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Revision 0A

241-Z-361 Tank Characterization Report

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management
Project Hanford Management Contractor for the
U.S. Department of Energy under Contract DE-AC06-96RL13200
Fluor Hanford
P.O. Box 1000
Richland, Washington

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Division: Nuclear Material Stabilization Project

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Environmental Quality Management

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EXECUTIVE SUMMARY

Tank 241-Z-361 is an underground settling tank at the Plutonium Finishing Plant (PFP) at the U.S. Department of Energy's Hanford Site in Richland, Washington. The tank served as the primary solids settling tank for low-salt liquid waste for several buildings at PFP from 1949 to 1973.

The contents of Tank 241-Z-361 were characterized to provide information to address an unreviewed safety question (USQ) related to the tank conditions. The USQ is discussed in detail in the *Justification for Continued Operation for Tank 241-Z-361* (PHMC 1999). The effort to characterize the residual sludge in the tank was conducted in two phases. Phase I activities included initial opening of the tank, collection of a headspace vapor sample for laboratory analysis, and conducting a video camera survey of the tank interior. Phase II activities included collection of two full-thickness core samples of the sludge for laboratory analysis, collection of additional tank headspace vapor samples for laboratory analysis, and conduct of nondestructive assay surveys of two dry wells that penetrate the sludge.

The characterization results were evaluated and reported at two levels. The first report was the recommendation for a regulatory path forward to address the tank contents. This document was required under the *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement or TPA), Milestone 15-37B (Ecology et. al. 1994). The milestone document provided evaluation of a focused set of data to allow assessment of any imminent hazards that may be presented by the sludge in Tank 241-Z-361. The report specifically evaluated the

potential for a criticality accident, potential presence of flammable concentrations of vapors in the headspace, potential mobility of the radioisotopes in the sludge, and qualitative assessment of the apparent structural integrity of the tank itself. The report concluded that there were no imminent threats posed by the tank or its contents in their present condition, and that the conditions are not likely to change in the near future. The sludge is, however, identified as a principal threat waste based on its content of plutonium and other toxic metals (e.g., mercury and chromium). As a principal threat waste, the sludge will ultimately require remediation under the site restoration activities established by the *Comprehensive Environmental Response, Compensation, and Liability Act of 1980* (CERCLA). A non-time-critical removal action was recommended in the milestone document as the preferred regulatory pathway to address the sludge. Based on the newly available information, the USQ on the tank was closed in January 2001.

This tank characterization report presents the second level of reporting of the Tank 241-Z-361 sludge characterization activity. The measurements and observations of the sludge and headspace vapors collected during both phases of field investigation at the tank are summarized and discussed in detail in this document.

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ACRONYMS

%LEL	lower explosive limit
AAMS	Aggregate Area Management Study
AEA	alpha energy analysis
APC	alpha proportional counter
ASTM	American Society for Testing and Materials
CAM	continuous air monitor
CAS#	Chemical Abstract Services number
CCl_4	carbon tetrachloride
CCS	counter control standard
CCV	continuing calibration verification
CERCLA	*Comprehensive Environmental Response, Compensation, and Liability Act of 1980*
CG	combustible gas
CGM	combustible gas meter
COC	chain of custody
cps	counts per second
CRQL	contract required quantitation limit
DBBP	dibutyl dibutyl phosphonate
DBP	dibutyl phosphate
DBP-d	dibutyl phosphate-derivitized
DCB	decachlorobiphenyl
DL	detection limit
DOE	U.S. Department of Energy
dpm	disintegrations per minute
DQA	Data Quality Assessment
DQO	Data Quality Objective
ECE	electrical conductivity of extract
EDA	exploratory data analysis
EPA	U.S. Environmental Protection Agency
FSAR	Final Safety Analysis Report
GC/MS	gas chromatography/mass spectrometry
GEA	gamma energy analysis
GFAA	graphite furnace atomic absorption
HEPA	high efficiency particulate air (filter)
HPGe	high purity germanium
HRLS	high rate logging system
IC	ion chromatography
ICP	inductively coupled plasma
ICV	initial calibration verification
ID	inside diameter
IDLH	immediately dangerous to live and health
IRQ	inter-quartile range
JCO	Justification for Continued Operation
KOH	potassium hydroxide
LCL	lower control limit

LCS	laboratory control sample
MDA	minimum detectable activity
MDC	minimum detectable concentration
MDL	method detection limit
MS	matrix spike
MSD	matrix spike duplicate
N/A	not analyzed
ND	not detected above reporting limit
NDA	nondestructive assay
NDR	no data reported
NEVS	nonelectrical vapor sampling
NH_3	ammonia
NIST	National Institute of Standards and Technology
NO_x	nitrogen oxides
O_2	oxygen
OD	outer diameter
PCB	polychlorinated biphenyl
PFP	Plutonium Finishing Plant
PID	photoionization detector
ppbv	parts per billion by volume
ppm	parts per million
ppmv	parts per million by volume
PQL	practical quantitation limit
PRF	Plutonium Reclamation Facility
PUF	polyurethane filter
QA	quality assurance
QC	quality control
RCRA	*Resource Conservation and Recovery Act of 1976*
RPD	relative percent difference
RPP	River Protection Project
SAP	Sampling and Analysis Plan (vapor)
SAS	Special Analytical Services
SDG	sample delivery group
STT	sorbent tube train
TAP	toxic air pollutant
TBP	tri-n-butylphosphate
TCD	thermal conductivity detection
TCLP	Toxicity Characteristic Leachate Procedure
TCX	tetrachloro-m-xylene
TIC	tentatively identified compound
TNMOC	total nonmethane organic carbon
TOV	total organic vapor
TSAP	Tank Sampling and Analysis Plan (sludge)
TSCA	*Toxic Substances Control Act*
UCL	upper confidence level or upper control limit
UHC	underlying hazardous constituent

USQ	unreviewed safety question
UTS	Universal Treatment Standard
VOA	volatile organic analysis
VOC	volatile organic compound
VPRQL	Vapor Program Required Quantitation
WSCF	Waste Sampling and Characterization Facility
WSR	Wilcoxon Signed Rank

1.0 INTRODUCTION

1.1 HISTORICAL INFORMATION SUMMARY

1.1.1 Tank Physical Description

Tank 241-Z-361 is a rectangular underground structure, constructed of reinforced concrete, and located near the east end of the south fence line of the Plutonium Finishing Plant (PFP) between Building 241-Z and the retention basin, 73.15 m (240 ft) south of 236-Z (*Z-Plant Source Aggregate Area Management Study Report* [DOE-RL 1992]). Figure 1-1 shows a plan view of the tank. The interior of the tank is 7.92 m (26 ft) long and 3.96 m (13 ft) wide and varies in depth between 5.18 m (17 ft) deep at the inlet (north end) and 5.49 m (18 ft) deep at the outlet (south end). The tank base is 22.86 cm (9 in) thick, with grout and waterproofing added for a total thickness of 30.48 cm (12 in). All walls are 30.48 cm (12 in) thick and the roof is 25.40 cm (10 in) thick. The top of the tank was sealed with 0.635 cm (¼ in) mastic and approximately 10.16 cm (4 in) of concrete was poured over the mastic with 5.08 cm by 5.08 cm (2 in by 2 in) 14-gauge reinforcement mesh. The elevation of the top of the tank is 204.98 m (672 ft 6 in). Grade level elevation is 205.59 m (674 ft 6 in). Figure 1-2 presents a side view of the tank looking west.

The interior of the tank was lined with 0.9525 cm (3/8 in) carbon steel on the bottom and up the sides to within 15.24 cm (6 in) of the roof. A protective coating was placed between the liner and the concrete as a corrosion barrier. Two 15.24-cm (6-in) stainless-steel pipes lead into the tank (from the retention basin and 241-Z) at the north end of the tank and one 20-32 cm (8-in) stainless-steel pipe forms the discharge at the south end of the tank. Baffle boxes were installed around the inlet and discharge pipes and attached to the liner. The elevation of the bottom of the inlet piping is 669 ft above mean sea level and that of the bottom of the discharge pipe is 668 ft above mean sea level.

The tank roof has three large penetrations and eight riser penetrations (Figure 1-1). A 0.91-m (3-ft) manhole exists at the north end of the tank. A second manhole is centered near the outside wall of the tank, and the third large penetration is a 1.22-m (4-ft) diameter concrete plug in the geometric center of the tank roof. There are two 20.32-cm (8-in) risers, one 5.08-cm (2-in) riser, and one 7.62-cm (3-in) riser built into the southwest corner of the tank and one 7.62-cm (3-in) riser built into the northeast corner of the tank. One 15.24-cm (6-in) riser penetration was installed through the concrete plug, and two 20.32-cm (8-in) riser penetrations were installed north of the center plug. All eight risers are capped or flanged closed and no equipment remains installed in the tank.

One of the south end 20.32-cm (8-in) risers had a dry well installed. This dry well has since been removed or corroded away, as it is not visible in the photographs of the interior of the tank. The inlet and outlet pipes have been isolated and plugged or flanged 0.61 m (2 ft) outside the outer wall of the tank. The reinforced concrete poured over the top of the tank was removed from over the two manholes when the tank was opened for sampling and photography in the mid 1970s. The manhole covers were subsequently reinstalled, covered with weather covers, and buried. The tank is covered with approximately 0.61 m (2 ft) of soil.

Figure 1-1. Plan View of Tank 241-Z-361 Indicating Location and Identification of Tank Riser Pipes

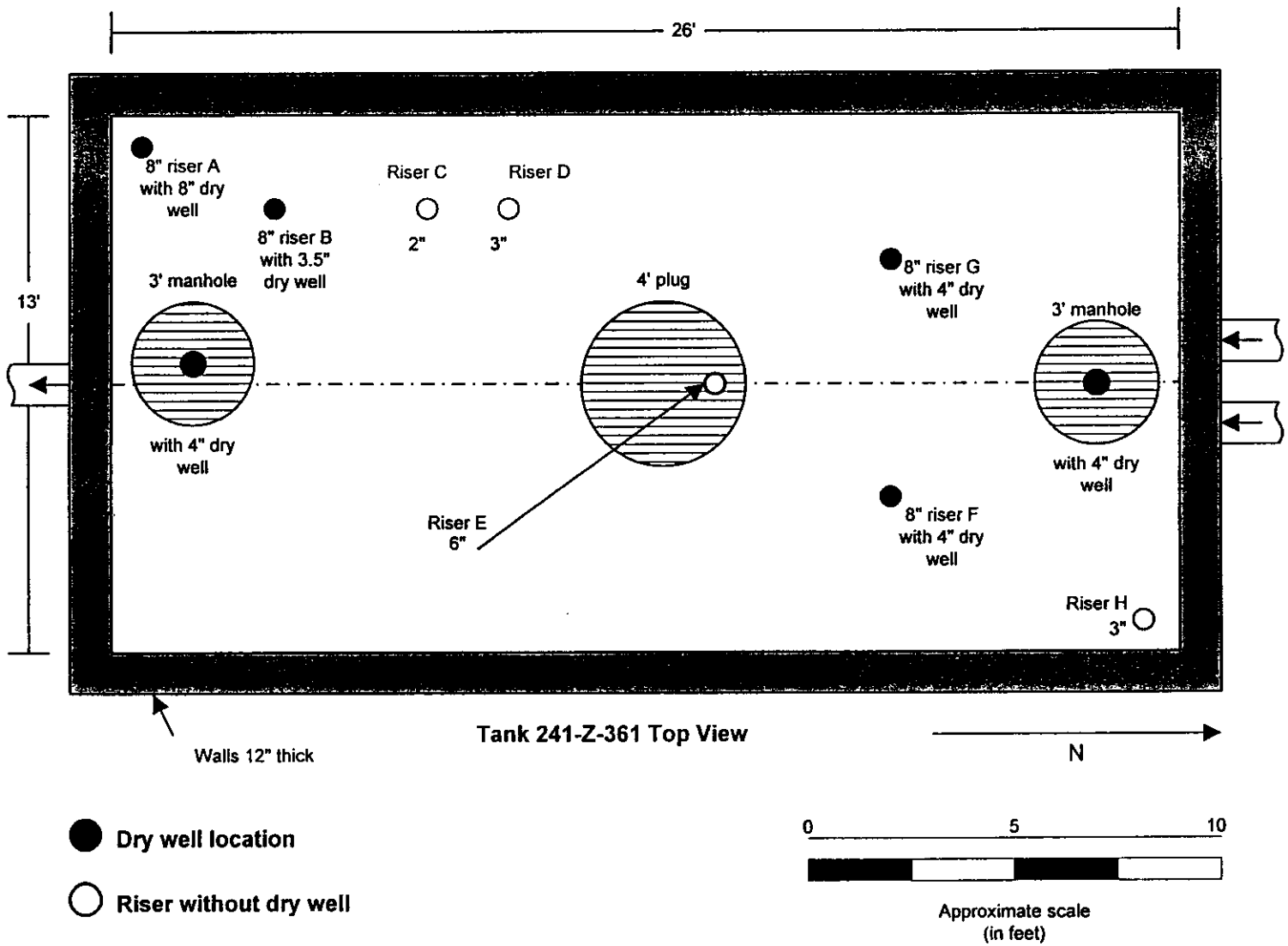
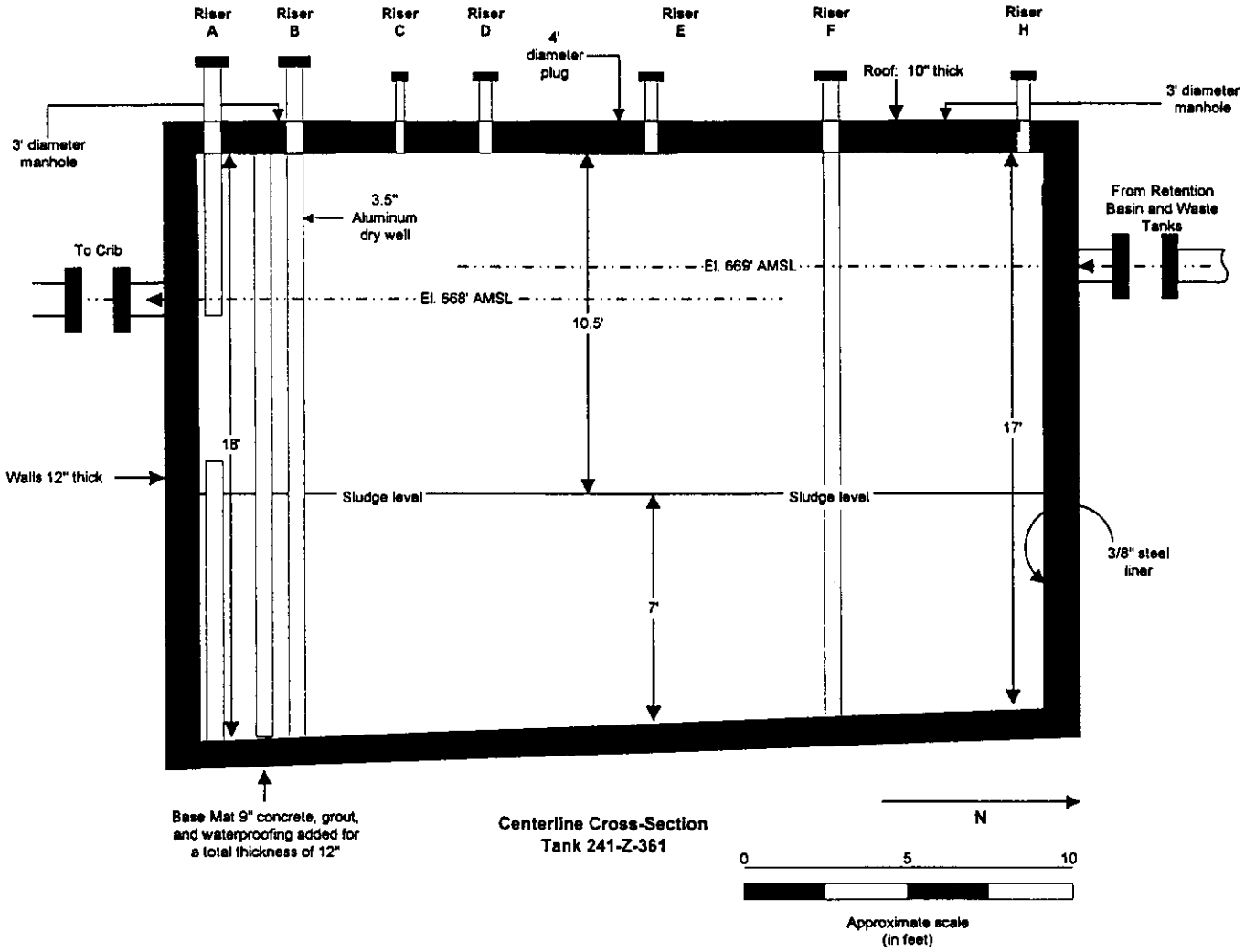


Figure 1-2. Side View of Tank 241-Z-361



1.1.2 Tank Operational History

Tank 241-Z-361 served as a primary solids settling tank for low-salt liquid waste from the 234-5Z, 236-Z, and 242-Z buildings. Tank 241-Z-361 was in service from 1949 to 1973. In 1975, all but approximately 800 L (210 gal) of the supernatant was pumped from the tank and was routed to the 216-Z-1A tile field and the 216-Z-1, -2, -3, and -12 cribs for disposal to ground (DOE-RL 1992). The tank was sealed in 1985 to prevent gas-phase communication with the surface. Approximately 239 cm (94 in) of sludge remains in the tank (about 75 m^3 [82.05 yd^3]). The sludge was expected to be dominated by the water insoluble components of effluent from Buildings 234-Z, 236-Z, and 232-Z. The sludge was believed to contain between 26 and 75 kg of plutonium (Freeman-Pollard 1994) with a probable inventory of 26.8 kg. A previous assessment of material unaccounted for estimated the tank contents as 31.2 kg plutonium (Lipke et. al. 1997).

1.1.3 Historical Process Records

The effluents that flowed through the system included incinerator caustic off-gas scrubber solution and liquid waste streams from the Plutonium Reclamation Facility (PRF), Waste Treatment Facility, and PFP laboratory. In addition to the drain lines from the process facilities, an unquantified (but large) volume of process water was discharged from retention basins to the cribs via Tank 241-Z-361.

When the tank was in use, the contents were neutralized by adding fly ash, and later sodium hydroxide, to raise the pH to the 8 to 10 range. Liquid samples collected in March 1975, however, had a pH as low as 4. Before this characterization, it was assumed that the pH was greater than 2, which renders the plutonium mostly insoluble.

Documentation about the individual chemical processes at PFP is sketchy. Although records describing the finishing process and the reclamation process for the radionuclides, especially plutonium, are quite complete, any discussions about additives such as organic reagents and solvents are very limited. Because all laboratory waste was discharged into the 241-Z-361 settling tank and the laboratories tested the individual processes (finishing and reclamation) on a bench scale, the same types of organics were discharged through the tank as were generated from process activities.

Large amounts of water were flushed through Tank 241-Z-361 and the discharges to the tank were generally dilute. Therefore, any constituents that were soluble in water were likely to have been washed to the cribs, leaving water-insoluble solids. In addition, materials sent to Tank 241-Z-361 were steam jetted. Compounds with low boiling points and high vapor pressures would likely have been vaporized and released through system vents. Except for the laboratory-generated waste streams, there were no processes that discharged reactants reasonably capable of generating large exothermic reactions. The laboratory chemicals discharged would have been in small quantity and well-diluted and, therefore, not likely to present a significant hazard. However, some organic materials used were heavier than water and may have settled in layers within the tank sludge.

Several facilities in the vicinity of PFP (234-5Z) may have contributed to the sludge in Tank 241-Z-361. The PFP was built in 1948 and began processing plutonium in mid-1949. The incinerator (232-Z) operated from December 1961 until May 1973. The PRF (236-Z) began operations in May 1964. The Waste Treatment Facility (242-Z), which reclaimed americium, operated from August 1964 until August 1976. Waste from some of these processes went through transfer lines to the Sump Tanks (241-Z-D4, 241-Z-D5, 241-Z-D7, 241-Z-D8) in Building 241-Z. Waste from Sump Tank 241-Z-D6 went to Tank 241-Z-361, whereas waste streams from the other sump tanks were directly discharged to the appropriate cribs, trenches, and ditches. Table 1-1 provides typical sources and amounts of plutonium and chemical contaminants added to Tank 241-Z-361.

Table 1-1. Typical Low-Salt Aqueous Process Streams in the Plutonium Finishing Plant (Circa 1969)

Stream	Drain	Source	Thousands of Gallons/Year	Plutonium Grams/Year	Chemical Contaminant
Uncontaminated lab wastes	D-4, 5	Cooling water for equipment in labs	127	0	None
Contaminated lab wastes	D-4, 5	Lab drains	174	100	Miscellaneous lab chemicals
Waste treatment aqueous waste	D-6	Ion exchange process	86	60	Principally Al, Ca, Mg, nitrate
Incinerator scrubber solution	D-6	Spent caustic from scrubber	6	600	Considerable Na
Reclamation condensate	D-6	Process concentrators	54	12	Slight
Fluorinator off-gas jet	D-6	Water for vacuum jet	1,906	100	Hydrogen fluoride
		Total	**2,353**	**872**	

Source: *Tank 241-Z-361 Process and Characterization History* (Jones 1997).

1.1.4 Historical Characterization Data

Several historical documents were reviewed to obtain a better understanding of the operations at PFP. Summaries of these documents are provided below.

- *History and Stabilization of the Plutonium Finishing Plant (PFP) Complex, Hanford Site* (Gerber 1997)

  Gerber (1997) provides a historical view of operations at Z-Plant and includes references to particular chemicals used. However, the individual waste streams and the flow of these waste streams are not addressed. For the chemical constituents, this document appears to be largely based on DOE-RL (1992).

- *Tank 241-Z-361 Process and Characterization History* (Jones 1997)

Jones (1997) interviewed operations personnel from Z-Plant and used historical documents, where available, such as laboratory books, notes, and memos to specify the operations and waste that potentially discharged to Tank 241-Z-361. Jones (1997) provides a list of known and suspected chemicals in the sludge.

- *Z-Plant Source Aggregate Area Management Study Report* (DOE-RL 1992)

  The Aggregate Area Management Study (AAMS) Report (DOE-RL 1992) lists specific waste streams from each location and provides as much detail about the contents of the waste streams as possible. It appears, however, that historical documents, such as those used in Jones (1997), were not incorporated in the AAMS report.

- *Inventory of Chemicals Used at Hanford Site Production Plants and Support Operations (1944-1980)* (Klem 1990)

  Klem (1990) consists of tables of chemicals used at the Hanford Site and lists these by locations. A list for Z-Plant is included.

Tank 241-Z-361 was characterized in the mid- to late-1970s as described in *Results of 361-Z Sludge Characterization* (Dodd 1976) and Jones (1997). The main focus of characterization was the plutonium content of the sludge, the distribution of plutonium, and the presence of various nuclear poisons. At this time, no vapor samples had been taken from Tank 241-Z-361.

The sludge was found to vary greatly in solids content, but to be on average 30% solid material with the remainder being mostly water. The sludge was deposited in layers from the various operating campaigns and exhibits considerable variability in consistency. There was no indication of free organic layers which could provide significant vapor-phase organic compound concentrations. Tables 1-2 and 1-3 provide historical data and probable nonradioactive components present in the sludge based on past sampling of Tank 241-Z-361. (Note: this sampling was performed using a thief-type sampler at given depths.)

Table 1-2. Component Concentrations in Air Dried Tank 241-Z-361 Solids

Component	Northeast Core (g/L)	Southwest Core (g/L)	Center Manhole Bottle (g/L)	
			Sample #8	Sample #9
Aluminum	71.8	304.0	---	290.3
Calcium	345.0	460.0	322.4	213.6
Cadmium	<3.8	<3.4	<0.4	0.9?*
Iron	230.9	562.2	59.0	74.0
Sodium	18.6	40.5	6.3	200.4
Silicon	10.5	10.4	4.4	8.3
Oxygen	20.	200.	---	---
Hydrogen	0.6	60.	---	---
Carbon	46.	87.2	---	---
Chloride	---	34.2	---	---
Fluoride	---	3.9	---	---

Source: Lipke et al. (1997) and PHMC (1998).
*Documented in PHMC (1998) with "?".

The large volumes of water discharged through this tank should have washed away soluble components. Based on bench-scale tests, the addition of water to the tank would not dissolve the plutonium or other solids (Jones 1997). Dodd (1976) indicated the sludge samples were very difficult to dissolve, even in a solution of 12M nitric acid and 0.5M hydrofluoric acid. This demonstrates the highly insoluble nature of the residual tank solids.

As stated previously, the tank was believed to contain between 26 and 75 kg of plutonium (Freeman-Pollard 1994). Both volume discharge records and limited sludge sampling data strongly suggest the plutonium was distributed in relatively simple layers, with plutonium concentrations varying only within a factor of 2 from the mean of 0.4g/L (29 kg total inventory). A 1997 criticality study concluded that, although the plutonium inventory is potentially sufficient to generate a criticality, plutonium distribution within the sludge makes an inadvertent criticality extremely unlikely (Lipke et. al. 1997).

Little is known regarding the routine acidity of the wastes sent to the settling tank, other than the general operations guidance that the waste water was to be neutralized before transfer into Tank 241-Z-361. In March 1975, the pH of one sample from tank sludge/solids was measured at 4.0. The corroded carbon steel liner also indicates that some wastes were not completely neutralized or the acidic flushes of sump tank 241-Z-D7 caused a low tank pH, or both.

Table 1-3. Known and Probable Components of 241-Z-361 Tank Sludge

Type of Component	Component	Probable Source
Known metals	Al	Waste treatment
	Na	Incinerator off-gas treatment
	Ca	Waste treatment
	Si	Incinerator off-gas treatment
	Cd	Most likely an analytical artifact
Known nonmetals	F^-	Hydrogen fluorinator
	Cl^-	Waste treatment
	C (organic or total?)	Incinerator off-gas treatment
	H_2O	All
	H^+	All
Probable metals	Pb	Incinerator off-gas treatment
	Mg	Waste treatment
	Mn	Waste treatment
	Cr	Corrosion of stainless-steel equipment
	Ni	Corrosion of stainless-steel equipment
	Ag	Lab film processing
Probable nonmetals	NO_3^-	Waste treatment
	NO_2^-	Radiolysis of NO_3^-
	SO_4^{2-}	Waste treatment
	PO_4^{3-}	Degradation of TBP
	CO_3^{2-}	Incinerator off-gas treatment
Probable organics	CCl_4	Waste process
	DBBP	Waste treatment
	TBP	Waste treatment
	DBP	Degradation of TBP
	MBP	Degradation of TBP
	Butanol	Degradation of TBP
	Urea	Incinerator off-gas treatment
	Lard oil (Triolein)	Waste treatment
	Oxalic acid	Waste treatment
	Acetic acid	Incinerator off-gas treatment
	Benzene	Incinerator off-gas treatment
	Phthalic acid	Incinerator off-gas treatment
Known radionuclides	Pu	All
	Am-241	Decay of Pu-241
	U	Waste treatment

Source: Jones (1997).

Although some organic materials have likely entered the tank, carbon was found in only a few samples, and then at concentrations of about 1% total carbon. Only one sample exhibited a higher concentration (i.e., 6%). The carbon could be from fly ash in the incinerator scrubber solution, carbonate from neutralization and absorption into caustic solution, or from organic compounds. No separate organic phase has been identified in the tank.

Historical discharge records, in Appendix D of Jones (1998), provided cumulative discharges of plutonium to Tank 241-Z-361 from 1952 through 1972. Between 1952 and 1957, yearly plutonium discharges were generally less than 100 g/yr. Discharges increased dramatically between 1957 and 1965 (on the order of several hundred to 1,000 g/yr) and then slowed down again between 1965 and 1972, with yearly discharges generally less than 200 g/yr. Based on these data, one could conclude that three strata exist within the tank, corresponding to distinctly different plutonium concentrations. Visual characteristics of sludge samples, however, suggest that even more strata may be present.

1.2 SCOPE OF CHARACTERIZATION WORK CONDUCTED

The *241-Z-361 Sludge Characterization Sampling and Analysis Plan* (TSAP) (EQM 1999a) identifies the type, quantity, and quality of data needed to support characterization of the sludge remaining in Tank 241-Z-361. The procedures described in the TSAP were based on results of the *241-Z-361 Sludge Characterization Data Quality Objectives* (DQO) (EQM 1999b) process for the tank. Characterization data were required to evaluate the need for an early removal action and, as required, to determine the appropriate methods for (1) removal of the sludge from Tank 241-Z-361, (2) stabilization and packaging of the sludge, and (3) sludge disposal.

Characterization activities at Tank 241-Z-361 were conducted as part of the Hanford Site remedial activities under the *Comprehensive Environmental Response, Compensation, and Liability Act of 1980* (CERCLA). Characterization of Tank 241-Z-361 supported cleanup under the CERCLA past-practice sites listed in the *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) (Ecology et. al. 1994) and was not part of single- or double-shell tank farm operations.

Sampling activities were conducted in 1999 under the requirements of the *Tank 241-Z-361 Vapor Sampling Analysis Plan* (SAP) (Hill et. al. 1998) for Phase I activities and conducted in 1999 and 2000 under the TSAP (EQM 1999a) for Phase II activities. A description of these activities follows.

Phase I Activities:

- Performance of a load test of the tank roof to determine load restrictions for characterization activities.

- Initial opening of the first sealed riser, venting excess pressure (if any), installation of a high efficiency particulate air- (HEPA-) filtered passive vent, monitoring for combustible vapors, and collection of a tank headspace vapor sample for analysis of volatile organic compounds.

- An inspection of the inside of the tank using a video camera.

Phase II Activities:

- Collection of two full-depth sludge core samples from the tank for detailed chemical analysis.
- Additional tank headspace vapor samples were collected during sludge sampling to evaluate the potential for release of volatile compounds by disturbance of the sludge.
- Down-hole nondestructive assay (NDA) techniques were used in the existing aluminum dry wells in Tank 241-Z-361 to provide additional information on the distribution of radioisotopes in the sludge.

Results of Phase I and II activities are presented in this Tank Characterization Report. Results for the Phase I headspace vapor sample are presented in Section 2.0. Results for Phase II field and laboratory measurements of headspace vapor samples are presented in Sections 3.2.2 and 3.2.3, respectively. The NDA results and correlation to sludge composite samples are discussed in Section 3.8.

1.3 CURRENT TANK STATUS

As part of the 1997 *Plutonium Finishing Plant Chemical Hazard Assessment* (BWHC 1997), a review of Tank 241-Z-361 determined that the potential hazards associated with the tank had not been evaluated in the formulation of the current PFP authorization basis. Potential hazards postulated during the 1997 review included hydrogen concentration increase in the tank headspace, structural failure of the tank as a result of its age and degradation, and potential for inadvertent criticality due to time-related phenomena. On October 15, 1997, the U.S. Department of Energy (DOE) declared an unreviewed safety question (USQ) on the tank (Wagoner 1997).

In the October 15 letter, the DOE directed the preparation of a Justification for Continued Operation (JCO). Phase I of this JCO (PHMC 1998) provided a basis for DOE to approve the controls needed to open the tank safely and to sample the tank headspace atmosphere. Phase II of this JCO (PHMC 1999) addressed core sampling and safe storage. This two-phased approach allowed flammable gas concerns to be resolved and the physical condition (dry or wet) of the sludge to be assessed before core sampling activities were authorized. The noted activities were conducted as authorized under the various revisions of the JCO. The various data obtained by these characterization activities are the subject of this report. The data also are discussed in detail in the "Submittal of Documentation in Fulfillment of Hanford Federal Facility Agreement and Consent Order Milestone M-15-37B" (FH 2000).

The characterization data clearly indicate that the hazards associated with Tank 241-Z-361 are not nearly as severe as was postulated when the USQ was declared in 1997 (Wagoner 1997). Only very low concentrations of potentially flammable gas were detected, far below the lower flammable limit. No indications of imminent structural failure of the tank were found, and

workable limits for tank roof loading were defined. Criticality was determined to be extremely unlikely for the sludge in its current configuration. Safety analysis of the newly available data resulted in the conversion of the JCO into an addendum to the *Plutonium Finishing Plant Final Safety Analysis Report* (DOE-RL 1997) in which a number of safety controls were relaxed as appropriate based on the new data. In addition, the USQ on the tank was closed (DOE-RL 2001).

Tank 241-Z-361 is currently inactive, passively ventilated through HEPA filters, and isolated from PFP systems. The tank is cordoned off to restrict personnel access, and a permanent fence is planned for construction. In the future, the tank contents will be retrieved, processed, and dispositioned (potentially in-place). These activities are not authorized by the Facility Safety Analysis Report (FSAR) Addendum (DOE-RL 1997); however, the FSAR Addendum does address activities that may occur during the period of storage before field remediation activities commence. All of these activities have been conducted previously or were authorized for performance during Phase I or II of the JCO (PHMC 1998 and 1999). The activities allowed under this FSAR Addendum include:

- Storing the tank contents,
- Opening the tank and performing atmospheric sampling and other dome-intrusive activities,
- Local waste-disturbing activities such as taking samples of the tank contents by grab sampling or push mode core sampling,
- Characterizing the tank contents through nondestructive analysis such as neutron or gamma logging,
- Measuring the height of tank waste,
- Performing additional structural evaluations, and
- Conducting maintenance activities such as testing or replacing HEPA filters.

Activities necessary for field remediation of the tank will need to be authorized under a future revision of the PFP FSAR (DOE-RL 1997).

2.0 PHASE I TANK VAPOR CHARACTERIZATION

Phase I activities were intended to resolve the primary safety questions regarding the potential for presence of combustible gases in the tank, pressurization of the tank, and general structural stability. Phase I activities also provided some preparation for the planned Phase II sludge sampling actions. The results of the Phase I activities are described in the following sections.

2.1 PROCEDURE/METHOD SUMMARY

The SAP (Hill et. al. 1998) described a subset of Phase I activities to investigate the actual hazards associated with the tank. These activities involved controls needed for opening the tank safely, worker protection, resolving flammable gas issues, and assessing the condition of the tank contents. These activities were further broken down into discrete tasks.

Task 1: Baseline Survey, work area and riser preparation included the preparation of the work area by removing physical obstructions, placarding and posting, installing air containment (glovebag), and performing a baseline survey.

Task 2: Vent and Install Breather Filter involved the relief of initial tank pressure, reducing and maintaining flammable gases to levels which met the current tank controls for invasive activities, and installation of a breather filter with an integral vapor sampling port on the riser.

Task 3: Tank vapor sampling included the collection of representative vapor samples from the tank head space for chemical analysis. Resulting data were used to define air emissions and safety requirements for future tank sludge core sampling.

Task 4: Tank interior videography encompassed the collection of videos of the tank interior to enable engineering evaluation of the physical condition of the tank and to answer structural integrity issues.

Task 5 : Work area cleanup comprised the restoration of the area to the original condition, closed risers, removed waste, and restored original access control posting.

The specific methodology and approach for field measurements and vapor sample analysis are discussed below.

2.2 FIELD MEASUREMENTS

During each task, field health and safety monitoring was conducted to protect the workers. All health and safety field activities were performed with continuous health physics and industrial hygiene support. Radiological monitoring of surfaces and the workplace was performed using alpha and beta/gamma survey instruments. The workplace was monitored by continuous air monitors (CAMs) for potential airborne alpha contamination. Industrial hygiene personnel measured the flammable gas (lower explosive limit [%LEL]), organic vapors, and oxygen of glove-bag air, tank riser vapor space, and workplace air using a combustible gas meter (CGM). Draeger tubes were used to monitor carbon tetrachloride, ammonia, and acid vapor. The following paragraphs summarize the monitoring performed.

2.2.1 Personal Monitoring

Personal monitoring utilized a personal sample pump with dual sorbent tubes: one charcoal and one carbon bead. The personal sampling system was placed on selected individuals while performing work tasks to measure general employee exposure. Personal monitoring for organic solvents (1-butanol, acetone, benzene, hexane, and toluene) was conducted during tank venting. The Waste Sampling and Characterization Facility (WSCF) Laboratory analyzed the samples collected.

2.2.2 Area Monitoring

Area monitoring involved the collection and analysis of samples in the general area where work took place. Area monitoring provided a general overview of the potential for employee exposure. Initial area monitoring was performed for combustible gas (CG) and oxygen (O_2) levels and for total organic vapor (TOV) using direct reading instruments. The area inside the enclosure was monitored for organic solvents during tank venting before installation of the glove bag and breather filter. The tent was monitored for O_2, TOV, and ammonia (NH_3) during collection of vapor samples. An area sample for TOV was taken outside the glovebag during videography. Area monitoring for CG, TOV, and O_2 of the greenhouse also was conducted during tank clean-up activities.

2.2.3 Source Monitoring

Source monitoring consists of the collection of samples at the supposed source. This type of monitoring is used to determine the highest potential to which employees could be exposed. Source monitoring also is useful in providing an estimate of the frequency and magnitude of any release. Source monitoring was conducted initially for baseline levels of TOV, NH_3, CG, and O_2 during installation of breather filter for NH_3 and TOV, and continuously inside the glovebag and at the breather filter during tank venting and tank cleanup for CG, O_2, and TOV. The tank dome space and breather filter were monitored for CG, O_2, TOV, and NH_3 during collection of vapor samples. The dome space, 3 ft into the riser, and the breather filter locations were monitored for NO_x, NH_3, TOV, CG, and O_2 during videography. Samples were taken for carbon tetrachloride (CCl_4) at the glovebag during tank venting and for acid test during tank venting and installation of the breather filter.

Personal, area, and source monitoring involves the use of tools and instrumentation identified in Table 2-1 for the collection of field monitoring vapor data. Results of the field monitoring measurements are presented in Tables 2-2 through 2-7. The results are broken down by task that impacted the type and amount of monitoring necessary. Radiological health and safety survey and air monitoring radiological data were obtained in accordance with the Health and Safety Plan (TSAP) (EQM 1999a). These data were not part of this data quality assessment (DQA).

Table 2-1. Field Monitoring Tools

Tool	Need	Main feature(s)
Continuous air monitor	Airborne alpha	Continuous monitoring for alpha emitting airborne particulates
Portable radiological survey instruments	Alpha and beta surface contamination	Health and safety radiological control
Combustible gas indicator	Flammable gases	Nonspecific detector for combustible gases measures gas concentrations as a percentage of lower explosive limit; visual and audible alarms
Oxygen meter	Oxygen deficiency	Direct readout in percent oxygen; visual and audible alarms
Organic vapor meters/analyzers	Toxic gas/vapor	Nonspecific gas and vapor detection for organic and some inorganic compounds; sensitivity related to ionization potential
Indicator (Draeger) tubes	Carbon tetrachloride, ammonia, acid test	Quantitative accuracies are variable; real time/semireal time results
Sampling media, containers, and pumps	Organic solvents, ammonia	Collects personal sample in the "breathing zone" to evaluate the exposure level of the person sampled; requires laboratory analysis; most accurate method for measuring exposure

Table 2-2. Baseline Survey of Riser H on Z-361, Survey ID: 99-0891,-0892,-0893

Date – Time	Analytes			
	TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
4/26/99 – 1000	0	0	20.9	0
4/27/99 – 1030	0	0	20.9	0
4/28/99 – 0900	0	0	20.9	<5

Table 2-3. Vent Z –361 Tank and Install Breather Filter, Survey ID: 99-0894

Location	Date – Time	Analytes				
		CCl_4 (ppm)	TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
Glove bag/ riser	4/29/99 – 1015	NDR	2	0	NDR	0
	4/29/99 – 1038	0	2	0	NDR	0
	4/29/99 – 1125	0	NDR	NDR	NDR	--
	4/29/99 – 1130	0	2	0	NDR	0
Open riser	4/29/99 – 1140	0	2.3	0	NDR	0
	4/29/99 – 1140	Acid test – positive				
Breather filter	4/29/99 – 1135	0	NDR	NDR	19.1	NDR
	4/29/99 – 1150	Acid test – positive				

NDR = no data reported

Table 2-4. Vent/Open Tank 241-Z-361 Laboratory Analyses

Location	Sample Number	Analytes					
		1-Butanol (ppm)	Acetone (ppm)	Benzene (ppm)	Hexane (ppm)	Toluene (ppm)	NH_3 (ppm)
Area inside the enclosure	DFS-42999-01	<0.18	<0.22	<0.17	<0.40	<0.14	NDR
	DFS-42999-02	NDR	NDR	NDR	NDR	NDR	<0.05
Person at stepoff pad	DFS-42999-03	<0.18	<0.23	<0.17	<0.11	<0.14	NDR
	DFS-42999-04	NDR	NDR	NDR	NDR	NDR	<0.03
Person at industrial hygiene sampling table	DFS-42999-05	<0.19	<0.24	<0.18	<0.12	<0.15	NDR
	DFS-42999-06	NDR	NDR	NDR	NDR	NDR	<0.04
Person at bottle cart	DFS-42999-07	<0.19	<0.24	<0.18	<0.12	<0.15	NDR
	DFS-42999-08	NDR	NDR	NDR	NDR	NDR	<0.04
Blanks	DFS-42999-09	<0.004	<0.004	<0.004	<0.004	<0.004	NDR
	DFS-42999-10	NDR	NDR	NDR	NDR	NDR	<0.0005

ppm = parts per million

Table 2-5. Tank 241-Z-361 Vapor Sampling, Survey ID: 99-0916

Location	Date	Analytes			
		TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
Breather filter	5/7/99	0	0	21.0	0
Tent	5/7/99	0	0	21.0	0
Dome space	5/7/99	4	0	19.2	0

Table 2-6. In–Tank Video, Survey ID: 99-0927

Location	Date	Analytes				
		NO_X	TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
3 ft into riser	5/12/99	0	0.9	0	19.4	0
Initial breather filter	5/12/99	0	0	0	20.9	0
Area outside of glovebag	5/12//99	NDR	0	NDR	NDR	NDR
Dome space	5/12/99	0	3.4	1	18.5	0

Table 2-7. 241-Z-361 Tank Clean-Up, Survey ID: 99-0901

Location	Date – Time	Analytes			
		TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
Glove bag/ riser	4/30/99 – 0933	12	0	19.4	0
	4/30/99 – 0957	8.4	0	19.8	NDR
	4/30/99 – 1002	7.4	NDR	NDR	NDR
	4/30/99 – 1010	NDR	0	19.9	NDR
	4/30/99 – 1015	5.8	NDR	NDR	NDR
Greenhouse	4/30/99 – 0940	0	0	20.9	NDR
	4/30/99 – 1015	NDR	0	21	NDR
	4/30/99 – 1030	0	0	21	NDR
Breather filter	4/30/99 – 0949	0	0	20.9	0
	4/30/99 – 1002	NDR	0	21	NDR

2.3 PHASE I TANK VAPOR SAMPLES

2.3.1 Sampling

Phase I tank vapor samples were taken on May 7, 1999. Sampling activities began by carefully removing the riser flange to allow controlled venting of any pressure while maintaining a non-flammable atmosphere within the glovebag. The glovebag design provided for active purging using an inert gas if necessary to maintain nonflammability within the glovebag. After the riser was opened, a breather filter was installed. The breather filter assembly contained an integral vapor sampling port which facilitated collection of tank dome-space vapor samples without the need for additional containment. Vapor samples were collected through a Teflon® sampling tube which extended from the breather filter assembly down to approximately 0.30 m (1 ft) from the sludge surface.

The following samples were collected during the sampling event:

- Five SUMMA® canisters (one ambient air field blank, one system blank, and three tank samples)
- Six nitrous oxide (NO_X) sorbent trains (four tank samples, one field blank, and one trip blank)
- Eight tributyl phosphate/dibutyl phosphate (TBP/DBP) sorbent tubes (five tank samples, one field blank, one field blank spike, and one trip blank)

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® SUMMA is a registered trademark of Molectrics, Inc., Cleveland, Ohio.

- Seven acetic acid sorbent tubes (four tank samples, two field blanks, and one trip blank)
- Seven ammonia sorbent tubes (four tank samples, one field blank, and one trip blank.

The samples were analyzed for a series of chemical and radiological parameters including potential toxic air pollutants (TAP) and flammable gas constituents listed in Table 2-8.

The *Tank 241-Z-361 Waste Characterization Data Quality Objective: Headspace Vapor and Tank Structure* (Field and Banning 1998) and Hill et. al. (1998) define the type, quantity and quality of data needed to satisfy the objectives of the Vapor Sampling Project. Table 2-8 summarizes the analytes, methods of analysis, procedures, practical quantitation limits (PQLs), precision, and accuracy required by the DQO and SAP.

Samples of various volumes were drawn through a set of two prefilters (2-micron pore size). The WSCF Laboratory analyzed the particulate filters for total alpha radioactivity, total beta radioactivity, and gamma emitters (using gamma energy analysis [GEA]). Laboratory results from all filters showed a maximum activity of 4.E-06 μCi/sample beta and less than the minimum detectable concentration (MDC) for alpha (7.0E-7 μCi/sample). Similarly, the GEA results were all less than the MDC. The MDC for determination of gamma emitters by GEA varies by radionuclide and the specific count time for a sample. However, all GEA MDCs were less than 3.75 pCi/sample. Evaluation of the data by tank farm radiological control (WMH 1999a) concluded that the portions of the sampling system downstream of the filters would be considered releasable to the analytical laboratories, pending external surveys. No quality control (QC) data were supplied with the radiological results; therefore, no further evaluation of the data was performed.

2.3.2 Laboratory Analysis

Special Analytical Services (SAS) (Numatec Hanford Company) at the vapor laboratory in the 600 Area Weather Station Complex was the primary analytical laboratory for gas chromatography/mass spectroscopy (GC/MS), GC/thermal conductivity detection (TCD), and ion chromatography (IC) methods. The WSCF Laboratory also performed GC/MS analyses.

Table 2-9 summarizes the laboratory results for the required target analytes as specified in the DQO and SAP (Field and Banning 1998 and Hill et. al. 1998). Although not required by the SAP, analytes which were above PQLs (positive results) also are provided for information in Table 2-10. Results for all analytes (including undetected and tentatively identified compounds [TICs]) provided by the laboratory as part of the analysis method are provided in the *Tank Vapor Sampling and Analysis Data Package for Tank 241-Z-361 Sampled May 7,1999* (WMH 1999b). Results for blanks (trip, field, system blanks) are presented in Table 2-11 (Section 2.3.3).

Table 2-8. Chemical and Radiological Analytical Requirements[1]

Analyses				Criteria		
Primary Analyte	Procedure/ Laboratory	Prepara-tion	Container	PQL	Precision[2]	Accuracy
CCl_4, benzene, n-butanol	LA-523-404/ SAS	Direct	SUMMA®	5 ppbv	±25%	70-130%
DBP, TBP	LA-523-428/ SAS	Solvent extraction	Sorbent tube	5 ppbv	±25%	70-130%
CH_4, H_2, N_2O	LA-523-409/ SAS	Direct	SUMMA®	50 ppbv	±25%	70-130%
NO, NO_2	LA-533-400/ SAS	Water extraction	Sorbent train	5 ppmv	±25%	70-130%
Acetic acid	LA-533-404/ SAS	Water extraction	Sorbent train[3]	50 ppbv	±25%	70-130%
NH_3	LA-533-402/ SAS	Water extraction	Sorbent train[3]	10 ppmv	±25%	70-130%
Total alpha Total beta	LA-508-415/ WSCF	Direct	Particle filter	1 µCi/ filter	±25%	Not defined in SAP
GEA	No procedure specified[4]	Direct	Particle filter	1 µCi/ filter	±25%	Not defined in SAP

1. Analytical requirements from SAP (Hill et al. [1998], Tables 1-7 and 2-2).
2. Precision is defined here as relative percent difference between replicate analyses, or as relative standard deviation of continuing calibration verification results if replicate analysis are not possible.
3. System contains individual sorbent media sections for NH_3 and acetic acid.
4. WSCF Laboratory used procedure LA-508-462.

ppbv = parts per billion by volume
ppmv = parts per million by volume

Table 2-9. Results for Target Analytes (2 sheets)

Sample Number(s)	Target Analytes[1]	PQL	Sample Result(s) Reported	Units	Comment – Flag[2]
VV-9001-A03-S128	Carbon tetrachloride	5	160	ppbv	1 sample, confirmed by analysis of duplicate – flag NF
	Benzene	5	5	ppbv	1 sample – flag Q
	n-Butanol	5	5	ppbv	1 sample – flag Q
V9001-A19-P001 V9001-A19-P005	Tributyl phosphate	38	38 38	ppbv	2 samples – flag M*, star used to designate that the QC criteria were estimated with less than 30 determinations.
	Dibutyl phosphate	46	46 46	ppbv	2 samples – flag M*, star used to designate that the QC criteria were estimated with less than 30 determinations.

Table 2-9. Results for Target Analytes (2 sheets)

Sample Number(s)	Target Analytes[1]	PQL	Sample Result(s) Reported	Units	Comment – Flag[2]
V9001-A04-S129	Hydrogen	~20	50	ppmv	One sample – flag Q
	Methane	~20	50	ppmv	One sample – flag M
	Nitrous oxide	~20	110	ppmv	One sample – flag NF
VS9001-A06-TR01b VS9001-A07-TR02b VS9001-A08-TR03b VS9001-A09-TR04b	Nitrogen dioxide	0.88	0.88 0.88 0.88 0.88	ppmv	4 samples – flag Q
VS9001-A06-TR01d VS9001-A07-TR02d VS9001-A08-TR03d VS9001-A09-TR04d	Nitric oxide	1.4	1.4 1.4 1.4 1.4	ppmv	4 samples – flag Q
VS9001-A12-AA01 VS9001-A12-AA02 VS9001-A12-AA03 VS9001-A12-AA04	Acetic acid	47	<49 <48 53 77	ppbv	4 samples-Flag, all samples flagged by lab due to low LCS "*" recovery and Field blank contamination "F", two samples also flagged M.
V9001-A06-TR01a V9001-A07-TR02a V9001-A08-TR03a V9001-A09-TR04a	Ammonia	4.8	4.8 4.8 4.8 4.8	ppbv	4 samples– flag Q
V9001-F3 U,D V9001-F4 U,D V9001-F5 U,D V9001-F6 U,D V9001-F7 U,D V9001-F8 U,D V9001-F9 U,D U = upstream or D = downstream of air sample flow	Total alpha	7E-07	< 7.0E-7	µCi/ sample	7 sets of sample filters, all results reported as < DL – flag NF
	Total beta	Not reported	4.8E-06 (maximum result)	µCi/ sample	7 sets of sample filters – flag NF
	Gamma spectroscopy	< 3.8	< 3.8	pCi/ sample	7 sets of filter samples analyzed. All MDCs reported were <3.8 pCi/sample – all flagged U

1. Target analytes per Hill et al. (1998)
2. Flags: NF = no flag on data
   M or U = target analyte was not detected in sample
   Q = target analyte was detected, but at concentrations less than vapor program quantitation limit (VPRQL)
   F = field blank contamination

DL = detection limit
LCS = laboratory control sample

Table 2-10. Results for Nontarget Analytes Detected Above PQL

Sample Number	Other Detected Compounds	PQL	Sample Result	Units	Comment – Flag*
VV-9001-A03-S128	Acetone	5	20	ppbv	1 sample – flag NF
	Chloroform	5	1300	ppbv	1 sample, result from diluted sample (DF-48) – flag D
	n-butane	5	120	ppbv	1 sample – flag NF
	Freon 11	5	610	ppbv	1 sample, result from diluted sample (DF-48) – flag D
	N-Pentane	5	5.5	ppbv	1 sample – flag NF
	Dichloromethane	5	16	ppbv	1 sample – flag NF
	2-Methylpentane	5	8.7	ppbv	1 sample – flag NF
	Toluene	5	7.1	ppbv	1 sample – flag NF
	Tetrachloroethylene	5	2000	ppbv	1 sample, result from diluted sample (DF-48) – flag D
V9001-A04-S129	Carbon dioxide	50	13,000	ppmv	1 sample – flag NF
V9001-A04-S129	TNMOC	0.2	12	ppmv	1 sample – flag NF

*Flags:
NF = no flag on data
D = target analyte reported is the result of a secondary dilution
M = target analyte was absent (not detected) in sample
Q = target analyte was detected, but at concentrations less than VPRQL
TNMOC = total nonmethane organic carbon

2.3.3 Field, System, and Trip Blank Results

Vapor sampling was conducted in accordance with the *Quality Assurance Project Plan for Sampling and Analysis of Waste Tank Headspace Vapors* (Keller 1994) and included the collection of field, system, and trip blanks. Field, system and trip blank results are presented in Table 2-11.

Field, system, and trip blanks were collected to assess the cleanliness of the sampling equipment, the effectiveness of the sample decontamination process, and potential sampling environment contaminant contribution. Nonelectrical vapor sampling site (NEVS) system blanks were collected in the field by pulling ambient air through the NEVS sample lines into a clean SUMMA® canister. A field blank was collected by sampling ambient air upwind of the tank. The NEVS system blank was analyzed for the same chemical analytes as actual tank vapor samples collected during use of the equipment. A trip blank traveled with the sorbent tubes from the laboratory, to the sampling site, and back to the laboratory. Blank results are summarized in Table 2-11. Acetone was detected above the PQL in the SUMMA® ambient air field blank. Acetone also was detected (but below the PQL) in the NEVS system blank. Acetic acid sorbent tubes for field blanks showed slightly positive results (above PQL), especially in the breakthrough sections of the tubes. The laboratory flagged the acetic acid results "F" for contamination in the field blank.

Table 2-11. Blank Results for Phase I Vapor Sampling of Tank 241-Z-361

Blank Sample No.	Blank Type	Reported Results/Units	Flag*
V9001-A01-S124	SUMMA® ambient air field blank	Acetone 6.0 ppbv 1-Butanol 5 ppbv n-Octane 5 ppbvn Nonane 5 ppbvn Decane 5 ppbv All other analytes not detected	NF Q Q Q Q M
V9001-A02-S127	SUMMA® NEVS system blank	Acetone 5 ppbv All other analytes not detected	Q M
V9001-A10-TR05	STT field blank	Ammonia 4.8 ppmv Nitrogen dioxide 0.88 ppmv Nitric oxide 1.4 ppmv	Q Q M
V9001-A11-TR06	STT trip blank	Ammonia 4.8 ppmv Nitrogen dioxide 0.88 ppmv Nitric oxide 1.4 ppmv	Q M Q
V9001-A16-AA05	Acetic acid sorbent field blank	<49 ppbv sample section 83 ppbv breakthrough section	**MF **F
V9001-A17-AA06	Acetic acid sorbent field blank	88 ppbv sample section 79 ppbv breakthrough section	**F **F
V9001-A18-AA07	Acetic acid sorbent trip blank	<47 ppbv sample section 111 ppbv breakthrough section	**MF **F
V9001-A24-P006	PUF field blank	DBP <46 ppbv TBP <38 ppbv	M M
V9001-A26-P008	PUF trip blank	DBP <46 ppbv TBP <38 ppbv	M M

*Flags:

**	=	specific QC problem discussed in the sample narrative
F	=	contamination found in the field blank
NF	=	no flag on data
M	=	target analyte was absent (not detected) in sample
Q	=	target analyte was detected, but at concentrations less than VPRQL

PUF = polyurethane filter
STT = sorbent tube train

2.4 QUALITY ASSURANCE/QUALITY CONTROL SUMMARY AND DATA QUALITY ASSESSMENT FOR PHASE I VAPOR SAMPLES

The quality assurance (QA)/QC data review included an evaluation of accuracy. Accuracy for these samples was evaluated by considering laboratory control samples, matrix spikes, surrogates or internal standards as appropriate to the method performed, initial and continuing calibrations. Precision was evaluated by looking at duplicate sample analysis when available. Findings are summarized in Tables 2-12 and 2-13 and are discussed below. Completeness also was evaluated using the definitions provided in the SAP (Hill et. al. 1998). All requested analyses for all samples provided were performed by the laboratory; therefore, completeness was 100%.

Table 2-12. QA/QC Summary for Phase I Vapor Samples Organic and Permanent Gas Analyses

Quality Control	Analytes			
	VOC in SUMMA®	TNMOC	Permanent Gases – CO, H_2, CH_4, CO_2, N_2O	DBP/TBP
Surrogate recovery	N/A	N/A	N/A	X
Blanks	O*	O	O*	O
Calibration	O*	O	O	O
Internal standards	O	N/A	N/A	O
Relative percent difference (RPD)	O	O	O	N/A
Laboratory control sample (LCS)	O	O	O	O
Matrix spike	N/A	N/A	N/A	X

N/A = not analyzed
O* = QC parameter is within required limits with notation
O = QC parameter is within required limits
X = QC parameter is not within required limits

Table 2-13. QA/QC Summary for Phase I Vapor Samples Inorganic Analyses

Quality Control	Inorganic Analytes		
	Acetic Acid	Ammonia	Nitric Oxide Nitrogen Dioxide
LCS	X	O	O*
Calibration (ICV, CCV)	O	O	O
RPD	X	O	O
Method performance blank	O	O*	O*
Matrix spike/matrix spike duplicate	O	O	O

O* = QC parameter is within required limits with notation
O = QC parameter is within required limits
X = QC parameter is not within required limits
ICV = initial calibration verification
CCV = continuing calibration verification

2.4.1 Data Quality Assessment Findings

A discussion of the pertinent DQA findings by analysis follows.

Volatile organic compounds (VOCs) in SUMMA® - Acetone was detected in the field and trip blanks slightly above 5 ppbv and acetone was detected in the samples. The blanks were likely contaminated by laboratory use of the chemical. Acetone was not an analyte of interest in the SAP (Hill et. al. 1998) and its presence in the blanks has no consequences for data use.

Three of the target compounds (Freon 11, chloroform, tetrachloroethylene) required dilutions. The dilutions were still above the calibration range, but were not analyzed with a second dilution. Therefore, concentrations are considered to be estimated.

Permanent gases – Hydrogen was detected in the zero air blank at about the same concentration (18 ppmv) as seen in the sample (26 ppmv). The decision level of hydrogen that was established in the SAP (Hill et. al. 1998) was 1% (approximately 10,000 ppmv). Thus, the potential error in estimating the hydrogen levels is insignificant compared to the decision level.

TBP and DBP – The required recovery range for surrogates is 70 to 130% or ±3 standard deviations of the average of at least 30 samples. Using the criteria of 70 to 130%, recoveries for the surrogate octachloronaphthalene (used in this analysis to monitor TBP recovery) ranged from 87 to 99%, well within the recovery range. Surrogate recoveries for the DBP surrogate (i.e., diphenylphosphate) ranged from 48 to 67%, which is below the lower end of the recovery range of 70%. Analyte recoveries for LCSs ranged from 70 to 88%. Field spike (matrix spike) recoveries for TBP were slightly lower (i.e., 68%) than the 70% control limit, whereas DBP recovery was 83% and within limits.

Although some QC data fell outside the general QC limits of 70 to 130%, the QC data all fell within the alternative limits of ±3 standard deviations of the mean of several test runs. The alternative recovery range of ±3 standard deviations of the average requires at least 30 samples to establish. The sample results for TBP and DBP were flagged (M*) by the laboratory to indicate that, although all of the results were within ±3 standard deviations of the mean, fewer than 30 samples (only 11) were analyzed in order to establish the alternative QC limits. The reported sample data did not detect any TBP or DBP; the levels reported were the lowest calibration point for the analyses. The data are useable for this project.

Acetic acid - The acetic acid determination method was still under development when the samples were analyzed. Two different LCS standard preparations were analyzed to examine the effect of the methanol/water rinse on the recovery of the LCS standard. For the LCS rinse preparation, a sorbent tube was rinsed with 3-mL portions of methanol and deionized water just before placement of the control standard. LCS rinse recovery was 11%. LCS no-rinse (rinse omitted) recovery was 23%. The results were flagged (*) by the laboratory due to low LCS recoveries. RPD for one set of duplicates was 28%. The required range is ±25%. The action limit determined in the SAP (Hill et. al. 1998) is 57.4 ppbv. This level is near the results that were determined during sampling (<48, <49, 53, and 77 ppbv). These results should be considered qualitative as well as any conclusions as to whether acetic acid is above or below the action limit. Based on the low recoveries and failure to meet QC criteria these data are not suitable to determine if acetic acid is above the action level.

Ammonia - Ammonia was detected in the method performance blank, but the concentration was less than the PQL (5 ppmv). The potential error is insignificant compared to the decision levels for ammonia based on flammability (i.e., LEL 15%) or for health and safety (immediately dangerous to life and health [IDLH] = 500 ppm). The data are acceptable for this project.

Nitric oxide – A peak was detected in the method performance blank, but the concentration was less than the PQL. The samples contained less than 1.4 ppmv, a level far below the decision level for nitric oxide based on health and safety (IDLH=100 ppm). Nitric oxide is not flammable. The data are acceptable for this project.

2.4.2 Summary

Tank headspace monitoring was performed before and during initial opening of the tank and installation of the HEPA-filtered vent, as well as during subsequent removal of equipment. Review of the data and associated QC lead to the conclusion that the data are suitable for the intended use to assess the tank headspace vapor constituents and to determine if the headspace gases present a safety issue due to potentially flammability or toxicity. Although small amounts of numerous gases were found as presented in Tables 2-9 and 2-10, the maximum concentration of flammable vapor reported was 1% of the lower flammability limit using a field combustible gas meter calibrated to hydrogen. In addition, there were no concentrations of gas that presented a health hazard to personnel during core sampling activities.

2.5 VIDEO PHOTOGRAPHY

Physical conditions inside Tank 241-Z-361 were recorded using a video camera inserted into the tank through an open tank riser pipe during the Phase I characterization activities. The recorded images were compared to existing still photographs of the tank interior. The conditions observed and apparent changes over time are discussed in the following sections.

2.5.1 Preexisting Still Photography

The conditions inside Tank 241-Z-361 had been evaluated during scoping activities based on a composite series of still photographs taken in early 1975, shortly after the supernatant liquid was removed from the tank. A still camera was inserted into the tank through the south manhole and a panoramic series of photographs was taken. The only available complete photomosaic composite photograph of the tank in 1975 exists in a photocopy of another document (RHO-ST-44) and does not produce an acceptable subsequent photocopy. However, the available copy is satisfactory for comparison of free liquid present on the surface of the sludge. One color photograph, taken in 1975 and used as part of the photomosaic, is available and is shown in Figure 2-1. The photograph in Figure 2-1 was taken from the south manhole looking toward the northeast corner of the tank. The photographs indicate that the sludge surface was relatively level and that little free liquid was apparent atop the sludge. The most significant observation is the absence of the aluminum pipes, or dry wells, that were subsequently placed in Risers B, F, and G and through the manhole covers in the north and south manholes (see Figure 1-1 for plan view of tank top).

2.5.2 Current Video Photography

Lockheed Martin Hanford Company personnel from the Tank Characterization Project Operations group performed an internal video camera inspection of Tank 241-Z-361 in May 1999. This inspection was conducted by inserting a video camera and lighting system into Riser E, located approximately in the center of the tank roof. The camera was rotated 360 degrees to allow observation of all four walls of the tank and was elevated and lowered to the extent possible to provide a view of the sludge surface and the tank roof. Figure 2-2 is a composite sketch of features of particular interest based on review of the video record of the tank interior. Figure 2-3 is a video capture image of a portion of the east wall of the tank photographed previously and shown in Figure 2-1. Riser F is just outside the left side of this video image.

The visually apparent comparisons and contrasts between the still photographs taken in 1975 and the current video recording are summarized below.

- Presence of additional dry wells in 1999 video record. The dry wells presently located in Risers B, F, and G and the north and south manhole covers are conspicuously absent in the earlier photograph. These dry wells were installed subsequent to the removal of the tank from service; however, no records of the installation of the dry wells, or of the reason for installation and the results of any related activities, could be located. A 2-in diameter "liquid level well" is visible in the 1975 photographs. This pipe appears to be connected to Riser B, C, or D. This pipe may be the same 3.5-in aluminum dry well currently present in Riser B; however, parallax in the photomosaic view makes exact location of this feature uncertain. The dry wells that have been directly inspected (i.e., those in Risers B, F, and G) were determined to be made of aluminum tubing.

- Similarity in apparent sludge surface level. The level of the sludge surface, and consequently the apparent depth of the sludge in the tank, is very similar in the two sets of photographs. The level is estimated by comparing the relative distance from the sludge surface to the roof of the tank in both photographs. This observation suggests that the level of waste in the tank has not changed substantially over the years between 1975 and 1999.

Figure 2-1. Photograph of a Portion of the Interior of Tank 241-Z-361, Taken in 1975

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Figure 2-2. Composite Sketch Based on 1999 Video of Tank 241-Z-361

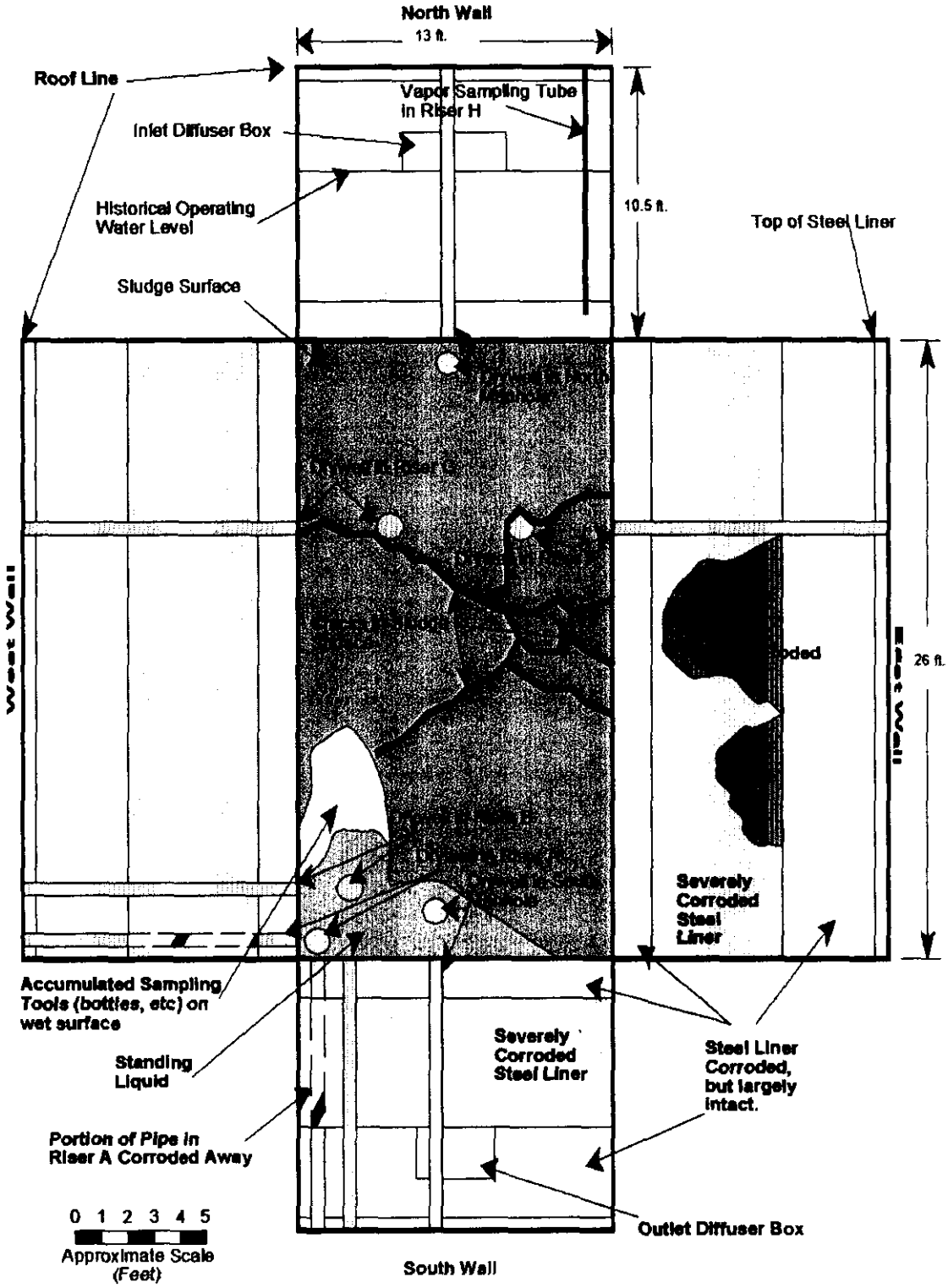
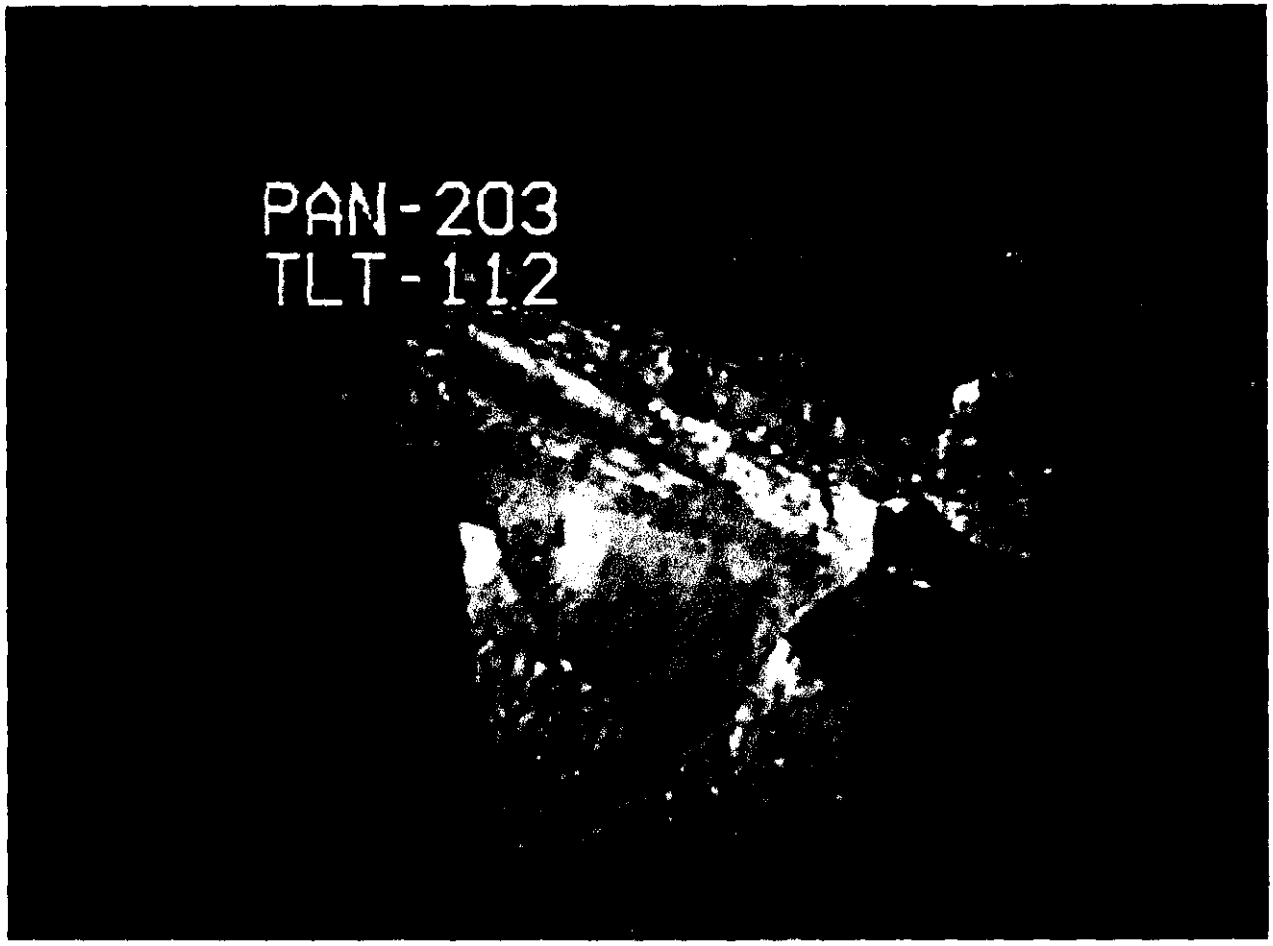


Figure 2-3. In-tank Video Capture of the Interior of Tank 241-Z-361, Taken in 1999



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- Similarity in area coverage of free liquid on the sludge surface. The video record made in 1999 was compared to the photomosaic of the tank interior taken in 1974 or 1975, after removal of the supernatant liquid (RHO-ST-44). These two records indicate that residual free liquid in the tank was confined largely to an area in the extreme south end of the tank and that little liquid was apparent at the sludge surface elsewhere in the tank. Free liquid also is visible in the 1999 video in disturbed areas immediately surrounding the dry wells in Risers F and G and in the north manhole, as well as in cracks in the sludge surface in the northern portion of the tank.

- Corrosion of the pipe in Riser A. An 8-in diameter steel pipe waste installed in the tank in Riser A, apparently during tank construction, and is described as a "monitoring well" (RHO-ST-44). This pipe is absent across the elevation range corresponding to the severe corrosion of the steel liner (see Figure 2-3).

- Corrosion of steel tank liner. The carbon steel liner of the tank exhibits severe corrosion over an elevation range from approximately 1.6 ft above the sludge surface to the historical operating water level at the elevation of the tank outlet pipe, a distance of approximately 4.7 ft. Severely corroded steel is still in place in some portions of the tank wall across this zone. In other areas, the steel appears to be gone and remnants of what appears to be plastic sheeting that likely underlies the steel liner is visible peeling away from the tank side. The steel liner and plastic sheeting are both absent in an irregularly-shaped area near the middle of the east tank wall. In this area, the concrete is severely corroded and coarse aggregate (i.e., aggregate of diameter less than 3 in) is completely exposed as can be seen in Figure 2-3.

The video record of the tank interior identified another feature that raises concern for the long-term structural stability of the tank. A continuous crack is visible in the tank roof slab, ranging in width from approximately 0.25 in to 0.75 in. This crack extends across the full width of the roof slab from the east wall to the west wall of the tank at approximately right angles to the tank walls, approximately one-third of the distance from the north end of the tank to the south end.

3.0 PHASE II TANK SLUDGE CHARACTERIZATION

Phase II sludge characterization activities were conducted in accordance with EQM (1999a). Full-depth core samples were collected from locations at Risers E (Core 263) and F (Core 264). See Figure 1-1 for tank plan view and relative riser locations. This section presents the results of the sampling and analysis activities.

3.1 PROCEDURE/METHOD SUMMARY

Phase II activities were planned according to the TSAP (EQM 1999a). The Phase II activities included collection of two core samples. Strata in the core segments were composited and analyzed by radiological and chemical analyses. The strata were composited based on total alpha analysis of each stratum, statistical assessment, visual assessment of the stratum and a documented compositing plan (EQM 1999c). The sludge composite samples were analyzed by Hanford Site laboratories. Any deviations from the methods are discussed in the subsection pertaining to the particular method.

The drainable liquid was collected and analyzed by the 222-S Laboratory according to EQM (1999a). Due to the small quantity of drainable liquid, approved deviations from the original plan were required. These deviations are discussed later in this chapter.

In addition to the sludge composite samples, tank headspace vapor samples were collected during the sludge sampling to evaluate the potential for release of volatile compounds by disturbance of the sludge. Section 3.2.3 summarizes and evaluates these results.

Down-hole NDA was used to augment the data for the radiological constituent distribution; these results are discussed in Section 3.8.

3.2 FIELD MEASUREMENTS

3.2.1 Radiological Measurements

Between July 6, 1999, and March 15, 2000, four radiological contamination surveys were performed on the 241-Z-361 tank, equipment, and areas surrounding the tank. The surveys were performed using a portable alpha monitor, measuring both direct and removable contamination.

The initial survey was completed July 6, 1999, and consisted of 30 direct measurements taken before, during, and after the installation of the helical piers. Ten measurements were taken before driving piers, 10 measurements were taken after the piers were started, and 10 measurements were taken after pier installation was complete. All direct alpha measurements were less than 500 disintegrations per minute (dpm) per/100 cm^2.

The second survey of tools, equipment, and dirt used in the installation of pilings for a bridge over the tank was performed on July 8, 1999. The direct alpha measurements were all less than 500 dpm/100cm^2.

A third survey of the tank and surrounding area for release from the Characterization Project Office to PFP was performed between March 9 and March 14, 2000. The items surveyed include Risers A through H, the eight riser locations for soil, and 36 nine square-meter grids (this includes soil above or around tank). The results of the survey are listed in Table 3-1.

Table 3-1. Radiological Survey Results for Risers A through H and Soil Associated with Tank 241-Z-361

Description	Total (dpm/100cm^2)		Removable (dpm/100cm^2)
Riser A	N/A	700	<20
Riser B	N/A	350	<20
Riser E	N/A	2100	<20
Risers C, D, F, H	N/A	0	<20
Riser G max	N/A	14,000	<20
36 3x3 meter grids	100	<500	NA
8 riser locations	N/A	<500	NA

Dose rate measurements on Risers A through H and on the 36 nine square-meter grids were less than 0.2 mrem/hr neutron dose and less than 0.4 mrem/hr deep dose (gamma).

A final survey of the nine bollards and post caps which surrounded the tank was completed on March 15, 2000. The direct alpha measurements of the nine bollards and post caps were all less than 500 dpm/100cm^2.

3.2.2 Field Vapor Measurements

3.2.2.1 Field Monitoring

All Phase II field monitoring activities were accompanied by continuous health physics and industrial hygiene technician support, as required by the sampling procedures and the Health and Safety Plan (EQM 1999a, Appendix C).

Industrial hygiene technicians monitored for the presence of flammable gases in the tank headspace and workplace air using a combustible gas meter. In addition to flammability monitoring, a photoionization detector (PID) was used to monitor for volatile organic compounds, Draeger tubes were used to monitor carbon tetrachloride and chloroform vapor, and a direct reading instrument was used to monitor ammonia.

Radiological monitoring of surfaces and workplace air was performed using alpha and beta/gamma survey instruments and continuous air monitors for workplace alpha contamination. These health and safety radiological surveys were not part of this DQA.

3.2.2.2 Radiation Release and Screening of Vapor Samples

Radiological screening of vapor samples was performed twice during the sampling/analytical sequence. The first screening was at the tank riser. The River Protection Project (RPP) Characterization Project Radiological Control released the SUMMA® canisters and particulate filters from the jobsite by direct measurement and measurement of smear samples. Radiation and contamination surveys were performed in accordance with HNF-IP-0718, Section 6.1, "Release Surveys for Materials and Equipment." No data were supplied for these activities; therefore, review of these data was not part of the DQA.

The second evaluation was the analysis of the particulate filters by an onsite, fixed laboratory at WSCF. The reason for the particulate filter radiological testing was to document that no particulate radioactivity was introduced into the sampling train media. Analysis of the particulate filters was performed by WSCF in accordance with Procedure LA-508-415.

The tank headspace vapor samples of various volumes were drawn through a set of two particulate prefilters (2-micron pore size). The laboratory analyzed particulate filters for total alpha, total beta, and gamma-emitting radionuclides (using GEA). From the sampling event of September 22, 1999, laboratory results from all filters show a maximum beta activity of 7.1 E-06 μCi per sample and a maximum alpha activity of 2.2 E-06 μCi per sample. The filters from the September 27, 1999, sampling event show a maximum beta activity of 4.0 E-06 μCi/sample and a maximum alpha activity of 1.4 E-06 μCi per sample. GEA results were not reported (WMH 1999a) for these samples. Evaluation of the data by Tank Farms radiological control personnel (WMH 1999a, Appendix B) concluded that the portions of the sampling system downstream of the filters would be considered releasable, per HNF-IP-0718 pending external surveys. No raw data or QC data were provided for these gross alpha and gross beta results, so no DQA was performed.

No gamma spectroscopy data were provided in the final data report (WMH 1999a, Appendix B); however, the project radiological control data reviewer stated that all gamma results were less than MDC, except for one. That one result was near the detection limit, deemed an anomaly by the reviewer, and dismissed.

3.2.2.3 Field Vapor Monitoring Results

Tables 3-2 to 3-4 provide the field monitoring results during different stages of the core sampling process. The raw data are provided in Appendix B.

Table 3-2. Field Monitoring Results of Initial Measurements at the Breather Filter and Riser F*

Location/ Survey No.	Date – Time	Analytes			
		TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
Breather filter/ 99-1367 and 99-1372	6/29/99 – 0945	NDR	0	18.8	NDR
	6/29/99 – 0950	4	NDR	NDR	NDR
	6/29/99 – 0952	NDR	NDR	NDR	<5
	7/1/99 – 0850	NDR	2	18.7	NDR
	7/1/99 – 0855	32.4	NDR	NDR	NDR
	7/1/99 – 0858	NDR	NDR	NDR	<5
Breather filter/ 99-1553	8/13/99 – 1400	4.3	0	18.8	0
Top of Riser F/ 99-1372	7/1/99 – 0920	NDR	1	18.7	NDR
	7/1/99 – 0925	5.1	NDR	NDR	NDR
	7/1/99 – 0927	NDR	NDR	NDR	<5
3 ft inside Riser F/ 99-1372	7/1/99 – 0930	NDR	2	19.1	NDR
	7/1/99 – 0933	5.8	NDR	NDR	NDR
	7/1/99 – 0935	NDR	NDR	NDR	<5

* Data from Survey ID: 99-1553, "Remove Liquid from Tank Dry Wells" and Survey ID: 99-1367 and 99-1372, "Riser Prep."

Table 3-3. Field Monitoring Results at Drill String Location During Core Sampling and Drill String Removal* (2 sheets)

Location/ Survey No.	Date	Time	Analytes			
			TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
Drill string during core sampling/ 99-1874	9/22/99	1355	0.5	0	20.5	<5
	9/22/99	1425	15.2	2	17.3	<5
	9/22/99	1445	0	2	20.5	<5
	9/22/99	1458	6.6	2	16.6	<5
	9/22/99	1511	3.4	2	17.8	<5
	9/22/99	1517	6.6	2	17.4	<5
	9/22/99	1839	3.4	2	18.5	<5
	9/22/99	1845	NDR	2	16.4	NDR
	9/22/99	1855	7.6	2	16.5	<5
	9/22/99	1855	NDR	2	16.3	NDR
	9/22/99	1911	8.4	2	14.3	<5
	9/22/99	1925	NDR	2	18.5	NDR
	9/22/99	1926	3.5	NDR	NDR	<5
	9/22/99	1943	6.2	2	16.2	<5
	9/22/99	1949	3.2	2	17.1	<5

Table 3-3. Field Monitoring Results at Drill String Location During Core Sampling and Drill String Removal* (2 sheets)

Location/ Survey No.	Date	Time	Analytes			
			TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
	9/22/99	1955	7.4	2	16.5	<5
	9/22/99	2000	4.2	1	14.5	<5
	9/22/99	2009	7.5	1	18.2	<5
	9/22/99	2032	7.5	2	17.6	NDR
	9/22/99	2038	7.1	0	11.1	NDR
	9/22/99	2048	8.1	1	12.5	NDR
	9/22/99	2056	6.2	3	12.5	NDR
	9/22/99	2117	3.3	3	16.4	NDR
	9/22/99	2120	0	3	17.2	NDR
	9/22/99	2133	1.7	3	16.3	NDR
	9/22/99	2128	5.1	NDR	6.2	NDR
Drill string during Removal/ 99-1877	9/23/99	1150	15	3	13	<5
	9/23/99	1200	1.3	1	20.6	<5
	9/23/99	1205	2.2	0	20.4	NDR
	9/23/99	1208	3.4	0	20.4	NDR
	9/23/99	1212	3.9	0	19.7	NDR
	9/23/99	1215	2.2	0	20.3	NDR
	9/23/99	1232	4.2	0	20.5	NDR
	9/27/99	NDR[1]	NDR[1]	NDR[1]	NDR[1]	NDR[1]

* Data from Survey ID: 99-1874 "Push Mode Sample 361-Z" and Survey ID: 99-1877, "Pull Drill String from 361-Z."

[1]No industrial hygiene survey data were supplied for sampling date 9/27/99.

Table 3-4. Field Monitoring Results at Various Locations* (2 sheets)

Location/Survey No.	Date	Time	Analytes			
			TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
Dry well/99-1553	8/13/99	1500	NDR	0	NDR	NDR
	8/13/99	1720	NDR	0	NDR	NDR
	8/13/99	1500	NDR	NDR	19.2	NDR
	8/13/99	1720	NDR	NDR	18.8	NDR
Dome space/99-1874	9/22/99	1110	2.7	2	19	<5
	9/22/99	1315	NDR	3	18.7	NDR
	9/22/99	1330	3.9	2	19	<5
	9/22/99	1345	NDR	3	18.9	NDR
	9/22/99	1400	NDR	3	18.7	NDR
	9/22/99	1415	NDR	2	18.6	NDR
	9/22/99	1430	NDR	3	18.5	NDR

Table 3-4. Field Monitoring Results at Various Locations* (2 sheets)

Location/Survey No.	Date	Time	Analytes			
			TOV (ppm)	CG (%LEL)	O_2 (%)	NH_3 (ppm)
	9/22/99	1445	NDR	2	18.5	NDR
	9/22/99	1500	NDR	2	18.5	NDR
	9/22/99	1515	NDR	2	18.5	NDR
	9/22/99	1830	NDR	2	18.5	NDR
	9/22/99	1845	NDR	2	18.5	NDR
	9/22/99	1900	NDR	2	19	NDR
	9/22/99	1915	NDR	2	18.5	NDR
	9/22/99	1930	NDR	2	19	NDR
	9/22/99	1945	NDR	2	14.3	NDR
	9/22/99	2001	NDR	1	19	NDR
	9/22/99	2005	NDR	1	19	NDR
	9/22/99	2030	NDR	1	19	NDR
	9/22/99	2045	NDR	3	19	NDR
	9/22/99	2100	NDR	1	19	NDR
	9/22/99	2115	NDR	2	18.5	NDR
	9/22/99	2130	NDR	3	19	NDR
Dome/ 99-1877	9/23/99	1145	3	2	18.3	<5
	9/23/99	1215	NDR	2	18.5	NDR
	9/23/99	1230	NDR	2	18.5	NDR
	9/27/99		NDR	NDR[1]	NDR	NDR[1]

*Data from Survey ID: 99-1553, "Remove Liquid from Tank Dry Wells" and Survey ID: 99-1874 "Push Mode Sample 361-Z" and Survey ID: 99-1877, "Pull Drill String from 361-Z."

[1]No industrial hygiene survey data were supplied for sampling date 9/27/99.

3.2.3 Laboratory Vapor Measurements

The purpose of the vapor sampling was to determine whether organic constituents are released in vapor form during the disturbance of sludge while sampling. The headspace vapor samples were collected from the sampling tube placed in Riser H of the tank during the preliminary Phase I headspace sampling described in Section 2.3. One headspace vapor sample was collected during the collection of each sludge core segment as described in the TSAP (EQM 1999a). The purpose of collecting these vapor samples was to determine whether or not changes in the headspace vapor constituents might indicate the presence of substantial quantities of volatile organic compounds within the sludge. The results indicate that no dramatic changes in headspace volatile concentration occurred during the sludge sampling effort. Although this was not a direct measurement of the volatile organic content of the sludge, the data indicate that no large volume of volatile compounds was disturbed and released during the sludge sampling.

Table 3-5 shows the sequence of sampling activities for the system, along with sample collection times. Cleanliness of the system was verified through collection and analysis of a field blank. The field blank included all sampling components and consisted of ambient air collected through the sampling components into the canister. One ambient vapor SUMMA® sample was taken per core as a field blank. Table 3-5 identifies the collection of six discrete SUMMA® canister samples during the sampling event.

Table 3-5. List of Samples and Quality Control for the System

Sample Code	Sample/Activity Description	Sampler Position During Collection	Sampling Duration
Amb-01	Field blank/one ambient SUMMA® sample per core	At the vented riser, not connected to tank riser sampling location	1 min
Risers #1 through 5	Collect SUMMA® samples during collection of 5 segments from selected risers	At riser sampling location	1 min

3.2.3.1 Analytical Requirements for Tank Vapor Samples

Vapor samples collected from Tank 241-Z-361 were analyzed for selected compounds in accordance the requirements in Table 3-6 as specified in the TSAP (EQM 1999a).

Table 3-6. Chemical Analytical Requirements for Tank Vapor Samples

CAS #	Analyte	Procedure	Prep	Container	PQL	Precision[1]	Accuracy
--	Gamma Emitters	not specified[2,3]	None	Particle filter	1 µCi/ filter	not specified	not specified
--	Radon Daughters	LA-508-415[3]	None	Particle filter	1 µCi/ filter	not specified	not specified
127-18-4	1,1,2,2-Tetrachloroethene	GC/MS 8260B LA-523-404[3]	None	SUMMA®	5 ppbv	± 25%	70-130%
120-82-1	1,2,4-Trichlorobenzene						
67-64-1	2-Propanone (Acetone)						
71-43-2	Benzene						
56-23-5	Carbon tetrachloride						
67-66-3	Chloroform						
75-09-2	Dichloromethane						
71-36-3	n-Butanol						
76-01-7	Pentachloroethane						
108-88-3	Toluene						
79-01-6	Trichloroethylene						
75-69-4	Trichloromonofluoro-methane						
1330-20-7	Xylene						

1. As specified in the Phase I Vapor Sampling SAP (Hill et. al. 1998)
2. Specifications from Phase II TSAP (EQM 1999a)
3. WSCF Laboratory used procedure LA-508-462.

CAS # = Chemical Abstract Services number

3.2.3.2 Vapor Sample Results

The volatile organic compounds detected in the headspace vapor samples are summarized in Table 3-7. This table includes compounds that were tentatively identified as well as those for which calibrated quantitation was performed. The summary in Table 3-7 does not include compounds that were detected in laboratory blanks or were otherwise identified by the laboratory to be of uncertain quality. These can be found in the summary laboratory report (WMH 1999b).

One ambient condition blank, or field blank (identified by the sample identifiers AMB-xx), was collected before the vapor sampling associated with each sludge core. The sample identifiers identify the riser from which a sludge core was being removed at the time of the vapor sample collection and the specific sequential core segment associated with the vapor sample. For example, the identifier 00F-01 indicates the vapor sample associated with the first segment of the core from Riser F.

3.2.4 QA/QC Summary and Data Quality Assessment

This assessment was performed on data presented in the completed data report provided by the laboratory (WMH 1999a). The assessment includes an evaluation of the following parameters:

- Precision through evaluation of duplicate analyses
- Accuracy through evaluation of standard recovery, instrument performance, and analyte quantitation
- Representativeness of the sampling and analysis action
- Completeness of the sampling and analysis
- Comparability of the analytical data to previous information

Table 3-8 is a summary of the QC parameter review for these samples. For those parameters with an "X" in the "DQA Result" column, further discussion is provided in this section.

3.2.4.1 Precision

Sample V98018-23-00E-01 was analyzed in duplicate by the laboratory. Positive detection of ten compounds was reported in each duplicate sample. The RPD for the concentrations reported in the duplicate pairs ranged from 1% to 22%. All but one of the reported RPDs were less than 20%. The one RPD exceeding 20% was for acetone, which was detected in the field blank. The measurement precision between the duplicate sample analyses is adequate and indicates good repeatability between laboratory measurements.

3.2.4.2 Accuracy

The data report indicates that analytes were quantified using six-point calibration with the exception of acetonitrile, which was quantified using a four-point calibration. This does not affect the project data because acetonitrile was not a compound of interest and was not detected in the samples. Standard recoveries were reported to be within the laboratory's control limits. CCV was generally within limits. CCV standards which were out of limits were for compounds which were not of interest to the project.

Table 3-7. Summary of Volatile Organic Compounds[1] Detected in Headspace Samples (2 sheets)

Compound (CAS #) / Samples	AMB-E[2]	00E-01	00E-02	00E-03	00E-04	00E-05	AMB-F[3]	00F-01	00F-02	00F-03	00F-04	00F-05
	Sample Results (all units ppbv; duplicate analyses in parenthesis)											
N-butane (106-97-8)	ND	110 (91)	120	120	120	120	ND	94	79	31	55	99
Freon 11 (75-69-4)	ND	760 (815)	820	820	820	820	ND	810	830	240	430	810
N-pentane (106-66-0)	ND	6.9 (6.8)	7.6	8.0	7.9	8.6	ND	5.6	5.3	ND	ND	7.4
Dichloromethane (75-09-2)	***ND***	***18 (15)***	***19***	***20***	***19***	***19***	***ND***	***21***	***20***	***6.9***	***12***	***22***
2-Methyl-pentane (107-83-5)	ND	8.9 (7.6)	9.7	10	10	10	ND	6.8	6.5	ND	5	8
Chloroform (67-66-3)	***ND***	***1000 (1050)***	***1100***	***1100***	***1100***	***1100***	***ND***	***1100***	***1100***	***320***	***570***	***1100***
Carbon tetrachloride (56-23-5)	***ND***	***140 (135)***	***150***	***150***	***150***	***150***	***ND***	***140***	***140***	***49***	***79***	***150***
Tetrachloroethylene (127-18-4)	***ND***	***1900 (1959)***	***2000***	***2000***	***2000***	***1900***	***ND***	***2000***	***2000***	***320***	***1100***	***2000***
1-Butanol (71-36-3)	***ND***	***37 (30)***	***40***	***40***	***38***	***38***	***ND***	***29***	***29***	***12***	***18***	***30***
Acetone (67-64-1)	***9.1***	***6.7 (5.4)***	***ND***	***ND***	***9.4***	***5.2***	***9.1***	***7***	***ND***	***8.8***	***16***	***ND***
	Tentatively Identified Compounds[4]											
Isobutane (75-28-5)	ND	220	520	500	220	460	ND	430	350	75	200	220
2,2-dimethyl propane (463-82-1)	ND	64	110	120	74	110	ND	110	80	ND	74	64
2-Methyl-butane (78-78-4)	ND	ND	98	92	ND	93	ND	73	73	ND	ND	ND
2,2-dimethyl butane (75-83-2)	ND	94	100	110	100	100	ND	83	83	ND	ND	89
1,2-Dichloroethylene (540-59-0)	ND	63	67	68	64	63	ND	58	54	ND	ND	63
Methylcyclopentane (96-37-7)	ND	180	200	200	190	190	ND	160	150	ND	85	160
Trichloroethylene (79-01-6)	***ND***	***850***	***880***	***880***	***830***	***820***	***ND***	***740***	***730***	***350***	***520***	***750***
Dichlorofluoromethane (75-43-4)	ND	ND	58	ND	ND	55	ND	ND	ND	ND	ND	ND
1,1,2-trichloro-1,2,2-trifluoroethane (76-13-1)	ND	ND	ND	ND	ND	ND	ND	ND	ND	48	43	ND

Table 3-7. Summary of Volatile Organic Compounds[1] Detected in Headspace Samples (2 sheets)

Compound (CAS #) / Samples	AMB-E[2]	00E-01	00E-02	00E-03	00E-04	00E-05	AMB-F[3]	00F-01	00F-02	00F-03	00F-04	00F-05
	Sample Results (all units ppbv; duplicate analyses in parenthesis)											
C3 Aliphatic Hydrocarbon	ND	ND	ND	130	ND	200	ND	200	ND	ND	ND	ND
C4 Aliphatic Hydrocarbon	ND	210	ND	ND	280	110	ND	ND	710	ND	ND	250
Unknown	ND	110	110	110	110	110	ND	93	91	ND	45	100

[1]Analytes in ***bold italics*** text are the target analytes listed in Table 3-6.
[2]AMB-E = Ambient Blank Sample V9018-021-AMB-01, associated with Riser E vapor samples
[3]AMB-F = Ambient Blank Sample V9019-091-AMB-01 associated with Riser F vapor samples.
[4]Concentration estimated
ND = not detected above reporting limit

Table 3-8. QA/QC Summary for Phase II VOC Vapor Samples in SUMMA® Canisters

QC Parameter	DQA Result
Blanks	X
Calibration	O*
Internal standards	X
RPD	O*
LCS	O
Completeness	X
Comparability	O
Representativeness	O

O = QC parameter is within required limits
O* = QC parameter is within required limits with notation
X = QC parameter is not within required limits

Response of the chlorobenzene-d5 internal standard was outside the laboratory's 50 to 150% recovery limit in some dilutions. Response for the fluorobenzene internal standard was consistently within limits. The laboratory method (method number LA-523-404) specifies that TIC concentrations will be estimated using the chlorobenzene-d5 response factor. The concentrations of TICs were estimated using the response factor for the fluorobenzene internal standard. The impact of estimating TIC concentrations using one internal standard versus the other is not clear, but likely has small effect on the estimated concentrations. The impact of low chlorobenzene-d5 response on quantitation of other compounds is not discussed in the data report narrative. A low response for internal standards may result in overestimating analyte concentrations.

The data were quantified using two separate sets of mass spectrometer integration parameters: one for the polar compounds and another for nonpolar. This was not discussed in the data report narrative, but was explained by laboratory staff during this DQA. Because the two separate integrations were performed consistently from initial calibrations through continuing calibration verification and the quantification of the unknowns, there is no apparent adverse effect on the data presented. In addition, the internal standard summary tables were not presented for the polar calculations.

The instrument was not calibrated for all compounds of interest for the project. Numerous TICs were identified in the samples, including trichloroethylene which was identified as a compound of interest for the sampling and analysis activity based on having been reported previously as a TIC during the Phase I vapor sampling and analysis. The concentrations of TICs are qualitative estimates, because no calibration standard was used for these compounds.

The reported quantitation limits for the compounds analyzed are consistent with the requirements of the TSAP (EQM 1999a) and are sufficiently low. The samples were appropriately analyzed at additional dilutions to quantify high concentration analytes. No residual out-of-range chromatographic peaks were identified in the diluted samples.

Apparently, contamination of blank air by n-decane produced some anomalous chromatographic responses at secondary dilution. A small peak was observed in numerous sample chromatograms at a retention time of approximately 35 minutes in the initial 2× sample dilution. In the subsequent 48× dilutions, a magnified peak is observed at the same retention time. This is consistent with the increased mass of n-decane in the diluted sample resulting from contamination in the laboratory blank air.

Some of the diluted samples exhibited a larger response for a compound with a retention time of approximately 35 minutes than reported in undiluted samples. The compound was identified as octamethylcyclotetrasiloxane by the spectral library. The compound response did not conflict with target analyte responses. This compound is most likely column bleed from a degrading column. The compounds 1-butanol (3.3 ppbv) and 2-octanone (12 ppbv) were also detected in a lab blank on October 14, 1999. 1-Butanol was detected in the vapor samples, but was not flagged as such on the data summary pages. 2-Octanone was not detected in any of the samples. Acetone was detected in field blanks and sporadically in tank vapor samples at a similar concentration. The reported acetone appears to be blank-related in all cases. These data defects do not have any significant effect on the data or its use.

Internal standard area counts for chlorobenzene-d5 were low and not within the required 50 to 150% limits set by the laboratory for some of the samples. The low recovery of chlorobenzene-d5 did not affect the quantitation of the target analytes present in the samples. All target analytes detected were quantitated relative to the fluorobenzene internal standard. Fluorobenzene internal standard recoveries were consistently within required limits.

3.2.4.3 Representativeness

Based on the sampling method, the analytical results appear to be representative of the headspace of the tank near the sludge surface. The vapor sampling port established in Riser H of the tank includes a vapor sampling tube which extends from the riser top to within 12 in of the sludge surface. Most of the volatile contaminants of potential concern in Tank 241-Z-361 have vapor densities greater than 1 (density of air = 1); therefore, these vapors would be expected to settle and be present at their highest concentration near the sludge surface. This sampling location should enhance the potential for detection of dense vapor constituents.

This sampling approach was never considered sufficiently sensitive to detect small changes in sludge volatile content or to detect disturbance of small quantities of volatile liquids. Similarly, the approach was not intended to directly correlate to any specific VOC content in the sludge. The purpose of the sampling was rather to quantify changes in the headspace volatile concentration that may have been affected by disturbance of the sludge and to provide additional identification of volatile contaminants of concern. To this end, the headspace vapor sampling and analysis indicated that no large release of volatile compounds resulted from the sludge disturbance, and the contaminants identified were consistent with the list of contaminants detected in the original headspace vapor samples.

