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**Characterization of the Radioactive
Sludge from the
ORNL MVST Waste Tanks**

**J. M. Keller
J. M. Giaquinto**



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Chemical and Analytical Sciences Division

Characterization of the Radioactive Sludge from the ORNL MVST Waste Tanks

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J. M. Giaquinto

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Prepared by the
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TABLE OF CONTENTS

LISTOFTABLES	ii
LISTOFFIGURES	iii
ACKNOWLEDGMENTS	iv
ABBREVIATIONSANDACRONYMS
EXECUTIVESUMMARY	vii
1.0 Introduction
2.0 Sample Collection Activities	2
3.0 Analytical Methodology	2
3.1 <u>Sample Preparation</u>	3
3.2 <u>Metal Analysis</u>
3.3 <u>Anion Analysis</u>	6
3.4 <u>Radiochemical Analysis</u>	7
3.5 <u>Criticality Controls</u>	8
4.0 Quality Assurance	9
5.0 Summary of Inorganic and Radiochemical Analytical Results	10
5.1 <u>Description of Data Tables</u>	10
5.2 <u>Discussion of MVST Sludge Characteristics</u>	45
5.4 <u>RCRA Characteristics for the MVST System</u>	50
5.5 <u>TRU Classifications for LLLW System</u>	52
5.6 <u>Distribution of Fissile Material in LLLW System</u>	52
REFERENCES	55
APPENDIXA	A-1
APPENDIXB	B- 1
QC Acceptance Criteria for Radioactive Liquid/Solid Waste Samples	B-1

LIST OF TABLES

Table 1	Summary of MVST Tanks in the ORNL LLLW System	2
Table 2	Analytical Data for Sludge in Tanks W-24	13
Table 3	Analytical Data for Sludge in Tanks W-25	17
Table 4	Analytical Data for Sludge in Tanks W-26	21
Table 5	Analytical Data for Sludge in Tanks W-27	25
Table 6	Analytical Data for Sludge in Tanks W-28	29
Table 7	Analytical Data for Sludge in Tanks W-29	33
Table 8	Analytical Data for Sludge in Tanks W-30	37
Table 9	Analytical Data for Sludge in Tanks W-31	41
Table 10	Assumption Used for Major Compounds in MVST Sludge	45
Table 11	Summary of Quality Checks for MVST Sludge Data	46
Table 12	Distribution of Beta Activity in the MVST Sludge	49
Table 13	Summary of Actinide Elements in MVST Sludge	49
Table 14	Summary of RCRA Regulatory Limits	51
Table 15	Summary of Denature Ratios for MVST Sludge	53
Table 16	Example of Converting Atom % to Weight % for W-31 Sludge	54

LIST OF FIGURES

Figure 1	Distribution of Major Compounds in the MVST Sludge	47
Figure 2	Distribution of Uranium and Thorium in the MVST Sludge	47
Figure 3	Distribution of Uranium in the MVST Sludge in 1996, 2000, and 2001	48
Figure 4	Distribution of Plutonium Alpha Activity in the MVST Sludge	54

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ABBREVIATIONS AND ACRONYMS

ALARA	As Low As Reasonably Achievable
CAO	Carlsbad Area Office
CASD	Chemical and Analytical Sciences Division
CVAA	Cold Vapor Atomic Absorption
DQO	Data Quality Objective
EPA	Environmental Protection Agency
GC/MS	Gas Chromatography/Mass Spectrometry
GC	Gas Chromatography
GFAA	Graphite Furnace Atomic Absorption
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
IDL	Instrument Detection Limit
LCS	Laboratory Control Sample
LLW	Liquid Low-Level Waste
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MVST	Melton Valley Storage Tanks
NTS	Nevada Test Site
ORNL	Oak Ridge National Laboratory
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Program Plan
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
RH-TRU	Remote Handled Transuranic Waste
RMAL	Radioactive Materials Analytical Laboratory (Building 2026)
TC	Total Carbon
TCLP	Toxicity Characteristic Leaching Procedure
TDS	Total Dissolved Solids
TIC	Total Inorganic Carbon or Tentatively Identified Compounds
TIMS	Thermal Ionization Mass Spectrometry
TOC	Total Organic Carbon
TRU	Transuranic
TWCP	Transuranic Waste Characterization Program
WAC	Waste Acceptance Criteria
WIPP	Waste Isolation Pilot Plant

EXECUTIVE SUMMARY

Over the last several years most of the sludge and liquid from the Liquid Low-Level Waste (LLLW) tanks at ORNL has been transferred and consolidated in the Melton Valley Storage Tanks (MVST). The contents of the MVST tanks at the time the sludge samples were collected for this report included the original inventory in the MVSTs along with the sludge and liquid from the Bethel Valley Evaporator Service Tanks (BVEST), Old Hydrofi-acture (OHF) tanks, and the Gunitite and Associated Tanks (GAAT). During the summer of 2001 full core samples of sludge were collected from the MVST tanks. The purpose of this sampling campaign was to characterize and validate that the current radiochemical and chemical contents of the MVST sludge, which was needed to meet the contract agreements prior to the transfer of the waste to another DOE contractor for processing. This report only discusses the analytical characterization of the sludge from the MVST waste tanks.

The isotopic data presented in this report supports the position that fissile isotopes of uranium (^{233}U and ^{235}U) and plutonium (^{239}Pu and ^{241}Pu) were “denatured” as required by the administrative controls stated in the ORNL LLLW waste acceptance criteria (WAC). In general, the MVST sludge was found to be hazardous by RCRA characteristics based on total analysis of chromium, mercury, and lead. Also, the alpha activity due to transuranic isotopes was well above the 100 nCi/g limit for TRU waste. The characteristics of the MVST sludge relative to the WIPP WAC limits for fissile gram equivalent, plutonium equivalent activity, and thermal power from decay heat, were estimated from the data in previous reports and found to be far below the upper boundary for any of the remote-handled transuranic waste (RH-TRU) requirements for disposal of the waste in WIPP. Therefore, the WIPP WAC limits were not evaluated for this set of samples.

Characterization of the Radioactive Sludge from the ORNL MVST Waste Tanks

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1 .0 Introduction

The active ORNL Liquid Low Level Waste (LLLW) system consists of the set of waste tanks summarized in Table 1. As indicated in Table 1, this report only discusses the analytical characterization data for the Melton Valley Storage Tanks (MVST). The characterization data summarized in this report was needed to verify the current composition of the sludge present after the transfer of sludge from the Bethel Valley Evaporator Service Tanks (BVEST), Gunite and Associated Tanks (GAAT), and the Old Hydrofracture (OHF) tanks. The data in this report can also be used to address waste processing options, support the performance assessment (PA) requirements for the Waste Isolation Pilot Plant (WIPP), evaluate the waste characteristics with respect to the waste acceptance criteria (WAC) for WIPP and Nevada Test Site (NT'S), address criticality concerns, and to meet DOT requirements for transporting the waste.

The samples and data collected for this project were performed during May and June of 2001. The level of quality assurance approximates what is required for regulatory measurements with the understanding that, when needed, sample size requirements were reduced, and steps were taken to reduce sample handling to ensure radiation exposures were as-low-as-reasonably-achievable (ALARA). Some procedure modifications were required to handle chemical matrix problems due to the high levels of sodium nitrate, uranium, and thorium present. Any deviations from procedures or problems observed with the tank samples were documented in the data files maintained by the laboratory. The regulatory holding time requirements for mercury and the organic measurements were complied with unless noted differently in the data tables. The Quality Control (QC) Acceptance Criteria for measurements used on this project are summarized in Appendix B.

Table 1 Summary of MVST Tanks in the ORNL LLLW System

Tanks	Data Presented in this report	
	Liquid	Sludge
MVST TANKS		
W-24	none	✓
W-25	none	✓
W-26	none	✓
W-27	none	✓
W-28	none	✓
w-29	none	✓
w-30	none	✓
w-3 1	none	✓

2.0 Sample Collection Activities

A detailed description on the background, operation of the LLLW system, and the sample collection techniques has been presented in previous reports and will not be discussed here (see Sections 2 and 3 of Reference 1). The staff from the Liquid and Gaseous Waste Operations (LGWO) provided all sample collection support and delivered the samples to the analytical laboratory. The tank location for sludge samples collected in 2000 are illustrated in Appendix A and there were only minor changes in the tank contents for this sampling campaign. The documentation for chain-of-custody was prepared, maintained for each sample collected, and stored with the data files by the analytical laboratory.

3.0 Analytical Methodology

The information and data collected from these studies are used to support various activities. The activities include demonstration of regulatory compliance, measurements to support processing options, and to meet data needs for risk assessments and other safety related assessments such as

criticality. Standardized analytical procedures are used to the extent possible to ensure broad acceptance of the data generated. Unless stated otherwise, the U. S. Environmental Protection Agency (EPA) methods are used for the analyses of constituents listed as, hazardous under the Resource Conservation and Recovery Act (RCRA), which includes all the inorganic and organic measurements presented in this report. In general the EPA Guidance Manual, *Test Methods for Evaluating Solid Waste*² (SW-846), is used for inorganic and organic methods. Some modifications of the standard procedures are necessary to handle the high radiation levels and the high salt/solids content. Some procedure modifications are required to generate valid data, these changes were usually needed to correct for chemical or other matrix related interferences All **deviations from** the standard procedures are documented in the raw data files and can be provided upon request to data users.

3.1 Sample Preparation

The interstitial liquid samples collected from the sludge were obtained by centrifuging a well mixed suspended sludge. The clarified liquids were then digested by the SW-846 Method 3015, *Microwave Assist&d Acid Digestion of Aqueous Samples and Extracts*, This sample preparation for aqueous samples was then used for all mercury analyses by CVAA.. Based upon results from a collaborative study³ with Argonne National Laboratory - East (ANL-E), Method 3015/3051 demonstrated excellent recovery for mercury and was used to prepare tank samples for mercury determination.

The primary method for digesting the sludge samples was SW-846 Method 3051, *Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils*. This sample preparation is considered to be a total digestion for metals and radionuclides by regulatory agencies and yields good results for most metals and radionuclides of interest. This digestion gave poor performance on two of the metals of interest, silver and silicon. Although nitric acid is excellent for dissolving silver compounds, there is usually enough chloride present in waste samples to form an insoluble silver chloride (AgCl) precipitate. If the chloride concentration is increased sufficiently, a silver chloride complex (AgCl_3^{-2}) forms which is soluble in the aqueous environment. Improved matrix spike recovery and defensible data for silver were obtained in earlier sampling campaigns using a separate sample digestion discussed in the earlier reports.

If the total silicon content in the sludge must be known to develop waste treatment options such as vitrification, another sample digestion is required. A simple nitric acid treatment will not dissolve most siliceous materials. The SW-846 Method 3052, *Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices*, provides the necessary digestion chemistry to yield good silicon data. Sludge samples were prepared for measurement of total silicon, by taking approximately 0.5 g of sludge and mixing with 7 mL of concentrated nitric acid and 3 mL of hydrofluoric acid in a fluorocarbon microwave vessel. The samples were digested for 10 minutes at full power (1200 watts) holding the digestion temperature at 190°C and then cooled to room temperature. The acid solution was then treated with excess boric acid and heated to 80°C for ten minutes to complex any free fluoride. This digestion mixture is cooled, filtered into a 50 mL volumetric flask, and diluted to volume with ASTM Type II water. Care must be exercised to ensure the digestion solution is cooled to room temperature prior to opening the sealed microwave vessel or there may be a significant loss of the volatile SiF_4 . The free fluoride is complexed with the boron to protect the sample introduction system to the ICP-AES and to prevent a high silicon background from the instrument glassware. This sample digestion with hydrofluoric acid should not be used for radiochemical measurements, especially for measurement of lanthanides or actinides.

Most of the metal and radionuclide data presented in this report are based upon a Method 3051 digestion with approximately a 0.5 gram sludge sample and 10 mL of concentrated nitric acid. After the microwave digestion is completed and the solution cooled to room temperature, the sample is filtered into a volumetric flask and diluted to 50 mL with ASTM Type II water or better. Any residue remaining after the nitric acid digestion consisted of mostly SiO_2 and was discarded.

3.2 Metal Analysis

Three analytical measurement methods were used to determine **all of the metals** included in this report. Most of the metals are **first** determined by SW-846 Method 6010A, *Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES)*. There are several elements of interest for which the ICP-AES has insufficient detection limits, and these elements must be determined by Method 7000A, *Atomic Absorption Methods*. The Radioactive Materials Analytical Laboratory (RMAL) uses a Graphite Furnace Atomic Absorption (GFAA) Spectrometer or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) for elements that require better sensitivity. The elements lead (Pb), thallium (Tl) and antimony (Sb) were analyzed by ICP-MS (Method 6020). The GFAA methods were used for arsenic (Method 7060A) and selenium (Method 7740). All the mercury measurements are done by either Method 7470A, *Mercury in Liquid Waste (Manual Cold-Vapor Technique)*, or Method 7471A, *Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)*. The samples discussed in this report were prepared for mercury analysis by the microwave technique discussed in section 3.1, the sample preparation specified in the mercury methods (7470A and 7471A) were not used.

The level of radioactivity in most LLLW tank samples required that the analytical systems used for metal measurements be modified for operation in a radiochemical hood or glove box. Custom instrument configurations are necessary to ensure contamination control and worker safety. All work was performed in radiochemical laboratories which are operated under strict radiation protection programs, with the use of protective clothing and routine contamination monitoring. Both an ICP-AES system and a GFAA system can generate dry, dusty particles which are difficult to contain and are highly hazardous when radioactive. A detailed description of the RMAL **setup** for these instruments are given in Appendix B of Reference 1.

The instrument detection limits (IDL) for various metals with undiluted aqueous samples are listed in data tables along with the results. For sludge samples, these detection limits must be increased by a factor that represents the dilution that **results from** the sample preparation. For all the MVST sludge samples approximately 0.5 g of **sample** was digested and then diluted to 50 mL which results in about a 100 fold dilution for the sample, and thus a 100 fold increase in the detection limits.

The analytical error for the metal measurements depends upon the analytical method, the concentration level, and the chemical matrix. Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and inductively coupled plasma-mass spectrometry (ICP-MS) are both multi-element measurement techniques that are designed for the best average performance for all elements analyzed. In general, these measurement techniques are not optimized for any single element. The sample introduction system for ICP instruments adds additional variability due to changes in sample density, viscosity, and solids content between samples and/or calibration standards. Overall, the expected analytical error for ICP measurements range from $\pm 4-6\%$ at concentrations above 10 times the detection limit to $\pm 20-50\%$ near the detection limit. These error estimates are typical for both ICP-AES and ICP-MS measurements.

Graphite Furnace AA instruments are generally optimized for a specific element and usually provide lower detection limits and better precision. The expected analytical error for GFAA measurements range from 3-5% for concentrations greater than 10 times the detection limit to 20-40% near the detection limit. One advantage of GFAA analysis is that the measurements are normally well above the method's detection limits. The mercury measurements were done by Cold Vapor Atomic Absorption (CVAA), which is very selective and sensitive for mercury. The analytical errors for CVAA measurements are similar to GFAA work.

3.3 Anion Analysis

The determination of the inorganic anions was needed for the development of process treatment options, to provide information to explain the distribution and chemical behaviors observed in the waste tanks, and to ensure the major chemical constituents were identified in the waste for which data was used to calculate the mass and charge balance for each sample. The common inorganic anions; including fluoride, chloride, bromide, phosphate, nitrate, nitrite, and sulfate; were measured by ion chromatography (IC) with a Dionex Model DX600 system. In addition, several water soluble organic acids were measured along with the inorganic anions. These organic acids were measured in their ionized form and included **formate**, acetate, citrate, and oxalate. Both the citrate and the oxalate can form strong complexes with many metals and change the solution chemistry of these metals in the waste. The ion chromatography system used for measurements on these radioactive

samples was configured such that the components that come into contact with radioactivity were isolated in a radiochemical hood for contamination control,

From past observations, the nitrate content dominates both the mass and charge balance calculations with sludge samples taken from the active LLLW tanks. There are many other anions present in the waste, some of which are measured directly by ion chromatography and others which can be estimated from the metal data such as chromate, dichromate, permanganate, and others. The carbonate is estimated from the total inorganic carbon measurement.

The anion measurement technique was ion chromatography. For simple water samples, without complex chemical matrix problems, the empirical analytical error for ion chromatography measurements ranges from 4-6% for concentrations above 10 times the detection limits to 20-40% near the detection limit. The measurement of anions present at concentration much lower ($< 1/25$) than other anionic species present may increase the overall error of the measurement.

3.4 Radiochemical Analysis

The only standard radiochemical methods useful for radioactive waste characterization are EPA Method 600/900.0, *Gross Alpha and Beta Radioactivity in Drinking Water*, and EPA Method 600/901.1: *Gamma Emitting Radionuclides in Drinking Water*. The EPA Method 6001905.0, *Radioactive Strontium in Drinking Water*, gave poor performance with the chemical matrix found in ORNL LLLW supernatant and sludge samples. The EPA method for gross alpha/beta measurements uses gas-flow proportional counting. In general, this counting technique requires drying a sample at elevated temperatures onto a metal (usually stainless steel) plate, which resulted in the loss of cesium chloride from the MVST samples and yielded poor gross beta results. To avoid this problem, all gross beta measurements reported are based on measurements by liquid scintillation counting. Other than the gamma spectroscopy measurements, all of the radionuclide measurements were done with in-house procedures. The method detection limits for radiochemical measurements are dependent on both sample matrix and count time and are not listed here. In general, the radiochemical measurements used count times to yield at least 1% (10,000 counts) counting statistics. The expected errors for the radiochemical data range from $\pm 5-10$ % for gross alpha/beta

and gamma emitter measurements to $\pm 10-20\%$ for radionuclides that require chemical separations before counting (i.e. ^{99}Tc , ^{90}Sr , ^{129}I , and ^{237}Np).

