
C	--	--	--	--	--	67		--	--
SO ₄	--	--	3	1	--	--		--	25
Cl ⁻	--	--	--	--	--	--		--	--
TOTAL	95	92	92	94	89	81		89	84
% Recovery	7.5	30.3	43.7	0.3	0.3	1.4	0.007	3.1	1.4

(a) After material has been heated in air for approximately 3 h at specified temperature.

TABLE 9. Chemical Composition of Typical Incinerator Residues Based on 46 Simulations

<u>Compound</u>	<u>Composition, wt%</u>			
	<u>Average</u>	<u>Std. Dev.</u>	<u>Low</u>	<u>High</u>
Al ₂ O ₃	15	4.5	2.0	20.1
BaO	0.8	0.6	0.3	3.1
CaO	3.3	1.1	1.7	8.2
Fe ₂ O ₃	0.4	0.1	0.2	0.9
K ₂ O	0.8	0.3	0.3	1.6
MgO	4.4	1.9	0.6	10.9
Na ₂ O	1.5	1.0	0.6	4.0
PbO	8.0	16.0	0.1	61.7
SiO ₂	21.0	5.0	4.0	27.0
TiO ₂	20.0	7.0	2.5	29.0
ZnO	1.0	1.0	0.5	2.4
C	8.0	4.0	3.6	21.9
S	0.3	0.2	0.1	0.9
P ₂ O ₅	5.0	5.0	0.4	25.2
Other	11.0	1.0	9.3	14.4

SURROGATE SLUDGE-ASH MIXTURE FOR IMMOBILIZATION STUDIES

Data at the Rocky Flats Plant indicate that the ratio of sludge production to incinerator ash production will be about 3:1. This ratio is obtained from sludge and ash production rates as obtained by flowsheets, estimates provided by operations supervision as to the expected yearly operation time of the waste facility, and projections of sludge and ash generation rates. A composite waste of sludge and ash blended at this ratio was made to be used as a reference blend for immobilization development studies. The mixture was made from nonradioactive sludge produced by the Rocky Flats Plant during its cold operational-phase checkout of the waste facility and duplicated actual production sludge to the extent possible. The nonradioactive ash used was produced by the rotary-kiln incinerator at the Rocky Flats Plant. Then, appropriate reagents were added to bring the mix to the approximate composition indicated by the flowsheets for sludge composition and by the incinerator simulation for ash composition. The sludge-ash composition, along with the flowsheet and ash simulation values, are shown in Table 10.

TABLE 10. Waste Compositions for Immobilization Studies

	Weight Percent									
	<u>Al</u>	<u>Ca</u>	<u>Fe</u>	<u>K</u>	<u>Mg</u>	<u>Na</u>	<u>Si</u>	<u>P</u>	<u>Ti</u>	<u>H₂O</u>
Sludge Flow-sheet	0.1	3.4	13.0	2.3	0.3	6.0	15.4	1.1	0	10
Ash Simulation Average	7.9	2.4	0.3	0.7	2.7	1.1	9.8	2.2	12.0	0
<u>Blended Waste</u>										
<u>3:1 Sludge/Ash:</u>										
Calculated	2.1	3.2	9.8	1.9	0.9	4.8	14.0	1.4	3.0	7.5
Actual	3.0	4.6	9.1	1.2	2.0	3.0	15.5	1.9	3.6	7.5(a)

(a) Indicates calculated value.

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CONCLUSIONS

Selection of technologies for immobilization of TRU wastes will depend in part upon the chemical and physical nature of the wastes. Thus, it is necessary to characterize the wastes of interest to establish the constraints that the wastes will impose on the candidate immobilization systems. Process sludge and incinerator ash were selected for detailed study because of their importance in the TRU waste inventory and because of proposed waste acceptance criteria that would exclude as-generated sludge and ash. Although a study of actual TRU-contaminated ash produced from an operating incinerator was not possible, an approximation of the waste composition was made along with an estimate of the variations. Actual sludge from the two sites was studied to determine variations at each location.

These studies indicate that there can be rather large differences in the composition of the TRU waste in question. In the case of incinerator residues, large variations in the chemical composition may be expected if the feed is not homogenized to some extent. For example, the burning of lead-impregnated glove-box gloves may result in a residue containing an extremely high lead content. With sludge, analyses indicated variations in some metallic constituents at a given site and significant variations in constituents such as Ca, Na and Al between sites. Some constituents, such as CO_3^{2-} , were present in large quantities at one location and not detected at the other. The actinide content of the sludges was quite low in every case, and ash is not expected to cause any significant changes.

A knowledge of these variations will make it possible to develop and characterize immobilization products and processes and to examine their sensitivity to variations in the waste. Then, the immobilization systems can be more meaningfully compared to any set of evaluation criteria in performing a comparative assessment of the alternatives.

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APPENDIX

TABLE A.1. Rocky Flats Plant Cold Sludge and Ash Analysis

<u>Element</u>	<u>Sludge, wt%</u>	<u>Ash, wt%</u>
Al	1.4	15.3
Ba	---	1.0
Ca	9.8	1.0
Cr	0.2	0.1
Cu	0.1	---
Fe	6.2	1.0
K	1.0	0.3
Mg	3.9	0.4
Na	8.3	0.8
P	3.1	0.4
Pb	---	0.1
Si	10.4	17.1
Sr	0.3	---
Ti	---	1.8

TABLE A.2. Rocky Flats Plant Sludge Analysis

Analysis	Weight Percent					
	I	II	III	IV	V	VI
Al	2.0	1.0	1.0	0.5	0.5	0.5
Ca	8.7	19.4	12.2	7.7	7.8	3.8
Fe	6.2	4.8	4.9	5.2	4.7	4.0
K	0.6	0.6	0.6	6.5	1.2	0.6
Mg	1.0	1.0	1.8	10.0	10.0	1.2
Na	6.6	10.5	10.0	9.0	10.0	14.0
Si	10.0	10.0	10.0	5.0	5.0	7.0
NO ₃ ⁻	4.2	8.0	8.1	9.1	6.3	8.4
PO ₄ ³⁻	0	0	0	3.2	6.9	8.9
SO ₄ ²⁻	—	—	—	0.2	0.4	0.3
Cl ⁻	0.2	0.2	1.5	0.7	0.3	0.8
F ⁺	0	0	0	0	0	0
H ₂ O	61.0	55.0	60.2	49.9	57.5	19.4
Pu (ppm)	22	39	5	23	4	5
Am (ppm)	32	5	1	1	1	6
U (ppm)	1700	198	561	2	1	1

TABLE A.3. Los Alamos National Laboratory Sludge Analysis

Analysis	Weight Percent			
	I	II	III	IV
Al	0.4	0.3	0.4	0.4
Ca	6.0	7.2	7.6	7.7
Fe	1.1	0.8	1.2	1.1
K	<0.3	<0.3	<0.3	<0.3
Mg	0.3	0.3	0.3	0.3
Na	<0.3	<0.3	<0.3	<0.3
Si	3.2	3.9	2.3	2.4
NO ₃ ⁻	0	0	0.8	0.6
PO ₄ ³⁻	0.4	0.4	1.5	1.4
CO ₃ ²⁻	8.1	9.9	11.5	10.4
SO ₄ ²⁻	0	0	0.1	0.1
Cl ⁻	0	0	0	0
F ⁻	0.1	0.1	0.1	0.1
H ₂ O	71.6	71.0	67.5	70.2
Pu (ppm)	1	1	2	1
Am (ppm)	0.1	0.1	0.1	0.1
U (ppm)	71	104	81	69

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