All samples submitted to the laboratory were analyzed for volatile organic compounds. Not all requested contaminants of concern identified in the sampling and analysis plan were analyzed quantitatively, however. Of the 13 volatile compounds requested for analysis in Table 3-6, 10 were analyzed quantitatively. Of the remaining three compounds requested, one (trichloroethylene) was reported as tentatively identified with an estimated concentration. Trichloroethylene, a common solvent, was reported as a TIC in the Phase I vapor sampling results and was specifically requested for quantitative analysis during this activity. It was again reported as a TIC. The other two compounds (1,2,4 -trichlorobenzene and pentachloroethane) were not reported or quantified in the data report. The specific compounds identified in the sampling and analysis plan for quantitative analysis and the number of those compounds reported in the analytical report are shown in Table 3-9. This results in an analytical completeness of 77% for the specific compounds requested for analysis.

Table 3-9. Contaminants of Concern Quantified in Analyses

Compound Requested in SAP (CAS #)	Detected in Vapor?	Quantified?
1,1,2,2-Tetrachloroethene (127-18-4)	Not reported	No
1,2,4-Trichlorobenzene (120-82-1)	Not reported	No
2-Propanone (Acetone) (67-64-1)	Yes	Yes
Benzene (71-43-2)	No	Yes
Carbon tetrachloride (56-23-5)	Yes	Yes
Chloroform (67-66-3)	Yes	Yes
Dichloromethane (75-09-2)	Yes	Yes
N-Butanol (71-36-3)	Yes	Yes
Pentachloroethane (76-01-7)	Not reported	No
Toluene (108-88-3)	No	Yes
Trichloroethylene (79-01-6)	Yes (TIC)	No (TIC)
Trichloromonofluoromethane (Freon 11) (75-69-4)	Yes	Yes
Xylene (1330-20-7)	No	Yes

Environmental Quality Management (EQM) was informed (telephone communication, Markus Stauffer to Chuck Miller, no date) that the laboratory had not analyzed for those compounds for the following reasons:

- The compounds are not on the "standard" analytical method list for vapors at WSCF;
- The laboratory has no standards for those compounds; and
- The standards would be prohibitively expensive to obtain.

3.2.4.4 Comparability of the Analytical Data

The vapor analyses for the Tank 241-Z-361 project were performed using common analytical instruments and calibration techniques. The data are reported in units of ppbv and in units of milligrams per cubic meter (mg/m^3). These reporting units are common units and allow direct comparison to previous vapor concentration measurements. The results reported are consistent in magnitude to the results of the Phase I and Phase II tank vapor sampling results.

The analysis of the Phase II vapor samples collected during sludge sampling produced a larger list of tentatively identified compounds than was reported in the Phase I vapor results. The reason for this difference is not apparent.

After reviewing the laboratory method LA-523-404 and comparing it to the reference method (EPA Method TO-14A), variations in calibration and quantification were identified. Although laboratory method LA-523-404 appears to be a valid analytical method, there are differences between the methods and a dramatically different target analyte list. Therefore, it should be noted that the method used was EPA Method TO-14 as modified by LA-523-404.

3.2.4.5 Documentation

The analytical data package contains copies of the chain-of-custody (COC) documents prepared for the project. The documents clearly identify the analytical sample numbers and the date and time of sample collection. Documentation was adequate.

3.2.4.6 Conclusions

Although some deficiencies in the data package were identified (as described in the preceding sections), the vapor analytical data appear to be adequate for the intended purpose. The intended uses were to identify contaminants of potential concern for subsequent sludge analysis and to evaluate the potential for release of substantial quantities of volatile compounds during disturbance of the sludge by the core sampling.

The most notable deficiencies in the data package are related to incomplete quantitative analysis of the compounds specified in the sampling and analysis plan. This deficiency does not necessarily jeopardize assessment of the tank contents.

The headspace vapor data indicate that no large quantity of volatile organic compounds was displaced and/or released by the sludge disturbance during core sample collection.

3.3 SLUDGE CORE SAMPLE PREPARATION

Two sludge core samples were collected from Tank 241-Z-361 using a push mode core sampling apparatus provided and operated by Lockheed Martin Hanford Company (now CH2M Hill Hanford Group) personnel. The core sampling device is truck-mounted and provides a hydraulic power source to push the core sampler into the sludge material. The core samples were collected in individual samplers that contain an intact core segment approximately 2.54 cm (1.0 in) in diameter and 48 cm (19 in) in length. The sludge depth was determined to be approximately 212 cm (84 in) thick at the locations sampled (i.e., near the middle of the tank). Four full core segments and one partially-filled segment were collected from each sample location. Each core segment, contained in an individual core sampler, was transported to the Hanford 222-S Laboratory to be extruded from the samplers, described, and prepared for chemical analysis. The extrusion process, preparation of composite samples, and results of chemical analysis of sludge are described in the Sections 3.3 through 3.7. Details of the extrusion process, alpha activity analysis of individual strata, and preparation of sludge composite samples for subsequent analyses are described in EQM (1999c).

3.3.1 Sample Extrusion

The core sample segments collected from Tank 241-Z-361 were extruded from the core samplers using remote manipulators inside the shielded hot cell at the 222-S Laboratory. As the segments were extruded, the visual characteristics (i.e., color, texture, and apparent wetness) were described and any unique strata were identified. The thickness of each stratum was measured and the strata were subsequently collected in separate sample jars. Duplicate subsamples were collected from each stratum sample for determination of total alpha activity. The total alpha activity analysis was intended to identify strata of exceptionally high alpha activity (i.e., plutonium content) and also to allow the laboratory staff to determine sample handling requirements. The results of the total alpha activity analyses of the strata are presented in Section 3.3.2, below.

Nineteen visually unique strata were identified during extrusion of core segments from Core 263 (Riser E). Eleven unique strata were identified during extrusion of the core segments from Core 264 (Riser F). For Core 264 (Riser F), a 32-cm (12.5-in) long zone of no sample recovery (i.e., unfilled core sampler) extending from approximately 85 cm (33.5 in) to 117 cm (46 in) below the sludge surface was noted.

Figure 3-1 is a graphical presentation of the approximate thickness and relative position of the strata identified in Cores 263 and 264. The qualitative descriptions of the sludge presented in Figure 3-1 are based on visual observations during core segment extrusion.

3.3.2 Total Alpha Activity Analysis

The push mode core segments collected between September 22 and September 27, 1999, were analyzed for total alpha activity by the 222-S Laboratory. The samples were analyzed in accordance with the TSAP (EQM 1999a). The purpose of this analysis was to determine which strata could be composited. Composite analysis results are discussed in Sections 3.4 through 3.7 of this report. All precomposite data and logic for compositing are discussed in the compositing plan (EQM 1999c).

Core 263 consisted of five core segments removed from Tank 241-Z-361, Riser E, on September 22, 1999. From the five core segments, 19 samples were taken for total alpha analysis, one from each visually unique stratum that was identified during extrusion. Each of the 19 samples had two subsamples taken for analysis, thereby providing duplicate analysis on each stratum.

Core 264 consisted of five core segments removed from Tank 241-Z-361, Riser F, on September 27, 1999. From the five core segments, 11 samples were taken for total alpha analysis, one from each visually unique stratum that was identified during extrusion. Each of the 11 samples had two subsamples taken for analysis, thereby providing duplicate analysis on each stratum.

3.3.2.1 Total Alpha Data Quality Assessment

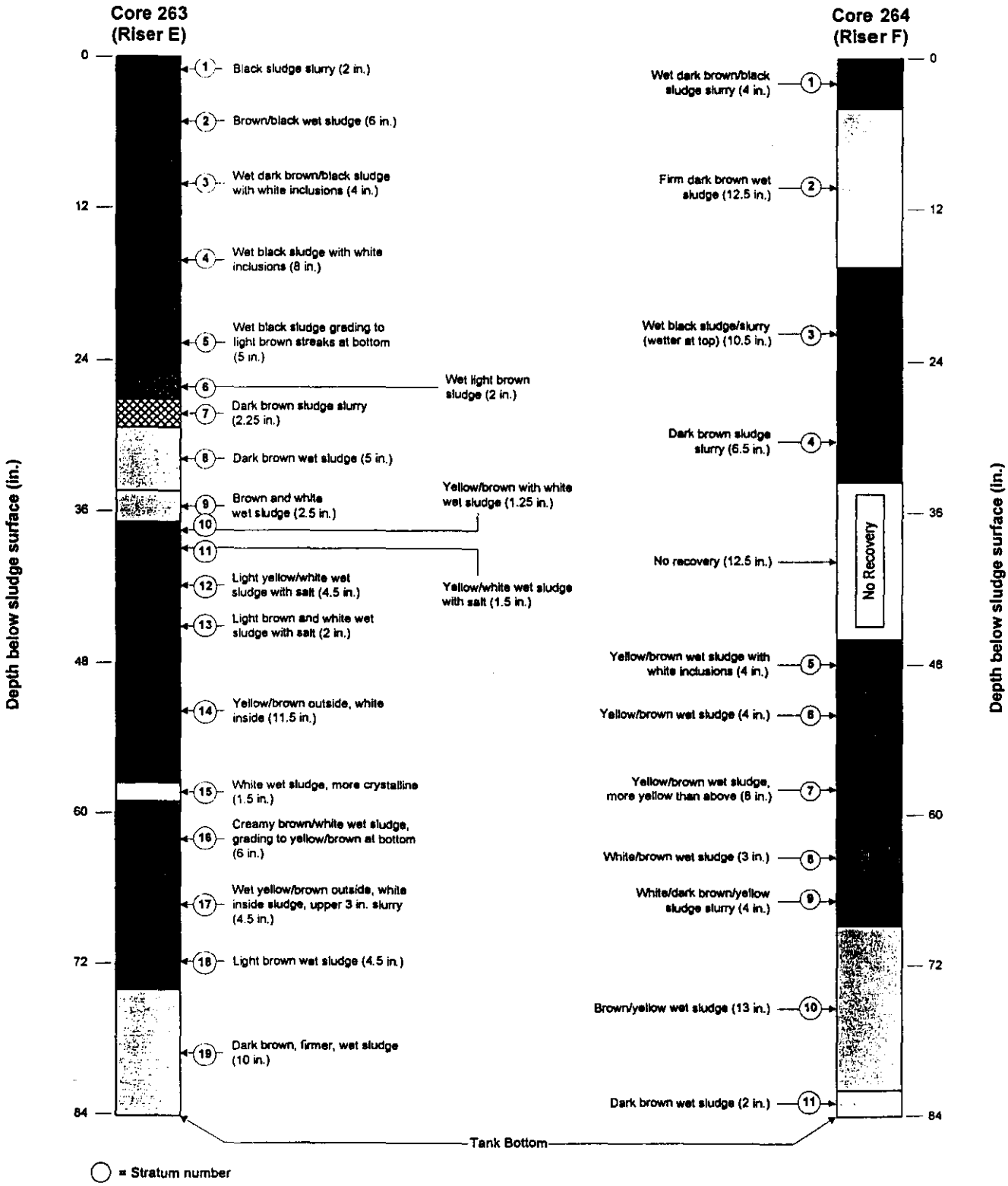
The total alpha activity concentration measurements (generated by 222-S Laboratory) were evaluated in a manner consistent with American Society for Testing and Materials (ASTM) Standard D6233-98 (ASTM 1998). A project-specific DQA checklist was developed for this project to facilitate rapid data assessment. The objective of the assessment was to determine the usability of the total alpha analyses to support the preparation of composite samples for further analysis.

The assessment, using the checklist as a guide, indicated that the total alpha data generated for this project met the analytical quality assurance requirements specified in the sampling and analysis plan, with some exceptions as documented in the following sections.

3.3.2.2 Sample Receipt

No discrepancies were noted in sample receipt. All samples were documented on the COC form; the COC was signed and dated with time of receipt recorded. All samples were analyzed for total alpha as specified in the TSAP (EQM 1999a). Sufficient documentation exists to trace the samples analyzed to a specific core, segment, and stratum. No discrepancies were identified in sample labeling, packaging, or handling upon sample receipt, and analytical holding times for total alpha analysis were met.

Figure 3-1. Strata Identified in Cores 263 and 264 During Extrusion



3.3.2.3 Quality Control

Method-specific quality control is discussed in the following sections.

Laboratory Control Samples (LCSs). All LCSs were within control limits and run at the proper frequency of at least one for every 20 samples (5% of samples) or one for every analytical batch. However, the LCSs were not taken through the preparation step (i.e., the fusion process). Failure to perform preparation on the LCS does not satisfy the requirements of the TSAP (EQM 1999a). Comparison of the precomposite alpha QC sample with the post-composite QC sample results (Section 3.4.4) show both good precision and accuracy. The post-composite alpha analysis included both pre and postfusion LCSs. The results of which are well within the TSAP requirements. Therefore this has no impact on data usability.

Method Blanks. Method blanks were run at the proper frequency of at least one for every 20 samples (5% of samples) or one for every preparation batch. A low level of alpha activity was detected in four of the 15 laboratory method blanks. The background contamination exceeded the MDC of the instrument, affecting a total of 14 samples. The level of contamination is low compared to results (less than 1%) and has no significant effect on data quality.

Duplicates. Duplicates were run at the proper frequency of at least one for every 20 samples (5% of samples) or one for every preparation batch. The RPD for sixteen pairs of duplicate analyses ranged from 0.4 to 34.6% with only three duplicate pairs exceeding the RPD objective of less than 20%. The three exceedencies were 21.7, 23.2, and 34.6%. This level of precision is considered acceptable given the apparent heterogeneity of the sludge. In addition to the batch QC, the TSAP (EQM 1999a) required two subsamples from each stratum of each segment to be analyzed. The analysis demonstrated acceptable precision between replicate analyses for each stratum.

Matrix Spikes. All matrix spike (MS) recoveries were between 70 and 130 % and were run at the proper frequency of at least one for every 20 samples (5% of samples) or one for every preparation batch. However, the MSs were not taken through the preparation step (i.e., fusion). This does not satisfy the requirements of the TSAP (EQM 1999a). As discussed under the LCS section, the prefusion MSs run for the post-composite total alpha analysis are well within TSAP requirements. Therefore this has no impact on data usability.

Standards Preparation. During the assessment, several concerns were noted in the area of standard recertification. The standards (75B53-A through 75B53-F) used in the initial calibration of the detectors for the gas flow proportional instrument were created by the Standards Laboratory on May 12, 1995. According to the *Standards Laboratory Radiation Source Certificate* created for these standards, sources are certified for one year after the date of preparation. Laboratory procedure LO-150-061, *Criteria for the Recertification of Standards,* discusses criteria used to establish the expiration dates and recertify radioactive standards (i.e., 5 yr or 4 half-lives). This procedure indicates that the standards will be statistically evaluated annually and a certificate generated showing the date the certified value was evaluated and recertified. These standards were being used for initial calibrations of detectors 13, 14, 15,

and 16 for the gas flow proportional instrument. No documentation was presented to verify that recertification of the standards had been performed. The americium-241 standards used for the continuing calibration verification checks were prepared on January 12, 1993. Inadequate documentation existed for the process of radiochemical standard expiration and certification. However, a statistical review of the standards was performed by the laboratory which indicates an approximately 1% change in the calibration standards over time. Therefore, this does not have any significant impact on the data quality.

3.3.2.4 Instrument Calibration

Method-specific calibration is discussed in the following sections.

Initial Calibration. As discussed in the standard preparation section, the americium-241 efficiency calibration was performed on October 14, 1995, for detector 16 and on October 28, 1997, for detector 15. The efficiencies generated in 1995 and 1997 are used for the calculation of total alpha activity for the core samples. A current efficiency calibration is especially important because the source used was not recertified since 1995. The remaining two detectors had efficiency calibrations in 1999.

Continuing Calibration. Control charts reviewed during the assessment indicated that continuing calibrations, LCSs, and MS recoveries were in control during October and November 1999. With the exception of efficiency verification control charts provided for Detector 16, from 1996 to 1999, control charts covering a longer period of time (e.g., 6 months to a year) were not available. The control limits for the control charts are fixed limits and are established statistically using procedure LQ-150-001.

Background Counts. The laboratory met the following requirements of the TSAP (EQM 1999a) for instrument background acquisitions:

- Backgrounds are acquired daily on each detector used for sample analysis.
- Backgrounds are within laboratory control limits that were available for October and November 1999.

Cross Talk. During initial evaluations of the procedures, the issue of detector cross-talk assessment was raised. It was reported that one of the detectors in the laboratory exhibits significant beta-to-alpha cross talk, but that detector would not be used for Tank 241-Z-361 samples. The laboratory was unable to verify that the affected detector was not used for these samples.

3.3.2.5 Sample Information

The laboratory met the requirements of the TSAP (EQM 1999a) for sample identification. The detector identification and efficiency used on each sample, background counts, count duration, sample volumes, calculated sample activities, uncertainties and MDC values were all available for review. All detection limits were below the PQLs for total alpha as identified in the TSAP, and all sample results were substantially above the MDC. The total alpha results are presented in Table 3-10.

Table 3-10. Summary of Total Alpha Sample Results (2 sheets)

Core	Segment	Stratum*	Sample ID	Total Alpha Result (µCi/g)
263	1	1	S99Z000109	44.20
			S99Z000110	51.70
		2	S99Z000107	25.90
			S99Z000108	31.40
	2	3	S99Z000113	24.60
			S99Z000211	26.70
		4	S99Z000111	24.20
			S99Z000112	26.60
		5	S99Z000117	27.30
			S99Z000118	27.30
		6	S99Z000115	33.20
			S99Z000116	32.80
	3	7	S99Z000121	26.20
			S99Z000122	25.20
		8	S99Z000119	28.60
			S99Z000120	27.80
		9	S99Z000125	39.70
			S99Z000126	43.70
		10	S99Z000123	34.10
			S99Z000124	34.20
		11	S99Z000129	18.30
			S99Z000130	18.30
		12	S99Z000127	7.930
			S99Z000128	7.010
		13	S99Z000131	19.90
			S99Z000132	19.60
	4	14	S99Z000135	19.00
			S99Z000136	21.40
		15	S99Z000133	6.160
			S99Z000134	6.950
		16	S99Z000137	44.10
			S99Z000138	25.30

Table 3-10. Summary of Total Alpha Sample Results (2 sheets)

Core	Segment*	Stratum*	Sample ID	Total Alpha Result (µCi/g)
	5	17	S99Z000141	7.120
			S99Z000142	6.670
		18	S99Z000139	8.000
			S99Z000140	6.560
		19	S99Z000143	4.400
			S99Z000144	3.950
			S99Z000212	3.750
264	1	1	S99Z000167	22.50
			S99Z000168	21.30
		2	S99Z000165	14.00
			S99Z000166	13.90
	2	3	S99Z000169	29.10
			S99Z000170	28.50
	3	4	S99Z000171	31.00
			S99Z000172	29.30
	4	5	S99Z000175	25.60
			S99Z000176	22.40
		6	S99Z000173	31.50
			S99Z000174	35.10
		7	S99Z000179	24.20
			S99Z000180	25.40
		8	S99Z000177	35.20
			S99Z000178	41.70
	5	9	S99Z000183	23.00
			S99Z000184	23.10
		10	S99Z000181	2.550
			S99Z000182	2.480
		11	S99Z000185	8.720
			S99Z000186	8.730

*See Section 3.3.3 regarding numbering of strata.

3.3.2.6 Conclusions

Based on this evaluation of the checklist items, the net impact on the data usability appears to be as follows:

- LCSs and MSs were within control limits and indicate acceptable accuracy for each batch.
- Continuing calibration control charts reviewed indicate systems are in control.
- Analyses showed acceptable precision between duplicate and replicate analyses within each stratum.
- Data appear to be usable for assessment of relative variability among samples and can be used to support the compositing plan.

3.3.3 Composite Sample Preparation

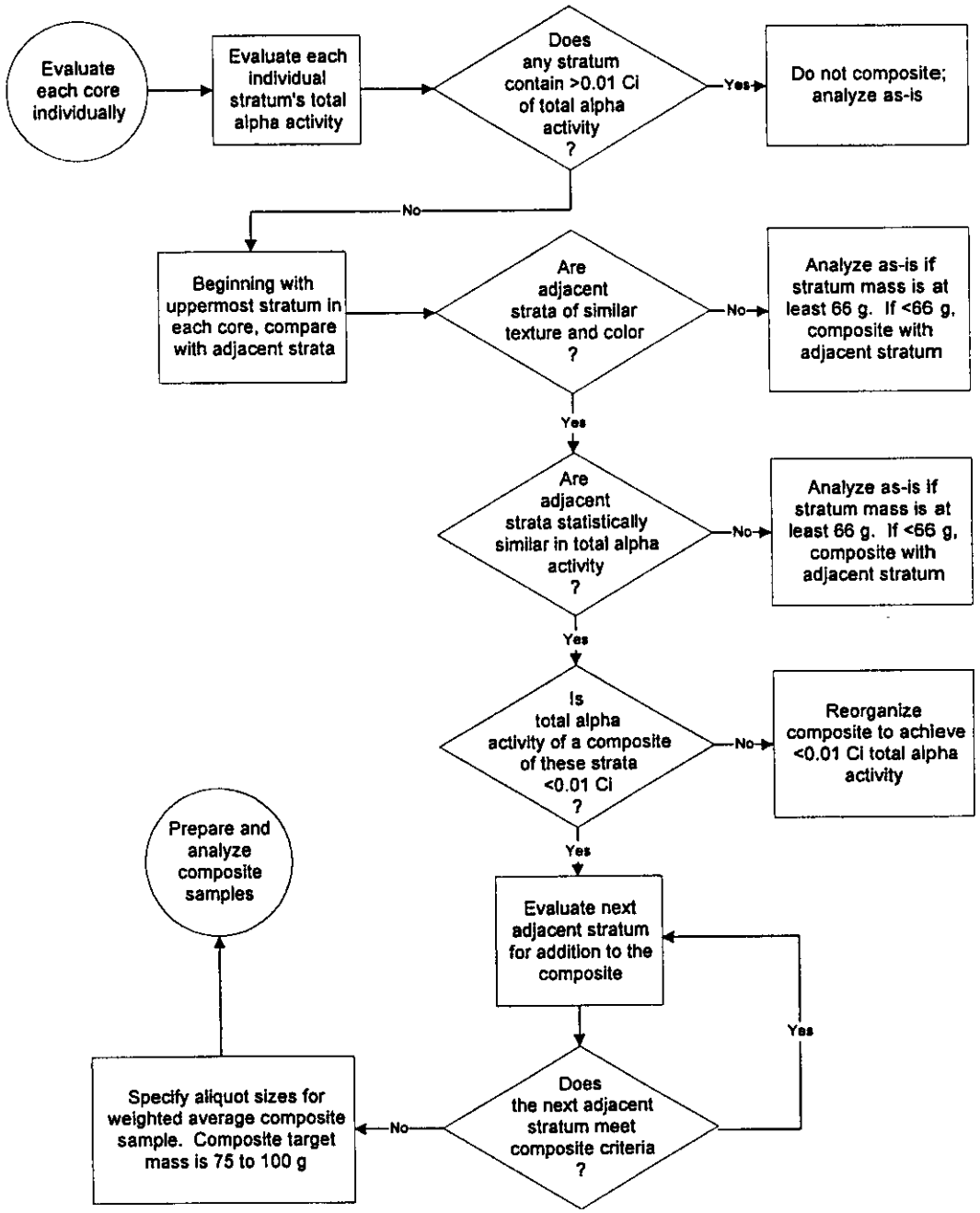
Composite samples were prepared from the strata identified in each core. Only adjacent strata in each core profile were considered for compositing. Strata to be composited were identified based on the logic shown in Figure 3-2 considering the following criteria:

- The laboratory limit for total alpha activity in an open container in the laboratory hoods is 0.01 Ci.
- The strata to be composited should be similar in color and texture.
- The strata to be composited should be statistically similar in alpha activity, based on the pointwise confidence intervals determined from the duplicate analyses of each stratum.

The minimum target composite mass required for complete analysis was 66 g of material, with a target composite mass of 75 to 100 g, to account for potential loss of sample to the container (i.e., material not readily recovered from the containers) and to allow for possible reruns.

A total of eight composite samples were defined for Core 263, and five composite samples were defined for Core 264. The composite samples were identified in sequential order from the top of the sludge surface downward. The sequential composite sample identifiers for each core (i.e., 263-1 through 263-8 and 264-1 through 264-5, respectively) are used throughout the remainder of this report. Descriptions of the composite samples and their constituent strata are described below (see Figure 3-1 for the relative position of the strata and initial numbering sequence).

Figure 3-2 . Logic for Compositing Strata for Radionuclide and Chemical Analyses



3.3.3.1 Core 263 Composite Samples

Composite 263-1 — Strata #1, #2, #3, and #4 are similar in color, texture, and alpha activity concentration. Strata #3 and #4 contain a few visible inclusions of varying color; however, the presence of such inclusions cannot be discounted in the other two strata. These four strata are combined on the basis of their similarity in color, texture, and alpha activity concentration.

Composite 263-2 — Strata #5, #6, #7, and #8 are similar in color, texture, and alpha activity concentration. Although it has a slightly higher alpha activity concentration than the other three strata, Stratum #6 is too small to analyze individually. Therefore, it is combined with the other three strata.

Composite 263-3 — Strata #9 and #10 are similar in color, texture and alpha activity concentration and are combined.

Composite 263-4 — Strata #11, #12, and #13 are similar in color and "salt" texture. Although the pointwise alpha activity concentration confidence intervals for Strata #11 and #13 do not overlap with Stratum #12, both #11 and #13 are each too small to analyze individually and, therefore, are combined with Stratum #12, based on their visual similarity.

Composite 263-5 — Strata #14 and #15 are similar in color. The strata vary slightly in texture and the pointwise alpha activity concentration confidence intervals do not overlap; however, Stratum #15 is too small to analyze individually and neither #14 nor #15 is similar to the overlying or underlying strata. Therefore, these two strata are combined.

Composite 263-6 — Stratum #16 is distinctly different in color and texture and the range of alpha activity concentration from the adjacent overlying and underlying strata. Because it is sufficiently large to be analyzed individually, Stratum #16 is not combined with any other strata.

Composite 263-7 — Strata #17 and #18 are similar in color, texture, and alpha activity concentration. Therefore, they are combined based on their similarities.

Composite 263-8 — Stratum #19 is distinctly different in color from the overlying adjacent stratum. Because it is sufficiently large to analyze individually, Stratum #19 is not combined with any other sample.

3.3.3.2 Core 264 Composite Samples

Composite 264-1 — Strata #1 and #2 are similar in color and texture. The total alpha activity pointwise confidence intervals do not overlap; however, Stratum #1 is not large enough to analyze individually. Therefore, it is combined with Stratum #2.

Composite 264-2 Strata #3 and #4 are combined based on their similarity in color, texture and total alpha activity concentration.

Composite 264-3 Strata #5, #6, and #7 are similar in color and texture, Stratum #5 does have some white inclusions which are not described in Strata #6 and #7. The total alpha activity concentration in all three strata are similar; therefore, the three strata are combined based on their visual similarity and alpha activity.

Composite 264-4 Strata #8 and #9 are similar in color and texture. The pointwise confidence intervals for total alpha activity concentration do not overlap; however, Stratum #9 is too small to analyze individually. Therefore, it is combined with Stratum #8 based on visual similarity.

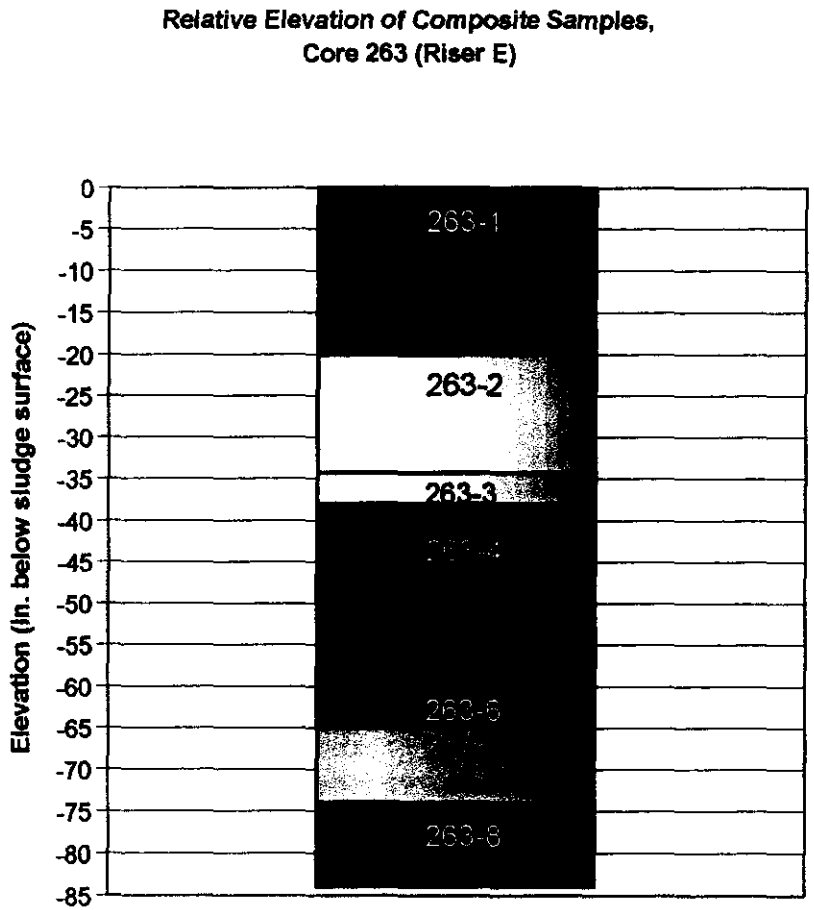
Composite 264-5 Strata #10 and #11 are similar in color and texture. The pointwise confidence intervals for total alpha activity concentration do not overlap; however, Stratum #11 is too small to analyze individually. Therefore, it is combined with Stratum #10.

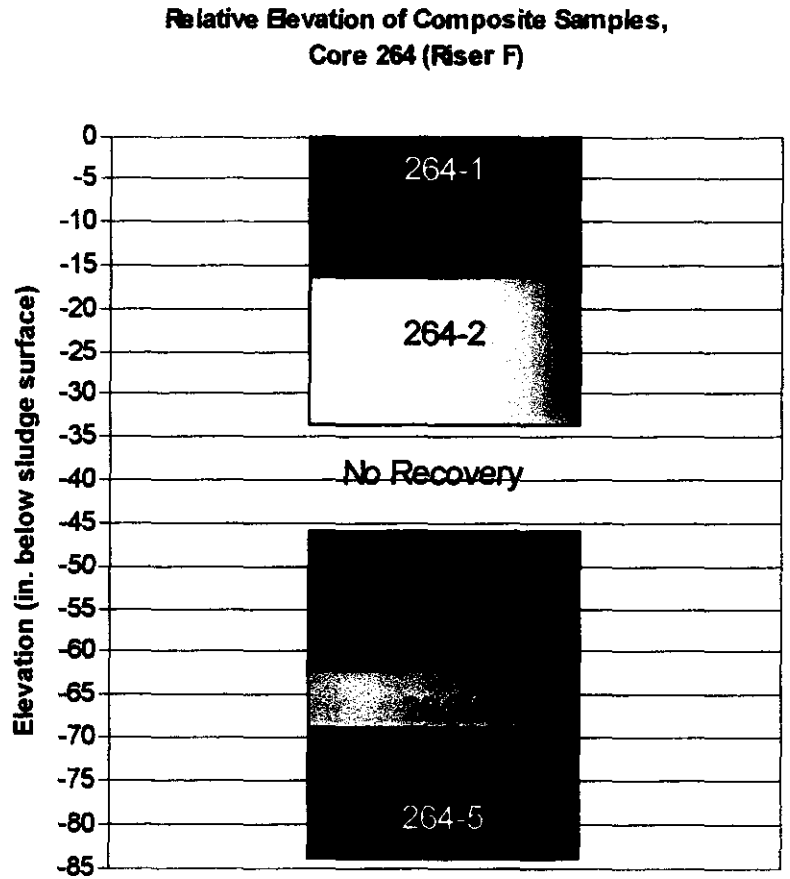
Table 3-11 and Figure 3-3 show the relative depths and thickness of the resulting composite samples in each core.

Table 3-11. Tank 241-Z-361 Sludge Composite Samples, Including Depth from Sludge Surface and Thickness

Sludge Composite Sample	Depth (below surface of sludge)				Composite Sample Thickness	
	Top		Bottom			
	(cm)	(in.)	(cm)	(in.)	(cm)	(in.)
Core 263 (Riser E)						
263-1	0	0	51	20	51	20
263-2	51	20	87	34.25	36	14.25
263-3	87	34.25	97	38	10	3.75
263-4	97	38	117	46	20	8
263-5	117	46	150	59	33	13
263-6	150	59	165	65	15	6
263-7	165	65	188	74	23	9
263-8	188	74	213	84	25	10
Core 264 (Riser F)						
264-1	0	0	42	16.5	42	16.5
264-2	42	16.5	85	33.5	43	17
No Recovery	85	33.5	117	46	32	12.5
264-3	117	46	157	62	41	16
264-4	157	62	175	69	18	7
264-5	175	69	213	84	38	15

Figure 3-3. Depth of Sludge Composite Samples for Cores 263 and 264 for Tank 241-Z-361





The sludge was observed to be 2.13 m (84 in) thick at the two locations where core samples were collected. The sludge surface is approximately level, indicating that the sludge thickness at the north (i.e., inlet) and south (i.e., outlet) ends of the tank is approximately 2.00 m (79 in) and 2.30 m (90 in), respectively. The calculated volume of wet sludge remaining in Tank 241-Z-361 is approximately 67 m^3 (2366 ft^3, 17,698 gal).

3.4 RADIOLOGICAL ANALYSES OF SLUDGE COMPOSITE SAMPLES AND SUMMARY OF QA/QC

3.4.1 Evaluation of Radionuclides

The concentration and distribution of the radionuclides in sludge from Tank 241-Z-361 were evaluated using laboratory analyses of two full-depth core samples collected from the sludge. The following sections present the results of these measurements.

3.4.2 Laboratory Analysis of Sludge Cores

As noted in Section 3.3.1, 19 distinct strata were identified in Core 263 and 11 strata in Core 264. Each stratum was analyzed for total alpha activity to ensure safe handling of the sludge samples in the laboratory and to develop a compositing plan (EQM 1999c). After evaluation of the total alpha analyses, strata of similar alpha activity and similar gross features (i.e., visual estimate of color and texture) were combined into composite samples for subsequent analyses in accordance with the sample compositing plan. Eight composite samples were prepared from Core 263, and five composite samples were prepared from Core 264. The composite samples are identified sequentially, proceeding from the surface of the sludge to the bottom of the tank, as composite samples 263-1 through 263-8 and composite samples 264-1 through 264-5, respectively.

3.4.3 Fusion Preparation

The 13 composite samples were divided among three preparation batches for fusion digestion for the radionuclide analysis. For each batch, the laboratory ran a postfusion method blank, a postfusion method standard, and a duplicate. The method blank was prepared using the same reagents as used for the samples. The method standard was prepared using the same reagents as used for the sample and spiking with the appropriate standard. For the duplicate, a second aliquot of a selected sample was taken after fusion and analyzed with the sample batch. In addition, a prefusion MS was prepared using a second aliquot spiked with an appropriate standard. For two of the three batches, a prefusion LCS was prepared.

In addition to the QC mentioned above, for the total alpha, total beta, neptunium-237, and inductively coupled plasma/mass spectrometry (ICP/MS) analysis, postfusion MSs were run. The QC results are further discussed within each analytical section. The evaluation of the QC data includes both preanalysis and postanalysis of QC data from the 222-S Laboratory. The following radiological analyses were performed on the sludge composite samples:

- Total alpha
- Total beta
- Strontium-90
- Technetium-99
- Neptunium-237
- Plutonium and americium by alpha energy analysis (AEA)
- Plutonium, americium, and uranium by ICP/MS

3.4.4 Total Alpha/Total Beta by Gas Proportional Counting

3.4.4.1 Laboratory Approach

Total alpha and total beta were performed by Method 9310 implemented by laboratory procedure LA-508-101. The total alpha/total beta analyses were performed on the fusion digest aliquots of the composite sludge samples in three analytical batches. Each analytical batch included method standard, method blank, duplicate, and postfusion MS samples. For total alpha, in addition to the QC samples mentioned above, the following QC samples were also run, a prefusion LCS and a postfusion spike sample were included in two of the three analytical batches.

Results of the total alpha and total beta analyses are presented in Tables 3-12 and 3-13.

3.4.4.2 Total Alpha and Total Beta QC Summary

Preanalysis Evaluation of QC Data. Before analysis of composite samples, EQM evaluated the QC data provided by the laboratory on January 23, 2000, which listed the yearly average and standard deviation for each of the Counter Control Standards (CCSs) for each detector. Qualitative examination of the QC results indicated that the standards had not degraded over time. The decay corrected activities for each of the CCS samples also were provided by the laboratory.

The 1999 average dpm for each of the eight detectors was compared to the known activity levels in the standard and percent biases from the expected levels were calculated. The percent bias for the detectors ranged from 2.2 to 14% for americium-241 and 10.6 to 19.8% for cesium-137.

Further discussion with the laboratory regarding the apparent positive bias revealed that it might be partially accounted for applying a laboratory derived ratio. The ratio is derived by comparing the efficiency for a small diameter source (the CCSs) to the calibrated efficiency, which is measured with a larger diameter source (like the samples). The sample result in dpm is calculated using the calibrated detector efficiency, which is derived using flood mount standards that are directly comparable to the samples.

On January 26, 2000, EQM recommended that it would be acceptable to count Tank 241-Z-361 samples for total alpha and total beta on the detectors listed above. If the positive bias observed in the CCS data could not be resolved by the application of the derived ratios, then the result would simply be a more conservative (i.e., higher) estimate of the alpha content of the samples.

Table 3-12. Total Alpha Results for Digested Sludge Composite Samples from Tank 241-Z-361

Composite	Composite Result (µCi/g)	Duplicate (µCi/g)	Detection Limit (µCi/g)	Counting Error (%)
263-1	37.4	N/A	0.00828	1.10
263-2	36.2	34.6	0.00803	1.10
263-3	42.6	N/A	0.00825	1.02
263-4	18.9	N/A	0.00827	1.54
263-5	25.4	N/A	0.0195	1.31
263-6	25.2	26.2	0.0195	1.31
263-7	7.3	N/A	0.0195	2.47
263-8	3.87	N/A	0.019	3.30
264-1	16.9	15.7	0.0151	1.53
264-2	37.9	N/A	0.0147	1.00
264-3	30.2	N/A	0.0145	1.12
264-4	27.8	N/A	0.0151	1.19
264-5	6.33	N/A	0.0148	2.49

Table 3-13. Total Beta Results for Digested Sludge Composite Samples from Tank 241-Z-361

Composite	Composite Result (µCi/g)	Duplicate (µCi/g)	Detection Limit (µCi/g)	Count Error (%)
263-1	2.35	N/A	0.0228	2.30
263-2	2.13	1.96	0.0221	2.36
263-3	2.45	N/A	0.054	3.50
263-4	0.837	N/A	0.0228	4.02
263-5	1.38	N/A	0.0676	5.26
263-6	1.35	1.34	0.0677	5.38
263-7	0.454	N/A	0.0675	12.4
263-8	0.146	N/A	0.0658	33.3
264-1	2.21	2.12	0.0502	3.58
264-2	3.96	N/A	0.0487	2.56
264-3	2.42	N/A	0.0483	3.36
264-4	2.18	N/A	0.0503	3.63
264-5	0.552	N/A	0.0491	9.14

Postanalysis Evaluation of QC Data. The QC samples for total alpha and total beta analyses included method standard, blank, duplicate, prefusion LCS, and postfusion MS samples. In addition, the total alpha analysis had a prefusion MS. The QC data are summarized in Tables 3-14 and 3-15.

A low level of alpha activity was detected in each of the total alpha preparation blanks. The level of activity was less than 1.0% of the lowest sample result (3.87 μCi/g) and was considered insignificant. No beta activity was detected in the blanks.

The RPD of the duplicates for the total beta Worklist 32247 was 20.561%, which exceeded the TSAP RPD requirement of less than 20% (EQM 1999a). All samples in Batch 32247 were rerun on Worklist 32279, except for composite sample 263-3. No explanation was noted in the data package for not including this composite sample with the rerun. The rerun duplicate RPD was less than 20%, and met the TSAP requirement. The results for composite sample 263-3 for total beta should be qualified (J) estimated, due to failure of the initial duplicate. All alpha total duplicate RPDs were within 20% as required by the TSAP.

All laboratory control and MS standard recoveries (pre and postfusion) for both alpha total and beta total are within 70 to 130% as required by the TSAP (EQM 1999a). Evaluation of the QC samples indicate that with the exception of sample 263-3 which is flagged (J) estimated, the data meet the QA requirements specified in the TSAP.

3.4.5 Strontium-90 by Gas Proportional Counting

3.4.5.1 Laboratory Approach

The strontium-90 analysis was performed on the fusion digest aliquots of the sludge composite samples. Following fusion of each sample, the strontium-90 in the sample is isolated using the strontium-90 separation procedure LA-220-101. Then the isolated strontium-90 is counted on a gas proportional counter. Recovery of the strontium by using a natural strontium carrier is determined gravimetrically. Strontium results are presented in Table 3-16.

3.4.5.2 Strontium-90 QC Summary

Preanalysis Evaluation of QC Data. EQM examined the QC data provided by the laboratory, which listed the yearly average and standard deviation for each CCS for each detector for 1996 through 1999. Qualitative examination of the QC results indicated that the standards had not degraded over time. A cesium-137 CCS is used for each detector and is adequate to determine the relative stability of the detector. The laboratory uses a strontium-90 derived efficiency to calculate the strontium-90 activity in the sample. The true activity for the cesium-137 CCS was provided by the laboratory.

The 1996, 1997, 1998 and 1999 average dpm for each detector was compared to the known activity levels in the standard and percent bias was calculated. The percent bias for detectors 10 and 12 ranged from –4.51 to –8.05%.

According to the LCS control charts for strontium-90 from 1996 through 1999, the percent recovery ranged between 98.02 to 111.42%. The percent recovery over the four year period is approximately 100%, which indicates no bias of the sample results due to chemistry.

Postanalysis Evaluation of QC Data. Analysis was performed in three analytical batches. Each analytical batch included method standard, method blank, duplicate, and prefusion MS samples. A prefusion LCS was included in two of the three analytical batches. The results of the QC analysis are summarized in Table 3-17.

Worklist 32190 was rerun due to the duplicate RPD of 31.61%, which exceeded the TSAP limit of 20% (EQM 1999a). The rerun duplicate RPD was 63.296% and also exceeded the TSAP limit. The high RPDs were due to the low strontium-90 activity present in the samples.

The prefusion LCS for Worklist 32190 was rerun due to the high percent recovery (202%). The rerun LCS met TSAP requirements. However, not all the samples from worklist 32190 were rerun, only sample 263-6. Due to the results of the other standard recoveries being within limits, no action is required. All standard recoveries (pre and postfusion) for the run were within 70 to 130% as required by the TSAP (EQM 1999a).

Strontium-90 activity was detected in all blanks except the blank for Worklist 32190. Blank activity ranged from 1 to 1000% of the strontium-90 results reported for the samples. The activity in the blanks was higher than the activity observed in the samples.

Table 3-14. Total Alpha QC Sample Summary (2 sheets)

Sample Type	Worklist	Sample	Actual Result (µCi/mL)	Found Result (µCi/mL)	Recovery (%)
Standard	32157	WL32157-STD	2.28e-04	2.17e-04	95.175
Standard	32181	WL32181-STD	2.28e-04	2.19e-04	96.053
Standard	32182	WL32182-STD	2.28e-04	2.28e-04	100.00

Sample Type	Worklist	Sample	Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)
Blank	32157	WL32157-Blk	2.63e-02	51.5	1.51e-02
Blank	32181	WL32181-Blk	3.66e-02	38.1	8.28e-03
Blank	32182	WL32182-Blk	3.76e-02	44.0	1.95e-02

Sample Type	Worklist	Sample	Duplicate Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	Sample Result (µCi/g)	RPD (%)
Duplicate	32157	S99Z000314	15.7	1.6	1.52e-02	16.9	7.362
Duplicate	32181	S99Z000307	34.6	1.1	8.11e-03	36.2	4.520
Duplicate	32182	S99Z000311	26.2	1.3	1.96e-02	25.2	3.891

Sample Type	Worklist	Sample	LCS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	LCS Recovery (%)
LCS (prefusion)	32157	N/A	N/A	N/A	N/A	N/A
LCS (prefusion)	32181	S00Z000011	111	1.4	4.15e-02	78.4
LCS (prefusion)	32182	S00Z000013	117	1.4	9.79e-02	83.6

Table 3-14. Total Alpha QC Sample Summary (2 sheets)

Sample Type	Worklist	Sample	MS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	MS Recovery (%)
MS (prefusion)	32157	S00Z000010	44.1	3.1	1.67e-01	98.1
MS (prefusion)	32181	S00Z000012	64.1	2.7	8.92e-02	100.3
MS (prefusion)	32182	S00Z000014	49.6	3.2	2.14e-01	87.5

Sample Type	Worklist	Sample	Actual Result (µCi/g)	Found Result (µCi/g)	Recovery (%)
Spike (postfusion)	32157	SPK S99Z000314	5.05E-02	4.53E-02	89.703
Spike (postfusion)	32181	SPK S99Z000307	5.05E-02	4.25E-02	84.158
Spike (postfusion)	32182	SPK S99Z000311	5.05E-02	4.44E-02	87.921

Table 3-15. Total Beta QC Sample Summary (2 sheets)

Sample Type	Worklist	Sample	Actual Result (µCi/mL)	Found Result (µCi/mL)	Recovery (%)
Standard	WL32147 (Rerun)	WL32147-STD	3.37e-03	3.37e-03	99.407
Standard	WL32148	WL32148-STD	3.37e-03	3.46e-03	102.671
Standard	WL32149	WL32149-STD	3.37e-03	3.37e-03	100
Standard	WL32179	WL32179-STD	3.37e-03	3.35e-03	99.407

Sample Type	Worklist	Sample	Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)
Blank	WL32147 (Rerun)	WL32147-Blk	<3.29e-02	500.0	6.76e-02
Blank	WL32148	WL32148-Blk	<4.34e-02	208.9	5.02e-02
Blank	WL32149	WL32149-Blk	<3.80e-02	500.0	5.42e-02
Blank	WL32179	WL32179-Blk	<1.10e-02	500.0	2.28e-02

Table 3-15. Total Beta QC Sample Summary (2 sheets)

Sample Type	Worklist	Sample	Duplicate Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	Sample Result (µCi/g)	RPD (%)
Duplicate	WL32247 (Rerun)	S99Z000307	2.4	3.50	5.31e-02	2.95	20.561
Duplicate	WL32248	S99Z000314	2.12	3.68	5.06e-02	2.21	4.157
Duplicate	WL32249	S99Z000311	1.34	5.40	6.79e-02	1.35	0.743
Duplicate	WL32279	S99Z000307	1.96	2.48	2.24e-02	2.13	8.313

Sample Type	Worklist	Sample	Actual Result (µCi/g)	Found Result (µCi/g)	Percent Recovery (%)
Spike (postfusion)	WL32247 (Rerun)	SPK S99Z000307	1.36e-01	1.41e-01	103.676
Spike (postfusion)	WL32248	SPK S99Z000314	1.36e-01	1.47e-01	108.088
Spike (postfusion)	WL32249	SPK S99Z000311	1.36e-01	1.45e-01	106.618
Spike (postfusion)	WL32279	SPK S99Z000307	1.36e-01	1.31e-01	96.324

Table 3-16. Strontium-90 Results for Sludge Composite Samples from Tank 241-Z-361

Composite	Composite Result (µCi/g)	Duplicate (µCi/g)	Detection Limit (µCi/g)	Counting Error (%)
263-1	0.00829	N/A	0.000698	11.2
263-2	0.00562	0.00557	0.000672	14.2
263-3	0.00249	N/A	0.000687	26.1
263-4	0.0184	N/A	0.000683	6.85
263-5	0.0145	N/A	0.000746	8.19
263-6	0.000649	0.00125	0.000779	92.4
263-7	0.00319	N/A	0.000741	22.7
263-8	0.00236	N/A	0.000709	27.7
264-1	0.0306	0.0295	0.000795	5.55
264-2	0.044	N/A	0.000753	4.42
264-3	0.0187	N/A	0.000746	7.08
264-4	0.00556	N/A	0.000771	15.6
264-5	0.00709	N/A	0.000758	13.0

The laboratory reported that, since no activity was detected in the blanks during total beta analysis, the contamination was introduced during the strontium-90 analysis. Because of the high activity of americium in the samples, precipitation of the alpha emitters was performed twice to ensure no interference from alpha in the beta counting. The activity in many of the blanks was greater than that observed in the samples. Therefore, due to the blank contamination, the actual sample results may be lower than they actually are. All sample and blank activity was above the PQL of 0.2 µCi/g listed in the TSAP, except for composite sample 264-1 which had an average strontium-90 activity of 3.01E-02 µCi/g.

The evaluation of the QC samples indicates that, because of the blank contamination found in all blanks, the strontium-90 results should be flagged (J) estimated for samples 263-1, 263-2, 263-3, 263-4, 264-1, 264-2, 264-3, 264-4. Sample 263-6 should be flagged (UJ).

3.4.6 Technetium-99 by Liquid Scintillation Counting

3.4.6.1 Laboratory Approach

Technetium analysis was performed on the fusion digest aliquots of the sludge composite samples. Following laboratory procedure LA-438-101, the sample is digested, then technetium-99 is extracted from the aqueous acid solution using a 30% Aliquot-336 in xylene. The organic phase is washed with nitric acid and sodium hydroxide to remove most interfering metals. The washed organic phase is then added to scintillation cocktail, the cocktail is placed in a scintillation counting vial and counted. Results for the technetium-99 analyses are presented in Table 3-18.

Table 3-17. Strontium-90 QC Summary (2 sheets)

Sample Type	Worklist	Sample	Actual Result (µCi/mL)	Found Result (µCi/mL)	Recovery (%)
Standard	32161	WL32161-STD	8.23e-04	7.83e-04	95.140
Standard	32189	WL32189-STD	8.23e-04	8.04e-04	97.691
Standard	32190 (rerun)	WL32186-STD	8.23e-04	8.27e-04	100.486
Standard	32375	WL32375-STD	8.22e-04	7.56e-04	91.971

Sample Type	Worklist	Sample	Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)
Blank	32161	WL32161-Blk	4.62e-04	89.4	8.01e-04
Blank	32189	WL32189-Blk	7.94e-03	37.6	3.45e-03
Blank	32190 (rerun)	WL32190-Blk	<1.84e-03	500.0	3.78e-03
Blank	32375	WL32375-Blk	6.53e-03	49.5	3.91e-03

Sample Type	Worklist	Sample	Duplicate Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	Sample Result (µCi/g)	RPD (%)
Duplicate	32161	S99Z000314	2.95e-02	5.7	7.95e-04	3.06e-02	3.661
Duplicate	32189	S99Z000307	5.57e-03	14.3	6.77e-04	5.62e-03	0.894
Duplicate	32190 (rerun)	S99Z000311	3.81e-03	20.5	7.76e-04	2.77e-03	31.611
Duplicate	32375	S99Z000311	1.25e-03	51.0	7.74e-04	6.49e-04	63.296

Table 3-17. Strontium-90 QC Summary (2 sheets)

Sample Type	Worklist	Sample	MS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	MS Recovery (%)
MS (prefusion)	32161	S00Z000010	6.81e-02	3.6	7.89e-04	84.5
MS (prefusion)	32189	S00Z000012	4.85e-02	4.0	6.72e-04	97.5
MS (prefusion)	32190 (rerun)	S00Z000014	5.23e-02	4.0	7.43e-04	110.8
MS (prefusion)	32375	S00Z000014	4.05e-02	4.7	7.64e-04	89.3

Sample Type	Worklist	Sample	LCS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	LCS Recovery (%)
LCS (prefusion)	32161	N/A	N/A	N/A	N/A	N/A
LCS (prefusion)	32189	S00Z000011	2.16e-01	4.3	3.48e-03	96.1
LCS (prefusion)	32190 (rerun)	S00Z000013	4.56e-01	3.0	3.83e-03	202.9
LCS (prefusion)	32375	S00Z000013	2.05e-01	4.7	3.89e-03	91.1

Table 3-18. Technetium-99 Results for Sludge Composite Samples from Tank 241-Z-361

Composite	Composite Result (µCi/g)	Duplicate (µCi/g)	Detection Limit (µCi/g)	Counting Error (%)
263-1	0.015	N/A	0.0014	5.44
263-2	0.0178	0.0203	0.00135	5.04
263-3	0.0279	N/A	0.00138	4.24
263-4	0.0134	N/A	0.00138	5.64
263-5	0.017	N/A	0.00152	5.17
263-6	0.00125	0.00172	0.000718	8.19
263-7	0.0119	N/A	0.00153	5.85
263-8	< 0.00149	N/A	0.00149	9.11
264-1	0.00349	0.00435	0.00139	8.06
264-2	0.0107	N/A	0.00122	5.79
264-3	0.0227	N/A	0.0014	4.6
264-4	0.0223	N/A	0.00143	4.68
264-5	0.00262	N/A	0.00143	8.5

Preanalysis Evaluation of QC Data. EQM examined the QC data provided by the laboratory, which included the control charts for the instrument control check standards and the technetium-99 LCSs for 1999. Qualitative examination of the QC results indicated that the standards have not degraded over time. The true activity for the technetium–99 standard (decay corrected) was provided by the laboratory.

The 1999 average dpm for each detector was compared to the known activity levels in the standard and percent biases from the expected levels were calculated. The percent bias for instrument LS 6000 was 0.35% and for instrument LS 6500, 0.53%.

According to the LCS control charts for technetium-99 from 1999, the mean percent recovery is 103.64%. This does not indicate any bias of the sample results due to the separation chemistry.

Postanalysis Evaluation of QC Data. The QC samples for this analysis include method standard, blank, duplicate, prefusion LCS, and prefusion MS samples. The QC data are summarized in Table 3-19.

All blank results were less than the detection limits. Two of the duplicate RPDs exceeded the liquid scintillation TSAP requirement of 15% (EQM 1999a). One for Worklist 32185 was 21.939%; the other for Worklist 32605 was 31.650%. For both samples, the high RPDs are attributed to low sample activity levels, only 2 to 4 times above detection limits. The laboratory reanalyzed composite sample 263-6 four times, with only the last set of results reported.

Table 3-19. Technetium-99 QC Summary (2 sheets)

Sample Type	Worklist	Sample	Actual Result (µCi/mL)	Found Result (µCi/mL)	Recovery (%)
Standard	32191	WL32191-STD	3.43e-03	3.10e-03	90.379
Standard	32192 (rerun)	WL32192-STD	3.43e-03	3.62e-03	105.539
Standard	32185	WL32185-STD	3.43e-03	3.75e-03	109.329
Standard	32605	WL32605-STD	3.43e-03	3.40e-03	99.125

Sample Type	Worklist	Sample	Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)
Blank	32191	WL32191-Blk	<6.91e-03	11.1	6.91e-03
Blank	32192 (rerun)	WL32192-Blk	<7.43e-03	10.0	7.43e-03
Blank	32185	WL32185-Blk	<1.47e-03	10.3	1.47e-03
Blank	32605	WL32605-Blk	<7.16e-04	9.5	7.16e-04

Sample Type	Worklist	Sample	Duplicate Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	Sample Result (µCi/g)	RPD (%)
Duplicate	32191	S99Z000307	2.03e-02	4.78	1.35e-03	1.78e-02	13.123
Duplicate	32192 (rerun)	S99Z000311	1.65e-02	5.21	not reported	1.63e-02	1.220
Duplicate	32185	S99Z000314	4.35e-03	7.730	1.41e-03	3.49e-03	21.939
Duplicate	32605	S99Z000311	1.72e-03	7.76	7.09e-04	1.25e-03	31.650

Table 3-19. Technetium-99 QC Summary (2 sheets)

Sample Type	Worklist	Sample	MS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	MS Recovery (%)
MS (prefusion)	32191	S00Z000012	6.35e-02	2.9	1.33e-03	89.8
MS (prefusion)	32192 (rerun)	S00Z000014	1.16e-01	55.9	1.49e-03	193.4
MS (prefusion)	32185	S00Z000010	5.32e-02	3.23	1.43e-03	97.1
MS (prefusion)	32605	S00Z000014	4.36e-02	2.49	7.08e-04	82.2

Sample Type	Worklist	Sample	LCS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	LCS Recovery (%)
LCS (prefusion)	32191	S00Z000011	2.71e-01	3.19	6.98e-03	104.5
LCS (prefusion)	32192 (rerun)	S00Z000013	3.02e-01	3.13e	8.00e-03	116.3
LCS (prefusion)	32185	N/A	N/A	N/A	N/A	N/A
LCS (prefusion)	32605	N/A	N/A	N/A	N/A	N/A

All standard recoveries (pre and postfusion) were within 80 to 120% for LCSs and 70 to 130% for MSs as required by the TSAP, except the MS for Worklist 32192, which had a recovery of 193.4%. The MS was rerun along with composite sample 263-6 and its duplicate. The recovery on the MS rerun was 82.2, which is within TSAP limits (EQM 1999a).

Evaluation of the QC samples indicates that, with the exception of two duplicate RPDs, the QC samples meet the requirements of the TSAP. Although it exceeded the TSAP requirements, the MS for the worklist was rerun and both the method standard and the prefusion LCS were within TSAP requirements (EQM 1999a). Therefore, no qualifiers need to be applied to the samples.

3.4.7 Neptunium-237 by Alpha Proportional Counting

3.4.7.1 Laboratory Approach

Neptunium analysis was performed on fusion digest aliquots of the sludge composite samples. After fusion of the sample, a chemical separation was performed using procedure LA-933-141.

The laboratory uses an efficiency of 0.500 in the calculation of sample activity for neptunium-237. This efficiency is established using an americium-241 standard. The alpha proportional counters (APCs) (1, 2, and 3) are calibrated to give an efficiency of 0.500 ± 0.020 by setting the system parameters appropriately. The system is then monitored by running a plutonium-239 CCS with each batch or once per week, whichever is more frequent. If the calculated efficiency does not fall within the range of 0.500 ± 0.020, the detector is tagged out of service. Therefore, the initial efficiency, as well as the efficiency for all batches analyzed is 0.500 ± 0.020. Because the detector is maintained within this specification, calculations are made using the average efficiency of 0.500. The results for neptunium-237 are presented in Table 3-20.

Table 3-20. Neptunium-237 Results for Sludge Composite Samples from Tank 241-Z-361

Composite	Composite Result (µCi/g)	Duplicate (µCi/g)	Detection Limit (µCi/g)	Counting Error (%)
263-1	0.00302	N/A	0.00368	77.5
263-2	0.00293	< 0.0024	0.00357	77.5
263-3	< 0.00196	N/A	0.00367	500
263-4	< 0.00229	N/A	0.00368	353
263-5	< 0.00225	N/A	0.00488	92.4
263-6	< 0.00225	< 0.00225	0.00488	132
263-7	< 0.00224	N/A	0.00487	92.4
263-8	< 0.00218	N/A	0.00475	86.9
264-1	< 0.00614	0.00729	0.00697	152
264-2	0.00523	N/A	0.00676	79.2
264-3	< 0.00404	N/A	0.00671	467
264-4	0.00426	N/A	0.00699	98.7
264-5	< 0.00601	N/A	0.00683	152

As reported by the laboratory, one sample (i.e., 264-1) had a result slightly over the detection limit. For four of the samples (263-1, 263-2, 264-2 and 264-4), positive neptunium–237 activity was detected at a level below the calculated detection limit. All other samples had no detectable neptunium-237 activity, and the results were reported as less than the calculated detection limit.

3.4.7.2 Np-237 QC Summary

Preanalysis Evaluation of QC Data. The 1999 averages of the APC detector efficiencies are as follows: 0.4963 for detector 1, 0.4906 for detector 2, and 0.4976 for detector 3. These averages are within the 0.500 ± 0.02 limits provided by the laboratory. According to the LCS control charts for neptunium-237 for 1999, the median percent recovery is 79.02% with statistical warning limits of 55.9 to 102.5% and out of control limits of 43.8 to 114.2%. Because the median recovery is approximately 80% (rather than 100%), this indicates a potential 20% average negative bias of the sample results. The TSAP lists a required recovery of between 70 and 130% for LCSs (EQM 1999a).

The cause of bias is most likely a combination of procedural losses specific to the neptunium-237 separation procedure and the use of americium-241 (alpha energy approximately 5.5 MeV) and plutonium-239 (alpha energy approximately 5.2 MeV) to establish the instrument counting efficiency. The alpha energy of neptunium-237 is approximately 4.8 MeV and the counting efficiency would be expected to be lower than that for americium-241or plutonium-239.

Postanalysis Evaluation of QC Data. Analyses were performed in three analytical batches. The QC samples are method standard, blank, duplicate, prefusion LCS, prefusion MS, and postfusion MS samples. The QC sample results are summarized in Table 3-21.

All blank results were less than the detection limits. Because all duplicate results and their associated samples were at or less than the detection limits, the RPD results are not applicable. Although the standard recoveries (both pre and postfusion) are negatively biased (between 83.1 and 72.0%), all are within 70 to 130% as required by the TSAP. Evaluation of the QC samples indicates that the data meet the TSAP requirements (EQM 1999a). No action required.

3.4.8 Plutonium Isotopic (Plutonium-238, Plutonium-239/240) and Americium-241 by Alpha Energy Analysis

3.4.8.1 Laboratory Approach

Plutonium isotopic analysis (plutonium-238 and plutonium-239/240) and americium-241 analysis were performed by methods LA-953-104 and LA-943-101, respectively. Sample results are presented in Tables 3-22, 3-23, and 3-24.

The plutonium and americium analyses were performed on the fusion digest aliquots of the sludge composite samples. The analyses were performed together in three analytical batches, but the plutonium and americium were reported on separate worklists.

Table 3-21. Neptunium-237 QC Summary (2 sheets)

Sample Type	Worklist	Sample	Actual Result (µCi/mL)	Found Result (µCi/mL)	Recovery (%)
Standard	32159	WL32159-STD	2.31e-02	1.74e-02	75.325
Standard	32185	WL32185-STD	2.31e-02	1.86e-02	80.519
Standard	32186	WL32186-STD	2.31e-02	1.87e-02	80.952

Sample Type	Worklist	Sample	Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)
Blank	32159	WL32159-Blk	<4.49e-03	351.6	6.97e-03
Blank	32185	WL32185-Blk	<1.39e-02	207.7	1.84e-02
Blank	32186	WL32186-Blk	<1.13e-02	221.9	2.45e-02

Sample Type	Worklist	Sample	Duplicate Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	Sample Result (µCi/g)	RPD (%)
Duplicate	32159	S99Z000314	7.29e-03	60.6	7.02e-03	<6.14e-03	N/A
Duplicate	32185	S99Z000307	<2.40e-03	285.4	3.61e-03	2.93e-03	N/A
Duplicate	32186	S99Z000311	<2.25e-03	73.5	4.90e-03	<2.25e-03	N/A

Table 3-21. Neptunium-237 QC Summary (2 sheets)

Sample Type	Worklist	Sample	LCS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	LCS Recovery (%)
LCS (prefusion)	32159	N/A	N/A	N/A	N/A	N/A
LCS (prefusion)	32185	S00Z000011	3.03e-01	10.1	1.84e-02	78.6
LCS (prefusion)	32186	S00Z000013	2.97e-01	10.7	2.45e-02	77.08

Sample Type	Worklist	Sample	MS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	MS Recovery (%)
MS (prefusion)	32159	S00Z000010	6.25e-02	11.3	6.98e-03	82.3
MS (prefusion)	32185	S00Z000012	6.51e-02	9.58	3.60e-03	82.5
MS (prefusion)	32186	S00Z000014	5.51e-02	11.1	4.87e-03	71.96

Sample Type	Worklist	Sample	Actual Result (µCi/g)	Found Result (µCi/g)	Recovery (%)
Spike (postfusion)	32159	SPK S99Z000314	2.31e-02	1.84e-02	79.654
Spike (postfusion)	32185	SPK S99Z000307	2.31e-02	1.70e-02	73.593
Spike (postfusion)	32186	SPK S99Z000311	2.31e-02	1.92e-02	83.117

Table 3-22. Plutonium-238 Results for Solid Samples

Composite	Composite Result (µCi/g)	Duplicate (µCi/g)	Detection Limit (µCi/g)	Counting Error (%)
263-1	2.3	N/A	1.75	4.09
263-2	< 1.7	< 1.66	1.7	6.57
263-3	< 2.25	N/A	2.25	5.92
263-4	< 0.968	N/A	0.968	6.12
263-5	< 1.14	N/A	1.14	6.14
263-6	< 1.12	< 1.16	1.12	6.61
263-7	< 0.377	N/A	0.377	6.62
263-8	< 0.201	N/A	0.201	100
264-1	< 0.67	0.659	0.67	4.98
264-2	< 1.68	N/A	1.68	4.78
264-3	< 1.54	N/A	1.54	7.41
264-4	< 1.52	N/A	1.52	7.53
264-5	< 0.331	N/A	0.331	7.09

Table 3-23. Plutonium-239/240 Results for Solid Samples

Composite	Composite Result (µCi/g)	Duplicate (µCi/g)	Detection Limit (µCi/g)	Counting Error (%)
263-1	26.8	N/A	1.75	1.7
263-2	28.6	27.2	1.7	1.64
263-3	42.4	N/A	2.25	1.52
263-4	17.7	N/A	0.968	1.53
263-5	23.7	N/A	1.14	1.49
263-6	23.8	25.4	1.12	1.5
263-7	7.26	N/A	0.377	1.53
263-8	4.21	N/A	0.201	1.5
264-1	9.28	9.35	0.67	1.74
264-2	29.1	N/A	1.68	1.63
264-3	26.5	N/A	1.54	1.63
264-4	26.1	N/A	1.52	1.67
264-5	6	N/A	0.331	1.6

Table 3-24. Americium-241 Results for Solid Samples

Composite	Composite Result (µCi/g)	Duplicate (µCi/g)	Detection Limit (µCi/g)	Counting Error (%)
263-1	14.1	N/A	1.47	2.31
263-2	13.9	13.3	1.35	2.34
263-3	8.84	N/A	1.26	2.69
263-4	3.86	N/A	0.648	2.95
263-5	4.67	N/A	0.495	2.59
263-6	4.26	4.15	0.483	2.64
263-7	1.35	N/A	0.186	2.96
263-8	0.306	N/A	0.0658	3.77
264-1	7.46	7.76	0.779	2.34
264-2	13.4	N/A	1.49	2.41
264-3	5.66	N/A	1.16	3.23
264-4	4.81	N/A	0.975	3.41
264-5	1.04	N/A	0.226	3.6

As reported by the laboratory, no tracers (plutonium-236 or americium-243) were added during the fusion preparation. Because multiple dilutions are made to the sample aliquot both during the fusion and separation procedures, if tracer were added prefusion, a large quantity of tracer would be required to provide an adequate counting response. The quantity of tracer needed is not readily available commercially and it would be extremely costly to purchase; therefore, the laboratory practice is to not add tracer during the fusion process. The recoveries of the LCS and MS were used to determine acceptable procedural recovery for sample batches.

After the fusion preparation, a radiochemical separation of the plutonium and americium was performed. The separated material was then mounted on a 1-in filter and counted. The counting process is twofold. First, the sample is counted for gross alpha. If this result shows significant alpha activity, the mount is then counted by alpha spectrometry. By calculating relative isotopic tracer fractions (tracer counts to the sum of all alpha counts determined by alpha spectrometry) and applying the tracer fractions to the results of the total alpha determination, the percent recovery of the tracers can be determined. This result is not reported, but is used by the analyst to monitor the separation performance and to identify any interferences with a particular sample matrix.

The gross alpha measurement is not used to calculate the reported analytical results. The analytical results are generated by using the known activity of the tracer added during the separation along with the relative isotopic tracer fraction and the fraction of isotope of interest (e.g., plutonium-239) to calculate activity for the isotope.

All plutonium-238 results were reported as less than the calculated detection limit, except for the duplicate results of composite sample 263-1. The plutonium-238 detection limits did not meet the PQL of 0.02 µCi/g listed in the TSAP (EQM 1999a) due to the large dilution required for the

high activity of plutonium-239/240 in the samples. Due to the fact that the plutonium-238 results were non-detects, this has no impact on data use. All plutonium-239/240 and americium-241 sample results were well above the PQL of 0.02 μCi/g listed in the TSAP and the calculated detection limits.

3.4.8.2 Plutonium Isotopic (Plutonium-238, Plutonium-239/240) and Americium-241 QC Summary

Preanalysis Evaluation of QC Data. Before analysis of the sludge composite samples, EQM examined the QC data provided by the laboratory, which included control charts for each detector for the alpha spectroscopy system and LCS control charts for the plutonium and americium for 1999. Qualitative examination of the QC results indicated that the standards had not degraded over time.

According to the LCS control charts for plutonium and americium from 1999, the median percent recovery for plutonium-239/240 was 103.2%, whereas the median percent recovery for americium-241 was 101.6%. No bias was indicated due to the separation chemistry. EQM examined control charts for the alpha spectroscopy instrumentation CCSs. These standards are counted with each batch or once per week, whichever is more frequent. The laboratory limit is ±5 channels from the expected channel. Each channel corresponds to 0.0046 MeV, so the five-channel range is approximately ± 0.023 MeV.

The backgrounds for the alpha spectroscopy are counted for eight hours once per week with the limit being four counts per channel. If the counts are higher than four per channel, the detector is tagged out of service until the background can be demonstrated to be back in control. Because the background on the alpha spectroscopy is maintained below four counts per channel, background is not subtracted from the sample analytical counts. Detectors with a background over four counts per channel are tagged out of service.

Postanalysis Evaluation of QC Data. The QC for this analysis are method standard, blank, duplicate, prefusion LCS and prefusion MS samples. The QC results are summarized in Tables 3-25 and 3-26.

The LCS and MS for plutonium-isotopic analysis were taken through the normal fusion and separation process and were spiked with plutonium-239. All other QC was started postfusion and taken through the separation process.

All blank results were less than the detection limits. All duplicate RPDs were within 20% required by the TSAP (EQM 1999a). All standard recoveries (pre and postfusion) were within 70 to 130%, as required by the TSAP.

Based on evaluation of the QC samples, no qualifiers need to be applied to the samples.

3.4.9 Plutonium, Americium, and Uranium by ICP/MS

3.4.9.1 Laboratory Approach

ICP/MS spectrometry analysis was performed on the fusion digest aliquots of the sludge composite samples. Results for uranium-235, uranium/plutonium-238, plutonium-240, and plutonium/americium-241 were determined directly from the fusion digest. The uranium results are reported in Tables 3-27 and 3-28.

Two sets of results were reported for plutonium-239, plutonium-240, and plutonium/americium-241 by the laboratory. The first set of results are presented in Tables 3-29, 3-31, and 3-33. Detection limits for these results did not meet the PQL of 2.4 μg/g listed in the TSAP because of the dilution required to reduce the potassium concentration from the fusion matrix (EQM 1999a). Because of criticality issues associated with the plutonium in the tank, the plutonium results were reanalyzed to lower the detection limits. In order to reduce the detection limits, the fusion aliquots were separated using the plutonium/americium chemical separation method. However, no tracers were added. The plutonium fraction of each sample collected from the separation was analyzed by ICP/MS. However, because tracers were not used, no method was available to determine whether differences in plutonium-239 and plutonium-240 results between alpha energy analysis (AEA) and ICP/MS were due to analytical uncertainties or losses during the chemical separation process. The results for chemically separated plutonium and americium are presented in Tables 3-30, 3-32, and 3-34.

The concentration of plutonium-241 also was calculated by applying isotopic ratios from the ICP/MS and AEA results. As discussed earlier, because tracers were not used, no method was available to determine whether differences in plutonium-239 and plutonium-240 results between AEA and ICP/MS analyses were due to analytical uncertainties or to losses during the chemical separation process. Therefore, for the plutonium-241 calculation, the chemically separated results obtained from the ICP/MS analyses (Tables 3-30 and 3-32) were used only to determine ratios of the isotopes.

The plutonium-241 calculations, as reported by the laboratory, are described below.

First, a result for plutonium-239 (μg/g) from AEA was calculated using the ratio of plutonium-239/plutonium-239 + plutonium-240 from the ICP/MS results:

1. The ICP/MS results (Tables 3-30 and 3-32) for plutonium-239 and plutonium-240 in units of μg/g were converted to units of μCi/g using the specific activities of 6.20e-02 μCi/μg for plutonium-239 and 2.27e-01μCi/μg for plutonium-240.

2. The converted ICP/MS results in units of μCi/g were summed (i.e., plutonium-239 + plutonium-240).

3. The ratio of (plutonium-239/plutonium-239 + plutonium-240) determined above was multiplied by the plutonium-239/240 μCi/g result from AEA (Table 3-23) to obtain an accurate result for plutonium-239 in μCi/g from the AEA results.

Table 3-25. Plutonium 239/240 QC Summary (2 sheets)

QC Type	Worklist	Sample	Actual Result (µCi/mL)	Found Result (µCi/mL)	Recovery (%)
Standard	32160	WL32160-STD	1.26e-04	1.25e-04	99.206
Standard	32187	WL32187-STD	1.26e-04	1.28e-04	101.587
Standard	32188	WL32188-STD	1.26e-04	1.26e-04	100.00

QC Type	Worklist	Sample	Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)
Blank	32160	WL32160-Blk	<3.41e-01	100	3.41e-01
Blank	32187	WL32187-Blk	<1.87	100	1.87
Blank	32188	WL32188-Blk	<1.66	100	1.66

QC Type	Worklist	Sample	Dup Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	Sample Result (µCi/g)	RPD (%)
Duplicate	32160	S99Z000314	9.35	1.8	6.58e-01	9.28	0.751
Duplicate	32187	S99Z000307	27.2	1.7	1.66	28.6	5.018
Duplicate	32188	S99Z000311	25.4	1.8	1.16	23.8	6.504

Table 3-25. Plutonium 239/240 QC Summary (2 sheets)

Sample Type	Worklist	Sample	LCS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	LCS Recovery (%)
LCS (prefusion)	32160	N/A	N/A	N/A	N/A	N/A
LCS (prefusion)	32187	S00Z000011	78.8	1.6	4.67	91.3
LCS (prefusion)	32188	S00Z000013	87.9	1.5	4.86	101.9

Sample Type	Worklist	Sample	MS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	MS Recovery (%)
MS (prefusion)	32160	S00Z000010	26.4	1.6	1.60	100.6
MS (prefusion)	32187	S00Z000012	44.3	1.6	2.26	91.5
MS (prefusion)	32188	S00Z000014	40.4	1.5	2.11	97.2

Table 3-26. Americium-241 QC Summary (2 sheets)

QC Type	Worklist	Sample	Actual Result (µCi/mL)	Found Result (µCi/mL)	Recovery (%)
Standard	32158	WL32158-STD	1.07e-04	1.12e-04	104.673
Standard	32183	WL32183-STD	1.07e-04	9.88e-05	92.336
Standard	32184	WL32184-STD	1.07e-04	1.23e-04	114.953

QC Type	Worklist	Sample	Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)
Blank	32158	WL32158-Blk	<3.36e-01	100	3.36e-01
Blank	32183	WL32187-Blk	<1.93	100	1.93
Blank	32184	WL32188-Blk	<1.29	100	1.29

QC Type	Worklist	Sample	Dup Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	Sample Result (µCi/g)	RPD (%)
Duplicate	32158	S99Z000314	7.76	2.45	2.45	7.46	3.942
Duplicate	32183	S99Z000307	13.3	2.45	13.3	13.9	4.412
Duplicate	32184	S99Z000311	4.15	2.63	4.15	4.26	2.616

Table 3-26. Americium-241 QC Summary (2 sheets)

Sample Type	Worklist	Sample	LCS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	LCS Recovery (%)
LCS (prefusion)	32158	N/A	N/A	N/A	N/A	N/A
LCS (prefusion)	32183	S00Z000011	40.6	2.18	3.92	75.7
LCS (prefusion)	32184	S00Z000013	57.3	2.19	4.32	106.7

Sample Type	Worklist	Sample	MS Result (µCi/g)	Relative Counting Error (%)	Detection Limit (µCi/g)	MS Recovery (%)
MS (prefusion)	32158	S00Z000010	18.2	2.75	1.52	101.4
MS (prefusion)	32183	S00Z000012	22.8	2.16	1.76	83.8
MS (prefusion)	32184	S00Z000014	17.0	2.32	1.44	119.4

Table 3-27. Uranium-235 by ICP/MS

Composite	Composite Result (µg/g)	Duplicate (µg/g)	Detection Limit (µg/g)
263-1	< 19.74	N/A	19.75
263-2	< 19.15	< 19.3	19.14
263-3	< 19.68	N/A	19.68
263-4	< 19.73	N/A	19.73
263-5	< 19.68	N/A	19.69
263-6	< 19.7	< 19.8	19.7
263-7	< 19.65	N/A	19.65
263-8	< 19.14	N/A	19.14
264-1	< 19.46	< 19.6	19.46
264-2	< 18.88	N/A	18.88
264-3	< 18.74	N/A	18.74
264-4	< 19.51	N/A	19.51
264-5	< 19.05	N/A	19.05

Table 3-28. Uranium-238 by ICP/MS

Composite	Composite Result (µg/g)	Duplicate (µg/g)	Detection Limit (µg/g)
263-1	49.85	N/A	19.75
263-2	21.38	23.4	19.14
263-3	20.83	N/A	19.68
263-4	32.56	N/A	19.73
263-5	21.49	N/A	19.69
263-6	< 19.7	< 19.8	19.7
263-7	26.36	N/A	19.65
263-8	24.09	N/A	19.14
264-1	58.37	56.4	19.46
264-2	62.62	N/A	18.88
264-3	34.04	N/A	18.74
264-4	21.13	N/A	19.51
264-5	32.23	N/A	19.05

Table 3-29. Plutonium-239 by ICP/MS*

Composite	Composite Result (µg/g)	Duplicate (µg/g)	Detection Limit (µg/g)
263-1	331	N/A	24.68
263-2	388	360	23.93
263-3	578	N/A	24.6
263-4	253	N/A	24.67
263-5	329	N/A	24.61
263-6	328	347	24.62
263-7	114	N/A	24.56
263-8	69.07	N/A	23.93
264-1	147	148	24.32
264-2	414	N/A	23.6
264-3	428	N/A	23.42
264-4	402	N/A	24.39
264-5	104	N/A	23.82

* These data were used in the criticality assessment (DOE-RL 2001).

Table 3-30. Chemically Separated Plutonium-239 by ICP/MS

Composite	Composite Result (µg/g)	Duplicate (µg/g)	Detection Limit (µg/g)
263-1	362	N/A	0.33
263-2	357	371	0.319
263-3	551	N/A	0.328
263-4	218	N/A	0.329
263-5	300	N/A	0.329
263-6	331	337	0.329
263-7	102	N/A	0.328
263-8	70.84	N/A	0.319
264-1	123	123	0.325
264-2	372	N/A	0.315
264-3	320	N/A	0.313
264-4	384	N/A	0.326
264-5	94.42	N/A	0.318

Table 3-31. Plutonium-240 by ICP/MS*

Composite	Composite Result (µg/g)	Duplicate (µg/g)	Detection Limit (µg/g)
263-1	25.5	N/A	24.68
263-2	<23.93	<24.2	23.93
263-3	37.23	N/A	24.6
263-4	<24.67	N/A	24.67
263-5	<24.6	N/A	24.61
263-6	<24.62	25.3	24.62
263-7	<24.56	N/A	24.56
263-8	<23.93	N/A	23.93
264-1	<24.32	<24.5	24.32
264-2	28.48	N/A	23.6
264-3	30.13	N/A	23.42
264-4	27.63	N/A	24.39
264-5	<23.82	N/A	23.82

* These data were used in the criticality assessment (DOE-RL 2001).

Table 3-32. Chemically Separated Plutonium-240 by ICP/MS

Composite	Composite Result (µg/g)	Duplicate (µg/g)	Detection Limit (µg/g)
263-1	45.41	N/A	0.33
263-2	40.95	44.1	0.319
263-3	60.97	N/A	0.328
263-4	25.28	N/A	0.329
263-5	30.15	N/A	0.329
263-6	31.13	30.8	0.329
263-7	10.19	N/A	0.328
263-8	3.543	N/A	0.319
264-1	15.21	15.2	0.325
264-2	44.23	N/A	0.315
264-3	33.51	N/A	0.313
264-4	36.28	N/A	0.326
264-5	8.459	N/A	0.318

Table 3-33. Plutonium/Americium-241 by ICP/MS

Composite	Composite Result (µg/g)	Duplicate (µg/g)	Detection Limit (µg/g)
263-1	<24.68	N/A	24.68
263-2	<23.93	<24.2	23.93
263-3	<24.6	N/A	24.6
263-4	<24.67	N/A	24.67
263-5	<24.6	N/A	24.61
263-6	<24.62	<24.7	24.62
263-7	<24.56	N/A	24.56
263-8	<23.93	N/A	23.93
264-1	<24.32	<24.5	24.32
264-2	<23.6	N/A	23.6
264-3	<23.42	N/A	23.42
264-4	<24.39	N/A	24.39
264-5	<23.82	N/A	23.82

Table 3-34. Chemically Separated Plutonium/Americium-241 by ICP/MS

Composite	Composite Result (µg/g)	Duplicate (µg/g)	Detection Limit (µg/g)
263-1	0.87	N/A	0.33
263-2	0.69	0.673	0.319
263-3	0.803	N/A	0.328
263-4	0.366	N/A	0.329
263-5	0.381	N/A	0.329
263-6	<0.329	0.332	0.329
263-7	<0.328	N/A	0.328
263-8	<0.319	N/A	0.319
264-1	<0.325	0.46	0.325
264-2	0.808	N/A	0.315
264-3	0.398	N/A	0.313
264-4	0.408	N/A	0.326
264-5	<0.318	N/A	0.318

The plutonium-239 result in µCi/g was converted to µg/g using the specific activity of *6.20e-02 µCi/µg*.

Next, the ratio of plutonium-241 (Table 3-34)/plutonium-239 (Table 3-30) was calculated from the ICP/MS μg/g results.

Finally, the ratio of plutonium-241/plutonium-239 calculated above was multiplied by plutonium-239 (μg/g) from AEA (from Step 4) to obtain the calculated result for plutonium-241 in μg/g.

The results of this calculation are presented in Table 3-35. Composite samples which had no detected concentration of plutonium-241 in the plutonium/americium separated samples were not included.

Some plutonium-240, all uranium-235 and plutonium/americium-241 results were less than detection limits. In addition, as discussed earlier, the PQL of 2.4 μg/g required in the TSAP (EQM 1999a) was not met. This was due to the dilution required. All uranium/plutonium-238 results were above detection limits, except for composite sample 263-6, which was a nondetect. Two sets of results were reported for plutonium-241. These results are presented in Table 3-35. The results in column one are the chemically separated results (also presented in Table 3-34). The results in the second column are the results calculated as discussed above.

Table 3-35. Plutonium-241 in Sludge Composite Samples from Tank 241-Z-361

Composite	Pu-241 Results from Pu/Am Sep./ICP/MS (μg/g)	Pu-241 Results Calculated from AEA* (μg/g)
263-1	8.70E-01	7.12E-01
263-2	6.90E-01	6.28E-01
263-3	8.03E-01	7.09E-01
263-4	3.66E-01	3.36E-01
263-5	3.81E-01	3.55E-01
264-2	8.08E-01	7.10E-01
264-3	3.98E-01	3.84E-01
264-4	4.08E-01	3.32E-01

* These data were used in the criticality assessment (DOE-RL 2001).

In comparing the two sets of analytical data generated for plutonium-239, plutonium-240, and plutonium-241, the results compare favorably. However, due to the absence of tracers to confirm the ion exchange column efficiency for the chemically separated radioisotopes, data from the unseparated runs for plutonium-239 and plutonium-240 were considered most representative and most conservative for use in the criticality assessment (DOE-RL 2001). The calculated plutonium-241 results presented in Table 3-35 were used in the criticality assessment.

3.4.10 Plutonium, Americium, and Uranium by ICP/MS QC Summary

Postanalysis Evaluation of the ICP/MS Uranium QC Data. The QC for this analysis are method standard, blank, duplicate, prefusion LCS, and prefusion MS samples. The QC data are presented in Table 3-36. The standard recoveries, spike recoveries and RPDs met the specifications in the TSAP (EQM 1999a). Low levels of uranium-235 and uranium-238 were detected in the preparation blanks. The level of uranium-235 and uranium-238 contamination were less than 0.6% of the uranium-238 sample results and the uranium-235 calculated detection limit. Because all uranium-235 sample results were nondetects, no results were included in the QC summary tables.

Postanalysis Evaluation of the Plutonium QC Data. The QC samples for this analysis are method standard, blank, duplicate, prefusion LCS, prefusion MS, and postfusion MS samples. The QC data are presented in Table 3-37. The standard recoveries, spike recoveries and RPDs met the specifications in the TSAP. Low levels of plutonium-239 were detected in the preparation blanks. The level of plutonium-239 contamination was less than 0.001% of the reported sample results. These levels were considered insignificant and do not impact data usability.

3.5 DATA VALIDATION SUMMARY OF SLUDGE COMPOSITE SAMPLES

EQM reviewed the data validation report for radiochemistry data package No. 241-Z-361-222S. The data validation was conducted in accordance with *Data Validation Procedure for Radiological Analysis* (WHC 1993). The validation report was compared against the requirements in the following sections of the data validation procedure: Total Alpha/Beta, Strontium-90, Alpha Spectrometry, Liquid Scintillation and ICP/MS Validation Requirements.

Because the laboratory performed the neptunium-237 analysis by alpha proportional counter, the criteria for gross alpha/gross beta were used to evaluate the data validation of neptunium-237.

Within each section, the following criteria were examined: initial calibration, continuing calibration, background counts, laboratory blanks, laboratory control or blank spike, laboratory duplicates, holding times, sample result quantitation samples, and minimum detectable activities.

For the initial calibration and continuing calibration check standards, the requirement is for standards to be National Institute of Standards and Technology (NIST)-traceable and certificates provided. This documentation was not provided with the data package; therefore, it could not be verified.

Table 3-36. ICP/MS Uranium-238 QC Summary (2 sheets)

Sample Type	Worklist	Sample	Actual Result (µg/mL)	Found Result (µg/mL)	Recovery (%)
ICV	32166	ICV-32166	0.0207	2.11e-02	101.932
	32179	ICV-32179	0.0207	2.09e-02	100.966
	32141	ICV-32141	0.0207	2.00e-02	96.618

Sample Type	Worklist	Sample	Result (µg/g)	Detection Limit (µg/g)
Blank-prep	32166	Blank-prep-32166	<0.0989	0.0989
Blank-prep	32179	Blank-prep-32179	<0.0989	0.0989
Blank-prep	32141	Blank-prep-32141	0.122	0.122

Sample Type	Worklist	Sample	Duplicate Result (µg/g)	Sample Result (µg/g)	RPD (%)
Duplicate	32166	S99Z000307	23.4	21.4	8.929
Duplicate	32179	S99Z000311	<19.8	<19.7	N/A
Duplicate	32141	S99Z000314	56.4	58.4	3.484

Sample Type	Worklist	Sample	LCS Result (µg/g)	LCS Recovery (%)
LCS (prefusion)	32166	S00Z000011	1025	102.5
LCS (prefusion)	32179	S00Z000013	976.6	97.7
LCS (prefusion)	32141	N/A	N/A	N/A

Table 3-36. ICP/MS Uranium-238 QC Summary (2 sheets)

Sample Type	Worklist	Sample	MS Result (µg/g)	MS Recovery (%)
MS (prefusion)	32166	S00Z000012	213.8	98.5
MS (prefusion)	32179	S00Z000014	182.4	84.6
MS (prefusion)	32141	S00Z000010	241.9	93.1

Sample Type	Worklist	Sample	Actual MS Result (µg/g)	Found MS Result (µg/g)	MS Recovery (%)
MS (postfusion)	32166	S99Z000307	0.020	1.89e-02	94.5
MS (postfusion)	32179	S99Z000311	0.020	1.92e-02	96.2
MS (postfusion)	32141	S99Z000314	0.020	2.00e-02	100.1

Table 3-37. ICP/MS Plutonium-239 QC Sample Summary (2 sheets)

Sample Type	Worklist	Sample	Actual Result (µg/mL)	Found Result (µg/mL)	Recovery (%)
ICV	32140	ICV-32140	1.00e-02	1.15e-02	115.0
	32165	ICV-32165	1.00e-02	1.10e-02	110.0
	32178	ICV-32178	1.00e-02	1.11e-02	111.0
	32449	ICV-32449	1.00e-02	1.06e-02	106.0
	32450	ICV-32450	1.00e-02	1.09e-02	109.0
	32451	ICV-32451	1.00e-02	1.03e-02	103.0

Sample Type	Worklist	Sample	Result (µg/g)	Detection Limit (µg/g)
Blank-prep	32140	Blank-prep/32140	<0.124	0.124
Blank-prep	32165	Blank-prep/32165	<0.124	0.124
Blank-prep	32178	Blank-prep/32178	<0.124	0.124
Blank-prep	32449	Blank-prep/32449	2.06E-04	2.06E-04
Blank-prep	32450	Blank-prep/32450	3.00E-04	3.00E-04
Blank-prep	32451	Blank-prep/32451	3.21E-04	3.21E-04

Sample Type	Worklist	Sample	Duplicate Result (µg/g)	Sample Result (µg/g)	RPD (%)
Duplicate	32140	S99Z000314	148	147	0.678
Duplicate	32165	S99Z000307	360	388	7.487
Duplicate	32178	S99Z000311	347	328	5.630
Duplicate	32449	S99Z000320	371	357	3.846
Duplicate	32450	S99Z000324	337	331	1.796
Duplicate	32451	S99Z000327	123	123	0.0

Table 3-37. ICP/MS Plutonium-239 QC Sample Summary (2 sheets)

Sample Type	Worklist	Sample	MS Result (µg/g)	MS Recovery (%)
MS (prefusion)	32140	S00Z000010	425.5	101.4
MS (prefusion)	32165	S00Z000012	633.0	90.8
MS (prefusion)	32178	S00Z000014	585.0	92.3

Sample Type	Worklist	Sample	LCS Result (µg/g)	LCS Recovery (%)
LCS (prefusion)	32140	N/A	N/A	N/A
LCS (prefusion)	32165	S00Z000011	128.8	92.5
LCS (prefusion)	32178	S00Z000013	1315	94.5
LCS (prefusion)	32449	S00Z000020	1203	86.46
LCS (prefusion)	32450	S00Z000019	1282	92.07
LCS (prefusion)	32451	N/A	N/A	N/A

Sample Type	Worklist	Sample	Actual Result (µg/g)	Found Result (µg/g)	Recovery (%)
Spike (postfusion)	32140	S99Z000314	0.02008	1.93E-02	96.116
Spike (postfusion)	32165	S99Z000307	0.02008	1.99E-02	99.104
Spike (postfusion)	32178	S99Z000311	0.02008	1.85E-02	92.131
Spike (postfusion)	32449	S99Z000320	0.02008	1.89E-02	94.124
Spike (postfusion)	32450	S99Z000320	0.02008	1.84E-02	91.633
Spike (postfusion)	32451	S99Z000320	0.02008	1.75E-02	87.151

The criteria used for data validation were compared against the TSAP to ensure the data met the TSAP requirements. The data validation procedure has the following requirements for laboratory duplicates:

"The relative percent difference (RPD) must be less than or equal to 20% for water samples ($\leq$35% for soils) if the sample concentration is greater than 5 times the RDL."

"For sample results less than five times the RDL, the range between the primary and duplicate sample results must be less than or equal to the RDL for water samples ($\leq$ 2× RDL for soils)."

Although the TSAP RPD requirements are $\leq$20% for AEA analysis, <15% for liquid scintillation, and <20% for GPC and ICP/MS, and the requirements for RDLs above are technically correct. The RPDs were reviewed against the above criteria in addition to TSAP requirements in determining data usability.

The TSAP requirements for LCS and MS % recovery were inserted into the data validation criteria and the data was reviewed against these criteria.

Because no RDLs were specified in the TSAP, when the validation criteria references RDL requirements, instrument detection limits were used for the purposes of the data review.

3.5.1 Total Alpha/Beta Data Validation Requirements and Summary

3.5.1.1 Initial Calibration

Verify that the counting system used for sample analysis meet the following criteria:

- *Each counting system used for sample analysis was efficiency calibrated within one year prior to sample analysis. If not calibrated within one year, then the continuing calibration requirements listed must be met.*
- *Calibration standards are NIST-traceable and certificates are provided.*
- *Self-absorption curves were prepared for each counting system from a series of planchets with weights ranging from 0 to 150 mg and the counting error for net counts is less than 5% for each planchet.*
- *Efficiency of each detector at 0% solids must be at least 20% or greater for either total alpha or gross beta.*

Two of the three detectors used for total alpha and beta analysis for this project were calibrated within one year before the analysis date. The third detector was last calibrated in October 1997. Daily reliability check results were control charted since the calibration date and show the stability of the detector efficiency since the last calibration.

Self-absorption curves were not generated for total alpha and total beta analyses. The high activities of the samples required dilutions of 100×; so no self-absorption curves were generated for total alpha and total beta. This was discussed with the laboratory by EQM prior to the start of analysis and has no impact on data usability.

The raw calibrations were examined and the detector efficiencies for the APCs and GPCs that were used for total alpha, total beta, strontium-90 and neptunium-237 were greater than or equal to 20% at zero percent solids.

3.5.1.2 Continuing Calibration

Verify that the continuing instrument calibration meets the following criteria:

- *Acceptable continuing calibration checks have been performed at least once per analytical run, sample batch, or daily, whichever is more frequent.*

- *Check standards are NIST-traceable and certificates are provided.*

All criteria were met.

3.5.1.3 Background Counts

Verify that the instrument background counts meet the following criteria:

- *Performed within one week prior to sample analysis.*
- *Performed on each detector used for sample analysis.*
- *Within the laboratory control limits.*

Daily detector background checks were performed on each detector and control charted. The checks were within limits.

All criteria were met.

3.5.1.4 Laboratory Blanks

Verify that the laboratory blank analysis meets the following criteria:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*

- *Prepared at the same time and analyzed with the samples using the same procedure.*

- *Results are less than or equal to the MDA and RDL.*

All criteria were met with the exception of the total alpha blanks (WL32157, WL32181 and WL32182). These blanks had total alpha activity that exceeded the detection limits. However, because the blank activity was less than 1% of the lowest sample result (3.87 μCi/g), no action was required.

3.5.1.5 Laboratory Control or Blank Spike Samples

Verify that the following criteria are met and that the laboratory provided the following information:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*
- *Prepared at the same time and analyzed with the samples using the same procedure.*
- *LCS activity is between 5 and 30 times the associated RDL value.*
- *The actual LCS concentration and the amount of spike added for the LCS were provided by the laboratory.*
- *Results are within the required TSAP recovery limits of 70% to 130%.*

All criteria were met.

3.5.1.6 Laboratory Duplicates

Verify that the following criteria are met and that the laboratory provided the following information:

- *The laboratory has conducted a duplicate analysis sample at frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.*
- *The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.*
- *The RPD must be less than or equal to 20% for water samples (≤ 35% for soils) if the sample concentration is greater than five times the RDL.*
- *For samples results less than five times the RDL, the range between the primary and duplicate sample results must be less than or equal to the RDL for water samples (≤2× RDL for soils).*

All criteria was met with the exception of the duplicate for total beta worklist 32247, which exceeded the 20% TSAP requirement. All samples in this batch were rerun with the exception of composite sample 263-3. The results for sample 263-3 for total beta should be qualified (J) estimated. All alpha total RPDs were within limits.

3.5.1.7 Holding Times

Verify that all samples were preserved properly (if applicable) and analyzed within 180 days.

All criteria were met.

3.5.1.8 Sample Result Quantitation and Minimum Detectable Activities

Verify the laboratory has reported the following information for each sample:

- *Sample identification*
- *Detector identification and efficiency*
- *Background counts*
- *Count duration*
- *Planchet weights*
- *Sample volume*
- *Alpha and beta crosstalk factors (if applicable)*
- *Calculated sample activities, uncertainties, and MDA values*
- *Required detection limits*

All criteria were met with the exception that no cross talk correction was used for the total alpha and beta analysis and no documentation was provided to the validator concerning cross talk correction. Because the MS results for the analysis indicate that the impact on sample results was negligible, no action was taken.

3.5.2 Strontium-90 Data Validation Requirements

3.5.2.1 Initial Calibration

Verify that the counting system used for sample analysis meet the following criteria:

- *Each counting system used for sample analysis was efficiency calibrated within one year prior to sample analysis. If not calibrated within one year, then the continuing calibration requirements listed must be met.*

- *Calibration standards are NIST-traceable and certificates are provided.*

- *Self-absorption curves were prepared for each counting system from a series of planchets with weights ranging from 0 to 150 mg and the counting error for net counts is less than 5% for each planchet. The calibration reference standard should be prepared from a traceable solution of Y-90, Sr-90, Sr-89, Tc-99, or Cs-137.*

- *Efficiency of each detector at 0% solids must be at least 20% or greater for either total alpha or gross beta and the method of determining empirical efficiencies for non-calibrated isotopes is described.*

The detector used for strontium-90 analysis for this project was calibrated in 1992. Daily reliability check results were control charted and show the stability of the detector efficiency since the last calibration; therefore, continuing calibration requirements have been met.

Self-absorption curves were not generated for strontium-90 analysis. The high activities of the samples require dilutions of 100×, therefore, no self-absorption curves were generated. However, as stated in the report, due to the high activity of the samples and the relatively low variation in counting efficiency over the range of sample weights that are being used, no action was taken.

The raw calibrations were examined and the detector efficiencies for the APCs and GPCs that were used for total alpha, total beta, strontium-90 and neptunium-237 were greater than or equal to 20% at zero percent solids.

3.5.2.2 Continuing Calibration

Verify that the continuing instrument calibration meets the following criteria:

- *Acceptable continuing calibration checks have been performed at least once per analytical run, sample batch, or daily, whichever is more frequent.*

- *Check standards are NIST-traceable and certificates are provided.*

A daily reliability check using cesium-137 was analyzed and control charted for the detector that was used for strontium-90 analysis.

All criteria were met.

3.5.2.3 Background Counts

Verify that the instrument background counts meet the following criteria:

- *Performed within one week prior to sample analysis.*
- *Performed on each detector used for sample analysis.*
- *Within the laboratory control limits.*

All criteria were met.

3.5.2.4 Laboratory Blanks

Verify that the laboratory blank analysis meets the following criteria:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*

- *Prepared at the same time and analyzed with the samples using the same procedure.*

- *Results are less than or equal to the MDA and RDL.*

The validation report indicated that all criteria were met with the exception of two blanks, WL32189 and WL32375. These blanks had strontium-90 activity which exceeded their detection limits, and therefore, composite samples 263-1, 263-2, 263-3 and 263-4 were qualified as estimated (J) by the validator. However, in section 3.4.5.2 of the TCR, the discussion from the laboratory case narrative indicates that strontium activity was detected in all blanks except the one for Worklist 32190. Therefore, due to the evaluation of the QC samples, samples 264-1, 264-2, 264-3, and 264-4 should be qualified (J) estimated and sample 263-6 should be qualified (UJ) in addition to the samples flagged by the validators.

3.5.2.5 Laboratory Control or Blank Spike Samples

Verify that the following criteria are met and that the laboratory provided the following information:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*
- *Prepared at the same time and analyzed with the samples using the same procedure.*
- *LCS activity is between five and 30 times the associated RDL value.*
- *Results are within the required TSAP recovery limits of 70 to 130%.*

All criteria were met.