3.5 Criticality Controls

The current ORNL waste acceptance criteria (WAC) for liquid-low level waste requires that the fissile isotopes of uranium and plutonium be isotopically diluted with ^{238}U and ^{232}Th , respectively. These administrative controls require that the ratio of the ^{238}U mass divided by the fissile equivalent mass (FEM) for uranium be greater than 100. The ^{235}U FEM is a useful scale for criticality calculations that normalizes the fission probability for each fissile isotope to ^{235}U . These FEM factors, designated as f_{35} for ^{235}U mass factors, are discussed and listed in the Appendix A, Table 1 of the ORNL Procedure NCS-1.0, *Nuclear Criticality Safety Program*.

The major fissile isotopes of concern in the ORNL waste tanks are ^{233}U , ^{235}U , and ^{239}Pu . The fissile isotope ^{241}Pu is also present in the waste but the mass is usually several orders of magnitude lower and below a level that would influence the isotopic dilution ratio for plutonium. Other fissile isotopes present in the ORNL waste include isotopes of neptunium, americium, and curium, but the actual mass present in the waste has been too low for major concern, and the low concentration would make it difficult and expensive to measure by mass spectrometry.

The data presented in this report for isotopic dilution ratios (also referred to as denature ratios) reflect both the current ORNL standard practices for disposal of fissile isotopes of uranium and plutonium. The administrative controls which were in effect when the waste was generated were different than current practices.

All calculations dealing with isotopic dilution for criticality safety are based on isotope mass ratios and must not be confused with activity ratios. For any data discussed in this report that uses ^{232}Th relative to isotopic mass ratios, the total thorium concentration and the ^{232}Th concentration are the same value.

The current administrative requirements for criticality control are more conservative than past practices and require that the following conditions be satisfied for uranium,

$$\frac{(^{238}\text{U}) - 200(^{233}\text{U})}{(^{235}\text{U})} \geq 110 \quad (1)$$

$$\frac{(^{238}\text{U}) - 110(^{235}\text{U})}{(^{233}\text{U})} \geq 200 \quad (2)$$

The administrative controls for plutonium require a dilution ratio of 200 parts thorium to one part plutonium (past practices only required a ratio of 100).

4.0 Quality Assurance

Quality assurance during the sampling activities was primarily addressed by the use of approved procedures for sampling the sludge phase found in each waste tank. These procedures provide detailed instructions for the collection, labeling, and shipping of each sample. Chain-of-custody forms were used to track individual samples from their collection point to the analytical laboratory.

The QA for the sludge characterization was based upon the RMAL Radioactive 'Waste Characterization QA Plan'⁴ which defines the basis for quality assurance and quality control used for the analysis of the waste tank samples. The QA plans discuss staff qualification requirements, laboratory participation in performance demonstration programs, quality control acceptance criteria for analytical methods, sample management, and most other laboratory operations. The QA plans implemented at the RMAL for waste characterization meet both the WIPP and the Nevada Test Site (NTS) QA requirements for inorganic, organic, and radiochemical measurements.

5.0 Summary of Inorganic and Radiochemical Analytical Results

5.1 Description of Data Tables

A summary of the inorganic, physical, and radiochemical analytical results for MVST sludge samples are presented in Table 2 through Table 7. Also, MVST data⁵ collected in 1996 was included in these data tables for comparison. These tables are arranged in a similar format to facilitate comparing data from different tanks and to group information into useful units. The analytical data presented in these tables are the consolidation of data from a single project which had a fixed set of analytical requirements. Any parameter reported with a dash (“-”) indicates that the data was not measured for that sample.

The first section, “Physical properties and miscellaneous data”, includes unrelated information that does not fit well into other table groups. The first parameters entered in a column include the RMAL request and sample numbers, which are laboratory filing codes used to track sample information. The next set of data includes information on the moisture or water content and the solids content of the sample. The group is completed with data on the inorganic and organic carbon content. For MVST waste tank samples the inorganic carbon can be assumed to be all carbonate and bicarbonate. The Total Organic Carbon (TOC) provides an upper limit on the organic content in the tank waste but does not include volatile organic compounds. Most of the liquid waste in the active system has been through an evaporator which removes the highly volatile organic compounds from the waste.

The next two sections include groups of metals; the “RCRA metals” are separated out for quick reference. The regulatory limit for the concentrations are listed in parentheses next to each RCRA metal. For the liquid samples the RCRA regulatory limits are used directly, since the supernatant would be defined as the TCLP leachate in the determination of waste characteristics for hazardous waste. The RCRA metal sludge data represents total metal measurements, as defined by EPA. Exceeding the RCRA regulatory limits listed for the sludge samples only indicates that the waste has the potential to be classified as hazardous. The sludge waste should only be classified as RCRA waste if the final waste form fails the TCLP leaching test.

The remaining metals are grouped under “Process metals”, which includes the common Group IA & IIA metals along with elements that could effect chemical processing, criticality concerns, and stabilization techniques such as grouting or vitrification. For the sludge data, all the metals are reported on a “as received” (wet weight) basis.

The section “Semi-quantitative metals by ICP-MS” includes additional metals identified in a full mass range scan by inductively-coupled plasma - mass spectrometry. This measurement helps ensure all major elements have been identified in the waste. Each element reported is not calibrated but is based upon a response factor from a curve generated from a few elements across the mass range. Therefore, these elemental concentrations are listed as estimates only.

The “Anions by ion chromatography” section includes the inorganic anions, several common water soluble organic acids. Two of the organic acids included, citrate and oxalate, are also classified as complexing agents.

The “Beta/gamma emitters” section summarizes the radionuclides that emit gamma-rays and beta particles. This section includes the gross beta activity, radionuclides identified by gamma spectrometry, and several “pure” beta emitters of interest. Many of the “pure” beta emitters (^3H , ^{14}C , and ^{90}Sr) require radiochemical separations prior to measurement by either liquid scintillation or gas-flow proportional counting. The ^{99}Tc was measured by ICP-MS without any prior chemical separation.

The “Alpha emitters” section summarizes the actinide elements in the waste. This section includes the gross alpha activity, an estimate of the activity for each alpha emitter identified in a gross alpha spectrum, and plutonium isotopes determined by alpha spectrometry after a radiochemical separation. For supernatant samples, an estimate of the $^{232}\text{Th}/^{239}\text{Pu}$ mass ratio is included in this section to address criticality concerns if enough thorium is present to calculate the ratio. For the sludge samples, this mass ratio is included with the plutonium mass spectrometry data.

The remaining sections include “Uranium isotopes by TIMS” , “Plutonium isotopes by TIMS”, and “Uranium isotopes by ICP-MS”. These sections summarize the uranium and plutonium data measured by thermal ionization mass spectrometry and, for more recent measurements, the uranium isotopes by ICP-MS. Also, included in these sections are the isotopic mass dilution or “denature” ratios for uranium and plutonium based on the requirements in place when the waste was generated (see section 3.5). The plutonium section for the sludge samples also includes the activity for each plutonium isotope, which was calculated **from** the mass spectrometry data.

Table 2' Analytical Data for Sludge in Tanks W-24

Characteristic (Analysis)	1996 w-24 s	2000 w-24 s	2001 W-24 S	IDL'
Physical properties and miscellaneous data				
Request number	7749c	10224.	12057	
Sample number	960806-006	000509-001	010716-010	
pH	12.8	9.8	9.96	
Water ^a (%)	51.2	64.0	57.6	
TS ^b (mg/g)	488	360	424	
TSS ^c (mg/g)		253	-	
TDS ^d (mg/g)		107		
Bulk density (g/mL)	1.37	1.315	1.276	-
TC ^e (mg/Kg)	13700	28800	22000	15
TIC ^f (mg/Kg)	13700	8790	8500	15
TOC ^g (mg/Kg)	< 15	20000	13400	15
RCRA Metals (±10%)				
Ag ^h (100)' (mg/Kg)	< 1.9			0.012
As (100) (mg/Kg)	< 5.3	< 0.8	< 1.7	0.005
Ba (2000) (mg/Kg)	75.5	86.2	107	0.001
Cd (20) (mg/Kg)	13.9	21.1	37.1	0.168
Cr (100) (mg/Kg)	61.6	236	276	0.013
Hg (4) (mg/Kg)	38.0	74.0	49.9	0.002
Ni (1000) (mg/Kg)	45.2	76.3	90.0	0.078
Pb (100) (mg/Kg)	303	435	606	0.341
Se (20) (mg/Kg)	< 5.3	< 0.8	< 4	0.005
Tl (18) (mg/Kg)	< 5.3	< 0.8	< 4	0.005
Process metals (±10%)				
Al (mg/Kg)	3330	3540	4130	0.035
B (mg/Kg)	4.35	9.52	5.72	0.01
Be (mg/Kg)	4.45	7.28	6.42	0.001
Ca (mg/Kg)	51200	42400	55400	0.03
Co (mg/Kg)	2.42	22.9	42.3	0.039
Cu (mg/Kg)	28.5	67.4	70.6	0.006
Cd (mg/Kg)	0.900	0.409	< 4	0.005
Fe (mg/Kg)	1250	2990	3010	0.014
K (mg/Kg)	13400	7980	9 6 0 0	0.5
Mg (mg/Kg)	9280	6330	9270	0.049
Mn (mg/Kg)	84.7	911	7 6 1	0.002
Mo (mg/Kg)		15.1	56.8	0.038
Na (mg/Kg)	48800	35200	40000	0.075
P (mg/Kg)	1240		-	0.13
Sb (mg/Kg)	< 19	< 41	< 4	0.509
Si ^k (mg/Kg)	3820	2340	3490	0.022
Sr (mg/Kg)	283	208	247	0.001
Th (mg/Kg)	3270	7480	7290	0.376
Ti (mg/Kg)			30.0	0.010
U (mg/Kg)	6780	46500	33800	0.077
V (mg/Kg)	2.23	< 1	< 1	0.013
Zn (mg/Kg)	479	658	700	0.445
Zr (mg/Kg)			244	0.008

Characteristic (Analysis)	1996 w-24 s	2000 w-24 s	2001 W-24 S	IDL'
Semi-quantitative metals by ICP-MS ($\pm 30-50$ %, * indicates data from water leach)				
Au, gold (mg/Kg)	1.5	-		
Bi, bismuth (mg/Kg)	170	-		
Ce, cerium (mg/Kg)	6.5	-	24.4	
Er, erbium (mg/Kg)	0.25			
Eu, europium (mg/Kg)	1.1			
Ga, gallium (mg/Kg)	5.3	-		
Gd, gadolinium (mg/Kg)	1.2	-	8.61	
Ho, holmium (mg/Kg)	1.0	-		
I, iodine (mg/Kg)	* 13			
La, lanthanum (mg/Kg)	9.1		79.3	
Li, lithium (mg/Kg)	* 170		111	
Mo, molybdenum (mg/Kg)	* 2.1			
Nb, niobium (mg/Kg)	0.93			
Rb, rubidium (mg/Kg)	* 1.4	-		
Sn, tin (mg/Kg)	12	-		
Ti, titanium (mg/Kg)	21	-		
W, tungsten (mg/Kg)	1.0	-		
Zr, zirconium (mg/Kg)	8.4	-		
Anions by ion chromatography in water wash of sludge (± 10%)				
<u>Inorganic</u>				
Bromide (mg/Kg)	< 50	< 41	149	0.05
Chloride (mg/Kg)	2770	1560	1770	0.05
Chromate (mg/Kg)	< 20	18.2	36.4	0.05
Fluoride (mg/Kg)	103	468	216	0.05
Nitrate (mg/Kg)	165000	6 7 4 0 0	99700	0.10
Nitrite (mg/Kg)	2250	3920	5030	0.10
Phosphate (mg/Kg)	< 20	35.0	< 8	0.20
Sulphate (mg/Kg)	1370	2570	4690	0.10
<u>Organic</u>				
Acetate (mg/Kg)	242	1310	1250	0.05
Citrate (mg/Kg)	< 20	5.12	19.3	0.50
Formate (mg/Kg)	175	110	149	0.05
Oxalate (mg/Kg)	690	397	196	0.05
Phthalate (mg/Kg)	< 20	27.9	< 4	0.05

Characteristic Analysis)		1996 W-24 S	2000 W-24 S	2001 W-24 S	IDL'
Beta/gamma emitters (±10%)					
Gross beta	(Bq/g)	4.6e+06	4.9e+06	4.4e+06	
⁶³ Ni	(Bq/g)	< 2.5e+01			
⁶⁵ Ni	(Bq/g)	3.3e+03			
⁶⁰ Co	(Bq/g)	2.8e+04	3.0e+04	2.5e+04	
⁹⁰ Sr/ ⁹⁰ Y	(Bq/g)	1.4e+06	1.3e+06	1.2e+06	
⁹⁹ Tc	(Bq/g)	4.5e+02		7.9e+02	
¹²⁹ I	(Bq/g)			-	
¹³⁴ Cs	(Bq/g)	1.3e+04	-	3.3e+03	-
¹³⁷ Cs	(Bq/g)	5.3e+05	6.3e+05	5.5e+05	-
¹⁵¹ Sm	(Bq/g)	< 6.0e+02			-
¹⁵² Eu	(Bq/g)	8.9e+04	2.4e+05	6.9e+05	-
¹⁵⁴ Eu	(Bq/g)	3.8e+04	2.5e+05	2.2e+05	
¹⁵⁵ Eu	(Bq/g)	1.0e+04	5.1e+04	6.6e+04	
²²⁷ Ac	(Bq/g)	< 4.7e+03			
²⁴¹ Pu	(Bq/g)	1.4e+04			
Alpha emitters (±10%)					
Gross alpha	(Bq/g)	34000	200000	150000	
²³² Th	(Bq/g)	13	30	30	
²³³ U	(Bq/g)	1600	8900	640	
²³⁴ U	(Bq/g)	77	710	184	
²³⁵ U	(Bq/g)	2.6	15	9.9	
²³⁸ U	(Bq/g)	84	580	419	
²³⁷ Np	(Bq/g)	10			
²⁴¹ Am	(Bq/g)	3900	10400	15000	
²⁴⁴ Cm	(Bq/g)	22000	155000	105000	
²⁵⁰ Cf	(Bq/g)	< 100		-	
²⁵² Cf	(Bq/g)	< 100			
Total Pu alpha	(Bq/g)	6600	28000	22000	
²³⁸ Pu	(Bq/g)	4000	17000	12000	
²³⁹ Pu/ ²⁴⁰ Pu	(Bq/g)	2600	11000	9400	
²⁴² Pu	(Bq/g)				
TRU activity					
Pu+Am (3700)	(Bq/g)	10500	38400	37000	
Uranium isotopics by ICP-MS (±2%)					
²³³ U	(atom %)	0.067	0.0548	0.0546	0.001
²³⁴ U	(atom %)	0.005	0.0067	0.0024	0.001
²³⁵ U	(atom %)	0.543	0.4606	0.4157	0.001
²³⁶ U	(atom %)	0.006	0.0028	0.0032	0.001
²³⁸ U	(atom %)	99.379	99.475 1	99.5241	0.001
²³³ U/MS	(mg/Kg)	4.45	24.9	18.1	
²³⁵ U/MS	m	36.4	211	139	
²³⁸ U/ ²³⁵ U FEM	-	159	189	206	

Characteristic (Analysis)		1996 W-24 S	2000 W-24 S	2001 W-24 S	IDL'
Plutonium isotopics by TIMS (±1%)					
²³⁸ Pu (atom %)		0.63			
²³⁹ Pu (atom %)		87.14	-		
²⁴⁰ Pu (atom %)		10.81			
²⁴¹ Pu (atom %)		0.37			
²⁴² Pu (atom %)		1.05			
²⁴⁴ Pu (atom %)		< 0.01			
<u>Pu activity</u>					
²³⁸ Pu (Bq/g)		3800			
²³⁹ Pu (Bq/g)		1900			
²⁴⁰ Pu (Bq/g)		870			
²⁴¹ Pu (Bq/g)		14000			
²⁴² Pu (Bq/g)		1.5			
²⁴⁴ Pu (Bq/g)		< 0.1			
(²³⁹ Pu) (ng/g)		960			
²³² Th/ ²³⁹ Pu ^m		3920	613	761	
(a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., (l) Instrument detection limits, and (m) the ratio reported for year 2000 assumes all alpha activity is from ²³⁹ Pu.					