3.5.2.6 Chemical Recovery Factors

Verify the following:

- *Chemical carrier was added to every sample analyzed including blanks and all quality control samples.*
- *The amount and concentration of the chemical carrier added to each sample and recovered from each sample was reported along with a dilution log documenting traceability.*
- *The chemical recovery factor is within required limits of 30% to 130%.*

All criteria were met.

3.5.2.7 Matrix Spike Samples

Verify that MS analysis was conducted as follows:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group if a carrier was not used in the analysis*

- *Prepared at the same time and analyzed with the samples using the same procedure.*

- *Percent recovery is within the recovery limits of 70% to 130% unless sample concentration exceeds the spike concentration by a factor of four or more.*

All criteria were met.

3.5.2.8 Laboratory Duplicates

Verify that the following criteria are met and that the laboratory provided the following information:

- *The laboratory has conducted a duplicate analysis sample at frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.*

- *The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.*

- *The RPD must be less than 20% for water samples (≤ 20% for soils) if the sample concentration is greater than five times the RDL.*

- *For samples results less than five times the RDL, the range between the primary and duplicate sample results must be less than or equal to the RDL for water samples (≤2 × RDL for soils).*

All duplicates were within the specified limits with the exception of S99Z00311 and its duplicate. However, because the sample activity was less than its detection limit, no action was taken.

3.5.2.9 Holding Times

Verify that all samples were preserved properly (if applicable) and analyzed within 180 days.

All criteria were met.

3.5.2.10 Sample Result Quantitation and Minimum Detectable Activities

Verify the laboratory has reported the following information for each sample:

- *Sample identification*
- *Detector identification and efficiency*
- *Start date and time of each analysis step*
- *Sample and background counts*

- *Count duration*
- *Planchet weights*
- *Sample volume*
- *Chemical recovery factors, including the amount added to each sample (including QC samples) and recoveries*
- *Ingrowth and decay factors for all analysis*
- *Calculated sample activities, uncertainties and MDA values*
- *Required detection limits*

All criteria were met.

3.5.3 Neptunium-237 Analysis by Alpha Proportional Counter

For neptunium-237 by APC, no specific criteria were spelled out in the data validation procedure. Therefore, because an alpha proportional counter was used for neptunium-237 analysis, the criteria for total alpha/beta were used for this report.

3.5.3.1 Initial Calibration

Verify that the counting system used for sample analysis meet the following criteria:

- *Each counting system used for sample analysis was efficiency calibrated within one year prior to sample analysis. If not calibrated within one year, then the continuing calibration requirements listed must be met.*

- *Calibration standards are NIST-traceable and certificates are provided.*

- *Self-absorption curves were prepared for each counting system from a series of planchets with weights ranging from 0 to 150 mg.*

- *Efficiency of each detector at 0% solids must be at least 20% or greater for either total alpha or gross beta.*

Detectors used for neptunium-237 analysis were calibrated within one year before the analysis date.

The raw calibrations were examined and the detector efficiencies for the APCs and GPCs that were used for total alpha, total beta, strontium-90 and neptunium-237 were greater than or equal to 20% at zero percent solids.

3.5.3.2 Continuing Calibration

Verify that the continuing instrument calibration meets the following criteria:

- *Acceptable continuing calibration checks have been performed at least once per analytical run, sample batch, or daily, whichever is more frequent.*

- *Check standards are NIST-traceable and certificates are provided.*

All criteria were met.

3.5.3.3 Background Counts

Verify that the instrument background counts meet the following criteria:

- *Performed within one week prior to sample analysis.*
- *Performed on each detector used for sample analysis.*
- *Within the laboratory control limits.*

All criteria were met.

3.5.3.4 Laboratory Blanks

Verify that the laboratory blank analysis meets the following criteria:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*

- *Prepared at the same time and analyzed with the samples using the same procedure.*

- *Results are less than or equal to the MDA and RDL.*

All criteria were met.

3.5.3.5 Matrix Spike Samples

Verify that MS analysis was conducted as follows:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group if a carrier was not used in the analysis.*

- *Prepared at the same time and analyzed with the samples using the same procedure.*

- *Percent recovery is within the required TSAP recovery limits of 70 to 130% unless sample concentration exceeds the spike concentration by a factor of 4 or more.*

All criteria were met.

3.5.3.6 Laboratory Control or Blank Spike Samples

Verify that the following criteria are met and that the laboratory provided the following information:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*
- *Prepared at the same time and analyzed with the samples using the same procedure.*
- *LCS activity is between 5 and 30 times the associated RDL value.*
- *The actual LCS concentration and the amount of spike added for the LCS were provided by the laboratory.*
- *Results are within the required TSAP recovery limits of 70 to 130%.*

All criteria were met.

3.5.3.7 Laboratory Duplicates

Verify that the following criteria are met and that the laboratory provided the following information:

- *The laboratory has conducted a duplicate analysis sample at frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.*
- *The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.*
- *The RPD must be less than 20% for water samples (≤ 20% for soils) if the sample concentration is greater than five times the RDL.*

All criteria were met.

3.5.3.8 Holding Times

Verify that all samples were preserved properly (if applicable) and analyzed within 180 days.

All criteria were met.

3.5.3.9 Sample Result Quantitation and Minimum Detectable Activities

Verify the laboratory has reported the following information for each sample:

- *Sample identification*
- *Detector identification and efficiency*
- *Background counts*
- *Count duration*
- *Planchet weights*

- *Sample volume*
- *Alpha and beta crosstalk factors*
- *Calculated sample activities, uncertainties, and MDA values*
- *Required detection limits*

All criteria were met.

3.5.4 Alpha Spectroscopy

3.5.4.1 Initial Calibration

Verify that the counting system used for sample analysis meets the following criteria:

- *Each counting system used for sample analysis was efficiency calibrated within one year prior to sample analysis.*

- *Efficiency values are provided for each detector and were measured within one year of the sample analysis.*

- *Calibration standards are NIST-traceable and certificates are provided.*

- *Detectors were calibrated in the energy range of approximately 4 to 6 MeV with a maximum range of 2 to 8 MeV, and the standards were counted in order to accumulate a minimum of 2000 counts for each target radioisotope.*

As stated by the validators, all criteria were met. No documentation was provided with the data package, therefore, no verification could be provided.

3.5.4.2 Continuing Calibration

Verify that the continuing instrument calibration meets the following criteria:

- *Energy calibration and detector efficiencies were checked at least weekly prior to sample analysis and for each detector used for sample analysis.*

- *Detector efficiencies determined from the weekly checks are within the laboratory control limits.*

- *Tracer preparation, activity dilution log, and traceability is submitted with each data package.*

- *The activity, NIST certificates, and dilution log are submitted for each check standard used for weekly checks.*

All criteria were met.

3.5.4.3 Background Counts

Verify that the instrument background counts meet the following criteria:

- *Performed at least monthly on each detector used for sample analysis for each region of interest (ROI) monitored for the particular analysis.*
- *Within the laboratory control limits.*

All criteria were met.

3.5.4.4 Laboratory Blanks

Verify that the laboratory blank analysis meets the following criteria:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*

- *Prepared at the same time and analyzed with the samples using the same procedure, aliquot size, and counting time.*

- *Results are less than or equal to the MDA and RDL.*

All criteria were met.

3.5.4.5 Matrix Spike Samples

Verify that MS analysis was conducted as follows:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group if a tracer was not used in the analysis.*

- *Prepared at the same time and analyzed with the samples using the same procedure.*

- *Percent recovery is within the required recovery limits of 70 to130% unless sample concentration exceeds the spike concentration by a factor of four or more.*

All criteria were met.

3.5.4.6 Laboratory Control or Blank Spike Samples

Verify that the following criteria are met and that the laboratory provided the following information:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*
- *Prepared at the same time and analyzed with the samples using the same procedure.*

- *LCS activity is between five and 30 times the associated RDL value.*
- *The actual LCS concentration and the amount of spike added for the LCS were provided by the laboratory.*
- *Results are within the required recovery limits of 70% to130%.*

All criteria were met.

3.5.4.7 Laboratory Duplicates

Verify that the following criteria are met and that the laboratory provided the following information:

- *The laboratory has conducted a duplicate analysis sample at frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.*

- *The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.*

- *The RPD must be less than or equal to 20% for water samples (≤ 20% for soils) if the sample concentration is greater than five times the RDL.*

- *For samples results less than five times the RDL, the range between the primary and duplicate sample results must be less than or equal to the RDL for water samples (≤2× RDL for soils).*

All criteria were met.

3.5.4.8 Tracer Recovery

Verify that the following criteria are met:

- *Each sample was spiked with an appropriate tracer as applicable for the analytical method.*
- *Tracer activity and NIST-traceability and a dilution log were provided.*
- *Raw data was provided showing the amount of tracer added to each sample and the gross counts per minute of the tracer.*
- *Tracer recoveries are within the limits of 20% to 105%.*

All criteria were met.

3.5.4.9 Holding Times

Verify that all samples were preserved properly (if applicable) and analyzed within 180 days.

All criteria were met.

3.5.4.10 Sample Result Quantitation and Minimum Detectable Activities

Verify the laboratory has reported the following information for each sample:

- *Sample identification*
- *Detector identification and efficiency*
- *Gross sample counts, ROI, and channel by channel counts*
- *Gross tracer counts, ROI, and channel by channel counts*
- *Background counts monthly*
- *Count duration*
- *Sample spectra showing peak integration parameters and full width at half maximum (FWHM) values*
- *Planchet weights*
- *Sample volume*
- *Alpha and beta crosstalk factors*
- *Calculated sample activities, uncertainties, and MDA values*
- *Required detection limits*

All criteria were met.

3.5.5 Liquid Scintillation Data Validation Requirements

3.5.5.1 Initial Calibration

Verify that the counting system used for sample analysis meets the following criteria:

- *Each counting system used was factory calibrated at installation and after any maintenance or repair and a certificate of calibration is provided in the data package or the most recent calibration supplement.*
- *Calibration standards are NIST-traceable and certificates are provided.*

All criteria were met.

3.5.5.2 Continuing Calibration

Verify that the continuing instrument calibration meets the following criteria:

- *Acceptable continuing calibration checks have been performed with each analytical run, sample batch, or daily, whichever is more frequent. The results and control limits shall be reported with each SDG.*

- *Calibration checks are within the laboratory control limits.*

- *Calibration checks are performed at the same aliquot size as the samples.*

- *Efficiency check standards are NIST-traceable and certificates are provided.*

- *Quench monitoring values are reported with each data package and are within the laboratory control limits.*

All criteria were met.

3.5.5.3 Background Counts

Verify that the instrument background counts meet the following criteria:

- *Performed within on each counting system used for sample analysis and were performed with each analytical run, sample batch, or daily, whichever is more frequent. The results and control limits are reported with each SDG.*
- *The most recent background check was within the laboratory control limits.*

All criteria were met.

3.5.5.4 Laboratory Blanks

Verify that the laboratory blank analysis meets the following criteria:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*
- *Prepared, distilled and analyzed using the same procedure and aliquot size as the samples.*
- *Results are reported along with the laboratory control limits.*

All criteria were met.

3.5.5.5 Laboratory Control or Blank Spike Samples

Verify that the following criteria are met and that the laboratory provided the following information:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*

- *Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.*

- *LCS or BSS activity is less than 100 times the RDL.*

- *LCS or BSS traceability, concentration, and dilution log is provided.*

- *Results are within the required recovery limits of 80% to 120%.*

All criteria were met.

3.5.5.6 Matrix Spike Samples

Verify that the following criteria are met and that the laboratory provided the following information:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*
- *Prepared at the same time and analyzed in the same batch, using the same procedure as the associated samples.*
- *Percent recovery is within the limits of 70 to 130% unless sample concentration exceeds the spike concentration by a factor of four or more.*

All criteria were met.

3.5.5.7 Laboratory Duplicates

Verify that the following criteria are met and that the laboratory provided the following information:

- *The laboratory has conducted a duplicate analysis sample at frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.*
- *The duplicate analysis was prepared and analyzed in the same batch, using the same procedure and the same aliquot size as the associated samples.*
- *The RPD must be less than 15% for sample concentrations greater than five times the RDL.*
- *For samples results less than five times the RDL, the range between the primary and duplicate sample results must be less than or equal to the RDL.*

All criteria were met.

3.5.5.8 Holding Times

Verify that all samples were preserved properly (if applicable) and analyzed within 180 days.

All criteria were met.

3.5.5.9 Sample Preparation

Verify the laboratory has met the following criteria:

- *All tritium field and QC samples were distilled prior to analysis.*

- *Samples were analyzed within seven days after distillation.*

All criteria were met.

3.5.5.10 Sample Result Quantitation and Minimum Detectable Activities

Verify the laboratory has reported the following information for each sample:

- *Sample identification*
- *Detector identification and efficiency*
- *Background counts*
- *Count duration*
- *Data and time of all sample analysis*
- *Sample volume*
- *Calculated sample activities, uncertainties, and MDA value*
- *Required detection limits*

All criteria were met.

3.5.6 ICP/MS Data Validation Requirements

3.5.6.1 Tuning and Mass Calibration

Verify that the ICP/MS instrument was tuned before sample analysis and that the following criteria were met:

- *Instrument was tuned with a mixture of the target radioisotopes at a concentration level not greater than 10 times the RDL and the tuning results are reported along with the raw data.*
- *The observed versus tune mass response agree within 5%.*
- *Tuning standards are NIST-traceable, and certificates and a dilution log are provided.*

All criteria were met

3.5.6.2 Initial Calibration

Verify that the initial instrument calibration met the following criteria:

- *Each ICP/MS instrument used was calibrated at the beginning of each analytical run with a calibration mixture containing all radioisotopes of interest.*
- *Calibration standards are NIST-traceable and certificates are provided.*
- *Initial calibration verification (ICV) percent recoveries are within the control limits of 90% to 110%.*

Because an initial calibration verification recovery was outside QC limits (i.e., 115%), the plutonium-239 ICP/MS results for composite samples 264-1, 264-2, 264-3, 264-4, and 264-5 were qualified as estimates and flagged (J). Because an initial calibration verification recovery was outside QC limits (i.e., 111%), the plutonium-239 ICP/MS results for composite samples 263-5, 263-6, 263-7, and 263-8 were qualified as estimates and flagged (J).

3.5.6.3 Continuing Calibration

Verify that the continuing instrument calibration met the following criteria:

- *Continuing calibration checks were performed at a 10% frequency, or every two hours, whichever is most frequent.*
- *Check standards are NIST-traceable, and certificates and a dilution log are provided.*
- *Continuing calibration verification (CCV) percent recoveries are within the control limits of 90% to 110%.*

All criteria were met.

3.5.6.4 Laboratory Blanks

Verify that the laboratory blank analysis met the following criteria:

- *Initial calibration blank (ICB) analyzed after the initial calibration samples and before the laboratory and QC samples.*

- *Continuing calibration blank (CCB) analyzed at a 10% frequency, or every two hours, whichever is most frequent.*

- *Preparation Blank (PB) digested and analyzed with the laboratory and field samples.*

All criteria were met.

3.5.6.5 Laboratory Control or Blank Spike Samples

Verify that the following criteria are met and that the laboratory provided the following information:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group.*
- *Prepared at the same time and analyzed with the samples using the same procedure.*
- *LCS or BSS activity is <100 times the RDL value.*
- *Provided the actual LCS concentration or the spike concentration and the amount of spike added for the BSS.*
- *Results are within the required recovery limits of 80% to 120%.*

All criteria were met.

3.5.6.6 Matrix Spike Samples

Verify that the following criteria are met and that the laboratory provided the following information:

- *Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group*
- *Prepared and analyzed at the same time, in the same batch, using the same procedure as the associated samples.*
- *Provided the spike concentration and the amount of spike.*
- *Verify that the results are within the limits of 75% to 125% recovery unless the sample activity exceeds the spike activity by a factor of four or more.*

All criteria were met.

3.5.6.7 Laboratory Duplicates

Verify that the following criteria are met and that the laboratory provided the following information:

- *The laboratory has conducted a duplicate analysis sample at frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.*
- *The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.*
- *The RPD must be less than 20% for water samples (≤ 20% for soils) if the sample concentration is greater than five times the RDL.*
- *For samples results less than five times the RDL, the range between the primary and duplicate sample results must be less than or equal to the RDL for water samples (≤2× RDL for soils).*

All criteria were met.

3.5.6.8 Holding Times

Verify that all samples were preserved properly (if applicable) and analyzed within 180 days.

All criteria were met.

3.5.6.9 Sample Result Quantitation and Minimum Detectable Activities

Verify the laboratory has reported the following information for each sample:

- *Sample identification*
- *Instrument identification*
- *Sample Analysis date and time*
- *Sample analysis raw data*
- *Sample results and detection limits*
- *Sample preparation data*
- *Required detection limits*

All criteria were met.

3.6 RADIOLOGICAL DATA ASSESSMENT SUMMARY

3.6.1 Precision

Measurements of precision between the duplicate sample results were determined to be adequate, indicating good repeatability.

3.6.2 Accuracy

Percent recoveries for the method standards, prefusion LCSs, and prefusion MSs were within the required TSAP limits (EQM 1999a) and indicate acceptable accuracy for the laboratory measurements. The sludge composite sample results for total alpha were plotted against the summed results of plutonium-239, plutonium-240, and americium-241 and are presented in Figure 3-4. The results compare favorably, although a slight negative bias is noted for the total alpha results when compared to the summed plutonium/americium results. The bias possibly could be caused by attenuation of the total alpha due to self-absorption (self-shielding) on the planchet.

The total alpha and summed plutonium/americium results also were plotted using a regression curve. The plot is presented in Figure 3-5. The correlation coefficient is 0.9901 and shows an excellent fit between the total alpha and plutonium and americium results.

Next, a comparison was made between the plutonium-239/240 results by AEA (see Table 3-23) and ICP/MS (see Tables 3-29 and 3-31). Figure 3-6 is the regression curve for ICP/MS results on samples not chemically separated versus AEA. The ICP/MS results were converted from units of μg/g to μCi/g using the specific activity for each isotope. The plutonium-239 and plutonium-240 results were then summed in order to compare them against the plutonium-239/240 AEA results. The correlation coefficient for the regression curve is 0.9689 and shows an excellent fit between the AEA and ICP/MS results.

The chemically separated plutonium-239/240 results by ICP/MS (see Tables 3-30 and 3-32) also were plotted against the plutonium-239/240 from AEA. The graph is presented in Figure 3-7. The correlation coefficient is 0.988 and again shows an excellent fit between analytical results.

Finally, the results of the chemically separated versus nonseparated runs were plotted on a regression curve. The results are presented in Figure 3-8.

Figure 3-4. Comparison of Plutonium-239 + 240 + Americium-241 to Total Alpha

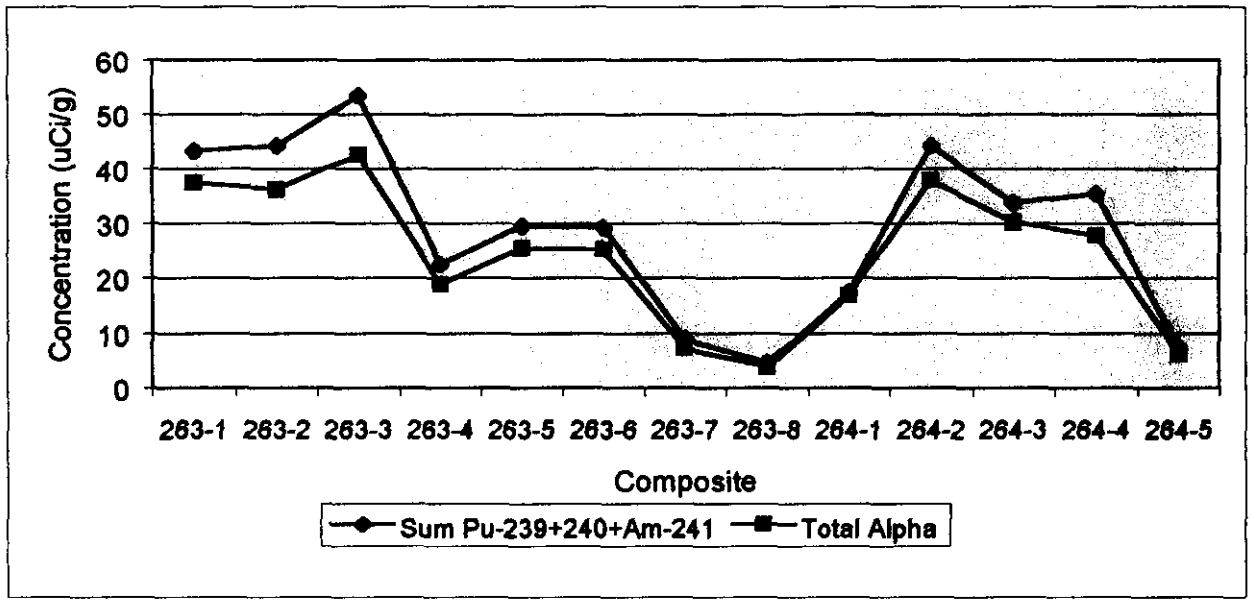


Figure 3-5. Regression Curve of Plutonium-239 + 240 + Americium-241 to Total Alpha

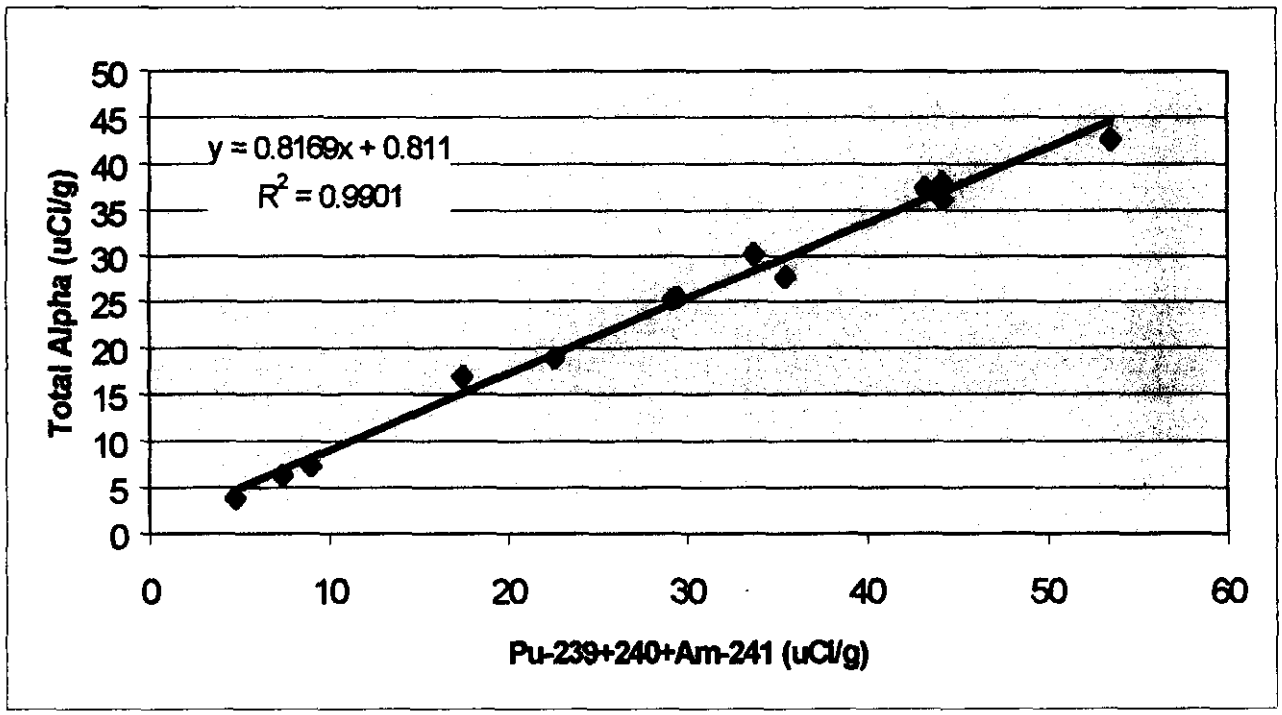


Figure 3-6. Regression Curve of AEA to ICP/MS Plutonium-239/240

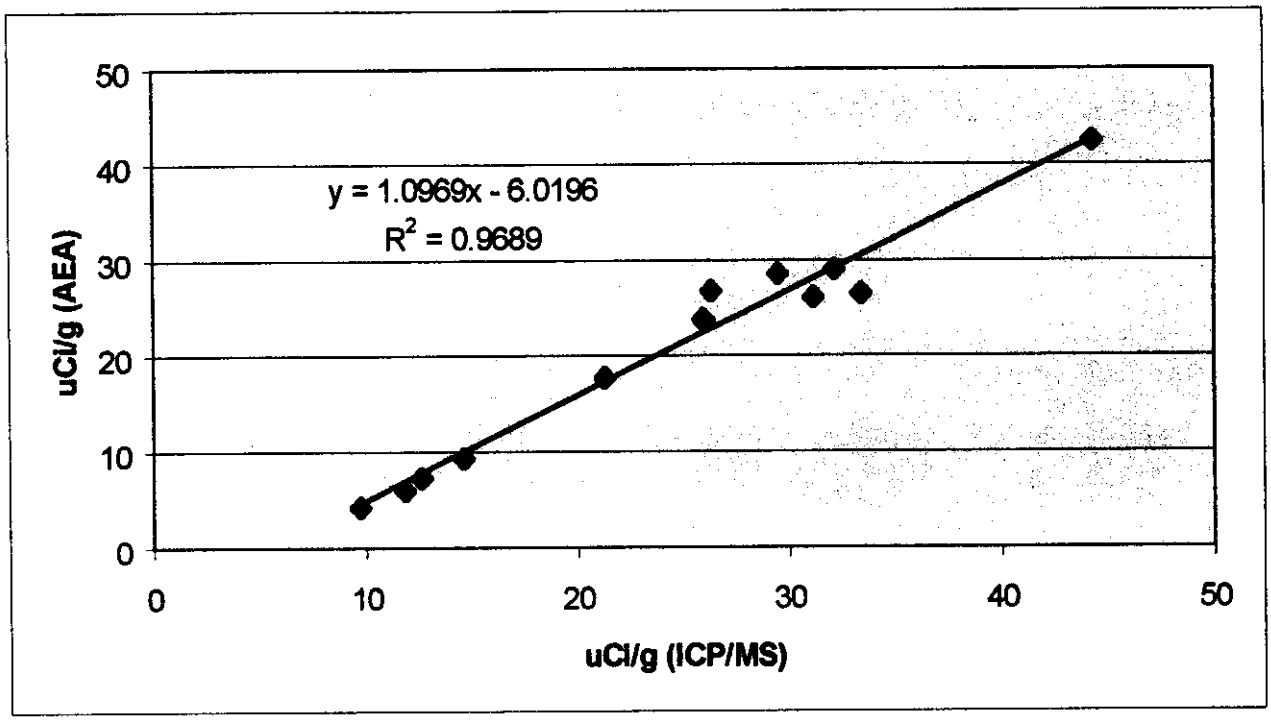


Figure 3-7. Regression Curve of AEA to Chemically Separated ICP/MS Plutonium-239/240

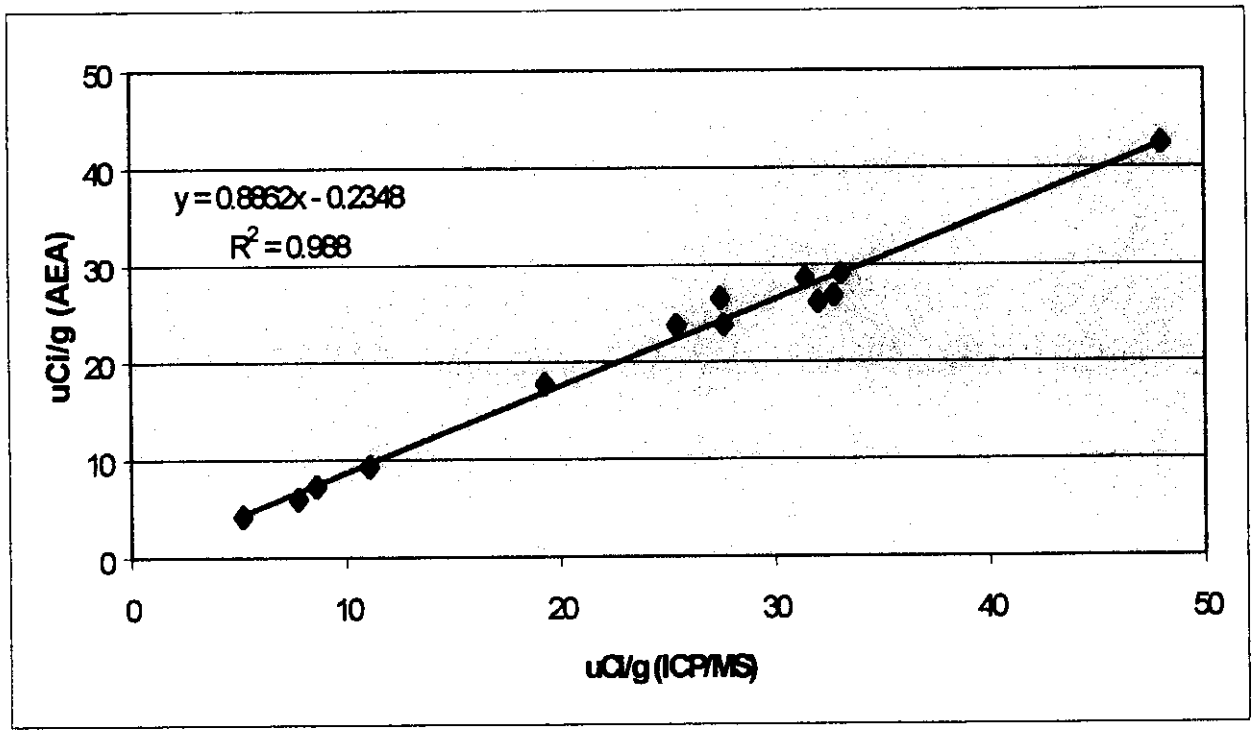
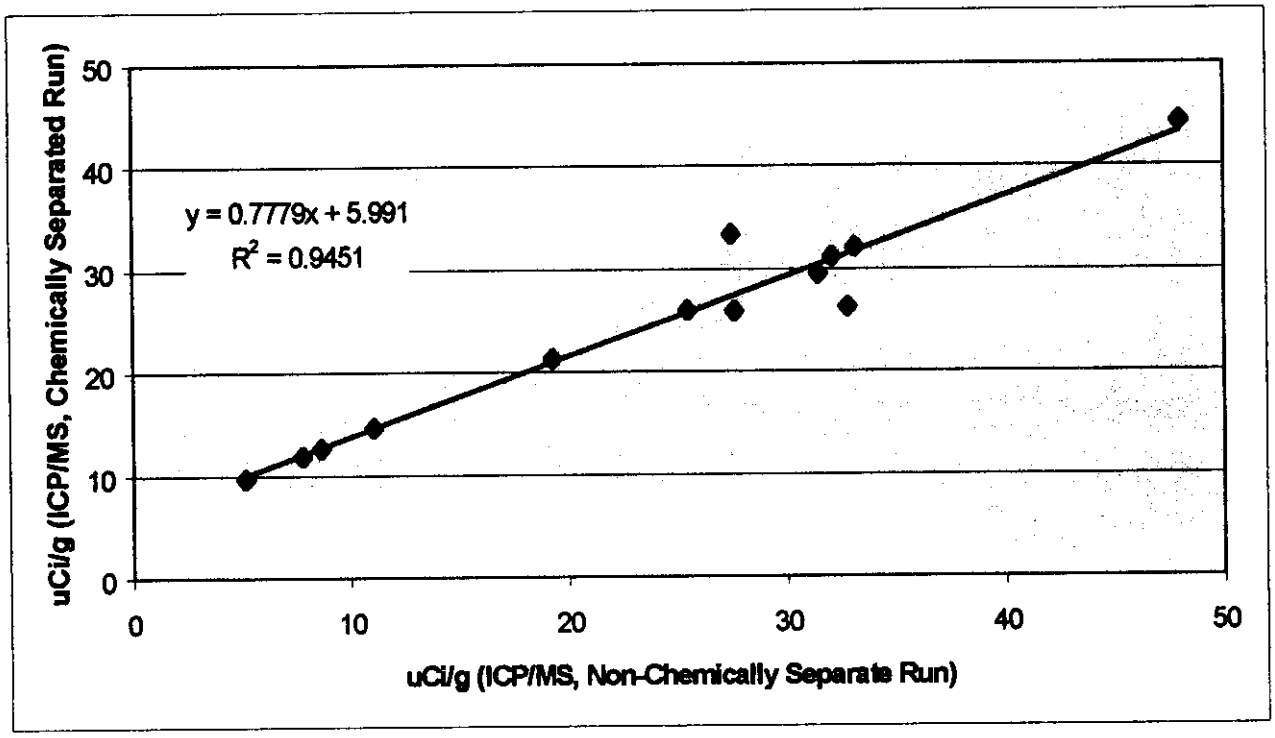


Figure 3-8. Regression Curve of Chemically Separated versus Nonchemically Separated Plutonium 239/240 by ICP/MS



3.7 CHEMICAL ANALYSES AND QA/QC SUMMARY OF SLUDGE COMPOSITE SAMPLES AND DRAINABLE LIQUID

For each analysis described in this section, the following subsections are presented:

- Analytical requirements,
- QC review, and
- Summary.

The analytical requirements subsection discusses the TSAP (EQM 1999a) and analytical method requirements. The QC review discusses the laboratory method used and a review of the QC data. Following the QC review, the results are summarized with reference to the QC data and the effect on the usability of the data is addressed. The review is not meant to be data validation.

According to the TSAP (EQM 1999a), third-party data validation was required for radiochemistry and polychlorinated biphenyl (PCB) analyses only. The review and validation result in the application of flags on selected results to indicate they are estimated. No data were rejected.

3.7.1 Volatile Organic Compounds

The volatile organic analysis (VOA) was performed on test portions taken from one stratum of each segment received. The sample aliquots for the VOA analysis were collected directly after extrusion and before any mixing of the segment was performed. The aliquots were approximately 0.1 g each. Additional separate aliquots were taken for duplicates and matrix spikes.

One drainable liquid sample was collected from Core 264, Segment 2. No other segments produced sufficient liquid for analysis. The liquid was very dark viscous liquid that was nearly sludge slurry. Duplicate and spike test portions were weighed for drainable liquid analysis.

3.7.1.1 Analytical Requirements

Samples were analyzed by purge and trap followed by Method 8260B as implemented by LA-523-118 Rev A/Mod 0. The precision and accuracy limits were identified in the TSAP (EQM 1999a).

3.7.1.2 Quality Control Review

Data for the thirteen analytes are presented in Tables 3-38 and 3-39. For the 12 sludge composite samples analyzed, the following summarizes the number of positive responses:

- 11 for n-butanol
- 12 for acetone
- 5 for benzene
- 6 for methylene chloride
- 1 for toluene

The positive hits for most of the analytes are above detection limits and are a factor of 10 above the method or hot cell blanks. However, there is a possibility that the acetone, n-butanol, methylene chloride, and benzene may be due to hot cell and/or laboratory contamination as discussed below.

There were multiple issues that create potential QC concerns. The first is that in the middle of extrusion of the first segment of core 263, a plastic melting operation was begun in the hot cell and this could possibly have contaminated the cores. No VOA sample was collected during collection of core 263 segment 1 due to this. Subsequent to this, blanks were placed in the hot cell. All hot cell blanks contained 1-butanol at up to 152 µg/kg. Method blanks analyzed with each analysis batch contained varying concentrations of common and uncommon contaminants (e.g., 1-butanol; 1,2,4-trichlorobenzene; benzene; toluene; and xylenes). Concentrations ranged from tenths of a ppb (µg/kg) to 35 µg/kg. Sample reporting limits ranged from 200 to 1000 µg/kg depending on the analyte. The elevated reporting limits are due to the low solids content, as well as the small test portion size of 0.1 g as opposed to the normal 5 g.

Table 3-38. Core 263 Volatile Organic Analysis[1,2]

Analyte	263-2		263-3		263-4		263-5	
	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit
1,2,4-Trichlorobenzene	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	< 1.75e+02	174.8
1-Butanol	1.63e+03	1320	1.56+03	1220	1.99e+03	1360	1.52e+03	874.1
Acetone	4.03e+03	526.3	1.39e+03	490.2	1.75e+03	543.4	7.88e+02	349.6
Benzene	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	1.57e+04	174.8
Carbon tetrachloride	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	< 1.75e+02	174.8
Chloroform	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	< 1.75e+02	174.8
Methylene chloride	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	< 1.75e+02	174.8
Pentachloroethane	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	< 1.75e+02	174.8
Tetrachloroethene	< 2.63e+02	263.2	< 2.45e+02	245	< 2.71e+02	271.7	< 1.75e+02	174.8
Toluene	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	< 1.75e+02	174.8
Trichloroethene	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	< 1.75e+02	174.8
Trichlorofluoromethane	< 2.63e+02	263.2	< 2.45e+02	245	< 2.72e+02	271.7	< 1.75e+02	174.8
Xylenes (total)	< 5.26e+02	526.3	< 4.90e+02	490.2	< 5.43e+02	543.4	< 3.50e+02	349.6

1. All results are in units of g/kg.
2. All data are estimated and flagged with J qualifier due to considerations discussed in Section 3.7.1.2.

The second concern was due to the replacement of the VOA vial caps. An issue regarding proper radiological work permits for loading alpha containing material out of the hot cell caused an 11- to 13-day delay between subsample collection and submittal of the samples to the laboratory. For alpha contamination control, the sample caps were changed twice. Removal of a cap for a reason other than to get the sample into the analysis process typically invalidates a VOA sample. The hot cell temperatures are elevated above normal room temperature. In addition, the samples were left unrefrigerated before analysis. These conditions likely resulted in loss of volatile organics from the samples.

The laboratory indicated that, because no method for refrigeration of the core segments from collection to extrusion exists, the lack of refrigeration was not an issue. However each segment is sealed in metal with o-ring seals on the valve and piston ends. In addition, had the VOA sample been immediately collected, removed, and stored under refrigeration, any interstitial volatiles would likely remain.

The project approved the analysis of the samples by purge and trap rather than headspace extraction method. The purge and trap is an acceptable Environmental Protection Agency (EPA) SW-846 method (EPA 1997).

The replacement of the caps and burning in the hot cell were not approved. Leaving the samples unrefrigerated was not approved. The hot cell blank contamination may have occurred in the hot cell during extrusion or during replacement of the caps. It is not clear how method blanks were contaminated.

3.7.1.3 Summary

Based on the following QC problems, the volatile data can only be taken as a lower estimate of concentration.

- Removing caps twice before analysis thus allowing release of volatiles,
- Potential hot cell and laboratory contaminants in samples and blanks, and
- No refrigeration from the time of extrusion to analysis.

The QC data were reported. However, given the fact that all data are estimated based on the above discussion, no detailed review was performed. No added benefit could be gained from further review. Table 3-39 summarizes the data.

3.7.2 Semivolatile Organic Compounds

The data are presented in Table 3-40. The table is ordered by composite number and provides the analyte, the composite sample result and detection limits, DQA flags, laboratory control sample percent recovery, and the duplicate results and relative percent differences. The data are reported on wet weight basis for the sludge. It is noted that, of the 13 composite results reported for each compound, there were 11 positive responses for the TBP and eight for the DBP and none for the dibutyl butyl phosphonate (DBBP). All positive responses for the TBP and the DBP were well below the sample specific reporting limit (i.e., factors of 20 to 100 times below the reporting limits). There are no regulatory limits for these constituents; therefore, the low concentrations detected do not affect the waste designation.

3.7.2.1 Analytical Requirements

The thirteen sludge samples were extracted and derivitized using LA-523-138 Rev B/Mod 2 and LA-523-116 Rev A/Mod 0. The derivitization allowed quantitation of the dibutyl phosphate (DBP-d) which does not chromatograph without derivitization. The other two semivolatile analytes of interest were DBBP and TBP. The samples were then analyzed by GC/MS using laboratory method LA-523-135 Rev A/Mod 0 which corresponds to Method 8270 C. Note that 8270C does not include any of the analytes of interest and, therefore, the calibrations for these analytes were performed in a manner consistent with the method.

3.7.2.2 QC Review

Detailed review for each type of QC sample is presented below. The percent recovery for one laboratory control sample for DBP-d was 2.04% and recoveries from one of the two matrix spike/matrix spike duplicates for the DBP was low (38 and 20%) but in line with previous laboratory control sample recoveries for the initial demonstration of performance for the method. The DBP recovery in the second matrix spike/matrix spike duplicate was very low (1 and 0.5%). The other laboratory control sample processed with the samples generated a 73.77% recovery for the DBP-d which is within the 70 to 130% recovery set by the SW-846 method (EPA 1997). Recoveries for the other two compounds in both laboratory control samples were acceptable. It appears that the low DBP recovery may be due to a derivitization problem.

The laboratory discussed this QC problem with the Z-361 project manager and the EQM chemists. No reanalysis was performed. The DBP is a degradation product from TBP and could be estimated by calculation. In addition, EPA has no risk data for DBP, nor is it on the Toxicity Characteristic or Underlying Hazardous Constituent (used for land disposal regulations) lists. Chelating compounds such as DBP, DBBP, and TPB may affect mobility of plutonium or uranium. These analytes were originally requested in the TSAP (EQM 1999a) to assess mobility of the plutonium. It was agreed in e-mails of March 11 and 12, 2000, that the samples would not be reanalyzed and the data would be reported with qualifiers.

Based on the poor recoveries of the DBP-d in one laboratory control sample and both pairs of matrix spike/matrix spike duplicates, all DBP data were flagged, as estimated (J) during the data review. Results on the other two analytes were not flagged because recoveries were acceptable.

The only deviation was not performing a reanalysis for the laboratory control sample failure noted in the above paragraphs. The deviation was approved by the project before the data were finalized (e-mails March 11 and 12, 2000).

No formal validation was performed, because none was required by the TSAP (EQM 1999a). A general review of the summarized QC was performed by EQM and the results are discussed below.

Table 3-39. Core 264 Volatile Organic Analysis[1,2]

Analyte	264-1			264-2			264-3			264-4			264-5		
		Result	Detection Limit		Result	Detection Limit		Result	Detection Limit		Result	Detection Limit		Result	Detection Limit
1,2,4-Trichlorobenzene	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4	<	1.49e+02	148.8	<	1.34e+02	133.7
1-Butanol	<	1.26e+03	1260		1.66e+03	766.9		1.04e+03	822.4		5.47e+03	744		1.73e+03	668.4
Acetone		2.27e+03	505		5.62e+03	306.7		3.15e+03	328.9		3.78e+03	297.6		1.28e+03	267.4
Benzene	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4		1.77e+02	148.8		3.93e+03	133.7
Carbon tetrachloride	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4	<	1.49e+02	148.8	<	1.34e+02	133.7
Chloroform	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4	<	1.49e+02	148.8	<	1.34e+02	133.7
Methylene chloride		1.25e+03	252.5		1.16e+03	153.3		8.74e+02	163.4	<	1.49e+02	148.8		7.17e+02	133.7
Pentachloroethane	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4	<	1.49e+02	148.8	<	1.34e+02	133.7
Tetrachloroethene	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4	<	1.48e+02	148.8	<	1.34e+02	133.7
Toluene	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4	<	1.49e+02	148.8		1.59e+02	133.7
Trichloroethene	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4	<	1.49e+02	148.8	<	1.34e+02	133.7
Trichlorofluoromethane	<	2.52e+02	252.5	<	1.53e+02	153.3	<	1.63e+02	163.4	<	1.49e+02	148.8	<	1.34e+02	133.7
Xylenes (total)	<	5.05e+02	505	<	3.07e+02	306.7	<	3.29e+02	328.9	<	2.98e+02	297.6	<	2.67e+02	267.4

1. All results are in units of g/kg.
2. All data are estimated and flagged with J qualifier due to discussions in Section 3.7.1.2.

Table 3-40. Semi-volatile Organic Analysis Results for Sludge Composite Samples from Tank 241-Z-361 (2 sheets)

Composite	Composite Result	Detection Limit	DQA Qualifier	LCS (%R)	Duplicate	RPD (%)
Dibutyl phosphate –derivatized (µg/kg)						
Core 263						
263-1	3	47	J	73.77	0.9	75.9
263-2	ND	49	J	2.04	ND	N/A
263-3	ND	43	J	73.77	N/A	N/A
263-4	0.4	45	J	73.77	N/A	N/A
263-5	ND	39	J	73.77	N/A	N/A
263-6	0.5	45	J	73.77	N/A	N/A
263-7	2	45	J	73.77	N/A	N/A
263-8	0.6	44	J	73.77	N/A	N/A
Core 264						
264-1	0.5	39	J	73.77	N/A	N/A
264-2	ND	41	J	2.04	N/A	N/A
264-3	0.3	46	J	2.04	N/A	N/A
264-4	ND	44	J	2.04	N/A	N/A
264-5	ND	49	J	2.04	N/A	N/A
Dibutylbutyl phosphonate (µg/kg)						
Core 263						
263-1	ND	17		101.4	0.252	N/A
263-2	ND	18		90.7	ND	N/A
263-3	ND	16		101.4	N/A	N/A
263-4	ND	16		101.4	N/A	N/A
263-5	ND	14		101.4	N/A	N/A
263-6	ND	17		101.4	N/A	N/A
263-7	ND	17		101.4	N/A	N/A
263-8	ND	16		101.4	N/A	N/A
Core 264						
264-1	ND	14		101.4	N/A	N/A
264-2	ND	15		90.7	N/A	N/A
264-3	ND	17		90.7	N/A	N/A
264-4	ND	16		90.7	N/A	N/A
264-5	ND	18		90.7	N/A	N/A

Table 3-40. Semi-volatile Organic Analysis Results for Sludge Composite Samples from Tank 241-Z-361 (2 sheets)

Composite	Composite Result	Detection Limit	DQA Qualifier	LCS (%R)	Duplicate	RPD (%)
Tri-n-butylphosphate (µg/kg)						
Core 263						
263-1	0.6	22		123	0.7	80
263-2	0.2	20		110.9	0.4	67
263-3	0.6	20		123	N/A	N/A
263-4	0.6	21		123	N/A	N/A
263-5	0.6	18		123	N/A	N/A
263-6	0.4	21		123	N/A	N/A
263-7	0.4	21		123	N/A	N/A
263-8	2	21		123	N/A	N/A
Core 264						
264-1	ND	18		123	N/A	N/A
264-2	0.4	19		110.9	N/A	N/A
264-3	2	22		110.9	N/A	N/A
264-4	1	21		110.9	N/A	N/A
264-5	ND	23		110.9	N/A	N/A

Surrogates. Surrogates are compounds of similar chemical composition that are spiked into each method blank, laboratory control sample, matrix spike/matrix spike duplicate, duplicate, and sample before extraction/derivitization. The percent recovery allows the accuracy of the method to be assessed for each QC sample and normal sample processed. The EPA method indicates that statistically based ranges should be calculated for the recoveries. The laboratory calculated ranges of concentration based on two standard deviations for all samples. Two surrogates were spiked into each sample: 2,4,6-tribromophenol derivitized (TBRP-d) and terphenyl-d14 (TPH-d14). Both surrogate concentrations in sample 264-5 were slightly below (33 and 30 µg/g) the lower limit of 33.3 µg/g.

Because the concentrations were just below the limits, no additional qualifiers were added beyond those previously discussed.

Matrix Spike/Matrix Spike Duplicate. As previously discussed, sludge composite samples 263-1 and 263-2 were spiked in duplicate and, as noted, the recoveries for one compound were low. Recoveries for DBBP and TBP were acceptable. The relative percent differences for spikes into sludge composite sample 263-1 were below the TSAP-required 20% upper limit. The relative percent differences for sludge composite sample 263-2 were slightly high (26 and 31%, respectively) for TBP and DBBP. Relative percent differences for the DBP-d were very high, as is expected given the previously discussed recoveries.

Duplicates. Sludge composite samples 263-1 and 263-2 were extracted and analyzed in duplicate. The relative percent differences were high for all compounds; however, this is due to the low concentrations (i.e., below reporting levels).

Method Blanks. Six method blanks were carried through preparation, derivitization, and analysis. The analytes of interest were undetected with the exception of two positive responses that were well below reporting limits: (0.4 μg/g DBP-d and 1.8 μg/g TBP).

Tunes. All tunes of the mass spectrometer were performed per SW-846 Method 8270C (EPA 1997) criteria and passed the criteria.

Initial and Continuing Calibration. All initial calibration criteria for the compounds of interest met the 15% difference for response. All system performance check compounds passed the minimum response criteria. The continuing calibration checks passed criteria with the exception of DBP-d run on March 8, 2000. Because the data are already estimated and are reported below the calibration level and reporting limit, no added data qualification is needed.

Internal Standard Areas. All internal standard areas met the criteria required by Method 8270 for the retention time windows and the area response, except for two method blanks, one laboratory control sample, three samples, and a duplicate (263-3, 263-4, 263-5, 263-1 and 263-1 duplicate). These samples have twice the amount of internal standard added. The data were calculated using the increased amount of internal standard and the data are within the limits for retention time and areas.

3.7.2.3 Summary

Based on the above information, the DPB-d is estimated due to the poor laboratory control sample and matrix spike/matrix spike duplicate recoveries and the failure of the CCC criteria for this compound. The results for all analytes were below reporting limits and calibration levels. The concentration of the DPF-d may have a low bias. Because these data are used to assess mobility and can be calculated from the TBP, the data are usable as estimated. DBBP and TBP data remain as reported, without flags.

3.7.3 Polychlorinated Biphenyls by Gas Chromatography

Analysis for PCBs was conducted to satisfy a regulatory requirement. PCBs were determined as Aroclors. However, concentrations of Aroclors were not totaled for each sludge composite sample, because a determination has not been made by the DOE, Richland Operations Office, and its contractors of how to include results that are "less than detection limit" in the total. In addition, the *Toxic Substances Control Act* (40 *Code of Federal Regulations* 261, the "MegaRule") requires that results be reported on a dry-weight basis; therefore, the moisture results (see Section 3.7.4) were used to correct the results for the "as-received" material (see Appendix C) for moisture content. Note that the dry-weight calculations were not to be performed by the laboratory, but are performed during this data quality assessment. Results for the sludge composite samples are presented in Table 3-41.

Table 3-41. Validated Data for Polychlorinated Biphenyl Concentration in Tank 241-Z-361

Composite	Interval (in.)		Aroclor (mg/kg dry weight)										Method Detection Limit (mg/kg dry weight)
	Top	Bottom	1232		1242		1248		1254		1260		
263-1	0.00	20.00	**		**	UJ	18.40		**		**		<3.45
263-2	20.00	34.25	**		**	UJ	8.17		**		**		<3.24
263-3	34.25	38.00	**		**	UJ	16.00		**		**		<2.68
263-4*	38.00	46.00	**		**	UJ	4.77		**		**		<2.09
263-5	46.00	59.00	**		**	UJ	**		**		**		<3.37
263-6	59.00	65.00	**		**	UJ	**		1.10		**		<0.68
263-7	65.00	74.00	**		**	UJ	**		4.07		**		<0.66
263-8	74.00	84.00	**		**	UJ	**		1.67		**		<0.58
264-1	0.00	16.50	**		**	UJ	24.20		**		**		<2.56
264-2	16.50	33.50	**		**	UJ	50.60	J	**		**		<4.29
264-3	46.00	62.00	**		**	UJ	160.00	J	**		**		<11.4
264-4	62.00	69.00	**	UJ	**	UJ	**	UJ	**	UJ	**	UJ	<3.04
264-5	69.00	84.00	**		**	UJ	7.74	J	**		**		<0.59

*Container was broken.
** Result is "less than detection limit."

3.7.3.1 Analytical Requirements

Sample preparation was requested by Methods 3540C, 3660, and 3665 (Soxhlet Extraction, Sulfur Cleanup, and Sulfuric Acid/Permanganate Cleanup, respectively). Analysis was requested by Method 8082 [Polychlorinated Biphenyls (PCBs) by Gas Chromatography] . These methods are implemented by laboratory methods LA-523-138 (includes soxhlet extraction and cleanups) and LA-523-427.

For each batch, the laboratory preparation method requires the following: (1) extraction of solid samples within 14 days of sampling and analysis of extracts within 40 days of extraction; (2) one method blank for each extraction batch; (3) one matrix spike and matrix spike duplicate for each analytical batch of 20 or fewer samples; (4) one laboratory control sample for each batch of 20 or fewer samples.

For each batch, the determinative method requires the quality control as specified in SW-846 Methods 8000B and 8082 (4th Edition, December 1996). The determinative quality control requirements are as follows: (1) a QC reference sample analyzed a minimum of one for each batch of twenty or fewer samples with a recovery of 80 to 120% of the certified value; (2) a calibration standard after each group of twenty samples; (3) at a minimum, analysis of a method blank, a matrix spike, a duplicate, and a laboratory control sample in each analytical batch; (4) surrogates added to each field sample and to the QC samples; (5) the laboratory control sample is a clean matrix, similar to the sample matrix, spiked with the same analytes at the same concentrations as the matrix spike. Also, the laboratory determinative method explicitly states that "Any matrix in which an extraction method is necessary will have surrogates added."

The TSAP (EQM 1999a) required one routine sample to be analyzed in duplicate for every twenty samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. In addition, at least one matrix spike sample was required for every twenty samples of similar matrix or preparation batch; therefore, at least one matrix spike sample was required for sludge composite samples. According to the TSAP, the required acceptance criteria for quality control data are 70 to 130% for laboratory control sample recovery, 75 to 125% for spike recovery, and less than 25 for the relative percent difference of duplicate samples. For both supernatant and sludge composite samples, the analytical requirements list an Action Limit for total PCBs of 50 parts per million. The low and high PQLs are 0.2 part per million.

Validation was required for PCB data (see Section 3.7.3.4)

3.7.3.2 Quality Control Review

Preparation of the sludge composites samples was performed by laboratory method LA-523-138 Rev B/Mod 2. The analysis was conducted by laboratory method LA-523-427 Rev A/Mod 1. The request for analysis of the supernatant was cancelled. (See HNF-1062, Attachment 4.)

The laboratory attempted to perform one extraction for both PCBs and semi-volatile organic compounds. For this approach, the surrogates for both classes of compounds were added to the same test portion before the extraction was performed. This resulted in interferences of the semi-volatile surrogates with the PCB analysis. The narrative of the data report (HNF-1692, Rev. 0B) noted that recovery of the decachlorobiphenyl (DCB) surrogate was high and recovery of the tetrachloro-m-xylene (TCX) surrogate was low. Although PCB results were not reported from the analysis of this extraction, the chromatograms were used to determine sample dilutions before analysis and the appropriate Aroclor to add for the matrix spike, as well as for confirmation of the Aroclor.

The second extractions were performed in three Worklists (#33160, 32646, and 32647). Analysis by gas chromatography was performed in three Worklists (#32649, 32650, and 33154).

Each extraction worklist included a laboratory control sample. Worklist #33160 included duplicate, matrix spike, and matrix spike duplicate samples. No extraction blank was noted on the worklist. The extraction was completed on March 24, 2000. Worklist #32646 included

extraction blank, duplicate, and matrix spike samples. No matrix spike duplicate (as required by the laboratory method) was noted on the worklist; however, this worklist meets the requirements of the TSAP (EQM 1999a). The extraction was completed on March 10, 2000. Worklist #32647 included blank, duplicate, matrix spike, and matrix spike duplicate samples. This extraction was initiated on March 13, 2000, and completed on March 16, 2000. The extractions generally were performed within two days; however, the third extraction was interrupted by an evacuation of the laboratory. Samples were not extracted within the 14-day holding time requirement; however, this is considered to be of no consequence to the project because PCBs are known as persistent compounds and not likely to have changed in character during the time period between sampling and extraction.

According to the narrative of the data report, the preparation of the first and second batches extended over two consecutive days because of limited glassware for performing the extractions. During extraction of the third batch, the test portion for sludge composite sample 264-2 (S00Z000036), its duplicate, matrix spike, and matrix spike duplicate were heating on the extractor for nearly 48 hours, rather than the usual 18 hours; however, the preparations did not go to dryness. In addition, on the second day, an insufficient amount of surrogate was added to the test portion for sample 264-5 (S00Z000039), which was expected to require a 20-fold dilution for analysis. Therefore, sludge composite sample 264-5 was reextracted on the third day with another method blank added to the preparation group. Note, however, that two blanks were not noted on the extraction worksheets included in the data report. All samples in this third preparation batch were analyzed in a single determinative batch.

Analysis was conducted on March 21, 2000; April 7, 2000; and April 6, 2000 for Worklists #32649, 32650, and 33154, respectively. Note that this is within the 40-day holding time requirement.

Regarding surrogate recovery, the narrative of the data report stated:

> The surrogate recoveries on the reanalysis were between 89.0 and 130.8% for the DCB and between 45.2 and 82.2% for TCX. All of these were within the method acceptance limits of 50 - 150% recovery, except for TCX for composite sample 264-4 (S00Z000038), which had the surrogate recovery of 45.2%. The DCB recovery for that sample was 103.4%. No Aroclors were observed in that sample at a detection limit of 1.0 ppm. Because at least one of the surrogate recoveries was within the acceptable limits, no additional analysis was requested.

It should also be noted that, according to the SW-846 Method 8082, TCX is the recommended surrogate when PCBs are analyzed as congeners; therefore, use of TCX as a surrogate was not required for this project because PCBs are being analyzed as Aroclors.

For preparation batch one, sludge composite sample 263-1 was run in duplicate; the relative percent difference was 16.9% which meets the TSAP (EQM 1999a) requirement. For preparation batch two, sludge composite sample 263-6 was run in duplicate and as the matrix spike duplicate; the relative percent differences were 102 and 25.1%, respectively. For preparation batch three, sludge composite sample 264-2 was run in duplicate and as the matrix

spike duplicate; the relative percent differences were 33.9 and -5561% (the large negative relative percent difference is due to the matrix spike recovery of -42.8% and matrix spike duplicate recovery of 39.8%; see below). Note that these relative percent differences do not meet the TSAP requirements.

Sludge composite samples 263-1 and 264-2 in batches one and three, respectively, were spiked with Aroclor 1248. The same Aroclor was used for the LCSs; recoveries were 96.0% and 104%, respectively, which meet the TSAP (EQM 1999a) requirements. Spike recoveries were 58.5 and -42.8% (negative 42.8%), respectively. Recovery for the MSD of sludge composite sample 264-2 was 39.8%. Note that these spike recoveries do not meet the TSAP requirements of 75 to 125%. The narrative of the data report attributes the poor recoveries "to matrix effects or sample variability which was further complicated by the need to use a small (~1 g) sample size."

Sludge composite sample 263-6 was spiked with Aroclor 1254. Aroclor 1254 was used for the laboratory control sample; recovery was 112%. This recovery meets the TSAP requirements (EQM 1999a). Spike recoveries for the MS and MSD were 122 and 157%, respectively. Note that the recovery for the matrix spike meets the TSAP requirements; however, the recovery for the MSD does not meet the TSAP requirements.

The TSAP required both low and high PQLs of 0.2 part per million. Although the data report identifies that most of the samples had nondetects for the Aroclors 1232, 1242, 1248, 1254, and 1260, in many cases the method detection limit for Aroclors in the samples was higher than the TSAP requirement (EQM 1999a). Specifically, for Worklist #32649, the method detection limits for the Aroclors was 1000 µg/kg, or 1.0 mg/kg (ppm), for sludge composite samples 263-1, 263-2, 263-3, 263-4, and 263-5, and blank sample S00Z000045. For Worklist #32650, the method detection limits were 400 µg/kg, or 0.4 mg/kg, for sludge composite sample 264-1. For Worklist #33154, the method detection limits were 1000 µg/kg, or 1.0 mg/kg, for sludge composite samples 264-2 and 264-4 and 4000 µg/kg, or 4.0 mg/kg, for sludge composite sample 264-3.

The narrative also states that:

> Some of the chromatograms have peaks identified that did not have the correct peak ratios for that Aroclor set. The chemist manually deleted these false positives before processing the data, but they still show up on the chromatograms. The sample summary sheets indicate "Operator disable compound identification" for Aroclors that were identified as false positives due to incorrect peak ratios.

3.7.3.3 Summary

Thirteen sludge composite samples were analyzed for PCBs. Not all QC samples required by the analytical methods were included in the worklists. Some requirements of the TSAP (EQM 1999a) were not met. However, the resulting data are considered sufficient for this project and were submitted for third-party data validation.

The TechLaw report stated: "Per Fluor Daniel instructions, no qualifiers were assigned based on holding times." If the data had been evaluated for the initial 14-day holding time, all undetected results would have been rejected and all detected results would have been qualified as estimates. However, PCBs are persistent and degrade slowly under environmental conditions. Therefore, the initial holding time has negligible influence on the data.

Instrument Performance and Calibrations. The report noted that the laboratory quantitated Aroclors on the basis of linear regression of five points, rather than by average response factor calibrations. Because the correlation coefficient of the linear regression for Aroclor 1242 was "less than 0.99% (sic)," the Aroclor results (which were nondetects) were qualified as estimates.

Blanks. All method blanks were acceptable with respect to Aroclors.

Accuracy. Requirements, as stated in the TechLaw report, are as follows: "matrix spike/matrix spike duplicate analyses are performed in duplicate using six compounds and must be within the established laboratory quality control limits...If spike recoveries are outside control limits, detected sample results less than 5 times the spike concentration are qualified as estimates...Sample results greater than five times the spike concentration require no qualification."

The report stated: "The results for Aroclor-1248 were qualified as estimates and flagged "J/UJ" in samples S00Z000036, S00Z000037, S00Z000038 and S00Z000039 due to MS/MSD recoveries outside QC limits. All other matrix spike/matrix spike duplicate recovery results were acceptable." Further information was offered in a later communication (Amy Ballow to Karl Pool, dated 15 May 2000), which stated:

> Typically, organic data is (sic) not qualified based solely on the results of MS/MSD data. Using professional judgement, only the Aroclor-1248 results were qualified because the matrix effect on the other Aroclors is unknown unless spiked. No additional qualifications were taken. Additionally, the laboratory did perform MS/MSD analyses for Aroclor-1254. (The samples only reported positive results for Aroclor-1248 and Aroclor-1254. Spiking the other Aroclors would not be required.)

For surrogate recovery, the TechLaw report stated the requirements as follows:

> When a surrogate compound recovery is outside the control window, all positively identified target compounds associated with the unacceptable surrogate recoveries are qualified as estimates...Nondetected compounds with surrogate recoveries less than the lower control limit are qualified as having an estimated detection limit...Undetected compounds with surrogate recoveries above the upper control limit require no qualification.

The report stated: "The non-detected results for all Aroclors in sample S00Z000038 were qualified as estimates and flagged "UJ" due to a low surrogate recovery for tetrachloro-m-xylene. All other surrogate recovery results were acceptable."

Precision. For RPD between MS and MSD samples, the TechLaw report stated the requirements as follows: "If RPD values are out of specification and the sample concentration is less than five times the spike concentration, all associated detected sample results are qualified as estimates...If RPD values are out of specification and the sample concentration is greater than five times the spike concentration, no qualification is required."

The report stated: "All matrix spike/matrix spike duplicate RPD results were acceptable."

The TechLaw report was questioned by the project because the MS/MSD for sample S00Z000032 was outside the relative percent difference criterion. TechLaw responded as follows:

> The MS/MSD analyses of sample S00Z000032 reported recoveries of 122 and 157%, respectively. The MSD analysis exceeded the QC limits of 50-150%. According to the "Data Validation Procedures for Chemical Analyses", action is taken on MS/MSD recoveries using surrogate recovery data. (See page 4-8 of guidelines). If MS/MSD recoveries >UCL [greater than upper control limit], then qualification is taken if the surrogate recoveries are <LCL [less than lower control limit] or >UCL [greater than upper control limit]. In this case, the surrogate recoveries were within criteria, and no action was taken. Additionally, the relative percent difference (RPD) for the MS/MSD analyses of sample S00Z000032 and all LCS recoveries were within criteria.

System Performance. For compound identification, the TechLaw report stated the requirements as follows:

> The identification of detected compounds are confirmed to investigate the possibility of false positives or false negatives. If the qualitative criteria are not net, detected results are qualified as follows: Misidentified peaks outside the retention time window are reported at the CRQL [contract required quantitation limit] if no interferences are noted. If the misidentified peak interferes with a target peak then the reported value is qualified as an estimate...If detected results have not been analyzed on dissimilar columns, the results are rejected...

The report stated: "All compound identification criteria were met." Method 8082, Section 1.5, states: "Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique." Although not included in the TechLaw report, it should be mentioned that second column confirmation was not performed. However, according to the narrative of the data report: "The original analysis results were used to determine sample dilution before analysis and the appropriate Aroclor to add for the matrix spike analysis." Therefore, identification of Aroclors present in the samples was performed on this "original analysis" and is sufficient for this project.

Analytical Detection Levels. The TechLaw report stated the requirements as follows: "Reported analytical detection levels meet the required criteria." The report stated: "No analytes were reported with the laboratory reported quantitation levels above the analyte specific CRQL."

Although TechLaw was not questioned about this previously, it should be noted that many samples had both nondetects and reported data with MDLs higher than the TSAP (EQM 1999a) requirement of the low and high PQLs of 0.2 ppm (see Section 3.7.3.1). However, the detection limits were well below the TSAP (i.e., TSCA) Action Limit. Failure to meet the TSAP requirement for PQLs does not impact the usability of the data.

In summary, the results for Aroclor 1248 were qualified as estimates and flagged (J/UJ) for sludge composite samples 264-2, 264-3, 264-4, and 264-5 because of MS/MSD recoveries outside QC limits. The nondetected results for all Aroclors in sludge composite sample 264-4 were qualified as estimates and flagged (UJ) because of low surrogate recovery for tetrachloro-m-xylene. All Aroclor 1242 results (all less than detection limit) for both cores were qualified as estimates and flagged (UJ), because the correlation coefficient of the five point curve used to quantitate Aroclor 1242 was less than 0.99. Although data flagged "J" are considered to be estimates, the data are useable for decision-making. All other results are considered to be accurate within the standard error associated with the methods.

3.7.4 Moisture Content

Determination of moisture content, or percent water, was performed to support treatment options. The results also are used to calculate PCBs on a dry-weight basis. Results for moisture content are presented in Table 3-42.

Table 3-42. Results for Moisture Content of Sludge Composite Samples from Tank 241-Z-361

Composite	Interval (in.)		Moisture (%)		Data Assessment
	Top	Bottom	Sample	Duplicate	Qualifier
263-1	0.00	20.00	71.0	72.9	
263-2	20.00	34.25	69.1		
263-3	34.25	38.00	62.7	63.4	
263-4*	38.00	46.00	52.2		J
263-5	46.00	59.00	70.3		
263-6	59.00	65.00	70.8		
263-7	65.00	74.00	69.5		
263-8	74.00	84.00	65.4		
264-1	0.00	16.50	84.4	84.6	
264-2	16.50	33.50	76.7		
264-3	46.00	62.00	65.0		
264-4	62.00	69.00	67.1		
264-5	69.00	84.00	66.3		

* Container was broken.

3.7.4.1 Analytical Requirements

Analysis was requested by a gravimetric method which is implemented by laboratory method LA-564-101: A known weight of sample is heated at a constant temperature for several hours before being cooled to room temperature in a dessicator and then weighed. The method specifies the constant temperature as 120°C, but allows for alternative drying temperatures (e.g., 180°C for drying total dissolved solids or 105°C for drying some samples) if specified in the worklist comments. For each batch, the method required one laboratory control sample standard and one duplicate for each ten samples or batch.

The TSAP required moisture content to be determined at 105°C (EQM 1999a). One routine sample was required to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. MS samples were not required. According to the TSAP, the required acceptance criteria for QC data are 80 to 120% for LCS recovery and less than 30 for the RPD of duplicate samples.

Validation of moisture data was not required by the TSAP.

3.7.4.2 QC Review

Moisture was determined by the required method LA-564-101 Rev G/Mod 3, although the data package does not confirm that the correct temperature was used for the procedure.

Worklist #32079 included the LCS standard and a duplicate sample. Percent moisture for the LCS standard was 58.1%. With an actual moisture content of 59.5% (as stated in the data package), the recovery for the standard was 97.647%. Sludge composite sample 263-1 was run in duplicate; the RPD was 2.641. Samples in this batch were 263-2, 263-5, 263-7, and 263-8. Worklist #32080 included the LCS standard and a duplicate sample. Percent moisture for the LCS standard was 57.3%; therefore, the recovery was 96.303%. Sludge composite sample 264-1 was run in duplicate; the RPD was 0.237. Samples in this batch were 263-6, 264-2, 264-3, 264-4, and 264.5.

Worklist #32318 included the LCS standard and a duplicate sample. Percent moisture for the LCS standard was 58.6; recovery for the standard was 98.487%. Sludge composite sample 263-3 was run in duplicate; the RPD was 1.11. The only other sample in this batch was 263-4.

The LCS standard is called "Terliq." It is composed of common waste tank salts and is prepared as described in laboratory procedure LR-332-111. The established moisture value is based upon drying at 120°C, so it is not surprising that the recovery for all three batches is low.

The sample container for sludge composite sample 263-4 (i.e., S99Z000221) was discovered to be broken when examined at the laboratory and before subsampling for laboratory analysis. The subsamples affected are as follows: S99Z000236; S99Z000240; S99Z000244; S00Z000283; S99Z0000296; S99Z000309; S99Z000322; S00Z000031. The laboratory was advised to take the

test portion for moisture content from the middle of the sample (personal communication, Chuck Miller to Ruth Esch, no date). Substantial moisture loss is expected to have occurred before the analysis, probably in the range of 13 to 32%, as may be surmised from examination of data for the other composite samples.

Loss of moisture, because of the breakage, may affect the results for all analyses of the subject subsamples, except for particle size analysis and particle size distribution. Even an analytical result that is not corrected for moisture content will have higher "as received" concentration than it would if the sample had not been affected by an unknown loss in moisture. Also, the loss of moisture is not a constant and cannot be quantified. Given the variability in the strata, the location of the test portion within the jar, and analysis variability, the data are not flagged but are noted in the summary tables. A comparison of the total alpha before and after compositing showed no effect because of the loss of moisture due to the broken jar.

3.7.4.3 Summary

Thirteen sludge composite samples were analyzed for moisture content. QC requirements were met. Although one sample may be compromised because of a broken container, the remaining data show that the moisture content varies from 62.7 to 84.4%. The data are useable for this project.

3.7.5 Particle Size Analysis/Particle Size Distribution by Laser Diffraction

Determination of particle size analysis and distribution was performed to support treatment options. Statistical data for particle size are presented in Table 3-43. The particle size data and distribution curves are presented in Appendix C.

Table 3-43. *Statistical Data for Particle Size Analysis of* Sludge Composite Samples from Tank 241-Z-361 (2 sheets)

Composite		Sample	Median	Mode	Mean	Range
			(µm)			
263	1	S00Z000027	188	236	167	9 - 450
263	2	S00Z000028	188	236	167	7 - 450
263	3	S99Z000239	113	157	103	6 - 170
263	4	S99Z000240	187	236	166	6 - 450
263	5	S99Z000241	186	237	166	5 - 450
263	6	S00Z000032	188	236	167	8 - 450
263	7	S99Z000242	188	236	167	7 - 450
263	8	S00Z000034	111	124	101	7 - 170
263	8	duplicate	188	236	167	7 - 450

Table 3-43. Statistical Data for Particle Size Analysis of Sludge Composite Samples from Tank 241-Z-361 (2 sheets)

Composite		Sample	Median	Mode	Mean	Range
			(μm)			
264	1	S00Z000035	181	237	161	0.2 - 450
264	2	S99Z000276	112	124	102	8 - 170
264	3	S00Z000037	184	237	164	5 - 450
264	4	S99Z000251	85	94	78	15 - 150
264	5	S99Z000252	84	93	74	13 - 130

3.7.5.1 Analytical Requirements

Analysis was requested by analytical method APHA 2560 which, according to the TSAP (EQM 1999a), is implemented by laboratory method LT-519-101. The particle size analysis is performed on a Horiba LA 910 Particle Size Analyzer. The instrument uses laser diffraction to determine the volume distribution of particles with size fractions ranging from 0.02 μm to 1020 μm. Test portions are diluted with enough deionized water to form a well-separated suspension. All measurements are performed at ambient laboratory temperature, typically about 25°C. A laboratory control sample is analyzed with each batch.

The TSAP acceptance criteria for particle size analysis is a relative percent difference of less than 30. Note that the LCS is marked as "N/A" or not applicable.

Validation was not required for particle size analysis and distribution data.

3.7.5.2 QC Review

Particle size analysis and distribution were determined by the required laboratory method; however, note that the correct number is LT-519-105. An aliquot of each sludge composite sample was analyzed for particle size analysis and distribution by laser diffraction of an aqueous suspension of the sample material. All samples were analyzed in one batch under Worklist #32018. The batch included one duplicate sample and one LCS standard. The standard results were median and mode equal to 0.947 m and mean equal to 0.955 μm; 88.5% of particles were in the 1.005 μm to less than 1.151 μm fraction and 11.5% were in the 1.151 μm to less than 1.318 μm fraction. The "known" mean value is 0.993 μm. The particle size analysis method evaluates the RPD of the "known" mean to the standard sample mean to determine if the standard results are acceptable. In this case, according to the narrative, the mean is within the 10% criterion specified in the method.

Particle size data are presented in Attachment 2 of the data report (FDH 2000b, pp. 101-120). In several instances, the subsamples that were originally identified for particle size analysis were of insufficient size, so test portions were removed from different subsamples. This is clearly identified on the instrument printouts and also is shown in a table included in the narrative.

DQA of the particle size data is atypical. For data from samples with distributions of "binned" values, the median and mode are more descriptive and likely more important than mean for describing the results; whereas for a well-sorted sample (such as the LCS standard), the mean is a simple statistic for evaluating the capability of the instrument to give consistent particle size measurements.

Sludge composite sample 263-8 (i.e., S00Z000034) was run as the duplicate sample. The RPD of the means was calculated as 49%. The results for summary descriptive statistics are as follows:

Composite	Median (μm)	Mode (μm)	Mean (μm)	Range – low	Range - high
263-8	111.171	124.401	100.607	6.720	174.6
263-8 dup	188.381	236.039	167.254	6.720	451.6

By inspection, it was noted that the duplicate sample is nearly identical to the particle size distribution for sludge composite sample 263-7 (S99Z000242), which is the composite immediately above the duplicate sample.

Composite	Median (μm)	Mode (μm)	Mean (μm)	Range – low	Range - high
263-8 dup	188.381	236.039	167.254	6.720	451.6
263-7	188.066	236.170	167.096	6.720	451.6

The issue of the high RPD of the duplicate means was discussed with the laboratory (personal communication, Ruth Esch to Chuck Miller, no date). Test portions were collected from each sludge composite subsample by aspirating a small quantity of the solid material by using a transfer pipette. For duplicate analysis, a second aliquot was aspirated from a different location within the composite subsample. The method of obtaining test portions is capable of collecting very small aliquots of the sample material. It is apparent that the test portions taken to be duplicate samples have very different particle size analysis results and particle size distributions. Because the overlying sludge composite sample is more like the duplicate test portion, there might be incomplete mixing of the composite samples with respect to small size particles. Also, the method of subsampling may preferentially extract certain particle sizes of material due to the diameter of the pipette. (Note: research on particle size analysis by elution was conducted and published by R.L. Jones and A. Beavers, University of Illinois, in the 1960s, exact publication date unavailable. The sorting of particle size classes was related to the diameter of the container, such as a pipette or separatory funnel, as the sample suspended in a fluid, such as water or air, moves through a series of containers of different diameter.) In addition, the data indicate that the size of the test portion for particle size analysis and the manner in which it is obtained do not result in a representative test portion.

Finally, this lack of representativeness for a small test portion estimated to be less than 100 mg (personal communication, Bill Winters to Joan Bartz, May 17, 2001) should be considered when evaluating the limitations of the remaining data for this project.

3.7.5.3 Summary

Thirteen sludge composite samples were analyzed for particle size analysis and distribution. QC requirements were met. The data are useable for this project.

3.7.6 Mercury by Cold-Vapor Atomic Absorption

Analysis for mercury was conducted to satisfy a regulatory driver. Results are presented in Table 3-44.

Table 3-44. Mercury Concentration in Sludge Composite Samples from Tank 241-Z-361

Composite	Interval (in.)		Mercury (µg/g)		Data Assessment Qualifier
	Top	Bottom	Sample	Duplicate	
263-1	0.00	20.00	177		
263-2	20.00	34.25	63.5		
263-3	34.25	38.00	20.5		
263-4*	38.00	46.00	36.0		
263-5	46.00	59.00	21.3		
263-6	59.00	65.00	18.6	14.1	J
263-7	65.00	74.00	23.4		
263-8	74.00	84.00	20.7		
264-1	0.00	16.50	65.1	69.6	
264-2	16.50	33.50	83.0		
264-3	46.00	62.00	26.8		
264-4	62.00	69.00	37.6		
264-5	69.00	84.00	67.7		

*Sample container was broken.

3.7.6.1 Analytical Requirements

Analysis was requested by Method 7470A (for liquid waste)/7471A (for solid or semisolid waste) which is implemented by laboratory method LA-325-106. Mercury in the test portion is reduced to elemental mercury, then volatilized from solution in a closed system. The mercury vapor passes through a quartz cell positioned in the light path of an atomic absorption spectrophotometer. Mercury concentration is measured as a function of absorbance at 253.7 nm.

For each batch, the method requires a preparation blank (which is the equivalent to the initial calibration blank and the continuing calibration blank) and a solid LCS which is digested with the batch. For each group of samples, the method requires at least one MS sample and one duplicate or MSD sample. For operation of the atomic absorption spectrophotometer, the method requires an initial calibration verification standard (i.e., a standard that is independent of the calibration standards), a continuing calibration verification (CCV) standard run at least every ten samples and at the end of each batch, and the preparation blank run as the initial and continuing calibration blank.

The TSAP (EQM 1999a) required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. In addition, at least one MS sample was required for every 20 samples of similar matrix or preparation batch; therefore, at least one MS sample was required for sludge composite samples. According to the TSAP, the required acceptance criteria for QC data are 80 to 120% for LCS recovery, 75 to 125% for spike recovery, and less than 20% for the RPD of duplicate samples. For sludge composite samples, the TSAP lists the Action Limits of 0.5 parts per million for total mercury and 0.025 parts per million as the Toxicity Characteristic Leaching Procedure (TCLP) Action Limit. It must be clarified that the 0.025 ppm is not TCLP but based on the Universal Treatment Standards (UTS) limits that are applied to the Underlying Hazardous Constituents (UHCs). In addition, both the low and high PQLs are 0.04 parts per million. For supernatant samples, the Action Limits are the same, but the low PQL is 0.02 part per million and the high PQL is 0.2 part per million.

Validation was not required for mercury data.

3.7.6.2 QC Review

The analysis was performed directly on the sludge composite samples by laboratory method LA-325-106 Rev. A/Mod 4. Only sludge composite samples were analyzed. The analytical request for supernatant was cancelled (FDH 2000c, Attachment 5).

Worklist #32006 included an initial calibration verification, an initial calibration blank, a CCV, and a continuing calibration blank. Recoveries for the initial and CCV standards were 107 and 106%, respectively. Mercury detected in the initial calibration blank was negligible in comparison to the sample concentrations. Sludge composite sample 264-1 was run as both the duplicate and MS samples. RPD of the duplicate samples was 6.67. MS recovery was 82.9%. Sludge composite samples included 264-2, 264-3, 264-4, and 264-5.

Worklist #32032 included an initial calibration verification, an initial calibration blank, a CCV, and a continuing calibration blank. Recoveries for the initial and CCV standards were 97.4 and 103%, respectively. Mercury detected in both the initial and continuing calibration blanks was negligible in comparison to the sample concentrations. Sludge composite sample 263-1 was run as the duplicate and MS samples. The analyst notes that this sample gave results that were outside the calibration range, even when diluted (60 times dilution factor). Therefore, sludge composite sample was rerun in Worklist #32417. (The reported data 212 μg/g and 221 μg/g for the duplicate samples, with a RPD of 4.29; recovery of the MS, 72.1%.) These data may be considered an estimate of the sample concentration. The review noted a records anomaly in that the run log lists test portion weights and instrument readings for sample #S99Z000246 which is apparently a transposition of #S99Z000264, (i.e., sludge composite sample 263-1). Sludge composite samples included 263-2, 263-3, 263-4, and 263-5.

Worklist #32033 included an initial calibration verification, an initial calibration blank, a CCV, and a continuing calibration blank. Recoveries for the initial and CCV standards were 107 and 109%, respectively. Mercury detected in the initial calibration blank was negligible in

comparison to the sample concentrations. Sludge composite sample 263-6 was run as the duplicate and MS samples. RPD for the duplicate samples was 27.647. Reanalysis was not requested based on duplicate sample results. Based on high RPD, the result for sample 263-6 is considered to be estimated. MS recovery was 90.5%. Sludge composite samples included 263-7 and 263-8.

Worklist #32417 included an initial calibration verification, an initial calibration blank, a CCV, and a continuing calibration blank. Recoveries for the initial and CCV standards were 108 and 93.7%, respectively. No mercury was detected in the initial or continuing calibration blank. Sludge composite sample 263-1 was rerun in duplicate in this worklist. RPD for the duplicates was 8.31. Both sludge composite sample 263-1 and a sample from another project were run as MS samples. MS recovery was 82.6% for sludge composite sample 263-1 (and 104% for the sample from the unrelated project).

Examination of the standard curves for each day of analysis indicates variability in the instrument response to calibration with the same standard from day to day. The cause of this response variability is not apparent. However, because the standard curve for each day was linear, this variability should not have a detrimental effect on quantitation of results. The laboratory project manager confirmed that the laboratory staff replaced tubing and cleaned the instrument optics sometime during the period of theses analyses which could account for the observed variation (personal communication, Ruth Esch to Chuck Miller, no date).

Limitations of the data include the following: Spike recoveries for sludge composite samples 263-1, 263-6, and 264-1 were 82.6% (Worklist #32417), 90.5% (Worklist #32033), and 82.9% (Worklist #32006), respectively. These generally low recoveries suggest that the data obtained for mercury concentration may be biased low. Also, the review could not identify a solid LCS (which is to be digested with the batch) for any of the worklists, as is required by the laboratory method. Results for a solid LCS might demonstrate whether or not such bias were present.

EPA SW-846 Method 7471A (for solid or semisolid waste) specifies that samples are weighed out in triplicate test portions of 0.2 g, whereas laboratory method LA-325-106 states that test portions are usually 0.04 g and that the number of replicates is determined by customer. (Note that the EPA SW-846 Hotline has allowed one digestion of 0.6 g instead of three separate digestions.) Test portions used for this project were generally between 0.04 and 0.06 g (weighed accurately on a four-place balance). The test portion for the reported result for sludge composite sample 263-1 was 0.0203 g. The smaller test portion was necessary so that the sample could be analyzed within the calibration range of the instrumentation. Because replicate analyses were not specified for this project, the only replicate analyses were the duplicates for QC purposes. This might be a concern because such small test portions may not be representative of the sludge, and replicate data do not exist to support whether or not the test portions were representative. Data for total organic and total inorganic carbon and for particle size analysis suggest that larger test portions may be necessary to represent this matrix.

3.7.6.3 Summary

Thirteen sludge composite samples were analyzed for mercury. Although a solid LCS was not digested for analysis as required, the remaining QC was performed and was acceptable; however, all MS results were between 82.6 to 90.5%, which may indicate that results are biased low but within allowable limits. The MDLs ranged from 0.600 to 3.94 μg/g. Results ranged from 18.6 to 177 μg/g, which exceeded the Action Limits for both total mercury and TCLP mercury. Therefore, achieving the low and high PQLs was unnecessary.

3.7.7 Metals by ICP Spectrometry

Metals were requested using two different preparations: acid leaching/digestion and fusion with potassium hydroxide (KOH). The resulting prepared test portions were analyzed by ICP spectrometry. The results for analysis of the acid leachate are presented in Tables 3-45 and 3-46. Table 3-45 addresses six of the eight toxicity characteristic metals. Mercury results are presented in Table 3-44. Selenium was not requested by the TSAP. Table 3-46 addresses the remainder of the metals determined in the acid leachate.

Results for analysis of the fusion preparation are presented in Table 3-47. These metals are primarily of interest in evaluating treatment options. Therefore, in Table 3-48, the results also are expressed as the metal oxide concentrations. This is sometimes referred to as the "whole rock analysis." Details on calculation of the metal oxide equivalent concentrations may be found in the narrative of the data report (FDH 2000c). Phosphorus determined using the acid leachate also is included in Table 3-48 because it is of interest in evaluating treatment options.

3.7.7.1 Analytical Requirements

Validation was not required for metals data.

Acid Leachate/Digestion. The acid leachate of the sludge composite samples and the supernatant were analyzed primarily for metals of regulatory interest related to waste designation. Drivers and data quality requirements for the metals determined in the acid leachate are given in Table 3-49.

Preparation of the samples was requested by Methods 3050A/3051, which is implemented by laboratory method LA-505-163. Analysis was requested by Method 6010B, which is implemented by laboratory method LA-505-161.

Fusion. The KOH-fused sludge composite samples were analyzed for total metals in support of treatment options and used to calculate results presented as oxides, i.e., whole rock analysis. The applicable practical quantitation limits are given in Table 3-50. There are no action limits.

Preparation of the samples was requested by Methods 3050A/3051, which are implemented by laboratory method LA-549-141. Analysis was requested by Method 6010B, which is implemented by laboratory method LA-505-161.

Table 3-45. Concentration of Toxicity Characteristic Metals by Nitric Acid Leaching of Sludge Composite Samples from Tank 241-Z-361*

Composite	Interval (in.)		Metal Concentration by Acid Leaching (µg/g)											
			Arsenic		Barium		Cadmium		Chromium		Lead		Silver	
	Top	Bottom	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL
263-1	0.00	20.00	<20.1	20.0	111	10.0	54.7	1.00	4030	2.00	367	20.0	34.6	2.00
263-2	20.00	34.25	<19.5	19.5	105	9.74	56.7	0.974	936	1.95	120	19.5	15.6	1.95
263-3	34.25	38.00	<9.8	9.8	104	4.90	45.0	0.49	691	0.98	75.8	9.80	24.4	0.98
263-4**	38.00	46.00	<10.1	10.1	111	5.06	32.8	0.506	710	1.01	32.0	10.1	59.3	1.01
263-5	46.00	59.00	<10.1	10.1	152	5.06	74.7	0.506	926	1.01	34.5	10.1	40.6	1.01
263-6	59.00	65.00	<19.9	19.9	116	9.96	1.48	0.996	1340	1.99	33.8	19.9	17.2	1.99
263-7	65.00	74.00	<19.8	19.8	131	9.92	2.15	0.992	2150	1.98	91.9	19.8	29.1	1.98
263-8	74.00	84.00	<40.2	40.2	197	20.0	21.4	2.00	10000	4.02	446	40.2	182	4.02
264-1	0.00	16.50	23.4	20.3	87.1	10.1	17.8	1.01	5910	20.3	136	20.3	17.8	2.03
264-2	16.50	33.50	<20.4	20.4	93.0	10.2	58.7	1.02	3090	2.04	263	20.4	22.4	2.04
264-3	46.00	62.00	<19.7	19.7	192	9.86	112	0.986	1110	1.97	53.6	19.7	60.3	1.97
264-4	62.00	69.00	<20.7	20.7	114	10.3	16.0	1.03	1220	2.07	49.4	20.7	17.2	2.07
264-5	69.00	84.00	<21.1	21.1	144	10.5	17.1	1.05	4260	2.11	173	21.1	128	2.11

*Selenium was not reported. Mercury is presented in Table 3-44.
** Container was broken.

Table 3-46. Concentration of Selected Metals by Nitric Acid Leaching of Sludge Composite Samples from Tank 241-Z-361

Composite	Interval (in.)		Metal Concentration by Acid Leaching (µg/g)																	
			Beryllium		Nickel		Zinc		Sulfur		Phosphorus		Potassium		Lithium		Uranium		Zirconium	
	Top	Bottom	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL
263-1	0.00	20.00	<1.00	1.00	1530	4.02	393	2.00	748	20.0	578	40.2	<100	100	75.1	2.00	115	100	75.7	2.00
263-2	20.00	34.25	<0.975	0.974	415	3.90	265	1.95	441	19.5	141	39	<97.5	97.4	222	1.95	<97.5	97.4	18.0	1.95
263-3	34.25	38.00	<0.49	0.49	86.7	1.96	124	0.98	448	9.80	133	19.6	<49.0	49.0	31.3	0.98	<49	49	3.89	0.98
263-4*	38.00	46.00	<0.506	0.506	56.7	2.03	58.5	1.01	499	10.1	173	20.3	<50.6	50.6	95.9	1.01	<50.6	50.6	5.69	1.01
263-5	46.00	59.00	<0.506	0.506	68.3	2.03	88.9	1.01	408	10.1	156	20.3	<50.6	50.6	230	1.01	<50.6	50.6	4.36	1.01
263-6	59.00	65.00	<0.996	0.996	210	3.98	177	1.99	433	19.9	139	39.8	<99.6	99.6	72.3	1.99	<99.6	99.6	<1.99	1.99
263-7	65.00	74.00	<0.992	0.992	325	3.96	303	1.98	423	19.8	169	39.6	<99.2	99.2	275	1.98	<99.2	99.2	3.84	1.98
263-8	74.00	84.00	<2.01	2.00	1580	8.04	622	4.02	1090	40.2	831	80.4	270	200	101	4.02	<201	200	36.3	4.02
264-1	0.00	16.50	<1.01	1.01	3360	40.5	263	2.03	683	20.3	656	40.5	<101	101	13.8	2.03	138	101	193	2.03
264-2	16.50	33.50	<1.02	1.02	1310	4.07	446	2.04	500	20.4	405	40.7	<102	102	374	2.04	142	102	48.0	2.04
264-3	46.00	62.00	<0.986	0.986	91.1	3.94	109	1.97	471	19.7	201	39.4	<98.6	98.6	94.2	1.97	<98.6	98.6	8.70	1.97
264-4	62.00	69.00	<1.03	1.03	196	4.14	180	2.07	362	20.7	135	41.4	<103	103	237	2.07	<103	103	3.31	2.07
264-5	69.00	84.00	<1.05	1.05	680	4.21	367	2.11	529	21.1	306	42.1	<105	105	89.9	2.11	<105	105	14.0	2.11

*Container was broken.

Table 3-47. Concentration of Metals of Interest by Fusion of Sludge Composite Samples from Tank 241-Z-361

Composite	Interval (in.)		Metal Concentration by Fusion (µg/g)																	
			Aluminum		Calcium		Chromium		Iron		Magnesium		Manganese		Silicon		Sodium		Titanium	
	Top	Bottom	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL	Result	DL
263-1	0.00	20.00	10100	1010	39300	2020	5050	202	44800	1010	7290	2020	565	202	2100	1010	11000	2020	276	202
263-2	20.00	34.25	26400	978	32600	1960	1260	196	28200	978	5030	1960	301	196	1640	978	39200	1960	<196	196
263-3	34.25	38.00	46500	1000	41200	2010	881	201	6970	1000	4910	2010	286	201	1540	1000	17200	2010	<201	201
263-4*	38.00	46.00	38200	1010	49500	2020	815	202	3730	1010	5120	2020	<202	202	1180	1010	7830	2020	<202	202
263-5	46.00	59.00	51200	1000	70100	2010	1420	201	3840	1000	6940	2010	<201	201	2170	1000	9610	2010	<201	201
263-6	59.00	65.00	27100	1010	60600	2010	1380	201	3850	1010	10800	2010	<201	201	1560	1010	8490	2010	<201	201
263-7	65.00	74.00	19900	1000	73100	2010	2320	201	6830	1000	8810	2010	422	201	2470	1000	5860	2010	<201	201
263-8	74.00	84.00	2690	978	103000	1960	8560	196	20600	978	6950	1960	<196	196	<978	978	9240	1960	<196	196
264-1	0.00	16.50	1390	994	28500	1990	6220	199	33000	994	4890	1990	771	199	2050	994	3290	1990	<199	199
264-2	16.50	33.50	10300	965	32800	1930	3210	193	31800	965	6320	1930	635	193	1780	965	23800	1930	216	193
264-3	46.00	62.00	39200	956	70100	1910	1270	191	4500	956	6350	1910	<191	191	1480	956	10700	1910	<191	191
264-4	62.00	69.00	30800	996	62100	1990	1400	199	4680	996	10700	1990	<199	199	1590	996	7040	1990	<199	199
264-5	69.00	84.00	12200	973	91600	1950	5040	195	12700	973	8530	1950	439	195	1950	973	6620	1950	<195	195

* Container was broken.

Table 3-48. Major Metals Expressed as Metal Oxide Concentration for Sludge Composite Samples from Tank 241-Z-361*

Composite	Interval (in.)		Metal Oxide Concentration (µg/g)									
			Aluminum	Calcium	Chromium	Iron	Magnesium	Manganese	Phosphorus	Silicon	Sodium	Titanium
	Top	Bottom	Al_2O_3	CaO	Cr_2O_3	Fe_2O_3	MgO	MnO	P_2O_3	SiO_2	Na_2O	TiO_2
263-1	0.00	20.00	18300	5440	7220	63100	11400	713	1300	3790	13100	402
263-2	20.00	34.25	49100	44500	1740	39600	7680	373	323	3170	50700	<327
263-3	34.25	38.00	86700	58600	1170	10000	7990	371	305	2990	21700	<335
263-4**	38.00	46.00	71600	70800	1270	5330	8660	<261	396	2800	10600	<337
263-5	46.00	59.00	96700	98100	2080	5490	11500	<260	357	4640	13000	<335
263-6	59.00	65.00	52500	87600	2100	5780	18600	<260	318	3440	10900	<335
263-7	65.00	74.00	37600	102000	3390	9760	14600	545	387	5280	7900	<335
263-8	74.00	84.00	5080	144000	12500	29500	11500	<253	1900	<2090	12500	<327
264-1	0.00	16.50	2990	38100	9130	57500	7840	961	1470	4750	4180	<332
264-2	16.50	33.50	19500	45900	4690	45500	10500	820	928	3810	32100	360
264-3	46.00	62.00	74100	98100	1860	6430	10500	<247	461	3170	14400	<319
264-4	62.00	69.00	58200	86900	2050	6690	17700	<257	309	3400	9490	<332
264-5	69.00	84.00	23100	128000	7370	18200	14100	567	701	4170	8920	<325

* Data taken from FDH (2000c), pages 17 and 18.

** Container was broken.

Table 3-49. Analytical Objectives for Analysis of Nitric Acid Leachate of the Sludge Composite and Supernatant Samples from Tank 241-Z-361

Metal	Driver for analysis			Action Limit (ppm)		Practical Quantitation Limit (ppm)			
	Waste Designation	RCRA[1] Parameter	Treatment Options			Sludge Composite Samples		Supernatant Samples	
				Total	TCLP[2]	Low	High	Low	High
Arsenic	X			100	5	100	8000	1	50
Barium	X			420	21	50	4000	0.5	25
Beryllium	X			24.4	1.22	5	400	0.05	2.5
Cadmium	X			2.2	0.11	5	400	0.05	2.5
Chromium	X		X	12	0.6	10	800	0.1	5
Lead	X			15	0.75	100	8000	1	50
Nickel	X			220	11	20	1600	0.2	10
Silver	X			2.8	0.14	50	4000	0.1	5
Zinc	X			86	4.3	10	800	0.1	5
Sulfur		X[3]		500		1	50	1	50
Potassium			X			500	40000	5	250
Lithium			X			100	8000	0.1	5
Uranium			X			10	800	5	250
Zirconium			X			10	800	0.1	5
Phosphorus			X			200	16000		

1. RCRA = Resource Conservation and Recovery Act of 1976
2. Although labeled as "TCLP" in the TSAP, this column also includes requirements to meet the UTS.
3. Data are used to evaluate sulfide content.

3.7.7.2 QC Review

Preparation of Samples and Determination of Metal Concentration. Sludge composite samples were acid leached according to laboratory method LA-505-163 Rev. B/ Mod 3. Samples were prepared in three Worklists (#32014, 32027, 32054). Metal concentrations were determined by the required method LA-505-161 Rev. C/Mod 5. Analysis was performed in three Worklists (#32128, 32168, 32573).

- Worklist #32014 included sludge composite samples 263-8, 264-1, 264-2, 264-3, 264-4, and 264-5. Analysis was performed in Worklist #32168.

- Worklist #32027 included sludge composite samples 263-1, 263-2, 263-3, 263-4, 263-5, 263-6, and 263-7. Analysis was performed in Worklist #32128.

- Worklist #32054 included the drainable liquid sample from core segment 2, the field blank associated with core 264, and the lithium bromide blanks associated with cores 263 and 264. Analysis was performed in Worklist #32573. These results are presented in Appendix C.

A separate test portion of each sludge composite sample was fused according to laboratory method LA-549-141 Rev. G/Mod 1. Samples were prepared in three Worklists (#32015, 32028, 32029). Metal concentrations were determined by the required method LA-505-161 Rev. C/Mod 5. Analysis was performed in three Worklists (#32138, 32177, 32911).

- Worklist #32015 included all sludge composite samples from core 264. Analysis was performed in Worklist #32138.

- Worklist #32028 included sludge composite samples 263-1, 263-2, 263-3, 263-4 and two samples for radionuclide analysis. Analysis was performed in Worklist #32911.

- Worklist #32029 included sludge composite samples 263-5, 263-6, 263-7, 263-8 and two samples for radionuclide analysis. Analysis was performed in Worklist #32177.

Table 3-50. Analytical Objectives for Analysis of the Fusion Digest of Sludge Composite Samples from Tank 241-Z-361

Metal	Practical Quantitation Limits (ppm)	
	Low	High
Aluminum	50	4000
Calcium	100	8000
Chromium	10	800
Iron	50	4000
Magnesium	100	8000
Manganese	10	800
Silicon	50	4000
Sodium	100	8000
Titanium	10	800

The following quality control was examined:

- Accuracy, as indicated by analysis of LCSs and MSs.
- Accuracy, as indicated by calibration with standards.
- Precision, as indicated by analysis of duplicate samples.
- Blanks, including method performance blanks, field blanks, and the lithium bromide blanks
- Result calculations.

Accuracy. LCS standards were prepared and analyzed for each batch. Digested LCSs were prepared for acid-digested batches, and a post digestion LCS (same as the ICV recovery) was analyzed for each fusion-digested batch. Recoveries were within the required range of 80 to 120%.

Predigestion spikes were prepared for acid-digested samples at the required frequency of one per 20 samples or one per batch. If the spike recovery was not within the required range of 75 to 125%, a post digestion spike was analyzed. Predigestion spikes were not prepared for fusion-digested samples. Only post digestion spikes were analyzed and reported, which is the usual laboratory protocol. Samples which did not meet the required recovery are listed below:

S99Z000293 (acid digest):	Cr	54.2%				
post digestion recovery:	Cr	97.7%				
S99Z000301 (acid digest):	Cr	-8.76%	Ni	12.3%		
post digestion recoveries:	Cr	98.1%	Ni	98.5%		
Drainable liquid sample						
S00Z000002 (acid digest)	Cr	-1.31 E+01%	Ni	50.2%	S	69.8%
post digestion recoveries	Cr	99.0%	Ni	99.5%	S	99.7%

See section on result calculations for explanation of low spike recoveries.

Calibration. Initial calibration data were not required to be provided. For each batch, ICV and CCV samples were analyzed. Also, initial calibration blanks and continuing calibration blanks were analyzed. These were reviewed and all were determined to be within required limits.

RPD. Duplicate samples were prepared for both acid and fusion digested samples at the required frequency of one in 20 samples or one per batch. The calculated RPDs were generally less than 20% as required with the exception of those for aluminum and iron in sample S99Z000314, the KOH fusion for core composite sample 264-1. The RPDs were 24% for aluminum and 35.8% for iron. The narrative of the data report identified that, for aluminum, there was a low concentration of the analyte (less than two-times the detection limit) in the sample. For iron, there was a high concentration of the analyte. Data usability is not affected.

Blanks. All reported method blanks were lower than sample concentrations and lower than detection limits. However, contamination was noted in field and lithium bromide blanks. The potassium concentrations in all three blanks were significant and suggest a contamination problem during preparation of the acid leachate/digest samples.

Sample	Blank type	Associated Core	Contaminant (μg/g) chromium	potassium*	nickel	zinc	zirconium
S00Z000003	field	264	1.490	82.40	2.710	3.060	1.910
S00Z000005	lithium bromide	263		65.50			1.020
S00Z000007	lithium bromide	264		81.50	2.630		

*Suggests a contamination problem during the acid digest preparation of the samples for Worklist #32054

Results calculation. Discrepancies were noted after recalculation of sample, sample duplicate, and spike concentrations from the raw (run) data provided. These discrepancies should not affect the decisions made from the data. Discrepancies are listed below. For the following, the first column of data is for the reported value and the second column is for the value as recalculated by EQM:

For S99Z000301 in units of μg/g:

	Reported concentration		Recalculated concentration	
Nickel				
sample	3360	*from 1:10 dilution*	2913	*undiluted sample*
duplicate	3400	*from 1:10 dilution*	2772	*undiluted sample*
RPD	1.18%		4.96%	
Recovery	12.3%	*(Result for post digestion spike should be 98%.)*	58.0%	*predigestion spike of 1:10 dilution*

	Reported concentration		Recalculated concentration	
Chromium				
sample	5910	*from 1:10 dilution*	5278	*undiluted sample*
duplicate	6000	*from 1:10 dilution*	5080	*undiluted sample*
RPD	1.18%		4.96%	
Recovery	-8.76%	*(Result for post digestion spike should be 98%.)*	56.2%	*predigestion spike of 1:10 dilution*

For (drainable liquid) S00Z000002 in units of μg/mL:

	Reported concentration		Recalculated concentration	
Chromium				
sample	3.95E+3	*from 1:10 dilution*	3.69E+3	*undiluted sample*
duplicate	3.81E+3	*from 1:10 dilution*	3.70E+3	*undiluted sample*
RPD	3.61%		0.3%	
Recovery	-1.31E+1%	*(Result for post digestion spike should be 99%.)*	40.8%	*predigestion spike of 1:10 dilution*
Nickel				
sample	1.41E+3	*from 1:10 dilution*	1.29E+3	*undiluted sample*
duplicate	1.35E+3	*from 1:10 dilution*	1.30E+3	*undiluted sample*
RPD	4.35%		0.2%	
Recovery	50.2%	*(Result for post digestion spike should be 99%.)*	73.7%	*predigestion spike of 1:10 dilution*
Sulfur				
sample	7.31E+3	*from 1:10 dilution*	7.28E+3	*undiluted sample*
duplicate	729	*from 1:10 dilution*	686	*undiluted sample*
RPD	0.27%		5.97%	
Recovery	69.8%	*(Result for post digestion* spike should be 99.7%.)	70.3%	*predigestion spike of 1:10 dilution*

Reported concentrations (listed above under "reported") for the sample and duplicate were diluted concentrations (dilution factor of 10). An error in reporting software caused the spike recoveries to be reported incorrectly (such that the dilution factor was incorrectly applied to the spike concentration). After the initial examination of data, the laboratory agreed to correct the error by reporting undiluted sample and duplicate concentrations and predigestion spike recoveries as shown in the "Recalculated" column.

In a revision to the data report (FDH 2000b), the laboratory did recalculate the spike recoveries, but did not consistently report the sample and duplicate results that went with the predigestion spike. Instead the 1:10 dilution results were reported. In order to report results in a consistent manner, the laboratory should have reported results for the undiluted sample, the undiluted duplicate, and the predigestion spike.

3.7.7.3 Summary

Thirteen sludge composite samples were prepared by two methods and analyzed for metals. The required QC was performed. In general, QC requirements were met; however, discrepancies were noted after recalculation of sample, duplicate, and spike concentrations for four analytes. However, these discrepancies should not affect decisions made for waste designation or treatment.

3.7.8 Arsenic, Cadmium, and Lead by Graphite Furnace Atomic Absorption Spectroscopy

Analysis of sludge composite samples by graphite furnace atomic absorption (GFAA) spectroscopy was unnecessary because the ICP data for arsenic, cadmium, and lead were suitable for this project.

3.7.9 Ammonium/Ammonia as Ammonia by Ion-selective Electrode

Determination of ammonium/ammonia concentration in the sludge was performed for health and safety purposes. Results are presented in Table 3-51.

Table 3-51. Ammonium/Ammonia Concentration of Sludge Composite Samples from Tank 241-Z-361

Composite	Interval (in.)		Ammonia (µg/g)	
	Top	Bottom	Sample	Duplicate
263-1	0.00	20.00	<493	<487
263-2	20.00	34.25	<482	
263-3	34.25	38.00	<498	
263-4*	38.00	46.00	<486	
263-5	46.00	59.00	<496	
263-6	59.00	65.00	656	
263-7	65.00	74.00	<512	
263-8	74.00	84.00	<513	
264-1	0.00	16.50	<498	<504
264-2	16.50	33.50	<491	
264-3	46.00	62.00	<494	
264-4	62.00	69.00	<490	
264-5	69.00	84.00	649	

* Container was broken.

3.7.9.1 Analytical Requirements

Analysis was requested by EPA Method 350.3 which is implemented by laboratory method LA-631-001. Solid samples are dissolved in water or acid. The ammonium ion is converted to ammonia by making the solution alkaline. A double standard addition method is used to quantify the ammonia using an ion-selective electrode (ISE). The method provides an option for

tank waste samples for which the electrode is suspended immediately (approximately 1/8-in) above the sample. For each batch, the analytical method required an initial calibration blank, a preparation blank, and an LCS. In addition, a CCV sample is analyzed after each batch. According to the method description, the second analysis of the LCS fulfills this requirement.

The TSAP (EQM 1999a) allowed for either an ion chromatography method or ISE method to be performed. The TSAP required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. In addition, at least one MS sample was required for every 20 samples of similar matrix or preparation batch; therefore, at least one MS sample was required for sludge composite samples. According to the TSAP, the required acceptance criteria for QC data are 80 to 120% for LCS recovery, 75 to 125% for spike recovery, and less than 20 for the RPD of duplicate samples. The analytical requirements list a low PQL of 15 ppm and a high PQL of 75 ppm. The TSAP required analysis of both supernatant and a water leachate from each composite sludge sample; however, only composite sludge samples were analyzed (FDH 2000c, Attachment 4.)

Validation was not required for ammonia data.

3.7.9.2 QC Review

Ammonia was determined by ISE according to the "tank waste" modification of laboratory method LA-631-001 Rev. D/Mod 2 which is discussed below. The only method deviation was as follows: not all water leachates from the sludge composite samples were acidified (this was done for only three of the thirteen samples).

Worklist #32355 included a preparation blank, two LCS standards, and the water leachate of one sludge composite sample (i.e., 263-1) which was run as both the duplicate and spike samples. The water leachate was not acidified as required by the method. Recovery for the standards was 115% for both. The preparation blank gave an MDL of less than 493 μg/g. Both the sample and duplicate results were less than this MDL; therefore, the RPD could not be calculated. Spike recovery was 90.6%.

Worklist #32595 included a preparation blank, two standards, and the water leachate of two sludge composite samples (i.e., 263-6 and 263-7). These water leachates were acidified as required by the method. The sludge composite sample 263-6 was run as the duplicate and spike samples. Recovery for the standards was 116% and 82.8%. The preparation blank gave an MDL of less than 523 μg/g. The sample and duplicate results were 656 μg/g and 595 μg/g, which gave an RPD of 9.75. The spike recovery was 96.7%. The second sample was less than 512 μg/g.

Worklist #32713 included a preparation blank, two standards, and the water leachate of five sludge composite samples (i.e., all composites from Core 264). Sludge composite sample 264-1 was run as the duplicate and spike samples. These water leachates were not acidified as required by the method. (See discussion later in this section.) Recovery for the standards was 107% and 88.9%. The preparation blank gave a detection limit of less than 498 μ/g. The sample and

duplicate results were both less than the MDL; therefore, the RPD could not be calculated. The spike recovery was 98.8%. One sample in this batch gave a result above the MDL; the result for sludge composite sample 265-5 was 649 μg/g NH_3.

Worklist #32720 included a preparation blank, two standards, and the water leachate of five sludge composite samples (i.e., 263-2, 263-3, 263-4, 263-5, and 263-8). Only the water leachate for sludge composite sample 263-8 was acidified as required by the method. No duplicate or MS samples were run. Recovery for the standards was 105% and 97.7%, respectively. The blank gave an MDL of less than 482 μg/g. The results of all samples in the batch were less than the MDL.

Upon review of the laboratory method for determination of ammonia, two procedural options were noted. The first option requires immersion of the electrode into the sample solution, followed by recording of the measurement and subsequent standard additions and associated measurements. The second option for "determination of ammonia for tank waste samples" specifies that the electrode be held in the air approximately 1/8-in above the sample. It is not apparent from the data package or narrative which option was used for the samples from Tank 241-Z-361.

The laboratory project manager verified with the analyst that these project samples were analyzed using the tank waste method (i.e., suspending the electrode above the sample), and that the QC samples were measured in the same manner. Although this practice is inconsistent with the reference method (Method 4500-NH3 G in Standard Methods for the Examination of Water and Wastewater, Seventeenth Edition) cited in the laboratory procedure, the instrument manufacturer, Orion Research, provided a summary of the practice (personal communication, Orion Research to Chuck Miller, no date). The saturated vapor method is typically used for aqueous samples containing either a surfactant or a nonpolar constituent that would cause wetting of the hydrophobic electrode membrane. This method also has application to solutions with high concentrations of interfering ions. The primary limitation to the saturated vapor method is that it is usually applied to solutions with relatively high ammonia concentrations; that is, greater than 14 mg/L.

Regarding acidification of the leachates before the analysis, the narrative of the data report states:

> Typically, samples would be acidified to preserve the ammonia in the ammonium form to reduce loss of analyte. However, the Laboratory has historically observed a greater loss of ammonia in tank samples during the process of acidification, so the first set of water digest samples were not acidified and an attempt was made to perform the ammonia analysis within a week of the water leach. The pH of the water leach solutions, tested with short-range pH paper, was approximately 7.5 to 8.
>
> All samples except composites 263-6, 263-7 and 263-8 were analyzed from unacidified solutions. For these three composites, a second water leach was prepared and acidified to determine if acidification minimized the loss of ammonia from the solutions...There was no significant difference between the results of the two analyses."

3.7.9.3 Summary

The ammonia results are consistent with the low sensitivity of the saturated vapor method. Only two of 13 sludge composite samples have detectable ammonia. Although the QC was acceptable, the data do not meet the high PQL of 75 ppm.

3.7.10 Total Organic and Total Inorganic Carbon by Coulometry

Analyses of total organic carbon and total inorganic carbon were performed to support treatment options. The parameters requested (as a result of the DQO process) were total organic carbon and total carbon, which is calculated by summing the results of the organic and inorganic results for each sample. Results for organic, inorganic, and total carbon are presented in Table 3-52.

3.7.10.1 Analytical Requirements

Analysis was requested by Method 9060 which is implemented by laboratory method LA-342-100. A test portion (suggested size 0.0500 g) is acidified, heated, and sparged in a reaction vessel. The evolved carbon dioxide is purified, then absorbed into a weak organic base. The resulting carbonic acid is titrated with electrical current by the coulometer to measure total inorganic carbon. In order to determine total organic carbon, persulfate oxidizer is added to the reaction vessel, a silver catalyst added, and the reaction vessel heated. The evolved carbon dioxide is purified, absorbed into a weak organic base, and titrated as described above. Total carbon is calculated by addition of the two results. For each batch, the method required one reagent blank (which is also considered to be the initial calibration blank), one LCS standard (which is also considered to be the initial calibration verification), a spike if requested by the customer, and a duplicate if requested by the customer. According to the procedure, the instrument baseline has been established to be equal to or less than 15 μg for total inorganic carbon and 40 μg for total organic carbon.

Table 3-52. Organic, Inorganic, and Total Carbon Results for Sludge Composite Samples from Tank 241-Z-361 (2 sheets)

Composite	Interval (in.)		Organic C	Inorganic C	Total C (calc.)	Data Assessment Qualifier
	Top	Bottom	Concentration (μg/g)			
263-1	0.00	20.00	8410	3080	11490	
263-2	20.00	34.25	1800	2520	4320	
263-3	34.25	38.00	*730*	*8490*	*9220*	
263-4*	38.00	46.00	*979*	*24500*	*25479*	J^O
263-5	46.00	59.00	1590	17600	19190	J^I J^T
263-6	59.00	65.00	*1500*	*24100*	*25600*	
263-7	65.00	74.00	946	5440	6386	J^O J^I J^T
263-8	74.00	84.00	849	13200	14049	J^O J^I J^T
264-1	0.00	16.50	1070	1520	2590	

Table 3-52. Organic, Inorganic, and Total Carbon Results for Sludge Composite Samples from Tank 241-Z-361 (2 sheets)

Composite	Interval (in.)		Organic C	Inorganic C	Total C (calc.)	Data Assessment Qualifier
	Top	Bottom	Concentration (µg/g)			
264-2	16.50	33.50	3540	1440	4980	
264-3	46.00	62.00	2360	17100	19460	$J^I J^T$
264-4	62.00	69.00	662	19000	19662	$J^I J^T$
264-5	69.00	84.00	3200	16800	20000	

*Container was broken.
Superscripts on J (estimated) qualifier are O, I, and T which identify organic, inorganic, and total carbon, respectively.
Bold italicized values are in database but are not representative of analyte concentrations. See Table 3-53.

The TSAP (EQM 1999a) required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. In addition, at least one MS sample was required for every 20 samples of similar matrix or preparation batch; therefore, at least one MS sample was required for sludge composite samples. According to the TSAP, acceptance criteria were provided for total organic carbon only; i.e., QC data are 82 to 106% for LCS recovery, 75 to 125% for spike recovery, and less than 20 for the RPD of duplicate samples. The analytical requirements for both total organic carbon and total carbon list a low PQL of 40 ppm and a high PQL of 2000 ppm for each. Note that no data acceptance criteria were provided for total inorganic carbon analysis.

Validation was not required for total organic carbon or total inorganic carbon data.

3.7.10.2 QC Review

Total organic carbon and total inorganic carbon were determined by coulometry according to the laboratory method LA 342-100 Rev. F/Mod 5. Analysis was performed directly on the sludge composite samples.

Samples were analyzed in five Worklists (#32035, 33082, 33090, 33131, 33139).

Worklist #32035 included, for each analyte, a reagent blank, an LCS standard, a sludge composite sample (263-6) run in triplicate, and two samples (263-7 and 263-8).

For total inorganic carbon, the results were as follows: The reagent blank result was 4.6 µg/g. Recovery for the LCS was 94.1%. Calculation of RPD used the original result (i.e., 13,200 µg/g) for both the duplicate and triplicate samples; RPDs were 10.8 and 18.6, respectively. Spike recovery was 166%. Results reported for the samples were 5440 µg/g for sludge composite sample 263-7 and 13,200 µg/g for sludge composite sample 263-8.

For total organic carbon, the results were as follows: The reagent blank result was 1.8 μg/g. Recovery for the LCS was 95.7%. Calculation of RPD used the original result (i.e., 505 μg/g) for both the duplicate and triplicate samples; RPDs were 62.5 and 1.80, respectively. Spike recovery was 130%. Results reported for the samples were 94,600 μg/g for sludge composite sample 263-7 and 84,900 μg/g for sludge composite sample 263-8.

For this worklist, spike recovery for both total inorganic carbon and total organic carbon were high. In addition, RPD for total organic carbon for one of the replicate pairs was high. Although not noted on the laboratory worklist, sludge composite sample 263-6 was rerun in Worklist #33139. However, data for sludge composite samples 263-7 and 263-8 were reported. Therefore, these data for both total inorganic carbon and total organic carbon would be qualified with a "J" as estimated due to high spike recovery.

Worklist #33082 included, for each analyte, a reagent blank, an LCS standard, a sludge composite sample (263-3) run in triplicate, and three samples (263-5, 264-3, 264-4).

For total inorganic carbon, the results were as follows: The reagent blank result was 6.0 μg/g. Recovery for the LCS was 95.7%. Calculation of RPD used the original result (i.e., 8780 μg/g) for both the duplicate and triplicate samples; RPDs were 8.30 and 2.92, respectively. Spike recovery was 40.7%. Results reported for the samples were 17,600 μg/g for sludge composite sample 263-5, 17,100 μg/g for sludge composite sample 264-3, and 19,000 μg/g for sludge composite sample 264-4.

For total organic carbon, the results were as follows: The reagent blank result was 12.9 μg/g. Recovery for the LCS standard was 92.7%. Calculation of RPD used the original result (i.e., 1400 μg/g) for both the duplicate and triplicate samples; RPDs were 37.7 and 28.7, respectively. Spike recovery was 93.5%. Results reported for the samples were 1590 μg/g for sludge composite sample 263-5, 2360 μg/g for sludge composite sample 264-3, and 662 μg/g for sludge composite 264-4.

The chemist noted that the results for sludge composite sample 263-3 were rejected because of low spike recovery for total inorganic carbon and because RPDs for replicate analyses for total organic carbon were above the 20% data objective. Sludge composite sample 263-3 was rerun in Worklist #33139; however, the remaining samples were not rerun and the data were reported in the database. Therefore, the reported data for total inorganic carbon would be qualified with a "J" as estimated due to low spike recovery.

Worklist #33090 included, for each analyte, a reagent blank, an LCS standard, a sludge composite sample (264-1) run in duplicate, and a spike sample. There were no additional samples in this worklist.

For total inorganic carbon, the results were as follows: The reagent blank result was 2.1 μg/g. Recovery for the LCS standard was 98.5%. RPD for duplicate analysis was 0.0, because the result for both analyses of sludge composite sample 264-1 was 1520 μg/g. Spike recovery was 99.1%.

For total organic carbon, the results were as follows: The reagent blank result was 7.0 μg/g. Recovery for the LCS standard was 89.4%. RPD for duplicate analysis was 2.76, based on an initial result of 1070 μg/g. Spike recovery was 96.0%.

This worklist met the data acceptance criteria for organic carbon; therefore, the data for sludge composite sample 264-1 are acceptable (no qualifiers).

Worklist #33131 included, for each analyte, a reagent blank, an LCS standard, a sludge composite sample (263-4) run in triplicate, and a spike sample. There were no additional samples in this worklist.

For total inorganic carbon, the results were as follows: The reagent blank result was 9.50 μg/g. Recovery for the LCS standard was 102%. Calculation of RPD used the original result (i.e., 24500 μg/g) for both the duplicate and triplicate samples; RPDs were 7.63 and 5.02, respectively. Spike recovery was 114%.

For total organic carbon, the results were as follows: The reagent blank result was 7.80 μg/g. Recovery for the LCS standard was 95.0%. Calculation of RPD used the original result (i.e., 979 μg/g) for both the duplicate and triplicate samples; RPDs were 57.0 and 97.2, respectively. Spike recovery was 97.0%.

For this worklist, RPD for total organic carbon was high. The sludge composite sample was not rerun and the initial result obtained in this batch was reported in the database. Therefore, the result would be qualified with a "J" as estimated due to high RPD.

Worklist #33139 included, for each analyte, a reagent blank, an LCS standard, a sludge composite sample (263-1) run in duplicate, a spike sample, and five samples (263-2, 263-3, 263-6, 264-2, 264-5).

For total inorganic carbon, the results were as follows: The reagent blank result was 3.80 μg/g. Recovery for the LCS standard was 102%. RPD for duplicate analysis was 0.647, based on an initial result of 3080 μg/g. Spike recovery was 100%. Results for the sludge composite samples were 2520 μg/g, 8490 μg/g, 24,100 μg/g, 1440 μg/g, and 16,800 μg/g, respectively.

For total organic carbon, the results were as follows: The reagent blank result was 14.8 μg/g. Recovery for the LCS standard was 93.0%. RPD for duplicate analysis was 11.4, based on an initial result of 8410 μg/g. Spike recovery was 113%. Results for the samples were 1800 μg/g, 730 μg/g, 1500 μg/g, 3540 μg/g, 3200 μg/g, respectively.

This worklist met the acceptance criteria for both total inorganic carbon and total organic carbon; therefore, the data are acceptable (no qualifiers) by the data validation "rules." However, it appears that these data for the reruns of samples 263-3 and 263-6 are not representative of the sample material.

Note that this worklist contains the reruns of sludge composite sample 263-3 from Worklist #33082 and sludge composite sample 263-6 from Worklist #32035. For sample 263-3, the new results were lower than the original results for both total inorganic carbon and total organic carbon. That is, the rerun results for total inorganic carbon ranged from 3.30 to 11.0% lower than the original triplicate analyses, and the rerun results for total organic carbon ranged from 23.6 to 61.0% lower than the original triplicate analyses. Conversely, for sample 263-6, the new results were higher than the original results for both analytes. That is, the rerun results for total inorganic carbon ranged from 55.8 to 82.6% higher than the original triplicate analyses and the rerun results for total organic carbon ranged from 55.6 to 202% higher than the original triplicate analyses. However, the rerun data for both sludge composite samples 263-3 and 263-6 were reported in the database.

The narrative of the data report (FDH 2000a) states that the results indicate that the total organic carbon was not evenly distributed in the tank. An alternative was offered as well:

> Another possible explanation might have been variability in the dryness of the sample throughout individual sample vials. However, for samples 263-3 and 263-4, the dryness of the sample should have affected both the TIC and the TOC. The small subsamples removed from the composite for analysis dried over time as the vial was opened and the material was smeared around on the sides while an attempt was made to get a representative aliquot. The very small sample size that was required for this particular analysis (<0.1 g) could cause elevated relative percent differences if one analysis aliquot was much drier than the second. This was the most probable cause for the elevated results for the rerun analysis of sample 263-6...the results for both TIC and TOC in the rerun were significantly higher than in the original analysis that was performed months earlier.

The original analysis of sludge composite sample 263-6 was performed on February 7, 2000, and the rerun was performed on April 4, 2000. Although the narrative offers a reasonable explanation for the higher results for the rerun, the suggestion that the sample no longer contained the same moisture content as when it was received is a good reason <u>not</u> to report the rerun results in the database.

Table 3-53 contains all replicate data for the total inorganic carbon samples that were obtained for the sludge composite samples from Tank 241-Z-361.

Table 3-53. Statistical Data (Standard Deviation, Average, and Relative Standard Deviation) for Replicate Carbon Analysis for Sludge Composite Samples from Tank 241-Z-361

Composite	Replicate 1	Replicate 2	Replicate 3	Replicate 4	Std Dev	Average	RSD (%)
Total Inorganic Carbon (µg/g)							
263-1	3080	3100	N/A	N/A	14.14214	3090	0.46
263-3	8780	9540	9040	8490	445.7484	8962.5	4.97
263-4*	24500	22700	23300	N/A	916.5151	23500	3.90
263-6	13200	14700	15900	***24100***	4876.73	16975	28.73
263-6**	13200	14700	15900	deleted	1352.775	14600	9.26
264-1	1520	1520	N/A	N/A	0	1520	0
Total Organic Carbon (µg/g)							
263-1	8410	7500	N/A	N/A	643.4672	7955	8.09
263-3	1400	956	1870	730	504.398	1239	40.71
263-4*	979	1760	1370	N/A	390.5001	1369.667	28.51
263-6**	505	964	496	***1500***	475.6682	866.25	54.91
263-6**	505	964	496	deleted	267.6397	655	40.86
264-1	1070	1100	N/A	N/A	21.2132	1085	1.96
Total Carbon calculated (µg/g)							
263-1	11490	10600	N/A	N/A	629.325	11045	6.70
263-3	10180	10496	10910	9220	719.3764	10201.5	7.05
263-4*	25479	24460	24670	N/A	538.0431	24869.67	2.16
263-6	13705	15664	16396	***25600***	5295.782	17841.25	29.68
263-6**	13705	15664	16396	deleted	1391.341	15255	9.12
264-1	2590	2620	N/A	N/A	21.2132	2605	0.81

Bold italicized values are in database but are not representative of analyte concentrations. Sample integrity is questionable for Composite 263-6, Replicate 4. (See quotation from data report narrative in this report.)
*Container was broken.
**Recalculated without compromised results from reanalysis.

The EPA Method 9060 upon which laboratory method LA-342-100 is based specifies that quadruplicate analysis is required and both the average and the range are to be reported. The method also requires homogenization of samples in a blender.

LA-342-100 states (Step 5.2.4):

> Duplicates will be run per customer's request. It is recommended, however, that a duplicate be run on all the samples to establish the homogeneity of a project. The relative percent difference (RPD) should be $\leq \pm$ 20%, depending upon the homogeneity. If the duplicate analysis fails, a triplicate may be run in order to avoid a sample/duplicate rerun. Consult the scientist in charge with any questions.

Appendix B of the laboratory method states that "Samples are not run in quadruplicate and neither the range or average is reported." The reason for change is noted as: "Due to analysis time and radiological concerns the samples are run at customer request and the analysis results are reported directly."

Regarding homogenization in a blender, the laboratory method states "Samples are not homogenized in a blender." The reason for the change is noted as "Due to radiological concerns it would not be feasible to blend samples." A laboratory supervisor described that:

> The Z-361 composites were made in the 11A hot cell. The homogenization [of the sludge composite samples] was done using a butter-type spatula (approximately ½ in. in diameter). Each sample used for the composite was stirred with a spatula for 5 – 10 minutes before taking a sample for the composite. The composite was also stirred for 5-10 minutes before subsampling for any analysis. This method of homogenization was used for the Z-361 project due to the PCB nature of the samples. All washings in the hot cell go directly to the 219-S tank system which is not TSCA regulated. Using the spatula method created only solid waste. Homogenizers would have to be washed and the waste could not be contained in the 11A hot cell without reaching the tank system (e-mail communication, John Prilucik to Joan Bartz, 2 May 2001).

The procedure used for homogenization of the samples was approved for the project, considering the radioactive nature of the samples, the need to conduct work in a hot cell, and the necessity of limiting waste generation to types for which the laboratory has a legal management pathway. The reporting of results, however, does not account for the variability in results that the EPA method seems to anticipate. Also, using small test portions (approximately 0.05 to 0.06 g) which may not be representative of the sludge composite sample is another shortcoming of the procedure. The data for these sludge composite samples illustrate that the inorganic and organic carbon concentrations would be better described if replicate analyses were performed and either all data were reported or the EPA requirement of reporting the average of replicate analyses and range was followed.

3.7.10.3 Summary

The required analytical method was performed on 13 sludge composite samples. QC samples were run at the required frequencies; however, results indicate that one or both of the following exist: (1) test portions are not representative of the sludge composite sample; and/or (2) composite sample was not homogenized. The DQO which were established for total organic carbon and total carbon were too restrictive, given the limitations of the analytical method with respect to test portion size and precision. The analysts used these criteria in deciding whether or not to reject the data and reanalyze the samples. Rather than using the single concentration value report by the laboratory, the use of all available replicate data is recommended (with the possible exception that the rerun data for sample 263-6 should be removed from the data set because the integrity of the sample is believed to have been compromised). This would be consistent with the EPA method requirement to analyze each sample in quadruplicate and report the mean and range.

3.7.11 Cyanide

Analysis of cyanide was conducted to satisfy a regulatory requirement. Results are presented in Table 3-54.

Table 3-54. Cyanide Concentration of Sludge Composite Samples from Tank 241-Z-361

Composite	Interval (in.)		Cyanide (µg/g)	
	Top	Bottom	Sample	Duplicate
263-1	0.00	20.00	< 0.738	<0.739
263-2	20.00	34.25	< 0.9	
263-3	34.25	38.00	< 0.787	
263-4*	38.00	46.00	< 0.883	
263-5	46.00	59.00	< 0.799	
263-6	59.00	65.00	1.41	1.02
263-7	65.00	74.00	1.01	
263-8	74.00	84.00	1.34	
264-1	0.00	16.50	< 0.695	<0.676
264-2	16.50	33.50	< 0.875	
264-3	46.00	62.00	< 0.766	
264-4	62.00	69.00	< 0.796	
264-5	69.00	84.00	< 0.65	

*Container was broken.

3.7.11.1 Analytical Requirements

Analysis was requested by Method 9012A which is implemented by laboratory method LA-695-103. For solid matrices, the method includes dissolution of insoluble cyanide compounds and treatment to remove interferences. Analyses are conducted by distillation of hydrogen cyanide from the solid samples, followed by spectrophotometric determination. For each batch, the analytical method required a continuing calibration blank and a CCV sample (i.e., LCS).

The TSAP (EQM 1999a) required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. In addition, at least one MS sample was required for every 20 samples of similar matrix or preparation batch; therefore, at least one MS sample was required for sludge composite samples. According to the TSAP, the required acceptance criteria for QC data are 80 to 120% for LCS recovery, 75 to 125% for spike recovery, and less than 20 for the RPD of duplicate samples. The analytical requirements list an Action Limit Total of 250 ppm and a low PQL of 8,000 ppm. The TSAP required analysis of both supernatant and sludge composite samples; however, only composite sludge samples were analyzed (FDH 2000c, Attachment 4.)

Validation was not required for cyanide data.

3.7.11.2 QC Review

The analysis was performed directly on the sludge composite samples. No deviations from the method were documented.

Worklist #32009 included a continuing calibration blank and a CCV sample. Sludge composite sample 264-1 was run as the duplicate and MS samples. The MS recovery was 101%; however, because the analytical result for the duplicate sample was less than the MDL, no RPD could be calculated. All results were less than the MDLs, which ranged from 0.650 to 0.875 μg/g.

Worklist #32038 included a calibration blank and a CCV sample. Sludge composite sample 263-1 was run as the duplicate and MS samples. The MS recovery was 108%; however, because the analytical result for the duplicate sample was less than the MDL, no RPD could be calculated. All results were less than the MDLs, which ranged from 0.738 to 0.900 μg/g.

Worklist #32039 included a continuing calibration blank and a CCV sample. Sludge composite sample 263-6 was run as the duplicate and MS samples. The MS recovery was 101%. The RPD of the duplicate samples was 32.1. Although this exceeds the criterion of less than 20%, the high RPD is expected because of the low analyte concentration in the sample material and does not result in a data qualifier. The other two samples (i.e., 263-7 and 263-8) also had detectable cyanide concentrations at less than two times the MDL. The data are acceptable.

3.7.11.3 Summary

The required analytical method was performed on 13 sludge composite samples. QC samples were run at the required frequencies. Results for MS samples meet the acceptance criteria. RPD for two of the three duplicate samples could not be calculated. RPD for the third duplicate sample was greater than 20%, but did not result in a data qualifier. For all samples, the low PQL objective of 8,000 ppm was met. Only three samples contained detectable cyanide, which was well below the Action Limit of 250 ppm. The data are acceptable for the intended use.

3.7.12 Anions by Ion Chromatography

Analysis of fluoride was conducted to satisfy a regulatory requirement, analysis of sulfate was requested to support the evaluation of reactive sulfide to satisfy a regulatory requirement, and analysis of the remaining anions was performed in support of evaluating treatment options for the waste. Results for all anions, except phosphate, are presented in Table 3-55. All phosphate results were less than the detection limit. (See Appendix C for detection limits.)

Table 3-55. Anion Concentration for Sludge Composite Samples from Tank 241-Z-361*

Composite	Interval (in.)		Anion Concentration (µg/g)												
			Fluoride			Chloride		Nitrite		Bromide		Nitrate		Sulfate	
	Top	Bottom	Sample	Q	Dup	Sample	Dup	Sample	Dup	Sample	Dup	Sample	Dup	Sample	Dup
263-1	0.00	20.00	4430		3840	647	552	519	443	1040	912	1000	868	1780	1610
263-2	20.00	34.25	10800			510		737		2710		435		1320	
263-3	34.25	38.00	7310			532		909		638		314		1310	
263-4**	38.00	46.00	2370			606		1020		1050		209		1240	
263-5	46.00	59.00	2980			482		863		2630		<166		1100	
263-6	59.00	65.00	3840	J	3560	907	946	1370	1450	1660	1740	346	348	1480	1770
263-7	65.00	74.00	5770			858		1540		1930		<166		1980	
263-8	74.00	84.00	5760			835		1530		1770		<167		1950	
264-1	0.00	16.50	1070		1300	718	732	362	322	280	287	1230	1290	1560	1630
264-2	16.50	33.50	3820			572		867		1150		<164		1130	
264-3	46.00	62.00	8300			565		527		5370		603		1400	
264-4	62.00	69.00	2850			612		986		3110		<164		974	
264-5	69.00	84.00	3820			562		971		1370		<170		1040	

*All phosphate concentrations were less than detection limit (<147 g/g).
**Container was broken.
dup = duplicate
J = estimated value
Q = data assessment qualifier

3.7.12.1 Analytical Requirements

Analysis was requested by Method 9056 which is implemented by laboratory method LA-533-105. A test portion is leached with water and particulates greater than 0.2 microns are filtered from the leachate. The leachate is then injected into an ion chromatograph. A separator column separates the test portion into its component ions. A suppressor column removes the background counter-ions and converts the anions into their acidic forms. The anions, in a low conducting eluent stream, are identified by a conductivity detector by the order of elution.

For each batch, the analytical method required calibration blank, duplicate, a laboratory control standard, and CCV samples. A preparation blank was required for the water extract samples. An MS, if analyzed, is spiked into the prepared leachate.

The TSAP required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the water extracts of the sludge composite samples. In addition, at least one MS sample was required for every 20 samples of similar matrix or preparation batch; therefore, at least one MS sample was required for the water extracts of the sludge composite samples. According to the TSAP, the required acceptance criteria for QC data are 80 to 120% for LCS recovery, 75 to 125% for spike recovery, and less than 20 for the RPD of duplicate samples.

The TSAP (EQM 1999a) and compositing plan (EQM 1999c) required analysis of the drainable liquid (i.e., supernatant) from the core segments. However, only segment two of Core 264 contained drainable liquid. As described in the data report, "Following the acid digestion for the metals analysis, the appearance of the remaining sample was described as sludge slurry, which was not appropriate to be analyzed by IC [ion chromatography]. An agreement to not perform the IC analysis on the drainable liquid was made via electronic mail." (FDH 2000b, Attachment 4.)

Validation was not required for anion data.

3.7.12.2 QC Review

The following anions, listed in order of elution, were determined by laboratory method LA-533-105 Rev. F/Mod 2: fluoride (F^-), chloride (Cl^-), nitrite (NO_2^-), bromide (Br^-), nitrate (NO_3^{-2}), phosphate (PO_4^{-3}), and sulfate (SO_4^{-2}). Analyses were conducted by direct analysis of the lithium bromide solutions associated with the sampling of each core, by direct analysis of the field blank associated with Core 264, and by analysis of a water extract of each sludge composite sample. Oxalate was measured simultaneously; however, because it was not a required analyte for this project, the data were not reported.

The lithium bromide solutions and field blank were analyzed in three Worklists (#32146, 32788, 32899); the sludge composite samples were analyzed in four Worklists (#32147, 32149, 32593, 32899).

Worklist #32146 included, for each anion, a calibration blank, an LCS, and a CCV sample. No duplicate or MS samples were included. The analyst rejected the phosphate analysis due to an LCS of 75.4%, which is below the required minimum of 80%. The analyst also rejected the bromide analysis, but provided no explanation. However, the bromide results of 55,100 μg/mL (Core 263) and 58,200 μg/mL (Core 264) were nearly twice what would be expected for the 0.3 M lithium bromide solution used in the core sampling, i.e., 24,000 μg/mL. Therefore, a dilution error is suspected. The anion results for the lithium bromide samples were less than MDL for all anions other than bromide. Although the MDLs were high for the lithium bromide samples, the MDLs meet the DQO for the low PQLs established in the TSAP (EQM 1999a). Data for fluoride, chloride, nitrite, nitrate, and sulfate are acceptable.

Worklist #32147 included, for each anion, a calibration blank, an LCS, and a CCV sample. A water extraction preparation blank also was included. Sludge composite sample 263-1 was run as the duplicate and postpreparation MS samples. Although the preparation blank contained 0.027 μg/g chloride, this contamination was negligible in comparison to the chloride concentration in the samples and does not result in any qualifiers. All other QC data meet the acceptance criteria. All data are acceptable.

Worklist #32149 included, for each anion, a calibration blank, an LCS, and a CCV sample. A water extraction preparation blank also was included. Sludge composite sample 264-1 was run as the duplicate and postpreparation MS samples. Although the preparation blank contained 0.630 μg/g chloride and 0.291 μg/g nitrite, this contamination was negligible in comparison to the chloride and nitrite concentrations in the samples and does not result in any qualifiers. All other QC data meet the acceptance criteria. All data are acceptable.

Worklist #32593 included, for each anion, a calibration blank, an LCS, and a CCV sample. A water extraction preparation blank also was included. Sludge composite sample 263-6 was identified as the duplicate sample; however, no routine sample 263-6 appears in the data package for this worklist. Although a sample labeled as an MS sample (in the data package) was analyzed, the data seem to indicate that the spike may not have been performed properly. Note, however, that the TSAP did not require duplicate and MS samples to be included in each batch, but only to be run at a 5% rate or one in 20 samples. Therefore, the data for the two sludge composite samples in this worklist, i.e., 263-7 and 263-8, are not limited by qualifiers. Although the calibration blank contained 0.157 μg/mL sulfate and the preparation blank contained 0.029 μg/g chloride, 0.299 μg/g nitrite, and 0.238 μg/g nitrate, the contamination was negligible in comparison to the chloride and nitrite concentrations in the samples. Note that nitrate, although found in the preparation blank, was not detected in the samples. The QC data meet the acceptance criteria. All data are acceptable.

Worklist #32788 included, for each anion, a calibration blank, an LCS, and a CCV sample. No duplicate or MS samples were included. Only phosphate was determined on the lithium bromide and field blank samples, because this worklist was a reanalysis as a result of the analyst's rejection of phosphate for Worklist #32146 (see above). The analytical results for phosphate were less than the MDLs of 1224 μg/mL for the lithium bromide samples and 0.120 μg/mL for the field blank. These results meet the DQO for phosphate in that they are less than the low PQL of 4000 ppm established in the TSAP. All data are acceptable.

Worklist #32899 included, for each anion, a calibration blank, an LCS, and a CCV sample. A water extract preparation blank also was included. Sludge composite sample 263-6 was run as the duplicate and MS samples. The preparation blank contained 0.018 μg/g chloride and 0.230 μg/g nitrite, which is negligible in comparison to the chloride and nitrite concentrations in the single sludge composite sample in this batch. All other QC data are acceptable with the exception of the spike recovery of 69.13% for fluoride. The laboratory attributed this low recovery to the high concentration of fluoride (i.e., 3840 μg/g) in the sample, "which made it difficult to add sufficient spike standard to have meaningful results." Essentially, the spike was swamped by the concentration of fluoride in the sample. Therefore, the fluoride result for sludge composite 263-6 is qualified by the data assessment as an estimated value. The bromide results of 24,100 μg/mL (Core 263) and 26,200 μg/mL (Core 264) for the lithium bromide samples are in agreement with the expected value of 24,000 μg/mL for 0.3 lithium bromide solution. These bromide results are acceptable.

3.7.12.3 Summary

The required analytical method was performed on 13 sludge composite samples, two lithium bromide solutions, and one field blank. QC samples were run at the required frequencies. RPD for duplicate samples meet the acceptance criteria. The only acceptance criterion that was not met for MS samples was a low recovery for fluoride for sludge composite sample 263-6. Therefore, this result is qualified as "estimated" by the data assessment. All samples meet the low PQL objective. Analytes present in preparation blanks are common water contaminants and were present in negligible amounts. Data are acceptable for the intended use.

3.7.13 p-Phthalic Acid as Phthalate by Ion Chromatography

Analysis for p-phthalic acid was required by a regulatory driver to consider UHCs when designating hazardous waste. All data are less than detection limit; however, the detection limit was too high to meet project requirements. The lack of definitive data for phthalate will not change the waste designation or the management for the sludge.

3.7.13.1 Analytical Requirements

Analysis was requested by Method 9056 which is implemented by laboratory method LA-533-115. This method is similar to LA-533-105, but is modified to use different instrumentation. (In addition, the laboratory was required to develop it for the analysis of phthalate.)

For each batch, the laboratory method required a calibration blank, an LCS, a CCV sample, and a preparation blank.

The TSAP (EQM 1999a) required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. In addition, MS samples were required at the same frequency. According to the TSAP, the required acceptance criteria for QC data are 80 to 120% for LCS recovery, 75 to 125% for spike recovery, and less than 20 for the RPD of duplicate samples. The DQOs noted the Action Limit Total for p-phthalic acid as 28 ppm; however, both the low and high PQLs were noted as "under development." The compositing plan (EQM 1999c) and TSAP required analysis of p-phthalic acid in both the sludge composite samples and the supernatant.

Validation was not required for phthalate data.

3.7.13.2 QC Review

p-Phthalic acid was determined by laboratory method LA-533-115 Rev. C/Mod 1. Analysis was conducted on a water extract of each sludge composite sample. As discussed previously, the supernatant, or drainable liquid, from the core sampling was not appropriate for analysis by ion chromatography; therefore, the analytical request for supernatant analysis was cancelled.

Samples were analyzed in three Worklists (#32240, 32242, 32603).

As analyzed by the laboratory, each worklist included the required calibration blank, LCS, CCV sample, and preparation blank, as well as duplicate and spike samples. Spike recoveries were 99.1, 99.1, and 88.2%, respectively, which meet the acceptance criteria. RPD for duplicate samples could not be calculated, because duplicate sample results were below MDL. All other QC results meet the acceptance criteria.

Phthalate results for all sludge composite samples were less than MDL. These MDLs ranged from 1100 to 4460 μg/g, which were approximately 40 to 160 times higher than the Action Limit of 28 ppm identified as the objective. The data report (FDH-2000c) notes that this analysis was a new method developed for this project; however, the method development was performed before the laboratory was informed of the Action Limit of 28 ppm. (The narrative of the data report states: "This analysis was a new method developed for this project. The development work was performed before obtaining the information concerning the required action limit of 28 μg/g listed in the TSAP." In order to produce a method for p-phthalic acid, developmental work began many months before the TSAP was completed. The Action Limit was not finalized until the TSAP was published, and there was not sufficient time to redevelop, review, and publish a method that would produce detection levels below the Action Limit.)

The laboratory had used a 0.5 g to 100 mL water extraction dilution so that the resulting detection limit was approximately 110 μg/g. The narrative of the data report states that "One analysis of the samples with no dilution gave indication of no detectable phthalate at this minimum detection limit." However, because of matrix interferences that affected spike recoveries, the samples were diluted during analysis, resulting in MDLs that were 10 to 40 times higher than the minimum detection limit (personal communication, Ruth Esch, Project Coordinator, to Chuck Miller, EQM, no date). After release of the Rev. 0 data package (FDH 2000c), the ion chromatograph was recalibrated, resulting in the possibility of an MDL of 55 μg/g if the samples could be analyzed without dilution. The possibility of using a two-times larger test portion for the water extraction was considered, because that would have resulted in a detection limit of 28 μg/g, but only if the extract could have been analyzed without dilution. Because it was known that dilution would have been needed due to matrix interferences, the laboratory could not attain the detection limit needed to meet the Action Limit objective. Therefore, no reanalysis was conducted.

3.7.13.3 Summary

Although the p-phthalic acid data do not meet the data quality objective of 28 ppm for the Action Limit, the data do provide an upper limit for phthalate in the sludge. However, as discussed previously, the lack of definitive data will not alter how the waste is managed.

3.7.14 Total Dissolved Solids

Analysis of total dissolved solids was conducted to support treatment options. The data were requested to estimate the water-soluble fraction of the sludge and for use in examining the internal consistency of the quantitative analysis of the water extracts. Analysis was requested for both sludge composite samples and supernatant samples.

3.7.14.1 Analytical Requirements

Analysis was requested by Method 160.1 which is implemented by LA-510-115. The parameter is determined by delivering a known volume of filtrate (or in this case, leachate) from a solid sample into a dry, tared vessel, drying the test portion at 180°C to a constant weight, cooling over a desiccant, then weighing the residue and vessel.

The TSAP (EQM 1999a) required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. MSs were not required for this parameter. According to the TSAP, acceptance criteria for QC data are 80 to 120% for LCS recovery and less than 20 for the RPD of duplicate samples. The analytical requirements list both the low and high PQL as 10 ppm.

Validation was not required for total dissolved solids data.

3.7.14.2 QC Review

Total dissolved solids was determined by the required method LA-510-115 Rev. 3/Mod 4. The analysis was performed on a water leachate of each sludge composite sample. The supernatant samples were deleted from the analytical request (FDH 2000c, Attachment 4).

Samples were analyzed in three Worklists (#32143, 32144, 31145). Each worklist included the LCS standard and a duplicate sample. The total dissolved solids standard, purchased from a commercial supplier has a certified value of 3000 mg/L; the recovery for the standard was 98.3%, 98.7%, and 98.3%, respectively. RPD could not be calculated for duplicates for any of the worklists because results were less than the detection limit of 0.00028 g/mL. Contrary to the narrative of the data report (which states "The reported detection limit met the requested low practical quantitation limit (PQL) listed in the TSAP."), the results did not meet the PQL objective of 10 ppm specified in the TSAP.

Samples were run twice. According to the narrative of the data report (FDH 2000c), "The analysis was reanalyzed with a larger sample size and there still were insufficient solids to report. Only the results of the second analysis were included in the raw data." The data package shows that, for the second analysis, 9 mL of leachate was used. The procedure suggests that "If unfamiliar with sample, use recommended sample size of about 10 mL." The test portion is introduced to a 35-mL glass vial containing a small wad (approximately 0.5 g) of glass wool. Apparently the initial analysis did not follow this recommendation, nor did the reanalysis. Presumably there would have been enough space in the vial for a much larger test portion.

3.7.14.3 Summary

The required analytical method was performed on the water leachates from 13 sludge composite samples. QC samples were run at the required frequencies. Because the results did not meet the PQL of 10 ppm and provide only an upper bound for soluble material in the sludge, a request was made for the laboratory to perform specific conductance measurement of the leachate to obtain an estimate of the total dissolved solid content of the samples.

3.7.15 Specific Conductance

Determination of specific conductance was requested by the project because the total dissolved solids data failed to meet project goals.

3.7.15.1 Analytical Requirements

Determination of specific conductance was requested after the analysis for total dissolved solids failed to provide adequate data for the project. (See above in Section 3.7.14.3) An e-mail (FDH 2000c, Attachment 3) stated that "The objective of determining the TDS...is to evaluate the measured results against the apparent total dissolved constituents. TDS...does not constitute a critical measurement and an estimate of the TDS based on specific conductance of the extract is sufficient to make the comparison." The laboratory was asked to report the results in units of millimhos/centimeter or millisiemens/centimeter.

Determination of specific conductance was requested for water leachate from all sludge composite samples; however, laboratory reruns for ammonia consumed substantial leachate volume. Therefore, sufficient leachate was available from only three sludge composite samples: 263-6, 263-7, and 263-8.

3.7.15.2 QC Review

The determination was performed in one batch according to laboratory method LA-512-107 Rev. A/Mod 1. Worklist #32604 included a continuing calibration blank, an initial calibration verification, and a duplicate sample. QC results were acceptable. Results for the samples analyzed were 130, 98.8 and 139 μmhos/cm, respectively.

For low ionic strength solutions such as these, the specific conductance or "electrical conductivity of extract" (ECE) can be used to estimate the total dissolved solids by the following relationship (from Bohn, McNeal, and O'Conner, 1979):

TSD (*in* mg/L) ~ ECE (*in* mmho/cm) * 640

Based on this relationship, the estimated total dissolved solids in the leachates from these sludge composite samples range from 63 to 89 mg/L.

Anions measured for sludge composite samples 263-6, 263-7, and 263-8 were compared to the ECE to evaluate the completeness of the anion evaluation according to the following relationship:

Sum of Anions (meq/L) ~ ECE (mmho/cm) * 10

The results of this evaluation are shown in Table 3-56.

Table 3-56. Comparison of Anion Concentration in Water Extracts to ECE for Three Sludge Composite Samples from Tank 241-Z-361

Anions	Concentration					
	Composite 263-6		Composite 263-7		Composite 263-8	
	(mg/L)	(meq/L)	(mg/L)	(meq/L)	(mg/L)	(meq/L)
Fluoride	19.2	1.01	28.8	1.52	29	1.52
Bromide	8.3	0.10	9.6	0.12	8.9	0.11
Nitrite	6.9	0.15	7.7	0.17	7.6	0.16
Chloride	4.5	0.11	4.3	0.13	4.2	0.12
Nitrate	1.7	0.03	<0.8	N/A	<0.8	N/A
Sulfate	7.4	0.15	9.9	0.21	9.7	0.20
Phosphate	<0.7	N/A	<0.7	N/A	<0.7	N/A
Phthalate	<5.7	N/A	<5.6	N/A	<5.6	N/A
Total Anions	**1.50 meq/L**		**2.15 meq/L**		**2.11 meq/L**	
ECE (mmho/cm) * 10	**1.30 meq/L**		**0.99 meq/L**		**1.39 meq/L**	

The sum of the quantified anions exceeded the estimated sum of the anions for all three sludge composite samples. Because the use of ECE to estimate ion concentrations is only semi-quantitative, the value of this comparison is for the relative values. This comparison indicates that the quantified anions exceed the estimate, indicating that the anions measured account for the majority of the soluble anions in the aqueous leachate of the sludge composite samples. If, for example, the sum of the anions accounted for only 10% of the estimated ions, then it would be likely that other ions were present in the leachate and had not been quantified.

3.7.15.3 Summary

Specific conductance was determined on the aqueous leachate from three sludge composite samples. An evaluation of these results in comparison to the sum of the anion concentrations in the same three samples indicates that the majority of the soluble anions have been quantified for the sludge samples.

3.7.16 Hydroxide by Titration

Analysis of hydroxide was required to support the evaluation of treatment options. All results are less than the detection limit.

3.7.16.1 Analytical Requirements

Analysis was requested by the Hanford-specific titration method which is implemented by laboratory method LA-211-102. Hydroxide concentration was determined by leaching each sludge composite sample with water, followed by a titration with a known amount of standardized nitric acid to a potentiometric endpoint. Interfering basic anions are precipitated with barium chloride before titrating. For each batch, the analytical method required a preparation blank, a reagent blank, and a standard.

The TSAP (EQM 1999a) required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore at least one duplicate was required for the sludge composite samples. The TSAP also specified MS samples at the same frequency. According to the TSAP, the required acceptance criteria for QC data are 80 to 120% for LCS recovery, 75 to 125% for spike recovery, and less than 20 for the RPD of duplicate samples. The TSAP required analysis of both supernatant and sludge composite samples; however, only sludge composite samples were available for analysis.

Validation was not required for hydroxide data.

3.7.16.2 QC Review

Hydroxide was determined by laboratory method LA-211-102 Rev. D/Mod 3. The analysis was not conducted on the supernatant (FDH 2000c, Attachment 4).

Water leachates of the sludge composite samples were analyzed in three Worklists: #32153, 32154, and 32155. Each worklist included a preparation blank and a standard. The data package did not include information for a reagent blank, so it is not clear if this method requirement was met. Sludge composite samples 263-1, 263-6, and 264-1 were run as the duplicate sample in the respective batch. The standard (i.e., LCS) recovery was acceptable for each batch. All results for preparation blanks and sludge composite samples were less than the MDL; therefore, RPD for duplicate samples could not be calculated. MSs were not performed.

3.7.16.3 Summary

The required analytical method was performed on the water leachates from 13 sludge composite samples. The preparation blank and standard were run as required in the method and duplicate samples were run as required in the TSAP. No MS samples were run (even though the laboratory method states that "Spikes, including matrix spikes, may be run if requested by the...customer"). Results did not meet the low PQL of 2,500 ppm; results for 10 out of 13 sludge composite samples did meet the high PQL of 8,400 ppm. Although all results are less than detection limits, the data may be used to establish an upper limit on the hydroxide concentration of the sludge.

3.7.17 pH

The pH was required as a RCRA parameter for waste designation. Results are presented in Table 3-57.

3.7.17.1 Analytical Requirements

Analysis was requested by Method 9040A for supernatant samples and by 9045B for sludge composite samples; these are implemented by laboratory method LA-212-105 (which is based on Method 9045C). pH is measured by electrode on a one-to-one reagent water to sample slurry of each sludge composite sample. For each batch, the analytical method required a standard pH buffer be run as the LCS.

Table 3-57. Results of pH Determination for Composite Sludge Samples from Tank 214-Z-361

Composite	Interval (in.)		pH	Duplicate
	Top	Bottom		
263-1	0.00	20.00	8.16	7.94
263-2	20.00	34.25	8.30	
263-3	34.25	38.00	8.49	
263-4	38.00	46.00	8.79	
263-5	46.00	59.00	8.67	
263-6	59.00	65.00	8.79	
263-7	65.00	74.00	8.68	
263-8	74.00	84.00	9.18	
264-1	0.00	16.50	8.60	7.91
264-2	16.50	33.50	8.06	
264-3	46.00	62.00	8.55	
264-4	62.00	69.00	8.48	
264-5	69.00	84.00	8.78	

The TSAP identified an LCS recovery of 0.1 pH and identified that acceptance criteria for spike recovery and RPD of duplicate samples was "N/A" or not applicable. The TSAP required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore at least one duplicate was required for the sludge composite samples. The TSAP also specified MS samples at the same frequency. The TSAP required analysis of both supernatant and sludge composite samples.

Validation was not required for pH data.

3.7.17.2 QC Review

The pH was determined by laboratory method LA-212-105 Rev. C/Mod 4. The determination was not conducted on the supernatant because only sludge composite samples were available for analysis (FDH 2000c, Attachment 4).

Worklist #32012 included a pH 6 buffer as the LCS. The result was within 0.1 pH unit of the known value. However, the method states, "Analyze laboratory control sample standards that are in the same pH region as the samples(s) and the calibration range. For example, use pH >7 standard for alkaline samples." Therefore, the wrong LCS was used for these samples. The data package does not identify the range over which the pH electrode/meter was calibrated. Sludge composite sample 264-1 was analyzed in duplicate with results of 8.60 and 7.91 pH units. The remaining samples ranged from 8.06 to 8.79 pH units.

Worklist #32045 included a pH 8 buffer as the LCS. The result was within 0.1 pH unit of the known value. This LCS was in the correct range for these alkaline samples. The data package does not identify the range over which the pH electrode/meter was calibrated. Sludge composite sample 263-1 was analyzed in duplicate with results of 8.16 and 7.94. The remaining samples ranged from 8.30 to 9.18 pH units.

As noted above, the TSAP identified the acceptance criterion for duplicates as "not applicable." The data report included RPDs for the duplicates of 8.36 and 2.73, respectively. Apparently, the "negative log" values for pH are averaged by the laboratory's data management system, which is an inappropriate manner in which to evaluate the data. To be mathematically correct, the hydrogen ion activity for each sample should be used, rather than the logarithmic values, in the RPD calculation. However, this results in RPDs of 132 and 49.7%, respectively.

Commonly, the acceptability of pH measurements for duplicate samples is determined by comparison to an absolute value such as was done for the LCS. Sludge materials exhibit characteristics and variability that are similar to soils. Usually, the goal for duplicate soil samples is ± 0.5 to 1 pH unit. Usually, the goal for water samples of low ionic strength is ± 0.1 pH unit and the goal for aqueous solutions of high ionic strength is ± 0.5 pH unit.

3.7.17.3 Summary

A one-to-one water slurry of each sludge composite sample was analyzed for pH in two batches. The results ranged from 7.91 to 9.18 pH units. The data are acceptable to confirm that the sludge is alkaline, as expected, and that the pH is well below the value of 12.5 which would cause the waste to be designated as corrosive.

3.7.18 Specific Gravity

Determination of specific gravity was performed to support treatment options. Results are presented in Table 3-58.

3.7.18.1 Analytical Requirements

Analysis was requested by 2710F (APHA 1995) a gravimetric method which, according to the TSAP, is implemented by laboratory method LA-510-112.

The TSAP (EQM 1999a) required one routine sample to be analyzed in duplicate for every 20 samples of similar matrix or preparation batch; therefore, at least one duplicate was required for the sludge composite samples. MS recovery and PQL objectives are not applicable to this parameter. No LCSs were required. According to the TSAP, the required acceptance criteria for RPD was less than 30%. The TSAP required analysis of both supernatant and sludge composite samples; however, only sludge composite samples were analyzed (FDH 2000c, Attachment 4).

Table 3-58. Specific Gravity for Sludge Composite Samples from Tank 241-Z-361

Composite	Interval (in.)		Specific Gravity (g/cm^3)		
	Top	Bottom	Sample	Duplicate	Replicates*
263-1	0.00	20.00	1.63	1.32	1.99; 1.40
263-2	20.00	34.25	1.39		
263-3	34.25	38.00	1.37		
263-4	38.00	46.00	1.19		
263-5	46.00	59.00	1.38		
263-6	59.00	65.00	1.08		
263-7	65.00	74.00	1.44		
263-8	74.00	84.00	1.36		
264-1	0.00	16.50	1.02	1.00	
264-2	16.50	33.50	1.38		
264-3	46.00	62.00	1.38		
264-4	62.00	69.00	1.45		
264-5	69.00	84.00	1.65		

*These values were rejected by the analyst; however, they may be within the range of actual specific gravity values for this composite sample.

Validation was not required for specific gravity.

3.7.18.2 QC Review

The determination was performed directly on the sludge composite samples by laboratory method LA-510-116 Rev C/Mod 2. The laboratory had noted that the analytical method designation was incorrect in the TSAP and confirmed this at the end of the project (e-mail from Ruth Esch to Mitzi Miller, March 16, 2000). The method is described as follows:

> The specific gravity of a sludge is the ratio of the mass of a volume of sludge to the mass of an equal volume of reagent water. The specific gravity is determined by displacing a known mass of material with a nonpolar liquid (such as hexadecane) to a fixed volume. The volume of the sludge can then be determined by subtracting the volume of nonpolar liquid added from the total volume. Once the volume corresponding to a known mass of sludge has been determined, the specific gravity can be computed.

Note that the method requires the use of a 5-place balance; therefore, resulting data contains four significant figures.

The method further notes that:

> A nonpolar liquid is used to displace the sample to a fixed volume, because Hanford Site waste (especially from tank farms) typically contains high levels of water soluble inorganics. If water were used to displace the samples, dissolution of some solids would likely occur. This dissolution would cause the specific gravity results to be biased high because the same mass of sample would occupy less volume. If a sample contains significant quantities of nonpolar organics, the results may be biased due to solvation effects. If the concentration of nonpolar species (that is, oil, grease, CCl_4, and so forth) is expected to exceed the concentration of polar components of a given sample, water should be used instead of a nonpolar liquid to displace the sample. In other words, the liquid that minimizes sample dissolution should be utilized.

The method QA includes one LCS (i.e., a 7.6 to 12.1 M solution of zinc chloride in 1% hydrochloric acid) and at least one duplicate per analytical batch. In addition, the method states "If a great accuracy and/or precision is necessary, and sufficient sample is available: replicates should be run and the average of the two results reported."

The determination of specific gravity was performed in three Worklists (#32010, 32040, 32766).

Worklist #32010 included the LCS and a duplicate. Recovery for the LCS was 104.6%. RPD for the duplicate (i.e., 264-1) was 1.980. Other sludge composite samples in this Worklist were 263-6, 264-2, 264-3, 264-4, and 264-5.

Worklist #32040 included the LCS and a duplicate. Recovery for the LCS was 101.6%. RPD for the duplicate (i.e., 263-1) was 34.81. Other sludge composite samples in this Worklist were 263-2, 263-3, 263-4, 263-5, 263-7, and 263-8.

Worklist #32766 included the LCS and a duplicate sample. Recovery for the LCS was 101.6%. RPD for the duplicate was 21.02. The sludge composite sample 263-1 was the only sample in this batch. It was rerun because of high RPD (i.e., 34.81) between the duplicate values when originally measured. The analyst noted "Use sample S00Z000027 and report as S99Z000264"; therefore, the database should be corrected to reflect the correct subsample for analysis.

The data report and narrative do not state what the displacing liquid was for these samples; however, by working through the calculation for one sludge composite sample from each worklist, the review confirmed that hexadecane was used as the displacement liquid.

3.7.18.3 Summary

Specific gravity was determined on 13 sludge composite samples with reported results ranging from 1.00 to 1.65. Although not all QC data met TSAP requirements, the specific gravity results are useable for this project.

3.8 DRY WELL LOGGING/NDA TESTING

3.8.1 Procedure/Method Summary

The NDA data report ("Report of Geophysical Logging at 241-Z-361" [WMTS 2000]) presents a summary of geophysical logging operations conducted in two risers of Tank 241-Z-361 by Waste Management Technical Services (now Duratek Technical Services). As discussed in the report, the purpose of the logging was to produce an estimate of the concentration of radionuclides of interest as a function of depth. An initial goal of the logging also was to identify and quantify nonradioactive elements such as chromium and mercury; however, the presence of large gamma flux (relative to detector sensitivity) in the dry wells prohibited this evaluation from being attained due to the methodology employed for neutron capture/spectral gamma logging.

The measurements were acquired in two dry wells which were installed within the risers several years ago. The dry wells consist of aluminum pipes that penetrated the waste contents. The dry wells are closed at the bottom, but are open at the top several inches below the tank roof. The dry well in Riser G has an inside diameter of 3.75 in and a wall thickness of 0.125 in. The dry well in Riser B has an inside diameter of 3.0 in, and a wall thickness of 0.125 in. It is not known if any previous measurements were acquired in these dry wells. Figure 1-2 illustrates the cross section. Figure 1-1 of Tank 241-Z-361 shows the plan view and the location of the risers (which contain the dry wells) in which log data were acquired.

The original scope of the logging operations was to utilize several logging techniques to characterize the sludge in Tank 241-Z-361. These techniques implemented passive neutron/ spectral gamma, neutron capture/ spectral gamma, and passive neutron/neutron moisture logging tools. Gamma-emitting radionuclides in the sludge, such as plutonium-239 and americium-241, could be identified from spectral gamma data acquired with the passive gamma high-purity germanium (HPGe). Passive neutron data are used to identify the presence of neutrons produced by spontaneous fission of plutonium isotopes and by (alpha, n) reactions (mainly with oxygen) from alpha decay of americium-241. The neutron log data also will provide background correction for the neutron moisture measurements acquired with an active neutron source. All logging tools utilized for the logging were calibrated with the Hanford Site calibration models.

Initial concerns regarding the utilization of the neutron capture/spectral gamma logging tool were associated with the potential for high neutron and gamma flux in the dry wells which would essentially saturate the tools and make them incapable of acquiring useful data. In addition to the three logging tools described above, a smaller gross gamma logging tool (called a Mount Sopris logging tool in the data report) was implemented to acquire initial data to determine if the anticipated high fields were present. This tool is not calibrated and was intended for acquiring gross gamma measurements for screening purposes only.

The initial logging operations were conducted on April 27, 2000, in Riser G. The dry well in this riser is constructed of 3.750 in inside diameter (ID) aluminum tubing with a wall thickness of 0.125 in. Upon placement of the passive neutron/spectral gamma-logging tool into the dry well within the riser, it was noticed that the fit was quite tight due to small annular space between the outside surface of the logging tool and the ID of the dry well tubing. The outside diameter (OD) of the logging tool is 3.625 in and, as mentioned previously, the ID of the casing is 3.750 in. Although not part of the original plan, plastic sleeving had been placed inside the dry well to prevent contamination of the logging tool and cable because activity of about 400 dpm were detected in swabs of the inside of the dry well tubing. It was decided not to log the borehole with the neutron capture/spectral gamma tool due the small annular space and plastic as there was a potential to lodge the tool inside the casing in the event that the plastic sleeving was torn and became gathered around the tool.

The small diameter tool, which has an OD of 1.875 in., was utilized to acquire the initial gamma activity measurements. The neutron moisture tool was utilized to acquire neutron activity measurements, both in the active and passive (americium-beryllium [AmBe] source removed) mode. All depth measurements were acquired relative to the top of the riser because all in-tank samples had been acquired relative to this datum (at the request of project scientists, the data were later normalized to ground surface). All data in this section are presented relative to ground surface. Measurements were not acquired above ground surface within the riser stickup (approximately 2 ft). Logging was conducted at a rate of 2 ft/min at sample intervals of 0.5 ft, and data were acquired at depths between 2.0 and 19.5 ft. The small diameter logging tool saturated at over 1,000,000 counts per second (cps) between depths of 13 and 21 ft. The count rate decreased dramatically as the tool was withdrawn from the tank at a depth of about 4 ft and entered the overburden covering the tank. Within the tank, the gamma count rate steadily increased to the depth at which the tool became saturated (approximately 13 ft). The small diameter tool is about half as efficient as the 35% HPGe spectral gamma; therefore, had the passive neutron/spectral gamma tool fit into the dry well, it also would have saturated (most likely high up in the dry well).

Riser G also was logged with the neutron moisture tool with the source installed and then removed. The neutron moisture logging tool is calibrated for vadose zone conditions of soil density and moisture content up to 20%. Because the conditions in the waste were different (sludge versus soil) and the moisture content values were well beyond the calibration values (up to 70%), the log data were reported in uncalibrated neutron count rate. The moisture count rates were corrected for the neutron count rates measured with the source removed. Measurements were acquired at a rate of 0.5 ft/min at 0.250 ft sample intervals. The depth interval between 10 and 21.5 ft was logged in the active mode (source installed), and a repeat survey was conducted

between depths of 14 and 21.5 ft. The depth interval logged in the passive mode (source removed) was between depths of 2.0 and 21.0 ft, and a repeat survey was conducted between depths of 12.0 and 21.0 ft. The neutron count rates (in the active mode) increased sharply from less than 100 cps to about 1,500 cps at a depth of 15.5 ft where the sludge was encountered. Within the sludge, the neutron count rates varied between 1,500 and 1,600 cps; some peaks and valleys occurred within the sludge and most likely reflected changing properties due to layering within the sludge.

Upon observation of the extremely high gamma ray count rates in Riser G, it was decided to implement a less sensitive wafer HPGe detector to acquire spectral gamma data in Riser G. This logging tool, which is designated the high rate logging system (HRLS), consists of a low-efficiency, 6-mm by 8-mm n-type HPGe detector. A tungsten shield can be utilized to extend the range of detection of the HRLS. It was anticipated that this detector, which is about one-two hundredths (1/200) the efficiency of the 35% HPGe, could acquire valid measurements in the sludge. The logging tool, which has an OD of 3.375 in, was enclosed in shrink-fit tubing and this allowed the plastic liner in the dry well to be removed without increasing the potential of contaminating the tool. Also, because the shrink-fit tubing fit the tool housing tightly, the potential for tearing and gathering loose tubing material around the tool was decreased.

Logging with the HRLS was conducted in Riser G on May 18, 2000, in accordance with logging procedures outlined in MAC-VZCP 1.7.10-1. Field verification spectra were collected before and after logging in the riser. Both verification spectra were within acceptable statistical limits and confirmed that the logging system was operational. Seven log runs were conducted in Riser G with the HRLS. Due to the high dead times encountered during the first four surveys, the standard HRLS housing was replaced with the housing containing an internal tungsten shield. The remaining log runs were conducted with this shielded detector. However, the tungsten shield decreased the gamma counts below the number required for a useable spectrum. After several more attempts to acquire data with stationary counts, high rate logging was discontinued following run 7.

Logging in Riser B was conducted on May 2, 2000, utilizing the small diameter total gamma and neutron moisture logging tools. The neutron moisture tool was used in the active and passive modes as it was in Riser G. The dry well within this riser was constructed of 3 in ID aluminum casing with a wall thickness of 0.250 in. This smaller size tubing (as compared to Riser G) prohibited the option of using the 3.375-in- and 3.625-in-OD spectral gamma logging tools.

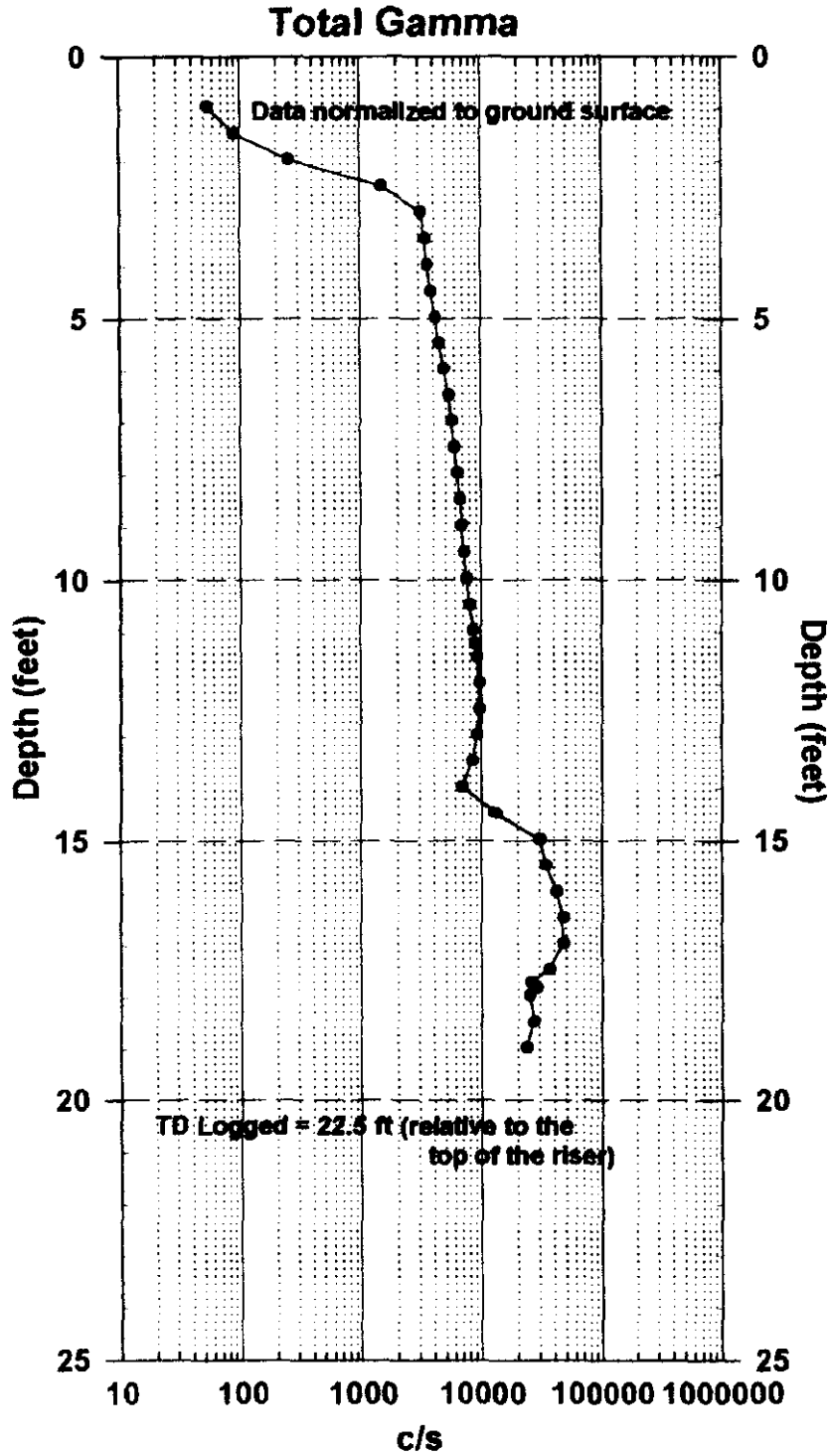
The total gamma data were acquired with the small diameter total gamma logging tool at a rate of 0.8 to 1.0 ft/min at a sample interval of 0.5 ft. Data were collected between depths of 4.0 and 22.5 ft, and a repeat survey was conducted between depths of 15.0 and 22.5 ft.

Data were collected with the neutron moisture tool in both the active and passive modes. In the active mode, data were acquired at a logging rate of 1.0 ft/min at a sample interval of 0.250 ft. The data were acquired between depths of 3.0 and 24.0 ft, and a repeat survey was conducted between depths of 15.0 and 24.0 ft. In the passive mode, the acquisition rate and sampling frequency was the same as the active mode. Log data were acquired between depths of 3.0 and 24.5 ft, and repeat surveys were conducted between depths of 15.0 and 24.5 ft, and between depths of 21.5 and 24.5 ft.

3.8.2 Summary of Results

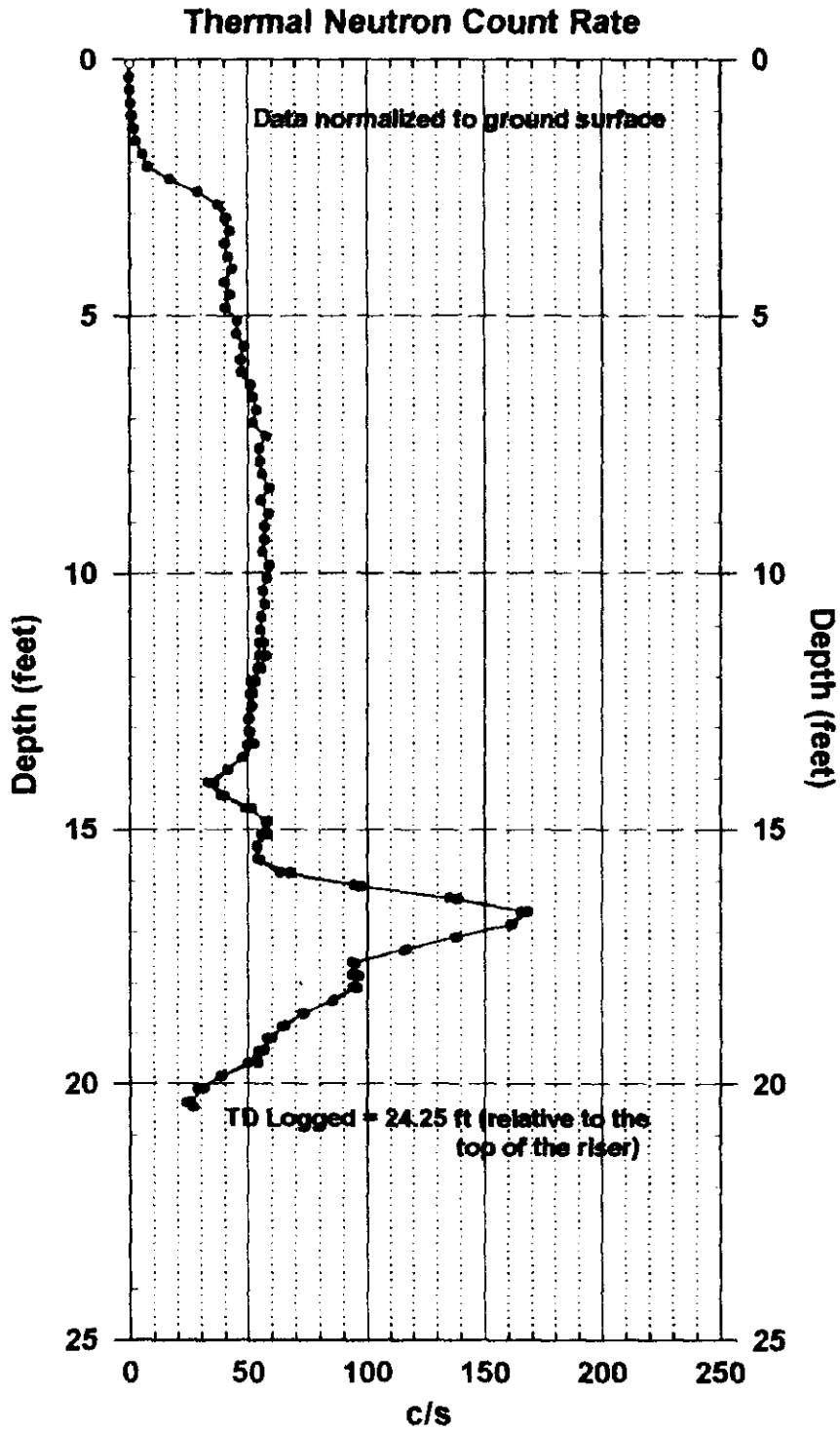
As discussed above, dry well logging data were obtained for Risers B and G. Presented below in Figures 3-9 through 3-18 are the graphical results of the logging. The data logs were acquired relative to the top of the riser from which they were obtained. They have been normalized to the ground surface so that the comparison from riser to riser is simplified. The surface of the sludge is taken to be about 13.7 ft from the surface of the ground. In the case of plutonium-239 and neptunium-237, only a few points were obtained.

Figure 3-9. Gamma Ray Profile of Tank 241-Z-361 from Riser B



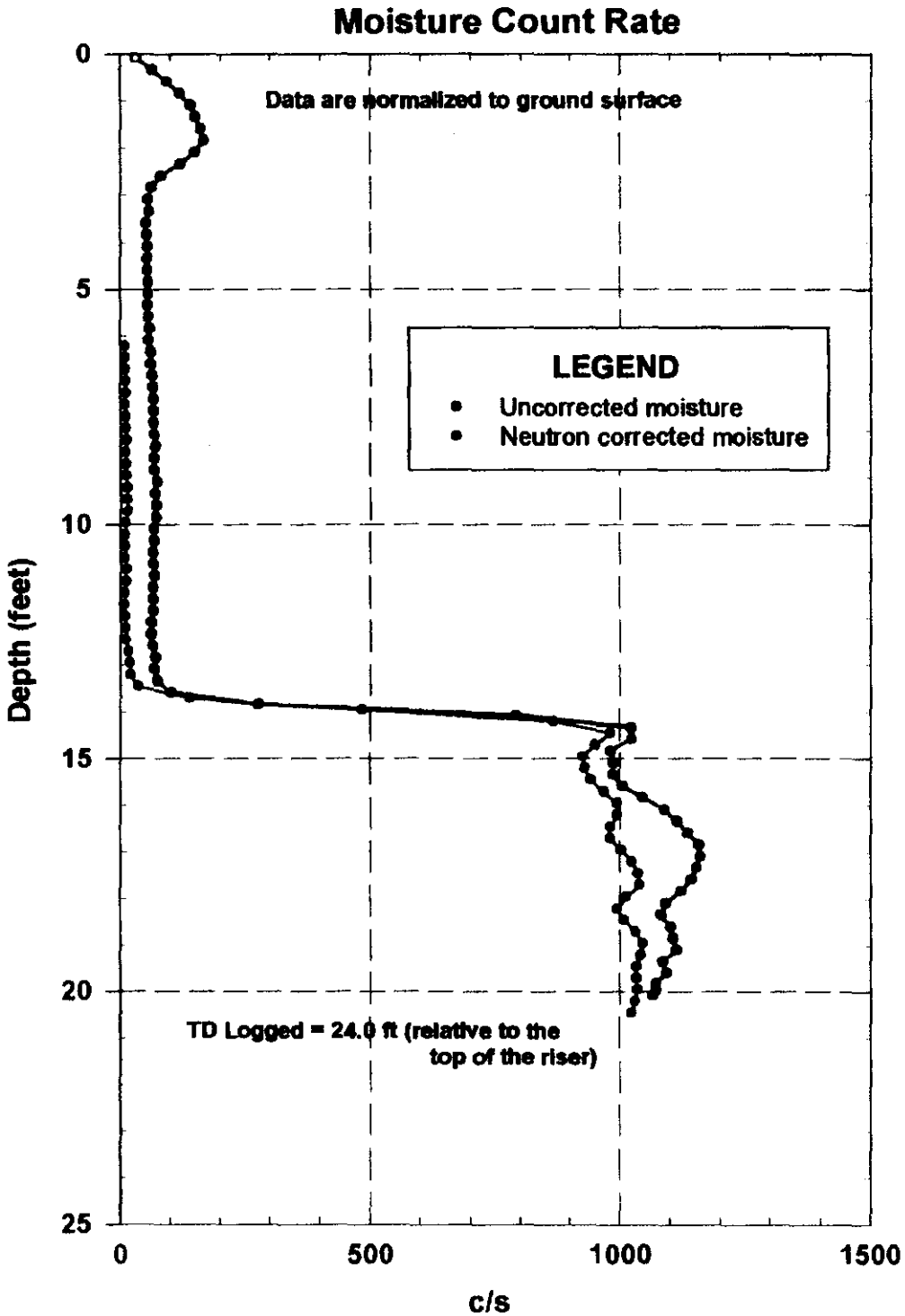
Source: RLS Gamma Ray Borehole Survey, Waste Management Technical Services, Riser "B," Tank 361, Gamma Ray Profile, Log Date: May 2, 2000.

Figure 3-10. Passive Neutron Profile of Tank 241-Z-361 from Riser B



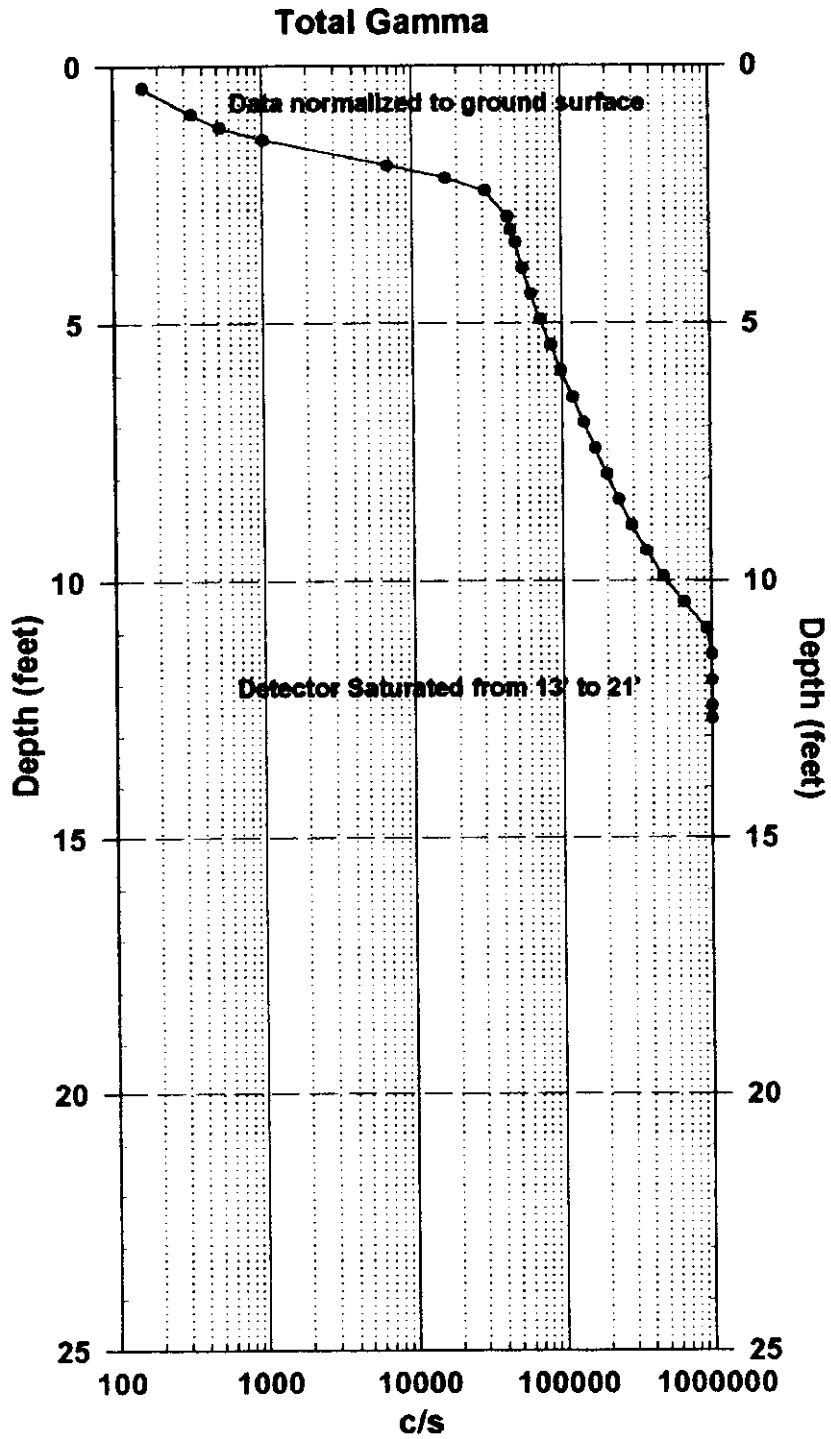
Source: RLS Passive Neutron Borehole Survey, Waste Management Technical Services, Riser "B," Tank 361, Passive Neutron Profile, Log Date: May 2, 2000.

Figure 3-11. Moisture Count Rate Profile of Tank 241-Z-361 from Riser B



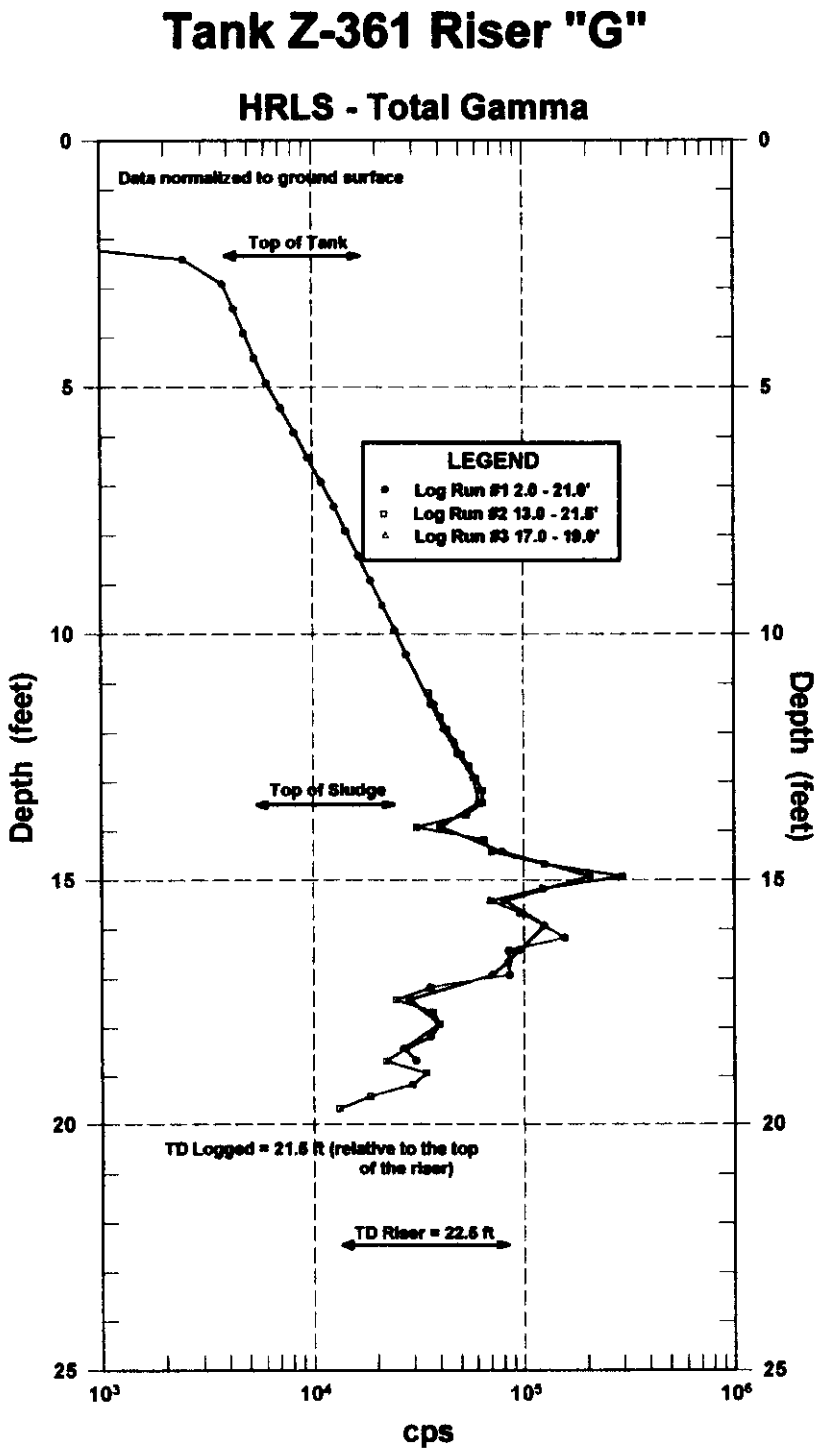
Source: RLS Moisture (c/s), Waste Management Technical Services, Project: PFP Logging, Log Date: May 2, 2000, Borehole: Riser B, Depth Datum: Top of Riser.

Figure 3-12. Gamma Ray Profile of Tank 241-Z-361 from Riser G



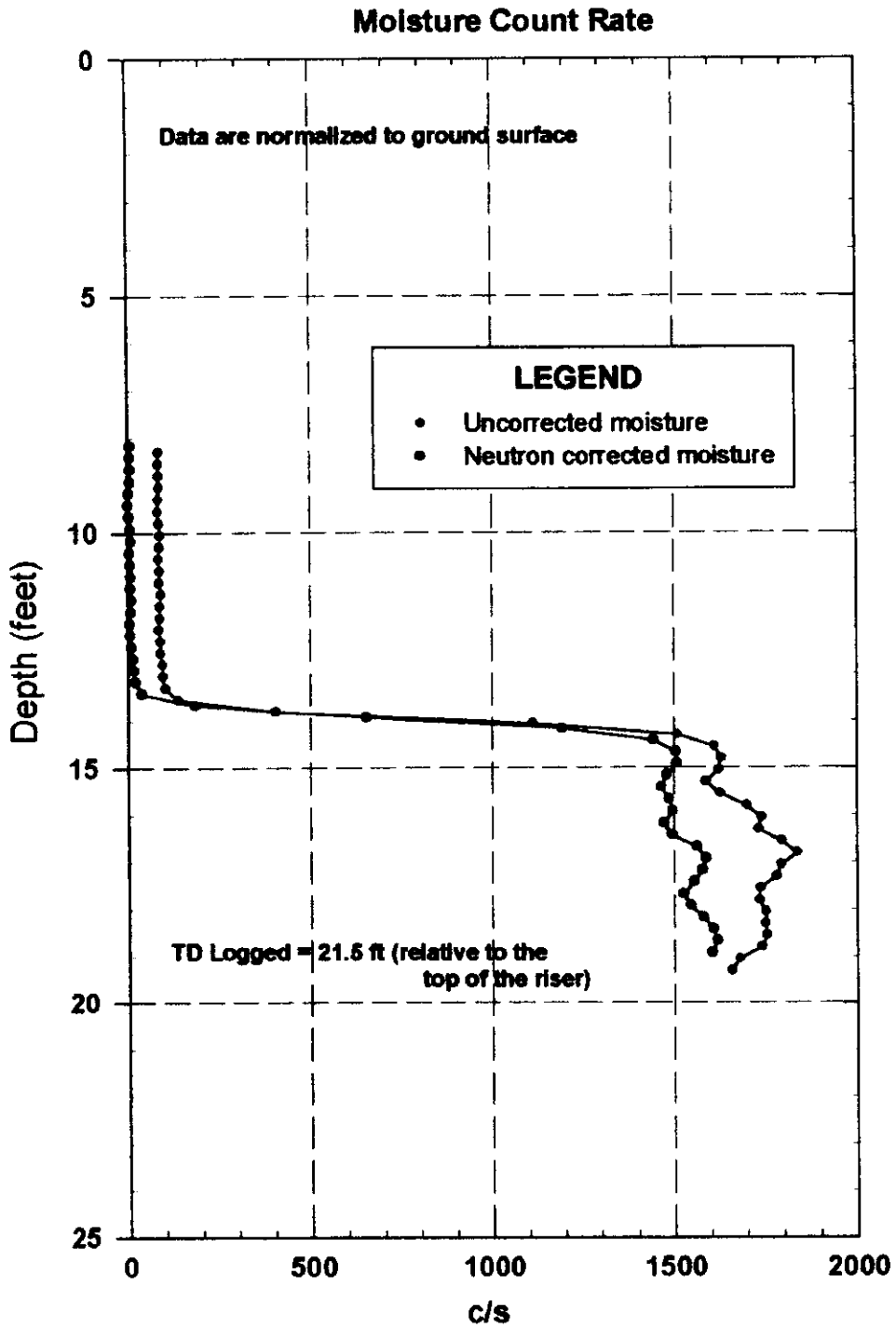
Source: RLS Gamma Ray Borehole Survey, Waste Management Technical Services, Riser "G," Tank 361, Gamma Ray Profile, Log Date: April 27, 2000.

Figure 3-13. HRLS Total Gamma Profile of Tank 241-Z-361 from Riser G



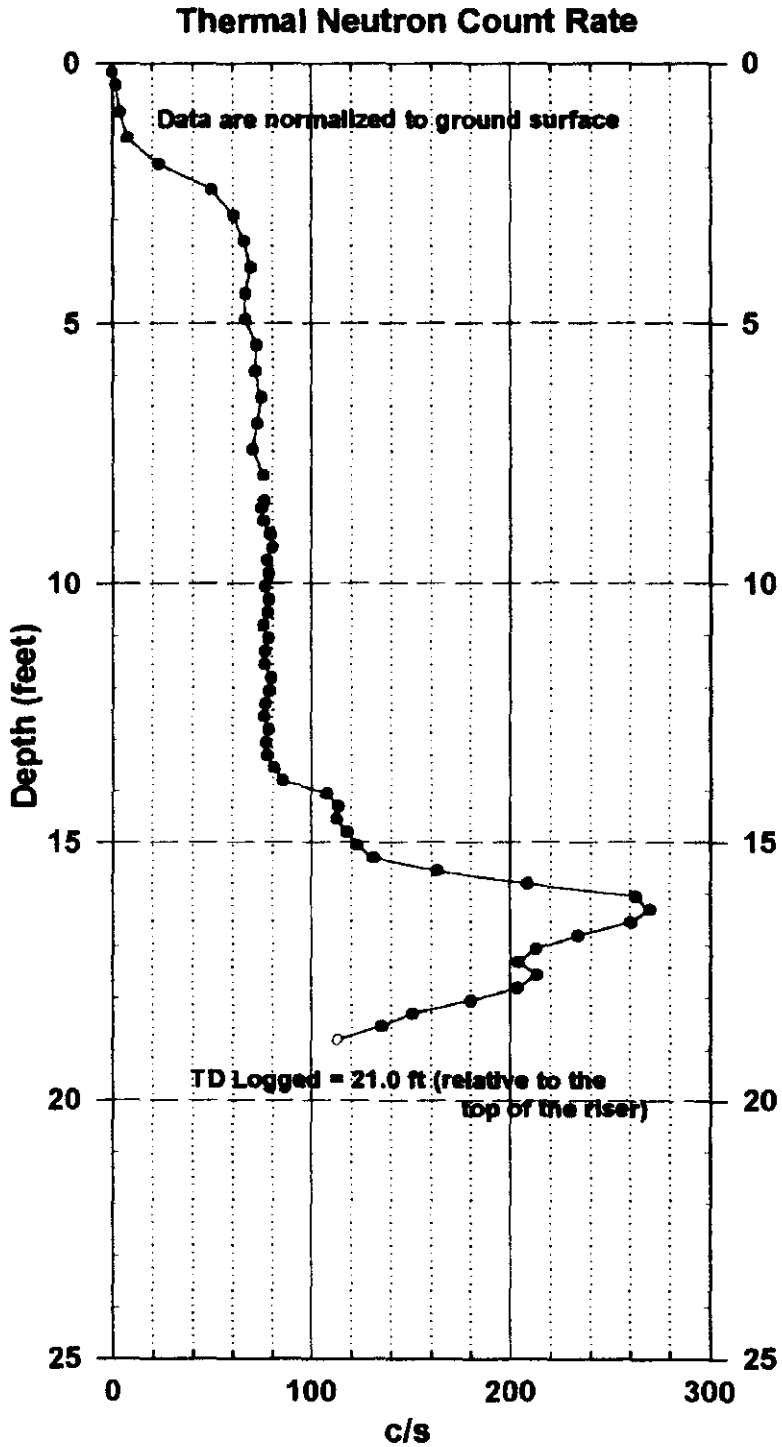
Source: Graphical representation of data found in Appendix E.

Figure 3-14. Moisture Count Rate Profile of Tank 241-Z-361 from Riser G



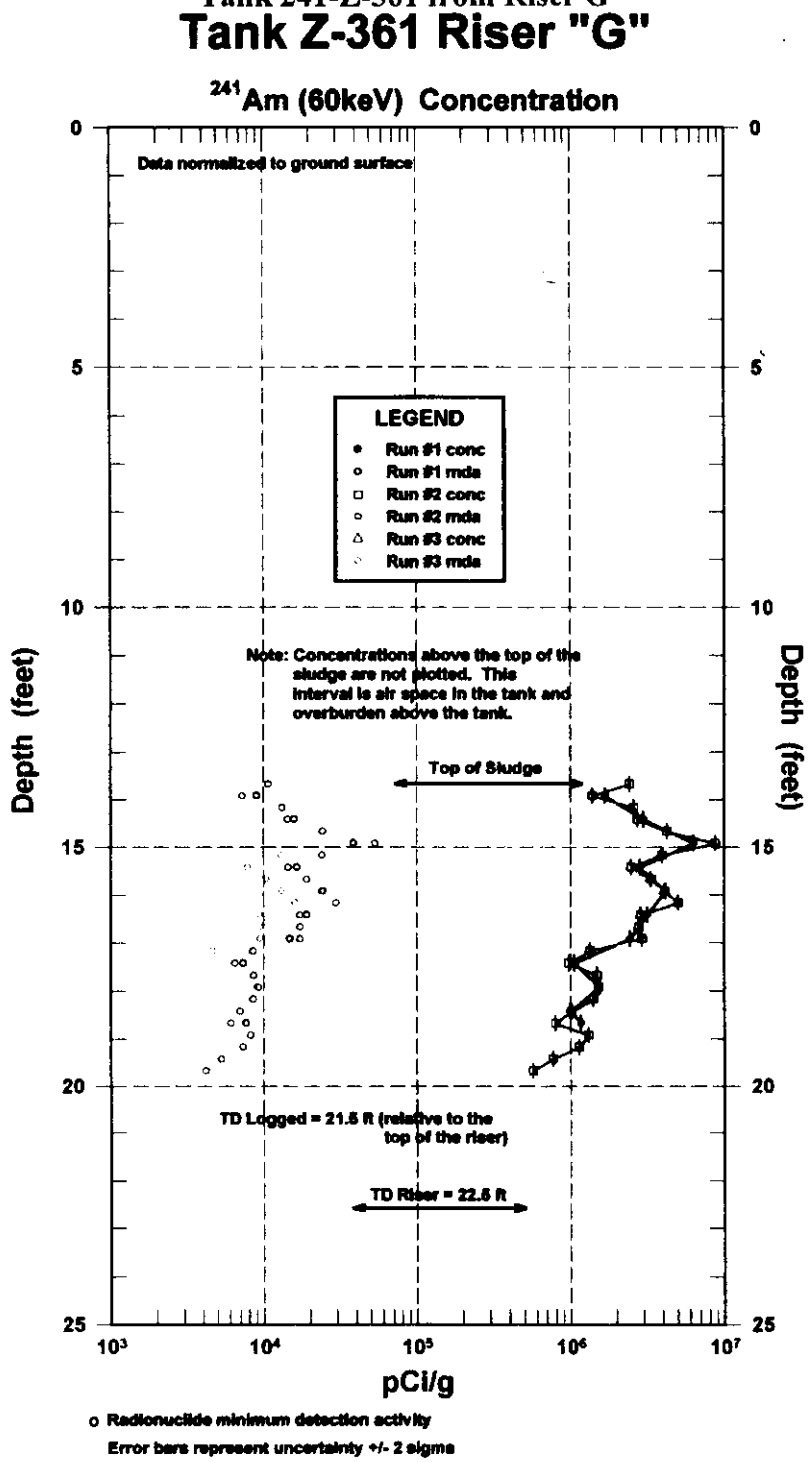
Source: RLS Moisture (c/s), Waste Management Technical Services, Project: PFP Logging, Log Date: April 27, 2000, Borehole: Riser G, Depth Datum: Top of Riser.