Table 3 Analytical Data for Sludge in Tanks W-25

Characteristic (Analysis)	1996 W-25 S	2000 W-25 S	2001 W-25 S	IDL'
Physical properties and miscellaneous data				
Request number	7749D	10224	12057	
Sample number	960822-036	000509-002	010716-011	
pH	12.6	11.0	11.8	
Water ^a (%)	50.9	59.5	55.4	
TS ^b (mg/g)	4 9 1	406	446	
TSS ^c (mg/g)		282		
TDS ^d (mg/g)		124		
Bulk density (g/mL)	1.36	1.331	1.359	
TC ^e (mg/Kg)	15700	20900	23300	15
TIC ^e (mg/Kg)	15700	11100	11000	15
TOC ^g (mg/Kg)	< 15	9800	12300	15
RCRA Metals (±10%)				
Ag ^h (100) (mg/Kg)	< 1.8		24.8	0.006
As (100) (mg/Kg)	< 1.3	< 0.9	< 1.7	0.011
Ba (2000) (mg/Kg)	105	73.8	106	0.001
Cd (20) (mg/Kg)	11.9	26.0	32.5	0.111
Cr (100) (mg/Kg)	92.1	190	197	0.008
Hg (4) (mg/Kg)	73.2	49.1	106	0.011
Ni (1000) (mg/Kg)	56.8	82.3	85.8	0.065
Pb (100) (mg/Kg)	442	454	679	0.341
Se (20) (mg/Kg)	< 1.3	< 0.9	< 0.8	0.005
Tl (18) (mg/Kg)	< 1.3	< 0.9	< 4	0.005
Process metals (±10%)				
Al (mg/Kg)	5810	2610	6580	0.048
B (mg/Kg)	3.76	7.34	3.65	0.024
Be (mg/Kg)	6.91	6.40	7.83	0.001
Ca (mg/Kg)	50800	56100	60000	0.017
Co (mg/Kg)	5.86	28.1	42.5	0.060
Cu (mg/Kg)	37.0	51.4	61.6	0.006
Cs ^j (mg/Kg)	0.857	< 0.09	< 4	0.005
Fe (mg/Kg)	1810	2160	2530	0.233
K (mg/Kg)	8850	9820	9230	0.178
Mg (mg/Kg)	7650	11500	10600	0.053
Mn (mg/Kg)	140	616	469	0.001
Mo (mg/Kg)		37.0	91.6	0.030
Na (mg/Kg)	52100	42000	46400	0.065
P (mg/Kg)	1850			0.13
Sb (mg/Kg)	114	< 50	< 4	0.509
Si ^k (mg/Kg)	8890	2670	5630	0.022
Sr (mg/Kg)	325	251	234	0.001
Th (mg/Kg)	9250	8960	10100	0.181
Ti (mg/Kg)			32.5	0.010
U (mg/Kg)	7660	30600	18000	0.105
V (mg/Kg)	3.85	< 1.2	< 1	0.013
Zn (mg/Kg)	285	623	458	0.390
Zr (mg/Kg)			331	0.008

Characteristic (Analysis)	1996 W-25 S	2000 W-25 S	2001 W-25 S	IDL'
Semi-quantitative metals by ICP-MS ($\pm 30-50$ %, * indicates data from water leach)				
Au, gold (mg/Kg)	0.28			
Bi, bismuth (mg/Kg)	250			
Ce, cerium (mg/Kg)	9.4		15.5	-
Er, erbium (mg/Kg)	0.02			
Eu, europium (mg/Kg)	2.1			
Ga, gallium (mg/Kg)	8.1			
Gd, gadolinium (mg/Kg)	1.7		5.52	
Ho, holmium (mg/Kg)	2.0			
I, iodine (mg/Kg)	* 12			
La, lanthanum (mg/Kg)	18		38.3	
Li, lithium (mg/Kg)	* 33		98.6	
Mo, molybdenum (mg/Kg)	* 2.0			
Nb, niobium (mg/Kg)	0.72			
Rb, rubidium (mg/Kg)	* 1.0			
Sn, tin (mg/Kg)	18			
Ti, titanium (mg/Kg)	47			
W, tungsten (mg/Kg)	0.61			
Zr, zirconium (mg/Kg)	16			
Anions by ion chromatography in water wash of sludge (± 10%)				
<u>Inorganic</u>				
Bromide (mg/Kg)	< 50	< 50	128	0.05
Chloride (mg/Kg)	2110	1630	1720	0.05
Chromate (mg/Kg)	95.5	29.5	89.1	0.05
Fluoride (mg/Kg)	118	251	155	0.05
Nitrate (mg/Kg)	162000	76600	120000	0.10
Nitrite (mg/Kg)	4967	3190	5440	0.10
Phosphate (mg/Kg)	< 20	< 10	< 10	0.20
Sulphate (mg/Kg)	1750	2590	2810	0.10
<u>Organic</u>				
Acetate (mg/Kg)	318	919	641	0.05
Citrate (mg/Kg)	< 20	25.5	52.7	0.50
Formate (mg/Kg)	247	130	174	0.05
Oxalate (mg/Kg)	521	643	238	0.05
Phthalate (mg/Kg)	< 20	18.4	< 5	0.05

Characteristic (Analysis)	1996 W-25 S	2000 W-25 S	2001 W-25 S	IDL'
Beta/gamma emitters (±10%)				
Gross beta @q/g	8.3e+06	4.3e+06	5.9e+06	
⁵⁹ Ni (Bq/g)	< 2.5e+01			
⁵³ Ni (Bq/g)	3.4e+03			
⁵⁰ Co (Bq/g)	2.5e+04	3.5e+04	2.2e+04	
⁹⁰ Sr/ ⁹⁰ Y (Bq/g)	3.2e+06	1.3e+06	2.3e+06	-
⁹⁹ Tc (Bq/g)	1.0e+02		9.1e+02	
¹²⁹ I (Bq/g)				
¹³⁴ Cs (Bq/g)	6.0e+03		< 1.8e+03	
¹³⁷ Cs (Bq/g)	4.7e+05	4.0e+05	3.5e+05	
¹⁵¹ Sm (Bq/g)	< 5.5e+02			
¹⁵² Eu (Bq/g)	7.1e+04	6.0e+05	3.0e+05	
¹⁵⁴ Eu (Bq/g)	3.7e+04	< 7.1e+04	1.0e+05	
¹⁵⁵ Eu (Bq/g)	8.4e+03	2.8e+04	2.2e+04	
²²⁷ Ac (Bq/g)	< 5.3e+03			
²⁴¹ Pu (Bq/g)	2.6e+04			
Alpha emitters (±10%)				
Gross alpha (Bq/g)	83000	130000	110000	
²³² Th (Bq/g)	38	36	41	
²³³ U (Bq/g)	2800	7200	3900	
²³⁴ U (Bq/g)	100	28	98	
²³⁵ U (Bq/g)	3.2	7.9	5.9	
²³⁸ U (Bq/g)	95	380	230	
²³⁷ Np (Bq/g)	10			
²⁴¹ Am (Bq/g)	9300	10300	8600	
²⁴⁴ Cm (Bq/g)	58000	90500	75000	
²⁵⁰ Cf (Bq/g)	< 100			
²⁵² Cf (Bq/g)	< 100			
Total Pu alpha (Bq/g)	13000	23000	22000	
²³⁸ Pu (Bq/g)	7700	14000	13000	
²³⁹ Pu/ ²⁴⁰ Pu (Bq/g)	4900	9500	9000	
²⁴² Pu (Bq/g)		-		
TRU activity (Bq/g)				
Pu+Am (3700) (Bq/g)	22300	33300	30600	
Uranium isotopics by ICP-MS (±2%)				
²³³ U (atom %)	0.103	0.0674	0.0614	0.001
²³⁴ U (atom %)	0.006	0.0004	0.0024	0.001
²³⁵ U (atom %)	0.597	0.3670	0.4147	0.001
²³⁶ U (atom %)	0.006	0.0048	0.0058	0.001
²³⁸ U (atom %)	99.289	99.5604	99.5157	0.001
²³³ U/MS (mg/Kg)	7.72	20.2	10.8	
²³⁵ U/MS (mg/Kg)	45.2	111	73.7	
²³⁸ U/ ²³⁵ U FEM	137	221	203	

Characteristic (Analysis)	1996 W-25 S	2000 W-25 S	2001 W-25 S	IDL'
Plutonium isotopics by TIMS (• 1%)				
²³⁸ Pu (atom %)	0.72	-		
²³⁹ Pu (atom %)	84.95	-		
²⁴⁰ Pu (atom %)	12.42	-		
²⁴¹ Pu (atom %)	0.40	-		
²⁴² Pu (atom %)	1.51	-		
²⁴⁴ Pu (atom %)	< 0.01	-		
Pu activity				
²³⁸ Pu (Bq/g)	7800	-		
²³⁹ Pu (Bq/g)	3400			
²⁴⁰ Pu (Bq/g)	1800			
²⁴¹ Pu (Bq/g)	26000	-		
²⁴² Pu (Bq/g)	3.8	-		
²⁴⁴ Pu (Bq/g)	< 0.1	-		
(²³⁹ Pu) (ng/g)	1700	-		
²³² Th/ ²³⁹ Pu	6320	-	1054	
(a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., (l) Instrument detection limits, and (m) the ratio reported for year 2000 assumes all alpha activity is from ²³⁹ Pu.				

Table 4 Analytical Data for Sludge in Tanks W-26

Characteristic (Analysis)	1996 W-26 S	2000 W-26 S	2001 W-26 S	I D L
Physical properties and miscellaneous data				
Request number	7749E	10224	12057	
Sample number	960830-044	000509-003	010716-012	
pH	9.7	9.1	9.4	
Water ^a (%)	50.9	56.1	52.9	
TS ^b (mg/g)	491	439	471	
TSS ^c (mg/g)		285		
TDS ^d (mg/g)		154		
Bulk density (g/mL)	1.38	1.363	1.675	-
TC ^e (mg/Kg)	13500	10300	11300	15
TIC ^f (mg/Kg)	11600	3790	3000	15
TOC ^g (mg/Kg)	1900	6510	8300	15
RCRA Metals ($\pm 10\%$)				
Ag ^h (100) ⁱ (mg/Kg)	< 1.9			0.006
As (100) (mg/Kg)	< 1.4	< 0.9	< 1.8	0.011
Ba (2000) (mg/Kg)	63.1	77.9	134	0.001
Cd (20) (mg/Kg)	19.8	21.9	48.2	0.111
Cr (100) (mg/Kg)	74.4	153	212	0.008
Hg (4) (mg/Kg)	12.7	58.3	89.5	0.011
Ni (1000) (mg/Kg)	42.8	74.5	94.3	0.065
Pb (100) (mg/Kg)	212	331	635	0.341
Se (20) (mg/Kg)	< 1.4	< 0.9	< 0.8	0.005
Tl (18) (mg/Kg)	< 1.4	< 0.9	< 4	0.005
Process metals ($\pm 10\%$)				
Al (mg/Kg)	1980	7130	7590	0.048
B (mg/Kg)	11.3	10.6	6.87	0.024
Be (mg/Kg)	1.85	5.63	5.33	0.001
Ca (mg/Kg)	45900	43200	61700	0.017
Co (mg/Kg)	2.69	21.1	41.5	0.060
Cu (mg/Kg)	29.0	70.6	63.7	0.006
Cs ^j (mg/Kg)	1.53	0.729	< 4	0.005
Fe (mg/Kg)	1010	2380	2740	0.233
K (mg/Kg)	25300	18400	17400	0.178
Mg (mg/Kg)	14700	12800	14500	0.053
Mn (mg/Kg)	102	180	278	0.001
Mo (mg/Kg)		46.4	72.4	0.030
Na (mg/Kg)	48900	40500	39700	0.065
P (mg/Kg)	1070			0.13
Sb (mg/Kg)	52.8	< 50	< 4	0.509
Si ^k (mg/Kg)	2100	7470	8330	0.022
Sr (mg/Kg)	254	195	216	0.001
Th (mg/Kg)	3280	4330	5880	0.181
Ti (mg/Kg)		-	30.2	0.010
U (mg/Kg)	19400	36900	30200	0.105
V (mg/Kg)	2.32	< 1.2	< 1	0.013
Zn (mg/Kg)	405	360	440	0.390
Zr (mg/Kg)			200	0.008

Characteristic (Analysis)	1996 W-26 S	2000 W-26 S	2001 W-26 S	IDL'
Semi-quantitative metals by ICP-MS (±30-50 %, * indicates data from water leach)				
Au, gold (mg/Kg)	0.92			
Bi, bismuth (mg/Kg)	78			
Ce, cerium (mg/Kg)	5.5		22.1	
Er, erbium (mg/Kg)	0.24			
Eu, europium (mg/Kg)	2.3			
Ga, gallium (mg/Kg)	4.0			
Gd, gadolinium (mg/Kg)	6.4		8.56	
Ho, hohnium (mg/Kg)	1.0	-		
I, iodine (mg/Kg)	* 12			
La, lanthanum (mg/Kg)	4.8	-	88.8	-
Li, lithium (mg/Kg)	* 76		116	
Mo, molybdenum (mg/Kg)	* 2.2			
Nb, niobium (mg/Kg)	0.22		-	
Rb, rubidium (mg/Kg)	* 2.5			
Sn, tin (mg/Kg)	7.3			
Ti, titanium (mg/Kg)	3.2			
W, tungsten (mg/Kg)	1.5			
Zr, zirconium (mg/Kg)	5.4			
Anions by ion chromatography in water wash of sludge (±10%)				
<u>Inorganic</u>				
Bromide (mg/Kg)	< 50	< 42	186	0.05
Chloride (mg/Kg)	3070	1800	1840	0.05
Chromate (mg/Kg)	< 20	4.94	12.5	0.05
Fluoride (mg/Kg)	< 50	119	64.3	0.05
Nitrate (mg/Kg)	214000	115000	144000	0.10
Nitrite (mg/Kg)	1652	2210	3150	0.10
Phosphate (mg/Kg)	< 20	< 8	< 10	0.20
Sulphate (mg/Kg)	2120	1520	1890	0.10
<u>Organic</u>				
Acetate (mg/Kg)	336	336	198	0.05
. Citrate (mg/Kg)	< 20	< 4	< 5	0.50
Formate (mg/Kg)	243	155	120	0.05
Oxalate (mg/Kg)	44.2	32.1	45.3	0.05
Phthalate (mg/Kg)	< 20	< 4	< 5	0.05

Characteristic Analysis)		1996 W-26 S	2000 W-26 S	2001 W-26 S	IDL ¹
Beta/gamma emitters (±10%)					
Gross beta	(Bq/g)	3.5e+06	4.0e+06	4.4e+06	-
²³⁹ Pu	(Bq/g)	< 3.0e+01	-	-	-
²⁴⁰ Pu	(Bq/g)	4.0e+03	-	-	-
⁶⁰ Co	(Bq/g)	5.8e+04	3.1e+04	3.3e+04	-
⁹⁰ Sr/ ⁹⁰ Y	(Bq/g)	7.1e+05	1.0e+06	1.3e+06	-
⁹⁹ Tc	(Bq/g)	1.2e+03	-	1.1e+03	-
²⁹¹ I	(Bq/g)	-	-	-	-
³⁴ Cs	(Bq/g)	1.2e+04	-	< 2.8e+03	-
³⁷ Cs	(Bq/g)	8.9e+05	8.1e+05	6.3e+05	-
⁵¹ Sm	(Bq/g)	< 5.8e+02	-	-	-
⁵² Eu	(Bq/g)	6.4e+05	3.9e+05	4.6e+05	-
⁵⁴ Eu	(Bq/g)	2.9e+05	1.7e+05	1.9e+05	-
⁵⁵ Eu	(Bq/g)	6.3e+04	< 1.8e+04	5.6e+04	-
²²⁷ Ac	(Bq/g)	< 9.3e+03	-	-	-
²⁴¹ Pu	(Bq/g)	1.5e+04	-	-	-
Alpha emitters (±10%)					
Gross alpha	(Bq/g)	52000	48000	68000	-
²³² Th	(Bq/g)	13	18	18	-
²³³ U	(Bq/g)	10000	10500	8200	-
²³⁴ U	(Bq/g)	180	640	210	-
²³⁵ U	(Bq/g)	4.0	12	8.8	-
²³⁸ U	(Bq/g)	240	460	370	-
²³⁷ Np	(Bq/g)	2	-	-	-
²⁴¹ Am	(Bq/g)	3900	4880	5000	-
²⁴⁴ Cm	(Bq/g)	28000	28000	40000	-
²⁵⁰ Cf	(Bq/g)	< 100	-	-	-
²⁵² Cf	(Bq/g)	< 100	-	-	-
Total Pu alpha	(Bq/g)	7600	9100	13000	-
²³⁸ Pu	(Bq/g)	5300	5200	8300	-
²³⁹ Pu/ ²⁴⁰ Pu	(Bq/g)	2300	3900	5100	-
²⁴² Pu	(Bq/g)	-	-	-	-
TRU activity Pu+Am (3700)	(Bq/g)	11500	14000	18000	-
Uranium isotopics by ICP-MS (±2%)					
²³³ U	(atom %)	0.152	0.0814	0.0773	0.001
²³⁴ U	(atom %)	0.004	0.0076	0.003 1	0.001
²³⁵ U	(atom %)	0.296	0.4800	0.4157	0.001
²³⁶ U	(atom %)	0.006	< 0.0001	0.0040	0.001
²³⁸ U	(atom %)	99.543	99.4310	99.4999	0.001
²³³ U/MS	(mg/Kg)	28.9	29.4	22.9	-
²³⁵ U/MS	(mg/Kg)	56.7	175	124	-
²³⁸ U/ ²³⁵ U FEM		202	171	194	-

Characteristic (Analysis)		1996 W-26 S	2000 W-26 S	2001 W-26 S	IDL'
Plutonium isotopics by TIMS (±1%)					
²³⁸ Pu	(atom %)	1.23			-
²³⁹ Pu	(atom %)	82.27			
²⁴⁰ Pu	(atom %)	15.11			
²⁴¹ Pu	(atom %)	0.57			
²⁴² Pu	(atom %)	0.81			
²⁴⁴ Pu	(atom %)	< 0.01			
<u>Pu activity</u>					
²³⁸ Pu	(Bq/g)	5400			
²³⁹ Pu	(Bq/g)	1300			
²⁴⁰ Pu	(Bq/g)	890	-		
²⁴¹ Pu	(Bq/g)	15000			
²⁴² Pu	(Bq/g)	0.8			
²⁴⁴ Pu	(Bq/g)	< 0.1			
(²³⁹ Pu)	(ng/g)	700	-		765
²³² Th/ ²³⁹ Pu		5730			
(a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., (l) Instrument detection limits, and (m) the ratio reported for year 2000 assumes all alpha activity is from ²³⁹ Pu.					