Figure 3-15. Passive Neutron Profile of Tank 241-Z-361 from Riser G



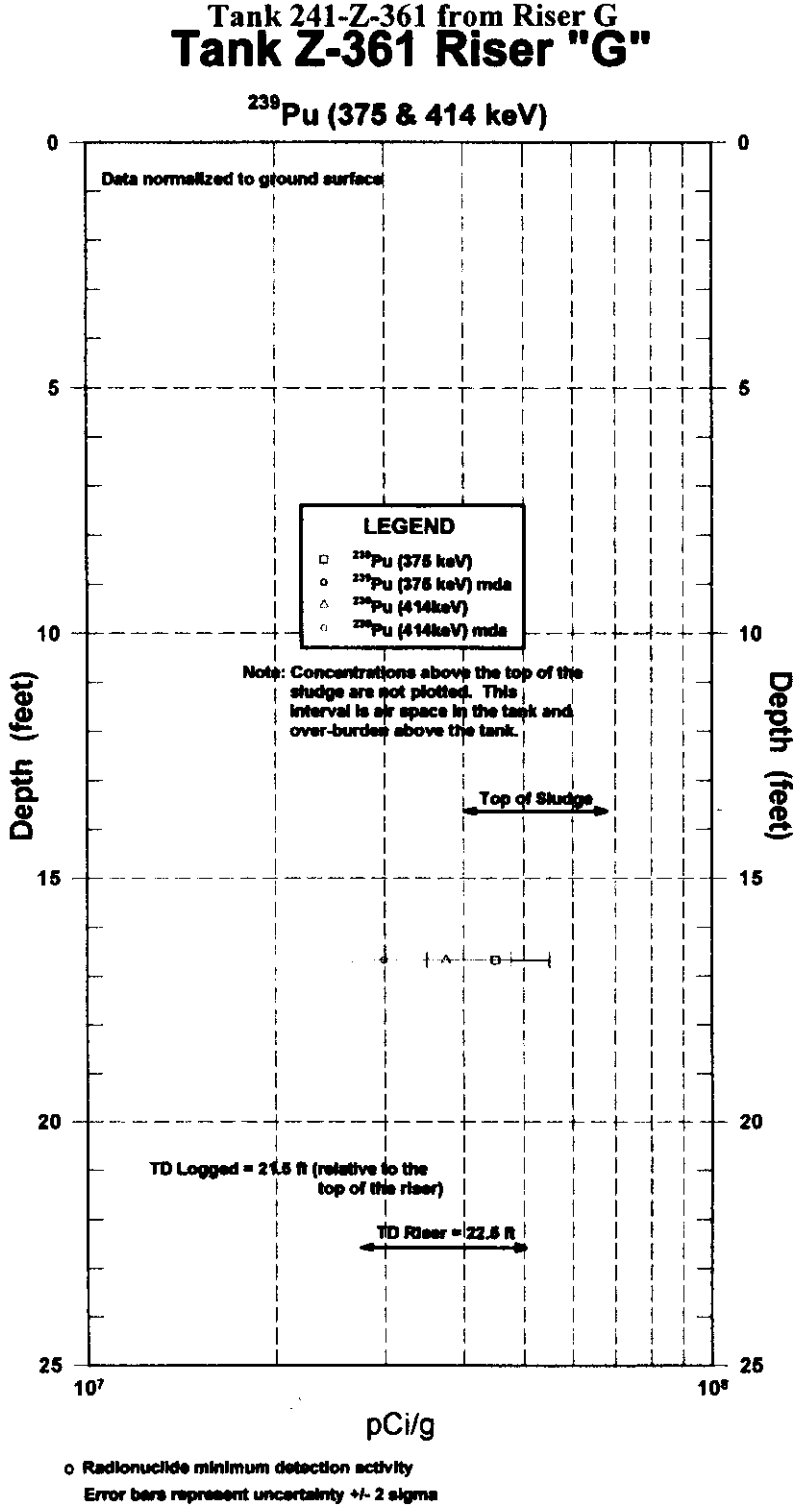
Source: RLS Passive Neutron Borehole Survey, Waste Management Technical Services, Riser "G," Tank 361, Passive Neutron Profile, Log Date: April 27, 2000.

Figure 3-16. Estimated Spectral Gamma Americium-241 Profile of Tank 241-Z-361 from Riser G



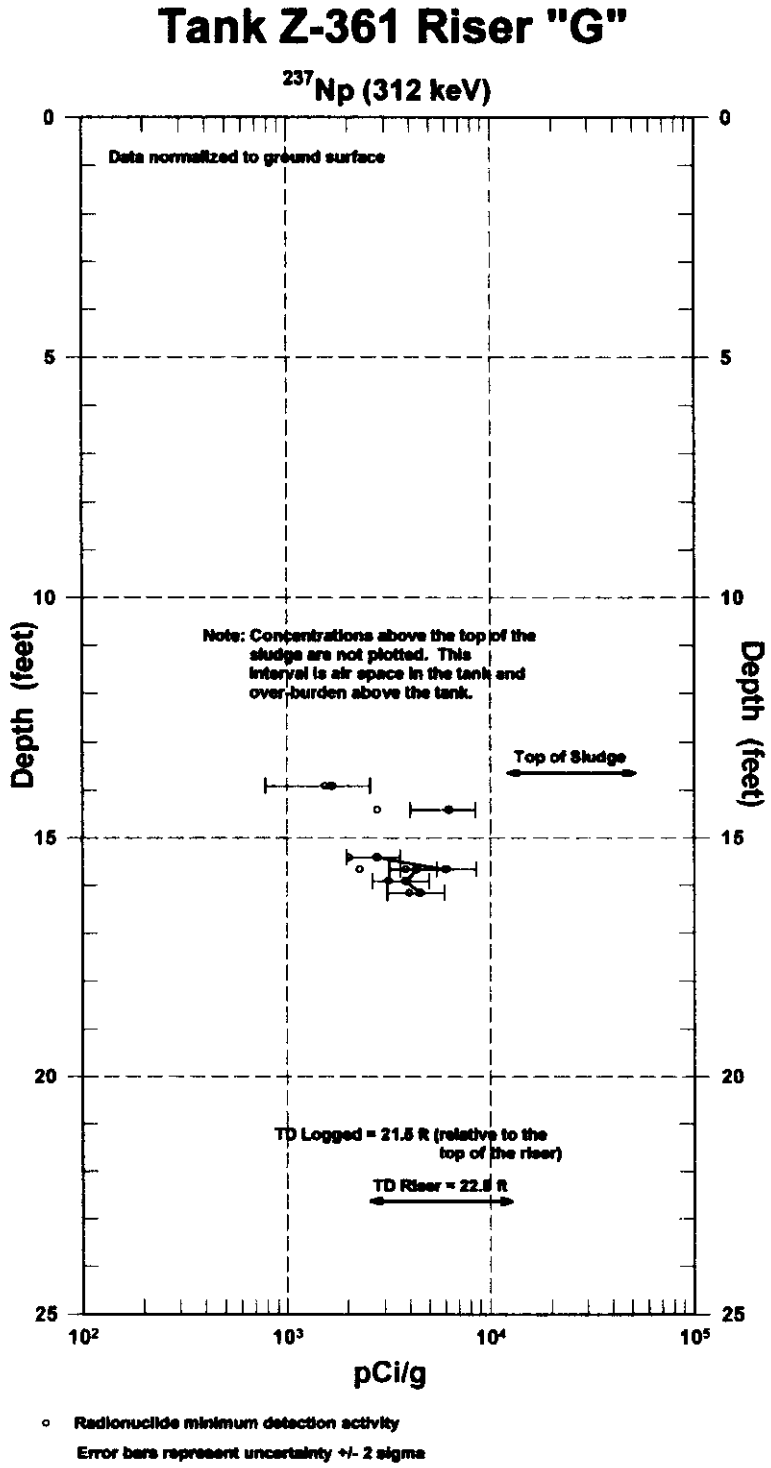
Source: Graphical representation of data found in Appendix E.

Figure 3-17. Estimated Spectral Gamma Plutonium-239 Content of a Single Depth in Tank 241-Z-361 from Riser G



Source: Graphical representation of data found in Appendix E.

Figure 3-18. Estimated Spectral Gamma Neptunium-237 at Several Depths in Tank 241-Z-361 from Riser G



Source: Graphical representation of data found in Appendix E.

3.8.3 Correlation To Sludge Composite Samples

Evaluation of the dry well logging data (WMTS 2000) was performed and the following comparisons and conclusions drawn.

3.8.3.1 Total Gamma Analysis

The passive total gamma logging results (see Figures 3-9, 3-11, 3-12, and 3-13) did not provide data that was useful in characterizing the tank waste. The gross gamma determination suffered from saturation of the detector due to the high levels of americium-241 in the waste. The HRLS gamma determination (Figure 3-13) provided the same spectral shape as seen for the americium-241 (Figure 3-16) determination. It is apparent from inspection of the laboratory data and the NDA plots that the americium-241 levels in the tank make the use of total gamma measurements of limited usefulness. It is possible that the use of specific shielding/detector configurations would generate information on the other gamma emitting radionuclides in the tank. However, the potential usefulness of those determinations is not clear. It would not be recommended that any future total gamma measurements be taken. Estimates of gamma field and calculations of adequate shielding and detector configuration for the other NDA determinations discussed below can be performed generally on process knowledge, laboratory, or dose rate data already available.

3.8.3.2 Neutron Moisture Determination

As stated in the data report (WMTS 2000), the neutron moisture tool was not calibrated for the expected moisture levels and matrix of the tank. As a result, only counts per second of thermalized neutrons were reported. Assuming that the measured net neutron counts per second are proportional to moisture, the neutron moisture determinations (Figures 3-11 and 3-14) showed a variable but generally increasing moisture level with increasing depth in the sludge. The active neutron moisture logging might be useful if the neutron moisture logging instrument were calibrated over the anticipated moisture levels to allow the counts per second to be converted to percent moisture in the sludge. However, it should be noted that laboratory data for moisture (Table 3-42) generally did not agree with the NDA data. The NDA data indicated a general increase in the moisture level with depth. In general, the laboratory data (Table 3-42) showed an unchanged to slight decrease in percent moisture as the depth of the sludge increased.

3.8.3.3 Spectral Gamma Determination of Americium-241

The measurement of americium-241 via its 60 KeV gamma ray emission would appear to be fairly accurate. A qualitative estimate of the potential accuracy of the NDA method was performed on the limited data available. The estimated concentration profile for americium-241 in the tank as measured in Riser G is shown in Figure 3-16. Tabulated in Table 3-59 is a comparison of the average NDA estimate of americium-241 activity in a specific depth interval to the laboratory data that approximately corresponded with that depth interval. An average estimated concentration of americium-241 from NDA was 3.7E6 pCi/g in Riser G in a depth interval that is compared to sludge composite sample 263-1 (Riser E). The estimated concentration from laboratory data was 1.4E7 pCi/g (sludge composite sample 263-1). Thus, the

estimate of americium-241 from NDA was about 26% of the laboratory data. A similar comparison between the NDA estimate and sludge composite sample 263-2 (Riser E) showed an NDA estimate of about 25% of the laboratory value. NDA results range from 25 to 59% of the laboratory results. Also presented in Table 3-59 is a comparison of the americium-241 NDA data to the laboratory data from Riser F.

Table 3-59. Comparison of Americium-241 Determined by Spectral Gamma NDA and Laboratory Analysis of Sludge Composite Samples (2 sheets)

Depth of Sludge (inches below surface[1])	NDA		Core 263			Core 264		
	Riser G NDA runs Am-241 (µCi/g)	Riser G NDA runs Am-241 (Average µCi/g)	Riser E Core 263 Analytical Sludge Composite Samples	Riser E Laboratory Data Am-241[2] (µCi/g)	Ratio of Riser G NDA data to Riser E Core Data	Riser F Core 264 Analytical Sludge Composite Samples	Riser F Laboratory Data Am-241[2] (µCi/g)	Ratio of Riser G NDA data to Riser F Core Data
0	2.4		263-1			264-1		
3	1.7		263-1			264-1		
3	1.4		263-1			264-1		
6	2.6		263-1			264-1		
9	3.0	3.7	263-1	14.1	0.26	264-1	7.5	0.50
9	2.7		263-1			264-1		
12	4.2		263-1			264-1		
15	6.3		263-1			264-1		
15	8.7		263-1			264-1		
18	3.9		263-1			264-2		
18	3.9		263-1			264-2		
21	2.8		263-2			264-2		
21	2.5		263-2			264-2		
21	2.5		263-2			264-2		
24	3.3		263-2			264-2		
24	3.3		263-2			264-2		
27	4.1	3.5	263-2	13.9	0.25	264-2	13.4	0.26
27	4.1		263-2			264-2		
27	4.1		263-2			264-2		
30	5.0		263-2			264-2		
30	5.0		263-2			264-2		
33	3.1		263-2			264-2		
33	2.9		263-2			264-2		
33	2.8		263-2			264-2		
36	2.8		263-3			NS		
36	2.8	2.8	263-3	8.8	0.32	NS		
39	2.5		263-4			NS		
39	2.9		263-4			NS		
39	2.9		263-4			NS		
42	1.4	1.9	263-4	3.9	0.48	NS		
42	1.3		263-4			NS		

Table 3-59. Comparison of Americium-241 Determined by Spectral Gamma NDA and Laboratory Analysis of Sludge Composite Samples (2 sheets)

Depth of Sludge (inches below surface[1])	NDA		Core 263			Core 264		
	Riser G NDA runs Am-241 (µCi/g)	Riser G NDA runs Am-241 (Average µCi/g)	Riser E Core 263 Analytical Sludge Composite Samples	Riser E Laboratory Data Am-241[2] (µCi/g)	Ratio of Riser G NDA data to Riser E Core Data	Riser F Core 264 Analytical Sludge Composite Samples	Riser F Laboratory Data Am-241[2] (µCi/g)	Ratio of Riser G NDA data to Riser F Core Data
45	1.1		263-4			NS		
45	9.7		263-4			NS		
48	1.5		263-5			264-3		
51	1.6		263-5			264-3		
51	1.5	1.3	263-5	4.7	0.28	264-3	5.7	0.23
54	1.4		263-5			264-3		
57	1.0		263-5			264-3		
57	1.0		263-5			264-3		
60	1.2		263-6			264-3		
60	7.9	1.1	263-6	4.3	0.25	264-3		
63	1.3		263-6			264-4		
66	1.1		263-7			264-4	4.8	0.23
69	7.6	0.8	263-7	1.4	0.59	264-4		
72	5.7		263-7			264-5	1.04	0.80

1. Based on NDA data, surface of sludge was approximately 13.67 ft below the ground surface around Tank 241-Z-361.
2. Data from Table 3-24

It is interesting that the NDA results range from 23 to 80% of the laboratory results for Riser F. Given the high dead time (due to high count rates) that was experienced by the americium-241 detector in this segment and the estimation of attenuation (shielding factors) for the risers, it is not surprising that the estimate of americium-241 is low. Any future counting should use a better shield and detector design to provide higher quality data and illustrates significant potential for these measurements.

It should also be noted that the NDA determination of americium-241 and the laboratory sample determinations are all performed at different locations in the tank. There are significant differences (approximately 3×) in the laboratory sample-determined americium-241 content of the tank between Riser E and F at similar depths. Thus, the estimated americium-241 content of the tank sludge by NDA measurements may be very close to the actual radionuclide content of the sludge at Riser G. It is not possible to accurately determine if there is a bias in the NDA data compared to laboratory data.

3.8.3.4 Passive Thermal Neutron Measurements

The measurement of thermal neutrons as a function of depth was expected to provide information as to the amount of fissile material in the sludge. This fissile material undergoes spontaneous fission at a known rate and, thus, it is theoretically possible to estimate the sludge content of fissile material by measuring the neutron flux.

There are confounding issues. The major issue is the production of neutrons by the interaction of alpha particles with the sludge, called alpha/n reactions. The passive thermal neutron measurements (Figures 3-10 and 3-15) generally showed a peak of neutron counts at the general depth of approximately 16.3 ft from the surface (approximately 31.5 in. below the sludge surface). This neutron peak is in the same sludge composite sample (same general depth) as the maximum plutonium content of the sludge as shown in the laboratory data for sludge composite sample 263-2 (20 to 34.25 in below the sludge surface).

The data could likely be improved if the alpha/n reactions from americium-241 were stripped out of the neutron counting profile. This could be accomplished by using americium-241 logging data to first estimate the americium-241 concentration of the sludge. The estimated neutron flux from the americium-241 could be stripped out of the neutron counting profile. That would leave mainly the contributions from the spontaneous fission of plutonium and other fissile materials (as well as the alpha/n reactions from other fissile materials). No attempt was made to estimate the fissile material content of the sludge from these data.

To better understand the relationship between thermal neutron activity and plutonium-239 concentrations, the following analysis is performed. A comparison is made between the thermal neutron activity in Risers B and G with plutonium-239/240 concentrations from Riser E. All data are normalized to the ground surface.

Figure 3-19 shows that the thermal neutron activity from Risers B and G and the plutonium-239/240 concentrations from Riser E all display similar qualitative changes with respect to depth. The data used in Figure 3-19 is presented in Table 3-60.

Many more data points are available from the thermal neutron activity (i.e., NDA measurements; approximately one measurement every three in) than for plutonium-239/240 on the sludge composite samples. The plutonium-239/240 concentrations on the sludge are the results of composite samples and represent spatial averages over the vertical regions that were composited. In presenting the plutonium-239 data, the value of the corresponding composite was assigned to each interval where data were available for the thermal neutron data sets.

Inspection of Figure 3-19 shows that the passive neutron NDA measurements generally coincide with the laboratory results for each sludge composite sample. These data suggest that passive neutron NDA may be a useful tool in evaluating the vertical profile of fissile materials in the sludge.

3.8.3.5 Spectral Gamma Determination of Plutonium-239

The NDA measurement of plutonium-239 was performed and reported in Riser G at a single depth near the peak thermal neutron location (about 16.7 ft, Figure 3-17). The one point provided an estimated concentration of plutonium-239 from NDA of 4.1E7 pCi/g (average of estimates from both 375 and 414 KeV gamma energies). The estimated concentration from laboratory data (from sludge composite sample 263-2 at corresponding depth) was 2.4E7 pCi/g. Thus, the estimate of plutonium-239 from NDA of a single point within the interval corresponding to sludge composite sample 263-2 was 170% of the laboratory data for the entire segment. Because nothing is known with regard to the vertical distribution of plutonium-239 within the composite, it is not possible to tell whether the NDA data are biased high or not. Future counting with a better shield design and longer counts, if needed, should provide higher quality data.

Figure 3-19. Graphical Comparison of NDA Passive Neutron Measurements on Risers B and G to Plutonium-239/240 Laboratory Data from Riser E Core

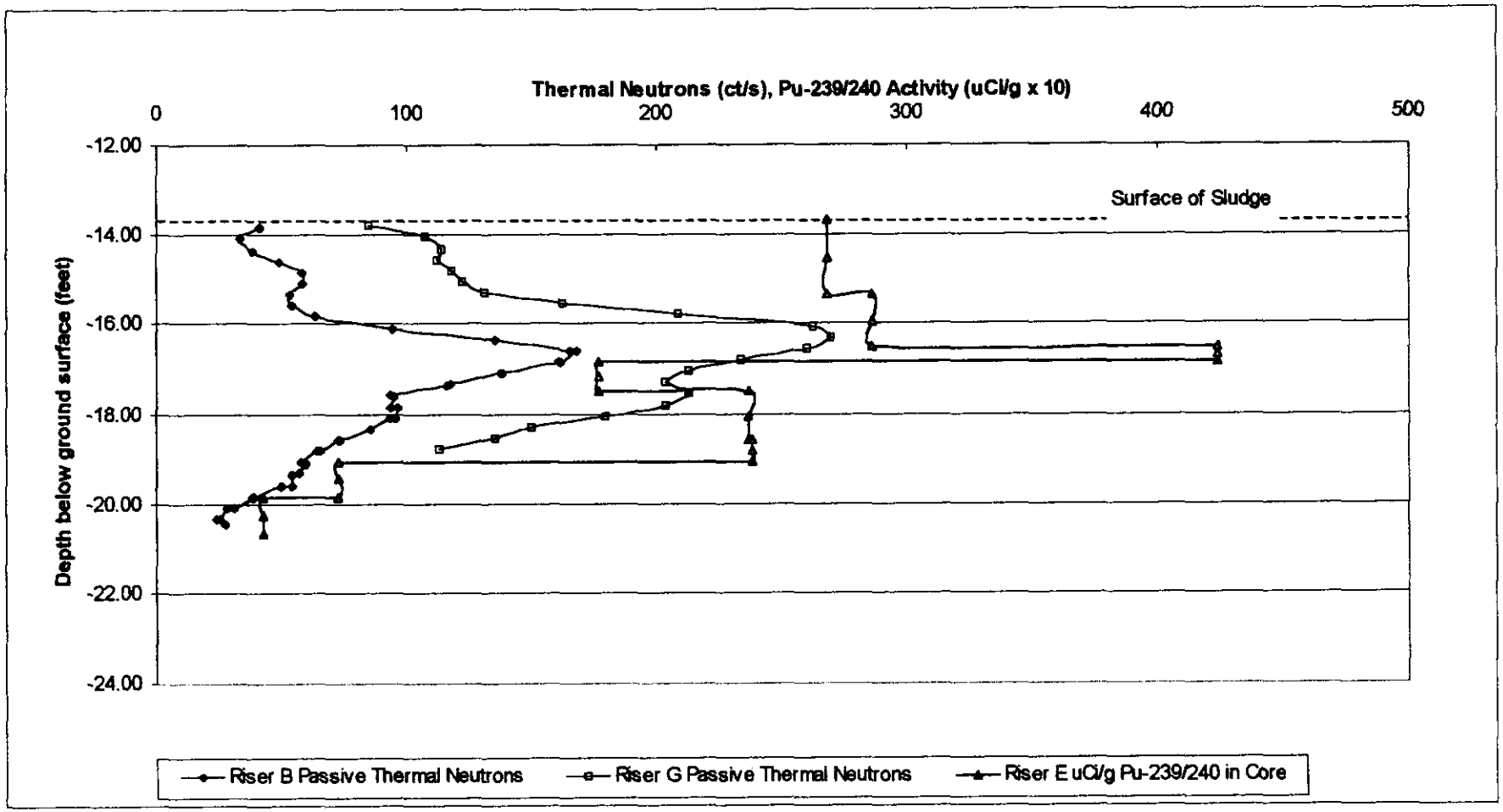


Table 3-60. Tabular Data for Figure 3-19[1] (2 sheets)

NDA Data				Laboratory Data[2]		
Depth From Ground Level Riser B (ft)	Riser B NDA Passive Neutron Gross Activity (cps)	Depth From Ground Level Riser G (ft)	Riser G NDA Passive Neutron Gross Activity (cps)	Sludge Composite Sample	Depth From Ground Level Riser E (ft)	Pu-239/240 Laboratory Result (µCi/g)
--	--	--	--	263-1	-13.67	26.8
-13.84	41	-13.80	85	263-1	-14.50	26.8
-14.09	33	-14.05	107	263-1	-15.34	26.8
-14.34	38	-14.30	114	263-2	-15.35	28.6
-14.59	49	-14.55	112	263-2	-15.93	28.6
-14.84	58	-14.80	118	263-2	-16.52	28.6
-15.09	58	-15.05	123	263-3	-16.53	42.4
-15.34	53	-15.30	131	263-3	-16.68	42.4
-15.59	54	-15.55	163	263-3	-16.84	42.4
-15.84	64	-15.80	209	263-4	-16.85	17.7
-16.09	94	-16.05	263	263-4	-17.17	17.7
-16.34	135	-16.30	270	263-4	-17.50	17.7
-16.59	166	-16.55	260	263-5	-17.51	23.7
-16.60	168	-16.80	233	263-5	-18.05	23.7
-16.84	162	-17.05	213	263-5	-18.59	23.7
-16.85	161	-17.30	204	263-6	-18.60	23.8
-17.09	138	-17.55	213	263-6	-18.84	23.8
-17.10	138	-17.80	204	263-6	-19.09	23.8
-17.34	117	-18.05	180	263-7	-19.10	7.26
-17.35	116	-18.30	151	263-7	-19.46	7.26
-17.59	94	-18.55	135	263-7	-19.84	7.26
-17.60	95	-18.80	113	263-8	-19.85	4.21
-17.84	94	--	--	263-8	-20.25	4.21
-17.85	96	--	--	263-8	-20.67	4.21
-18.09	96	--	--	--	--	--
-18.10	94	--	--	--	--	--
-18.34	86	--	--	--	--	--
-18.35	85	--	--	--	--	--
-18.59	74	--	--	--	--	--
-18.60	73	--	--	--	--	--
-18.84	66	--	--	--	--	--
-18.85	64	--	--	--	--	--

Table 3-60. Tabular Data for Figure 3-19[1] (2 sheets)

NDA Data				Laboratory Data[2]		
Depth From Ground Level Riser B (ft)	Riser B NDA Passive Neutron Gross Activity (cps)	Depth From Ground Level Riser G (ft)	Riser G NDA Passive Neutron Gross Activity (cps)	Sludge Composite Sample	Depth From Ground Level Riser E (ft)	Pu-239/240 Laboratory Result (µCi/g)
-19.09	58	--	--	--	--	--
-19.10	60	--	--	--	--	--
-19.34	57	--	--	--	--	--
-19.35	54	--	--	--	--	--
-19.59	54	--	--	--	--	--
-19.60	50	--	--	--	--	--
-19.84	38	--	--	--	--	--
-19.85	39	--	--	--	--	--
-20.09	31	--	--	--	--	--
-20.10	28	--	--	--	--	--
-20.34	25	--	--	--	--	--
-20.35	24	--	--	--	--	--
-20.46	27	--	--	--	--	--

1. Blanks in table reflect the fact that there are different numbers of data points recorded.
2. For purposes of graphing, the result of a particular sludge composite sample were repeated three times. Once at the top of the composite interval, at the midpoint, and at the bottom.

3.8.3.6 Spectral Gamma Determination of Neptunium-237

The NDA measurement of neptunium-237 was performed and reported in Riser G at a series of depths near the peak americium-241 location (about 15 ft, Figure 3-18). The NDA results ranged from 1669 to over 6000 pCi/g. Multiple NDA measurements were performed at depths of 14.4 ft to 15.92 ft. The average of these results was 4,226 pCi/g. These depths correspond to sludge composite sample 263-2 from Riser E and sludge composite sample 264-2 from Riser F. The laboratory data reported a result of 2,930 pCi/g for composite 263-2 and 5,230 pCi/g for composite 264-2. Both reported results were less than detection limits and both results had greater than 75% counting error reported with them. The level of agreement between the NDA and laboratory data is excellent. Future counting with a better shield design and longer counts, if needed, should provide higher quality data.

3.8.3.7 Conclusions

The data acquisition related to this tank is difficult when compared to the normal well logging performed by Waste Management Technical Services (now Duratek Technical Services). The material is settled sludge from the waste stream of the PFP facility and is not similar to the characteristic soil conditions encountered in the groundwater wells, boreholes associated with cribs, ditches, and ponds, or soil surrounding tank farm dry wells.

The Tank 241-Z-361 tank conditions require special calibrations for all of the detector systems. The neutron-moisture probe data were not presented as calibrated data due to the size and type of casing material and the high moisture content of the sludge. Monte Carlo computer calculations are required to extrapolate from the physical conditions in the calibration standards to the conditions in the tank and were not done due to funding limitations. The small diameter (Mount Sopris) logging tool, that was utilized to acquire gamma activity measurements, is used for gross gamma only, because it was not calibrated.

The wafer HPGe spectral gamma detector was calibrated, but most of the borehole corrections were derived from ratios of steel to aluminum mass attenuation coefficients. The errors in the concentration data may be largely due to the high gamma intensity from the 60 KeV americium signal that caused system dead time to be higher than normal limits and masked photo peaks of other radionuclides.

Sufficient data regarding the tank contents have been acquired to adequately configure suitable detectors to perform manmade radionuclide assays and potentially perform elemental analysis using neutron capture/ spectral gamma logging methods. The NDA measurements appear to have the capability of measuring neutron flux as a measure of spontaneous fission. In addition, the spectral gamma measurements of americium-241, plutonium-239, and neptunium-237 content of the sludge provide an indication that these measurement methods may be valuable in determining content of several key radionuclides in the sludge without sampling.

3.9 SLUDGE ANALYTICAL DATA EVALUATION

3.9.1 Chemical Constituents Concentration and Distribution

Two full-depth core samples were collected from risers in the tank. Multiple strata were detected in the vertical profile of the cores. Compositing of vertical layers of the cores for fixed laboratory analysis was performed based on similarities found in alpha activity results, as well as color and texture of the cores. The results for the sludge composite samples for each analyte are presented in this section. Results are presented for two suites, radionuclides and metals. For each analyte, three-dimensional plots are presented to show the spatial distribution of the results for both cores. Depths on the plot represent the average depth for each interval over which a composite was taken. The sludge occupies the lower third of the tank, and the three-dimensional plots represent the portion of the tank which the sludge occupies. Hence, the top of each plot represents the surface of the sludge.

Exploratory data analysis (EDA) plots are presented for each analyte as well. For each analyte, the EDA plot consists of a histogram, boxplot, density estimate, and normal-normal quantile plot. Histograms plot the frequency of observations within consecutive, equally sized intervals of concentrations/activities. They provide a discrete estimate of the shape of the distribution of the data from the composite sampling. Boxplots are another method of representing the distribution. The top and bottom of the box in the boxplot represent the inter-quartile range (IQR), identified by the 75th and 25th percentiles of the data, respectively. The horizontal line in the middle of the box represents the 50th percentile (the median). Vertical lines (called whiskers) extend to the most extreme data point which is no more than 1.5*IQR from the box. "Outliers" are represented on box plots by dots beyond the whiskers. The density estimate provides a continuous approximation of the probability distribution for the analytes from the composite samples. It is a complementary plot to the histogram. The normal-normal quantile plot pairs the sorted data with the corresponding quantiles of the standard normal distribution. If the data are normally distributed, then the plotted pairs will follow a straight line.

These EDA plots combine to provide a qualitative assessment of the form of the distribution. All of these plots are used together to assess possibilities for the construction of a statistical model for the distribution of each analyte. EDA plots are useful for identifying properties of the distribution that may guide the assessment of the distributional form. One property of interest is skew. Any distribution that is not symmetric about the mean is skewed. Another way of saying this is that, if the mean does not equal the median of a distribution, then the distribution is skewed. If the mean is greater than the median, the distribution is said to be right-skewed. On the other hand, if the mean is less than the median, the distribution is said to be left-skewed. Right-skew is a common property of many environmental data sets due to the presence of several elevated concentrations. If the skew is significant, it can change the possible distributional model that may be constructed for a data set.

3.9.2 Radionuclide Concentration and Distribution

Summary statistics for each of the analytes are presented in Table 3-61. The data represents a pooling of all core data without regard to depth or core distinctions. The minimum, median, mean, standard deviation, relative standard deviation, and relative standard error of the mean were calculated. The relative standard deviation was calculated as the standard deviation divided by the mean. This provides a measure of the spread of the distribution relative to the analyte-specific mean concentration. The relative standard error was calculated as the standard error divided by the mean (i.e., the standard deviation divided by the square root of the number of samples divided by the mean). This provides a measure of the spread of the sample mean's distribution relative to the analyte-specific mean concentration. Results from analysis of the core samples for radionuclides are presented in the following analyses. Radiological results are presented for gross alpha, americium-241, gross beta, neptunium-237, plutonium-238, plutonium-239, plutonium-239/240, plutonium-240, plutonium-241, plutonium/americium-241, strontium-89/90, technetium-99, uranium, uranium-235, and uranium-238.

Table 3-61. Summary Statistics for Pooled Radiological Data from All Sludge Composite Samples

Analyte	Units	Detect	N	Minimum	Median	Mean	Maximum	Std. Dev.	Relative Std. Dev.	Relative Std. Error of Mean
Americium-241	Ci/g	D	13	0.306	4.81	6.42	14.1	4.8	0.747	0.207
Gross Alpha	Ci/g	D	13	3.87	25.7	24.2	42.6	12.9	0.531	0.147
Gross Beta	Ci/g	D	13	0.146	2.04	1.71	3.96	1.06	0.617	0.171
Neptunium-237	Ci/g	D	4	0.00293	0.00364	0.00386	0.00523	0.0011	0.284	0.142
	Ci/g	N	9	0.00196	0.00225	0.00326	0.00614	0.00171	0.524	0.175
Plutonium-238	Ci/g	D	1	2.3	2.3	2.3	2.3	NA	NA	NA
	Ci/g	N	12	0.201	1.13	1.12	2.25	0.64	0.569	0.164
Plutonium-239/240	Ci/g	D	13	4.21	24.6	20.9	42.4	11.3	0.539	0.15
Plutonium-239	g/g	D	13	69.1	331	299	578	152	0.509	0.141
Plutonium-240	g/g	D	5	25.5	28.5	29.8	37.2	4.48	0.15	0.0672
	g/g	N	8	23.8	24.4	24.3	24.7	0.359	0.0148	0.00522
Plutonium-241*	g/g	NA	8	0.332	0.506	0.521	0.712	0.183	0.352	0.124
Plutonium/ Americium-241	g/g	N	13	23.4	24.4	24.2	24.7	0.444	0.0183	0.00508
Strontium-89, 90	Ci/g	D	13	0.00095	0.00709	0.0124	0.044	0.0127	1.03	0.285
Technetium-99	Ci/g	D	12	0.00148	0.0142	0.014	0.0279	0.0084	0.6	0.173
	Ci/g	N	1	0.00149	0.00149	0.00149	0.00149	NA	NA	NA
Uranium	g/g	D	3	112	134	130	142	15.6	0.121	0.0696
	g/g	N	10	49	98.9	95.4	201	44.1	0.462	0.146
Uranium-235	g/g	N	13	18.7	19.5	19.4	19.7	0.354	0.0183	0.00507
Uranium-238	g/g	D	12	20.8	29.3	33.7	62.6	14.8	0.438	0.127
	g/g	N	1	19.7	19.7	19.7	19.7	NA	NA	NA

*Results are estimated from plutonium/americium-241 results.

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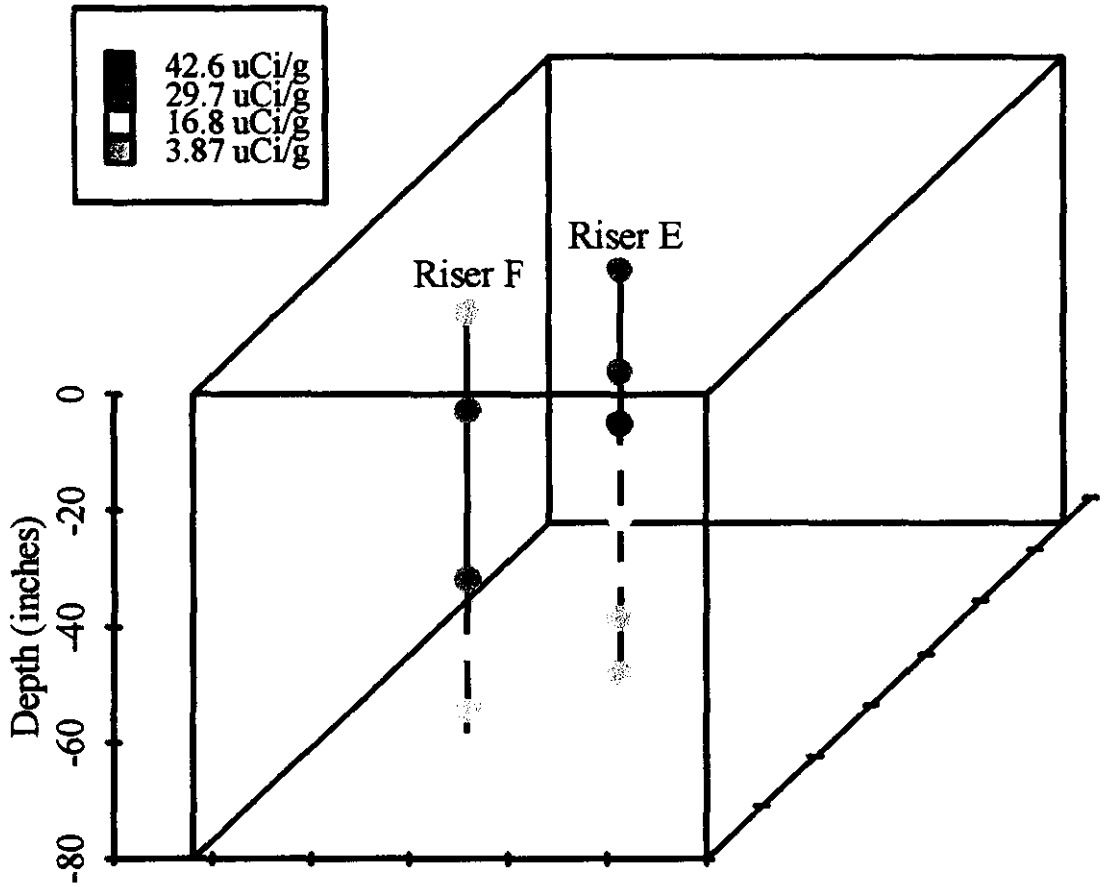
3.9.2.1 Gross Alpha

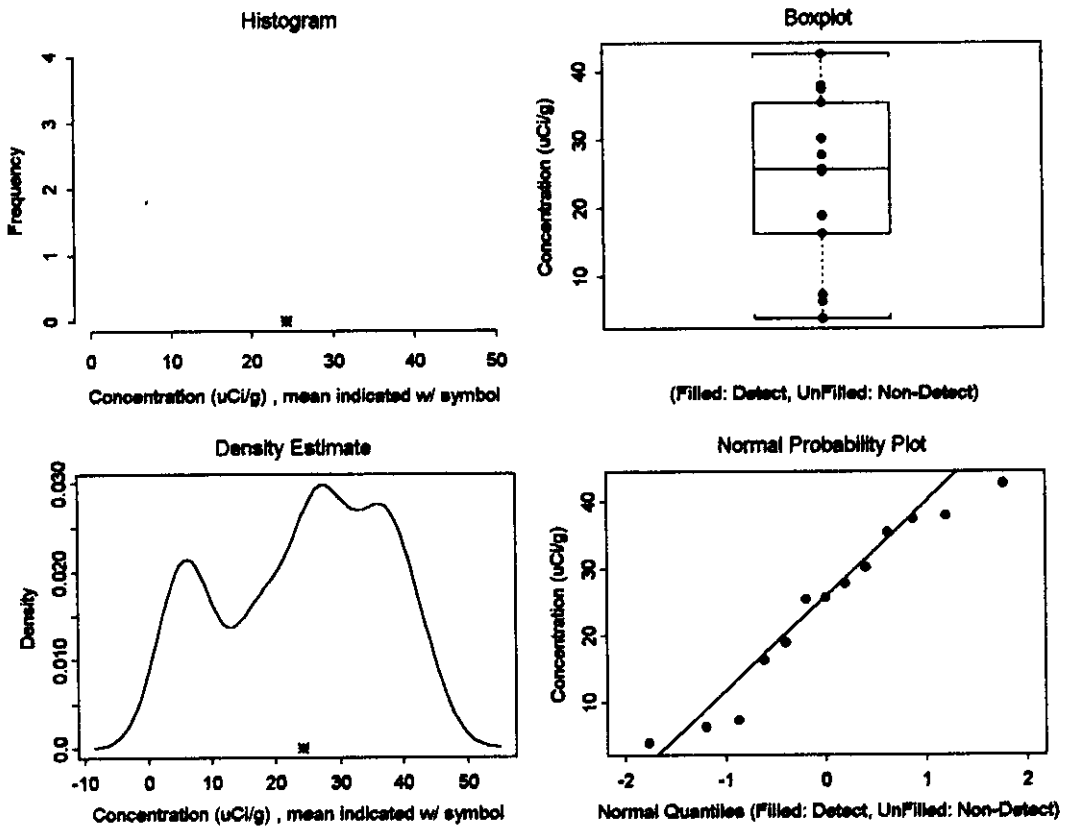
All results for gross alpha are detected values. Activities ranged from 3.87 μCi/g to 42.60 μCi/g. Figure 3-20 shows that for both cores, the lowest activities are found in the composite samples taken from the bottom of the tank. The highest activity of gross alpha is from the composite sample taken at the depths 34.25 to 38 in from Riser E. The lowest result is from the composite sample taken at the depths 74 to 84 in from Riser E.

Figure 3-20. Composite Sample Results for Gross Alpha



The exploratory data plots in Figure 3-21 show that the distribution of the composite samples for gross alpha is somewhat symmetric with respect to the mean. The distribution is approximately normal and there are no data values that seem to be outliers.

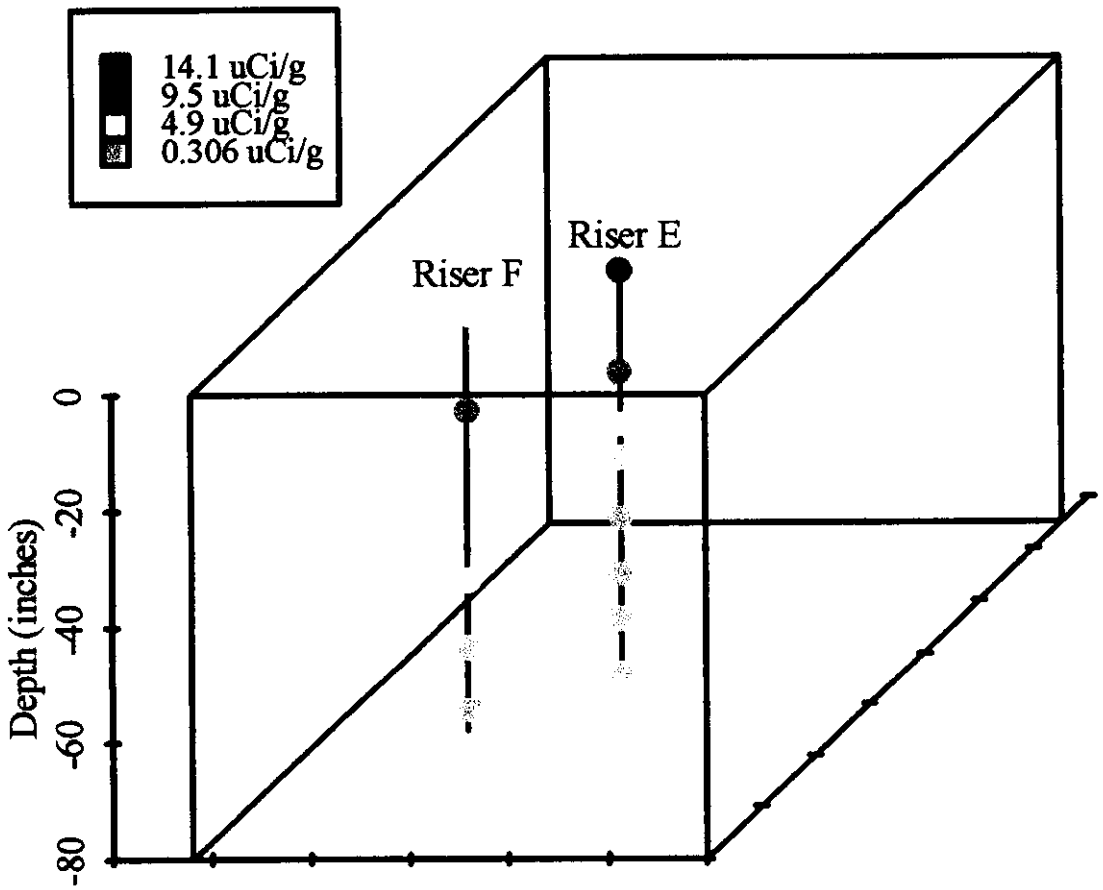
Figure 3-21. Exploratory Data Analysis for Gross Alpha



3.9.2.2 Americium-241

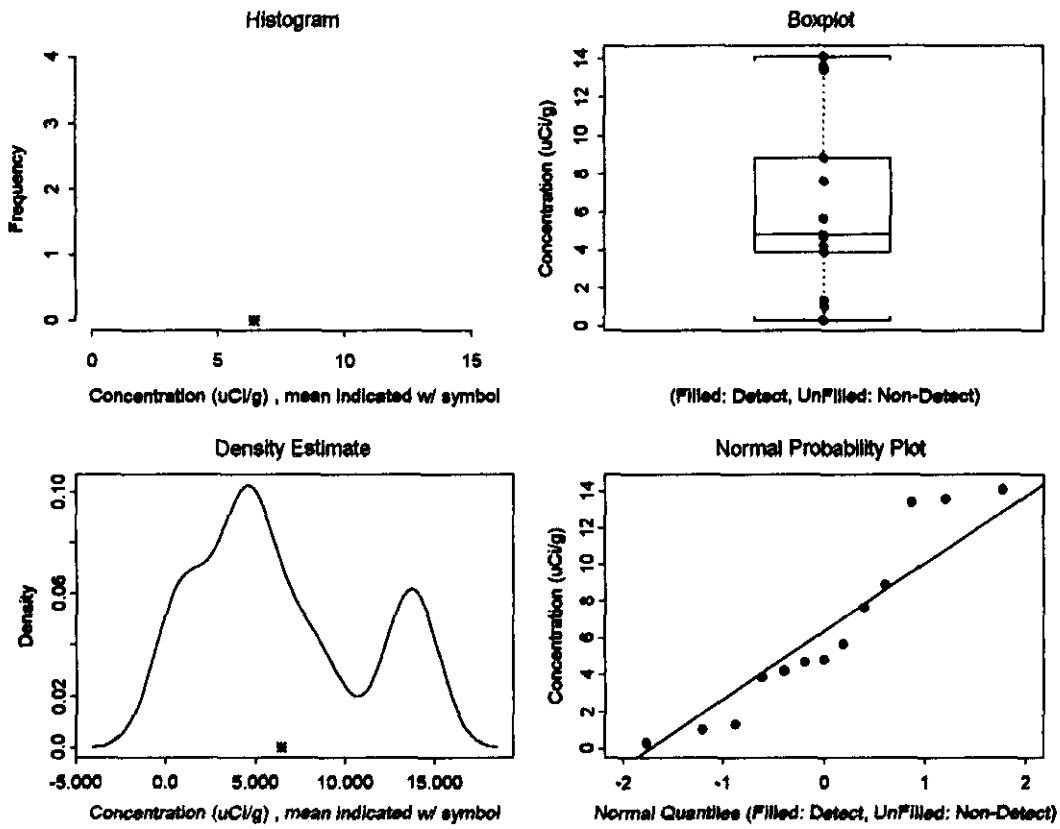
All results for americium-241 are detected values. Activities for americium-241 range from 0.306 μCi/g to 14.1 μCi/g. Figure 3-22 shows that most of the americium-241 in the two cores appears to be in the upper two-thirds of the sludge. The core samples from Riser E show a trend of increasing activities toward the surface of the sludge. The maximum result occurs in the composite sample taken from depths 0 to 20 in from Riser E. The maximum result from Riser F occurs at the second sample from the surface of the sludge. There is a weak trend of decreasing results with increasing depth of the core sample.

Figure 3-22. Composite Sample Results for Americium-241



The exploratory data plots in Figure 3-23 show that the distribution of the composite samples for americium-241 is possibly bimodal. This is not surprising given the distinct fluxes of heterogeneous wastes that were sent through the tank. The use of a normal model for the distribution of americium-241 in the tank is questionable.

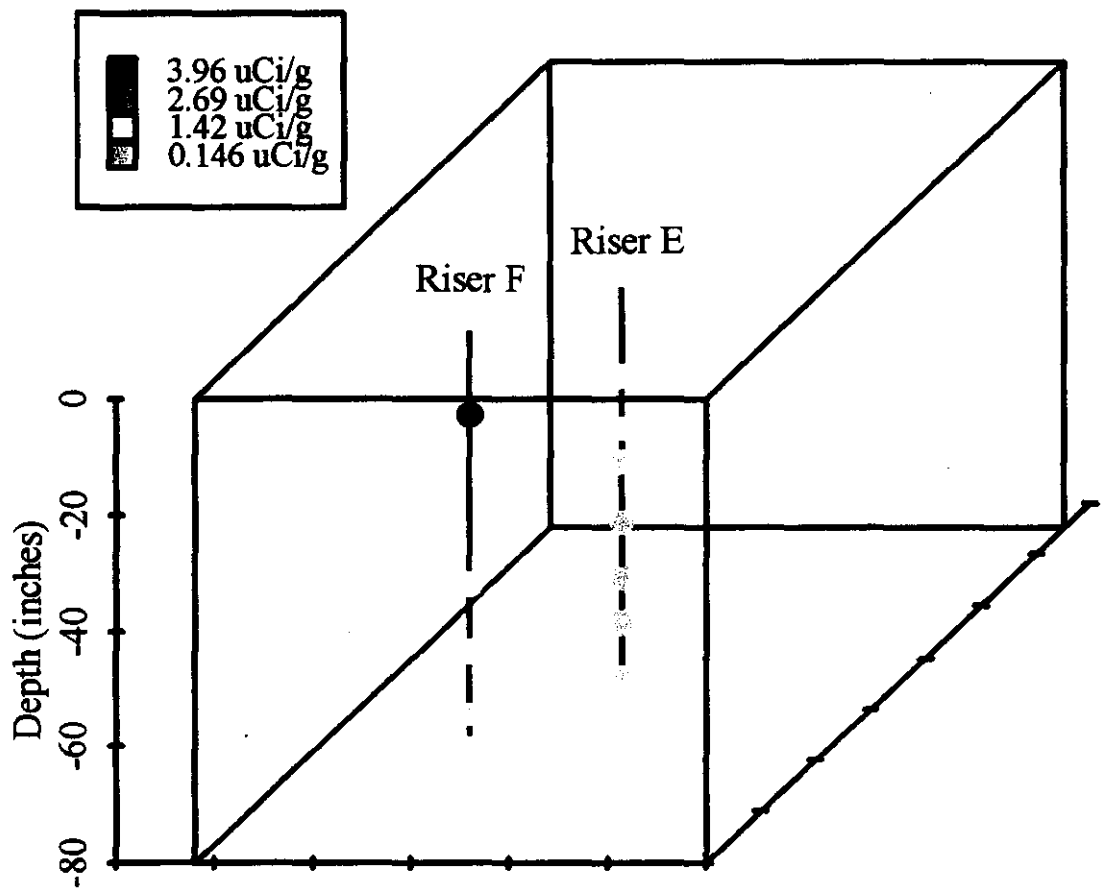
Figure 3-23. Exploratory Data Analysis for Americium-241



3.9.2.3 Gross Beta

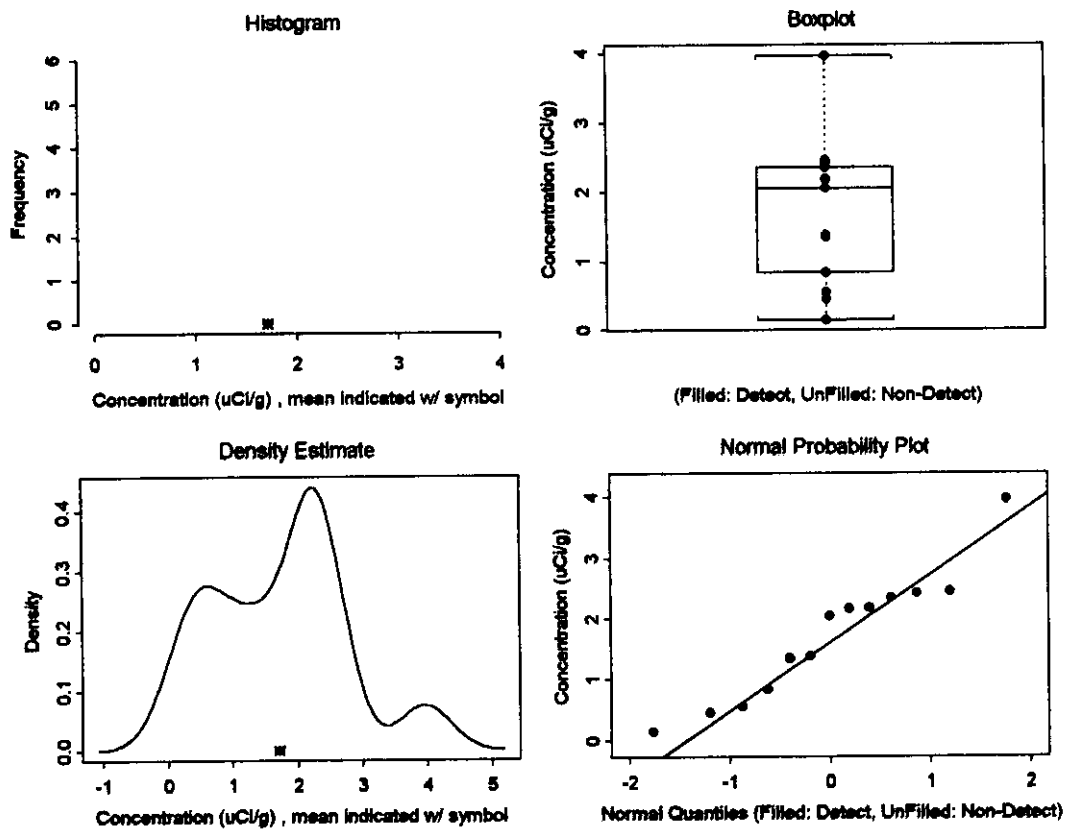
All results for gross beta are detected values. Activities for gross beta ranged from 0.146 μCi/g to 3.960 μCi/g. Figure 3-24 shows that for both cores, the highest activities are found in composites taken from the middle of the cores. The lowest activities for both risers are found in the composite samples taken from the bottom of the tank. The maximum value is found in Riser F at the composite depth of 16.5 to 33.5 in The minimum result is found in Riser E at the composite depth of 74 to 84 in.

Figure 3-24. Composite Sample Results for Gross Beta



The exploratory data plots in Figure 3-25 show that the distribution of the gross beta composite samples is approximately normal and there are no data values that seem to be outliers.

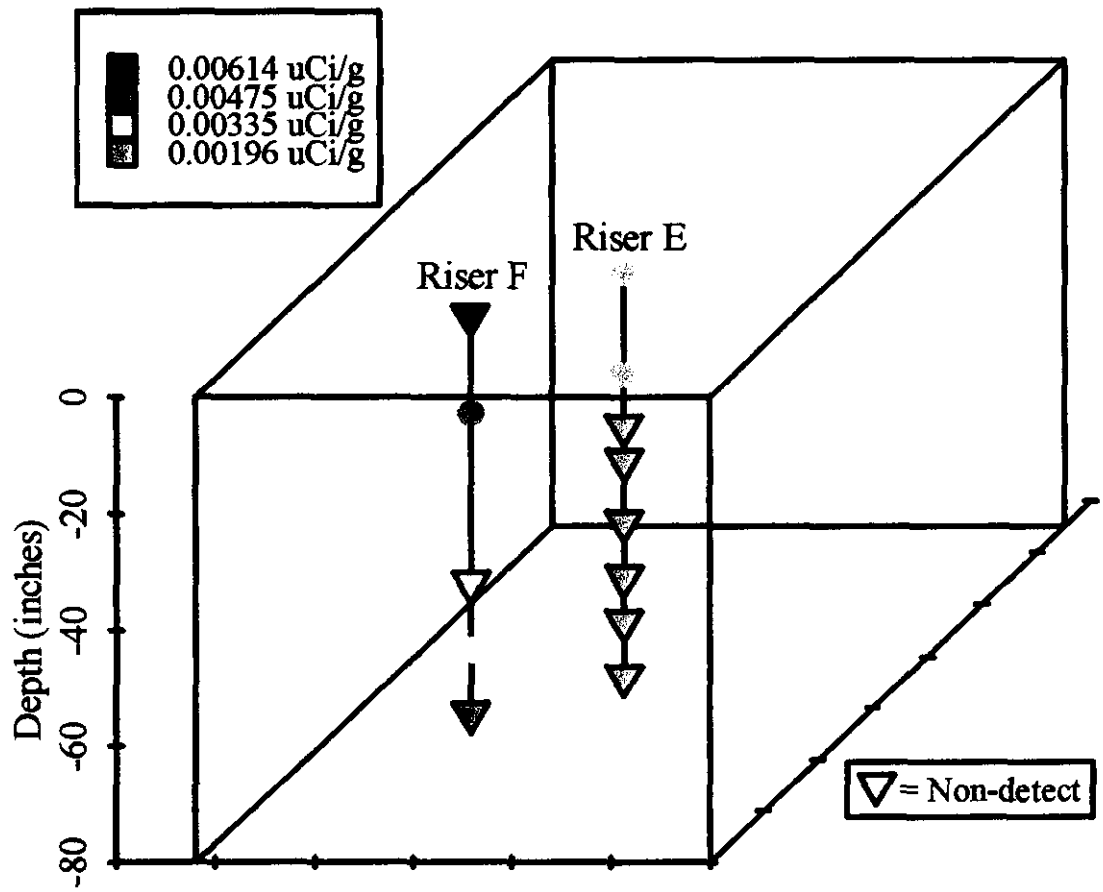
Figure 3-25. Exploratory Data Analysis for Gross Beta



3.9.2.4 Neptunium-237

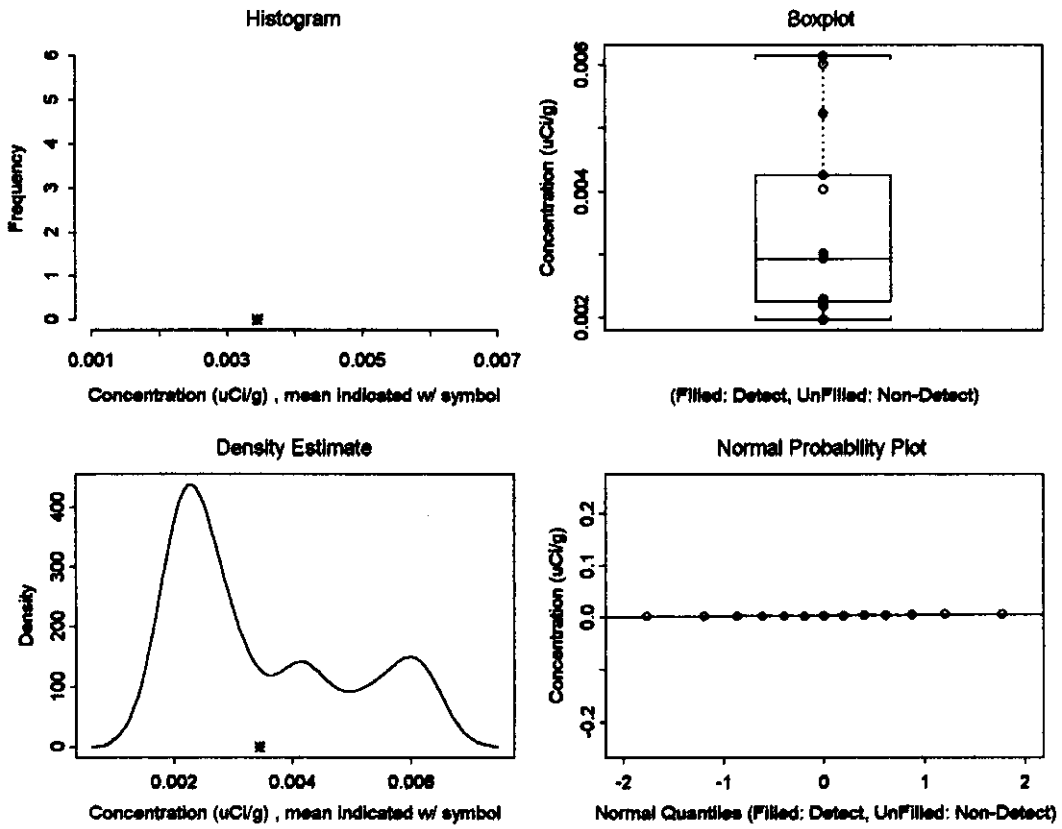
As shown in Figure 3-26, four of the 13 results for neptunium-237 are detected values. Activities for the detected values range from 0.0029 µCi/g to 0.0052 µCi/g. Detection limits for the nondetects range from 0.002 µCi/g to 0.0061 µCi/g.

Figure 3-26. Composite Sample Results for Neptunium-237



The exploratory data plots are shown in Figure 3-27. However, because only four of the results for neptunium-237 are detects, it is difficult to make any assertions about the distribution of the data from the composite samples.

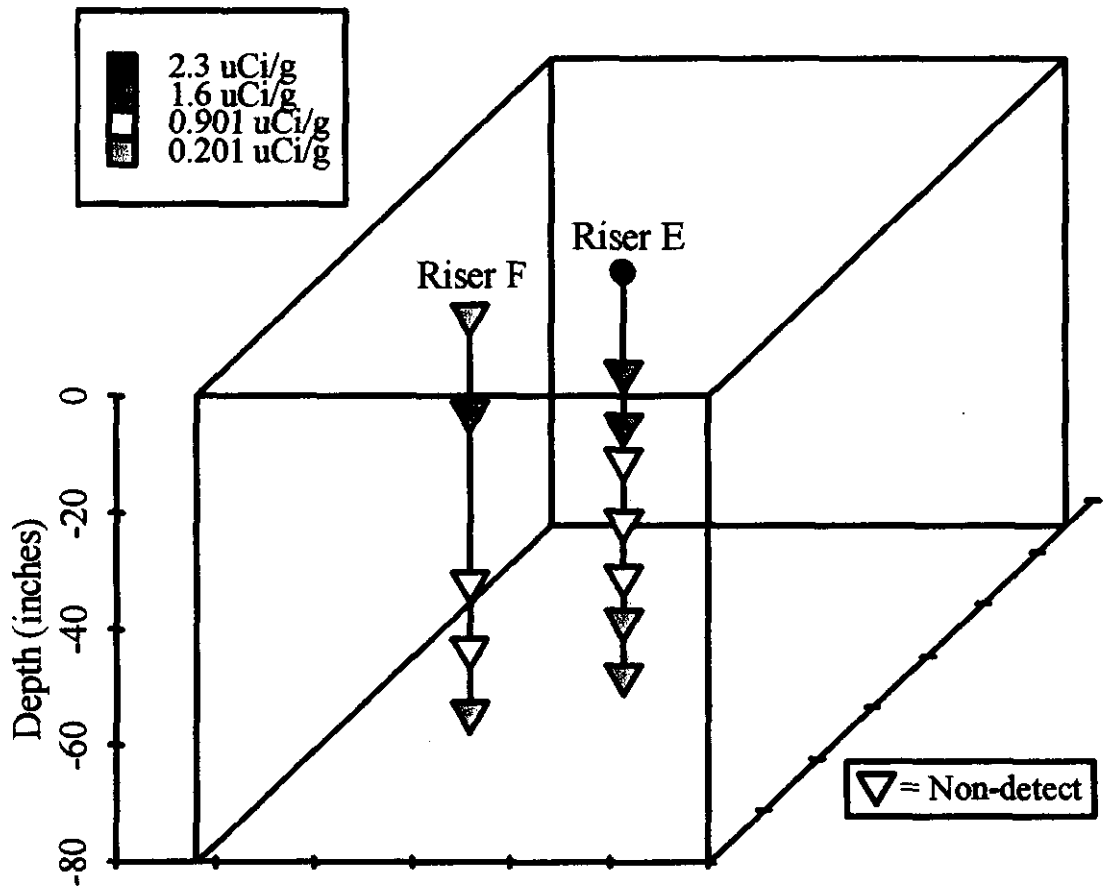
Figure 3-27. Exploratory Data Analysis for Neptunium-237



3.9.2.5 Plutonium-238

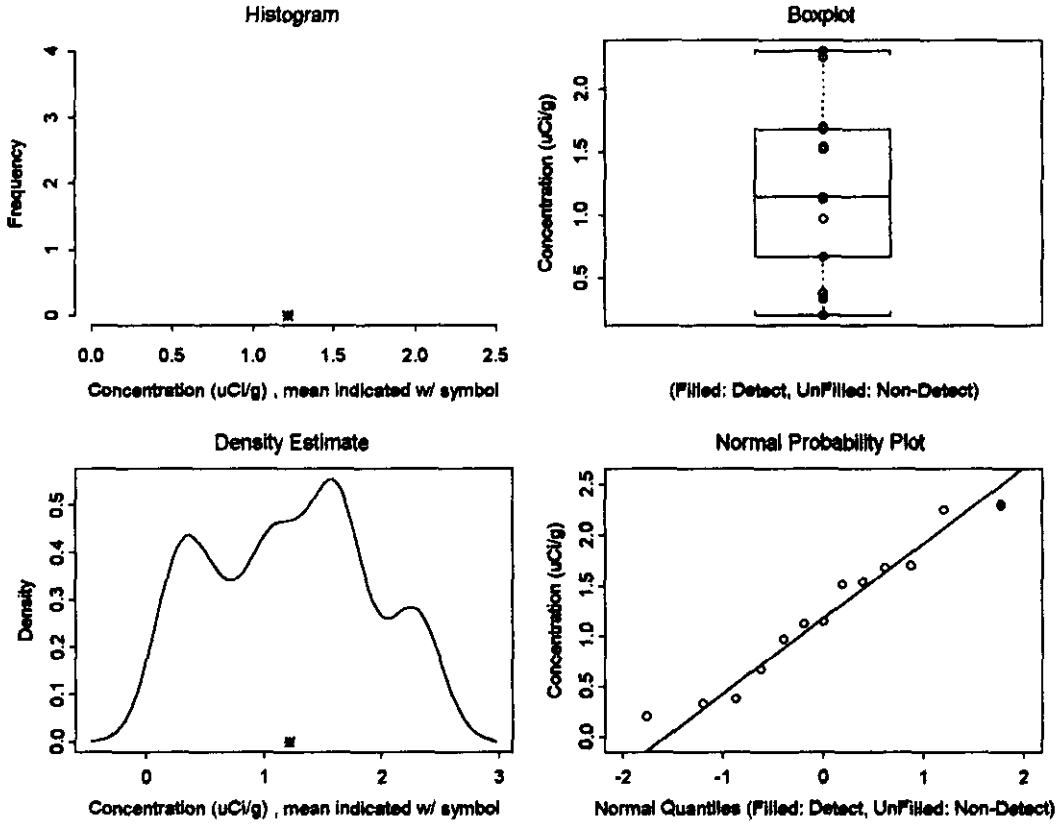
As shown in Figure 3-28, there is only one detected result for plutonium-238. It is an activity of 2.3 μCi/g found in Riser E in the composite taken from 0 to 20 in.

Figure 3-28. Composite Sample Results for Plutonium-238



The exploratory data plots are shown in Figure 3-29. However, because only one of the results for plutonium-238 is detected, it is difficult to make any assertions about the distribution of the data from the composite samples.

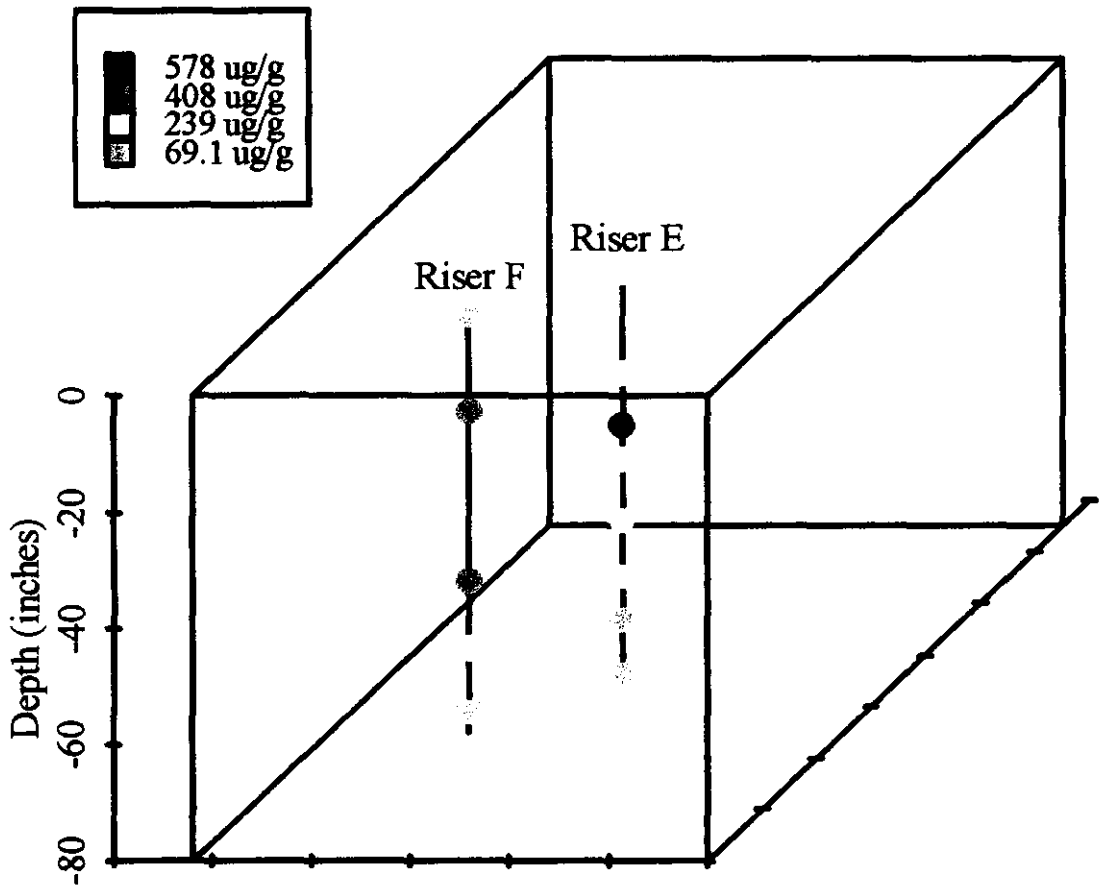
Figure 3-29. Exploratory Data Analysis for Plutonium-238



3.9.2.6 Plutonium-239 by ICP/MS-Fusion

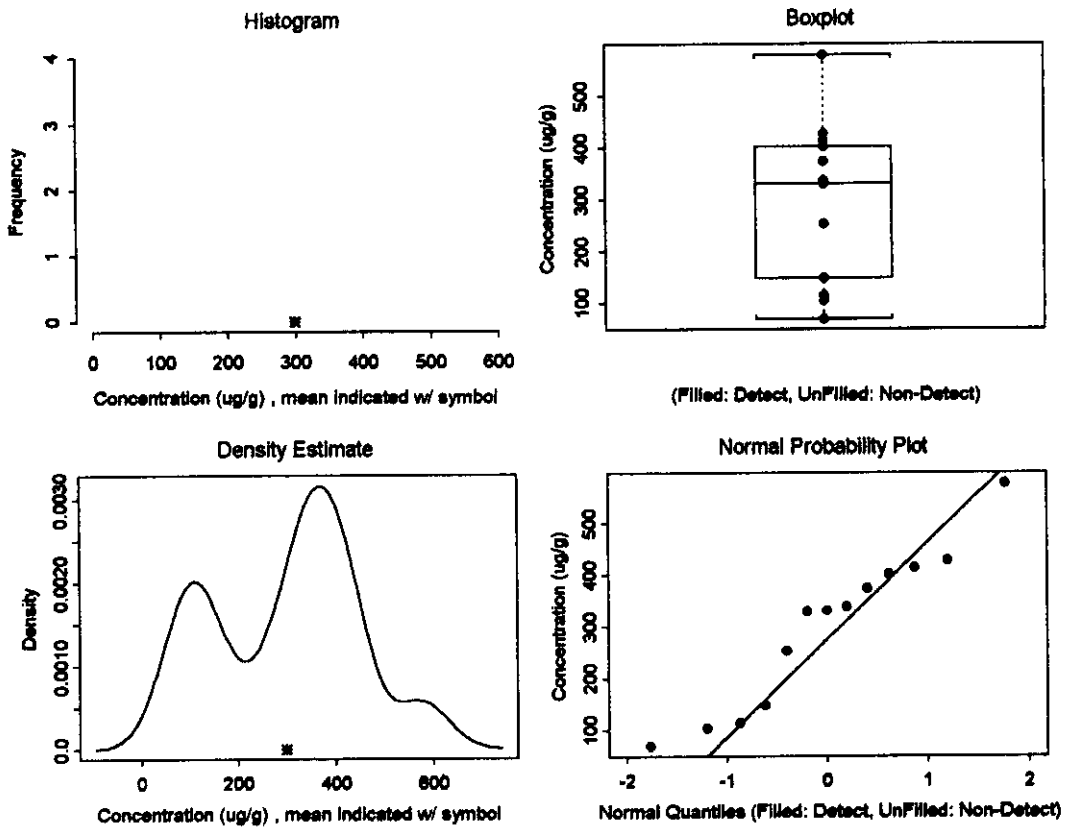
All results for plutonium-239 are detected values. The concentrations for plutonium-239 range from 69.1 μg/g to 578.0 μg/g. Figure 3-30 shows that for both cores, the highest activities are found in composites taken from the middle of the cores. The highest concentration is from the composite sample taken at the depths 34.25 to 38 in from Riser E. The lowest result is from the composite sample taken closest to the bottom of the tank.

Figure 3-30. Composite Sample Results for Plutonium-239 by ICP/MS-Fusion



The exploratory data plots in Figure 3-31 show that the distribution of the concentrations for plutonium-239 is slightly skewed to the left. The distribution is approximately normal and there are no data values that seem to be outliers.

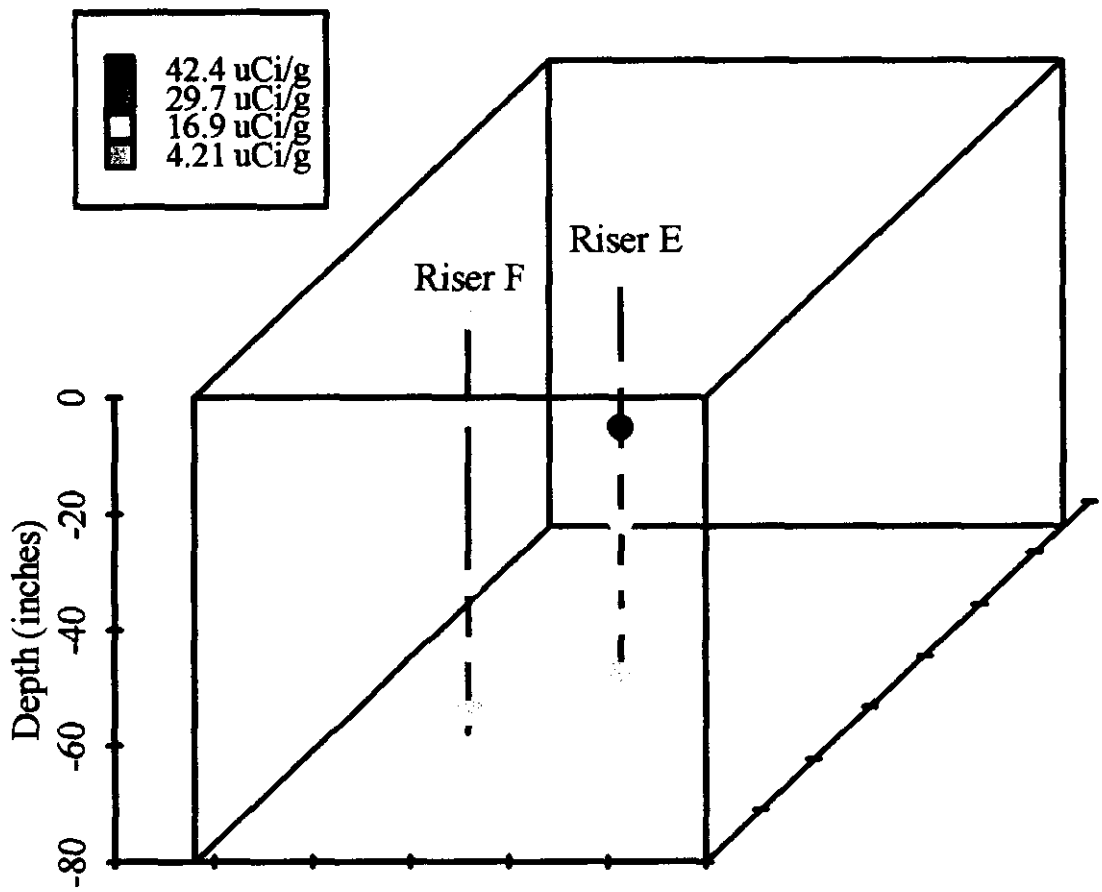
Figure 3-31. Exploratory Data Analysis for Plutonium-239 by ICP/MS-Fusion



3.9.2.7 Plutonium-239/240 by AEA

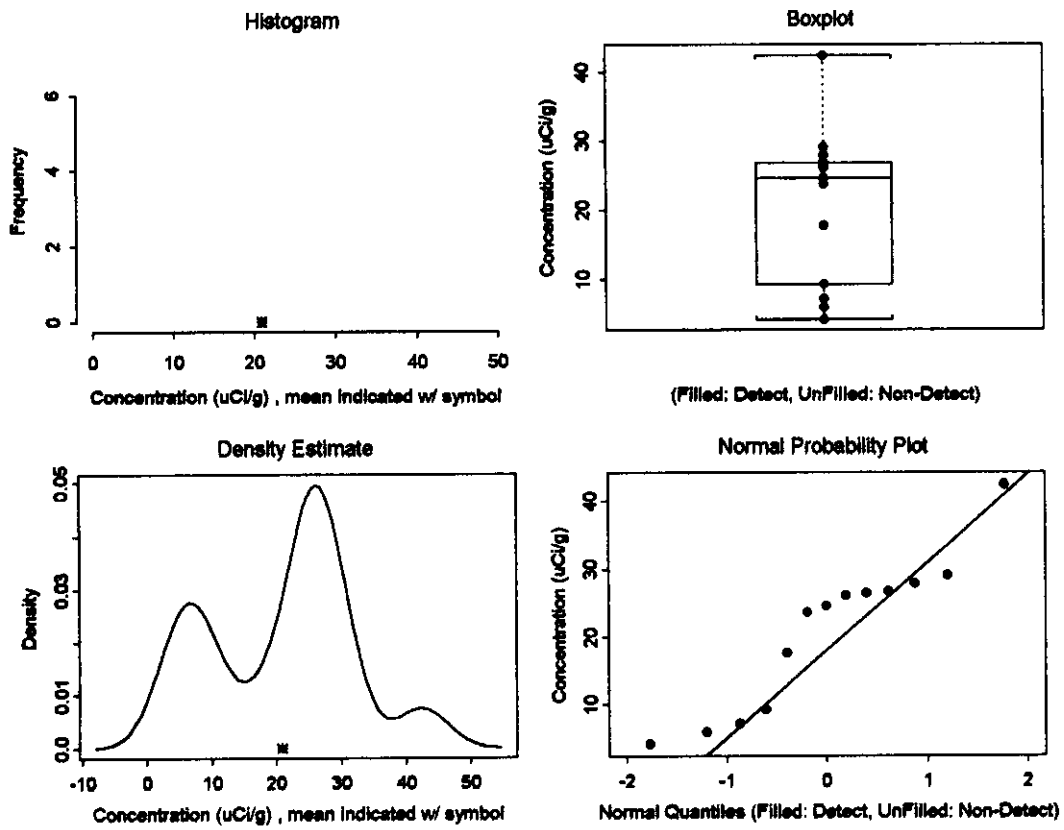
All results for plutonium-239/240 are detected values. The activities for plutonium-239/240 range from 4.21 μCi/g to 42.4 μCi/g. Figure 3-32 shows that for Riser F, the highest activities are found in the composites taken from the middle of the sludge and for Riser E, the highest activities are found in the top half of the core. Although the magnitude of results from Riser E is larger than those from Riser F, the cores' concentrations seem to be correlated, relative to depth. The highest activity is from the composite sample taken at the depths 34.25 to 38 in from the top of the sludge at Riser E. The lowest activities are from the composite samples taken near the bottom of the tank.

Figure 3-32. Composite Sample Results for Plutonium-239/240 by AEA



The exploratory data plots in Figure 3-33 show that the distribution of the concentrations for plutonium-239/240 is slightly skewed to the left and possibly bimodal. There are no data values that seem to be outliers.

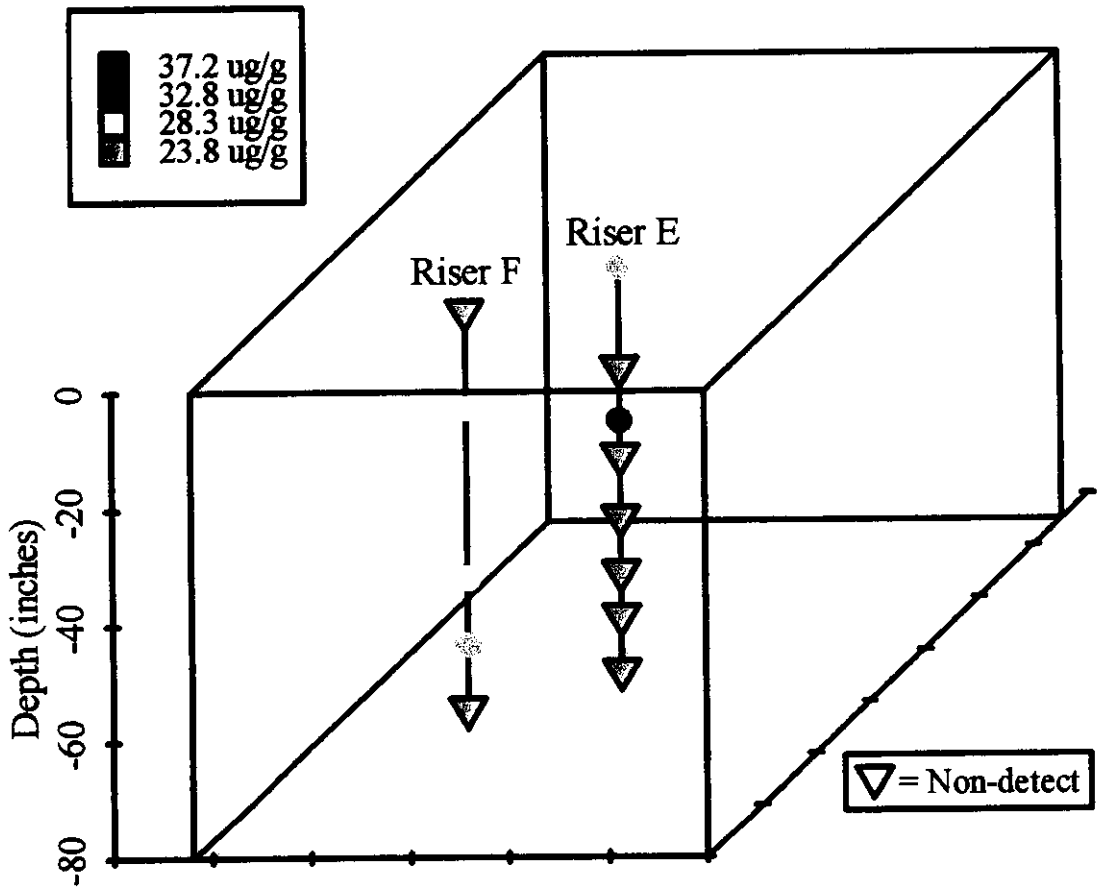
Figure 3-33. Exploratory Data Analysis for Plutonium-239/240 by AEA



3.9.2.8 Plutonium-240 by ICP/MS-Fusion

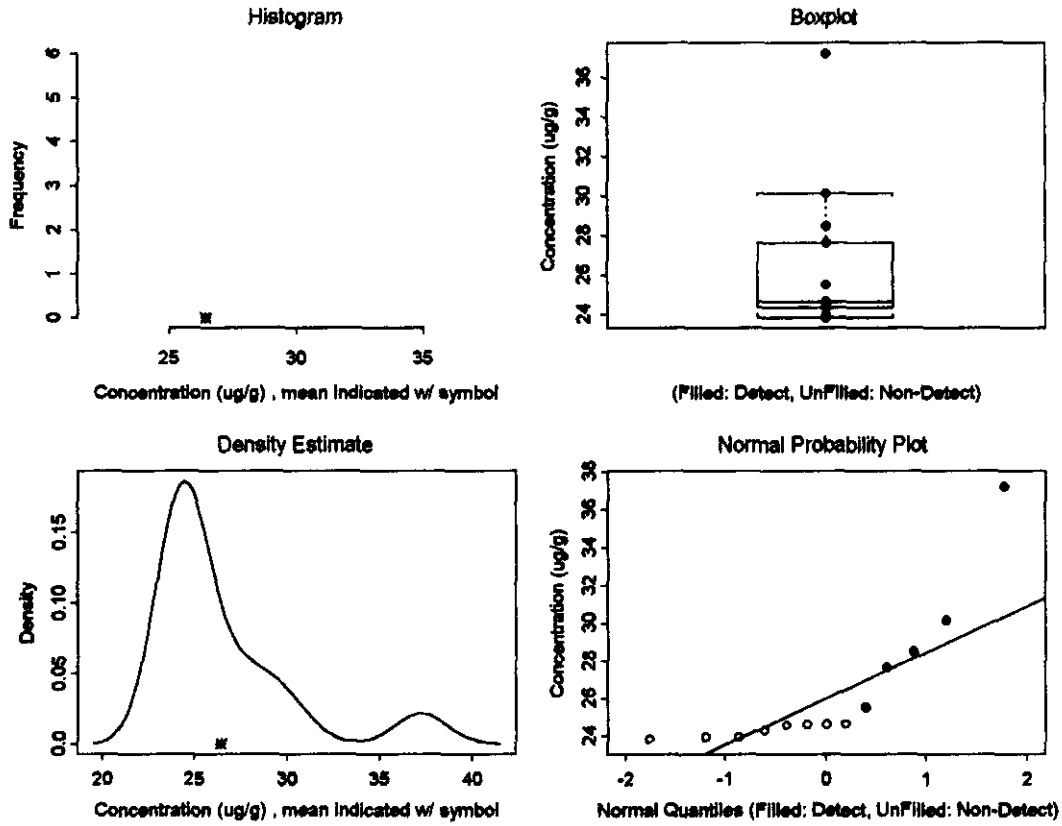
As shown in Figure 3-34, five of the 13 results for plutonium-240 are detected values. Concentrations for the detected values range from 25.5 µg/g to 37.23 µg/g. Detection limits for the nondetects range from 23.8 µg/g to 24.7 µg/g.

Figure 3-34. Composite Sample Results for Plutonium-240 by ICP/MS-Fusion



The exploratory data plots are shown in Figure 3-35. However, because only five of the results for plutonium-240 are detects, it is difficult to make any assertions about the distribution of the data from the composite samples.

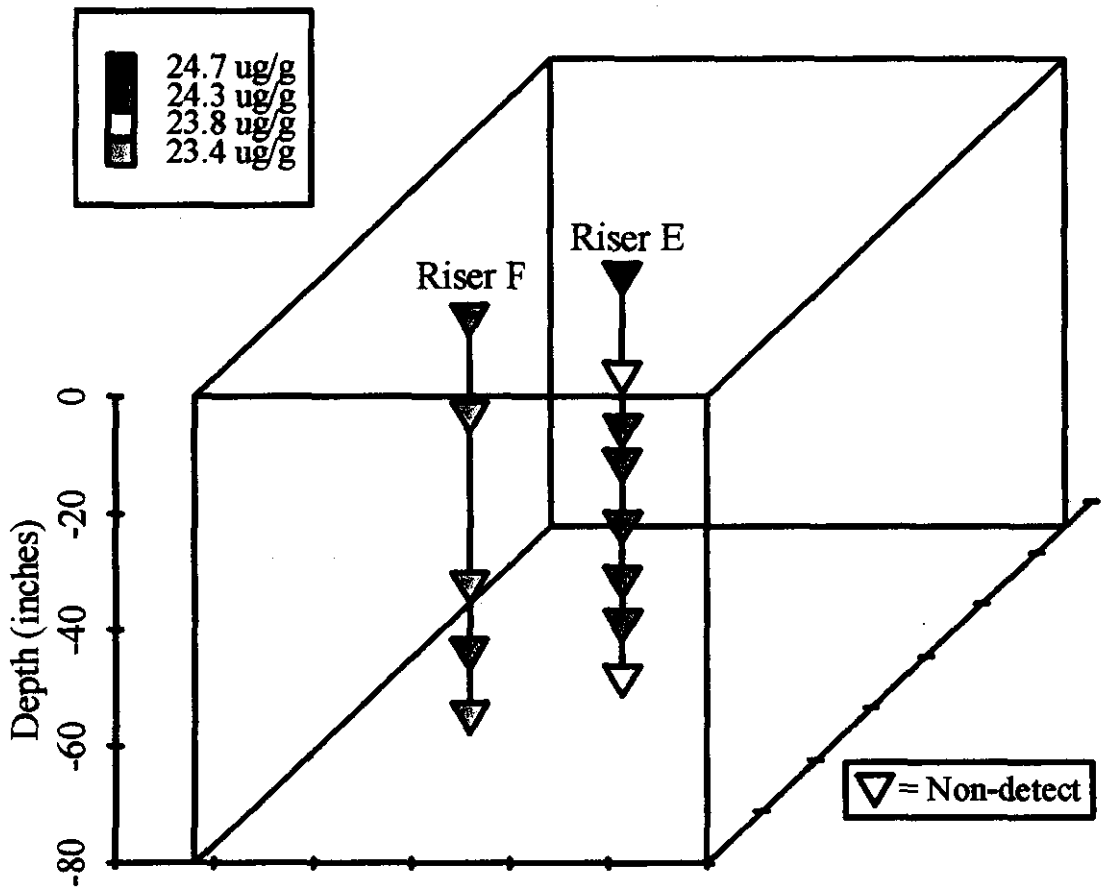
Figure 3-35. Exploratory Data Analysis for Plutonium-240 by ICP/MS-Fusion



3.9.2.9 Plutonium/Americium-241 by ICP/MS

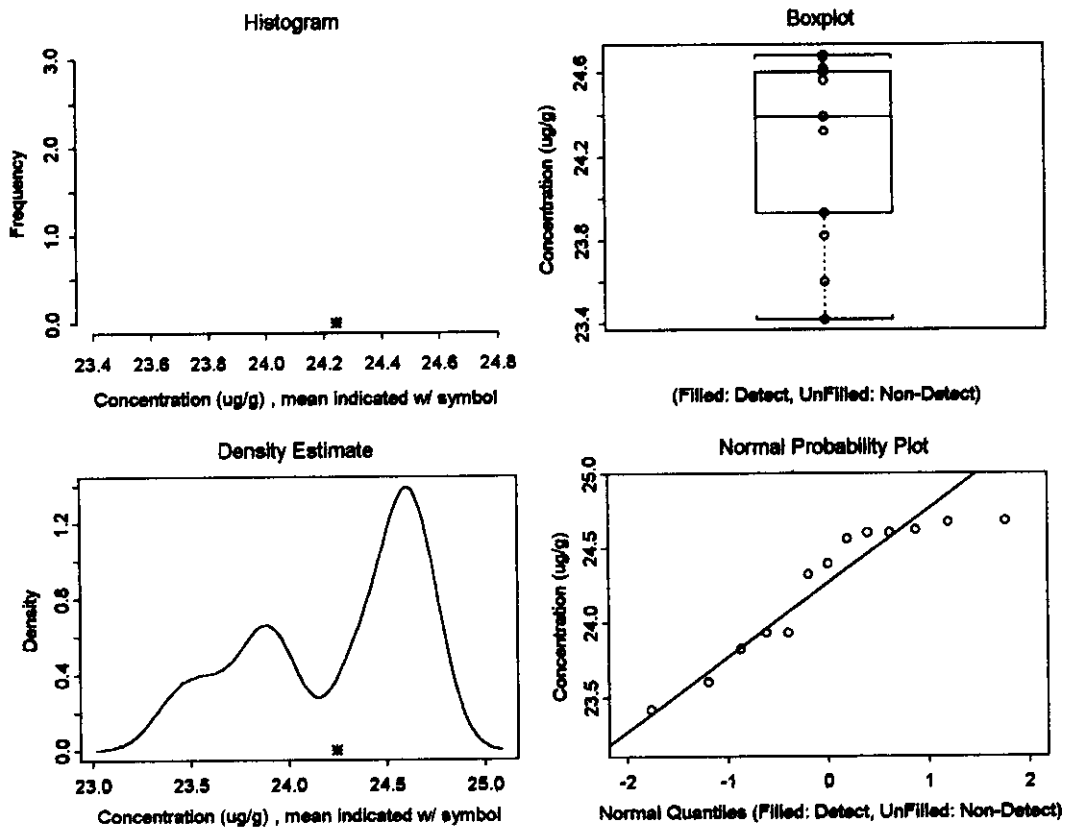
As shown in Figure 3-36, all samples for plutonium/americium-241are nondetects. Detection limits ranged from 23.4 μg/g to 24.7 μg/g.

Figure 3-36. Composite Sample Results for Plutonium/Americium-241 by ICP/MS



Because all results for plutonium/americium-241 are nondetects, it is difficult to make any assertions about the distribution of the data from the composite samples. The exploratory data graphs in Figure 3-37 represent the distribution of the detection limits for plutonium/ americium-241.

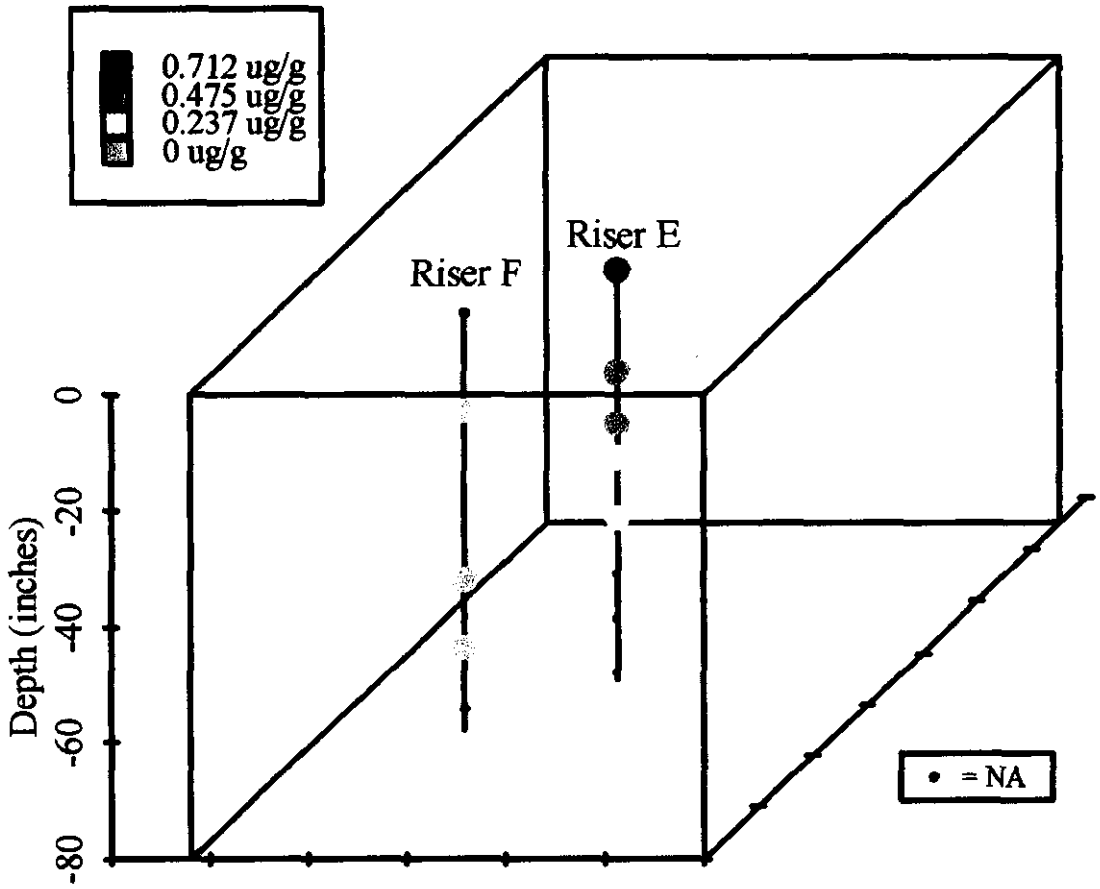
Figure 3-37. Exploratory Data Analysis for Plutonium/Americium-241 by ICP/MS



3.9.2.10 Plutonium-241 by Calculation

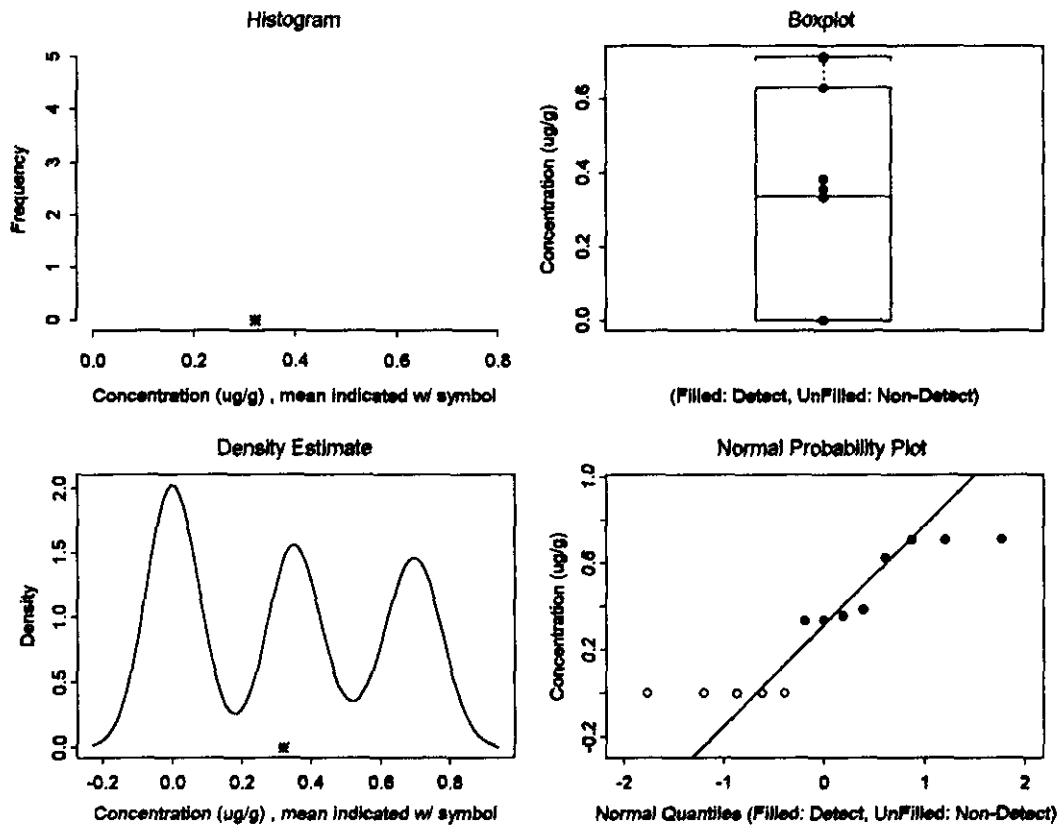
Results for plutonium-241 are estimated as discussed in Section 3.4.10, ICP/MS. Of the plutonium/americium-241 ICP/MS data that are used to estimate the plutonium-241 values, five of the 13 plutonium/americium-241 values are nondetects. Nondetected composite results for plutonium/americium-241 are not used to estimate plutonium-241, so only the eight detects are used. The nondetected values are represented with small black dots (Figure 3-38). The highest concentrations are estimated to be in the upper portion of Riser E.

Figure 3-38. Composite Sample Results for Plutonium-241



The exploratory data plots in Figure 3-39 show that the eight detected results for plutonium-241 do not appear to follow a normal distribution. There appears to be three distinct sets of data within these samples. The trimodel appearance of the data appears to be an artifact of detection limit issues.