Table 5 Analytical Data for Sludge in Tanks W-27

characteristic (Analysis)	1996 W-27 S	2000 W-27 S	2001 W-27 S	2001 W-27 H	IDL'
Physical properties and miscellaneous data					
Request number	7749F	10224	12057	12057	
Sample number	960904-248	000509-004	010716-013	010716-014	
pH	12.3	10.0	8.8	11.7	
Water ^a (mg/g)	54.9	68.9	72.7	58.5	
TS ^b (mg/g)	451	311	273	415	
TSS ^c (mg/g)		270			
TDS ^d (mg/g)		41.4			
Bulk density (g/mL)	1.44	1.246	1.169	1.340	
TC ^e (mg/Kg)	12400	10100	6200	6800	15
TIC ^f (mg/Kg)	10000	< 1000	3900	2700	15
TOC ^g m	2400	10100	2300	4000	15
RCRA Metals (±10%)					
Ag ^h (100)' (mg/Kg)	< 1.8				0.006
As (100) (mg/Kg)	< 1.4	< 1	< 1.6	< 1.4	0.011
Ba (2000) (mg/Kg)	41.8	64.2	94.6	58.1	0.001
Cd (20) (mg/Kg)	14.8	< 16	19.3	24.4	0.111
Cr (100) (mg/Kg)	55.3	132	267	83.0	0.008
Hg (4) (mg/Kg)	29.0	196	118	36.2	0.011
Ni (1000) (mg/Kg)	48.9	84.0	73.9	37.4	0.065
Pb (100) (mg/Kg)	157	317	804	200	0.341
Se (20) (mg/Kg)	< 1.4	< 1	< 0.7	< 0.7	0.005
Tl (18) (mg/Kg)	< 1.4	< 1	< 4	< 3	0.005
Process metals (±10%)					
Al (mg/Kg)	2250	7640	8580	5380	0.048
B (mg/Kg)	5.98	11.9	4.70	3.07	0.024
Be (mg/Kg)	1.10	17.7	8.67	2.32	0.001
Ca (mg/Kg)	43700	26700	23400	54100	0.017
Co (mg/Kg)	2.57	16.8	26.2	28.5	0.060
Cu (mg/Kg)	14.2	66.3	71.1	22.0	0.006
Cs ^j (mg/Kg)	0.892	1.81	< 4	< 3	0.005
Fe (mg/Kg)	935	3780	3670	1770	0.233
K (mg/Kg)	6970	4880	4560	7640	0.178
Mg (mg/Kg)	7820	4800	3670	5500	0.053
Mn (mg/Kg)	65.4	387	314	65.7	0.001
Mo (mg/Kg)		98.5	70.8	35.0	0.030
Na (mg/Kg)	58200	30900	23700	52300	0.065
P (mg/Kg)	1000				0.13
Sb (mg/Kg)	37.4	< 50	< 4	< 3	0.509
Si ^k (mg/Kg)	3860	6270	7750	13900	0.022
Sr (mg/Kg)	107	163'	87.8	122	0.001
Th (mg/Kg)	1290	17400	6400	2250	0.181
Ti (mg/Kg)			53.1	209	0.010
U (mg/Kg)	11700	29500	34300	3000	0.105
V (mg/Kg)	3.31	< 1.3	< 1	< 3.6	0.013
Zn (mg/Kg)	360	299	186	460	0.390
Zr (mg/Kg)			234	62.7	0.008

Characteristic (Analysis)	1996 W-27 S	2000 W-27 S	2001 W-27 S	2001 W-27 H	IDL'
Semi-quantitative metals by ICP-MS (±30-50 %, * indicates data from water leach)					
Au, gold (mg/Kg)	0.62				
Bi, bismuth (mg/Kg)	130				
Ce, cerium (mg/Kg)	7.2		34.5	22.1	
Er, erbium (mg/Kg)	0.12				
Eu, europium (mg/Kg)	0.80				
Ga, gallium (mg/Kg)	4.2				
Gd, gadolinium (mg/Kg)	1.9		4.4	8.56	
Ho, holmium (mg/Kg)	1.6				
I, iodine (mg/Kg)	* 6.8				
La, lanthanum (mg/Kg)	7.3		218	88.8	
Li, lithium (mg/Kg)	* 53		227	116	
Mo, molybdenum (mg/Kg)	* 2.0				
Nb, niobium (mg/Kg)	0.56				
Rb, rubidium (mg/Kg)	* 1.2				
Sn, tin (mg/Kg)	4.0			223	
Ti, titanium (mg/Kg)	99				
W, tungsten (mg/Kg)	1.3				
Zr, zirconium (mg/Kg)	4.0				
Anions by ion chromatography in water wash of sludge (±10%)					
<u>Inorganic</u>					
Bromide (mg/Kg)	< 50	< 41	140	37.8	0.05
Chloride (mg/Kg)	2280	1190	2450	688	0.05
Chromate (mg/Kg)	< 20	16.2	18.8	37.5	0.05
Fluoride (mg/Kg)	< 50	246	20.7	235	0.05
Nitrate (mg/Kg)	210000	62500	233000	45400	0.10
Nitrite (mg/Kg)	2283	2600	4380	2460	0.10
Phosphate (mg/Kg)	< 20	12.1	< 10	< 10	0.20
Sulphate m	549	943	530	1040	0.10
<u>Organic</u>					
Acetate (mg/Kg)	196	272	448	42.3	0.05
Citrate (mg/Kg)	< 20	14.1	< 5	< 4	0.50
Fonnnate (mg/Kg)	200	77.8	174	21	0.05
Oxalate	16.0	228	21.2	42.3	0.05
Phthalate (mg/Kg)	< 20	< 4.1	< 5	< 4	0.05

characteristic (Analysis)		1996 W-27 S	2000 W-27 S	2001 W-27 S	2001 W-27 H	IDL'
Beta/gamma emitters (±10%)						
<u>Gross beta</u>	(Bq/g)	1.6e+06	1.0e+07	5.3e+06	2.0e+06	
¹⁹ Ni	(Bq/g)	< 2.0e+01				
⁵³ Ni	(Bq/g)	1.7e+03				
⁵⁰ Co	(Bq/g)	1.2e+04	9.9e+03	7.1e+03	4.93+03	
⁹⁰ Sr/ ⁹⁰ Y	(Bq/g)	4.5e+05	4.0e+06	2.1e+06	6.5e+05	
⁹⁹ Tc	(Bq/g)	8.7e+01		< 2.3e+02	4.3e+02	
¹²⁹ I	(Bq/g)					
¹³⁴ Cs	(Bq/g)	< 8.1e+02		< 1.4e+03	< 1.1e+03	
¹³⁷ Cs	(Bq/g)	3.9e+05	6.0e+05	6.6e+05	4.2e+05	
¹⁵¹ Sm	(Bq/g)	< 5.7e+02				
¹⁵² Eu	(Bq/g)	4.1e+04	2.2e+05	9.9e+04	2.3e+04	
¹⁵⁴ Eu	(Bq/g)	1.7e+04	< 4.2e+04	2.9e+04	7.8e+03	
¹⁵⁵ Eu	(Bq/g)	< 2.7e+03	< 1.8e+04	5.1e+03	< 4.0e+03	
²²⁷ Ac	(Bq/g)	< 6.2e+03				
²⁴¹ Pu	(Bq/g)	6.5e+03				
Alpha emitters (±10%)						
<u>Gross alpha</u>	(Bq/g)	26000	110000	62000	24000	
²³² Th	(Bq/g)	5.2	71	26	9.1	
²³³ U	(Bq/g)	1000	6200	1360	396	
²³⁴ U	(Bq/g)	53	380	343	< 1	
²³⁵ U	(Bq/g)	2.5	11	17.2	0.8	
²³⁸ U	(Bq/g)	145	360	424	37.2	
²³⁷ Np	(Bq/g)	12				
²⁴¹ Am	(Bq/g)	2800	11600	6600	4000	
²⁴⁴ Cm	(Bq/g)	17000	77000	42000	14500	
²⁵⁰ Cf	(Bq/g)	< 100				
²⁵² Cf	(Bq/g)	< 100				
<u>Total Pu alpha</u>	(Bq/g)	3400	16000	11000	4300	
²³⁸ Pu	(Bq/g)	2200	10000	6000	2500	
²³⁹ Pu/ ²⁴⁰ Pu	(Bq/g)	1200	5800	4900	1800	
²⁴² Pu	(Bq/g)					
<u>TRU activity</u>						
Pu+Am (3700)	(Bq/g)	6200	27600	17600	8300	
Uranium isotopics by ICP-MS (±2%)						
²³³ U	(atom %)	0.025	0.0598	0.0113	0.0378	0.001
²³⁴ U	(atom %)	0.002	0.0057	0.0044	0.0000	0.001
²³⁵ U	(atom %)	0.308	0.5068	0.6360	0.3454	0.001
²³⁶ U	(atom %)	0.006	< 0.0001	0.0001	0.0000	0.001
²³⁸ U	(atom %)	99.660	99.4276	99.3481	99.6168	0.001
²³³ U/MS	(mg/Kg)	2.86	17.3	3.79	1.11	
²³⁵ U/MS	(mg/Kg)	35.6	148	215	10.2	
²³⁸ U/ ²³⁵ U FEM	-	296	1 7 2	155	255	

Characteristic (Analysis)		1996 W-27 S	2000 W-27 S	2001 W-27 S	2001 W-27 H	IDL'
Plutonium isotopics by TIMS (±1 %)						
²³⁸ Pu	(atom%)	1.08				
²³⁹ Pu	(atom%)	84.88				
²⁴⁰ Pu	(atom%)	12.64				
²⁴¹ Pu	(atom%)	0.49				
²⁴² Pu	(atom%)	0.91				
²⁴⁴ Pu	(atom%)	< 0.01				
Pu activity						
²³⁸ Pu	(Bq/g)	2400				
²³⁹ Pu	(Bq/g)	670				
²⁴⁰ Pu	(Bq/g)	370				
²⁴¹ Pu	(Bq/g)	6500				
²⁴² Pu	(Bq/g)	0.5				
²⁴⁴ Pu	(Bq/g)	co. 1				
(²³⁹ Pu)	(ng/g)	350				
²³² Th/ ²³⁹ Pu		4390		1336	1201	
<p>(a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., (l) Instrument detection limits, and (m) the ratio reported for year 2000 assumes all alpha activity is from ²³⁹Pu.</p>						

Table 6 Analytical Data for Sludge in Tanks W-28

Characteristic (Analysis)	1996 W-28 s	2000 W-28 s	2001 W-28 S	IDL'
Physical properties and miscellaneous data				
Request number	7749B	10224	12057	
Sample number	960724-060	000509-005	010716-015	
pH	12.3	11.0	8.7	
Water ^a (%)	47.3	61.7	65.4	
TS ^b (mg/g)	527	383	346	
TSS ^c (mg/g)		266		
TDS ^d (mg/g)		117		
Bulk density (g/mL)	1.37	1.306	1.190	
TC ^e (mg/Kg)	12800	8000	8900	15
TIC ^f (mg/Kg)	10200	< 1000	5300	15
TOC ^g (mg/Kg)	2600	8000	3600	15
RCRA Metals (±10%)				
Ag ^h (100) ⁱ (mg/Kg)	< 1.8			0.006
As (100) (mg/Kg)	< 5.0	< 1	< 2.5	0.011
Ba (2000) (mg/Kg)	43.3	64.3	76.9	0.001
Cd (20) (mg/Kg)	24.9	17.7	21.9	0.111
Cr (100) (mg/Kg)	54.8	170	277	0.008
Hg (4) (mg/Kg)	6.55	50.6	81.7	0.011
Ni (1000) (mg/Kg)	53.6	66.5	77.5	0.065
Pb (100) (mg/Kg)	195	396	758	0.341
Se (20) (mg/Kg)	< 5.0	< 1	< 1	0.005
Tl (18) (mg/Kg)	5.97	< 1	< 6	0.005
Process metals (±10%)				
Al (mg/Kg)	571	3860	6980	0.048
B (mg/Kg)	7.33	7.14	< 2.7	0.024
Be (mg/Kg)	1.36	7.62	7.54	0.001
Ca (mg/Kg)	45800	44600	37600	0.017
Co (mg/Kg)	3.53	23.9	36.3	0.060
Cu (mg/Kg)	28.0	57.0	70.6	0.006
Cs ^j (mg/Kg)	0.480	0.401	< 6	0.005
Fe (mg/Kg)	599	2180	3270	0.233
K (mg/Kg)	14600	8860	7900	0.178
Mg (mg/Kg)	14500	8760	7380	0.053
Mn (mg/Kg)	91.0	339	379	0.001
Mo (mg/Kg)		28.0	54.0	0.030
Na (mg/Kg)	61000	37200	32000	0.065
P (mg/Kg)	907			0.13
Sb (mg/Kg)	< 18	< 50	< 6	0.509
Si ^k (mg/Kg)	1080	3000	5620	0.022
Sr (mg/Kg)	151	175	139	0.001
Th (mg/Kg)	1360	4710	4820	0.181
Ti (mg/Kg)			30.0	0.010
U (mg/Kg)	18500	31200	41900	0.105
V (mg/Kg)	1.54	< 1.3	< 1.5	0.013
Zn (mg/Kg)	278	368	292	0.390
Zr (mg/Kg)			184	0.008

Characteristic (Analysis)	1996 W-28 s	2000 W-28 S	2001 W-28 S	IDL'
Semi-quantitative metals by ICP-MS ($\pm 30-50$ %, * indicates data from water leach)				
Au, gold (mg/Kg)	1.9	-		-
Bi, bismuth (mg/Kg)	12	-		
Ce, cerium (mg/Kg)	7.9	-	3.42	
Er, erbium (mg/Kg)	0.07	-		
Eu, europium (mg/Kg)	1.5	-		
Ga, gallium (mg/Kg)	3.1	-		
Gd, gadolinium (mg/Kg)	6.0	-	< 6	
Ho, holmium (mg/Kg)	0.97	-		
I, iodine (mg/Kg)	* 9.1	-		
La, lanthanum (mg/Kg)	2.0	-	212	
Li, lithium (mg/Kg)	* 170	-	172	
Mo, molybdenum (mg/Kg)	* 2.3	-		
Nb, niobium (mg/Kg)	0.30	-		
Rb, rubidium (mg/Kg)	* 1.9	-		
Sn, tin (mg/Kg)	5.9	-	207	
Ti, titanium (mg/Kg)	4.1	-		
W, tungsten (mg/Kg)	1.4	-		
Zr, zirconium (mg/Kg)	1.8	-		
Anions by ion chromatography in water wash of sludge (± 10%)				
<u>Inorganic</u>				
Bromide (mg/Kg)	< 50	< 44	57.3	0.05
Chloride (mg/Kg)	3460	1580	1210	0.05
Chromate (mg/Kg)	< 20	10.7	42.5	0.05
Fluoride (mg/Kg)	< 50	70.6	277	0.05
Nitrate (mg/Kg)	248000	98800	73500	0.10
Nitrite (mg/Kg)	1120	1750	2400	0.10
Phosphate (mg/Kg)	< 20	< 8.8	< 10	0.20
Sulphate (mg/Kg)	1773	973	988	0.10
<u>Organic</u>				
Acetate (mg/Kg)	325	349	70.1	0.05
Citrate (mg/Kg)	< 20	< 4.4	< 5	0.50
Formate (mg/Kg)	271	90.1	5.6	0.05
Oxalate (mg/Kg)	19.1	11.8	119	0.05
Phthalate (mg/Kg)	< 20	< 4.4	< 5	0.05