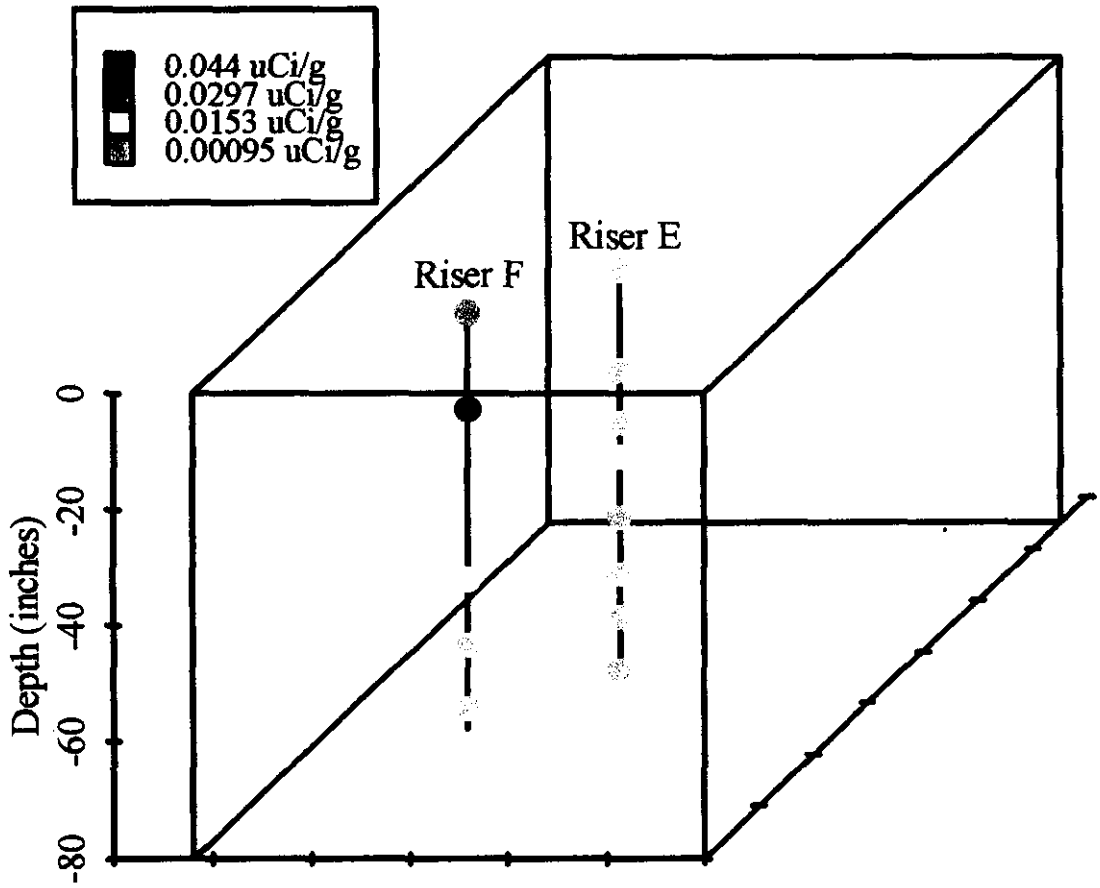
Figure 3-39. Exploratory Data Analysis for Plutonium-241



3.9.2.11 Strontium-89/90

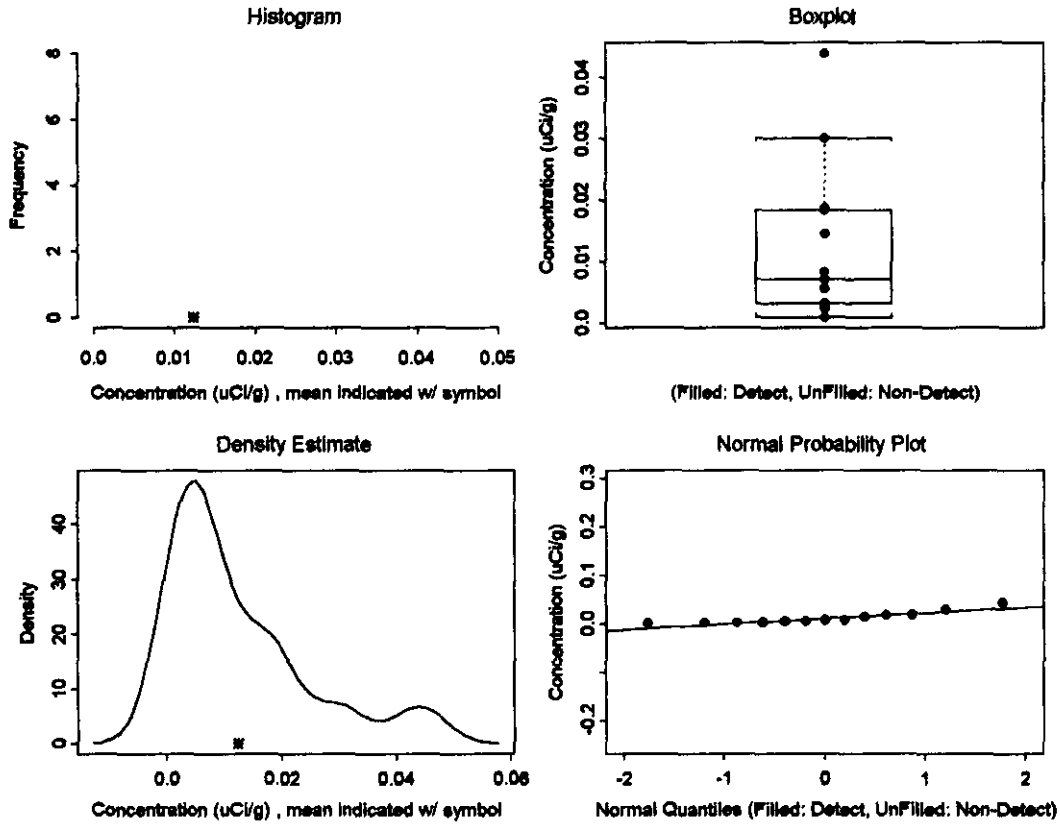
All composite samples for strontium-89/90 are detected values. The detected results for strontium-89/90 range from 0.00236 μCi/g to 0.044 μCi/g. The highest activity is from the composite sample taken at the depths 16.5 to 33.5 in from Riser F. The lowest activity is from the composite sample taken at the depths 59 to 65 in from Riser E. There does not appear to be a strong trend relating result and depth, although the lowest results are found at the bottom of the tank. Figure 3-40 shows that results from Riser E near the top and bottom of the tank are relatively lower than those found in the middle. The highest results from Riser F are found in the upper two-thirds of the tank with the highest result being found at a depth of 16.5 to 33.5 in below the sludge surface.

Figure 3-40. Composite Sample Results for Strontium-89/90



The exploratory data plots in Figure 3-41 show the distribution for the concentrations of strontium-89/90 is skewed to the right. The results are not normally distributed. There is one outlier in the data.

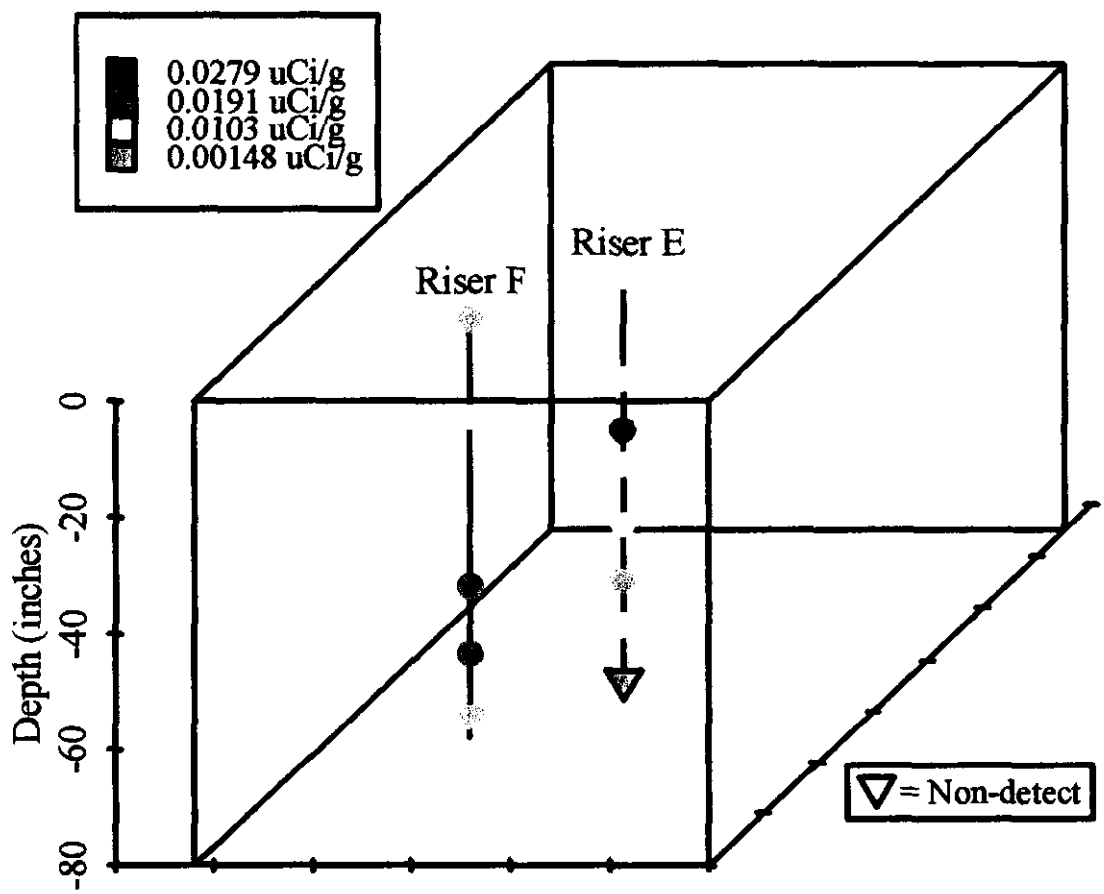
Figure 3-41. Exploratory Data Analysis for Strontium-89/90



3.9.2.12 Technetium-99

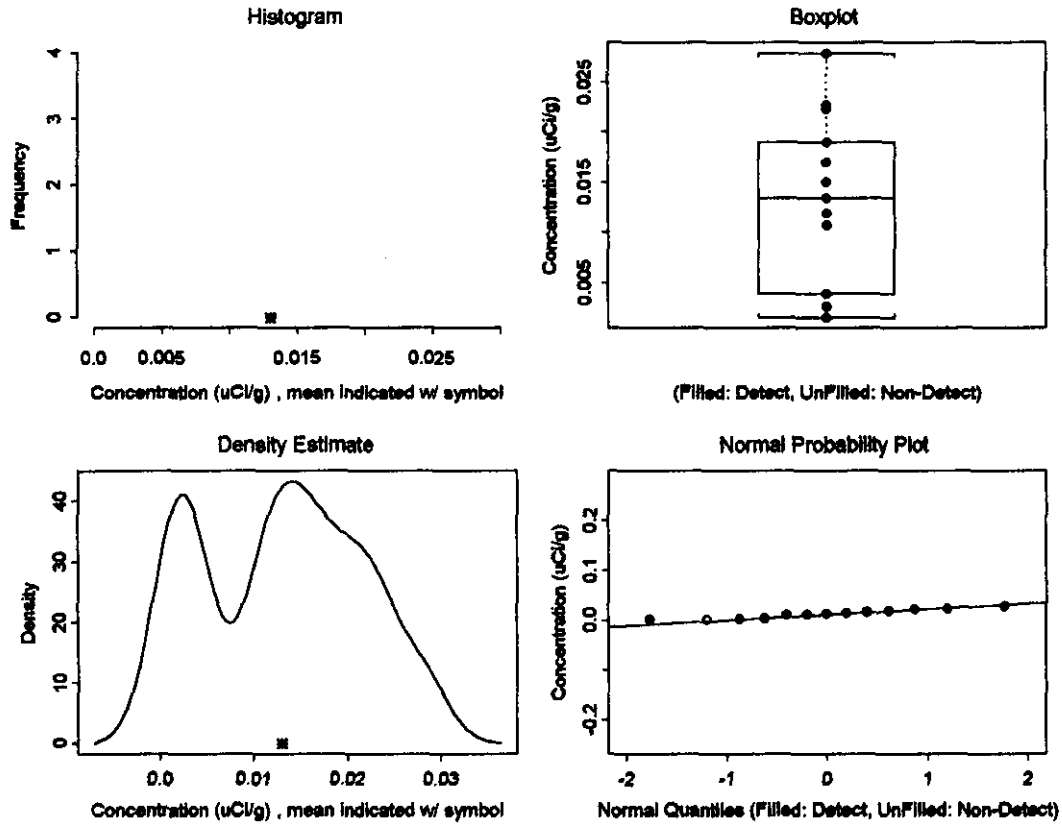
Twelve of the 13 results for technetium-99 are detected values. The activities for technetium-99 range from 0.00148 μCi/g to 0.0279 μCi/g. Figure 3-42 shows that the highest activities in both risers are found in the composite samples taken from the middle of the sludge. The maximum activity is from the composite sample from Riser E taken at the depths 34.25 to 38 in The lowest result is from the composite sample taken from Riser E at the depths 59 to 65 in.

Figure 3-42. Composite Sample Results for Technetium-99



The exploratory data plots in Figure 3-43 show the distribution of activities for technetium-99 is slightly skewed to the right. The results do not appear to be well approximated by a normal distribution. There are no outliers in the data.

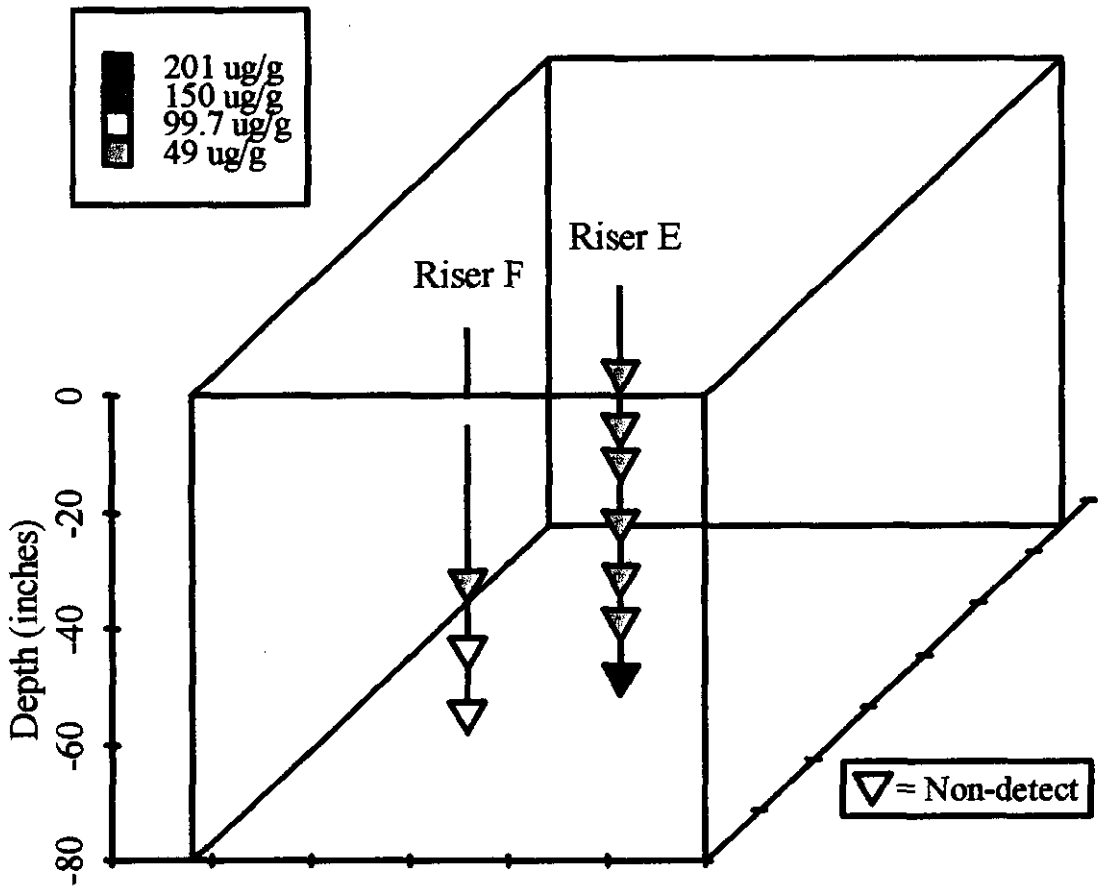
Figure 3-43. Exploratory Data Analysis for Technetium-99



3.9.2.13 Uranium

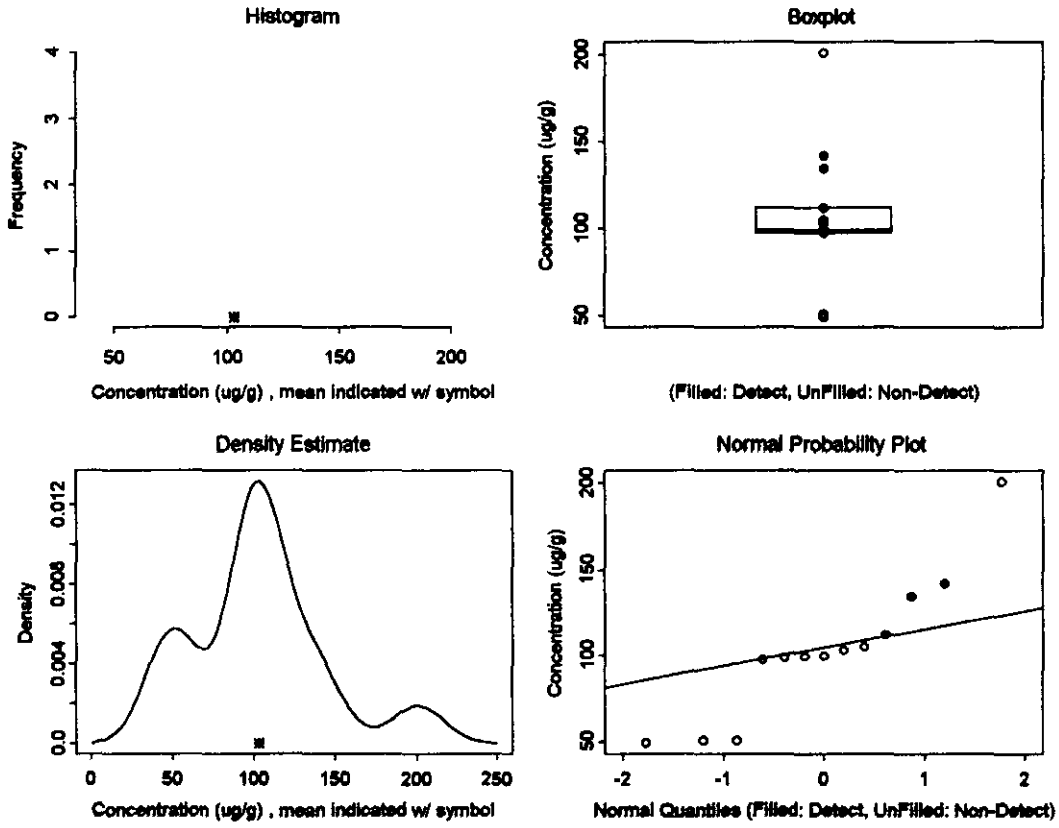
Three of the 13 results for uranium are detected values. Figure 3-44 shows that the detected values are at the top of the tank. Concentrations for the detected values range from 112 µg/g to 142 µg/g. Detection limits for the nondetected values range from 49 µg/g to 201 µg/g.

Figure 3-44. Composite Sample Results for Uranium



The exploratory data plots are shown in Figure 3-45. Because only three of the results for uranium are detects, it is difficult to make any assertions about the distribution of the data.

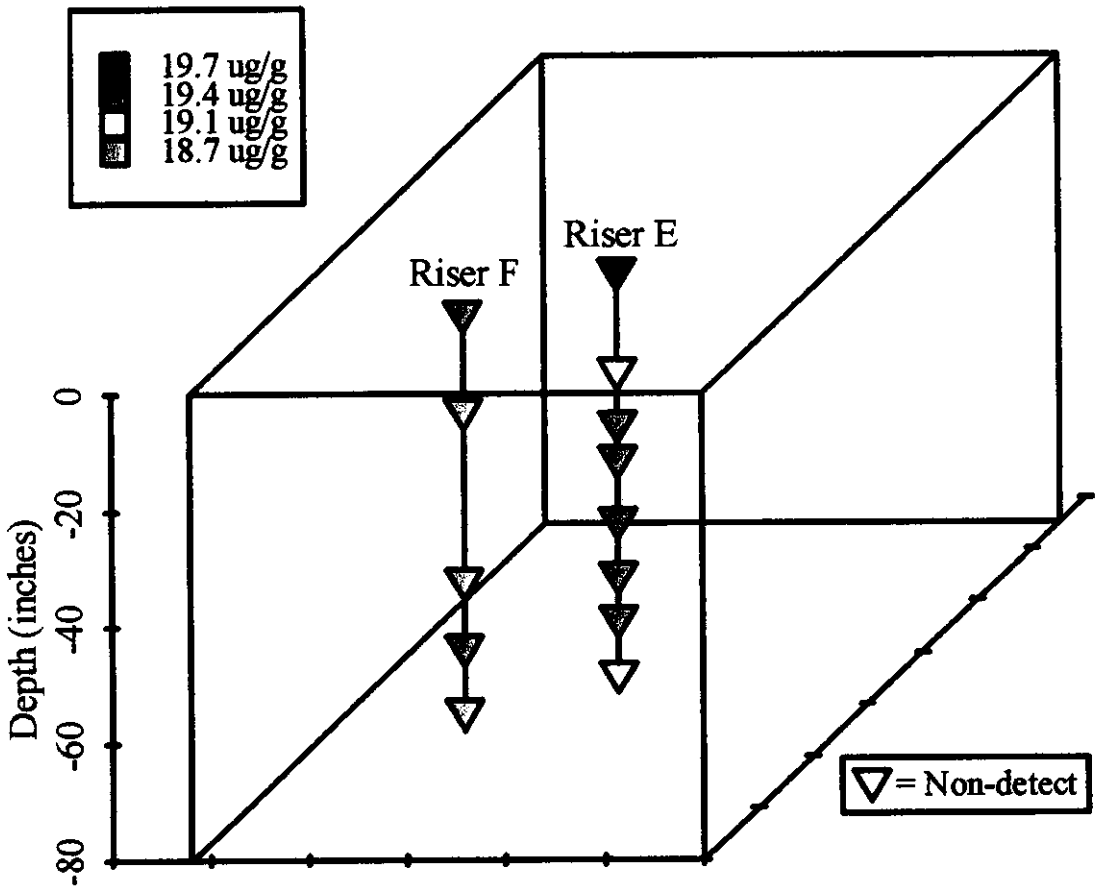
Figure 3-45. Exploratory Data Analysis for Uranium



3.9.2.14 Uranium-235

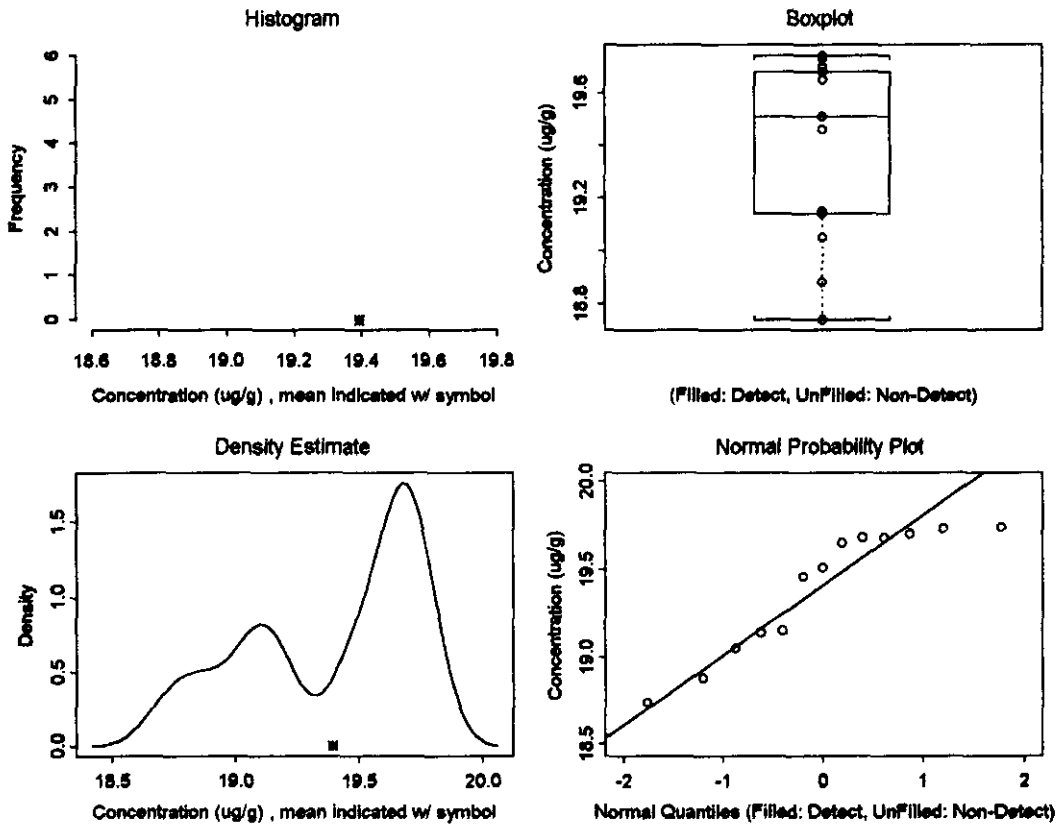
All composite samples for uranium-235 are nondetects. Detection limits, shown in Figure 3-46, range from 18.7 µg/g to 19.7 µg/g.

Figure 3-46. Composite Sample Results for Uranium-235



Because all results for uranium-235 are nondetects, it is difficult to make any assertions about the distribution of the data from the composite samples. The exploratory data plots in Figure 3-47 represent the distribution of the detection limits.

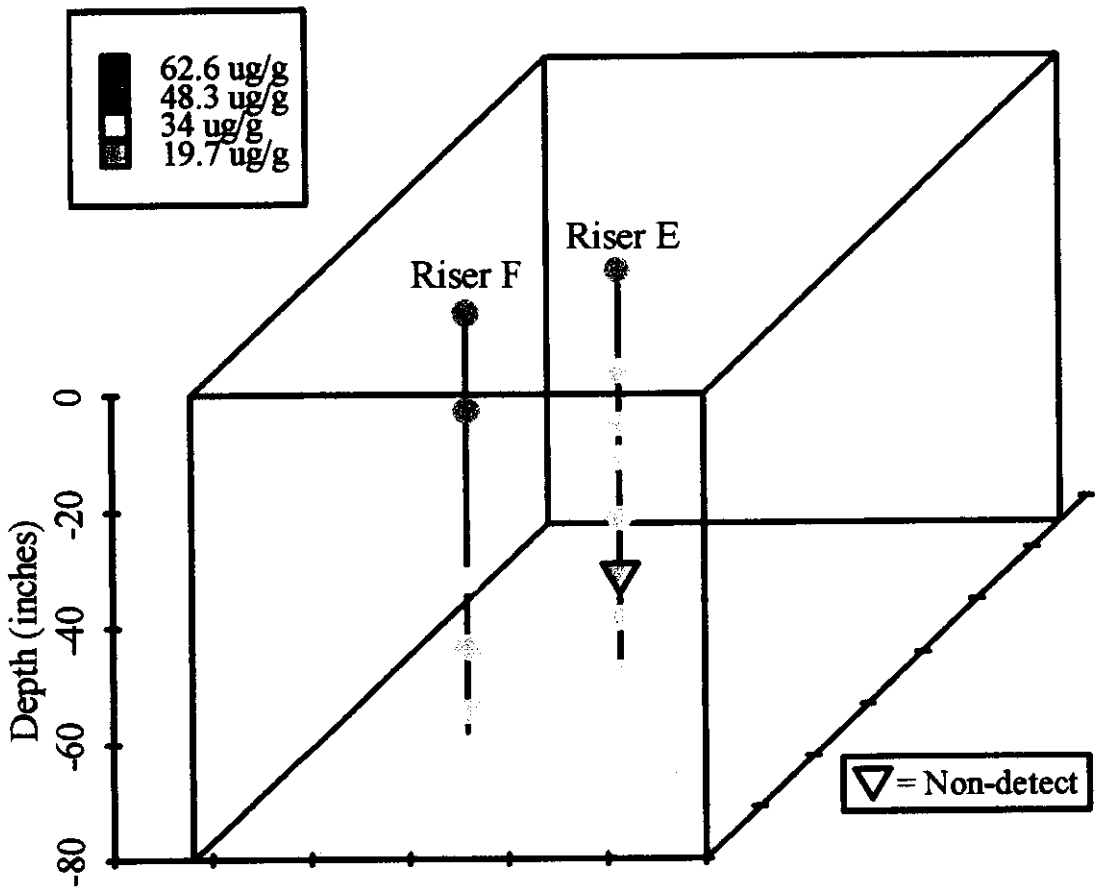
Figure 3-47. Exploratory Data Analysis for Uranium-235



3.9.2.15 Uranium-238

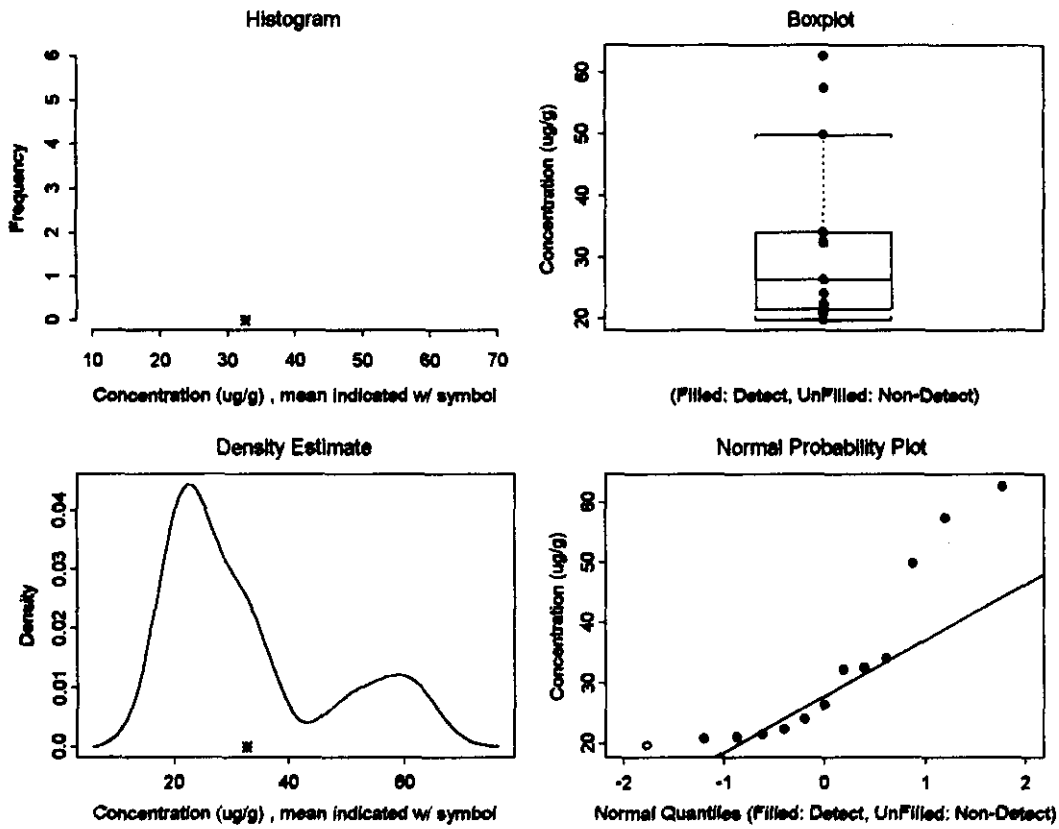
Twelve of the 13 results for uranium-238 are detected values. The detected values for uranium-238 range from 20.8 µg/g to 62.6 µg/g. The highest detected result is from the composite sample taken at the depth 16.5 to 33.5 in from Riser F. The lowest detected result was from the composite sample taken from Riser E at the depth 34.25 to 38 in. Figure 3-48 shows that the highest activities for Riser F are found in the top of the sludge.

Figure 3-48. Composite Sample Results for Uranium-238



The exploratory data plots are presented in Figure 3-49. The distribution of concentrations for uranium-238 is skewed to the right and possibly bimodal. There are several extreme values that increase the variability of the sample. The results do not seem to be well represented by a normal distribution. The two extreme values in the data occur at the top of Riser F.

Figure 3-49. Exploratory Data Analysis for Uranium-238



3.9.3 Assessment of the Horizontal and Vertical Strata for Radionuclides

Determining the extent of the vertical and horizontal strata of radionuclides is difficult due to limited data available for analysis. The core samples obtained from Risers E and F were approximately 5 ft apart (Figure 1-1). The horizontal extent can only be qualitatively described in the region between, and to some extent around, the areas where the core samples were taken. The vertical profile can only be assessed in the area where there are data. This limits the conclusions which can be drawn about the vertical strata to those areas between and around the boreholes. Furthermore, results of quantitative techniques such as kriging and tests for trend are somewhat questionable when sample sizes are small (n=13) and spatial representation (two cores relatively close together) is limited. Descriptive statistics and observation will guide the qualitative assessment of the horizontal and vertical strata.

Table 3-62 presents the maximum and minimum detected values for each of the analytes with the corresponding average depth (the midpoint of the depth interval) of the composite sample from which the result is obtained.

Table 3-62. Summary of Maximum and Minimum Analytical Results for Sludge Composite Samples from Tank 241-Z-361

Analyte	Number of Detects	Min	Depth of Min[a] (in.)	Max	Depth of Max[a] (in.)
Americium-241**	13	0.306	79	14.1	10
Gross Alpha**	13	3.87	79	42.6	36
Gross Beta**	13	0.146	79	3.96	25
Neptunium-237**	4	0.00293	36	0.00523	8
Plutonium-238**	1	2.3	10	2.3	10
Plutonium-239*	13	69.1	79	578	36
Plutonium-239/240**	13	4.21	79	42.4	36
Plutonium-240*	13	25.5	10	37.2	36
Plutonium/Americium-241*	NA	NA	NA	NA	NA
Strontium-89/90**	13	0.00095	62	0.044	25
Technetium-99**	12	0.00148	62	0.0279	36
Uranium*	3	112	10	142	25
Uranium-235*	0	NA	NA	NA	NA
Uranium-238*	12	20.8	36	62.6	25

*Units = µg/g
**Units = µCi/g
[a] Depth of max/min is the midpoint of the sludge composite sample interval

Information for the depth of the maximum detected result in Table 3-62 shows that for each analyte, the maximum detected result occurs in the upper half of the sludge. The global minimum values are typically in the bottom half of the sludge. For four analytes (neptunium-237, plutonium-238, plutonium-240, and uranium), fewer than half of the samples are detects, so the observation of the minimum in the top half of the sludge may not reflect the entire core for that analyte. In fact, nondetects occurred at depths below the minimum (depth of minimum) for all four of those analytes. The one nondetect for uranium-238 is in the bottom half of the sludge.

It is clear there are distinct strata in the vertical profile because the compositing was performed based on the ability to distinguish these strata from one another. It may be the case that the layers observed in the vertical profile of the core extend horizontally throughout the sludge. Based on the NDA results (Section 3.8) from Risers B and G, it would appear that neutron emitting radionuclides and americium-241 are indeed present in similar horizontal layers across the tank. This can be seen from Figures 3-10, 3-15, and 3-19 with regard to thermal neutron counts.

3.9.4 Upper Confidence Limits of the Mean

Upper confidence limits (UCLs) of the mean at 90 and 95% are calculated for the radionuclide data using a nonparametric bootstrapping method (Appendix D). Because many of the distributions do not appear to be normal, the nonparametric method was chosen over a method that requires the assumption of normality. The use of a nonparametric method for calculating UCLs obviates the need for verifying assumptions about the underlying distribution of the data. It is also particularly useful when the data appear to have a mixed distribution resulting from multiple releases of contaminants, as is certainly the case for the sludge tank. For results that are nondetects, the detection limit was used as a value. The method used to assign values to nondetects impacts the subsequent calculation of the UCL. Depending on the number of nondetects, the detection limits, and the variability both within and between the detected values and the nondetected values, the estimate of the UCL can increase or decrease as a consequence of different replacement schemes. Impacts of the different replacement schemes must be assessed on a per analyte basis.

In this nonparametric bootstrapping method, the data are sampled, with replacement, to create a new data set of the same size as the original data set. This process is repeated 5,000 times, so that there are 5,000 new data sets of the same size as the original. Then the 90th or 95th percentile of the 5,000 sample means is calculated. In this way, the variability of the sample mean is quantified using a method that is based only on the data collected. The nonparametric bootstrapping method of calculating UCLs eliminates the need for the construction or assumption of a statistical model to quantify the variability of the sample mean. Table 3-63 shows the values of the 90 and 95% UCLs on the mean. These UCLs can be considered equivalent to the critical values for a one-sided hypothesis tests on the mean at the alpha levels of 0.10 and 0.05, respectively.

3.9.5 Nonradiological Inorganic Analyte Concentration and Distribution

Summary statistics for each of the analytes are presented in Table 3-64. When analytical duplicate data were available, the results were averaged before performing statistical analysis. The minimum, median, mean, standard deviation, relative standard deviation, and relative standard error of the mean were calculated. The relative standard deviation was calculated as the standard deviation divided by the mean. This provides a measure of the spread of the distribution relative to the analyte-specific mean concentration. The relative standard error was calculated as the standard error divided by the mean (i.e., the standard deviation divided by the square root of the number of samples divided by the mean). This provides a measure of the spread of the sample mean's distribution relative to the analyte-specific mean concentration. Results from the samples sent to the fixed laboratory are presented in the following analyses.

Results are presented for ammonium and ammonia, arsenic, barium, beryllium, bromide, cadmium, chloride, chromium, cyanide, fluoride, hydroxide, lead, lithium, mercury, nickel, nitrate, nitrite, phosphate, phosphorus, potassium, silver, sulfate, sulfur, and uranium. Results for which an analyte was detected in the sample are presented separately from the nondetected samples. The summary statistics for the nondetected samples present information on the distribution of the detection limits.

Table 3-63. Estimated Upper Confidence Limits of Core Radionuclide Data

Analyte	Units	90% UCL	95% UCL
Americium-241	Ci/g	8.063	8.614
Gross Alpha	Ci/g	28.65	29.75
Gross Beta	Ci/g	2.089	2.176
Neptunium-237	Ci/g	0.003973	0.004145
Plutonium-238	Ci/g	1.457	1.522
Plutonium-239/240	Ci/g	24.67	25.96
Plutonium-239 by ICP/MS-fusion	g/g	351.2	364.5
Plutonium-240 by ICP/MS-fusion	g/g	27.81	28.19
Plutonium-241	g/g	0.6022	0.6185
Plutonium/Americium-241 by ICP/MS-fusion	g/g	24.39	24.43
Strontium-89/90	Ci/g	0.01681	0.01829
Technetium-99	Ci/g	0.01603	0.01686
Uranium	g/g	118.1	122.3
Uranium-235	g/g	19.51	19.54
Uranium-238	g/g	37.72	39.29

Table 3-64. Summary Statistics for Pooled Nonradiological Data from All Composite Samples* (2 sheets)

Analyte**	Detect	N	Minimum	Median	Mean	Maximum	Std. Dev.	Relative Std. Dev.	Relative Std. Error of Mean
Ammonia/Ammonium	D	2	625.500	637.250	637.250	649.000	16.617	0.026	0.018
	N	11	482.000	494.000	495.727	513.000	9.603	0.019	0.006
Arsenic	D	1	23.400	23.400	23.400	23.400	NA	NA	NA
	N	12	9.800	19.850	19.283	40.200	8.001	0.415	0.120
Barium	D	13	85.600	114.000	127.354	197.000	35.046	0.275	0.076
Beryllium	N	13	0.490	0.996	0.967	2.010	0.383	0.396	0.110
Bromide	D	13	283.400	1700.000	1899.108	5370.000	1334.655	0.703	0.195
Cadmium	D	13	1.480	32.800	39.260	112.000	31.878	0.812	0.225
Chloride	D	13	482.000	599.500	644.977	926.400	143.726	0.223	0.062
Chromium	D	13	691.000	1340.000	2796.385	10000.000	2724.819	0.974	0.270
Cyanide***	D	3	1.010	1.215	1.188	1.340	0.167	0.140	0.081
	N	9	0.650	0.787	0.788	0.900	0.087	0.110	0.037
Fluoride	D	13	1180.000	3820.000	4830.000	10800.000	2678.694	0.555	0.154
Hydroxide	N	13	8170.000	8220.000	8243.077	8510.000	86.351	0.010	0.003
Lead	D	13	32.000	91.900	143.038	446.000	132.969	0.930	0.258
Lithium	D	13	13.650	95.900	146.954	374.000	108.535	0.739	0.205
Mercury	D	13	16.380	35.970	50.282	169.600	42.298	0.841	0.233
Nickel	D	13	56.700	325.000	762.985	3380.000	967.207	1.268	0.352
Nitrate	D	7	209.000	435.000	586.386	1260.000	381.522	0.651	0.246
	N	6	164.000	166.000	166.167	170.000	2.229	0.013	0.005
Nitrite	D	13	342.100	909.000	937.169	1540.000	379.742	0.405	0.112
Phosphate	N	13	139.000	143.000	142.923	147.000	2.431	0.017	0.005
Phosphorus	D	13	133.000	173.000	307.577	831.000	232.892	0.757	0.210

Table 3-64. Summary Statistics for Pooled Nonradiological Data from All Composite Samples* (2 sheets)

Analyte**	Detect	N	Minimum	Median	Mean	Maximum	Std. Dev.	Relative Std. Dev.	Relative Std. Error of Mean
Potassium	D	1	270.000	270.000	270.000	270.000	NA	NA	NA
	N	12	49.000	99.400	88.008	105.000	22.971	0.261	0.075
Silver	D	13	15.600	29.100	49.754	182.000	50.277	1.011	0.280
Sulfate	D	13	974.000	1320.000	1413.385	1980.000	333.360	0.236	0.065
Sulfur	D	13	362.000	471.000	541.038	1090.000	197.185	0.364	0.101
Uranium	D	3	112.000	134.500	129.500	142.000	15.612	0.121	0.070
	N	10	49.000	98.900	95.410	201.000	44.115	0.462	0.146

*When analytical duplicate data were available, the results were averaged before performing statistical analysis.

**Units for all analytes are g/g

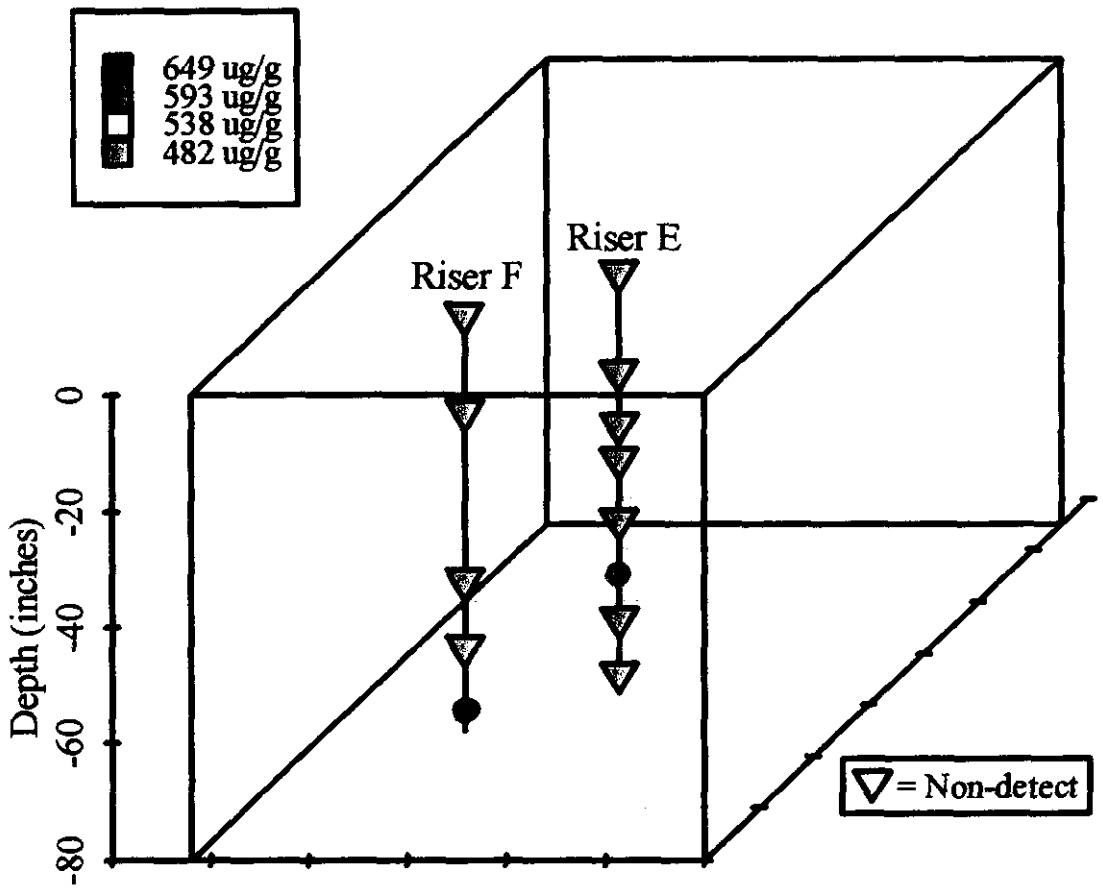
***The database supplied by the laboratory omitted the nondetect result of <0.796 for composite 264-4; therefore, the statistics were calculated for nine nondetect samples rather than 10. This will not affect decision-making.

3.9.5.1 Ammonia/Ammonium

Only two of the thirteen results for ammonia and ammonium are detected values. The detected concentrations for ammonia and ammonium are 625.5 µg/g and 649 µg/g. The nondetected concentrations for ammonia and ammonium range from 482 µg/g to 513 µg/g. Figure 3-50 shows that the detected concentrations are found in the composite samples taken from near the bottom of the sludge.

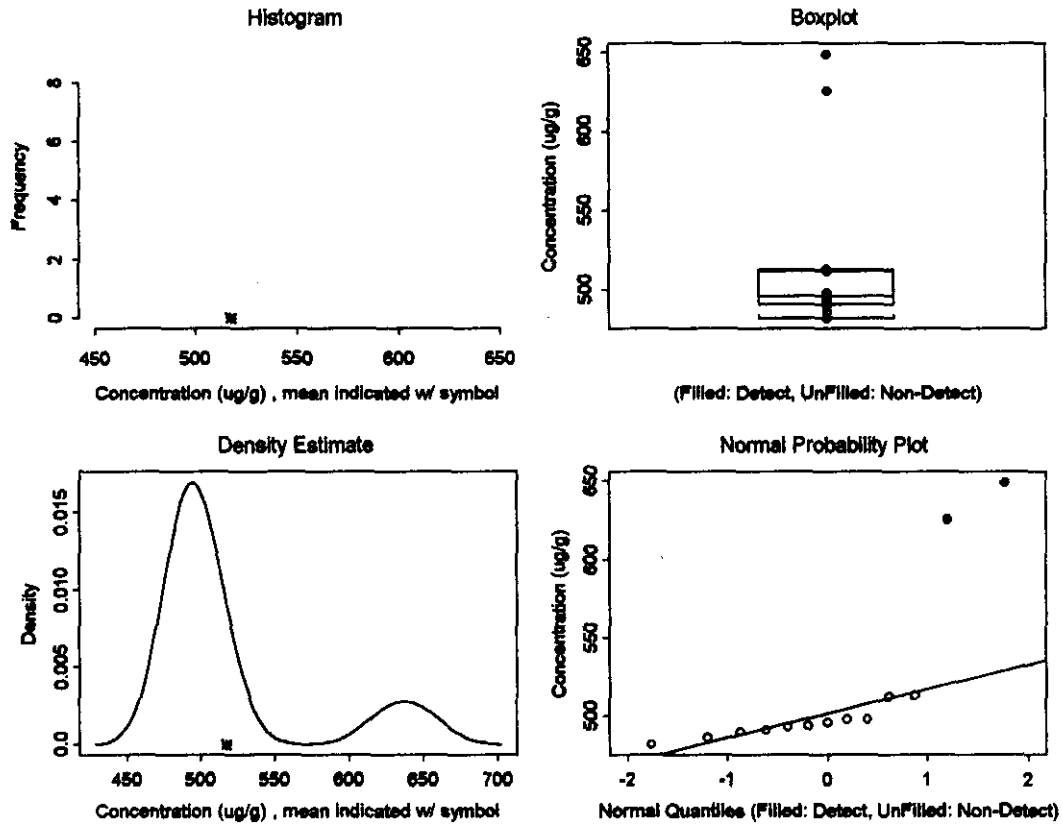
Under alkaline conditions, such as those in the tank, the conversion of ammonium ion to ammonia gas is favored. Therefore, it is not surprising that the only detections of this volatile gas are near the base of the cores. In addition, conversion pathways to nitrate and nitrite exist and may have been operative in the sludge.

Figure 3-50. Composite Sample Results for Ammonia and Ammonium



The exploratory data plots are shown in Figure 3-51. Because there are only two detected values for ammonia and ammonium, it is difficult to make any statements about the distribution. The distribution of nondetects appears to follow a normal distribution.

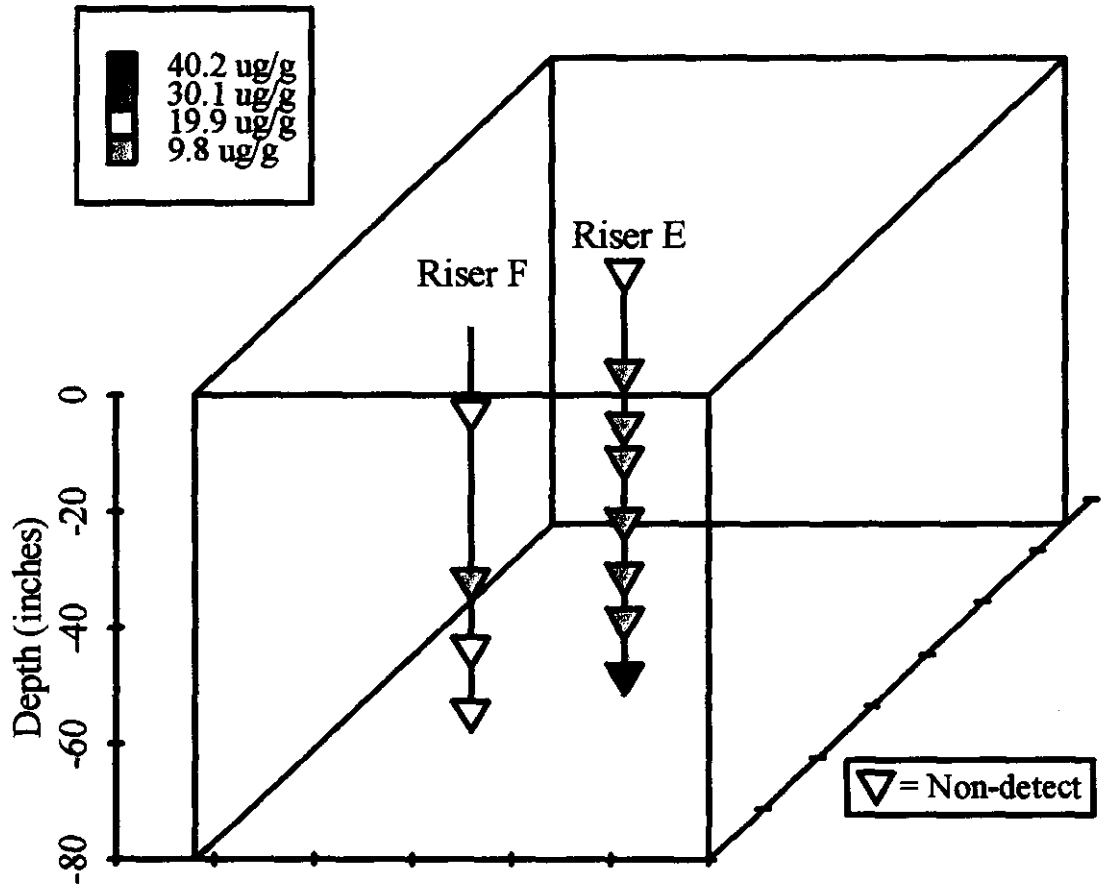
Figure 3-51. Exploratory Data Analysis for Ammonia and Ammonium



3.9.5.2 Arsenic

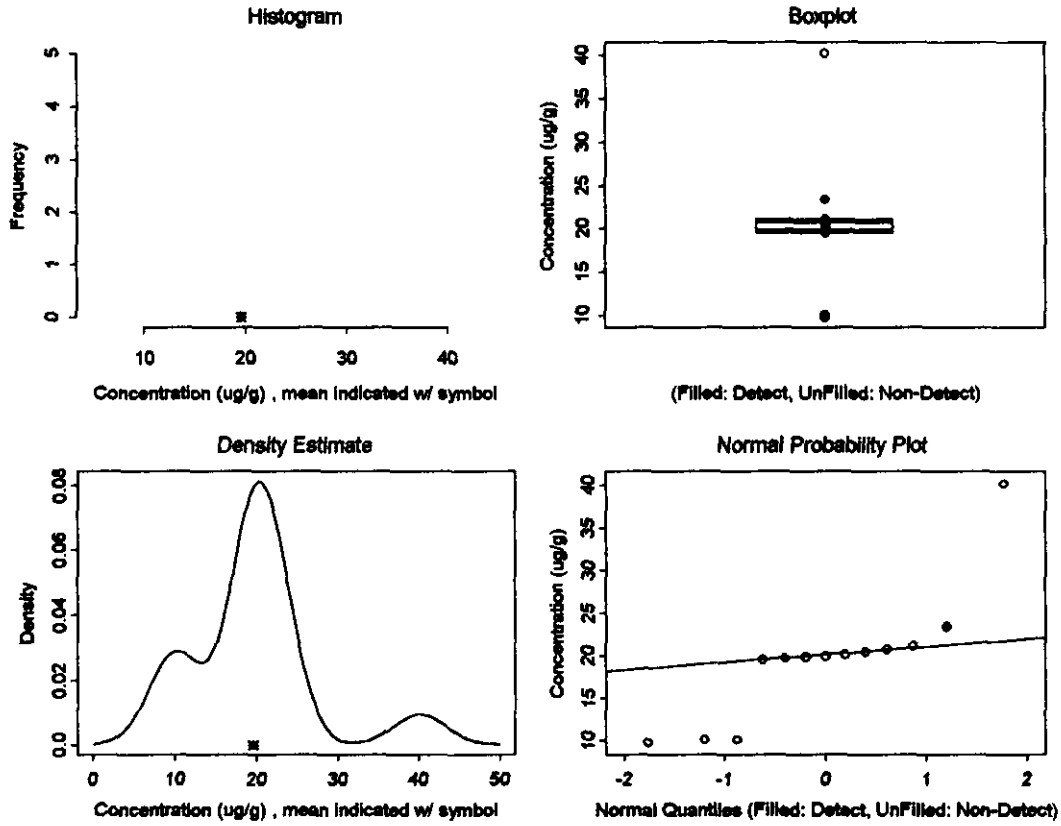
The composite sample results are shown in Figure 3-52. Only one of the 13 results for arsenic is a detected value. The detected value is located at the top of the sludge in Riser F and its concentration is 23.4 μg/g.

Figure 3-52. Composite Sample Results for Arsenic



The exploratory data plots are shown in Figure 3-53. Because there is only one detected value for arsenic, it is difficult to make any statements about the distribution.

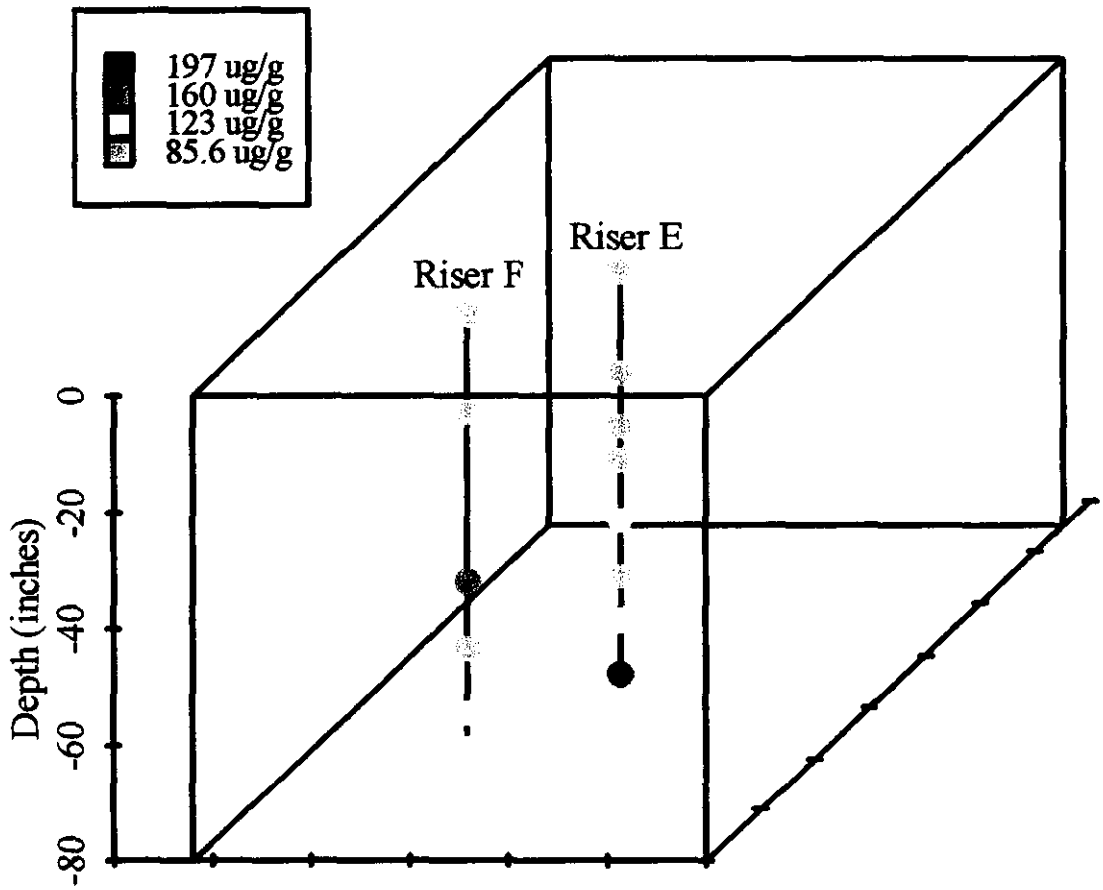
Figure 3-53. Exploratory Data Analysis for Arsenic



3.9.5.3 Barium

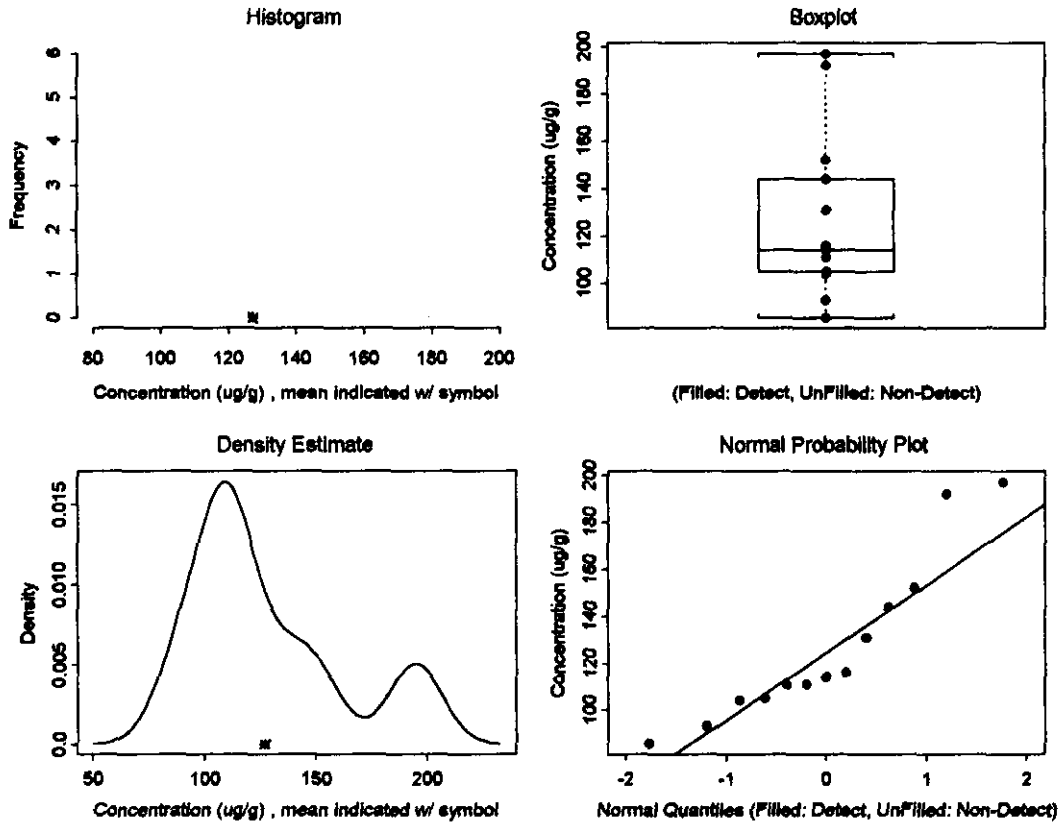
All 13 results for barium are detected values. The concentrations for barium range from 85.6 μg/g to 197.0 μg/g. Figure 3-54 shows that the highest concentrations are found in the composite samples taken from the bottom of the sludge in Riser E and the middle of the sludge in Riser F.

Figure 3-54. Composite Sample Results for Barium



The exploratory data plots are shown in Figure 3-55. The distribution of concentrations for barium is skewed to the right. The results do not appear to be well approximated by a normal distribution. There are no outliers in the data.

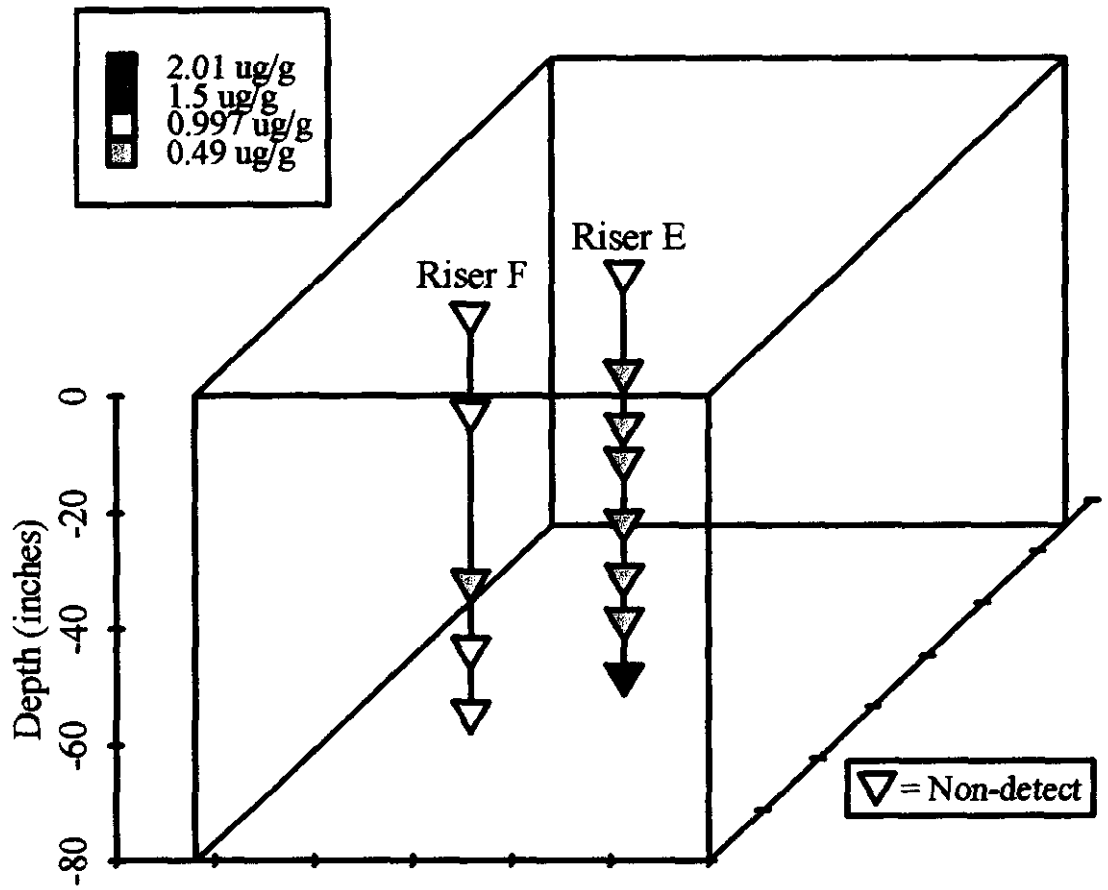
Figure 3-55. Exploratory Data Analysis for Barium



3.9.5.4 Beryllium

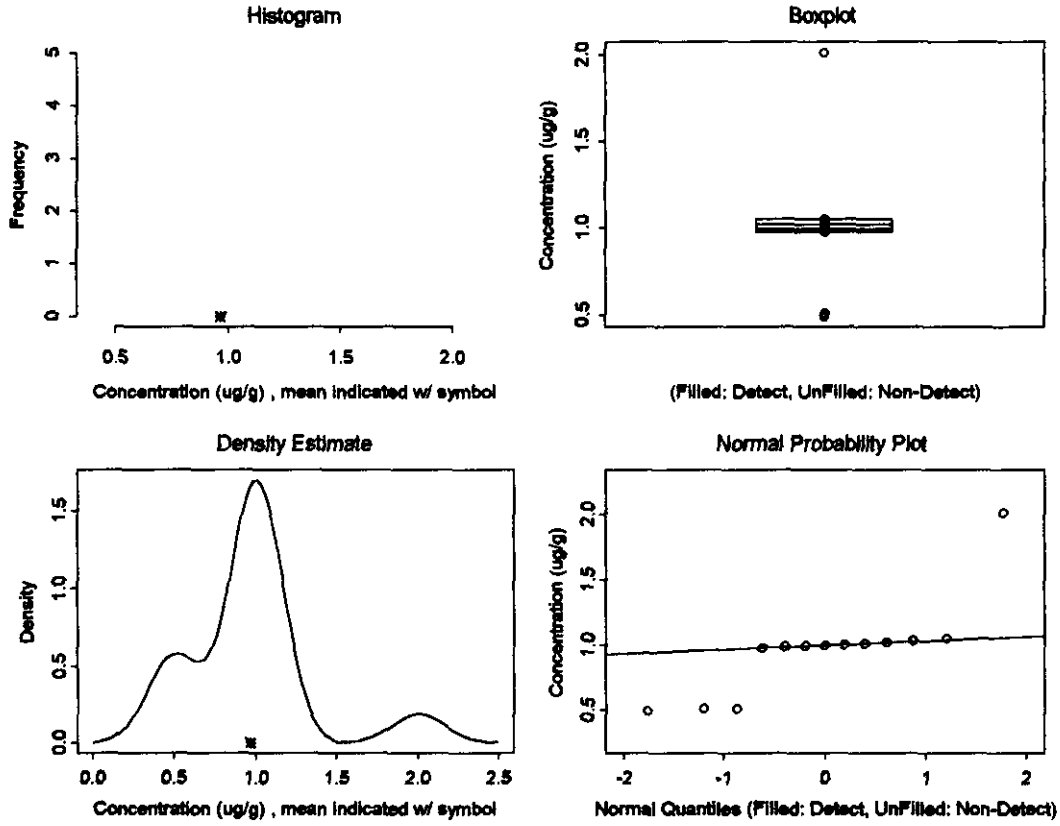
The composite sample results are shown in Figure 3-56. None of the thirteen results for beryllium are detected values. The detection limits for beryllium range from 0.490 μg/g to 2.010 μg/g.

Figure 3-56. Composite Sample Results for Beryllium



Because none of the thirteen results for beryllium are detected values, Figure 3-57 shows the distribution of the detection limits.

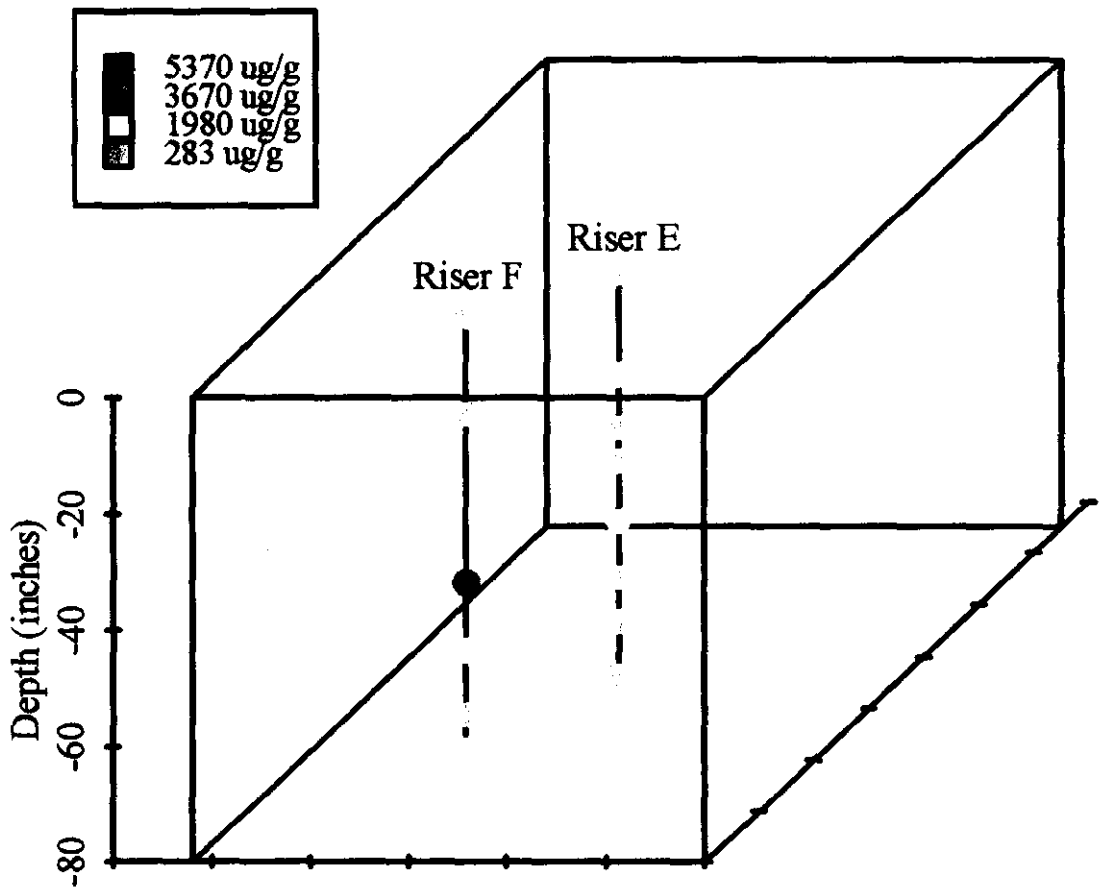
Figure 3-57. Exploratory Data Analysis for Beryllium



3.9.5.5 Bromide

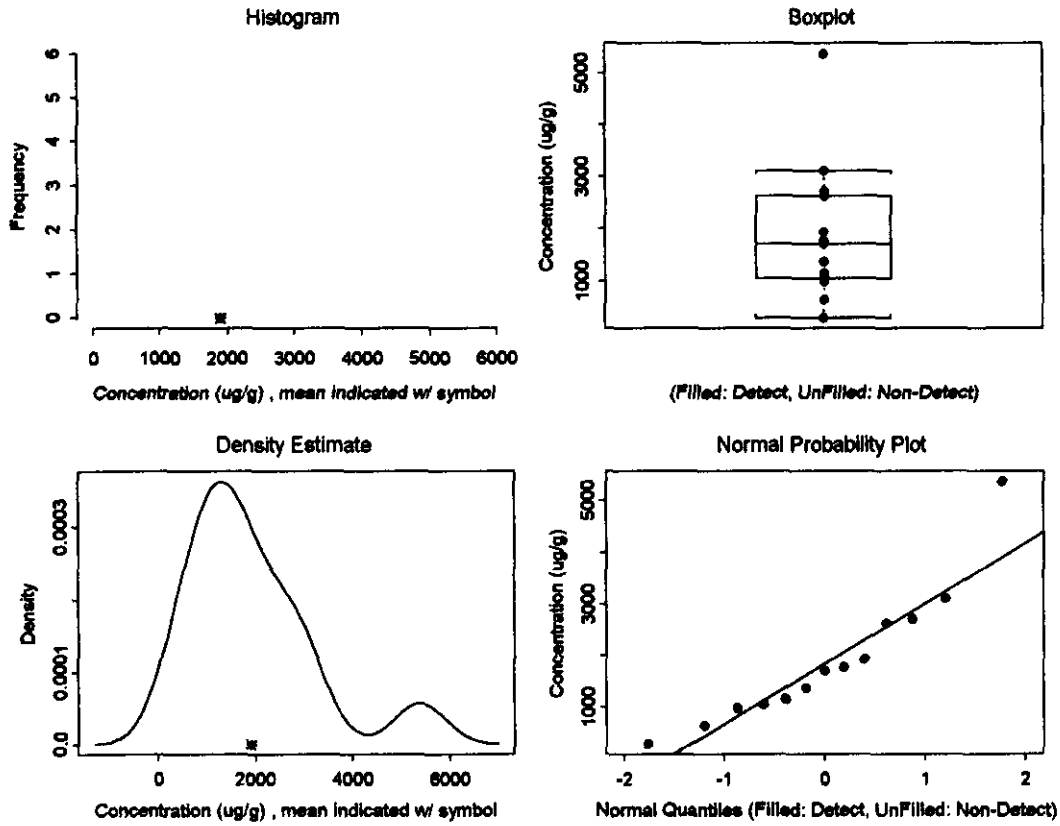
All 13 results for bromide are detected values. The concentrations for bromide range from 283.40 µg/g to 5370.0 µg/g. Figure 3-58 shows that the highest concentration is found in the composite samples taken from the middle of the sludge in Riser F.

Figure 3-58. Composite Sample Results for Bromide



The exploratory data plots are shown in Figure 3-59. The distribution of concentrations for bromide is skewed to the right. The results do not appear to be well approximated by a normal distribution. There is one outlier in the data.

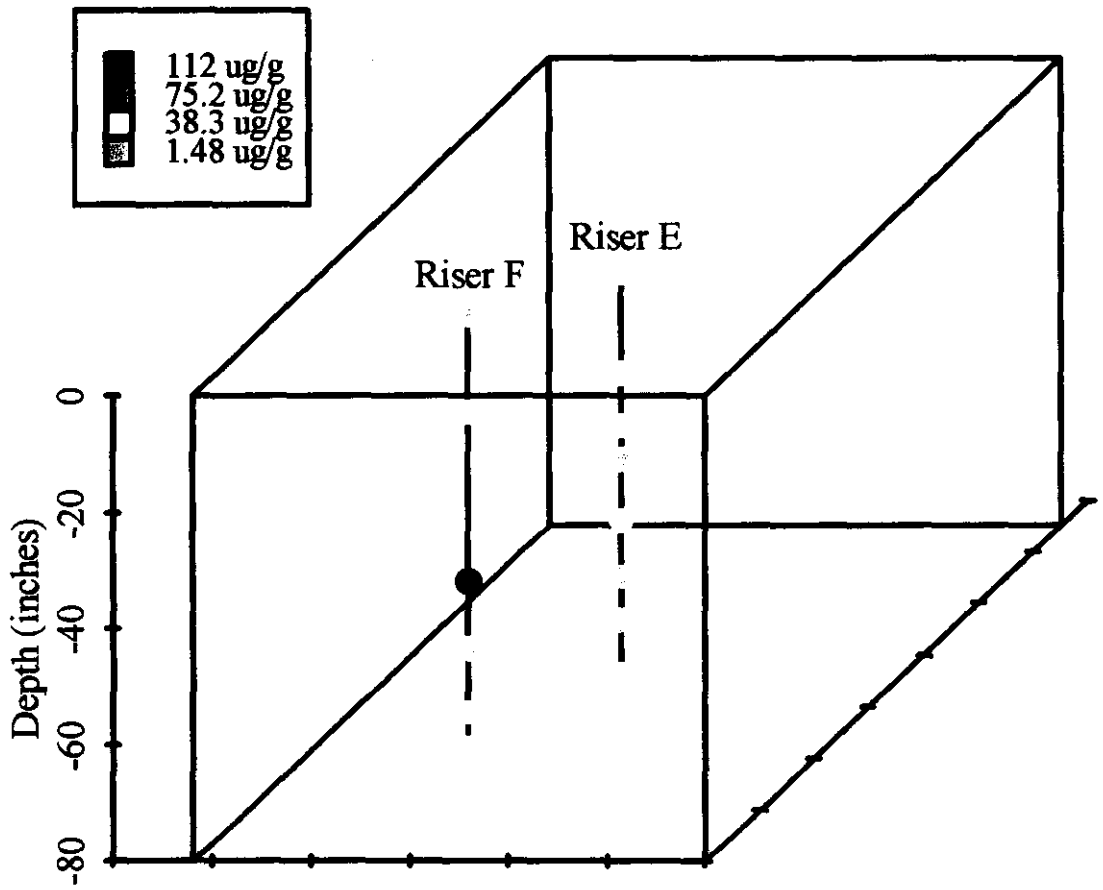
Figure 3-59. Exploratory Data Analysis for Bromide



3.9.5.6 Cadmium

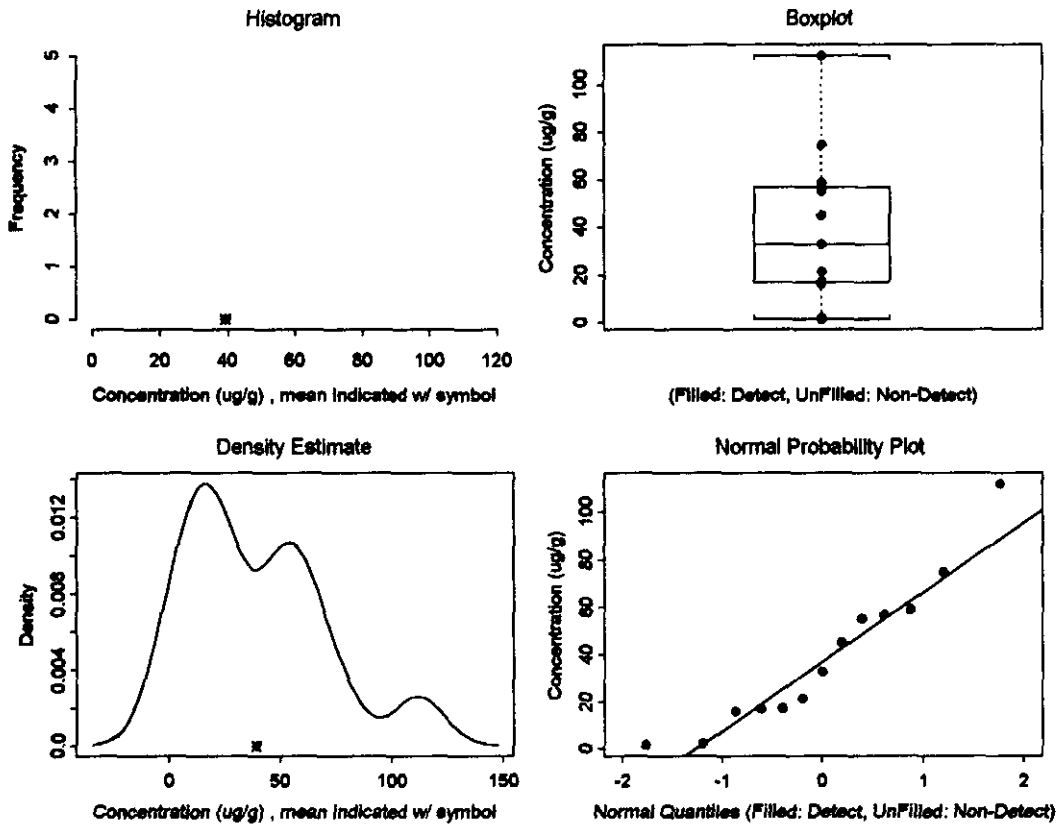
All results for cadmium are detected values. The concentrations for cadmium range from 1.480 µg/g to 112.0 µg/g. Figure 3-60 shows that the highest concentration is found in the composite samples taken from the middle of the sludge in Riser F.

Figure 3-60. Composite Sample Results for Cadmium



The exploratory data plots are shown in Figure 3-61. The distribution of concentrations for cadmium is slightly skewed to the right. The results do not appear to be well approximated by a normal distribution. There are no outliers in the data.

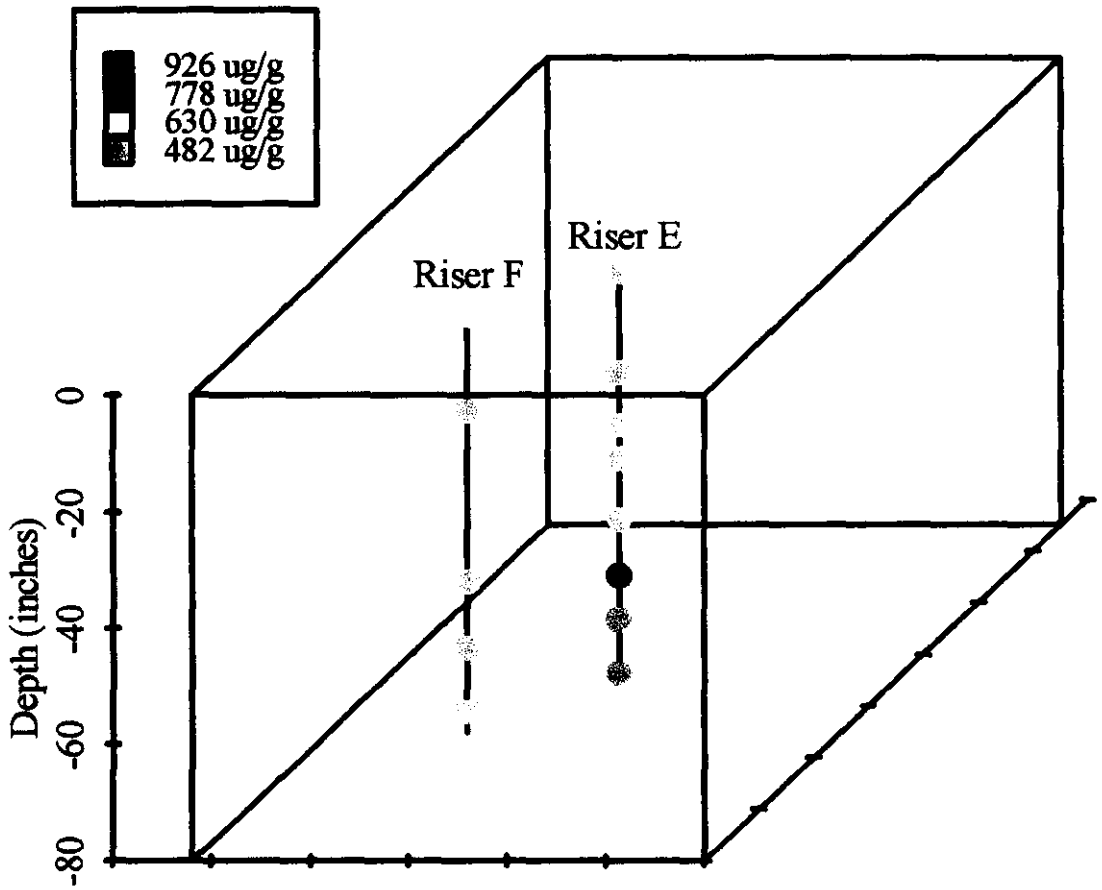
Figure 3-61. Exploratory Data Analysis for Cadmium



3.9.5.7 Chloride

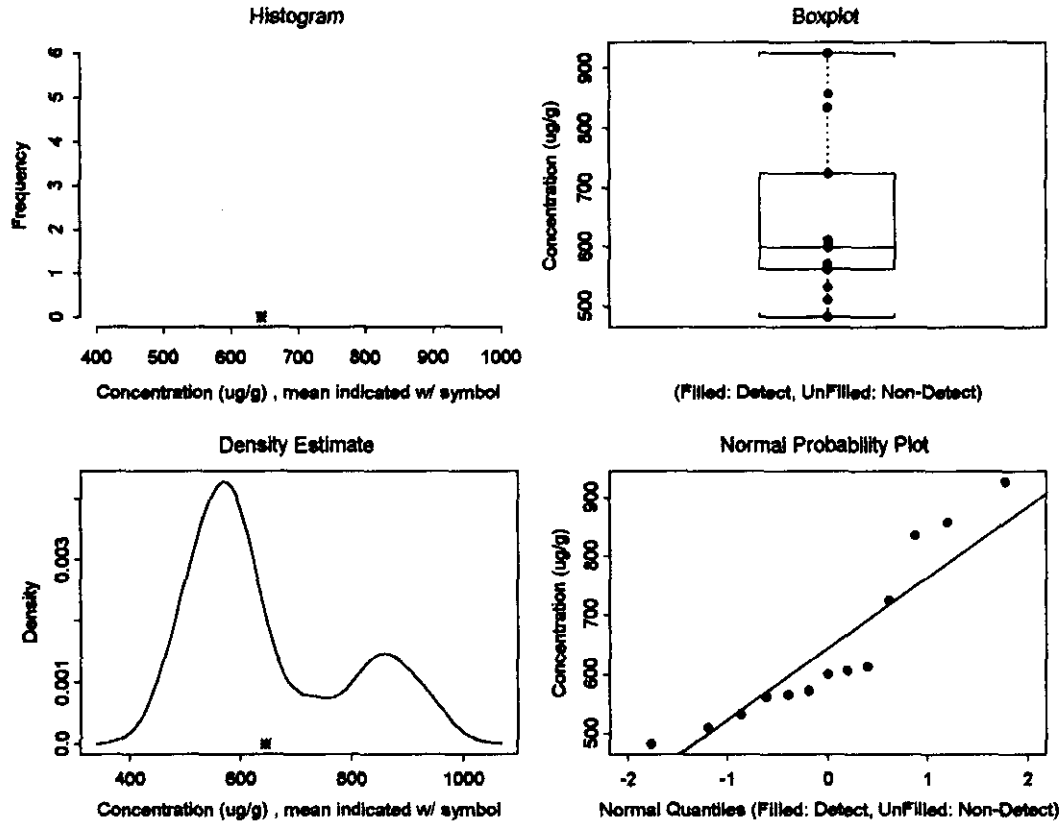
All 13 results for chloride are detected values. The concentrations for chloride range from 482.0 μg/g to 926.40 μg/g. Figure 3-62 shows that the highest concentrations are found in the composite samples taken from the bottom of the sludge in Riser E.

Figure 3-62. Composite Sample Results for Chloride



The exploratory data plots are shown in Figure 3-63. The distribution of concentrations for chloride is skewed to the right and possibly bimodal. The results do not appear to be well approximated by a normal distribution. There are no outliers in the data.

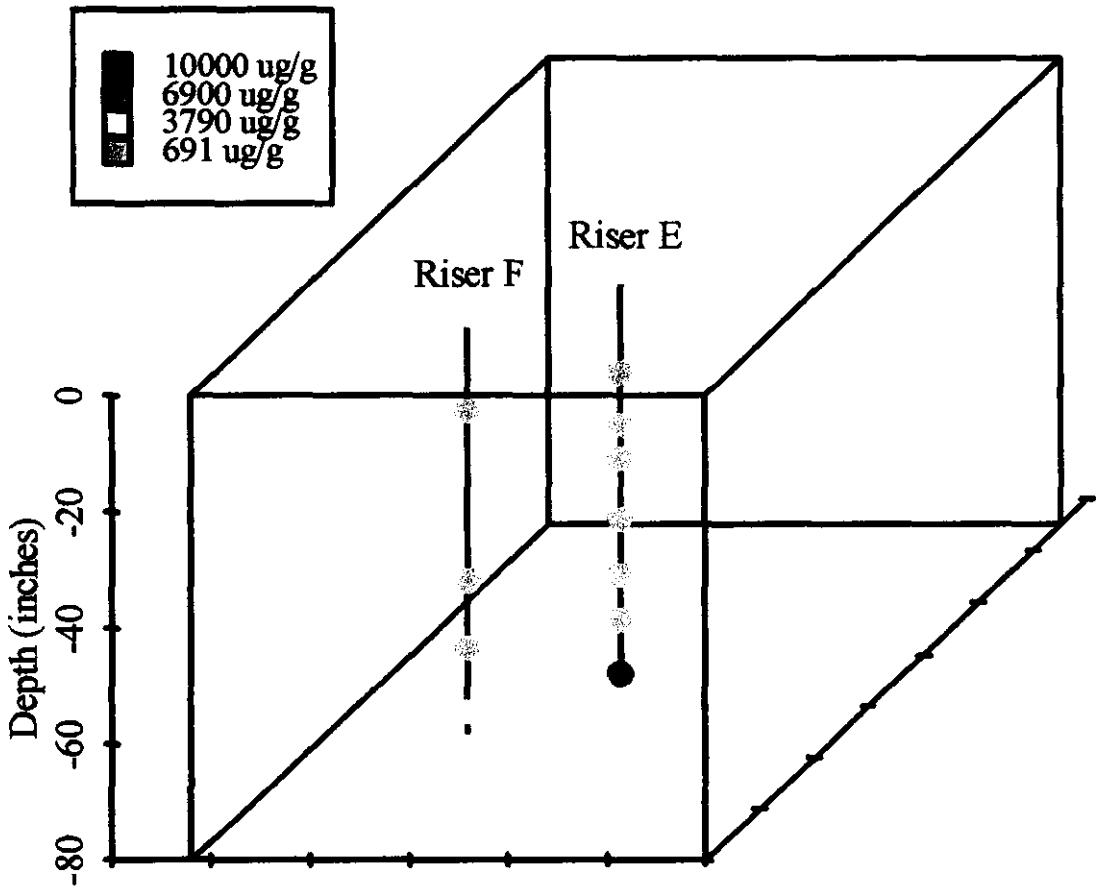
Figure 3-63. Exploratory Data Analysis for Chloride



3.9.5.8 Chromium

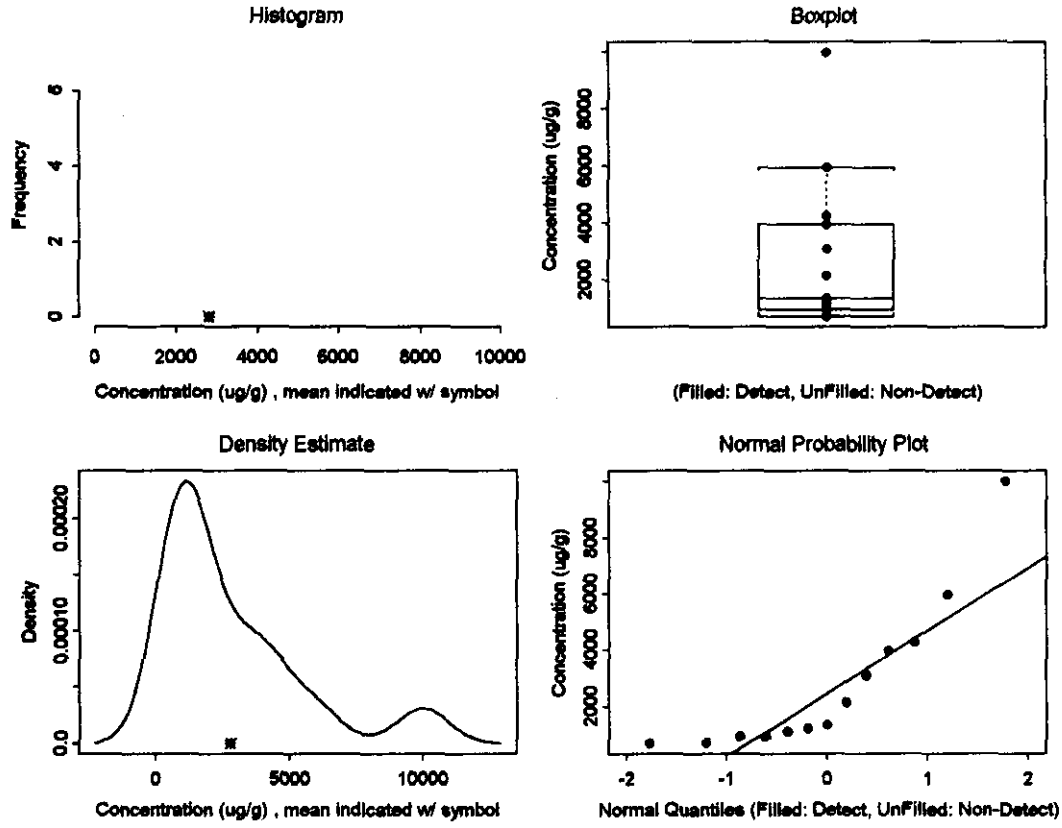
All results for chromium are detected values. The concentrations for chromium range from 691.0 µg/g to 10,000.0 µg/g. Figure 3-64 shows that the highest concentration is found in the composite sample taken at the bottom of the sludge in Riser E.

Figure 3-64. Composite Sample Results for Chromium



The exploratory data plots are shown in Figure 3-65. The distribution of concentrations for chromium is skewed to the right. The results do not appear to be well approximated by a normal distribution. There is one outlier in the data.

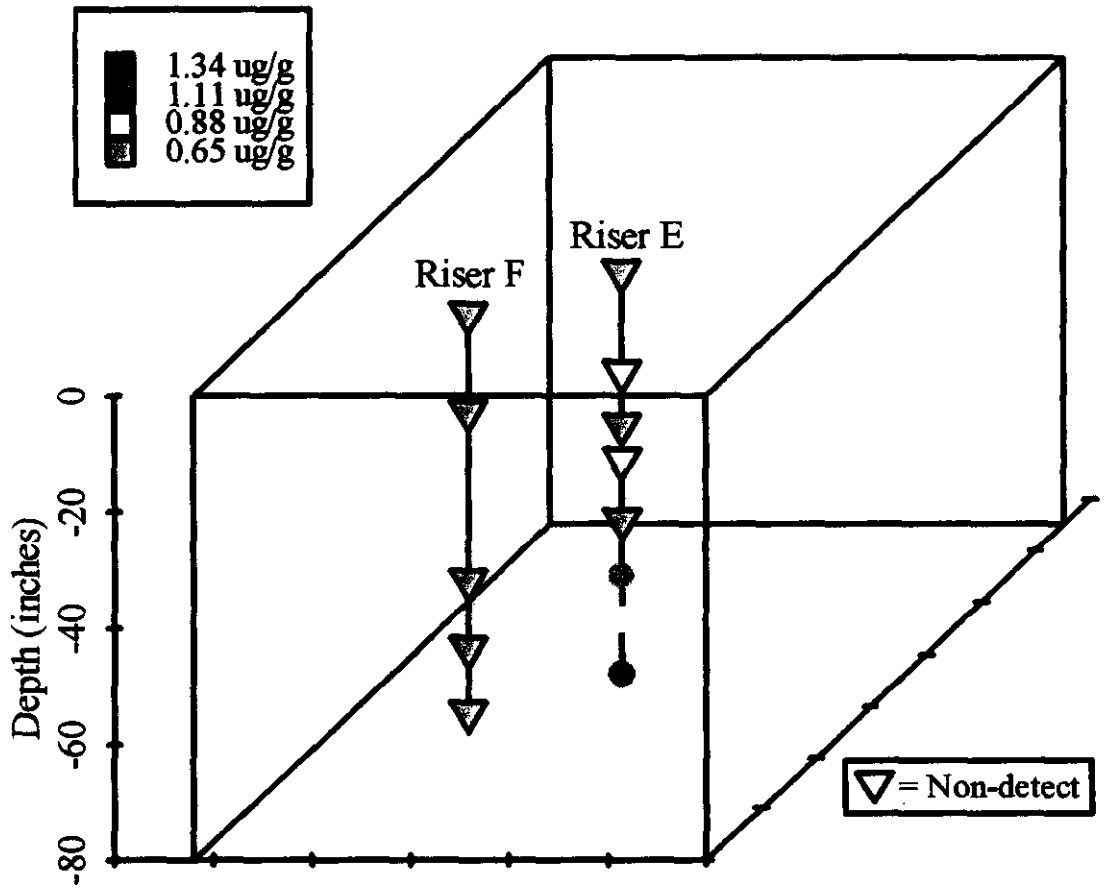
Figure 3-65. Exploratory Data Analysis for Chromium



3.9.5.9 Cyanide

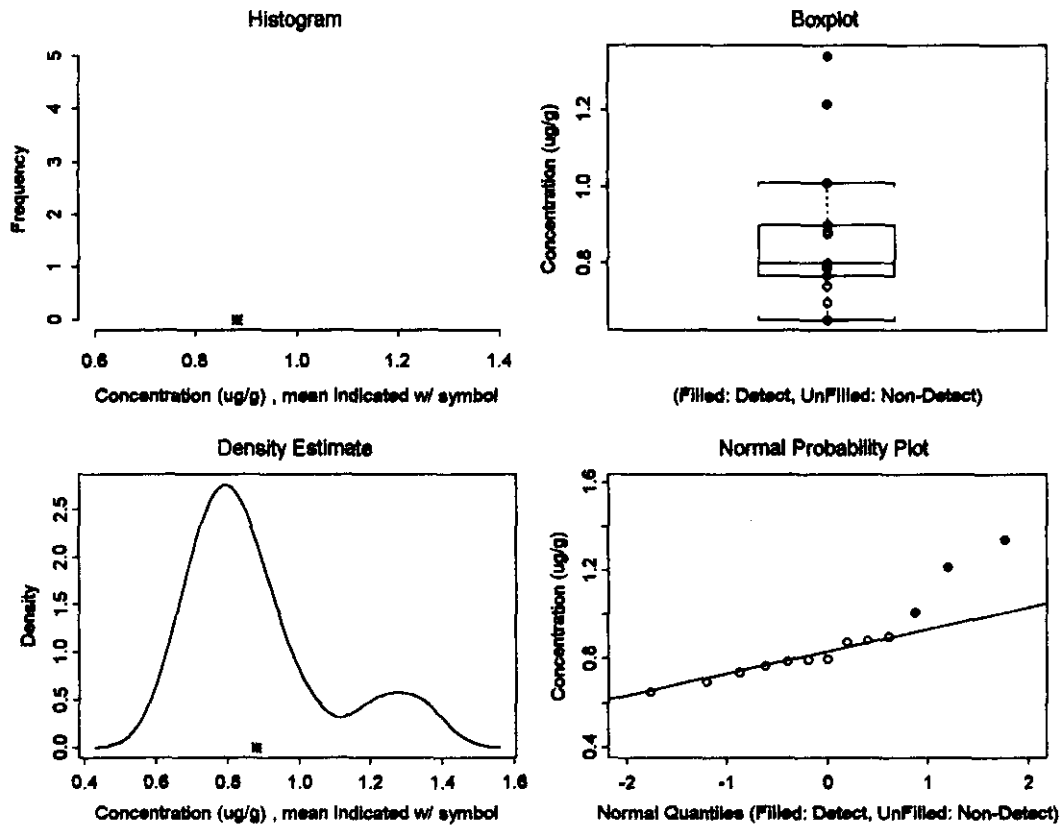
Three of the 13 results for cyanide are detected values. The concentrations for the detected values range from 1.010 µg/g to 1.340 µg/g. All nondetects were less than the lowest detected value. Figure 3-66 shows that the highest concentrations are found in the composite samples taken from the bottom of the sludge in Riser E.