Characteristic (Analysis)	1996 W-28 S	2000 W-28 S	2001 W-28 S	IDL'
Beta/gamma emitters (±10%)				
Gross beta (Bq/g)	3.1e+06	5.0e+06	4.8e+06	-
⁵⁹ Ni (Bq/g)	<2.5e+01			
⁵³ Ni (Bq/g)	3.3e+03		-	
⁵⁰ Co (Bq/g)	4.2e+04	1.5e+04	4.8e+03	
⁹⁰ Str/ ⁹⁰ Y (Bq/g)	7.0e+05	1.7e+06	1.8e+06	
⁹⁹ Tc (Bq/g)	1.2e+02		< 3.6e+02	-
¹²⁹ I (Bq/g)	4.1e - 02	-		
¹³⁴ Cs (Bq/g)	< 1.2e+03		< 9.3e+02	
¹³⁷ Cs (Bq/g)	3.1e+05	4.8e+05	2.7e+05	
¹⁵¹ Sm (Bq/g)	< 5.6e+02			
¹⁵² Eu (Bq/g)	8.0e+05	5.2e+05	1.2e+05	
¹⁵⁴ Eu (Bq/g)	2.7e+05	1.4e+05	3.2e+04	
¹⁵⁵ Eu (Bq/g)	7.0e+04	<2.0e+04	7.8e+03	
²²⁷ Ac (Bq/g)	< 6.7e+03			
²⁴¹ Pu (Bq/g)	1.2e+04			
Alpha emitters (±10%)				
Gross alpha (Bq/g)	44000	66000	58000	
²³² Th (Bq/g)	5.5	19	20	
²³³ U (Bq/g)	5200	4800	1680	
²³⁴ U (Bq/g)	130	360	338	
²³⁵ U (Bq/g)	3.8	11	20.3	
²³⁸ U (Bq/g)	230	390	518	
²³⁷ Np (Bq/g)	16			
²⁴¹ Am (Bq/g)	4600	7600	6200	-
²⁴⁴ Cm (Bq/g)	25000	43000	35000	
²⁵⁰ Cf (Bq/g)	< 100			
²⁵² Cf (Bq/g)	< 100			-
Total Pu alpha (Bq/g)	4400	11000	13000	
²³⁸ Pu (Bq/g)	2700	6400	7400	
²³⁹ Pu/ ²⁴⁰ Pu (Bq/g)	1700	4600	6100	
²⁴² Pu (Bq/g)	-			
TRU activity				
Pu+Am (3700) (Bq/g)	9000	18600	19200	
Uranium isotopics by ICP-MS (±2%)				
²³³ U (atom %)	0.08 1	0.0441	0.0115	0.001
²³⁴ U (atom %)	0.003	0.005 1	0.0036	0.001
²³⁵ U (atom %)	0.296	0.4827	0.6141	0.001
²³⁶ U (atom %)	0.007	0.0033	0.0001	0.001
²³⁸ U (atom %)	99.613	99.4647	99.3707	0.001
²³³ U/MS (mg/Kg)	14.7	13.5	4.72	
²³⁵ U/MS (mg/Kg)	54.1	149	254	
²³⁸ U/ ²³⁵ U FEM	249	186	160	

Characteristic (Analysis)		1996 W-28 S	2000 W-28 S	2001 W-28 S	IDL'
Plutonium isotopics by TIMS ($\pm 1\%$)					
²³⁸ Pu	(atom %)	< 1.06			
²³⁹ Pu	(atom %)	81.54			
²⁴⁰ Pu	(atom %)	15.93			
²⁴¹ Pu	(atom %)	0.70			
²⁴² Pu	(atom %)	0.76			
²⁴⁴ Pu	(atom %)	0.01			
Pu activity					
²³⁸ Pu	(Bq/g)	3000			
²³⁹ Pu	(Bq/g)	830			
²⁴⁰ Pu	(Bq/g)	600			
²⁴¹ Pu	(Bq/g)	12000			
²⁴² Pu	(Bq/g)	0.5			
²⁴⁴ Pu	(Bq/g)	< 0.1			
(²³⁹ Pu)	(ng/g)	440			
²³² Th/ ²³⁹ Pu		3750		851	
(a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., (l) Instrument detection limits, and (m) the ratio reported for year 2000 assumes all alpha activity is from ²³⁹ Pu.					

Table 7 Analytical Data for Sludge in Tanks W-29

Characteristic (Analysis)	1996 W-29 S	2000 W-29 S	2001 W-29 S	IDL ¹
Physical properties and miscellaneous data				
Request number	No Data	No Data	12057	-
Sample number			010716-016	
pH			12.0	
Water ^a (%)			56.5	
TS ^b (mg/g)			435	
TSS ^c (mg/g)				
TDS ^d (mg/g)				
Bulk density (g/mL)			1.336	
TC ^e (mg/Kg)			12000	15
TIC ^f (mg/Kg)			4100	15
TOC ^g (mg/Kg)			7700	15
RCRA Metals (±10%)				
Ag ^h (100) ⁱ (mg/Kg)				0.006
As (100) (mg/Kg)			< 1.6	0.011
Ba (2000) (mg/Kg)			86.0	0.001
Cd (20) (mg/Kg)			30.7	0.111
Cr (100) (mg/Kg)			149	0.008
Hg (4) (mg/Kg)			40.0	0.011
Ni (1000) (mg/Kg)			71.8	0.065
Pb (100) (mg/Kg)			434	0.341
Se (20) (mg/Kg)			< 0.7	0.005
Tl (18) (mg/Kg)			< 4	0.005
Process metals (±10%)				
Al (mg/Kg)			4060	0.048
B (mg/Kg)			2.06	0.024
Be (mg/Kg)			5.08	0.001
Ca (mg/Kg)			73700	0.017
co (mg/Kg)			40.2	0.060
cu (mg/Kg)			48.4	0.006
Cs ^j (mg/Kg)				0.005
Fe (mg/Kg)			1740	0.233
K (mg/Kg)			8040	0.178
Mg (mg/Kg)			10800	0.053
Mn (mg/Kg)			247	0.001
Mo (mg/Kg)			50.4	0.030
Na (mg/Kg)			37900	0.065
P (mg/Kg)				0.13
Sb (mg/Kg)			< 4	0.509
Si ^k (mg/Kg)			2940	0.022
Sr (mg/Kg)			287	0.001
Th (mg/Kg)			4860	0.181
Ti (mg/Kg)			36.1	0.010
U (mg/Kg)			24100	0.105
V (mg/Kg)			< 1	0.013
Zn (mg/Kg)			485	0.390
Zr (mg/Kg)			187	0.008

Characteristic (Analysis)	1996 W-29 S	2000 W-29 S	2001 w-29 s	IDL'
Semi-quantitative metals by ICP-MS ($\pm 30-50\%$, * indicates data from water leach)				
Au, gold (mg/Kg)				
Bi, bismuth (mg/Kg)				
Ce, cerium (mg/Kg)			1.76	
Er, erbium (mg/Kg)			-	
Eu, europium (mg/Kg)				
Ga, gallium (mg/Kg)				
Gd, gadolinium (mg/Kg)			4.34	
Ho, holmium (mg/Kg)				
I, iodine (mg/Kg)				
La, lanthanum (mg/Kg)			60.7	
Li, lithium (mg/Kg)			117	
Mo, molybdenum (mg/Kg)				
Nb, niobium (mg/Kg)				
Rb, rubidium (mg/Kg)			< 4	
Sn, tin (mg/Kg)				
Ti, titanium (mg/Kg)				
W, tungsten (mg/Kg)				
Zr, zirconium (mg/Kg)				
Anions by ion chromatography in water wash of sludge ($\pm 10\%$)				
<u>Inorganic</u>				
Bromide (mg/Kg)			120	0.05
Chloride (mg/Kg)			1380	0.05
Chromate (mg/Kg)			26	0.05
Fluoride (mg/Kg)			28.9	0.05
Nitrate (mg/Kg)			106000	0.10
Nitrite (mg/Kg)			1920	0.10
Phosphate (mg/Kg)			< 10	0.20
Sulphate (mg/Kg)			918	0.10
<u>Organic</u>				
Acetate (mg/Kg)			267	0.05
Citrate (mg/Kg)			< 4	0.50
Formate (mg/Kg)			99.1	0.05
Oxalate (mg/Kg)			40.6	0.05
Phthalate (mg/Kg)			< 4	0.05

Characteristic (Analysis)		1996 W-29 S	2000 W-29 S	2001 w-29 s	IDL'
Beta/gamma emitters (±10%)					
Gross beta	(Bq/g)			4.1e+06	
⁵⁹ Ni	(Bq/g)			-	
⁶³ Ni	(Bq/g)			2.5e+04	
⁶⁰ Co	(Bq/g)			1.4e+06	
⁹⁰ Sr/ ⁹⁰ Y	(Bq/g)			9.3e+02	
⁹⁹ Tc	(Bq/g)				
¹²⁹ I	(Bq/g)				
¹³⁴ Cs	(Bq/g)			3.3e+03	
¹³⁷ Cs	(Bq/g)			5.8e+05	
¹⁵¹ Sm	(Bq/g)			-	
¹⁵² Eu	(Bq/g)			1.5e+05	
¹⁵⁴ Eu	(Bq/g)			4.8e+04	
¹⁵⁵ Eu	(Bq/g)			1.8e+04	
²⁴⁴ Ac	(Bq/g)			-	
²⁴¹ Pu	(Bq/g)			-	
Alpha emitters (±10%)					
Gross alpha	(Bq/g)			64000	
¹³² Th	(Bq/g)			20	
¹³³ U	(Bq/g)			2310	
¹³⁴ U	(Bq/g)			32.5	
¹³⁵ U	(Bq/g)			9.7	
¹³⁸ U	(Bq/g)			298	
¹³⁷ Np	(Bq/g)				
¹⁴¹ Am	(Bq/g)			7500	
¹⁴⁴ Cm	(Bq/g)			42000	
¹⁵⁰ Cf	(Bq/g)			-	
¹⁵² Cf	(Bq/g)			-	
Total Pu alpha	(Bq/g)			11000	
²³⁸ Pu	(Bq/g)			6600	
²³⁹ Pu/ ²⁴⁰ Pu	(Bq/g)			4500	
²⁴² Pu	(Bq/g)			-	
TRU activity					
Pu+Am (3700)	(Bq/g)			18500	
Uranium isotopics by ICP-MS (±2%)					
²³³ U	(atom %)			0.0274	0.001
²³⁴ U	(atom %)			0.0006	0.001
²³⁵ U	(atom %)			0.5093	0.001
²³⁶ U	(atom %)			0.0007	0.001
²³⁸ U	(atom %)			99.4620	0.001
²³³ U/MS	(mg/Kg)			6.47	
²³⁵ U/MS	(mg/Kg)			121	
²³⁸ U/ ²³⁵ U FEM	-			185	

Characteristic (Analysis)		1996 W-29 S	2000 W-29 S	2001 W-29 S	IDL ^l
Plutonium isotopics by TIMS (±1%)					
²³⁸ Pu	(atom %)			-	-
²³⁹ Pu	(atom %)			-	-
²⁴⁰ Pu	(atom %)			-	-
²⁴¹ Pu	(atom %)			-	-
²⁴² Pu	(atom %)			-	-
²⁴⁴ Pu	(atom %)			-	-
Pu activity					
²³⁸ Pu	(Bq/g)			-	-
²³⁹ Pu	(Bq/g)			-	-
²⁴⁰ Pu	(Bq/g)			-	-
²⁴¹ Pu	(Bq/g)			-	-
²⁴² Pu	(Bq/g)			-	-
²⁴⁴ Pu	(Bq/g)			-	-
(²³⁹ Pu) ²³² Tn/ ²³⁹ Pu	(ng/g)			1014	-
(a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., (l) Instrument detection limits, and (m) the ratio reported for year 2000 assumes all alpha activity is from ²³⁹ Pu.					

Table 8 Analytical Data for Sludge in Tanks W-30

Characteristic (Analysis)	1996 W-30 S	2000 W-30 S	2001 w-30 s	IDL'
Physical properties and miscellaneous data				
Request number	No Data	No Data	12057	
Sample number			010716-017	
pH			10.7	
Water ^a (%)			55.0	
TS ^b (mg/g)			450	
TSS ^c (mg/g)				
TDS ^d (mg/g)				
Bulk density (g/mL)			1.411	
TC ^e (mg/Kg)			13600	15
TIC ^e (mg/Kg)			5200	15
TOC ^e (mg/Kg)			8500	15
RCRA Metals (±10%)				
Ag ^h (100) (mg/Kg)			-	0.006
As (100) (mg/Kg)			< 2	0.011
Ba (2000) (mg/Kg)			119	0.001
Cd (20) (mg/Kg)			15.2	0.111
Cr (100) (mg/Kg)			189	0.008
Hg (4) (mg/Kg)			67.3	0.011
Ni (1000) (mg/Kg)			66.5	0.065
Pb (100) (mg/Kg)			915	0.341
Se (20) (mg/Kg)			< 0.9	0.005
Tl (18) (mg/Kg)			< 5	0.005
Process metals (±10%)				
Al (mg/Kg)			5900	0.048
B (mg/Kg)			4.24	0.024
Be (mg/Kg)			7.00	0.001
Ca (mg/Kg)			43800	0.017
Co (mg/Kg)			33.6	0.060
Cu (mg/Kg)			52.9	0.006
Cs ^j (mg/Kg)			< 5	0.005
Fe (mg/Kg)			2740	0.233
K (mg/Kg)			10400	0.178
Mg (mg/Kg)			6670	0.053
Mn (mg/Kg)			277	0.001
Mo (mg/Kg)			63.9	0.030
Na (mg/Kg)			48000	0.065
P (mg/Kg)				0.13
Sb (mg/Kg)			< 5	0.509
Si ^k (mg/Kg)			10000	0.022
Sr (mg/Kg)			189	0.001
Th (mg/Kg)			5740	0.181
Ti (mg/Kg)			528	0.010
U (mg/Kg)			26300	0.105
V (mg/Kg)			< 1.2	0.013
Zn (mg/Kg)			262	0.390
Zr (mg/Kg)			478	0.008

Characteristic (Analysis)	1996 W-30 S	2000 W-30 S	2001 W-30 S	IDL'
Semi-quantitative metals by ICP-MS ($\pm 30-50\%$, * indicates data from water leach)				
Au, gold (mg/Kg)			-	
Bi, bismuth (mg/Kg)			2.60	
Ce, cerium (mg/Kg)				
Er, erbium (mg/Kg)				
Eu, europium (mg/Kg)				-
Ga, gallium (mg/Kg)			< 5	
Gd, gadolinium (mg/Kg)				
Ho, holmium (mg/Kg)				
I, iodine (mg/Kg)				
La, lanthanum (mg/Kg)			155	-
Li, lithium (mg/Kg)			148	
Mo, molybdenum (mg/Kg)				
Nb, niobium (mg/Kg)			775	
Rb, rubidium (mg/Kg)				
Sr, strontium (mg/Kg)				
Ti, titanium (mg/Kg)				
W, tungsten (mg/Kg)				
Zr, zirconium (mg/Kg)				
Anions by ion chromatography in water wash of sludge ($\pm 10\%$)				
<u>Inorganic</u>				
Bromide (mg/Kg)			135	0.05
Chloride (mg/Kg)			2350	0.05
Chromate (mg/Kg)			21	0.05
Fluoride (mg/Kg)			199	0.05
Nitrate (mg/Kg)			159000	0.10
Nitrite (mg/Kg)			5620	0.10
Phosphate (mg/Kg)			< 5	0.20
Sulphate (mg/Kg)			2210	0.10
<u>Organic</u>				
Acetate (mg/Kg)			531	0.05
Citrate (mg/Kg)			190	0.50
Formate (mg/Kg)			118	0.05
Oxalate (mg/Kg)			973	0.05
Phthalate (mg/Kg)			< 3	0.05

Characteristic Analysis)		1996 W-30 S	2000 W-30 S	2001 W-30 S	IDL ¹
Beta/gamma emitters (±10%)					
<u>Gross beta</u>	(Bq/g)			6.5e+06	
⁶³ Ni	(Bq/g)			-	-
⁶⁵ Ni	(Bq/g)				
⁶⁰ Co	(Bq/g)			1.6e+04	
⁹⁰ Sr/ ⁹⁰ Y	(Bq/g)			1.9e+06	
⁹⁹ Tc	(Bq/g)			5.8e+02	
¹²⁹ I	(Bq/g)			-	
¹³⁴ Cs	(Bq/g)			1.9e+04	-
¹³⁷ Cs	(Bq/g)			1.9e+06	
¹⁵¹ Sm	(Bq/g)			-	
¹⁵² Eu	(Bq/g)			7.4e+04	
¹⁵⁴ Eu	(Bq/g)			2.5e+04	-
¹⁵⁵ Eu	(Bq/g)			<3.8e+03	
²²⁷ Ac	(Bq/g)			-	
²⁴¹ Pu	(Bq/g)			-	
Alpha emitters (±10%)					
<u>Gross alpha</u>	(Bq/g)			55000	-
²³² Th	(Bq/g)			23	-
²³³ U	(Bq/g)			1940	-
²³⁴ U	(Bq/g)			301	-
²³⁵ U	(Bq/g)			10.9	-
²³⁸ U	(Bq/g)			325	
²³⁷ Np	(Bq/g)			-	
²⁴¹ Am	(Bq/g)			5500	
²⁴⁴ Cm	(Bq/g)			34000	
²⁵⁰ Cf	(Bq/g)				
²⁵² Cf	(Bq/g)			-	
<u>Total Pu alpha</u>	(Bq/g)			12000	
²³⁸ Pu	(Bq/g)			6200	
²³⁹ Pu/ ²⁴⁰ Pu	(Bq/g)			6100	
²⁴² Pu	(Bq/g)				-
<u>TRU activity</u>					
Pu+Am (3700)	(Bq/g)			17500	
Uranium isotopics by ICP-MS (±2%)					
²³³ U	(atom %)			0.0211	0.001
²³⁴ U	(atom %)			0.005 1	0.001
²³⁵ U	(atom %)			0.5239	0.001
²³⁶ U	(atom %)			0.0018	0.001
²³⁸ U	(atom %)			99.4482	0.00 1
²³³ U/MS	(mg/Kg)			5.44	
²³⁵ U/MS	(mg/Kg)			136	
²³⁸ U/ ²³⁵ U FEM	-			182	

Characteristic (Analysis)	1996 W-30 S	2000 W-30 S	2001 W-30 S	IDL ¹
Plutonium isotopics by TIMS (±1%)				
²³⁸ Pu (atom %)				
²³⁹ Pu (atom %)				
²⁴⁰ Pu (atom %)				
²⁴¹ Pu (atom %)				
²⁴² Pu (atom %)				
²⁴⁴ Pu (atom %)				
Pu activity				
²³⁸ Pu (Bq/g)			-	
²³⁹ Pu (Bq/g)			-	
²⁴⁰ Pu (Bq/g)			-	
²⁴¹ Pu (Bq/g)			-	
²⁴² Pu (Bq/g)			-	
²⁴⁴ Pu (Bq/g)			-	
(²³⁹ Pu) ²³² Th/ ²³⁹ Pu (ng/g)			-	
			1098	
(a) Free water content of sludge, (b) Total solids, (c) Total suspended solids, (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrochloric acid prep., (i) RCRA regulatory limits, (j) measured by ICP-MS or GFAA, (k) nitric-hydrofluoric acid prep., (l) Instrument detection limits, and (m) the ratio reported for year 2000 assumes all alpha activity is from ²³⁹ Pu.				

Table 9 Analytical-Data for Sludge in Tanks W-31

Characteristic (Analysis)	1996 w-31 s	2000 w-31 s	2001 w-31 s	2001 W-31 H	IDL'
Physical properties and miscellaneous data					
Request number	7749A	10224	12057	12057	
Sample number	960717-023	000509-006	010716-018	010716-019	
pH	9.9	9.7	9.3	9.3	
Water ^a (%)	51.4	72.5	58.5	64.3	
TS ^b (mg/g)	486	275	415	357	
TSS ^c (mg/g)		196			
TDS ^d (mg/g)		79.2			
Bulk density (g/mL)	1.44	1.210	1.331	1.283	
TC ^e (mg/Kg)	10200	5690	14000	11000	15
TIC ^e (mg/Kg)	5300	< 1000	9200	8700	15
TOC ^g (mg/Kg)	4900	5690	4800	2200	15
RCRA Metals (±10%)					
Ag ^h (100) ⁱ (mg/Kg)	< 1.9				0.006
As (100) (mg/Kg)	< 5.0	< 0.9	< 1.3	< 2.3	0.011
Ba (2000) (mg/Kg)	124	108	100	119	0.001
Cd (20) (mg/Kg)	9.03	< 15	148	< 12	0.111
Cr (100) (mg/Kg)	130	237	182	325	0.008
Hg (4) (mg/Kg)	70.7	65.4	70.2	76.0	0.011
Ni (1000) (mg/Kg)	104	70.3	89.1	74.0	0.065
Pb (100) (mg/Kg)	764	717	933	1450	0.341
Se (20) (mg/Kg)	< 5.0	< 0.9	< 0.6	< 1	0.005
Tl (18) (mg/Kg)	< 5.0	< 0.9	< 3	< 5	0.005
Process metals (±10%)					
Al (mg/Kg)	12700	7920	8700	7830	0.048
B (mg/Kg)	11.6	7.25	5.55	< 2.6	0.024
Be (mg/Kg)	21.0	8.91	15.6	6.79	0.001
Ca (mg/Kg)	24100	24700	43000	52100	0.017
Co (mg/Kg)	4.76	14.1	30.3	36.1	0.060
Cu (mg/Kg)	80.2	64.3	83.8	70.7	0.006
Cs ^j (mg/Kg)	0.543	0.395	< 3	< 5	0.005
Fe (mg/Kg)	2820	3420	2760	3960	0.233
K (mg/Kg)	8320	4570	7330	5660	0.178
Mg (mg/Kg)	2170	2090	4690	2330	0.053
Mn (mg/Kg)	247	268	270	262	0.001
MO (mg/Kg)		46.7	110	53.1	0.030
Na (mg/Kg)	60600	33700	46100	34900	0.065
P (mg/Kg)	4240				0.13
Sb (mg/Kg)	< 19	< 45	< 3	< 5	0.509
Si ^k (mg/Kg)	10200	7930	7940	7240	0.022
Sr (mg/Kg)	174	88.3	198	351	0.001
Th (mg/Kg)	20700	8350	13300	5470	0.181
Ti (mg/Kg)			22.3	26.0	0.010
U (mg/Kg)	19800	38600	24200	34000	0.105
V (mg/Kg)	7.18	< 1.1	< 1	< 1.4	0.013
Zn (mg/Kg)	125	92.2	187	129	0.390
Zr (mg/Kg)			437	202	0.008

Characteristic (Analysis)	1996 w-31 s	2000 w-31 s	2001 w-31 s	2001 W-31 H	IDL'
Semi-quantitative metals by ICP-MS (±30-50 %, * indicates data from water leach)					
Au, gold (mg/Kg)	2.6				
Bi, bismuth (mg/Kg)	1200				
Ce, cerium (mg/Kg)	20		278	40.3	
Er, erbium (mg/Kg)	0.85				
Eu, europium (mg/Kg)	0.54				
Ga, gallium (mg/Kg)	12				
Gd, gadolinium (mg/Kg)	0.75		< 4	< 5.3	
Ho, holmium (mg/Kg)	0.22				
I, iodine (mg/Kg)	* 20				
La, lanthanum (mg/Kg)	54		146	272	
Li, lithium (mg/Kg)	* 81		411	178	
Mo, molybdenum (mg/Kg)	* 1.4				
Nb, niobium (mg/Kg)	2.0				
Rb, rubidium (mg/Kg)	* 1.3				
Sn, tin (mg/Kg)	40		< 3	233	
Ti, titanium (mg/Kg)	34				
W, tungsten (mg/Kg)	1.3				
Zr, zirconium (mg/Kg)	51				
Anions by ion chromatography in water wash of sludge (±10%)					
<u>Inorganic</u>					
Bromide (mg/Kg)	< 50	< 50	78.9	63.8	0.05
Chloride (mg/Kg)	2570	817	1610	1220	0.05
Chromate (mg/Kg)	51.5	21.9	46.8	85.3	0.05
Fluoride (mg/Kg)	125	604	370	920	0.05
Nitrate (mg/Kg)	197000	51900	148000	89800	0.10
Nitrite (mg/Kg)	3470	2680	4710	4140	0.10
Phosphate (mg/Kg)	< 50	169	< 10	103	0.20
Sulphate (mg/Kg)	1090	919	1240	1140	0.10
<u>Organic</u>					
Acetate (mg/Kg)	237	< 50	149	191	0.05
Citrate (mg/Kg)	< 50	< 5	15.1	< 5	0.50
Formate (mg/Kg)	251	< 50	93.4	15.3	0.05
Oxalate (mg/Kg)	89.8	107	261	162	0.05
Phthalate (mg/Kg)	< 50	< 5	< 5	< 5	0.05

Characteristic (Analysis)		1996 w-31 s	2000 w-31 s	2001 w-31 s	2001 W-31 H	IDL'
Beta/gamma emitters (±10%)						
<u>Gross t s</u>	(Bq/g)	2.4e+07	6.4e+06	1.4e+07	7.6e+06	
⁵⁹ Ni	(Bq/g)	< 3.3e+01				
⁶³ Ni	(Bq/g)	4.4e+03				
⁶⁰ Co	(Bq/g)	2.2e+04	< 6.4e+03	9.2e+03	2.3e+03	
⁹⁰ Sr/ ⁹⁰ Y	(Bq/g)	1.1e+07	2.6e+06	6.3e+06	3.2e+06	
⁹⁹ Tc	(Bq/g)	1.4e+02		7.3e+02	< 3.3e+02	
¹²⁹ I	(Bq/g)	4.5e-02				
¹³⁴ Cs	(Bq/g)	2.5e+03		9.8e+02	< 1.6e+03	
¹³⁷ Cs	(Bq/g)	4.3e+05	5.7e+05	4.6e+05	6.4e+05	
¹⁵¹ Sm	(Bq/g)	< 6.0e+02				
¹⁵² Eu	(Bq/g)	3.0e+04	< 3.0e+04	4.9e+04	1.0e+04	
¹⁵⁴ Eu	(Bq/g)	2.0e+04	< 1.9e+04	1.9e+04	< 3.6e+03	-
¹⁵⁵ Eu	(Bq/g)	< 3.4e+03	< 1.1e+04	1.0e+04	< 3.0e+03	
²²⁷ Ac	(Bq/g)	< 5.8e+03				
²⁴¹ Pu	(Bq/g)	2.4e+04				
Alpha emitters (±10%)						
<u>Gross alpha</u>	(Bq/g)	160000	43000	92000	33000	
²³² Th	(Bq/g)	84	34	54	22	
²³³ U	(Bq/g)	5200	1970	2100	12	
²³⁴ U	(Bq/g)	310	860	200	180	
²³⁵ U	(Bq/g)	10	17	12	17	
²³⁸ U	(Bq/g)	244	480	300	420	
²³⁷ Np	(Bq/g)	21				
²⁴¹ Am	(Bq/g)	14000	2900	7900	3200	
²⁴⁴ Cm	(Bq/g)	110000	29000	65000	19000	
²⁵⁰ Cf	(Bq/g)	< 100				
²⁵² Cf	(Bq/g)	< 100				
<u>Total Pu alpha</u>	(Bq/g)	19000	9300	16000	8600	
²³⁸ Pu	(Bq/g)	13000	5200	9700	4200	
²³⁹ Pu/ ²⁴⁰ Pu	(Bq/g)	6200	4100	5800	4300	
²⁴² Pu	(Bq/g)					
<u>TRU activity</u>						
Pu+Am (3700)	(Bq/g)	33000	12200	23900	11800	
Uranium isotopics by ICP-MS (±2%)						
²³³ U	(atom %)	0.075	0.0146	0.0250	0.0001	0.001
²³⁴ U	(atom %)	0.007	0.0098	0.0036	0.0023	0.001
²³⁵ U	(atom %)	0.750	0.6333	0.6460	0.6471	0.001
²³⁶ U	(atom %)	0.004	< 0.0001	0.0000	0.0000	0.001
²³⁸ U	(atom %)	99.165	99.3423	99.3254	99.3504	0.001
²³³ U/MS	(mg/Kg)	14.5	5.52	5.92	co.1	
²³⁵ U/MS	(mg/Kg)	150	241	154	217	
²³⁸ U/ ²³⁵ U FEM	-	118	154	148	155	

Characteristic (Analysis)	1996 w-31 s	2000 w-31 s	2001 w-31 s	2001 W-31 H	IDL ¹
Plutonium isotopics by TIMS (±1%)					
²³⁸ Pu (atom%)	< 1.16		-		
²³⁹ Pu (atom%)	81.94			-	
²⁴⁰ Pu (atom%)	14.55			-	
²⁴¹ Pu (atom%)	0.34				
²⁴² Pu (atom%)	1.9				
²⁴⁴ Pu (atom%)	0.11		-	-	-
Pu activity					
²³⁸ Pu (Bq/g)	13000				
²³⁹ Pu (Bq/g)	3400				
²⁴⁰ Pu (Bq/g)	2200				
²⁴¹ Pu (Bq/g)	24000				
²⁴² Pu (Bq/g)	5.1				
²⁴⁴ Pu (Bq/g)	< 0.1		-		
(²³⁹ Pu) (ng/g)	1820	-			
²³² Th/ ²³⁹ Pu	13800		1908	1460	
(a) Free water content of sludge, (b) Total solids, (c) Total suspended solids (d) Total dissolved solids, (e) Total carbon, (f) Total inorganic carbon, (g) Total organic carbon, (h) nitric-hydrofluoric acid prep, (i) RCRA regulatory limits, (j) measured by I#-MS or &AA (k) nitric-hydrofluoric acid prep., (l) Instrument detection limits, and (m) the ratio reported for year 2000 assays all alpha activity is from ²³⁹ Pu.					

5.2 Discussion of MVST Sludge Characteristics

Determination of the mass and charge balance for a sludge sample typically has a larger error bar than what is observed with a supernatant sample. The assumptions required about the chemical form and the oxidation state of the species present in the sludge are not well-known, and many of the compounds in the sludge are mixed oxides which are not directly measured. The sludge is actually a slurry with a high water content. The interstitial liquid is in close contact with the sludge, and there are many ionic solubility equilibria. The anion data for the sludge samples discussed in this report are based on the water soluble anions that would be available to a water wash. The water wash does not account for the insoluble hydroxides, carbonates, and mixed oxides present in a sludge sample. The insoluble species do not contribute to the charge balance, and the cation charge is not used in the calculation, as indicated in Table 10. Most of the nitrate reported for the sludge is due to the interstitial liquid. Considering these limitations, the compounds listed in Table 10 were used to estimate the mass and charge balance.

Table 10 Assumption Used for Major Compounds in MVST Sludge

Cation	Chemical Form	Cation Charge Used	Gravimetric Factors
Al ³⁺	Al ₂ O ₃	0	1.890
Ca ²⁺	CaCO ₃	0	2.497
Fe ³⁺	Fe ₂ O ₃	0	1.430
K ⁺	K ⁺ NO ₃ ⁻	+1	2.586
Mg ²⁺	Mg(OH) ₂	0	2.399
Mn ²⁺	Mn(OH) ₂	0	1.619
Na ⁺	Na ⁺ NO ₃ ⁻	+1	3.697
Th ⁴⁺	Th(OH) ₄	0	1.293
UO ₂ ²⁺	UO ₂ ((OH) ₂ ·H ₂ O)	0	1.353

Table 11 summarizes the mass and charge balance for the MVST tank sludge samples. Considering the limitations of these calculations, the mass balance is within the analytical error ($\pm 20\%$) for these sludge samples. The charge balance is more influenced by the chemical form assumptions, and the results have a larger corresponding error range.

Table 11 Summary of Quality Checks for MVST Sludge Data

Tank	Mass Balance ($TS_{calc.}/TS_{meas.}$)	Charge Balance (M^+/A^-)	pH	$^{134}Cs+^{137}Cs$ (%)	$^{90}Sr/^{90}Y$ (%)	Beta Recovery (%)
W-24 S	0.97	0.92	10.0	18.9	64.0	95.6
W-25 S	0.98	0.89	11.8	7.5	84.4	94.3
W-26 S	0.98	0.83	9.4	17.9	63.3	94.9
W-27 S	0.95	0.28	8.8	15.1	82.2	97.6
W-27 H	0.99	2.66	11.7	26.8	71.1	92.6
W-28 S	0.99	1.07	8.7	7.7	88.2	86.0
w-29 s	0.98	0.97	12.0	18.1	75.0	92.5
w-30 s	0.91	0.79	10.7	36.0	61.8	95.5
w-31 s	0.95	0.76	9.3	4.1	95.2	95.2
W-31 H	1.03	0.86	9.3	10.4	89.4	94.6

The beta recovery results are listed in Table 11. As discussed before, the variability for the beta recovery is probably due to the analytical error on the ^{90}Sr measurement. Any measurement error for the ^{90}Sr activity would be doubled when considering the beta recovery calculation.

The distribution, by weight percent, of the major compounds from Table 8 are illustrated in Fig. 1 for each MVST sludge sample. The distribution of the total uranium and thorium concentrations in the MVST sludge samples are shown in Fig. 2, and the change in uranium concentration between sludge sample collected in 1996 and 2000 is shown in Fig. 3.