Figure 3-66. Composite Sample Results for Cyanide



The exploratory data plots are shown in Figure 3-67. Because there are only three detected values for cyanide, it is difficult to make any assertions about the distribution of concentrations in the sludge. The distribution of the nondetects appears to follow a normal distribution.

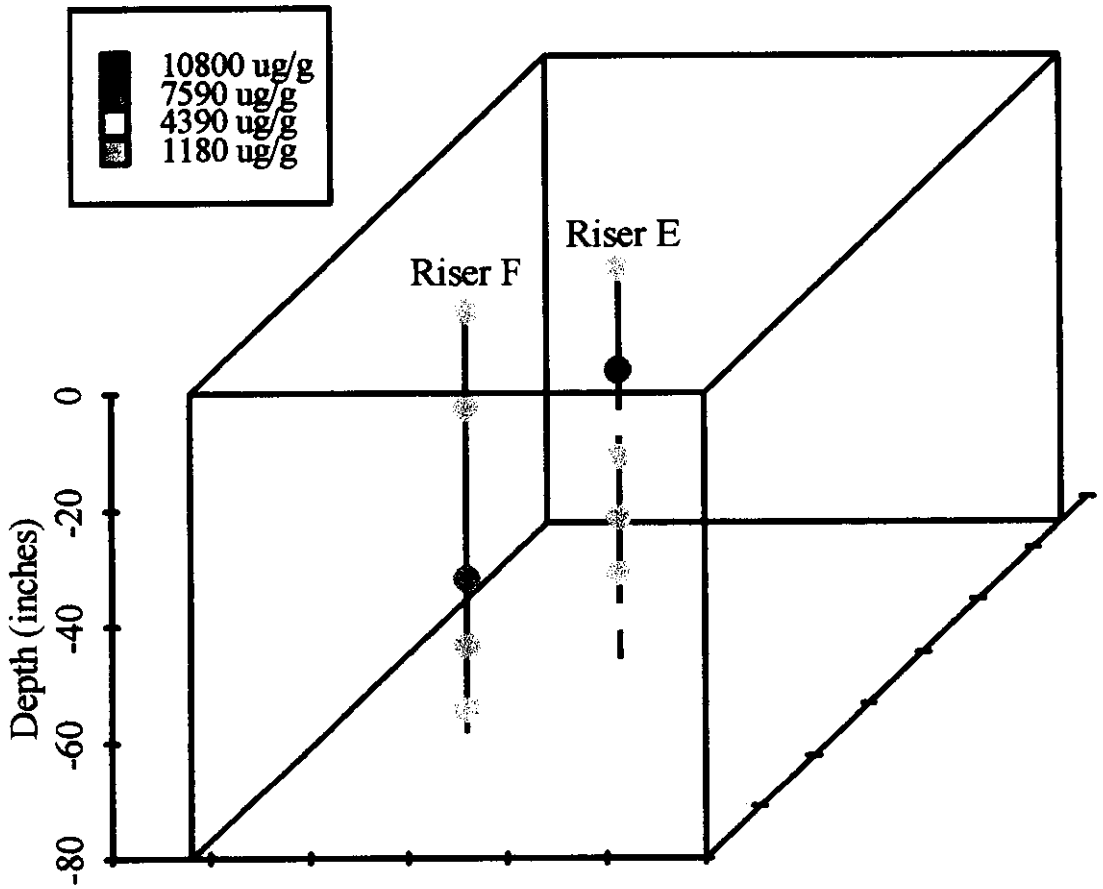
Figure 3-67. Exploratory Data Analysis for Cyanide



3.9.5.10 Fluoride

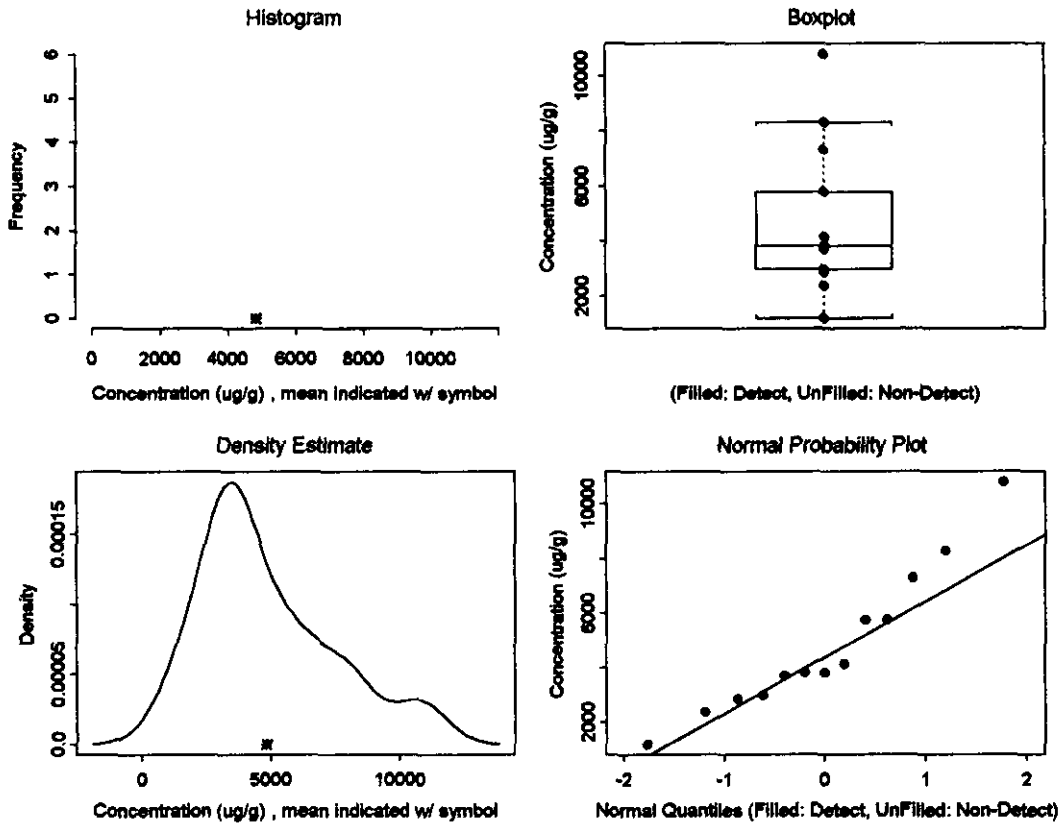
All results for fluoride are detected values. The concentrations for fluoride range from 1180.0 μg/g to 10,800.0 μg/g. Figure 3-68 shows that the highest concentrations are found in the composite samples taken from the middle of the sludge in Riser F, and from slightly above the middle of the sludge in Riser E.

Figure 3-68. Composite Sample Results for Fluoride



The exploratory data plots are shown in Figure 3-69. The distribution of concentrations for fluoride is skewed to the right. The results do not appear to be well approximated by a normal distribution. There is one outlier in the data.

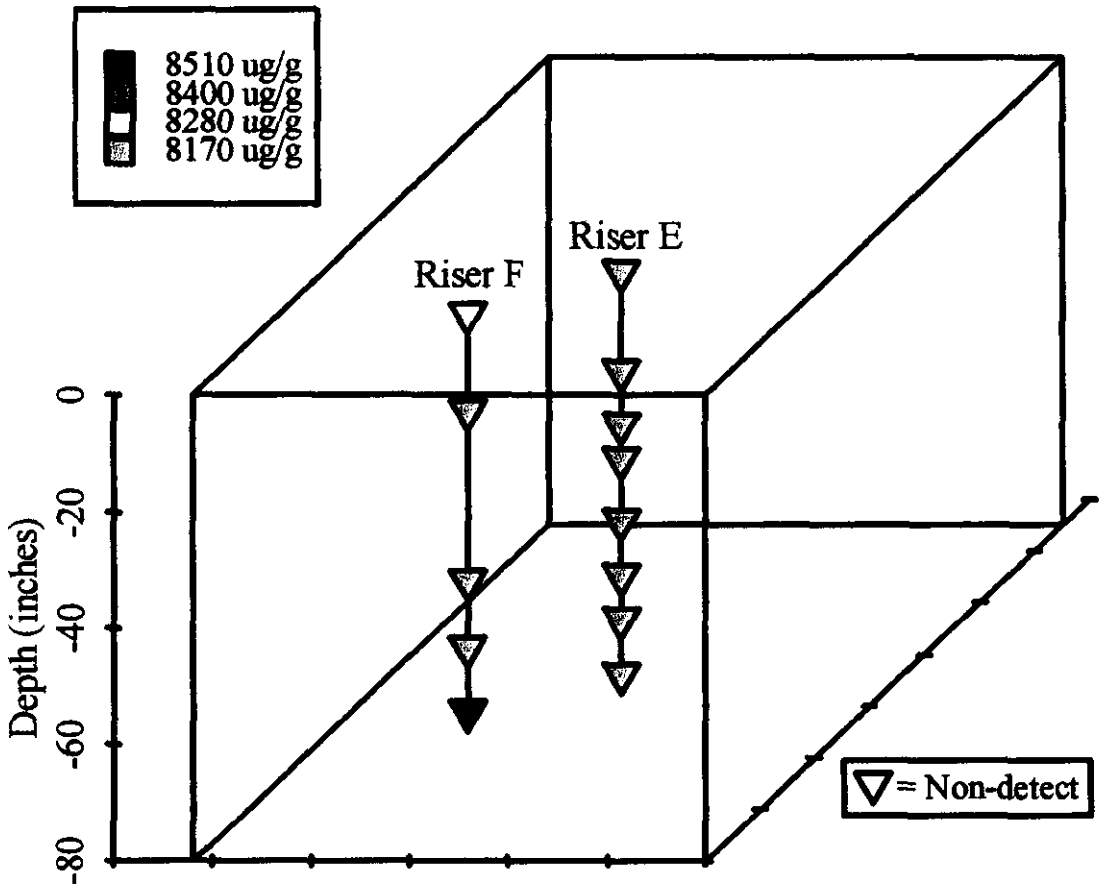
Figure 3-69. Exploratory Data Analysis for Fluoride



3.9.5.11 Hydroxide

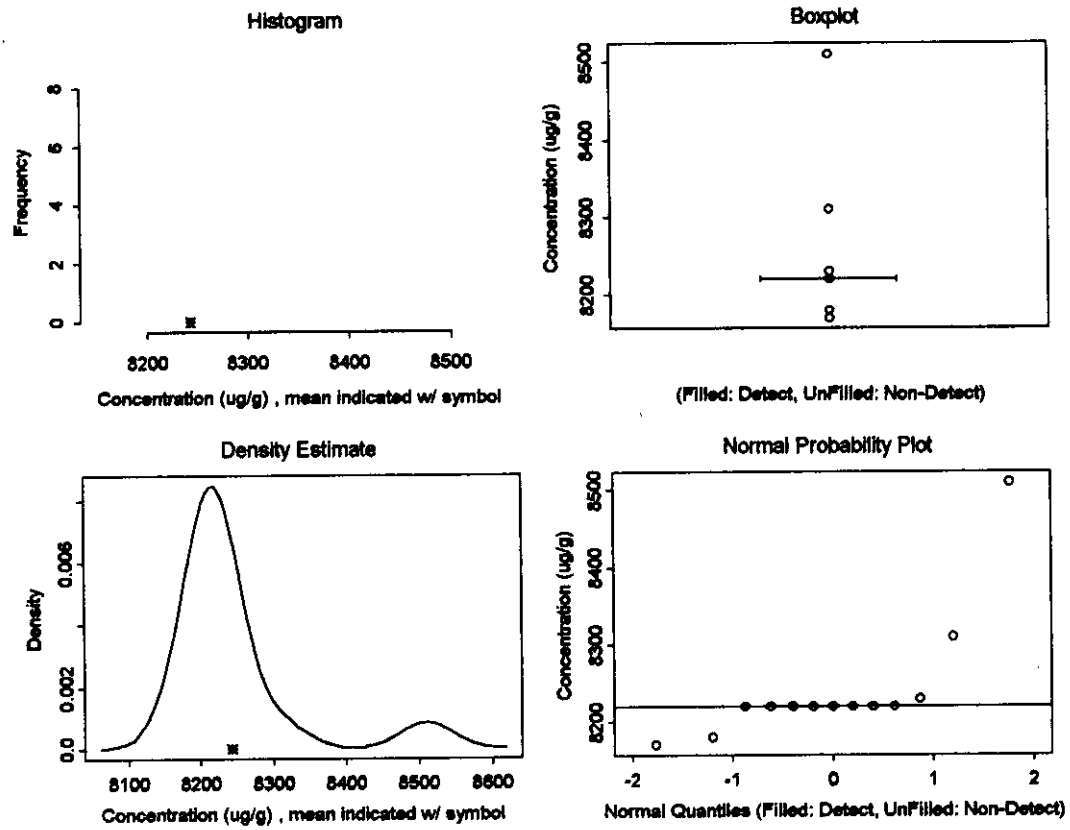
The composite sample results are shown in Figure 3-70. None of the results for hydroxide are detected values. The detection limits range from 8170.0 μg/g to 8510.0 μg/g.

Figure 3-70. Composite Sample Results for Hydroxide



The exploratory data plots are shown in Figure 3-71. Because none of the results are detected values, it is difficult to make any statements about the distribution of hydroxide.

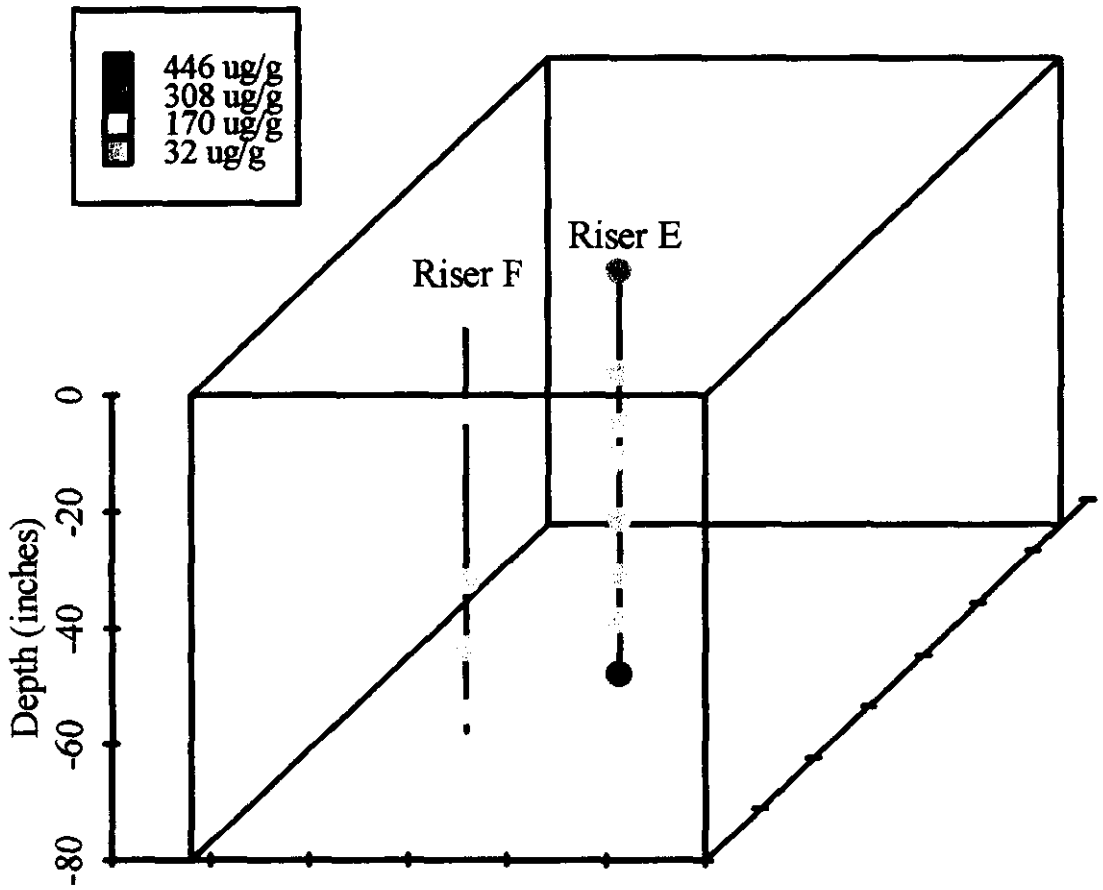
Figure 3-71. Exploratory Data Analysis for Hydroxide



3.9.5.12 Lead

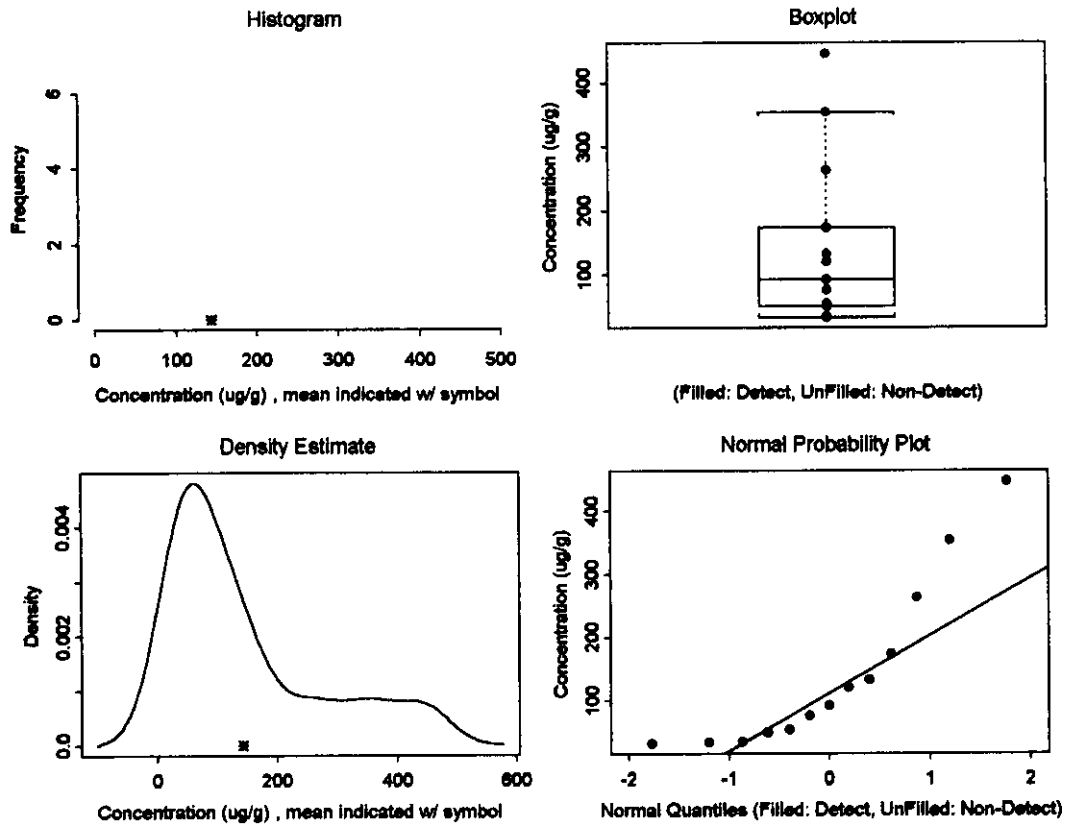
All results for lead are detected values. The concentrations for range from 32.0 μg/g to 446.0 μg/g. Figure 3-72 shows that the highest concentrations are found in the composite samples taken from the top and bottom of the sludge in Riser E.

Figure 3-72. Composite Sample Results for Lead



The exploratory data plots are shown in Figure 3-73. The distribution of concentrations for lead is skewed to the right. The results do not appear to be well approximated by a normal distribution. There is one outlier in the data.

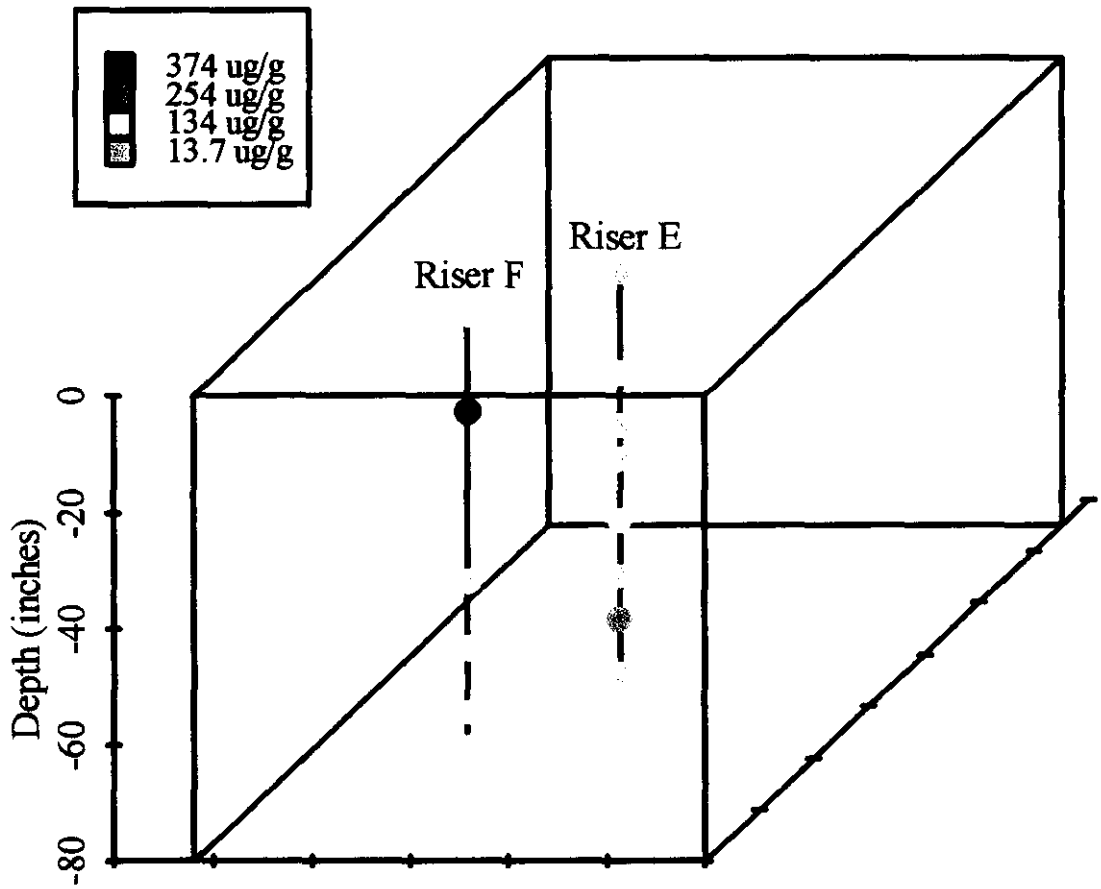
Figure 3-73. Exploratory Data Analysis for Lead



3.9.5.13 Lithium

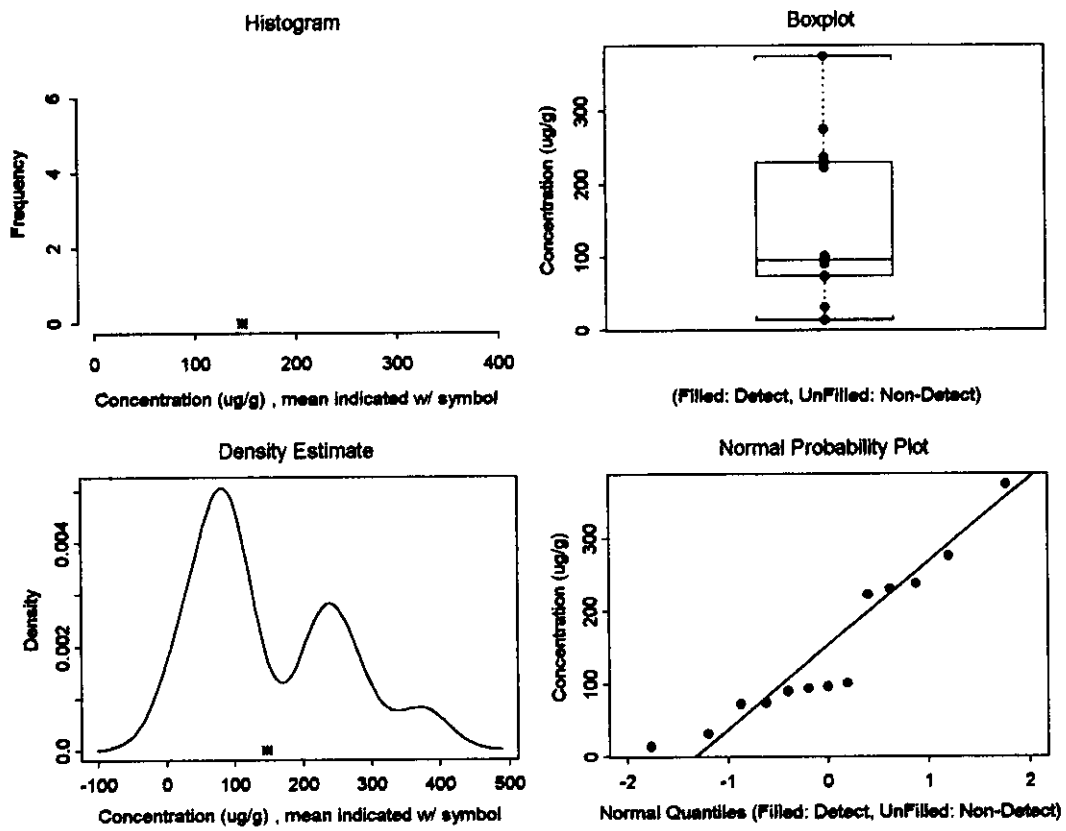
All 13 results for lithium are detected values. The concentrations for lithium range from 13.650 µg/g to 374.0 µg/g. Figure 3-74 shows that the highest concentration is found in the composite sample taken from 25 in below the surface of the sludge in Riser F.

Figure 3-74. Composite Sample Results for Lithium



The exploratory data plots are shown in Figure 3-75. The distribution of concentrations for lithium is skewed to the right and possibly bimodal. The results do not appear to be well approximated by a normal distribution. There are no outliers in the data.

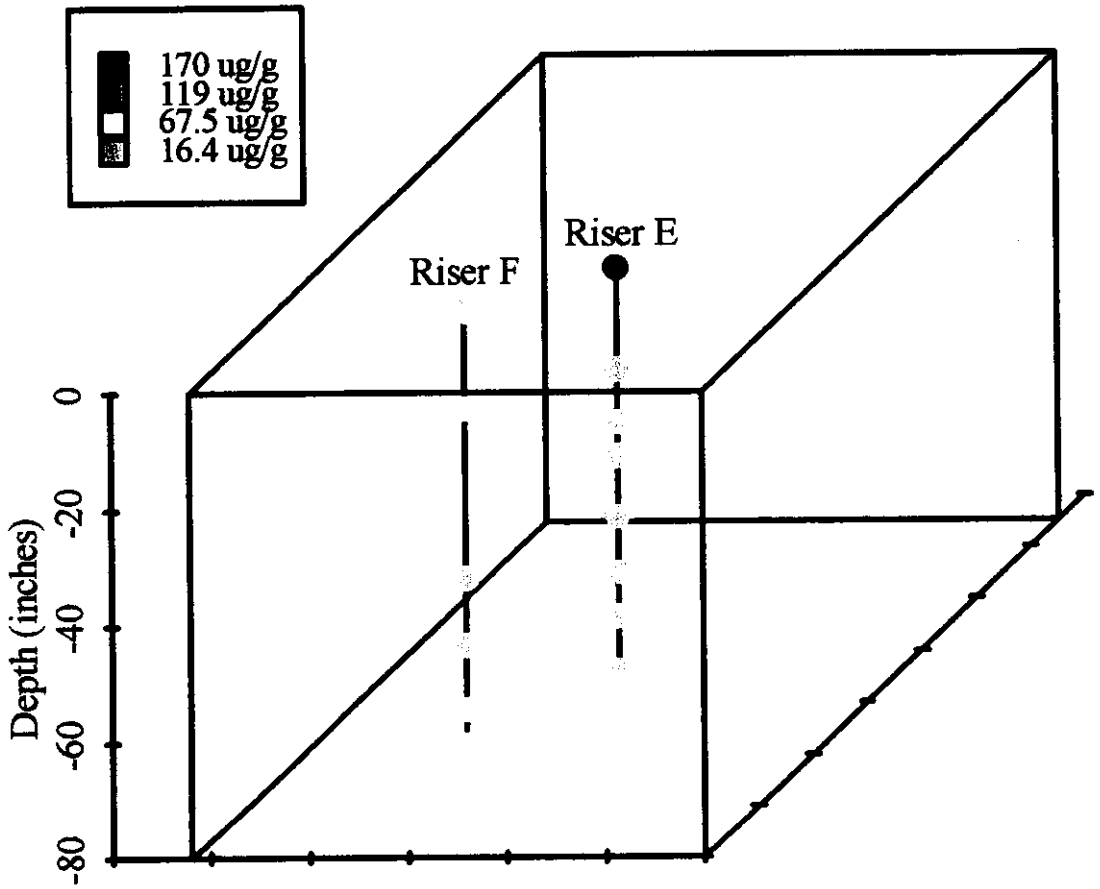
Figure 3-75. Exploratory Data Analysis for Lithium



3.9.5.14 Mercury

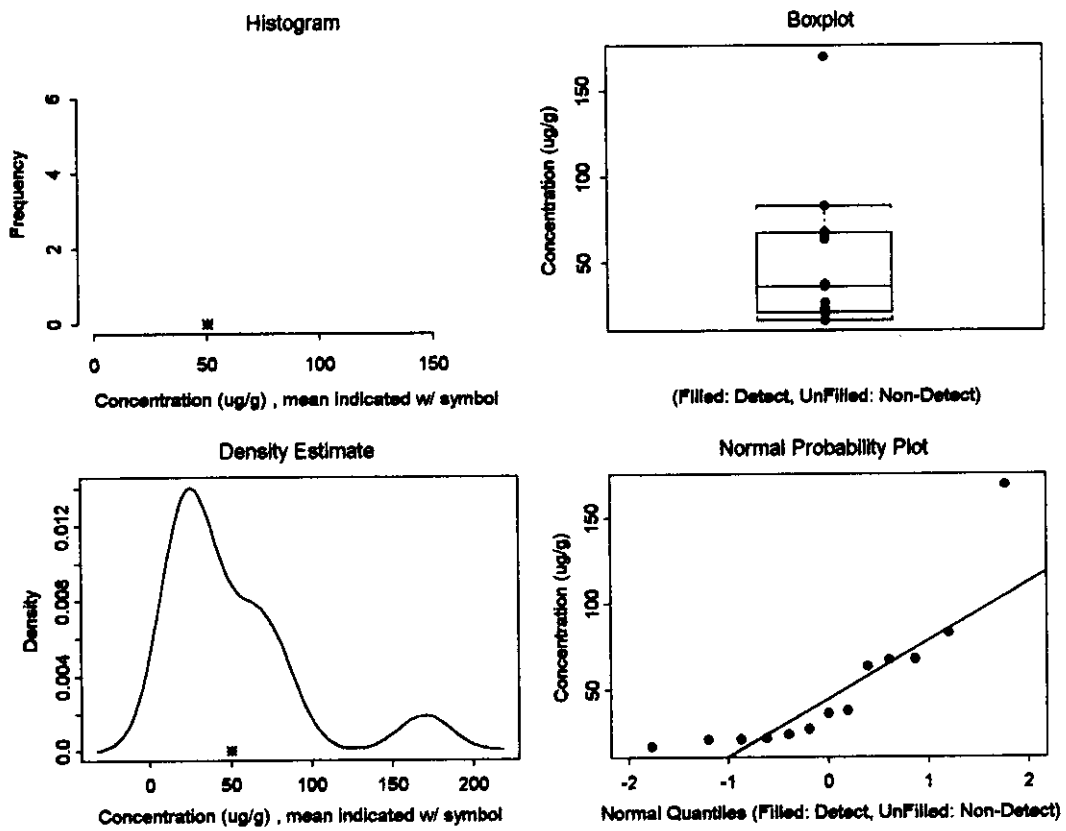
All 13 results for mercury are detected values. The concentrations for mercury range from 16.380 µg/g to 169.60 µg/g. Figure 3-76 shows that the highest concentration is found in the composite samples taken from the top of the sludge in Riser E.

Figure 3-76. Composite Sample Results for Mercury



The exploratory data plots are shown in Figure 3-77. The distribution of concentrations for mercury is skewed to the right. The results do not appear to be well approximated by a normal distribution. There is one outlier in the data.

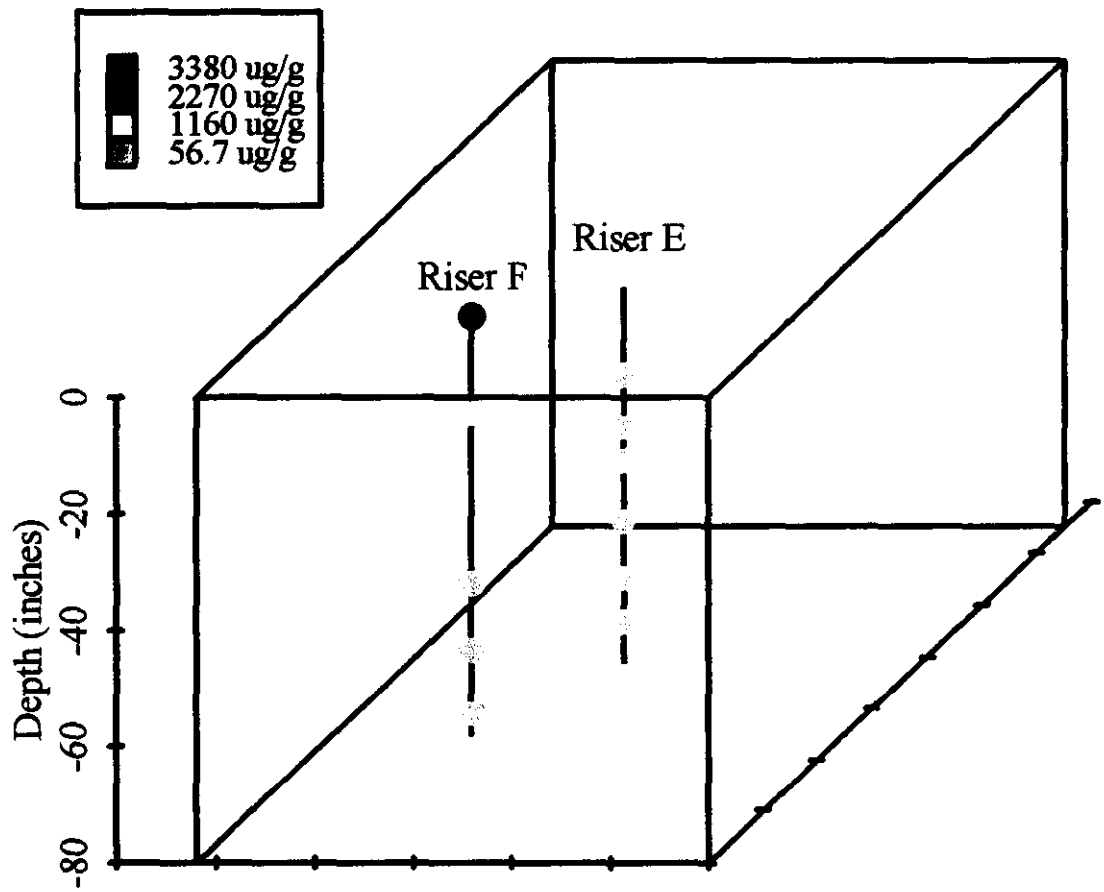
Figure 3-77. Exploratory Data Analysis for Mercury



3.9.5.15 Nickel

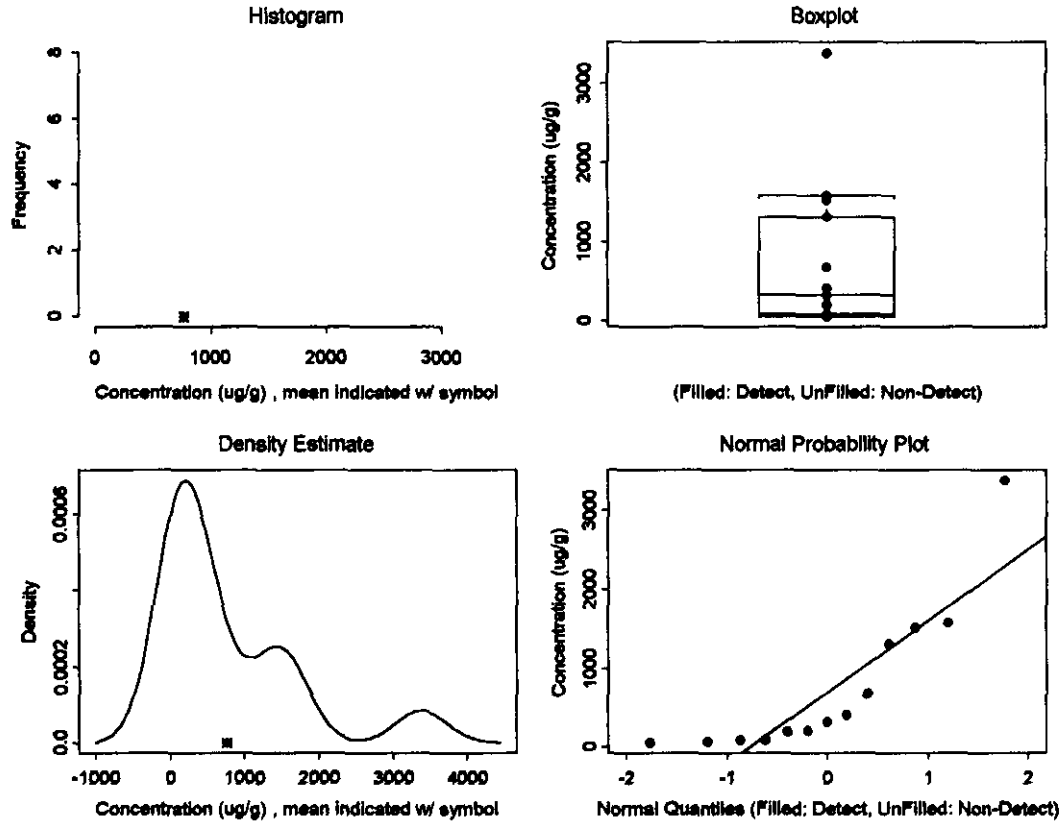
All 13 results for nickel are detected values. The concentrations for nickel range from 56.70 µg/g to 3380.0 µg/g. Figure 3-78 shows that the highest concentrations are found in the composite samples taken from the top of the sludge in Riser F and the composite samples taken from both the top and bottom of the sludge in Riser E.

Figure 3-78. Composite Sample Results for Nickel



The exploratory data plots are shown in Figure 3-79. The distribution of concentrations for nickel is skewed. The results do not appear to be well approximated by a normal distribution. There is one outlier in the data.

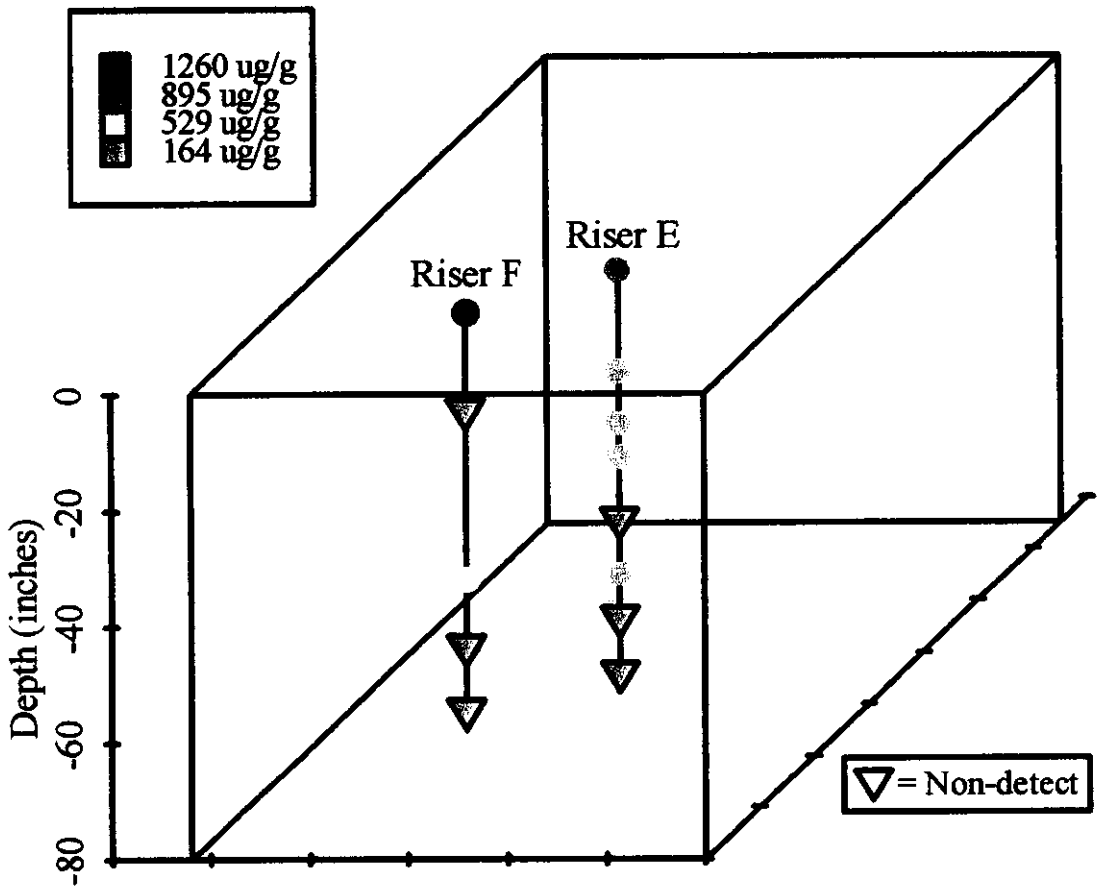
Figure 3-79. Exploratory Data Analysis for Nickel



3.9.5.16 Nitrate

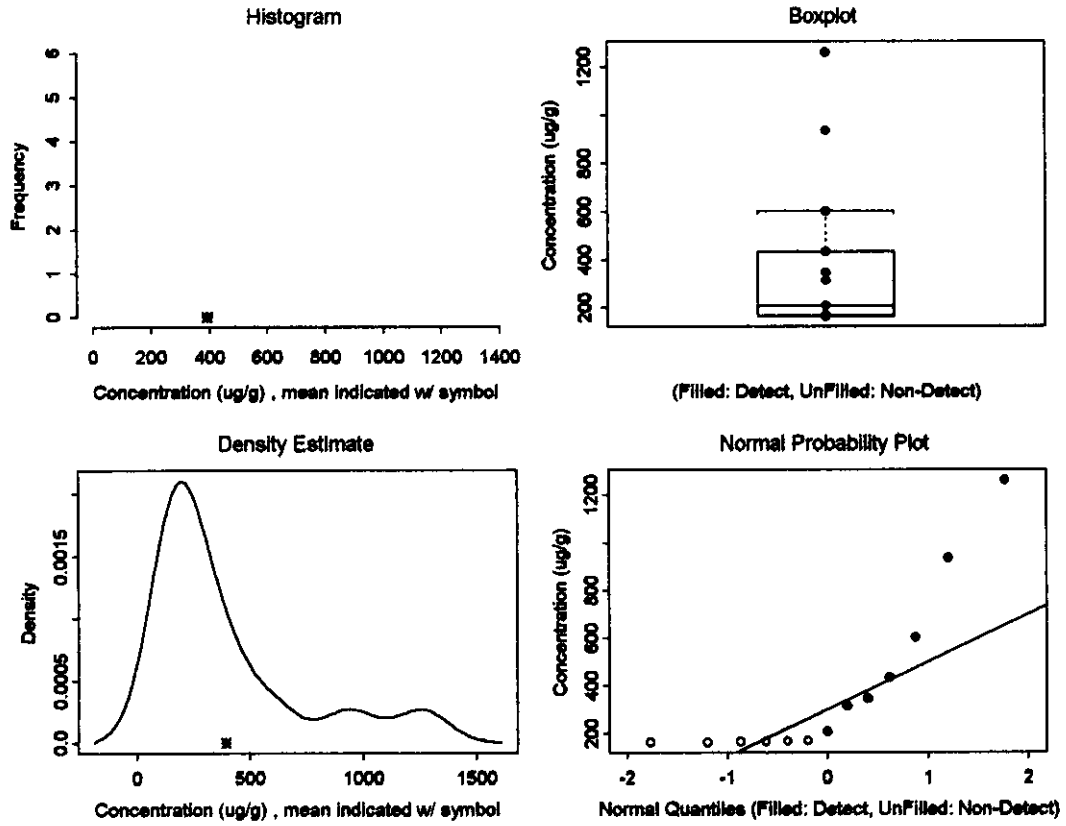
Seven of the 13 results for nitrate are detected values. The concentrations for the detected values range from 209.0 µg/g to 1260.0 µg/g. The detection limits range from 164.0 µg/g to 170.0 µg/g. Figure 3-80 shows that the highest concentrations are found in the composite samples taken from the top of the sludge in both risers.

Figure 3-80. Composite Sample Results for Nitrate



The exploratory data plots are shown in Figure 3-81. The distribution of concentrations for nitrate is skewed to the right. As a whole, the results do not appear to be well approximated by a normal distribution, although from the Normal Probability plot, it seems that the detected values could be reasonably modeled with a normal distribution. There are two outliers in the data.

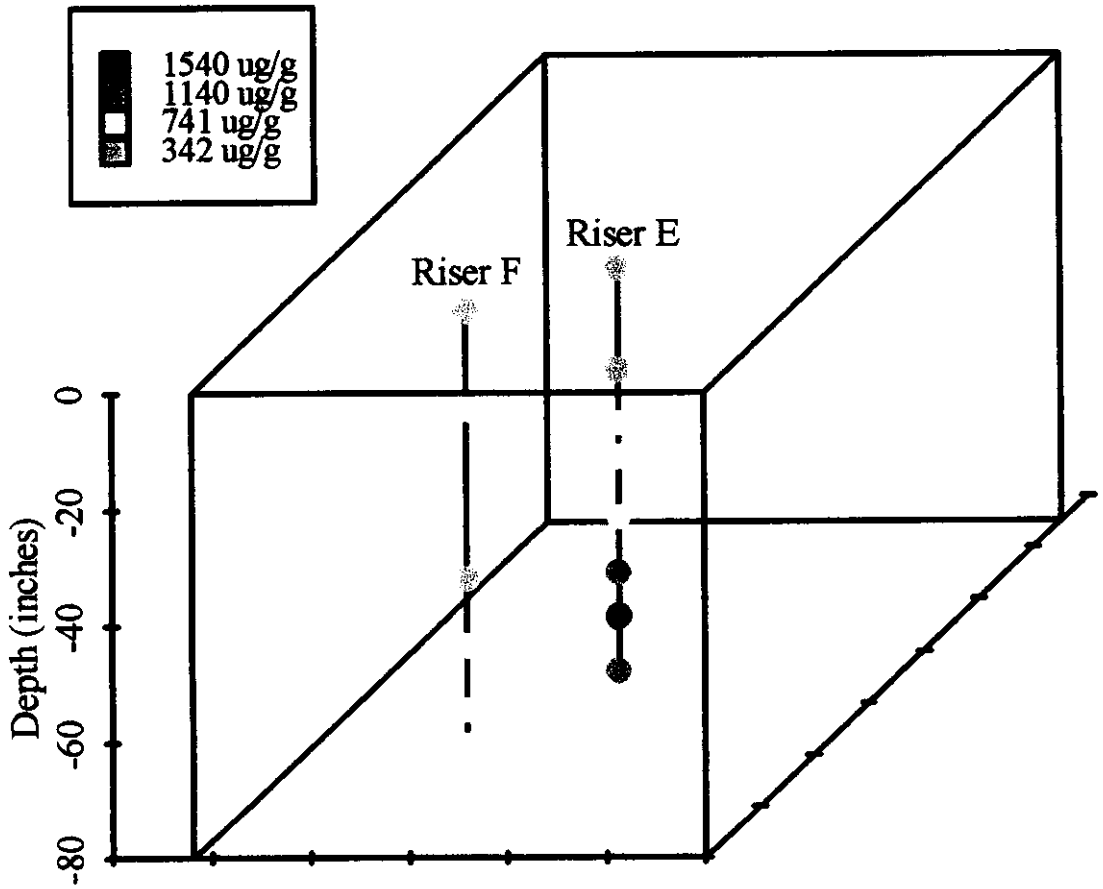
Figure 3-81. Exploratory Data Analysis for Nitrate



3.9.5.17 Nitrite

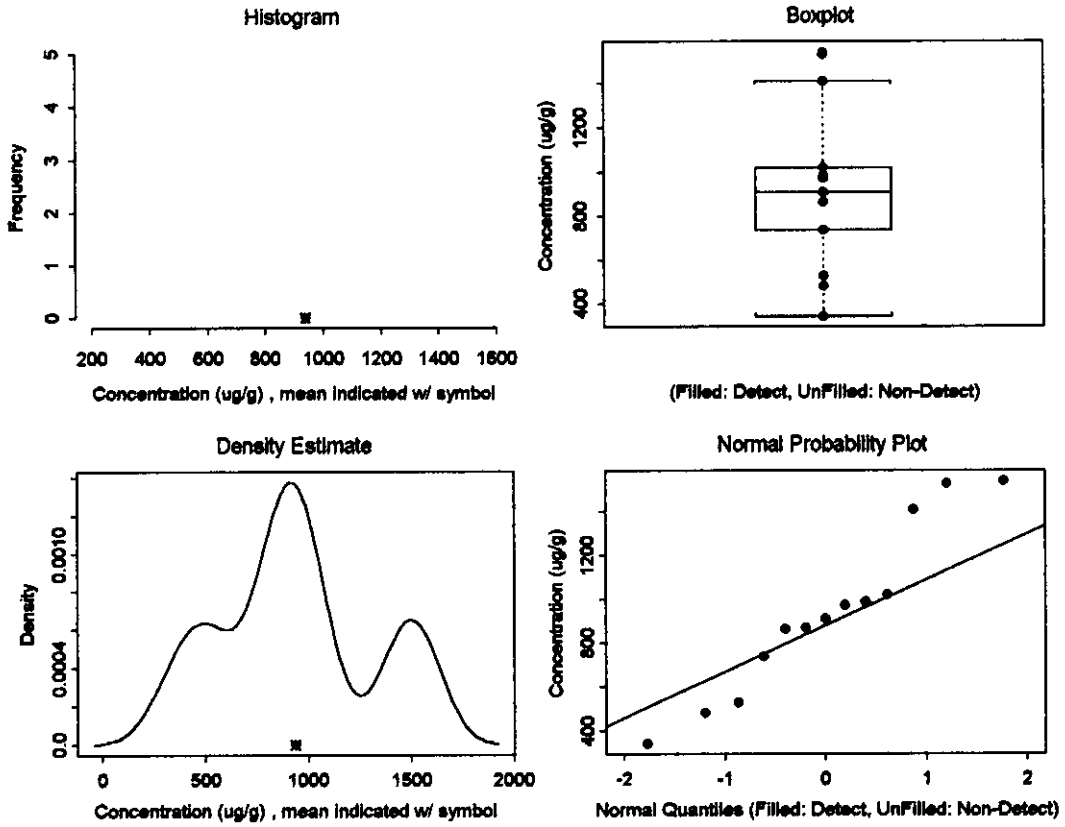
All 13 results for nitrite are detected values. The concentrations for nitrite range from 342.10 μg/g to 1540.0 μg/g. Figure 3-82 shows that the highest concentrations are found in the composite samples taken from the bottom of the sludge in Riser E.

Figure 3-82. Composite Sample Results for Nitrite



The exploratory data plots are shown in Figure 3-83. The distribution of concentrations for nitrite appears bimodal. The results do not appear to be well approximated by a normal distribution. There is one outlier in the data.

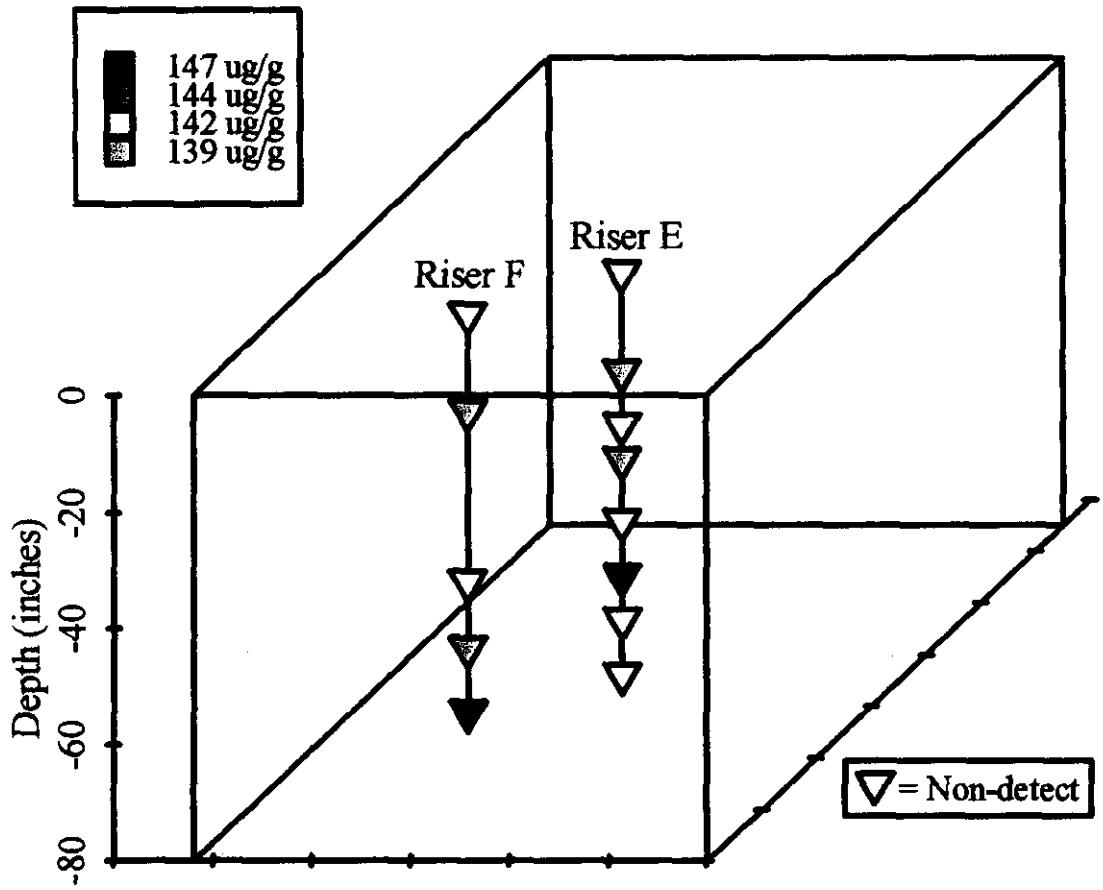
Figure 3-83. Exploratory Data Analysis for Nitrite



3.9.5.18 Phosphate

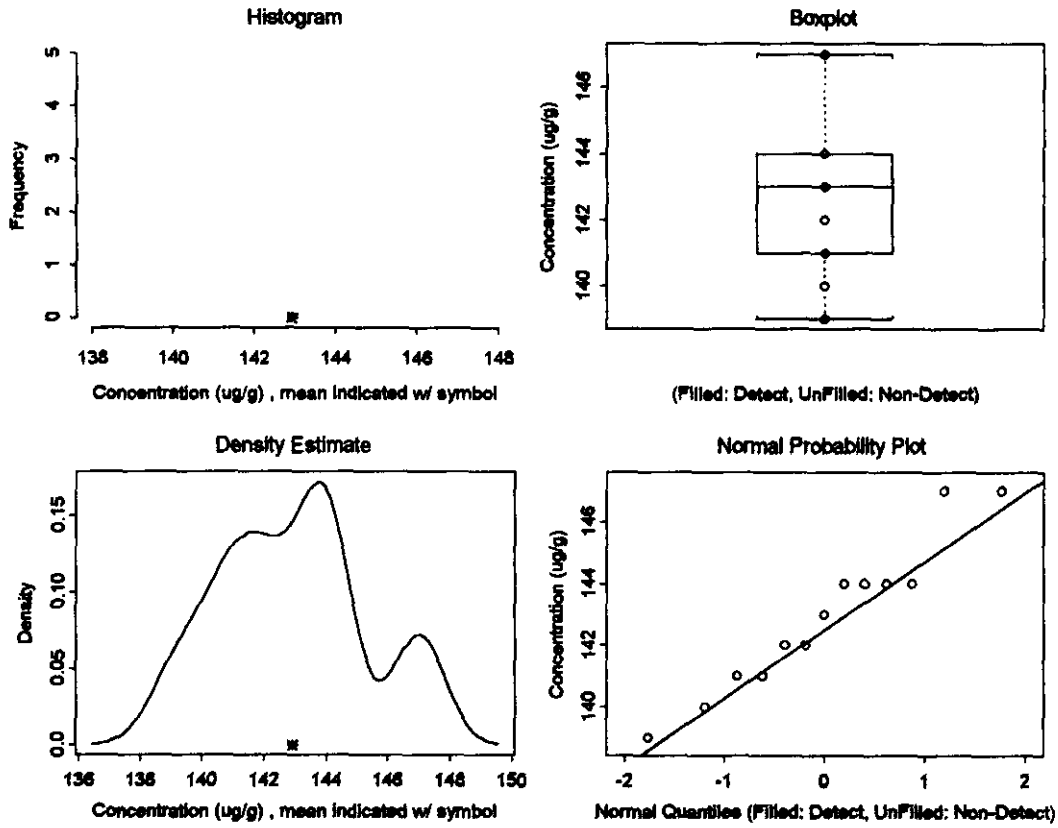
The composite sample results are shown in Figure 3-84. None of the thirteen results for phosphate are detected values. The detection limits for phosphate range from 139.0 µg/g to 147.0 µg/g.

Figure 3-84. Composite Sample Results for Phosphate



The exploratory data plots are shown in Figure 3-85. The distribution of *detection limits* for phosphate is reasonably approximated by a normal distribution.

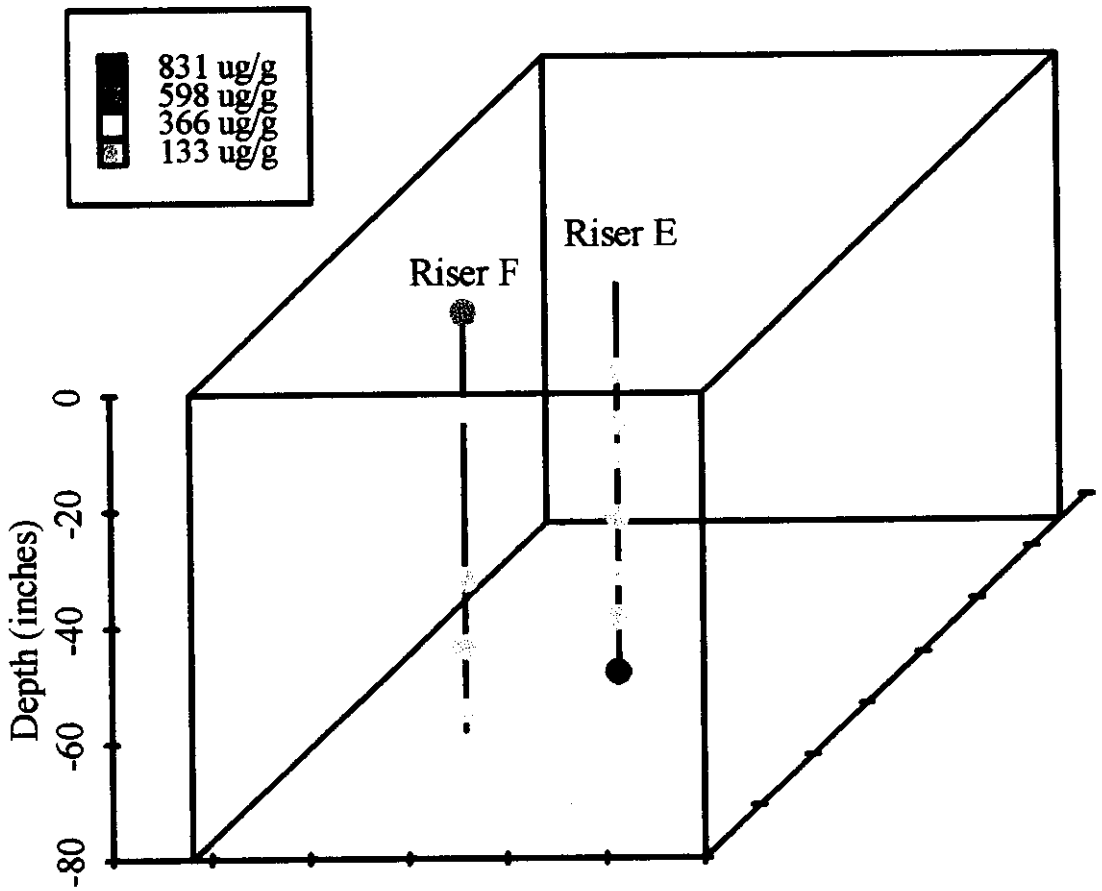
Figure 3-85. Exploratory Data Analysis for Phosphate



3.9.5.19 Phosphorus

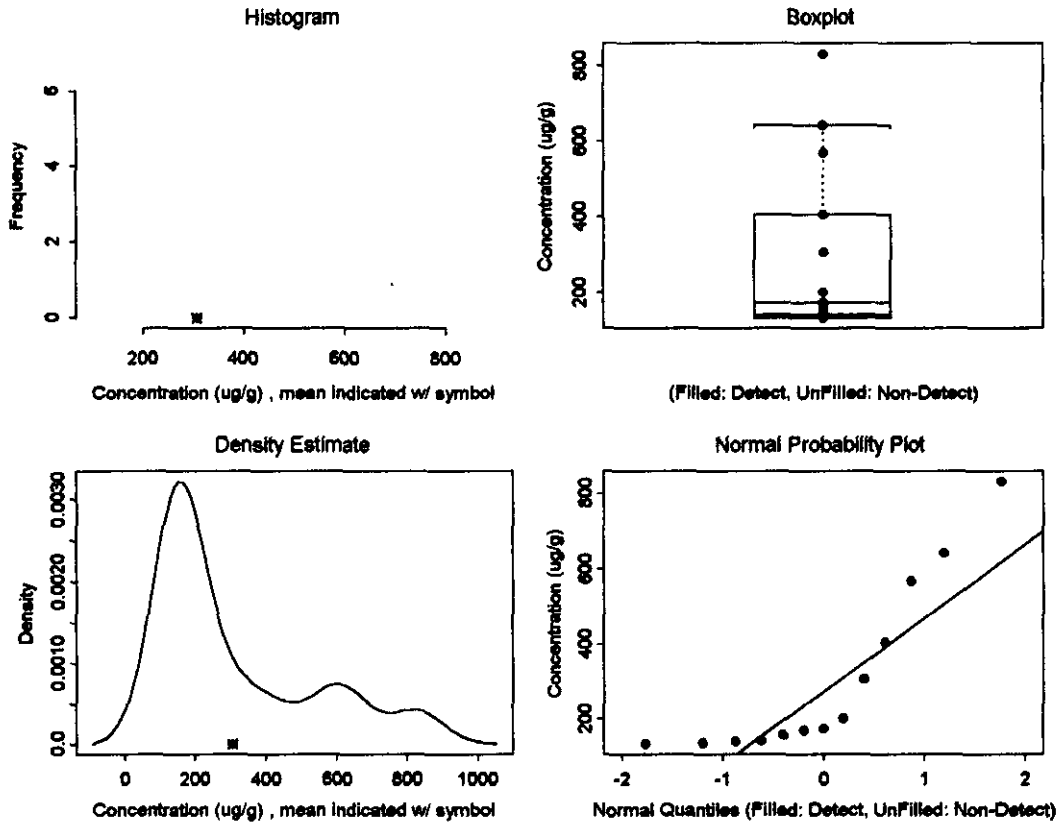
All 13 results for phosphorus are detected values. The concentrations for phosphorus range from 133.0 μg/g to 831.0 μg/g. Figure 3-86 shows that the highest concentrations are found in the composite samples taken from the top of the sludge in both risers and the bottom of the sludge in Riser E.

Figure 3-86. Composite Sample Results for Phosphorus



The exploratory data plots are shown in Figure 3-87. The distribution of concentrations for phosphorus is skewed to the right. The results do not appear to be well approximated by a normal distribution. There is one outlier in the data.

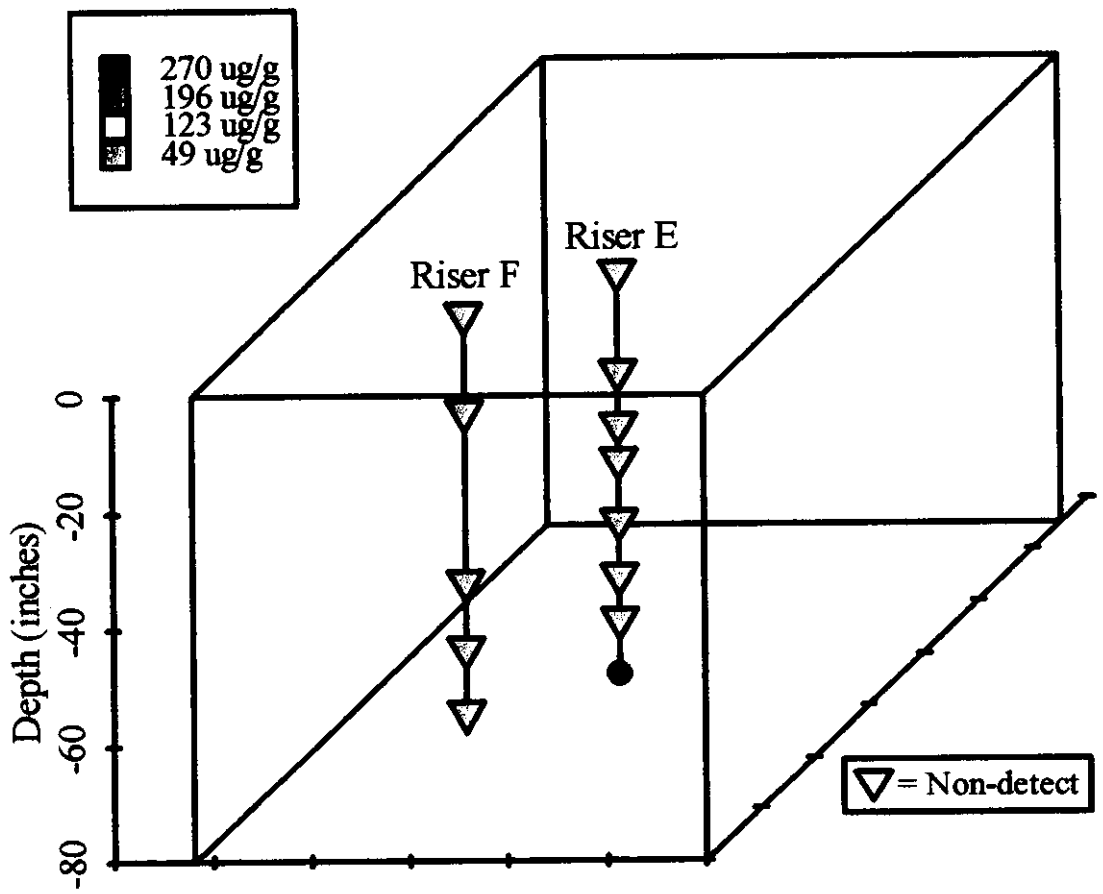
Figure 3-87. Exploratory Data Analysis for Phosphorus



3.9.5.20 Potassium

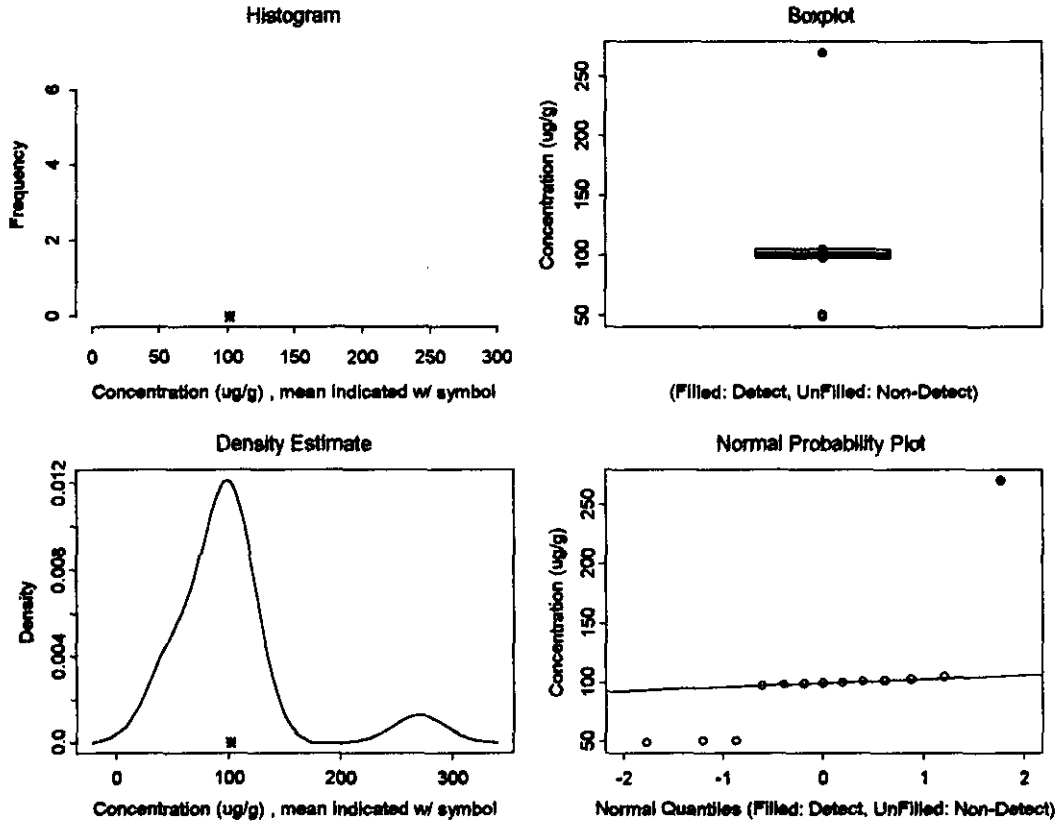
Only one of the 13 results for potassium is a detected value. The detected concentration for potassium is 270.0 μg/g. The detection limits for the other samples range from 49 μg/g to 105 μg/g. Figure 3-88 shows that this detected concentration is found in the composite sample taken from the bottom of the sludge in Riser E.

Figure 3-88. Composite Sample Results for Potassium



The exploratory data plots are shown in Figure 3-89. Because only one value was detected for potassium, there is little to be said about the distribution.

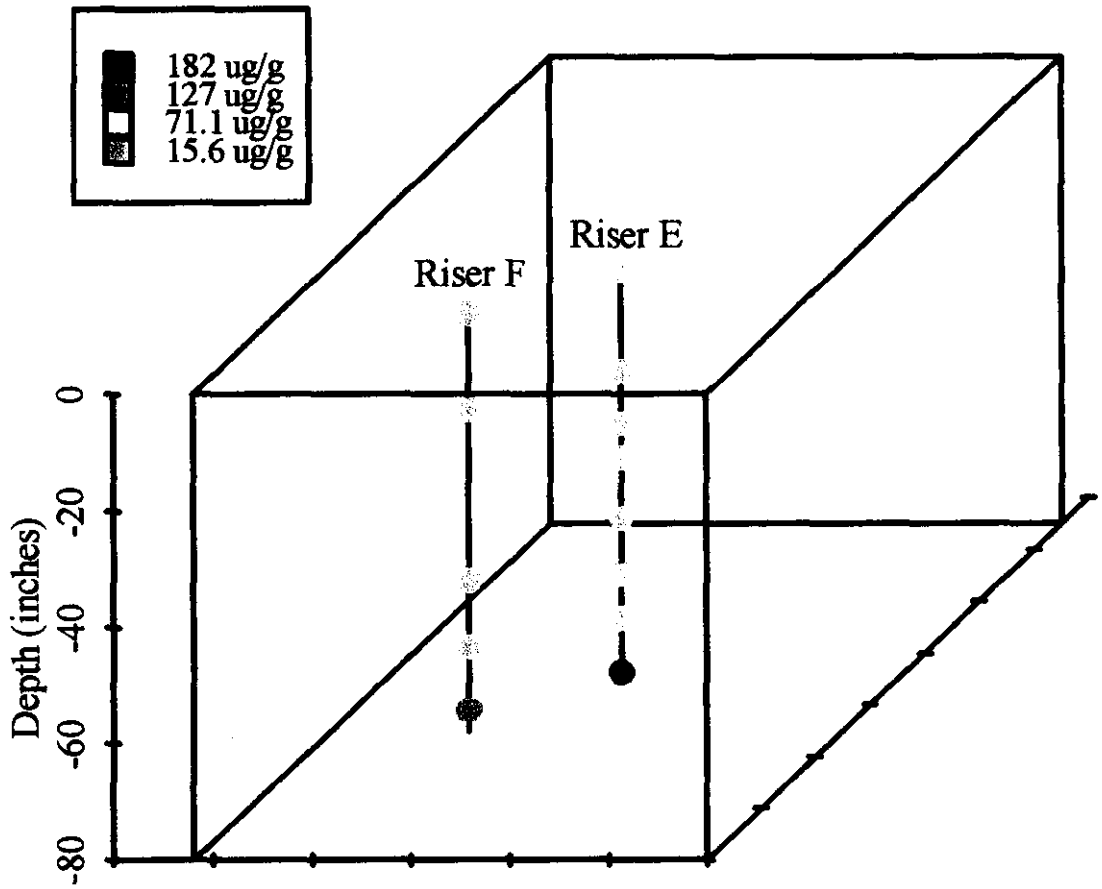
Figure 3-89. Exploratory Data Analysis for Potassium



3.9.5.21 Silver

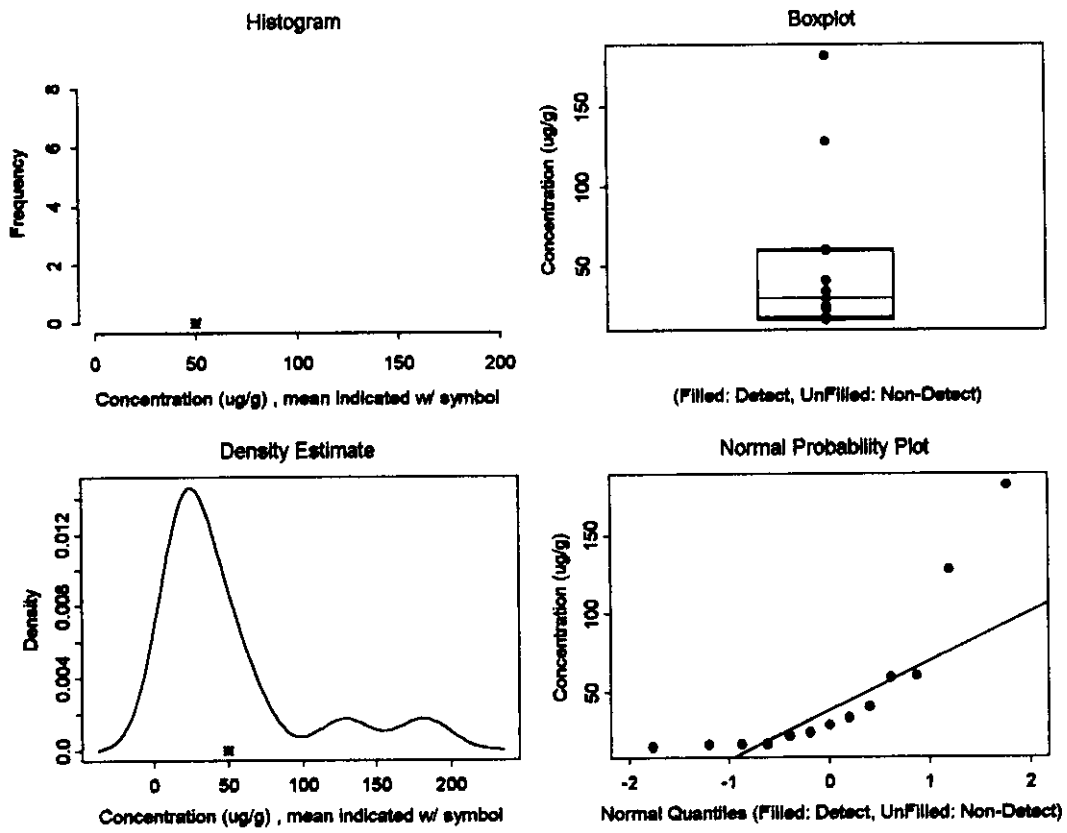
All 13 results for silver are detected values. The concentrations for silver range from 15.60 μg/g to 182.0 μg/g. Figure 3-90 shows that the highest concentrations are found in the composite samples taken from the bottom of the sludge in both risers.

Figure 3-90. Composite Sample Results for Silver



The exploratory data plots are shown in Figure 3-91. The distribution of concentrations for silver is skewed. The results do not appear to be well approximated by a normal distribution. There are two outliers in the data.

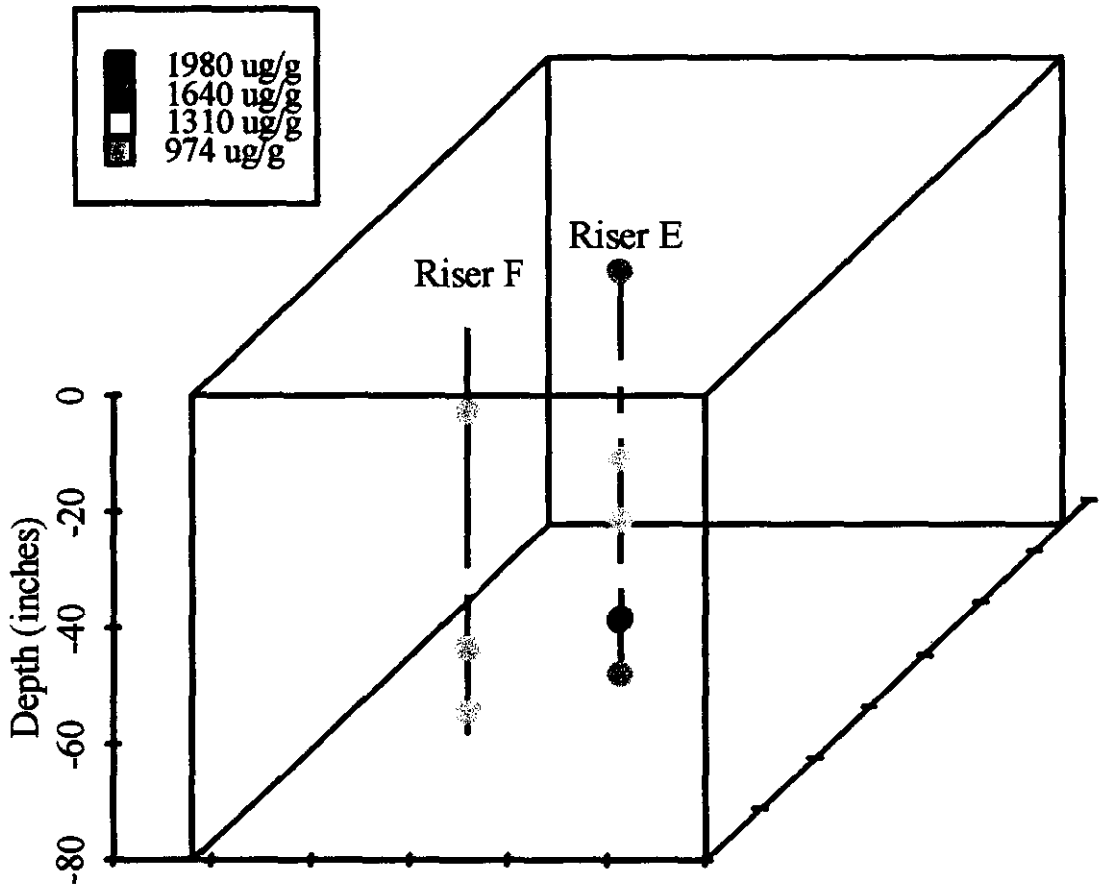
Figure 3-91. Exploratory Data Analysis for Silver



3.9.5.22 Sulfate

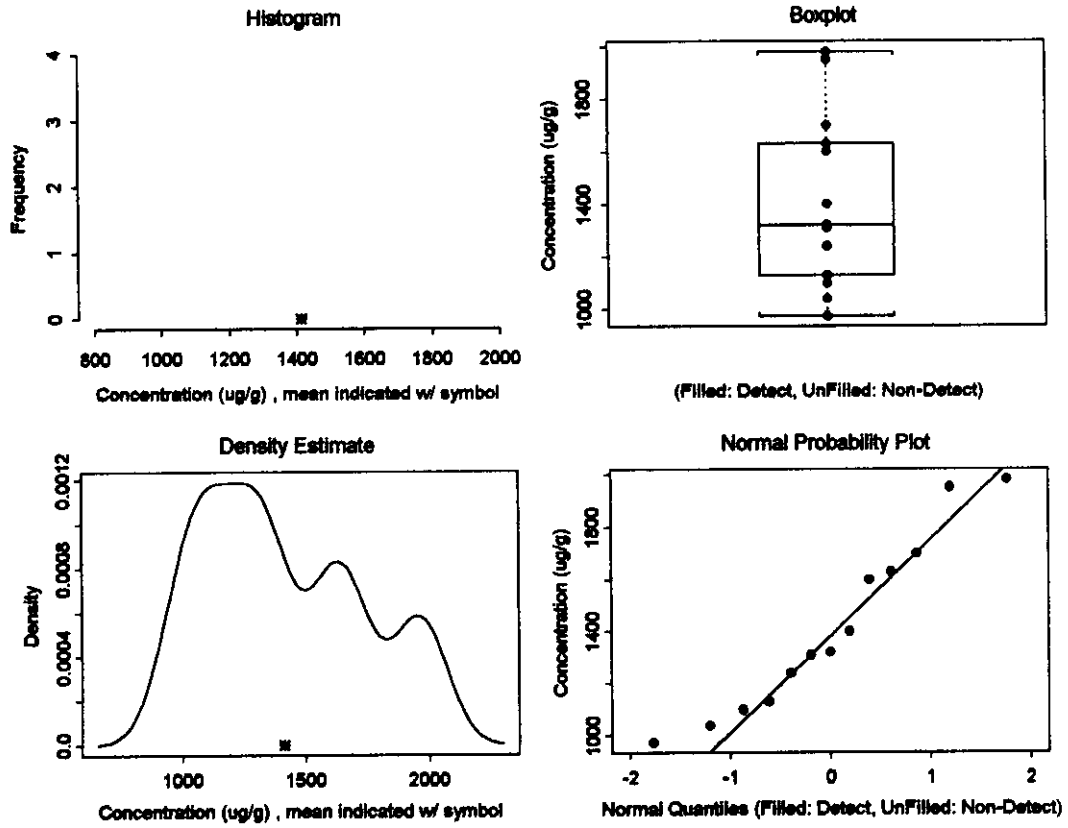
All 13 results for sulfate are detected values. The concentrations for sulfate range from 974.0 µg/g to 1980.0 µg/g. Figure 3-92 shows that the highest concentrations are found in the composite samples taken from near the bottom of the sludge in Riser E.

Figure 3-92. Composite Sample Results for Sulfate



The exploratory data plots are shown in Figure 3-93. The distribution of concentrations for sulfate is fairly symmetric. There are no outliers in the data.

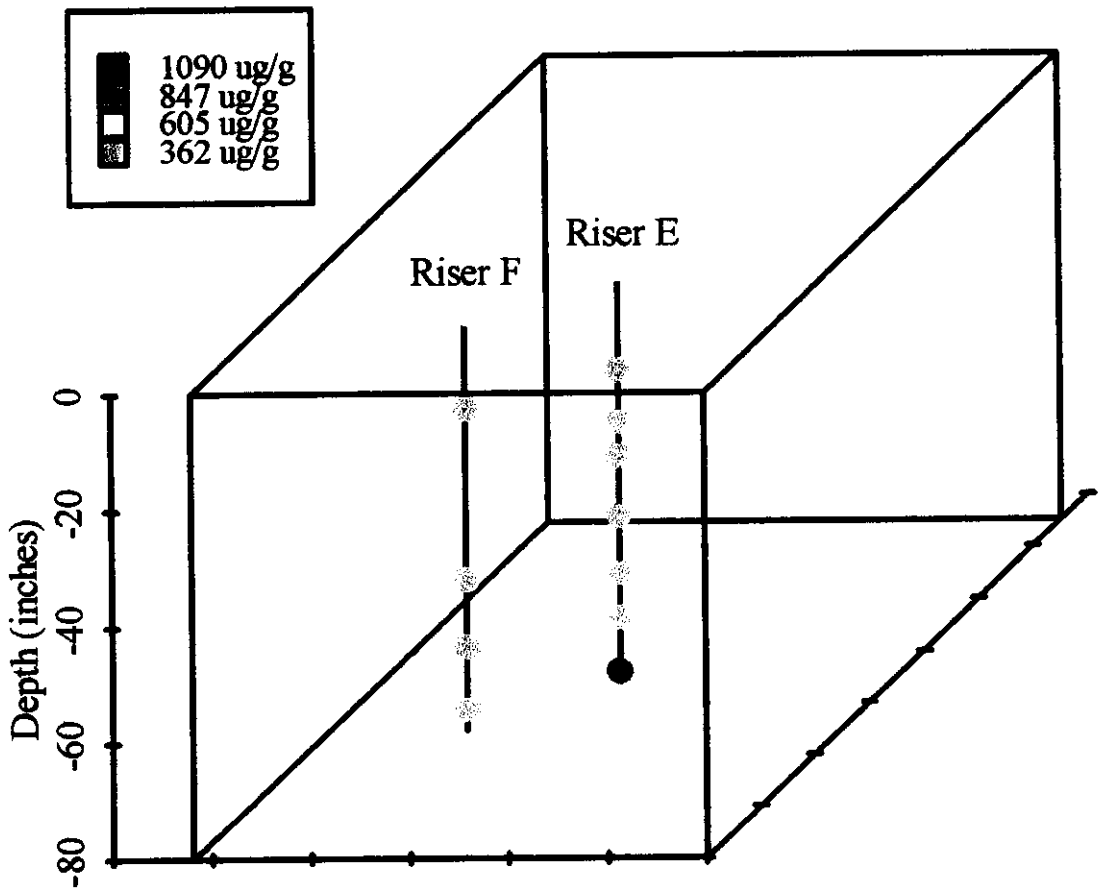
Figure 3-93. Exploratory Data Analysis for Sulfate



3.9.5.23 Sulfur

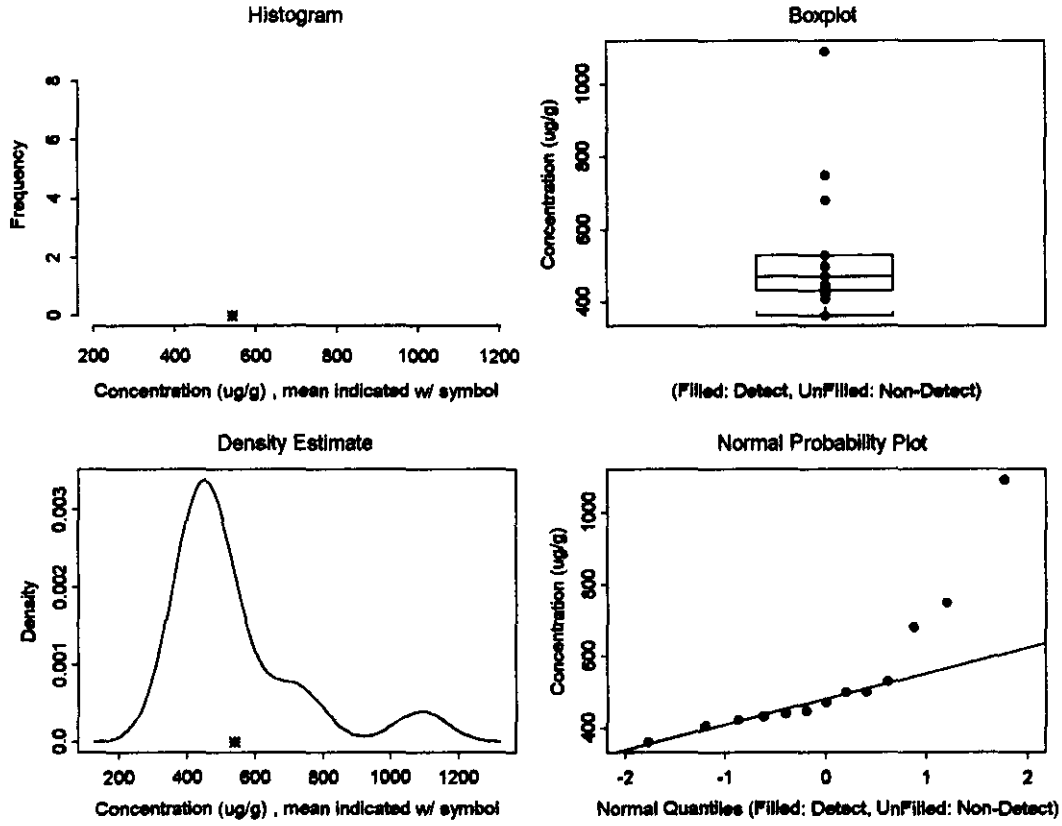
All 13 results for sulfur are detected values. The concentrations for sulfur range from 362.0 µg/g to 1090.0 µg/g. Figure 3-94 shows that the highest concentration is found in the composite sample taken from the bottom of the sludge in Riser E.

Figure 3-94. Composite Sample Results for Sulfur



The exploratory data plots are shown in Figure 3-95. The distribution of concentrations for sulfur is skewed to the right. The results do not appear to be well approximated by a normal distribution. There are three outliers in the data.

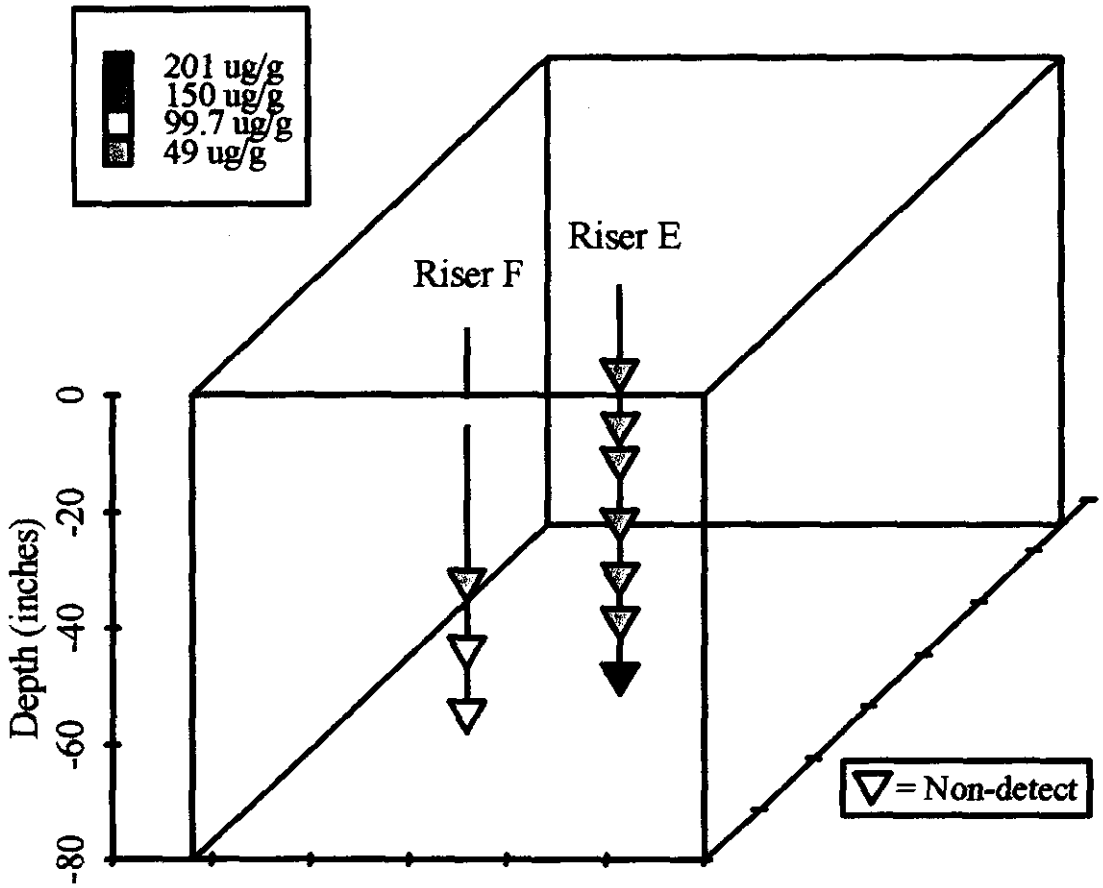
Figure 3-95. Exploratory Data Analysis for Sulfur



3.9.5.24 Uranium

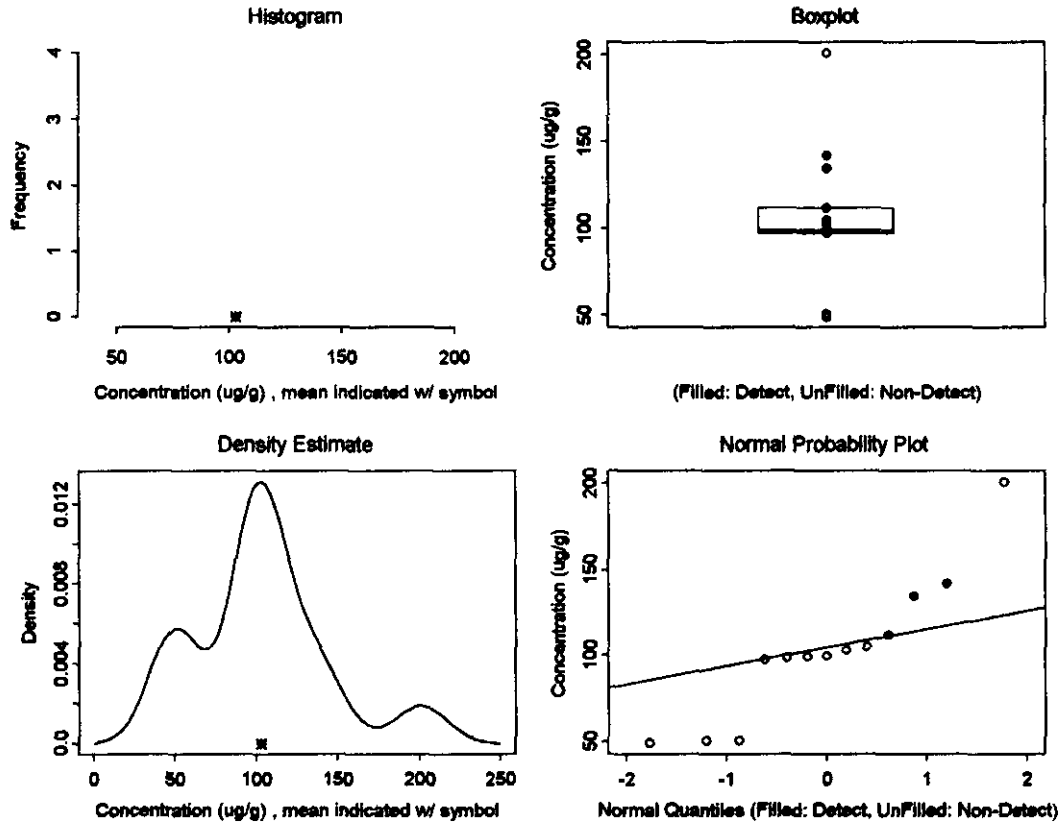
Three of the 13 results for uranium are detected values. Figure 3-96 shows that the detected values are at the top of the tank. Concentrations for the detected values range from 112 µg/g to 142 µg/g. Detection limits for the nondetected values range from 49 µg/g to 201 µg/g.

Figure 3-96. Composite Sample Results for Uranium



The exploratory data plots are shown in Figure 3-97. Because only three of the results for uranium are detected values, it is difficult to make any statements about the distribution.

Figure 3-97. Exploratory Data Analysis for Uranium



3.9.6 Assessment of the Horizontal and Vertical Strata for Nonradiological Inorganic Analytes

Determining the extent of the vertical and horizontal strata of inorganic analytes is difficult due to limited data available for analysis. With only two core samples, horizontal extent can only be qualitatively described in the region between, and to some extent around, the area where the core samples were taken. The vertical profile can only be assessed in the area where there are data as well. This limits the conclusions that can be drawn about the vertical strata to those areas between and around the boreholes. Descriptive statistics and observation will guide the qualitative assessment of the horizontal and vertical strata.

Table 3-65 presents the maximum and minimum detected values for each of the analytes with the corresponding average depth (the midpoint of the depth interval) of the composite sample from which the result is obtained.

Table 3-65. Maximum and Minimum Detected Values Nonradiological for Inorganic Analyte in Tank 241-Z-361*

Analyte	Number of Detects	Min (µg/g)	Depth of Min** (in.)	Max (µg/g)	Depth of Max [a] (in.)
Ammonia/Ammonium	2	625.5	62	649	77
Arsenic	1	23.4	8	23.4	8
Barium	13	85.6	8	197	79
Beryllium	0	NA	NA	NA	NA
Bromide	13	283.4	8	5370	54
Cadmium	13	1.48	62	112	54
Chloride	13	482	53	926.4	62
Chromium	13	691	36	10000	79
Cyanide	3	1.01	70	1.34	79
Fluoride	13	1180	8	10800	27
Hydroxide	0	NA	NA	NA	NA
Lead	13	32	42	446	79
Lithium	13	13.65	8	374	25
Mercury	13	16.38	62	169.6	10
Nickel	13	56.7	42	3380	8
Nitrate	7	209	42	1260	8
Nitrite	13	342.1	8	1540	70
Phosphate	0	NA	NA	NA	NA
Phosphorus	13	133	36	831	79
Potassium	1	270	79	270	79
Silver	13	15.6	27	182	79
Sulfate	13	974	66	1980	70
Sulfur	13	362	66	1090	79
Uranium	3	112	10	142	25

*When analytical duplicate data were available, the results were averaged before performing statistical analysis.

**Depth of max/min is the midpoint of the composite interval.

Table 3-85 shows that for the majority of analytes, the composite where the maximum concentration is located is below the composite where the minimum concentration is found. There are several analytes that are an exception to this observation. Cadmium, mercury, nickel, and nitrate all have their maximum value located in a composite that is above where the minimum value is located. This is the opposite of what is seen for the radionuclides in Table 3-62, where the depths of maximum activities/concentrations (for radionuclides) are found in the upper half of the sludge.

3.9.7 Quantitative Comparison of Inorganic Analytes to Action Limits

In an effort to quantify the relationship between the center of the distribution for each analyte and the corresponding action limits, UCL of the mean at 90 and 95% are calculated for the metals data using a nonparametric bootstrapping method. Because many of the distributions do not appear to follow a normal distribution, the nonparametric method was chosen over a method that requires the assumption of normality. The use of a nonparametric method for calculating UCLs reduces the need for verifying distributional assumptions about the underlying distribution of the data. It is also particularly useful when the data appear to have a mixed distribution resulting from multiple releases of contaminants, as is certainly the case for the sludge tank. For results that are nondetects, the detection limit was used as a value. The method used to assign values to nondetects impacts the subsequent calculation of the UCL. Depending on the number of nondetects, the detection limits, and the variability both within and between the detected values and the nondetected values, the estimate of the UCL can increase or decrease as a consequence of different replacement schemes. Impacts of the different replacement schemes must be assessed on a per analyte basis.