Figure 1 Distribution of Major Compounds in the MVST Sludge

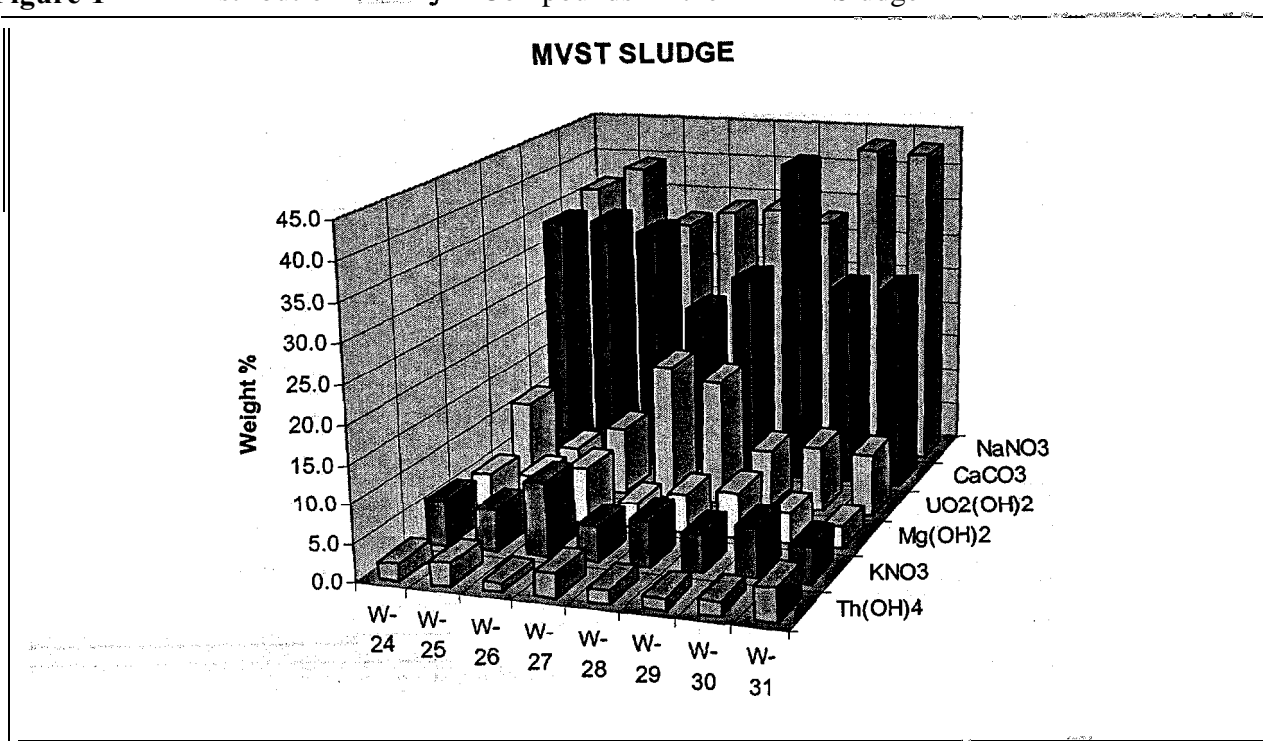


Figure 2 Distribution of Uranium and Thorium in the MVST Sludge

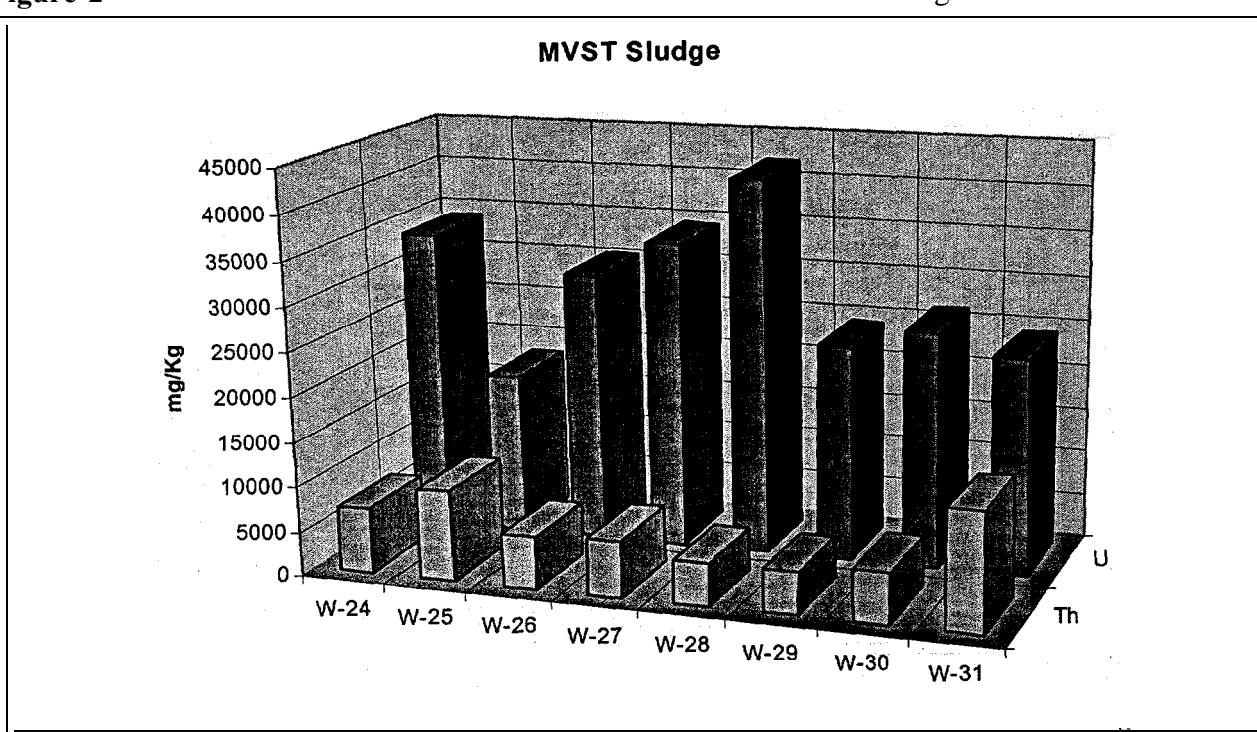
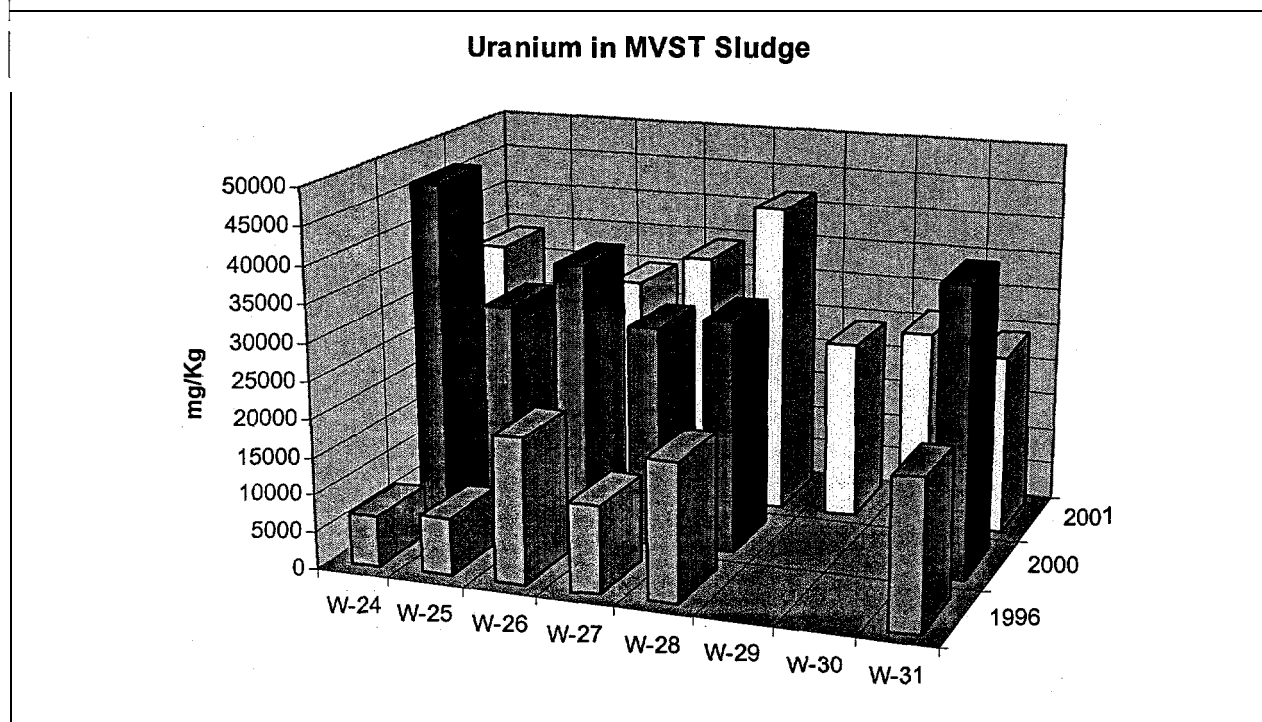


Figure 3 Distribution of Uranium in the MVST Sludge in 1996, 2000, and 2001



The distribution of the major beta emitters found in the MVST sludge samples are summarized in Table 12. The distributions of the beta activity are shown to be dependent upon the radionuclides present, which is a function of the age of the radioactive waste, and the pH of the supernatant found over the sludge. Under the typical basic conditions for ORNL waste tanks, the major difference in the beta distribution between the supernatant and the sludge is that the distribution of the longer lived fission products (^{90}Sr and ^{137}Cs) are reversed due to the differences in solubility. The Group IA metals (^{134}Cs and ^{137}Cs) and the radionuclides that form anionic species ($^{99}\text{TcO}_4^-$, ^{129}I , and $^{129}\text{IO}_3^-$) are more soluble in the supernatant. The solubility of the Group IIA metals (^{90}Sr) in the supernatant are a function of both pH and carbonate concentration. At high pH most of the other metals, lanthanides, and actinide elements form insoluble hydroxides and mixed oxides, which are found in the sludge. The ^{99}Tc activity is higher in the supernatant than the sludge. The source of most of the ^{99}Tc found in the sludge samples was the interstitial liquid, and not insoluble forms of technetium. The shorter lived radionuclides observed include the europium (^{152}Eu , ^{154}Eu , and ^{155}Eu) isotopes and to some extent ^{134}Cs .

Table 12 Distribution of Beta Activity in the MVST Sludge

Tank	pH	Percent of Total Beta Activity				
		⁹⁰ Sr/ ⁹⁰ Y (%)	¹³⁴ Cs+ ¹³⁷ Cs (%)	⁶⁰ Co (%)	⁹⁹ Tc (%)	^{152,154,155} Eu (%)
W-24 s	10.0	64.0	18.9	0.6	0.02	15.6
w-25 s	11.8	84.4	7.5	0.4	0.02	7.6
W-26 S	9.4	63.3	17.9	0.8	0.03	17.9
W-27 S	8.8	82.2	15.1	0.1	0.00	2.5
W-27 H	11.7	71.1	26.8	0.3	0.02	1.6
W-28 S	8.7	88.2	7.7	0.1	0.00	3.7
w-29 s	12.0	75.0	18.1	0.7	0.02	5.7
w-30 s	10.7	61.8	36.0	0.3	0.01	1.5
w-31 s	9.3	95.2	4.1	0.1	0.01	0.6
W-31 H	9.3	89.4	10.4	0.0	0.00	0.1

Table 13 Summary of Actinide Elements in MVST Sludge

Actinide	W-24S	W-25S	W-26S	W-27S	W-27H	W-28S	W-29S	w-30s	w-31s	W-31H
	(% α activity)									
²³² Th	0.02	0.04	0.03	0.04	0.04	0.03	0.03	0.04	0.06	0.07
²³³ U	4.34	3.55	12.14	2.20	1.67	2.94	3.64	3.56	2.32	0.04
²³⁴ U	0.12	0.09	0.32	0.55	0.00	0.60	0.05	0.56	0.22	0.56
²³⁵ U	0.01	0.00	0.01	0.02	0.00	0.03	0.01	0.02	0.01	0.05
²³⁸ U	0.28	0.20	0.56	0.69	0.16	0.90	0.47	0.60	0.33	1.34
²³⁷ Np										
²³⁸ Pu	8.08	11.95	12.36	9.73	10.53	12.92	10.43	11.40	10.65	13.40
²³⁹⁺²⁴⁰ Pu	6.33	8.27	7.59	7.95	7.58	10.65	7.11	11.21	6.37	13.72
²⁴¹ Am ^a	10.10	7.90	7.44	10.70	16.85	10.82	11.85	10.11	8.67	10.21
²⁴⁴ Cm	70.71	68.00	59.55	68.11	63.18	61.10	66.39	62.50	71.37	60.61
Gα	150000	110000	68000	62000	24000	58000	64000	55000	92000	33000

^a The “Am data is based on subtracting the ²³⁸Pu by ICP-MS from the alpha peak measured at 5.15 MeV (²³⁸Pu + ²⁴¹Am) in the alpha spectrum.

The distribution of the alpha activity is summarized in Table 13, which includes the percent alpha for each MVST sludge sample. In general, the alpha activity in the MVST system is strongly weighted by the ^{244}Cm , which has a high specific activity. The list of actinides in Table 13 required several radiochemical and inorganic analytical measurements to generate the best estimates for each of the alpha activities. The ^{232}Th activity is calculated from the total thorium measured by ICP-AES. The other thorium isotopes (^{228}Th , ^{229}Th , and ^{230}Th) are present in the ORNL sludge waste at such low mass, their presence would not effect the ICP-AES measurement. The uranium isotopes are measured by ICP-MS. The atom % results are converted to weight %, which is used to calculate the concentration of each uranium isotope **from** the total uranium results obtained by ICP-AES. The activity for each uranium radionuclide is then calculated **from** the specific activity for each isotope. The plutonium isotopes are first measured by ICP-MS, and the total plutonium alpha activity, measured after a chemical separation, is, used to calculate the activity for each isotope. The ^{244}Cm was measured directly by alpha spectrometry without any chemical separation. The ^{241}Am activity is determined by subtracting the ^{238}Pu activity from the sum of the $^{238}\text{Pu} + ^{241}\text{Am}$ measured by alpha spectrometry. Both ^{238}Pu and ^{241}Am have an alpha energy of about 5.50 MeV and can not be resolved by alpha spectrometry. There was no chemical separation of the plutonium and americium for this project because of cost concerns.

5.4 RCRA Characteristics for the MVST Svsstem

The RCRA regulatory limits are listed in Table 14, which also includes the limits for the EPA Toxicity Characteristic Leaching Protocol (TCLP) extract and the functional total metal limits for a solid or sludge waste. The total metal limits are a factor of twenty times higher than the TCLP extraction limits and are based on the 1:20 dilution used for the TCLP extraction procedure.

Table 14 Summary of RCRA Regulatory Limits

Metals	TCLP Extract and Liquids (mg/L)	Solid/Sludge Total Metal (mg/Kg)
Silver (Ag)	5	100
Arsenic (As)	5	100
Barium (Ba)	100	2000
Cadmium (Cd)	1	20
Chromium (Cr)	5	100
Mercury (Hg)	0.2	4
Nickel (Ni)	50	1000
Lead (Pb)	5	100
Selenium (Se)	1	20
Thallium (Tl)	0.9	18

If the RCRA metal concentrations are found to be below the total metal limits, the solid waste can not fail the TCLP leach test. If the RCRA metal concentrations exceed the total metal limits, the TCLP leach test must be done to determine if the solid waste is hazardous. For solid samples, the TCLP leach test is only valid for the final waste form ready for disposal. The total metal concentration data can be used as acceptable process knowledge if the final waste form only results in a dilution of the RCRA metal concentrations. Examples of waste forms that result in a dilution of a solid waste includes grouting (2 fold dilution) and vitrification (3 fold dilution). If the total metal limit is exceeded after stabilizing the waste, the TCLP leach test would be required for only the metals that had the potential to exceed the regulatory limits.

All of the MVST tank sludge samples exceed the total metal limits for lead and mercury, and two tanks are over or near the limit for chromium.. Most of the ORNL radioactive waste sludge samples, characterized to date, have exceeded the total metal limits for these three RCRA metals. Based on past experience, it is expected that solidification of the ORNL MVST sludge would fix these RCRA metals such that the final waste form would pass the TCLP leach test.

5.5 TRU Classifications for LLLW System

The DOE definition for Transuranic (TRU) Waste includes the following conditions,

- TRU activity ≥ 3700 Bq/g (100 nCi/g),
- TRU isotopes must be alpha emitting actinide with $Z > 92$ (uranium),
- TRU isotopes must have a half life ≥ 20 years.

This definition excludes all thorium and uranium isotopes. The short lived actinide ^{244}Cm ($t_{1/2} = 18.1$ years), which is common to ORNL waste, falls outside the TRU definition. Also, the plutonium isotope, ^{241}Pu , would be excluded from calculation of the TRU activity because it is a pure beta emitter. The primary actinide elements common to ORNL waste, that are present at sufficient levels to meet the TRU definition, include ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Am . There is some current work at the Radiochemical Engineering Development Center (Mark-42 fuel assembly processing) that could generate enough ^{243}Am to make a significant contribution to TRU alpha content of the waste. The remaining actinide elements present in ORNL waste are generally not available at high enough activity, and/or do not have a long enough half-life to meet the TRU definition.

None of the MVST supernatant samples which were analyzed in previous sampling campaigns had enough alpha activity to be considered as TRU waste. All of the MVST sludge that has been characterized to date has been classified as TRU waste based on only the plutonium and americium activity. The alpha activity reported is based on wet weight, if adjusted for dry weight the activity would almost double. The MVST sludge samples contained enough plutonium and americium activity to easily satisfy the WIPP waste acceptance criteria* for transuranic waste. Based on the TRU activity, any dilution of the sludge that would result from a solidification process such as grouting or vitrification would most likely not effect the TRU classification.

5.6 Distribution of Fissile Material in LLLW System

As discussed in section 3.5, the ORNL LLLW waste acceptance criteria (WAC) requires the fissile isotopes of uranium and plutonium to be diluted with ^{238}U and ^{232}Th , respectively. A summary of the dilution ratios for fissile material in the sludge samples is provided in Table 15. All the dilution ratios for the MVST sludge samples exceed the required dilution factors for the fissile isotopes of

uranium and plutonium. All the dilution ratios listed in Table 15 are based on equations discussed in section 3.5 of this report.

Table 15 Summary of Denature Ratios for MVST Sludge

Tank	$^{238}\text{U}/^{235}\text{U} f_{35}$ (> 110) ^a	$^{238}\text{U}/^{233}\text{U}$ (> 200) ^b	$^{232}\text{Th}/(^{239}\text{Pu}+^{240}\text{Pu})$ (> 200)
W-24	206	1017	7 6 1
w - 2 5	203	906	1054
W-26	194	718	765
W-27 S	155	2730	1336
W-27-H	255	1680	1201
W-28	160	2900	851
w-29	185	1640	1014
w-30	182	2060	1098
w-31 s	148	1190	1908
W-31 H	155	284000	1460

^a Calculation based on equation 1 in Section 3.5. ^b Calculation based on equation 2 in Section 3.5., ^c Conservative estimate of ^{239}Pu dilution since ^{240}Pu is included.

The dilution ratios listed in Tables 15 are based on the ratio of weight %, not the ratio of atom % given in the data tables. There is a small difference between atom %, reported for the uranium and plutonium, and weight %, which is needed for many calculations performed with the analytical data. To convert from atom % to weight %, we used the following equation,

$$W_i = \frac{a_i M_i}{\sum_i^n a_i M_i} \times 100\%$$

where, W_i = weight %,
 M_i = nuclidic mass
 a_i = atom %.

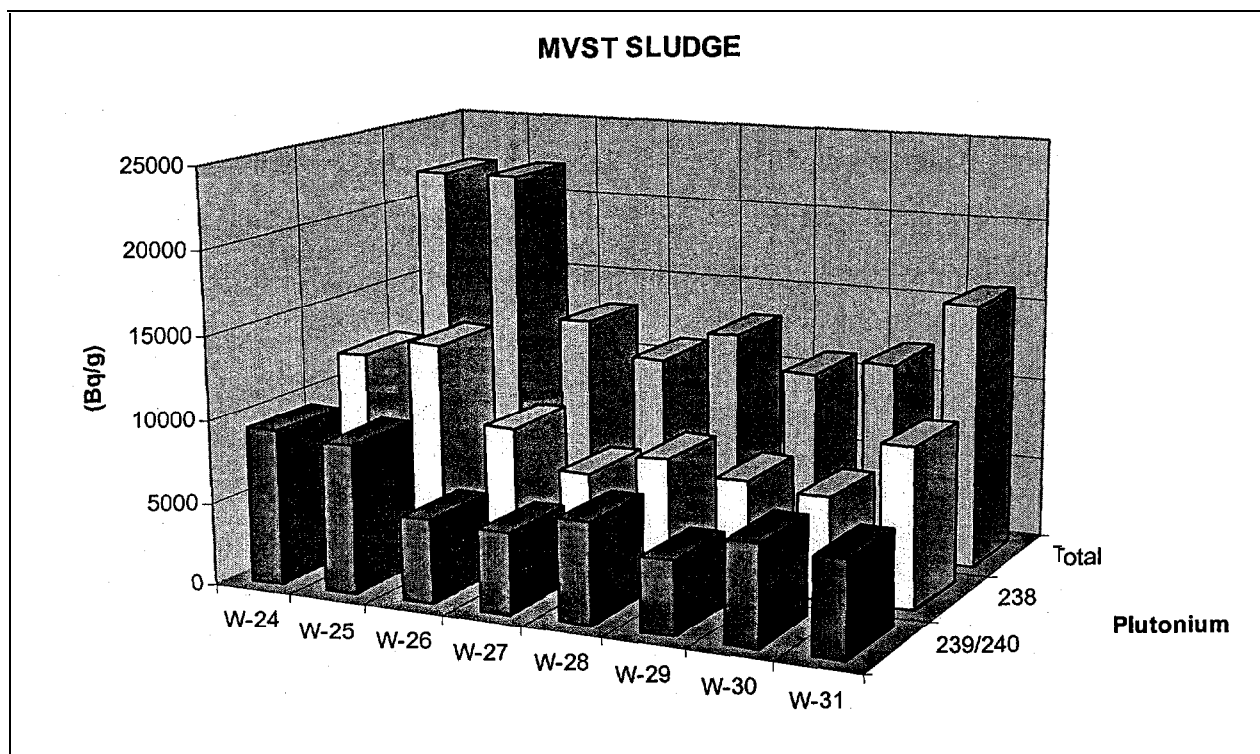
An example of this calculation is provided in Table 16, which shows there is not much difference between the atom % and the weight %.

Table 16 Example of Converting Atom % to Weight % for W-31 Sludge

Isotope	Nuclidic mass (g/mol)	atom %	($a_i M_i$)	weight %
^{233}U	233.039629	0.056	13.0502	0.0548
^{234}U	234.040947	0.004	0.9362	0.0039
^{235}U	235.043924	0.621	145.9623	0.6132
^{236}U	236.045563	0.002	0.4721	0.0020
^{238}U	238.050785	99.316	23642.2518	99.3260
Total		99.999	23802.6726	99.9999

The distribution of plutonium isotopes by alpha activity are illustrated in Fig. 4 for each of the MVST samples. One should note that the ^{238}Pu dominates the alpha activity and the ^{239}Pu is the major isotope by weight or concentration.

Figure 4 Distribution of Plutonium Alpha Activity in the MVST Sludge

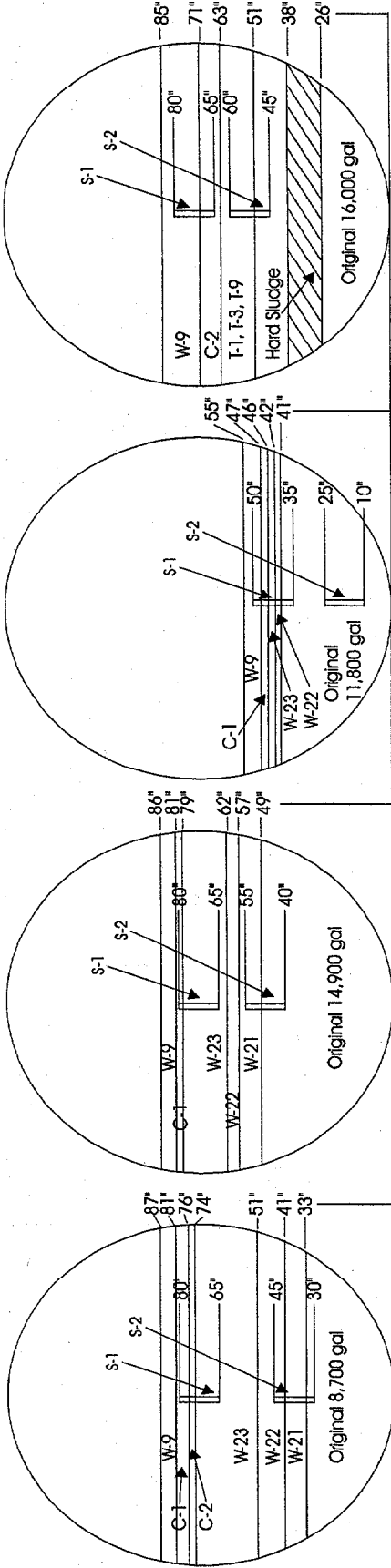


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APPENDIX A

The following diagram graphically illustrates where sludge samples were collected **from each MVST** tank during the 2000 sampling campaign. Several of the other **ORNL** waste tanks systems were transferred to the MVST system prior to the sludge sample being collected. This diagram also provides an estimate of which MVST tanks collected these transfers. There were only minor changes in the contents of these tanks prior to this sampling campaign. The sludge samples for this campaign consisted of a full core from the top to the bottom of the sludge layers.



Top of G3 Port to Bottom of Tank = 258"

W-24

Top of G3 Port to Bottom of Tank = 258"

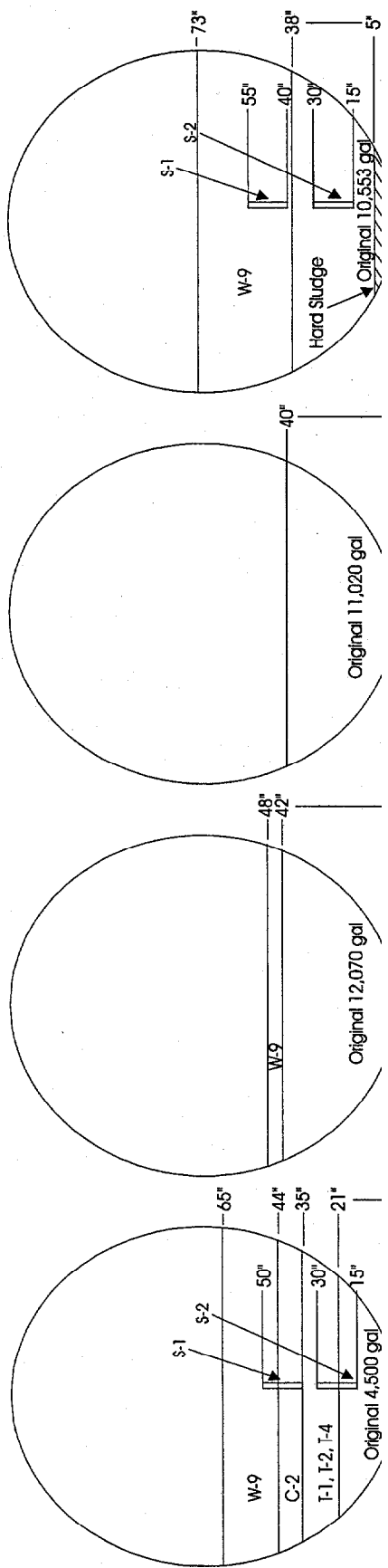
W-25

Top of G3 Port to Bottom of Tank = 255"

W-26

Top of G3 Port to Bottom of Tank = 257"

W-27



Top of G3 Port to Bottom of Tank = 256"

W-28

Top of G3 Port to Bottom of Tank = 258"

W-29

Top of G3 Port to Bottom of Tank = 248"

W-30

Top of G3 Port to Bottom of Tank = 248"

W-31

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APPENDIX B

Radioactive Materials Analytical Laboratory QC Acceptance Criteria for Radioactive Liquid/Solid Waste Samples

Analysis	Method (s) CASD-AM-	Quality Control Check (per batch)	SW-846 Acceptance Criteria (%D, %R, RPD)	RMAL Acceptance Criteria (%D, %R, RPD)
Metals by ICP-AES (inductively coupled plasma atomic emission spectroscopy)	SW846-6010A	high standard calibration verifications (ICV & CCV) ^a calibration blank & checks (ICB & CCB) ^b method blank (sample prep) matrix spike matrix spike duplicate or sample duplicate laboratory control sample (sample prep) serial dilution (if interference suspected) post digestion spike ^d	±5%D ±10%D <3 x IDL <3 x IDL ±20%D ±20 RPD none specified ±10%R ±20%D	±5%D ±10%D <3 x IDL <3 x IDL ±25%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid) ±20%D ±10%R ±25%D (liq.), ±30%D (solid)
Metals by ICP-MS (inductively coupled plasma- mass spectrometry)	SW846-6020	calibration verifications (ICV & CCV) calibration blank & blank checks (CCB) ^b method blank (sample prep) ^c matrix spike matrix spike duplicate or sample duplicate laboratory control sample (sample prep) ^c internal standard post digestion spike ^d	±10%D <3 x IDL none specified none specified ±20 RPD none specified 30-120% R ±10%D	±10%D <3 x IDL <10 x IDL ±25%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid) ±20%D ±30%D ±20%D
Metals by GFAA (graphite furnace atomic absorption)	SW846-7000A	high standard calibration verifications (ICV & CCV) method blank (sample prep) ^c matrix spike matrix spike duplicate laboratory control sample (sample prep) ^c serial dilution (if interference suspected) post digestion spike ^d	not required ±10%D (ICV), ±20%D (CCV) none specified none specified none specified none specified ±10%R ±15%D	±5%D ±10%D (ICV), ±20%D (CCV) <3 x IDL ±25%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid) ±25%D ±10%R ±25%D (liq.), ±30%D (solid)
Mercury by CVAA (cold vapor atomic absorption)	SW846-7471A SW846-7470	instrument blank calibration verification (ICV & CCV) ^a method blank (sample prep) ^c laboratory control sample (sample prep) ^c matrix spike matrix spike duplicate or sample duplicate post digestion spike ^d	none specified none specified none specified none specified none specified none specified none specified	<5 x IDL ±10%D <5 x IDL ±25%D ±25%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid) ±25%D (liq.), ±30%D (solid)
Carbon (total organic carbon, total carbon, total inorganic carbon)	SW846-9060	instrument blank calibration verification (ICV & CCV) matrix spike matrix spike duplicate	none specified none specified none specified none specified	<3 x IDL ±10%D (ICV.), ±20%D (CCV) ±25%D (liq.), ±30%D (solid) ±20 RPD (liq.), ±30 RPD (solid)
Anions by Ion Chromatography (IC)	SW846-9056	calibration verification (ICV & CCV) matrix spike sample duplicate	±10%D (ICV), ±5%D (CCV) none specified none specified	±10%D (ICV), ±15%D (CCV) ±25%D ±20 RPD
pH measurement	SW846-9040A SW846-9045B	check standard sample duplicate	none specified none specified	±10%D ±20%D

Analysis	Method (s) CASD-AM-	Quality Control Check (per batch)	SW-846 Acceptance Criteria (%D, %R, RPD)	RMAL Acceptance Criteria (%D, %R, RPD)
Total and dissolved solids (TS & TDS)	EPA600-160.2 EPA600-160.3	sample duplicate check standard	none specified none specified	±10 mg/ 10mL sample ±10%D
Carbonate and bicarbonate titration	AC-MM-1 003105	sample duplicate check standard	none specified none specified	±20 RPD ±20%D
Gross alpha/beta	EPA-900.0 RML-RA02 RML-RA12	background check calibration verification method blank (optional) ^f sample duplicate matrix spike	none specified none specified none specified none specified none specified	< 3sigma daily change ±10%D evaluated for contamination ±25 RPD (liq.), ±30 RPD (solid) ±25%D (liq.) & ±30%D (solid)
Nuclides by gamma spectrometry	EPA-901.1	background check calibration verification sample duplicate	none specified none specified none specified	< 3sigma daily change ±10%D ±25%D (liq.) & ±30%D (solid)
Sr-90 determination	RML-RA13 EPA-905.0	method blank (optional) ^f laboratory control sample matrix spike matrix spike duplicate or sample duplicate associated instrument QC	none specified none specified none specified none specified none specified	evaluated for contamination ^g 20%D ±25%D (liq.) & ±30%D (solid) ±25 RPD (liq.), ±30 RPD (solid) see gross alpha/beta criteria
Ce-99 determination	DOE Compendium RP550 RML-RA05	method blank (optional) ^f laboratory control sample matrix spike matrix spike or sample duplicate associated instrument QC	none specified none specified none specified none specified none specified	< 3 x IDL 20%D ±25%D (liq.) & ±30%D (solid) ● 25RPD (liq.), ±30RPD (solid) see ICP-MS criteria
I-131 determination	EPA-906.0	method blank (optional) ^f laboratory control sample matrix spike matrix spike duplicate or sample duplicate associated instrument QC	none specified none specified none specified none specified none specified	evaluated for contamination ^g 20%D ±25%D (liq.) & ±30%D (solid) ±25 RPD (liq.), ±30 RPD (solid) see gross alpha/beta criteria
Th Determination	EPA-901.1 RML-RA09	method blank (optional) ^f laboratory control sample matrix spike matrix spike duplicate or sample duplicate associated instrument QC	none specified none specified none specified none specified none specified	evaluated for contamination ^g 20%D ±25%D (liq.) & ±30%D (solid) ● 25RPD (liq.), ±30RPD (solid) see gamma spectrometry criteria
PCBs (polychlorinated-biphenyls)	SW846-8080	calibration verification (ICV & CCV) method blank (sample prep) surrogate standard matrix spike matrix spike duplicate sample duplicate laboratory control sample (sample prep) ^g	refer to method 8080 none specified none specified none specified none specified none specified none specified	to be specified ^h < regulatory limit (2ppm) ± 50-150%R ± 50-150%R to be specified ^h to be specified ^h

- a Initial calibration verification (ICV) is typically performed at the beginning of a run to check the calibration and must be independent of the calibration standards. The continuing calibration verification (CCV) must also be independent of the calibration standards, but may be the same standard as the ICV. The CCV is typically analyzed every 10 samples and at the end of the run for metals analysis or every 12 samples for organic analysis.
- b The calibration blank is an instrument blank used in the calibration to initially determine the blank value and therefore used as blank subtraction. The continuing calibration blank (CCB) is also an instrument blank which is analyzed every 10 samples and at the end of the run, but is not used in blank subtraction, but only to monitor instrument contamination.

- c Method blanks and laboratory control samples are only required if a sample preparation is performed before analysis. Sample preparation does not include dilutions or transfers to containers.
- d Post digestion spikes are not necessary if the pre-digestion spike is in control. If this control does not meet the QC acceptance criteria, the post digestion spike should be performed.
- e Acceptance criteria:
%D = % deviation from true value
%R = % recovery of true value
RPD = relative percent difference between two compared values
- f Method blanks for radiochemical analysis are used to monitor cross contamination. However, due to the levels of radioactivity present in samples at the **RMAL**, the effect of contamination may be insignificant in most cases. Therefore, the requirement to analyze a method blank for radiochemical analysis is **optional (i.e.** at the discretion of the chemist or supervisor).
- g Acceptance criteria for the method blanks performed for radiochemical analysis varies based upon the level of activity in the samples and the amount of background activity. A qualified chemist reviews the data from method blanks to determine if significant contamination is present.
- h The acceptance criteria for PCB analyses which are not identified in this table, shall be specified at a later date. Currently, the Analytical Methods Group group leader specifies the QC criteria if different **from** SW846 and if not specified by the sample generator.

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