In this nonparametric bootstrapping method, the data are sampled, with replacement, to create a new data set of the same size as the original data set. This process is repeated 5000 times, so that there are 5000 new data sets of the same size as the original. Then the 90th or 95th percentile of each of the 5000 sample means is calculated. In this way, the variability of the sample mean is quantified using a method that is based only on the data collected. The nonparametric bootstrapping method of calculating UCLs eliminates the need for the construction or assumption of a statistical model to quantify the variability of the sample mean. Table 3-66 shows the values of the 90 and 95% UCLs on the mean. These UCLs can be considered equivalent to the critical values for one-sided hypothesis tests on the means at the alpha levels of 0.10 and 0.05, respectively. Also listed in the table are the number of detected results in the samples and the action limit, if one was available. The data show that cadmium, chromium, lead, silver, and mercury (all RCRA metals) exceed the action limit. Thus, the sludge would be considered a RCRA toxic characteristic waste. The UHC nickel also exceeds the TCLP criteria.

Table 3-66. Comparison of Action Limits to UCL of the Mean for Nonradiological Inorganic Analyte in Sludge Composite Samples From Tank 241-Z-361

Analyte[1]	Number of Detects	Action limit[2] (ppm)	90% UCL[3] (ppm)	95% UCL[3] (ppm)
Ammonia/Ammonium	2	No action limit	537.1	543.2
Arsenic	1	100	***22.44***	***23.15***
Barium	13	420	139.6	143
Beryllium	0	24.4	1.092	1.155
Bromide	13	No action limit	2364	2532
Cadmium	13	2.2	***50.43***	***54.01***
Chloride	13	No action limit	694.9	711.5
Chromium	13	12	***3778***	***4110***
Cyanide	3	No action limit	0.9515	0.9728
Fluoride	13	No action limit	5767	6035
Hydroxide	0	No action limit	8272	8285
Lead	13	15	***189.3***	***203.5***
Lithium	13	No action limit	184.9	195.6
Mercury	13	0.5	***65.38***	***70.22***
Nickel	13	220	1100	1216
Nitrate	7	No action limit	514.6	560.7
Nitrite	13	No action limit	1066	1110
Phosphate	0	No action limit	143.8	144
Phosphorus	13	No action limit	388.5	415
Potassium	1	No action limit	122.2	128.9
Silver	13	2.8	***68.02***	***73.8***
Sulfate	13	No action limit	1529	1560
Sulfur	13	No action limit	613.3	634.7
Uranium	3	No action limit	117.9	121.9

1. Analytes in ***bold italics*** are RCRA Toxicity Characteristic metals.
2. Action limits as listed in Table 2-4 in the TSAP (EQM 1999a) are TCLP levels for total metals analysis.
3. Total metals concentration in sludge.

Hypothesis tests are another method for assessing the difference between the center of a distribution and an action limit. The data are examined for evidence that the null hypothesis can be rejected at some level of confidence. Because of the presence of nondetects and the lack of applicability of a normal model, a nonparametric hypothesis test is used. The Wilcoxon Signed Rank (WSR) test (Appendix D) is the nonparametric analog to the one-sample t-test. This test is used when a comparison between a threshold value (action limit) and the center of a distribution is of interest. In this case, WSR is a superior choice over the one-sample t-test for comparison of the center of the distribution to an action limit because the WSR test has a ranking scheme (Gehan ranking; Appendix D) that can accommodate the presence of nondetects. For this ranking scheme to be applicable, there must be no more than 50% nondetects in the data set of interest. Table 3-67 presents the results of the WSR test for the analytes in the inorganic analytes suite.

Table 3-67. Results of Wilcoxon Signed Rank Test

Analyte	Action limit*	WSR Statistic	P-value
Ammonia/Ammonium	No action limit	NA	NA
Arsenic	100	Too many nondetects	NA
Barium	420	-3.1458**	0.0008
Beryllium	24.4	Too many nondetects	NA
Bromide	No action limit	NA	NA
Cadmium	2.2	88	0.9996
Chloride	No action limit	NA	NA
Chromium	12	91	1
Cyanide	No action limit	NA	NA
Fluoride	No action limit	NA	NA
Hydroxide	No action limit	NA	NA
Lead	15	91	1
Lithium	No action limit	NA	NA
Mercury	0.5	91	1
Nickel	220	66	0.9268
Nitrate	No action limit	NA	NA
Nitrite	No action limit	NA	NA
Phosphate	No action limit	NA	NA
Phosphorus	No action limit	NA	NA
Potassium	No action limit	NA	NA
Silver	2.8	3.2157**	0.9993
Sulfate	No action limit	NA	NA
Sulfur	No action limit	NA	NA
Uranium	No action limit	NA	NA

* Action limits as listed in Table 2-4 in the TSAP (EQM 1999a) are TCLP levels for total metals analysis.

** Normal approximation was able to be used for calculation of p-value

For each analyte, the p-value represents the probability of observing such a data set (or one with a center greater than the action limit) under the assumption that the true center of the distribution equals the action limit. The small p-values reflect the corresponding small probability that such elevated levels of contamination would be randomly sampled by chance when in fact the center of the distribution of the analyte of interest equals (or is greater than) the action limit. *Another way of saying this is that small p-values are associated with analytes for which there is strong evidence that values in the samples do not exceed the corresponding action limits. Similarly, large p-values are associated with analytes for which there is little evidence that values in the samples do not exceed the corresponding action limits (e.g. the null hypothesis cannot be rejected).* The results of the WSR tests agree with the results of the non-parametric confidence intervals (Table 3-66). The WSR test results provide insufficient evidence to reject the null hypothesis. Failure to reject the null hypothesis can result from one of two situations. Either the analytes of interest exceed their corresponding action limits or these analytes do not exceed their action limits and we simply do not have enough data to show it. When the result of a hypothesis test is failure to reject the null, power analysis can be used to quantify the probability associated with each of the outcomes previously presented. Results of the EDA combined with the p-values that are close to one obviate the need for a power analysis and indicate that it is most probable that cadmium, chromium, lead, nickel, silver, and mercury exceed their corresponding action limits at the 0.05 significance level.

3.9.8 PCB Concentration and Distribution

No statistical evaluation of the PCB data was performed due to various undefined issues remaining to be resolved. These issues are discussed in Section 3.7.3. Figures 3-98 through 3-102 plot the results and detection limits for the Aroclor data, on the calculated dry-weight basis.

Figure 3-98. Composite Sample Results for Aroclor-1232

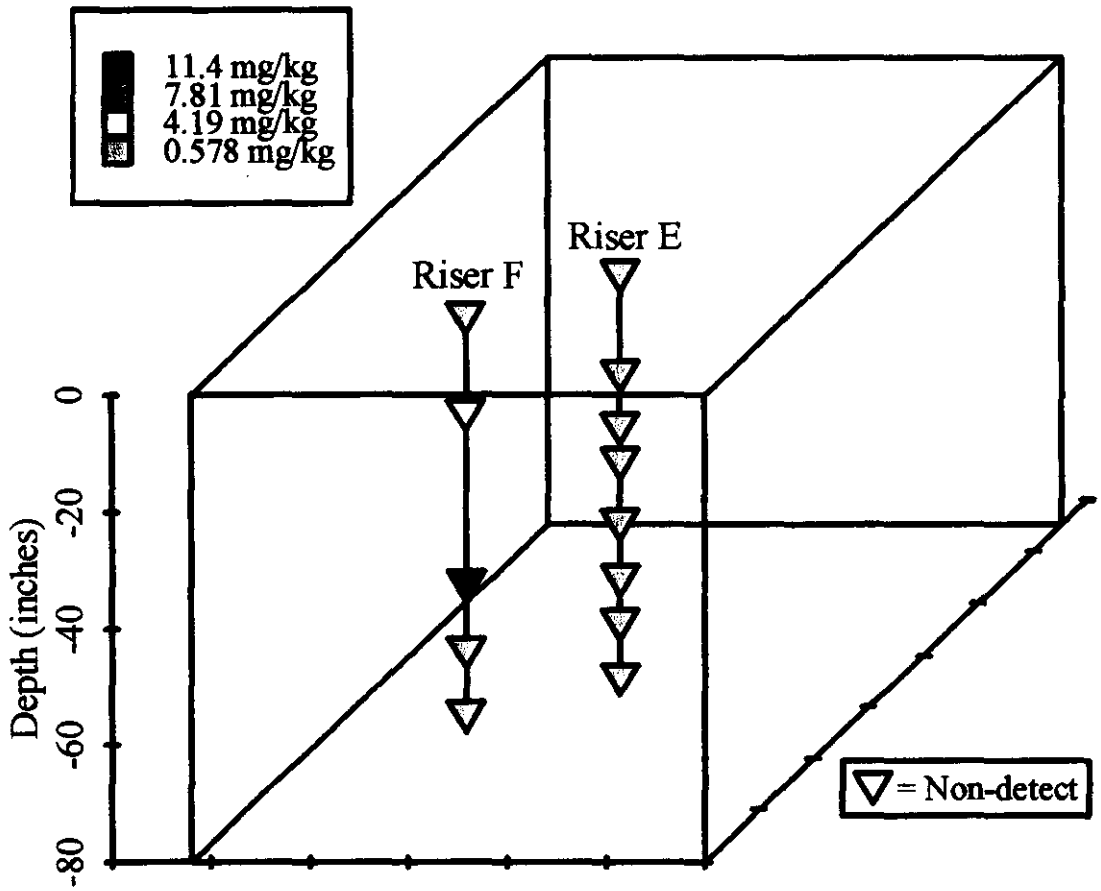


Figure 3-99 . Composite Sample Results for Aroclor-1242

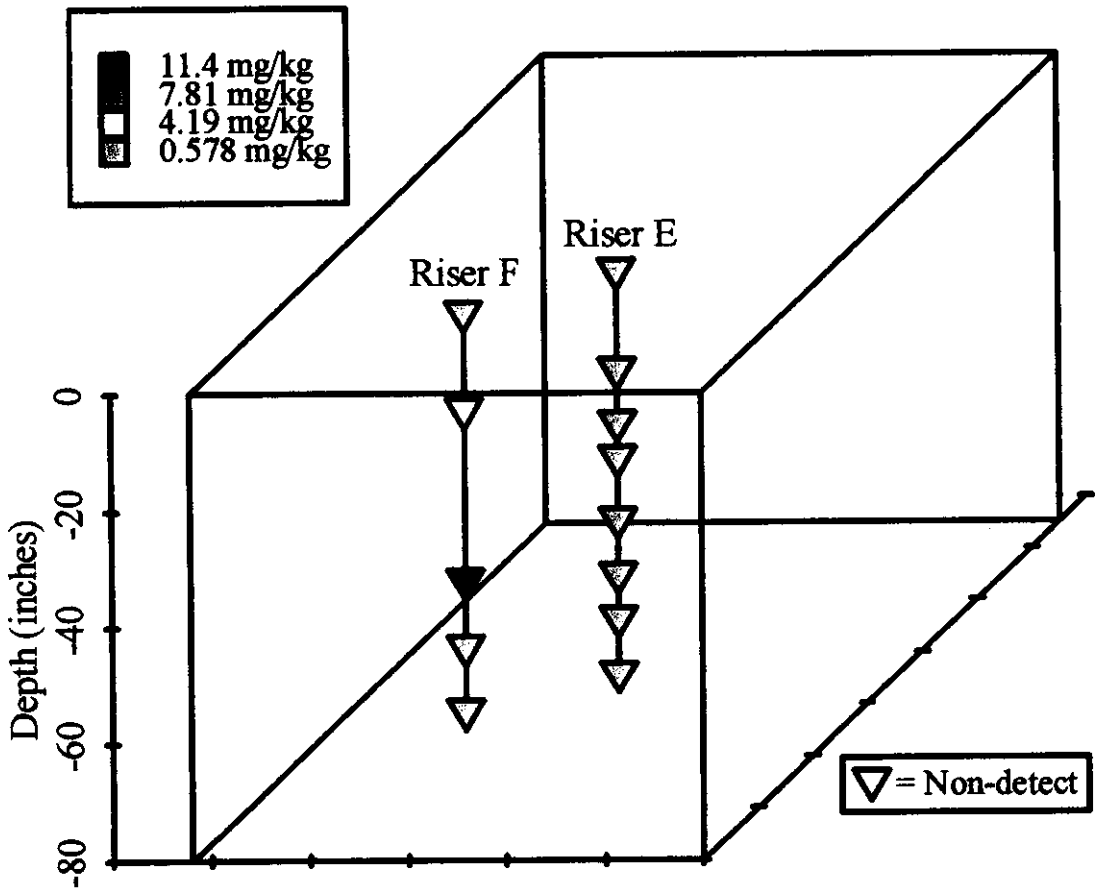


Figure 3-100. Composite Sample Results for Aroclor-1248

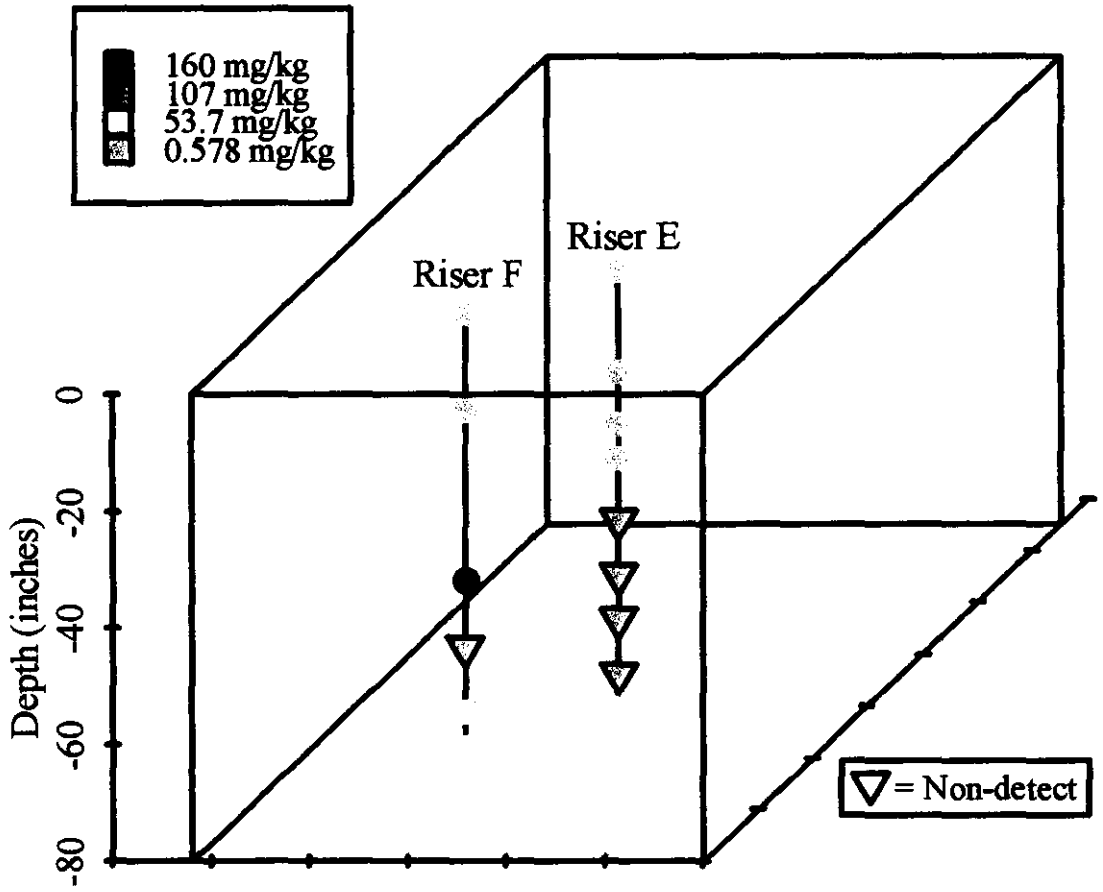


Figure 3-101. Composite Sample Results for Aroclor-1254

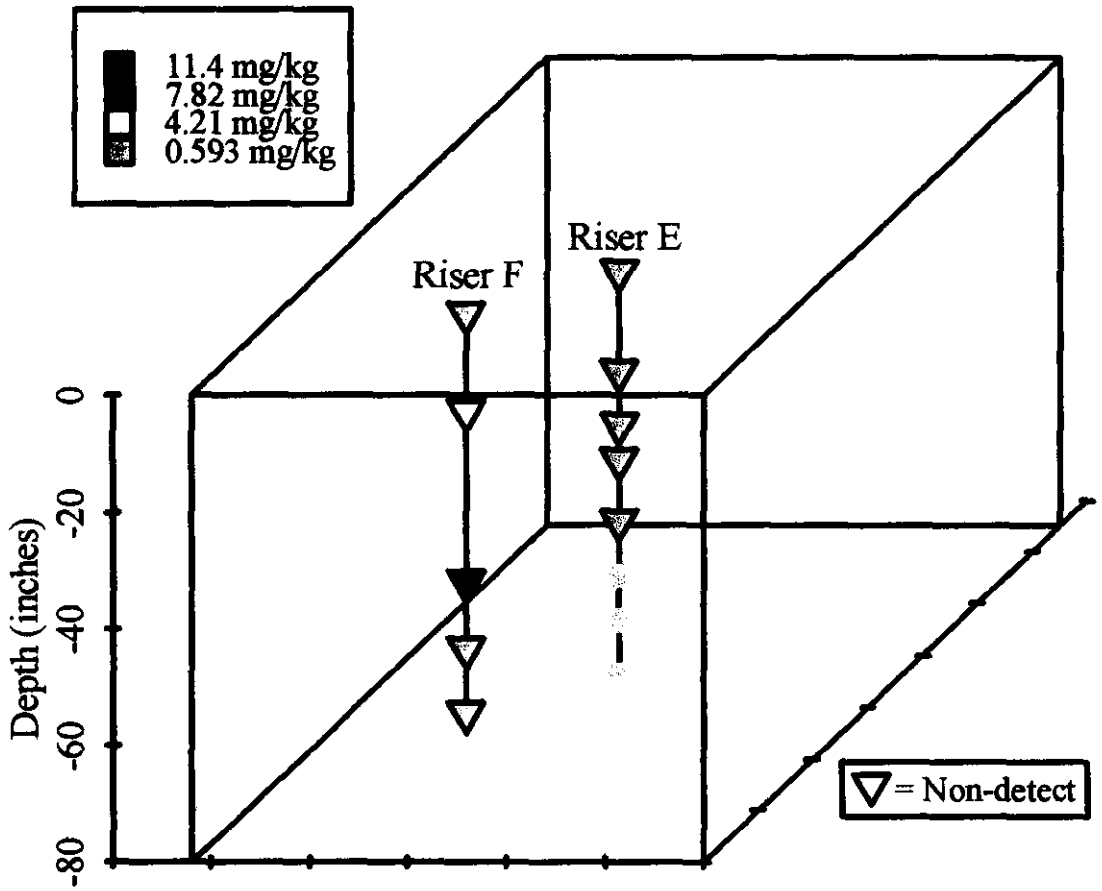
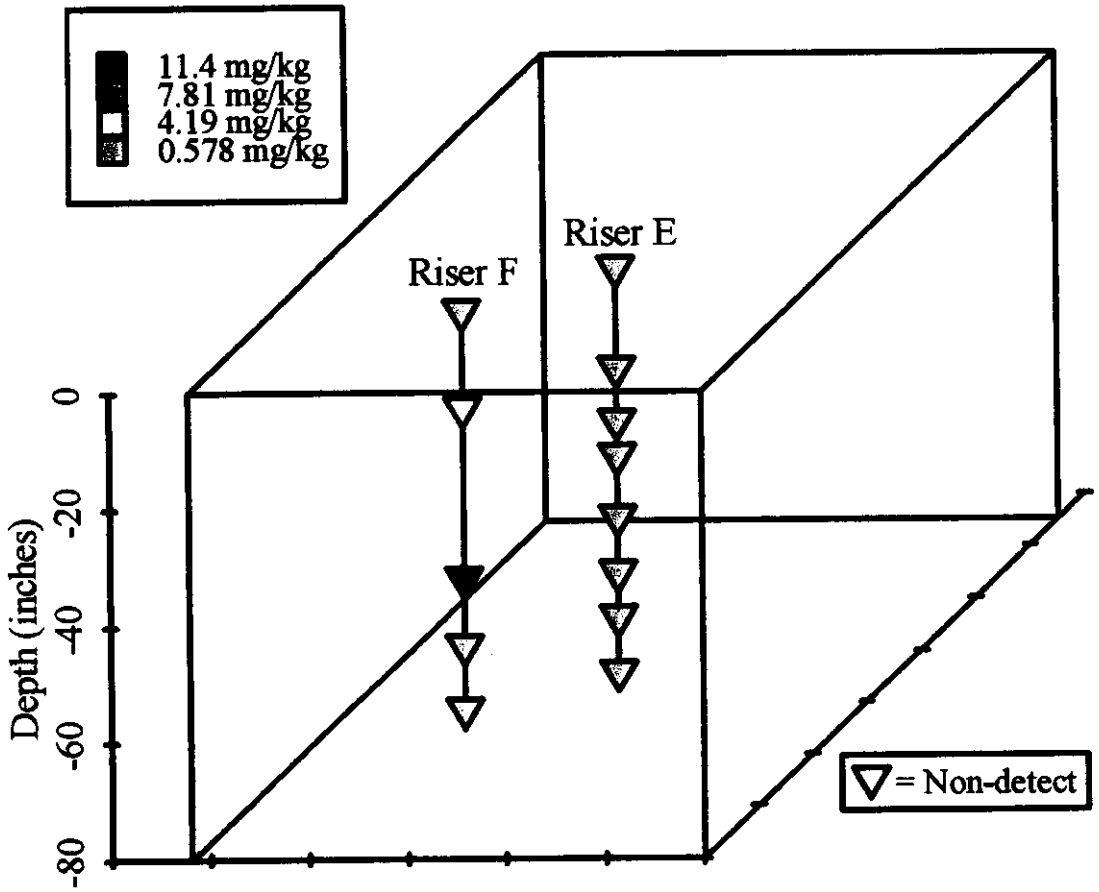


Figure 3-102. Composite Sample Results for Aroclor-1260



3.9.9 Uncertainties

Hypothesis tests are used to provide data-based decision rules that guide the assessment and characterization of constituents of concern. For each hypothesis test performed in the previous section, the null hypothesis of interest for each analyte can be stated in the following manner: "The concentration of the analyte exceeds the corresponding action limit." In this way the burden of proof is put on the data to show that the concentration of the analyte is below the specified action limit, if in fact this is the case. Each hypothesis test has a p-value associated with it. In the context of the null hypothesis stated above, the p-value represents the probability of obtaining a concentration equal to or lower than the one calculated from the sludge composite sample in subsequent sampling efforts. This probability is calculated under the assumption that the true average concentration in the tank equals the Action Limit.

There are two types of decision errors associated with any classical statistical hypothesis test. One is mistakenly concluding that the Action Limits have been met (Type I error). The other is mistakenly concluding that the Action Limits have not been met. Mistakenly concluding that the Action Limits have been met is equivalent to deciding that the sludge is "acceptable" in some sense when, in fact, the sludge is not acceptable (Type II error). Mistakenly concluding that the Action Limits have not been met is equivalent to concluding the sludge is not "acceptable" in some sense when in fact the sludge is acceptable. Whenever a decision is made based on data, there is an associated probability of making a mistake (Type I or Type II error). The p-value quantifies the probability of a Type I error based on the data that are available. For all analytes where hypothesis tests were performed and there is a corresponding Action Level (barium, cadmium, chromium, lead, mercury, nickel, silver, and sulfur) the small p-value provides strong evidence that the center of the distribution for each analyte exceeds the Action Limit.

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APPENDIX A

PHASE I AND PHASE II FIELD RADIOLOGICAL AND VAPOR DATA

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY

☐ Business Sensitive

ADMINISTRATION

Survey ID	99-0251	Survey Date	4-26-99	Page	1 of 2
Location Area:	200 W	Building:	224-B	Room:	Z-361 Other: [illegible]
Company Conducting Operation:	CH2M				

Agent	CAS	Limit	Reference
TOC	na	Some up to 3 ppm	HASP
O2	na	19.5-23.5%	OSHA
NH3	7664-41-7	Eff. TWA 25 PPM	ACGIH
CO	na	20%-21%	HASP/OSD

In Procedure (s): [illegible] SOP's - 2.1.1.2, 1.5, 2.2, 2.3, 2.4 HASP (HNF 2867)

COPY

Survey Time: BASELINE SURVEY OF AREA ON Z-361

Job Documents: W-S-99-00253 Work Package No.: N/A Other:

Requested (Name, Co., Phone): W KENNEDY

On-the-job Contact (Name, Co., Phone): B. PAZNOK

Surveyor(s): Name (Please Print):	TOM T WOODS	Signature: [signature]	Page 1 of 2 Date: 5-4-99
Reviewer: Name (Please Print):	L MARQUART	Signature: [signature]	Page 1 of 2 Date: 5/11/99
Reviewed By: Name (Please Print):		Signature:	Page ___ of ___ Date:
Entered Into System By:		Signature:	Date:

CALIBRATION

	Instrument No. 1		Instrument No. 2		Instrument No. 3		Detector Tube Pump
Instrument	PID-251		OVM 580-5		TMX-410		DRAGER HAND PUMP
ID No.	0001		P-165		CT06		HCM
Factory Cal	3-18-99		2-18-99		2-23-99		4-2-99
Battery (Y/N)	N/A		N/A		N/A		[redacted]
Zero/Gain	N/A		11.EEV		N/A		
Cal. Source ID	B-169-100 (25%LEL) PENTANE		B-003-100 100 PPM ISOBUTYLENE		(25%LEL) PENTANE		
Cal. Exp./Exp. Date	12-2000		12-2000		12-2000		
Cal. Source Value	25% LEL		100 PPM		25% LEL		
	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration
Date	4-26-99	4-26-99	4-26-99	4-26-99	N/A	N/A	4-26-99
Time	0715	1300	0715	1300	N/A	N/A	0715
As Found	25%	25%	97 PPM	101 PPM	N/A	N/A	[redacted]
Adjusted to	N/A	[redacted]	100 PPM	[redacted]	N/A	[redacted]	[redacted]
Cal Lab Check (Y/N)	N/A	N/A	N/A	N/A	N/A	N/A	Y
Cal By Initials	TTW	TTW	TTW	TTW	TTW	TTW	TTW
Cal Location (OFC/FLD)	OFC	OFC	OFC	OFC	OFC	OFC	OFC

Calibrated By: TT WOODS

Name (Please Print)

← see back

CCl4 tubes →

Detector Tube	Chemical	Catalog No.	Lot No.	Name
1	NH3	5A	[illegible]	[illegible]
2	CCl4	1A	[illegible]	[illegible]
3	ACID TEST	1B	[illegible]	[illegible]

Calibration Comments: O2 Cal - 20.9%

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY (Continued)			D Business Sensitive
Survey ID 99-0891	Survey Date 4-21-99	Page : 2 :	
METEOROLOGY			
Time 1000	Pressure 29.23 in Hg	Wind Speed 6 mph	
Temperature 52 °F	Humidity 32 %	Wind Direction W	
D Surface Conditions	Comments: NET TOWER REPORT FOR EAST AREA		
WORKPLACE / INSTRUMENT READINGS			
Operation / Task TID			
Eng. Controls N/A			
Admin Controls ROPES AND BARRIERS			

Reading No.	Instrument	Sample Type	Time	Location	Job Activity	Agent	Results / Units
#1	#1	S	1000	Z-361 Baseline	Baseline Survey	O_2/LEL	20.9% / 0%
2	#2	S	↓	Riser H closed.	↓	TOC	0 PPM
3	DT	S	↓	↓	↓	NH_3	0 PPM

Comments:

4-21-99

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY

D — Business Sensitive

ADMINISTRATION

Survey ID	[illegible]	Survey Date	4-27-99	Page	1 of 2
Location Area	200 W	Building	[illegible]	Room	Z 361

Company Conducting Operation: [illegible]

Agent	CAS	Limit	Reference	In Procedure (s):
T.O.C	n/a	[illegible]	HASP	[illegible]
O2	n/a	[illegible]	OSHA	SOPs - [illegible]
NH3	[illegible]	[illegible]	ACGIH	HASP (HNF-[illegible])
CO	n/a	[illegible]	HASP/OSD	

COPY

Survey Title: BASELINE SURVEY OF AREA IN CRN Z-361

Job Documents: HS-99-00023 — Other: N/A
Work Package No.:

Reviewer (Name, Co., Phone): W. KENNEDY

On-Charge Person (Name, Co., Phone): L. MARQUART

Surveyor(s):	TOM T WOODS	Signature:	Page 1 of 2 — Date: 5-4-99
Name (Please Print):			
Project Mt	L. MARQUART	Signature:	Page 1 of 2 — Date: 5/11/99
Name (Please Print):			
Reviewed By:			Page — of — — Date:
Name (Please Print):		Signature:	
Entered into System By:		Signature:	Date:

CALIBRATION

	Instrument No. 1	Instrument No. 2	Instrument No. 3	Detector Tube Pump
Instrument	MX-251	OVM 325-5	TPX-418	DRAGER HAND PUMP
ID No.	C004	P-163	G706	H028
Periodic Cal	3-18-99	3-18-99	3-23-99	4-2-99
Battery (Y/N)	N/A	N/A	N/A	
Lamp/Cell	N/A	11.7EV	N/A	
Cal. Source ID	5-169-100 (25% LEL) PENTANE	5-005-102 100 PPM ISOBUTYLENE	(25% LEL) PENTANE	
Cal. Expiration Date	12-2000	12-2000	12-2000	
Cal. Source Value	25% LEL	100 PPM	25% LEL	

	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration
Date	4-27-99	4-27-99	4-27-99	4-27-99	N/A	N/A	4-27-99
Time	0715	1300	0715	1300	N/A	N/A	0715
As Found	25%	25%	12 PPM	101 PPM	N/A	N/A	
Adjusted to	N/A		100 PPM		N/A		
24 Leak Check (Y/N)	N/A	N/A	N/A	N/A	N/A	N/A	Y
Cal By Initials	TTW	TTW	TTW	TTW	TTW	TTW	TTW
Cal Location (OFC/FLD)	OFC	OFC	OFC	OFC	OFC	OFC	OFC

Calibrated By: TT WOODS
Name (Please Print)

Detector Tube	Chemical	Catalog No.	Lot No.	Range
1	NH3	5/A	[illegible]	[illegible]
2	CO/CO2	1/A	[illegible]	[illegible]
3	ACID TEST	10	[illegible]	INDICATION [illegible]

Calibration Comments: O2 Cal - 20.5%

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY (Continued)

☐ Business Sensitive

Survey ID: 99-0892 | Survey Date: 4-27-99 | Page : of :

METEOROLOGY

Time:	1030	Pressure:	29.28	Wind Speed	6
Temperature	50.0°F	Humidity	38 %	Wind Direction	W

☐ Standard Conditions | Comments: HIT TOWER REPORT FOR EAST AREA

WORKPLACE / INSTRUMENT READINGS

Operation / Task: TFD

Eng. Controls: N/A

Admin. Controls: ROPES AND BARRIERS

Reading No.	Instrument	Sample Type	Time	Location	Job Activity	Agent	Results/Units
1	#1	S	1020	2-361 Fixer Hand	Pre-check	O2/LEL	20.9% / 0%
2	#2	S	↓	closed Flange.	↓	TOC	0 PPM
3	DT	S	↓	↓	↓	NH_3	0 PPM

4-27-99

Comments:

Industrial Hygiene Direct Reading Instrument Survey

Business Sensitive ☐ Yes ☐ No

COPY

Survey ID 99-0293 | Survey Date 4-28-99 | Page 1 of 2

Company Conducting Operation Lockheed Martin Hanford Company Other

Job Location 200 W Tank Farm / Facility Z-361 Room

Other Location 241-Z-361

Agent	CAS	Limit	Reference	IH Procedures
ORGANIC VAPORS (O.V.)	N/A	2 ppm @ 3 minutes	HASP 002, Rev. 3	Manufacturers
AMMONIA (NH_3)	7664-41-7	25 ppm / 35 ppm STEL	HASP/NIOSH	Operating Manuals
FLAMMABLE GAS (LEL)	N/A	10% / 25%	OSHA/BIO Section 5.11	HNF-Pro-409
OXYGEN (O_2)	N/A	19.5% - 23.5%	OSHA	
Other				

Survey Title Z-361 RISER BASELINE CHECK

Work Package Number 99-00033	Procedure Number N/A		Other N/A
Requestor	Company LMHC		Phone
On the job contact B. PRASNIK	Company LMHC		Phone 373-7757
Surveyor KL Jones	Signature	Page 1 of 2	Survey Date 4-28-99
Project IH Lauri J. Abrenovdt	Signature	Page 1 of 2	Review Date 4/28/99
Reviewed by	Signature	Page of	Review Date
Entered into the system by	Signature	Entered Date	

Calibration Data

	Instrument 1		Instrument 2		Instrument 3		Detector Pump
Instrument Brand/Type	Ind. Sci. TMX-410		Ind. Sci. MX-251		Therm. Env. 580 EZ/S		DRAGER PUMP
IHEL Inst. I.D. Number	IHEL OT31		IHEL		IHEL PIEY		IHEL H049
Periodic Calibration Date	2-11-99				11-27-98		3-15-99
Battery Check	☒ Yes ☐ No ☐ N/A		☐ Yes ☐ No ☐ N/A		☐ Yes ☐ No ☐ N/A		
Lamp / Gain	N/A		N/A		10.6 eV		
Calibration Source ID	8-049-101				9-005-102		
Cal. Effective/Exp. Date	2-19-98				1-6-99		
Calibration Source Value	25% LEL Pentane		25% LEL Pentane		100 ppm Isobutylene		
	Pre Cal	Post Cal	Pre Cal	Post Cal	Pre Cal	Post Cal	Pre Cal
Date	4-28-99	4-28-99			4-28-99	4-28-99	4-28-99
Time	0732	1215			0730	1213	0734
As Found	25%	24%			96.1 ppm	103 ppm	
Adjusted to	N/A				102 ppm		
Detector Pump check							☒ Yes ☐ No
"Calibrated by" Initials	KLJ	KLJ	KLJ	KLJ	KLJ	KLJ	KLJ
Calibration Location	Ofc Fld	Ofc Fld	Ofc Fld	Ofc Fld	Ofc Fld	Ofc Fld	Ofc Fld

Detector Tube Data

Detector Tube Chemical	Catalog Number	Lot Number	Range
Ammonia (NH_3)	CH20501	LL-1243	5 to 70 ppm

Calibrated by KL Jones | Comments:

KLJ 4-28-99

Industrial Hygiene Direct Reading Instrument Survey

Business Sensitive ☐ Yes ☐ No

Survey ID 99-0293 | Survey Date 4-22-99 | Page 2 of 2

Meteorology Data

373-2716 PNL Forecaster | Current Hanford conditions

Time 0900 | Pressure 29.30 in/Hg | Wind Speed 16

Temp 56° ☒ F ☐ C | Humidity 33 % | Wind Direction N

Standard Conditions ☐ Yes | Comments Weather data obtained from PNL

Workplace / Instrument Measurements

Operation Z-361 RISER BASELINE CHECK

Engineering Controls N/A

Administrative Controls HASP

Reading Number	Inst. #	Sample Type	Time	Location	Job Activity	Agent	Results
1	1	A	0845	Z-361 RISER	INITIAL START	O_2	20.9%
2	1	A				LEL	0 %
3	3	A				CV	0 ppm
4	DT	A				NH_3	<5 ppm

Comments

KCL 4-22-99

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY

Business Service

ADMINISTRATION

Survey ID	99-0294	Survey Date	4-29-99	Page	1 of 3
Location Area:	200-W	Building:	224-B	Room: 2-361	Other:
Company Conducting Operation:	LMHC				

Agent	CAS	Limit	Reference	In Procedure (s):
T.O.C	NA	Stop for 3 min.	HASP	HNF 4, 1.3.
O2	NA	19.5-23.5%	OSHA	SOP1-2.11.2 15.3.2, 7.3.2.2.4
NH3	7664-41-7	Ev. TWA 25PPM	ACGIH	HASP (HNF 2367)
CO	NA	25%-25%	HASP/OSD	

COPY

Survey Title: VENT 2-361 TANK AND INSTALL BREATHER FILTER

Job Documents: WS-99-00033 Work Package No.: N/A Other:

Requestor (Name, Co., Phone): W. KENNEDY

On-The-Job Contact (Name, Co., Phone): S. PRAZNIK

Surveyor(s): TOM T WOODS		Pages 1 thru 3	
Name (Please Print):	Signature:	Date: 5-3-99	
Project Int: L. MARQUART		Pages 1 thru 3	
Name (Please Print):	Signature:	Date: 5/11/99	
Reviewed By:		Pages — thru —	
Name (Please Print):	Signature:	Date:	
Entered Into System By:	Signature:	Date:	

CALIBRATION

	Instrument No. 1		Instrument No. 2		Instrument No. 3		Detector Tube Pump
Instrument	MSA 261		OVM 580-5		TMX-410		DRAGER HAND PUMP
ID No.	C001		P-165		0706		H088
Periodic Cal	3-18-99		3-18-99		3-25-99		4-2-99
Battery (Y/N)	N/A		N/A		N/A		[redacted]
Lamp/Gain	N/A		11.8EV		N/A		
Cal. Source ID	B-169-100 (25%LEL) PENTANE		S-005-100 100 PPM ISOBUTYLENE		(25%LEL) PENTANE		
Cal. Exp./Exp. Date	12-2000		12-2000		12-2000		
Cal. Source Value	25% LEL		100 PPM		25% LEL		
	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration
Date	4-29-99	4-29-99	4-29-99	4-29-99	N/A	N/A	4-29-99
Time	0715	1200	0715	1200	N/A	N/A	0715
As Found	25%	25%	95 PPM	101 PPM	N/A	N/A	[redacted]
Adjusted to	N/A	[redacted]	100 PPM	[redacted]	N/A	[redacted]	[redacted]
DT Leak Check (Y/N)	N/A	N/A	N/A	N/A	N/A	N/A	Y
Cal By Initials	TTW	TTW	TTW	TTW	TTW	TTW	TTW
Cal. Location (OFC/FLD)	OFC	OFC	OFC	OFC	OFC	OFC	OFC

Calibrated By: TT WOODS	Detector Tube	Chemical	Catalog No.	Lot No	Range
Name: (Please Print)	1	NH3	5/A	LH-1E31	5-70 PPM
	2	CO/CO2	1/A	LH-C-01	1-15 PPM
	3	ACID TEST	10	HL-0201	INDICATION ONLY

per Robert L. Marquart 5/24/01 CLH ATR

Calibration Comments: O2 Cal - 20.5%

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY (Continued)

☐ Business Sensitive

Survey ID: 94-0844 | Survey Date: 11-29-94 | Page 2 of 3

METEOROLOGY

Time: 11:00 | Pressure: 29.30 inHg mmHg | Wind Speed: 15 MPH

Temperature: 67 °F °C | Humidity: 26 % | Wind Direction: N

Comments: ☐ Standard Conditions

WORK PLACE/INSTRUMENT READINGS

Operation/Task: Venting 2-361 Tank

Eng. Controls: Glovebag, Enclosure

Admin Controls: Dome loading, Ropes, bariers

Reading No.	Instrument	Sample Type	Time	Location	Job Activity	Agent	Results/Units
1	#1	S	10:15	Glove bag / riser	removing bolts	CG	0 % LEL
2	#2	S	↓	↓	↓	TOC	2 ppm
3	DT	S	↓	↓	↓	NH3	0 ppm
				Rev 4-29-95		~~CCl2~~	~~0 ppm~~
4	#1	S	10:38	Glove bag / riser	removing bolts	CG	0 % LEL
5	#2	S	↓	↓	↓	TOC	2 ppm
6	DT	S	↓	↓	↓	NH3	0 ppm
7	DT	S	11:25	Glove bag / riser	↓	CCl2	0 ppm
8	#1	S	11:30	Glove bag / riser	reinstate riser	CG	0 % LEL
9	#2	S	↓	↓	↓	TOC	2 ppm
10	DT	S	↓	↓	↓	NH3	0 ppm
11	DT	S	↓	↓	↓	CCl2	0 ppm
12	DT	S	11:35	breather filter	↓	CCl2	0 ppm
13	#1	S	11:40	over open riser	covering riser	CG	0 % LEL
14	#2	S	↓	↓	↓	TOC	2.3 ppm
15	DT	S	↓	↓	↓	NH3	0 ppm
16	DT	S	↓	↓	↓	CCl2	0 ppm
17	DT	S	↓	↓	↓	Acid	pos.
18	DT	S	11:50	Breather filter	installing Breather filter	Acid	pos.
19	#1	S	11:55	Dome space	↓	CG	1 % LEL
20	#1	S	↓	↓	↓	O2	19 %

Comments: (Name / ID / Job Title / PPE / Reading No.)

N/A

A-6001-760R (02/98)

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY (Continued)

☐ Business Sensitive

Survey ID 99-0894 | Survey Date 4-29-99 | Page 3 of 3

METEOROLOGY

Time:	Pressure: inHg mmHg	Wind Speed: MPH
Temperature ° F ° C	Humidity: %	Wind Direction:

Comments: ☐ Standard Conditions

4-29-99

WORK PLACE/INSTRUMENT READINGS

Operation/Task

Eng. Controls

Admin Controls

Reading No.	Instrument	Sample Type	Time	Location	Job Activity	Agent	Results/Units
21	DT	S	11:55	Dome space	installing Breather filter	VOC	0 ppm
22	#2	S	↓	↓	↓	TOC	3.7 ppm

4-29-99

Comments: (Name / ID / Job Title / PPE / Reading No.)

N/A

A-6001-760R (02/98)

Industrial Hygiene Direct Reading Instrument Survey

Business Sensitive ☐ Yes ☒ No

Survey ID 99-0501 | Survey Date 4-30-99 | Page 1 of 2

COPY

Company Conducting Operation Lockheed Martin Hanford Company Other

Job Location 200W Tank Farm / Facility PFP Room

Other Location Z-361 TANK

Agent	CAS	Limit	Reference	IH Procedures
ORGANIC VAPORS (O.V.)	N/A	2 ppm @ 3 minutes	HASP 002, Rev. 3	Manufacturers
AMMONIA (NH_3)	7664-41-7	25 ppm / 35 ppm STEL	HASP/NIOSH	Operating Manuals
FLAMMABLE GAS (LEL)	N/A	10% / 25%	OSHA/BIO Section 5.11	HNF-Pro-409
OXYGEN (O_2)	N/A	19.5% - 23.5%	OSHA	
Other				

Survey Title Z-361 TANK CLEAN UP

Work Package Number ~~92-~~ N/A	Procedure Number N/A		Other N/A
Requestor N/A	Company LMHC		Phone N/A
On the job contact N/A	Company LMHC		Phone N/A
Surveyor KL Jones	Signature	Page 1 of 2	Survey Date 4-30-99
Project IH Lori T. Nergaard	Signature Lori T. Nergaard	Page 1 of 2	Review Date 5/4/99
Reviewed by	Signature	Page of	Review Date
Entered into the system by	Signature	Entered Date	

Calibration Data

	Instrument 1		Instrument 2		Instrument 3		Detector Pump
Instrument Brand/Type	Ind. Sci. TMX-410		Ind. Sci. MX-251		Therm. Env. 580 EZ/S		DRAGER PUMP
IHEL Inst. I.D. Number	IHEL OT31		IHEL		IHEL TI84		IHEL H049
Periodic Calibration Date	2-11-99				2-11-99		2-15-99
Battery Check	☒ Yes ☐ No ☐ N/A		☐ Yes ☐ No ☐ N/A		☐ Yes ☐ No ☒ N/A		
Lamp / Gain	N/A		N/A		10.6 eV		
Calibration Source ID	Z-079-101				9-005-102		
Cal. Effective/Exp. Date	2-19-99				1-6-99		
Calibration Source Value	25% LEL Pentane		25% LEL Pentane		100 ppm Isobutylene		
	Pre Cal	Post Cal	Pre Cal	Post Cal	Pre Cal	Post Cal	Pre Cal
Date	4-30-99	4-30-99			4-30-99	4-30-99	4-30-99
Time	0754	1420			0759	1420	0800
As Found	25%	22%			99 ppm	101 ppm	
Adjusted to	N/A				N/A		
Detector Pump check							☒ Yes ☐ No
"Calibrated by" Initials	KLJ	KLJ	KLJ	KLJ	KLJ	KLJ	KLJ
Calibration Location	Ofc Fld	Ofc Fld	Ofc Fld	Ofc Fld	Ofc Fld	Ofc Fld	Ofc Fld

Detector Tube Data

Detector Tube Chemical	Catalog Number	Lot Number	Range
Ammonia (NH_3)	CH20501	LL-1842	5 to 70 ppm
		KLJ 4-30-99	

Calibrated by KL Jones | Comments:

KLJ 4-30-99

Industrial Hygiene Direct Reading Instrument Survey

Business Sensitive ☐ Yes ☒ No

Survey ID 99-0901 | Survey Date 4-30-99 | Page 2 of 2

Meteorology Data

373-2716 PNL Forecaster | Current Hanford conditions

Time 1000 | Pressure 29.27 in/Hg | Wind Speed 4

Temp 63° ☒ F ☐ C | Humidity 40 % | Wind Direction N

Standard Conditions ☐ Yes | Comments Weather data obtained from PNL

Workplace / Instrument Measurements

Operation 2-361 TANK CLEAN UP

Engineering Controls N/A

Administrative Controls HASP

Reading Number	Inst. #	Sample Type	Time	Location	Job Activity	Agent	Results
1	1	S	0933	2-361 GLOVE BAG	INITIAL PRIOR TO REMOVAL	O_2	19.4 %
2	1	S				LEL	Ø %
3	3	S				OV	12 ppm
4	DT	S				NH_3	Ø ppm
5	1	A	0940	2-361 GREENHOUSE	GENERAL AREA CHECK	O_2	20.9 %
6	1	A				LEL	Ø %
7	3	A				OV	Ø ppm
8	1	S	0949	2-361 BAGHOUSE FILTER	INITIAL BASELINE	O_2	20.9 %
9	1	S				LEL	Ø %
10	3	S				OV	Ø ppm
11	DT	S				NH_3	Ø ppm
12	1	S	0957	2-361 GLOVE BAG	CONTINUOUS READING	O_2	19.2 %
13	1	S				LEL	Ø %
14	3	S				OV	8.4 ppm
15	1	S	1002	2-361 BAGHOUSE FILTER	CONTINUOUS READING	O_2	21.0 %
16	1	S				LEL	Ø %
17	3	S	1002	2-361 GLOVE BAG	CONTINUOUS ORGANIC	OV	7.4 ppm
18	1	S	1010		OXYGEN/LEL LEVELS	O_2	19.5 %
19	1	S				LEL	Ø %
20	3	S	1015	2-361 GLOVE BAG	CONTINUOUS ORGANIC	OV	5.8 ppm
21	1	A		2-361 GREENHOUSE	CONTINUOUS READING	O_2	21.0 %
22	1	A				LEL	Ø %
*23	3	A	1030	2-361 GREEN HOUSE	ORGANICS CHECK	OV	Ø ppm
24	1	A			CONTINUOUS READING	O_2	21.0 %
25	1	A				LEL	Ø %

4-30-99

Comments * BAG WAS COLLAPSED AND VENTED TO GREEN HOUSE.

COPY

IH Direct Reading Instrument Survey

Business Sensitive ☐

Survey ID: 99-0916 | Survey Date: 5/7/99 | HNF-PRO-409 Exposure Monitoring

Company Conducting Operation: Lockheed Martin Hanford Company (LMHC) | Other:

Job Location: 200 West | Farm: | Tank: Z361 | Other: 234-5Z

Agent	CAS	Limit	Reference	IH Procedures
TOV	N/A	2 PPM @ 3 Minutes	HASP 002, REV 3	Manufactures
NH3	7664-41-7	25 PPM / 35 PPM	HASP / NIOSH	Operating Manuals
Flammable Gas	N/A	10% / 25%	OSHA / EIO Section	HNF-PRO-409
O2	N/A	19.5%-23.5%	5.11	

Survey Title: VAPOR SAMPLE TANK Z361 | Work Package: WS9800056.7W

Procedure: N/A | Requestor: CPO

Company: Lockheed Martin Hanford Company (LMHC) | Job Contact: B PRAZNICK

Phone: 373-7751 | Surveyor: Randy Horner | Project IH: LauriMarquardt

Entered By: Randy Horner

Calibration

Instrument Type: OVM 580EZ | Calibration Gas: Isobutylene

IH Equipment Number: P183 | Calibration Source ID: FF16937

Pre-Cal Date: 12/22/98 | Cal Effect Date: 1/6/99

Battery Check: ☑ Yes ☐ Low | 10.6 .Ev | Cal Source Value: 99.7 PPM

Pre Cal Date: 5/7/99 | Post Cal Date: 5/7/99

Pre Cal Time: 6:30 | Post Cal Time: 16:00

Pre Cal As Found: 71 PPM | Post Cal As Found: 82 PPM

As Left: 100 PPM | Post Cal By: Randy Horner

Battery Check: ☑ Yes | Other: ☑ Field ☐

Pre Cal By: Randy Horner

Other: ☑ Field ☐

CSA 99 Add Instrument | ▸▸

Instrument Type: MX-251 | Calibration Gas: Pentane

IH Equipment Number: 0028 | Calibration Source ID: FF13625

Pre-Cal Date: 4/26/99 | Cal Effect Date: 2/19/98

Battery Check: ☑ Yes ☐ Low | Cal Source Value: 25 % LEL

Pre Cal Date	5/7/99	Post Cal Date	5/7/99
Pre Cal Time	6:30	Post Cal Time	16:00
Pre Cal As Found	23 % LEL	Post Cal As Found	23 % LEL
Adjusted To	25 % LEL	Post Cal By	Randy Horner
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Randy Horner	Click to Add Instrument	
Office ☑ Field ☐			

Instrument Type	Drager Pump	Calibration Gas	
DTEL Number	H055	Calibration Source ID	
Previous Cal Date	2/22/99	Cal Effect Date	
Battery Check	☐ Yes Lamp	Cal Source Value	

Pre Cal Date	5/7/99	Post Cal Date	5/10/99
Pre Cal Time	6:30	Post Cal Time	
Pre Cal As Found		Post Cal As Found	
Adjusted To		Post Cal By	
Leak Check	☑ Yes	Office ☐ Field ☐	
Pre Cal By	Randy Horner	Click to Add Instrument	
Office ☐ Field ☐			

Detector Tube

Chemical	Catalog Number	Lot Number	Range
Ammonia	CH20501	LL-1842	5-70 PPM

Get Current Weather Data From PNL

Standard Conditions ☐ Weather Time 8:00 Pressure 29.44 in./Hg Humidity 50 %

Wind Speed 13-21 mph Wind Direction SW Temperature 50 F ☑ C ☐

Operation	
Engineering Controls	Enclosure
Administrative Controls	HASP

DRI Readings

Instrument	Type	Location	Activity	Agent	Results
COMB	A	TENT	VAPOR SAMPLING	O2	21.0 %
OVM	A	TENT	VAPOR SAMPLING	TOV	0 PPM
DT	A	TENT	VAPOR SAMPLING	NH3	0 PPM
COMB	S	BREATHER FILTER	VAPOR SAMPLING	LEL	0 %
COMB	S	BREATHER FILTER	VAPOR SAMPLING	O2	21.0 %
OVM	S	BREATHER FILTER	VAPOR SAMPLING	TOV	0 PPM
DT	S	BREATHER FILTER	VAPOR SAMPLING	NH3	0 PPM
COMB	S	DOME SPACE	VAPOR SAMPLING	LEL	0 %
COMB	S	DOME SPACE	VAPOR SAMPLING	O2	19.2 %
OVM	S	DOME SPACE	VAPOR SAMPLING	TOV	4 PPM
DT	S	DOME SPACE	VAPOR SAMPLING	NH3	0 PPM
COMB	A	TENT	VAPOR SAMPLING	LEL	0 %

Comments

CONTINOUS MONITORING AT BREATHER FILTER 0 % LEL. CONTINOUS MONITORING INSIDE TENT 0 PPM TOV.

Tech Signature Sandy Horner

IH Signature

FDH Signature

IH Direct Reading Instrument Survey

Business Sensitive COPY

HNF-PRO-409 Exposure Monitoring

Survey ID: 99-0927 Survey Date: 5/12/99

Company Conducting Operation: Lockheed Martin Hanford Company (LMHC) Other:

Job Location: 200 West Farm: Tank: Z-361 Other: 234-5

Agent	CAS	Limit	Reference	IH Procedure
TOV	N/A	2 PPM @ 3 Minutes	HASP 002, REV 3	Manufactures
NH3	7664-41-7	35 PPM / 35 PPM	HASP / NIOSH	Operating Manuals
Flammable Gas	N/A	10% / 25%	OSHA / BIO Section	HNF-PRO-409
O2	N/A	19.5%-23.5%	5.11	

Survey Title: INTANK VIDEO Work Package: WS-99-00055

Procedure: N/A Requestor: CPO

Company: Lockheed Martin Hanford Company (LMHC) Job Contact: BOB FRAZNIK

Phone: 373-7751 Surveyor: Rick Melbihess Project IH: LauriMarquardt

Entered By: Rick Melbihess

Calibration

Instument Type	OVM 580EZ	Calibration Gas	Isobutylene
IHEL Number	PI-88	Calibration Source ID	9-005-102
Periodic Cal Date	12/23/99	Calibration Effect Date	1/6/99
Battery Check	✓ Yes Lamp: 10.6 .Ev	Cal Source Value	100 ppm

Pre Cal Date	5/11/99	Post Cal Date	5/12/99
Pre Cal Time	23:00	Post Cal Time	5:30
Pre Cal As Found	98.8 ppm	Post Cal As Found	101 ppm
Adjusted To	100 ppm	Post Cal By	Rick Melbihess
Leak Check	Yes	Office ✓ Field	
Pre Cal By	Rick Melbihess		
Office ✓ Field		Click To Add Instrument	

Instument Type	TMX-410	Calibration Gas	Pentane
IHEL Number	OT07	Calibration Source ID	9-068-105
Periodic Cal Date	5/23/99	Calibration Effect Date	03/11/99
Battery Check	✓ Yes Lamp: N/A	Cal Source Value	25%

Pre Cal Date	5/11/99	Post Cal Date	5/12/99
Pre Cal Time	23:00	Post Cal Time	5:30
Pre Cal As Found	25%	Post Cal As Found	25%
Adjusted To	25%	Post Cal By	Rick Melbihess
Leak Check	☐ Yes	Office ☑ Field ☐	
Pre Cal By	Rick Melbihess	Click To Add Instrumen	
Office ☑ Field ☐			

Instument Type	Drager Pump	Calibration Gas	
IHEL Number	HC55	Calibration Source ID	
Periodic Cal Date	7/2/99	Calibration Effect Date	
Battery Check	☐ Yes Lamp: N.A	Cal Source Value	

Pre Cal Date	5/11/99	Post Cal Date	5/12/99
Pre Cal Time	23:00	Post Cal Time	5:30
Pre Cal As Found		Post Cal As Found	
Adjusted To		Post Cal By	Rick Melbihess
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Rick Melbihess	Click To Add Instrumen	
Office ☑ Field ☐			

Detector Tube

Chemical	Catalog Number	Lot Number	Range
Ammonia	CH 20501	LL-1843	5-70
Nitric Oxide	80461		0-15.5

Get Current Weather Data From PN

Standard Conditions Weather Time 3:00 Pressure 29.11 in/Hg Humidity 71 %

Wind Speed 13 mph Wind Direction WNW Temperature 50 F ☑ C ☐

Operation	Waste/Environmental
Engineering Controls	Glove Bag
Administrative Controls	HASP

DRI Readings

Instrument	Type	Time	Location	Activity	Agent	Results
COMB	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	O2	20.9%
COMB	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	LEL	0%
OVM	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	TOV	0.0 PPM
DT	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	NH3	0 PPM
DT	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	NOX	0 PPM
COMB	S	2:10	Z-361	DOME SPACE SAMPLE	O2	18.5 %
COMB	S	2:10	Z-361	DOME SPACE SAMPLE	LEL	1 %
OVM	S	2:10	Z-361	DOME SPACE SAMPLE	TOV	3.4 PPM
DT	S	2:10	Z-361	DOME SPACE SAMPLE	NH3	0 PPM
DT	S	2:10	Z-361	DOME SPACE SAMPLE	NOX	0 PPM
COMB	S	4:00	Z-361	SAMPLE AT 3FT INTO RISER	O2	19.4 %
COMB	S	4:00	Z-361	SAMPLE AT 3FT INTO RISER	LEL	0 %
OVM	S	4:00	Z-361	SAMPLE AT 3FT INTO RISER	TOV	0.9 PPM
DT	S	4:00	Z-361	SAMPLE AT 3FT INTO RISER	NH3	0 PPM
DT	S	4:00	Z-361	SAMPLE AT 3FT INTO RISER	NOX	0 PPM
OVM	A	4:15	Z-361	AREA SAMPLE OUTSIDE OF GLOVEBAG	TOV	0.0 PPM

Comments

Tech Signature

IH Signature

FDH Signature

COPY

IH Direct Reading Instrument Survey

Business Sensitive ☐

Survey ID: 99-1367 | Survey Date: 6/29/99 | HNF-PRO-409 Exposure Monitoring

Company Conducting Operation: Lockheed Martin Hanford Company (LMHC) | Other:

Job Location: 200 West | Farm: | Tank: | Other: Z-PLANT 361

Agent	CAS	Limit	Reference	IH Procedures
TOV	N/A	2 PPM @ 3 Minutes	HASP 002, REV 3	Manufacturers
NH3	7664-41-7	25 PPM / 35 PPM	HASP / NIOSH	Operating Manuals
Flammable Gas	N/A	10% / 25%	OSHA / BIO Section	HNF-PRO-409
O2	N/A	19.5%-23.5%	5.11	

Survey Title: Riser Prep. | Work Package: WS-99-00279

Procedure: N/A | Requestor: CPO

Company: Lockheed Martin Hanford Company (LMHC) | Job Contact: Bob Prusak

Phone: 373-7751 | Surveyor: Dell Spaulding | Project IH: Laura Marquardt

Entered By: Dell Spaulding

Calibration

OV

Instrument Type	OVM 580EZ	Calibration Gas	Isobutylene
IHEL Number	P178	Calibration Source ID	9-005-102
Previous Cal Date	10/3/98	Calibration Effect Date	1/6/99
Battery Check	☑ Yes Lamp 10.6 .Ev	Cal Source Value	99.7 ppm

Pre Cal Date	7/1/99	Post Cal Date	7/1/99
Pre Cal Time	7:45	Post Cal Time	15:20
Pre Cal As Found	95.7 ppm	Post Cal As Found	96.4ppm
Adjusted To	100.0 ppm	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding		
Office ☑ Field ☐		Click To Add Instrument	

Instrument Type	Draeger Pump	Calibration Gas	NA
IHEL Number	H004	Calibration Source ID	NA
Previous Cal Date	4/6/99	Calibration Effect Date	
Battery Check	☑ Yes Lamp	Cal Source Value	NA

Pre Cal Date	6/29/99	Post Cal Date	6/29/99
Pre Cal Time	7:45	Post Cal Time	15:20
Pre Cal As Found	NA	Post Cal As Found	NA
Adjusted To	NA	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Instrument Type	LTX-310	Calibration Gas	Pentane
IHEL Number	IHEL OT27	Calibration Source ID	9-068-105
Periodic Cal Date	6/10/99	Calibration Effect Date	3 /11/99
Battery Check	☑ Yes Lamp N/A	Cal Source Value	25% LEL

LEL

Pre Cal Date	6/29/99	Post Cal Date	6/29/99
Pre Cal Time	7:45	Post Cal Time	15:20
Pre Cal As Found	25%	Post Cal As Found	25%
Adjusted To	25%	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Detector Tube

DT

Chemical	Catalog Number	Lot Number	Range
Ammonia	CH 20501	LL-1843	5-700 PPM

Standard Conditions ☐ Weather Time 10:00 Pressure 29.21 in/Hg Humidity 40 %

Wind Speed 11 mph Wind Direction NW Temperature 71 F ☑ C ☐

Operation	Waste/Environmental
Engineering Controls	None
Administrative Controls	HASP

DRI Readings

Instrument	Type	Time	Location	Activity	Agent	Results
COMB	S	9:45	Z-PLANT Z-361	Riser Prep. Pre-check at the breather filter port.	O2	18.8%
COMB	S	9:45	Z-PLANT Z-361	Riser Prep. Pre-check at the breather filter port.	LEL	0%
OVM	S	9:50	Z-PLANT Z-361	Riser Prep. Pre-check at the breather filter port.	TOV	4.0 PPM
DT	S	9:52	Z-PLANT Z-361	Riser Prep. Pre-check at the breather filter port.	NH3	<5 PPM

Comments

Job was stop do to WBGT reading above the Limits in HNF-PRO-121

Tech Signature

IH Signature

FDH Signature

COPY

IH Direct Reading Instrument Survey

Business Sensitive ☐

Survey ID: 99-1372 | Survey Date: 7/1/99 | HNF-PRO-409 Exposure Monitoring

Company Conducting Operation: Lockheed Martin Hanford Company (LMHC) | Other:

Job Location: 200 West | Farm: | Task: | Other: Z-PLANT 361

Agent	CAS	Limit	Reference	IH Procedures
TOV	N/A	2 PPM @ 3 Minutes	HASP 002, REV 3	Manufacturers
NH3	7664-41-7	25 PPM / 35 PPM	HASP / NIOSH	Operating Manuals
Flammable Gas	N/A	10% / 25%	OSHA / EIO Section	HNF-PRO-409
O2	N/A	19.5%-23.5%	5.11	

Survey Title: Riser Prep. | Work Package: WS-99-00279

Procedure: N/A | Requestor: CPO

Company: Lockheed Martin Hanford Company (LMHC) | Job Contact: Bob Prasnik

Phone: 373-7751 | Surveyor: Dell Spaulding | Project IH: LauriMarquardt

Entered By: Dell Spaulding

Calibration

Instrument Type: OVM 580EZ | Calibration Gas: Isobutylene

IH EL Number: PI78 | Calibration Source ID: 9-005-102

Periodic Cal Date: 10/3/98 | Calibration Effect Date: 1/6/99

Battery Check: ☑ Yes | Lamp: 10.6 .Ev | Cal Source Value: 99.7 ppm

Pre Cal Date: 7/1/99 | Post Cal Date: 7/1/99

Pre Cal Time: 8:00 | Post Cal Time: 14:00

Pre Cal As Found: 97.4 ppm | Post Cal As Found: 98.3ppm

Adjusted To: 100.0 ppm | Post Cal By: Dell Spaulding

Leak Check: ☑ Yes | Office ☑ Field ☐

Pre Cal By: Dell Spaulding

Office ☑ Field ☐

Click To Add Instrument

Instrument Type: Dragger Pump | Calibration Gas: NA

IH EL Number: H004 | Calibration Source ID: NA

Periodic Cal Date: 4/6/99 | Calibration Effect Date:

Battery Check: ☑ Yes | Lamp: | Cal Source Value: NA

Pre Cal Date	7/1/99	Post Cal Date	7/1/99
Pre Cal Time	8:00	Post Cal Time	14:00
Pre Cal As Found	NA	Post Cal As Found	NA
Adjusted To	NA	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Instrument Type	LTX-310	Calibration Gas	Pentane
THEL Number	THEL OT27	Calibration Source ID	9-068-105
Periodic Cal Date	6/10/99	Calibration Effect Date	3/11/99
Battery Check	☑ Yes Lamp N/A	Cal Source Value	25% LEL

Pre Cal Date	7/1/99	Post Cal Date	7/1/99
Pre Cal Time	8:00	Post Cal Time	14:00
Pre Cal As Found	25%	Post Cal As Found	25%
Adjusted To	25%	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Detector Tube

Chemical	Catalog Number	Lot Number	Range
Ammonia	CH 20501	LL-1843	5-700 PPM

Standard Conditions ☐ Weather Time 10:00 Pressure 29.02 in/Hg Humidity 36 %

Wind Speed 11 mph Wind Direction NNW Temperature 70 F ☑ C ☐

Operation	Waste/Environmental
Engineering Controls	None
Administrative Controls	HASP

DRI Readings

Instrument	Type	Time	Location	Activity	Agent	Results
COMB	S	8:50	Z-Plant 361	Riser Prep. Pre-check at the breather filter port.	O2	18.7 %
COMB	S	8:50	Z-Plant 361	Riser Prep. Pre-check at the breather filter port.	LEL	2%
OVM	S	8:55	Z-Plant 361	Riser Prep. Pre-check at the breather filter port.	TOV	32.4 PPM
DT	S	8:58	Z-Plant 361	Riser Prep. Pre-check at the breather filter port.	NH3	<5 PPM
COMB	S	9:20	Z-Plant 361	CHECK AT THE TOP OF RISER F	O2	18.7 %
COMB	S	9:20	Z-Plant 361	CHECK AT THE TOP OF RISER F	LEL	1%
OVM	S	9:25	Z-Plant 361	CHECK AT THE TOP OF RISER F	TOV	5.1 PPM
DT	S	9:27	Z-Plant 361	CHECK AT THE TOP OF RISER F	NH3	<5 PPM
COMB	S	9:30	Z-Plant 361	CHECK 3FT INSIDE RISER F	O2	19.1 %
COMB	S	9:30	Z-Plant 361	CHECK 3FT INSIDE RISER F	LEL	2%
OVM	S	9:33	Z-Plant 361	CHECK 3FT INSIDE RISER F	TOV	5.8 PPM
DT	S	9:35	Z-Plant 361	CHECK 3FT INSIDE RISER F	NH3	<5 PPM

Comments

Tech Signature

IH Signature 7/14/99

FDH Signature

APPENDIX B

PHASE II RADIOLOGICAL AND VAPOR DATA

COPY

IH Direct Reading Instrument Survey

Business Sensitive ☐

HNF-PRO-409 Exposure Monitoring

Survey ID: 99-1367 Survey Date: 6/29/99

Company Conducting Operation: Lockheed Martin Hanford Company (LMHC) Other:

Job Location: 200 West Farm: Tank: Other: Z-PLANT 361

Area	CAS	Limit	Reference	IH Procedures
TOV	N/A	2 PPM @ 3 Minutes	HASP 002, REV 3	Manufacturers
NH3	7664-41-7	25 PPM / 35 PPM	HASP / NIOSH	Operating Manuals
Flammable Gas	N/A	10% / 25%	OSHA / EIO Section	HNF-PRO-409
O2	N/A	19.5%-23.5%	5.11	

Survey Title: Riser Prep. Work Package: WS-99-00079

Procedure: N/A Requestor: CPO

Company: Lockheed Martin Hanford Company (LMHC) Job Contact: Bob Prusnik

Phone: 373-7751 Surveyor: Dell Spaulding Project IH: Lauri Marquardt

Entered By: Dell Spaulding

Calibration

Instrument Type	OVM 580EZ	Calibration Gas	Isobutylene
UHEL Number	F778	Calibration Source ID	9-005-102
Periodic Cal Date	10/3/98	Calibration Effect Date	1/6/99
Battery Check	☑ Yes Lamp	10.6 .Ev	Cal Source Value 99.7 ppm

Pre Cal Date	7/1/99	Post Cal Date	7/1/99
Pre Cal Time	7:45	Post Cal Time	15:20
Pre Cal As Found	95.7 ppm	Post Cal As Found	96.4ppm
Adjusted To	100.0 ppm	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Instrument Type	Drager Pump	Calibration Gas	NA
UHEL Number	H004	Calibration Source ID	NA
Periodic Cal Date	4/6/99	Calibration Effect Date	
Battery Check	☑ Yes Lamp		Cal Source Value NA

Pre Cal Date	6/29/99	Post Cal Date	6/29/99
Pre Cal Time	7:45	Post Cal Time	15:20
Pre Cal As Found	NA	Post Cal As Found	NA
Adjusted To	NA	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Instrument Type	LTX-310	Calibration Gas	Pentane
IHEL Number	IHEL 0727	Calibration Source ID	9-068-105
Periodic Cal Date	6/10/99	Calibration Effect Date	3/11/99
Battery Check	☑ Yes Lamp NA	Cal Source Value	25 % LEL

Pre Cal Date	6/29/99	Post Cal Date	6/29/99
Pre Cal Time	7:45	Post Cal Time	15:20
Pre Cal As Found	25 %	Post Cal As Found	25 %
Adjusted To	25 %	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Detector Tube

Chemical	Catalog Number	Lot Number	Range
Ammonia	CH 20501	LL-1843	5-700 PPM

Standard Conditions ☐ Weather Time 10:00 Pressure 29.21 in/Hg Humidity 40 %

Wind Speed 11 mph Wind Direction NW Temperature 71 F ☑ C ☐

Operation: Waste/Environmental

Engineering Controls: None

Administrative Controls: HASP

DRI Readings

Instrument	Type	Time	Location	Activity	Agent	Results
COMB	S	9:45	Z-PLANT Z-361	Riser Prep. Pre-check at the breather filter port.	O2	18.8%
COMB	S	9:45	Z-PLANT Z-361	Riser Prep. Pre-check at the breather filter port.	LEL	0%
OVM	S	9:50	Z-PLANT Z-361	Riser Prep. Pre-check at the breather filter port.	TOV	4.0 PPM
DT	S	9:52	Z-PLANT Z-361	Riser Prep. Pre-check at the breather filter port.	NH3	<5 PPM

Comments

Job was stop do to WBGT reading above the Limits in HNF-PRO-121

Tech Signature [signature]

IH Signature [signature] 7/14/99

FDH Signature _____

COPY

IH Direct Reading Instrument Survey

Business Sensitive

Survey ID: 99-1372 Survey Date: 7/1/99 HNF-PRO-409 Exposure Monitoring

Company Conducting Operation: Lockheed Martin Hanford Company (LMHC) Other:

Job Location: 200 West Farm: Tank: Other: Z-PLANT 361

Agent	CAS	Limit	Reference	IH Procedures
TOV	N/A	2 PPM @ 3 Minutes	HASP 002, REV 3	Manufacturers
NH3	7664-41-7	25 PPM / 35 PPM	HASP / NIOSH	Operating Manuals
Flammable Gas	N/A	10% / 25%	OSHA / BIO Section	HNF-PRO-409
O2	N/A	19.5%-23.5%	5.11	

Survey Title: Riser Prep. Work Package: WS-99-00279

Procedure: N/A Requestor: CPO

Company: Lockheed Martin Hanford Company (LMHC) Job Contact: Bob Franzik

Phone: 373-7751 Surveyor: Dell Spaulding Project IH: LaurieMarquardt

Entered By: Dell Spaulding

Calibration

Instrument Type: OVM 580EZ Calibration Gas: Isobutylene

IHEL Number: PT78 Calibration Source ID: 9-005-102

Periodic Cal Date: 10/3/98 Calibration Effect Date: 1/6/99

Battery Check: ☑ Yes Lamp: 10.6 .Ev Cal Source Value: 99.7 ppm

Pre Cal Date: 7/1/99 Post Cal Date: 7/1/99

Pre Cal Time: 8:00 Post Cal Time: 14:00

Pre Cal As Found: 97.4 ppm Post Cal As Found: 98.3ppm

Adjusted To: 100.0 ppm Post Cal By: Dell Spaulding

Batt Check: ☑ Yes Office: ☑ Field: ☐

Pre Cal By: Dell Spaulding

Office: ☑ Field: ☐

Click To Add Instrument

Instrument Type: Drager Pump Calibration Gas: NA

IHEL Number: H004 Calibration Source ID: NA

Periodic Cal Date: 4/6/99 Calibration Effect Date:

Battery Check: ☑ Yes Lamp: Cal Source Value: NA

Pre Cal Date	7/1/99	Post Cal Date	7/1/99
Pre Cal Time	8:00	Post Cal Time	14:00
Pre Cal As Found	NA	Post Cal As Found	NA
Adjusted To	NA	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Instrument Type	LTX-310	Calibration Gas	Pentane
INEL Number	INEL OT67	Calibration Source ID	P-068-105
Periodic Cal Date	6/10/99	Calibration Effect Date	3/11/99
Battery Check	☑ Yes ☐ Low N/A	Cal Source Value	25% LEL

Pre Cal Date	7/1/99	Post Cal Date	7/1/99
Pre Cal Time	8:00	Post Cal Time	14:00
Pre Cal As Found	25%	Post Cal As Found	25%
Adjusted To	25%	Post Cal By	Dell Spaulding
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Dell Spaulding	Click To Add Instrument	
Office ☑ Field ☐			

Detector Tube

Chemical	Catalog Number	Lot Number	Range
Ammonia	CH 20501	LL-1843	5-700 PPM

Standard Conditions	☐	Weather Time	10:00	Pressure	29.02	in/Hg	Humidity 36 %
Wind Speed	11	mph	Wind Direction	NNW	Temperature	70	F ☑ C ☐

Operation	Waste/Environmental
Engineering Controls	None
Administrative Controls	HASP

DRI Readings

Instrument	Type	Time	Location	Activity	Agent	Results
COMB	S	8:50	Z-Plant 361	Riser Prep. Pre-check at the breather filter port.	O2	18.7%
COMB	S	8:50	Z-Plant 361	Riser Prep. Pre-check at the breather filter port.	LEL	2%
OVM	S	8:55	Z-Plant 361	Riser Prep. Pre-check at the breather filter port.	TOV	32.4 PPM
DT	S	8:58	Z-Plant 361	Riser Prep. Pre-check at the breather filter port.	NH3	<5 PPM
COMB	S	9:20	Z-Plant 361	CHECK AT THE TOP OF RISER F	O2	18.7%
COMB	S	9:20	Z-Plant 361	CHECK AT THE TOP OF RISER F	LEL	1%
OVM	S	9:25	Z-Plant 361	CHECK AT THE TOP OF RISER F	TOV	5.1 PPM
DT	S	9:27	Z-Plant 361	CHECK AT THE TOP OF RISER F	NH3	<5 PPM
COMB	S	9:30	Z-Plant 361	CHECK 3FT INSIDE RISER F	O2	19.1%
COMB	S	9:30	Z-Plant 361	CHECK 3FT INSIDE RISER F	LEL	2%
OVM	S	9:33	Z-Plant 361	CHECK 3FT INSIDE RISER F	TOV	5.8 PPM
DT	S	9:35	Z-Plant 361	CHECK 3FT INSIDE RISER F	NH3	<5 PPM

Comments

Tech Signature

JH Signature 11/4/99

FDH Signature

COPY

IH Direct Reading Instrument Survey

Buisness Sensitive

08:25Ver:2.0

HNF-PRO-409 Exposure Monitoring

Survey ID: 99-1553 | Survey Date: 8/13/99

Company Conducting Operation: Lockheed Martin Hanford Company (LMHC) | Other:

Job Location: 200 West | Farm: 234-5 | Tank: | Other: Z-361

Agent	CAS	Limit	Reference	IH Procedure
TOV	N/A	2 PPM @ 3 Minutes	HASP 002, REV 3	Manufactures
NH3	7664-41-7	25 PPM / 35 PPM	HASP / NIOSH	Operating Manuals
Flammable Gas	N/A	10% / 25%	OSHA / BIO Section	HNF-PRO-409
O2	N/A	19.5%-23.5%	5.11	

Survey Title: REMOVE LIQUID FROM TANK DRYWELLS | Work Package: WS-99-00430

Procedure: N/A | Requestor: CPO

Company: Lockheed Martin Hanford Company (LMHC) | Job Contact: BOB FRAZNIK

Phone: 373-7751 | Surveyor: Rick Melbihess | Project IH: LauriMarquardt

Entered By: Rick Melbihess

Calibration

Instument Type: OVM 580EZ | Calibration Gas: Isobutylene

IHEL Number: IHEL PI-88 | Calibration Source ID: 8-259-19

Periodic Cal Date: 12/23/99 | Calibration Effect Date: 10/28/98

Battery Check: ☑ Yes | Lamp: 10.6 .Ev | Cal Source Value: 100 ppm

Pre Cal Date: 8/13/99

Pre Cal Time: 0:30

Pre Cal As Found: 92.3 ppm

Adjusted To: 100 ppm

Leak Check: ☑ Yes

Pre Cal By: Rick Melbihess

Office ☑ Field ☐

Post Cal Date: 8/13/99

Post Cal Time: 5:15

Post Cal As Found: 100 ppm

Post Cal By: Rick Melbihess

Office ☑ Field ☐

Click To Add Instrument

Instument Type: TMX-410 | Calibration Gas: Pentane

IHEL Number: IHEL OT-07 | Calibration Source ID: 9-068-105

Periodic Cal Date: 5/25/99 | Calibration Effect Date: 03/11/99

Battery Check: ☑ Yes | Lamp: N/A | Cal Source Value: 25%

Pre Cal Date	5/13/99	Post Cal Date	5/13/99
Pre Cal Time	0:30	Post Cal Time	5:15
Pre Cal As Found	25%	Post Cal As Found	25%
Adjusted To	25%	Post Cal By	Rick Melbihess
Leak Check	☑ Yes	Office ☑ Field ☐	
Pre Cal By	Rick Melbihess		
Office ☑ Field ☐		Click To Add Instrument	

Detector Tube

Chemical	Catalog Number	Lot Number	Range
Ammonia	CH 20501	ME-1782	5-70

Standard Conditions ☐ Weather Time 5:00 Pressure 29.13 in/Hg Humidity 56 %

Wind Speed 10 mph Wind Direction NW Temperature 60 F ☑ C ☐

Operation: Waste/Environmental

Engineering Controls: Glove Bag

Administrative Controls: HASP

DRI Readings

Instrument	Type	Time	Location	Activity	Agent	Results
COMB	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	O2	18.8 PPM
COMB	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	LEL	0%
OVM	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	TOV	4.3 PPM
DT	S	2:00	Z-361	INITIAL BREATHER FILTER SAMPLE	NH3	0 PPM
COMB	S	3:00	Z-361	3FT SAMPLE INTO RISER F	LEL	0%
COMB	S	3:00	Z-361	6FT SAMPLE INTO RISER F	O2	19.2 %
COMB	S	5:20	Z-361	3FT SAMPLE INTO RISER G	LEL	0%
COMB	S	5:20	Z-361	6FT SAMPLE INTO RISER G	O2	18.8 %
OVM	A	3:10	Z-361	OVM monitoring within containment tent	TOV	0.0 ppm

Comments

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY

☐ Business Sensitive

ADMINISTRATION

Survey ID	99-1874	Survey Date	9-22-99	Page	1 of 4

Location: Area: 200-W Building: 224-B Room: 361-Z Other

Company Conducting Operation: LMHC

Agent	CAS	LPM	Reference	IH Procedure (s):
T O C	n/a	Zero for 2 min.	HASP	IH-1.4, 1.5,
O2	n/a	19.5-23.5%	OSHA	SOPs - 2.11, 2.15, 2.3, 2.3, 2.4
N-O	Tech-41-7	8hr. TWA 25PPM	ACGIH	HASP (NW 2867)
CG	n/a	20%-21%	HASP/OSD	

COPY

Survey Task: PUSH MODE SAMPLE 361-Z

Job Documents: Work Package No.: N/A Other: N/A

Requestor (Name, Co., Phone): J. Lee

On-Site Contact (Name, Co., Phone): D. Harvey

Surveyor(s): Name (Please Print):	TOM T WOODS	Signature:	Pages 1 thru 4 Date: 10-1-99
Project Mt: Name (Please Print):	L. HARQUAROT	Signature:	Pages 1 thru 4 Date: 11/1/99
Reviewed By: Name (Please Print):		Signature:	Pages - thru — Date:
Entered into System By:		Signature:	Date:

CALIBRATION

	Instrument No. 1		Instrument No. 2		Instrument No. 3		Detector Tube Pump
Instrument	PGX-251		OVM 580-EZ K		TMX-410		DRAGER HAND PUMP
ID No.	O103		P-96		OT06		H100
Periodic Cal	9-15-99		11-22-98		8-22-99		9-21-99
Battery (Y/N)	N/A		N/A		N/A		[redacted]
Lamp/Gain	N/A		10.6EV		N/A		
Cal. Source ID	B-169-100 (25%LEL) PENTANE		5-005-103 100 PPM ISOBUTYLENE		(25%LEL) PENTANE		
Cal. Mfrc./Exp. Date	12-2000		12-2000		12-2000		
Cal. Source Value	25% LEL		100 PPM		25% LEL		
	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration
Date	9-22-99	9-22-99	9-22-99	9-22-99	9-22-99	9-22-99	9-22-99
Time	0715	2300	0715	2300	0715	2300	0715
As Found	21%	21%	95 PPM	70 PPM	21%	24%	[redacted]
Adjusted to	N/A	[redacted]	100 PPM	[redacted]	N/A	[redacted]	[redacted]
DT Leak Check (Y/N)	N/A	N/A	N/A	N/A	N/A	N/A	Y
Cal By Initials	TTW	TTW	TTW	TTW	TTW	TTW	TTW
Cal. Location (OFC/FLD)	OFC	OFC	OFC	OFC	OFC	OFC	OFC

Calibrated By: TT WOODS
Name: (Please Print)

Detector Tube	Chemical	Catalog No.	Lot No.	Range
1	N-O	S/A	LH-1D1	5-70 PPM
2				
3				

Calibration Comments: O2 Cal - 20.9%

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY (Continued)

Number 99-1874 | Survey Date 9-22-99 | Page 2 of 4

METEOROLOGY

Time 1200 | Pressure 29.14 | Wind Speed 5

Temperature 81 °F | Humidity 38 % | Wind Direction NW

Surface Condition | Comments MET TOWER REPORT FOR EAST AREA

WORKPLACE / INSTRUMENT READINGS

Operation / Task TSD

Eq Control NA

Area Control NONE AND BARRIER

Reading No.	Instrument	Sample Type	Time	Location	Job Activity	Agent	Reading
1	#1	S	1110	2-361 Dome	Pre-check	O2/LEL	20.9%/2%
2	#2/DT	S	↓		↓	TOC/NH3	0.7PPM/<5PPM
3	#1	S	1315		Pre-check	O2/LEL	18.7%/3%
4	#3	S	1330	R- @ 3' in Kist	Initial Bench	O2/LEL	19.5%/2%
5	#2/DT	S	↓	↓	↓	TOC/NH3	3.5PPM/<5PPM
6	#1	S	1345	Dome	15 min ✓	O2/LEL	18.7%/3%
7	#3	S	1355	Drill String	Add 17"	O2/LEL	20.5%/0%
8	#2/DT	S	↓	↓	↓	TOC/NH3	<5PPM/<5PPM
9	#1	S	1400	Dome	15 min ✓	O2/LEL	18.7%/3%
10	#1	S	1415	↓	↓	O2/LEL	18.6%/2%
11	#3	S	1425	Drill String	Drill Pointed Rd	O2/LEL	17.3%/2%
12	#2/DT	S	↓	↓	↓	TOC/NH3	15.7PPM/<5PPM
13	#1	S	1430	Dome	15 min ✓	O2/LEL	18.5PPM/3%
14	#3	S	1435	Drill String	~~Add 17"~~ / Break Drill Str.	O2/LEL	20.5%/2%
15	#2/DT	S	1445	↓	↓	TOC/NH3	[illegible]/<5PPM
16	#1	S	1445	Dome	15 min ✓	O2/LEL	18.5%/2%
17	#1	S	1500	↓	15 min	O2/LEL	18.5%/2%
18	#3	S	1458	Drill String	Remove Sample	O2/LEL	16.6%/2%
19	#2/DT	S	1459	↓	↓	TOC/NH3	6.6PPM/<5PPM
20	#3	S	1511	Drill String	Add sampler	O2/LEL	17.8%/2%
21	#2/DT	S	1511	Drill String	↓	TOC/NH3	3.4PPM/<5PPM
22	#1	S	1515	Dome	15 min	O2/LEL	18.5%/2%
23	#3	S	1517	Drill String	Add 17"	O2/LEL	17.4%/2%
24	#2/DT	S	1519	↓	↓	TOC/NH3	6.6PPM/<5PPM
25	#1	S	1830	Dome	15 min	O2/LEL	18.5%/2%
26	#3	S	1839	Drill String	Prior To Starting	O2/LEL	18.5%/2%
27	#2/DT	S	1839	↓	↓	TOC/NH3	3.4PPM/<5PPM
28	#1	S	1845	Dome	15 min	O2/LEL	18.5%/2%
29	#4	S		Drill String	Break in Drill String	O2/LEL	16.4%/2%
30	#2/DT	S	1856	↓	↓	TOC/NH3	7.6PPM/2%
31	#4	S	1855	↓	↓	O2/LEL	16.5%/2%

Comments #11 TMX 412 Nitic Down Ind Data CO

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY (Continued)

Number: 99-1974 | Date: 9-22-99 | Page 3 of 4

METEOROLOGY

Time: | Pressure: | Wind Speed:

Temperature: °F/°C | Humidity: | Wind Direction:

Surface Condition: | Comments: METEOROLOGY NEEDED FOR EAST AREA

WORKPLACE / INSTRUMENT READINGS

Operation / Task: TRO

Eng. Controls: N/A

Admin Controls: BOXES AND BARRIERS

Reading No.	Instrument	Sample Type	Time	Location	Job Activity	Agent	Reading Level
32	#4	S	1955	Drill String	Break Drill String	O2/LEL	16.3%/2%*
33	#1	S	1900	Dome	15 min.	O2/LEL	19.0%/2%
34	#4	S	1911	Drill String	Drill String	O2/LEL	14.3%/2%*
35	#2/DT	S	↓	Drill String	DS Break	TOC/NH3	8.4ppm/<5ppm
36	#1	S	1915	Dome	15 min	O2/LEL	18.5%/2%
37	#4	S	1925	Drill String	DS Break	O2/LEL	18.5%/2%*
38	#2/DT	S	1926	Drill String	DS Break	TOC/NH3	3.5ppm/<5ppm
39	#1	S	1930	Dome	15 min	O2/LEL	19.0%/2%
40	#4	S	1943	DS	DS Break	O2/LEL	16.2%/2%
41	#2/DT	S	1943	DS	DS Break	TOC/NH3	6.2ppm/<5ppm
42	#1	S	1945	Dome	15 min	O2/LEL	14.3%/2%
43	#4	S	1949	DS	DS Break	O2/LEL	17.1%/2%
44	#2/DT	S	1949	DS	DS Break	TOC/NH3	3.2ppm/<5ppm
45	#4	S	1955	DS	DS Break	O2/LEL	16.5%/2%
46	#2/DT	S	1955	DS	DS Break	TOC/NH3	7.4ppm/<5ppm
47	#4	S	2000	DS	DS Break	O2/LEL	14.5%/1%
48	#2/DT	S	2000	DS	DS Break	TOC/NH3	4.7ppm/<5ppm
49	#1	S	2001	Dome	15 min	O2/LEL	19.0%/1%
50	#4	S	2009	DS	DS Break	O2/LEL	19.2%/1%
51	#2/DT	S	2009	DS	DS Break	TOC/NH3	7.5ppm/<5ppm
52	#1	S	2015	Dome	DS Break	O2/LEL	19.0%/1%
53	#4	S	2026	DS	DS Break	O2/LEL	16.3%/3%
54	#2/DT	S	2026	DS	DS Break	TOC/NH3	7.5ppm/<5ppm
55	#1	S	2030	Dome	15 min	O2/LEL	19.0%/1%
56	#4	S	2032	DS	DS Break	O2/LEL	17.6%/2%
57	#2	S	2032	DS	DS Break	TOC/NH3	7.5ppm
58	#4	S	2038	DS	DS Break	O2/LEL	11.1%/0%
59	#2	S	2038	DS	DS Break	TOC	7.1ppm
60	#1	S	2045	Dome	15 min	O2/LEL	19.0%/3%
61	#4	S	2048	DS	DS Break	O2/LEL	12.5%/1%
62	#2	S	2048	DS	DS Break	TOC/	8.1 ppm

Comments: N/A.

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY (Continued) ☐ Business Sensitive

Survey ID 99-1874 | Survey Date 9-22-99 | Page 4 of 4

METEOROLOGY

Time: | Pressure: | Wind Speed

Temperature °F/°C | Humidity | Wind Direction

Sample Conditions | Comments

WORKPLACE / INSTRUMENT READINGS

Operation / Task

Eye Covers ROPES BANDS

Arm Covers N/A

Reading No.	Instrument	Sample Type	Time	Location	Job Activity	Agent	Results / Units
63	#4	S	2056	DS	DS Break	O2/LEL	12.5%/3%
64	#2	S	2056	DS	DS Break	TOC/	6.2 PPm
65	#1	S	2100	Dome	15 min ~~DS Break~~ DS	O2/LEL	19.0%/1%
66	#1	S	2115	↓	15 min	O2/LEL	19.6%/3%
67	#4	S	2117	DS	DS Break	O2/LEL	16.4%/3%
68	#2	S	2117	DS	DS Break	TOC	3.3 PPM
69	#4	S	2120	DS	DS Break	O2/LEL	17.2%/3%
70	#2	S	2120	DS	DS Break	TOC	0.0 PPm
71	#4	S	2128	DS	Sheilded Reciever	O2/LEL	6.2%/.
72	#2	S	2128	DS	↓	TOC	5.1 PPm
73	#1	S	2130	Dome	15 min	O2/LEL	19.0%/3%
74	#4	S	2133	DS	DS Break	O2/LEL	16.3%/3%
75	#2	S	2133	DS	DS Break	TOC	1.7 PPm

Comments:

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY

☐ Business Sensitive

ADMINISTRATION

Survey ID	[illegible]	Survey Date	9-23-99	Page	1 of 2
Location Area	200-W	Building: 224-B	Room: 361-Z	Other	
Company Conducting Operation:	LMHC				

Agent	CAS	Limit	Reference	In Procedure (s):
T.O.C	n/a	Scan for 2 min.	HASP	Inc.1.4, 1.5,
O2	n/a	19.5-23.5%	OSHA	SOPs - 2.11.2.13, 2.2
N-3	7664-41-7	Ev. TWA 25PPM	ACGIH	HASP (HF 2867)
CO	n/a	25%LEL	HASP/OSD	

COPY

Survey Task: Fall Out Sampling Room 361-Z

Job Documents: N/A
Work Package No.: Other: N/A

Requestor (Name, Co., Phone): J. Lee

On-The-Job Contact (Name, Co., Phone): D. Harvey

Surveyor(s): Name (Please Print): TOM T WOODS	Signature: Tom T Woods	Pages 1 thru 2 Date: 10-11-99	
Project Mt: Name (Please Print): L. MARQUART	Signature: Lyle D Marquart	Pages 1 thru 2 Date: 11/11/99	
Reviewed By: Name (Please Print):	Signature:	Pages - thru — Date:	
Entered into System By:	Signature:	Date:	

CALIBRATION

	Instrument No. 1		Instrument No. 2		Instrument No. 3		Detector Tube Pump	
Instrument	MSA-261		OVM 580-EZ		THX-410		DRAGER HAND PUMP	
ID No.	0103		P-86		0706		H100	
Periodic Cal	9-15-99		11-23-98		8-27-99		9-21-99	
Battery (Y/N)	N/A		N/A		N/A		[redacted]	
Lamp/Gain	N/A		10.6EV		N/A			
Cal. Source ID	S-169-100 (25%LEL) PENTANE		S-005-102 100 PPM ISOBUTYLENE		(25%LEL) PENTANE			
Cal. Effect/Exp. Date	12-2000		12-2000		12-2000			
Cal. Source Value	25% LEL		100 PPM		25% LEL			
	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration	Post Calibration	Pre Calibration	[redacted]
Date	9-23-99	9-23-99	9-23-99	9-23-99	9-23-99	9-23-99	9-23-99	
Time	0715	1515	0715	1515	0715	1515	0715	
As Found	25%	25%	33 PPM	99 PPM	25%	25%	[redacted]	
Adjusted to	N/A	[redacted]	100 PPM	[redacted]	N/A	[redacted]	[redacted]	
Off Leak Check (Y/N)	N/A	N/A	N/A	N/A	N/A	N/A	Y	
Cal By Initials	TTW	TTW	TTW	TTW	TTW	TTW	TTW	
Cal. Location (OK/OF/LD)	OFC	OFC	OFC	OFC	OFC	OFC	OFC	

Calibrated By: TT WOODS
Name: (Please Print)

Detector Tube	Chemical	Catalog No.	Lot No.	Range
1	N-3	SA	LH-1031	5-70 PPM
2				1-15 PPM
3				INDICATION ONLY

Calibration Comments: O2 Cal - 20.5%

INDUSTRIAL HYGIENE DIRECT READING INSTRUMENT SURVEY (Continued) | D Business Service

Survey ID: 99-1877 | Survey Date: 9-22-99 | Page 3 of 3

METEOROLOGY

Time: 1200	Pressure: 29.20	Wind Speed: 3	
Temperature: 80 °F	Humidity: 34 %	Wind Direction: W	
D Surface Condition	Comments: HFT TOWER REPORT FOR EAST AREA		

WORKPLACE INSTRUMENT READINGS

Operation/Task: TPO

Eng Controls: N/A

Area Controls: ROPES AND BARRIERS

Reading No	Instrument	Sample Type	Time	Location	Job Activity	Agent	Reading
1	#1	S	1145	2-361 - Dome	Pre-check	O2/LEL	20.7%/2%
2	#2/DT	S	↓	↓		TOC/NH3	3PPM/<5PPM
3	#1	S	1197	Inner Box (shielded)		O2/LEL	18.7%/0%
4	#1	S	↓	Outer Box		O2/LEL	20.8%/0%
5	#1	S	1150	Drill String		O2/LEL	21%/3%
6	#2/DT	S	↓	↓	↓	TOC/NH3	15PPM/<5PPM
7	#1	S	1200	Drill String	Pulling D.S.	O2/LEL	20.1%/1%
8	#2/DT	S	↓			TOC/NH3	1.3PPM/<5PPM
9	#1	S	1205			O2/LEL	20.8%/0%
10	#2	S	↓			TOC	2.2 PPM
11	#1	S	1208			O2/LEL	20.8%/0%
12	#2	S	↓			TOC	3.4 PPM
13	#1	S	1212			O2/LEL	20.7%/0%
14	#2	S	↓			TOC	3.9 PPM
15	#1	S	1215			O2/LEL	20.3%/0%
16	#2	S	↓	↓	↓	TOC	2.2 PPM
17	#3	S	1215	Dome	15 min ✓	O2/LEL	18.5%/2%
18	#3	S	1230	Dome	↓	O2/LEL	18.5%/2%
19	#1	S	1232	Drill String	Pull (core barrel)	O2/LEL	20.5%/1%
20	#2	S	↓	↓	↓	TOC	4.2PPM

Comments: N/A

Unable to download logged OVM data from datalogger. Seems to be a proble with the instruments datalogger. All breather filter readings were between 0.0 ppm and 5.4 ppm during the work task.	Tech Signature IH Signature 10/69 FDH Signature

APPENDIX C

CHEMICAL AND RADIOLOGICAL ANALYSES OF SLUDGE COMPOSITE SAMPLES

Table C-1. Radiological Sludge Composite Sample Results for Tank 241-Z-361* (2 sheets)

Composite	Americium-241 (µCi/g)		Gross Alpha of Digested Solid (µCi/g)		Gross Beta of Digested Solid (µCi/g)		Neptunium-237 (µCi/g)		Plutonium-238 (µCi/g)		Plutonium-239/240 (µCi/g)		Strontium-89/90 (µCi/g)		Technetium-99 (µCi/g)		Uranium-235 (µg/g)		Uranium-238 (µg/g)	
	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit
Core 263																				
263-1	14.1	1.47	37.4	0.00828	2.35	0.0228	0.00302	0.00368	2.3	1.75	26.8	1.75	0.00829	0.000698	0.015	0.0014	< 19.74	19.75	49.85	19.75
263-2	13.9	1.35	36.2	0.00803	2.13	0.0221	0.00293	0.00357	< 1.7	1.7	28.6	1.7	0.00562	0.000672	0.0178	0.00135	< 19.15	19.14	21.38	19.14
263-3	8.84	1.26	42.6	0.00825	2.45	0.054	< 0.00196	0.00367	< 2.25	2.25	42.4	2.25	0.00249	0.000687	0.0279	0.00138	< 19.68	19.68	20.83	19.68
263-4	3.86	0.648	18.9	0.00827	0.837	0.0228	< 0.00229	0.00368	< 0.968	0.968	17.7	0.968	0.0184	0.000683	0.0134	0.00138	< 19.73	19.73	32.56	19.73
263-5	4.67	0.495	25.4	0.0195	1.38	0.0676	< 0.00225	0.00488	< 1.14	1.14	23.7	1.14	0.0145	0.000746	0.017	0.00152	< 19.68	19.69	21.49	19.69
263-6	4.26	0.483	25.2	0.0195	1.35	0.0677	< 0.00225	0.00488	< 1.12	1.12	23.8	1.12	0.000649	0.000779	0.00125	0.000718	< 19.7	19.7	< 19.7	19.7
263-7	1.35	0.186	7.3	0.0195	0.454	0.0675	< 0.00224	0.00487	< 0.377	0.377	7.26	0.377	0.00319	0.000741	0.0119	0.00153	< 19.65	19.65	26.36	19.65
263-8	0.306	0.0658	3.87	0.019	0.146	0.0658	< 0.00218	0.00475	< 0.201	0.201	4.21	0.201	0.00236	0.000709	< 0.00149	0.00149	< 19.14	19.14	24.09	19.14
Core 264																				
264-1	7.46	0.779	16.9	0.0151	2.21	0.0502	< 0.00614	0.00697	< 0.67	0.67	9.28	0.67	0.0306	0.000795	0.00349	0.00139	< 19.46	19.46	58.37	19.46
264-2	13.4	1.49	37.9	0.0147	3.96	0.0487	0.00523	0.00676	< 1.68	1.68	29.1	1.68	0.044	0.000753	0.0107	0.00122	< 18.88	18.88	62.62	18.88
264-3	5.66	1.16	30.2	0.0145	2.42	0.0483	< 0.00404	0.00671	< 1.54	1.54	26.5	1.54	0.0187	0.000746	0.0227	0.0014	< 18.74	18.74	34.04	18.74
264-4	4.81	0.975	27.8	0.0151	2.18	0.0503	0.00426	0.00699	< 1.52	1.52	26.1	1.52	0.00556	0.000771	0.0223	0.00143	< 19.51	19.51	21.13	19.51
264-5	1.04	0.226	6.33	0.0148	0.552	0.0491	< 0.00601	0.00683	< 0.331	0.331	6	0.331	0.00709	0.000758	0.00262	0.00143	< 19.05	19.05	32.23	19.05

*For detection limit determinations, the laboratory uses the following calculations:

The detection limit (l_d) the level at which it is 95% sure that the material will be detected.
The critical level (L_c) is the level which must be exceeded before a sample can be said to contain any activity above background level.
If the critical level is not exceeded, a "less than" value will be calculated for the analytical result. The L_D value is used to determine the result.
These calculations are performed in accordance with *Detection Limits and Uncertainty Calculations for Radioisotopic Counting*, Procedure LA-508-202.

$$L_d = \frac{2.72}{T_b} + 2L_c$$

$$L_c = 2.33\sqrt{\frac{R_b}{T_b}6}$$

Where:

T_b = Background count time in minutes
R_b = Background count rate in counts per minute

The L_d counts-per-minute value is then used in the activity calculation as cpm and is divided by the fractional efficiency to convert it to dpm.

For example, if a sample count rate is below zero (worse case), the result is reported as less than the critical level count rate corrected for sample size, efficiency, etc. The detection limit is the count rate at 95% of any samples which exceed that rate are a real positive. It is 2.72/count time × 2 × critical level. Therefore, it is possible to have a less than that is less than half the detection level. It is also possible to have a positive result less than the detection level because the results are based on the actual count rate of the sample and the detection limits are based on the background count rate.

Table C-2. Results for Selected Nonchemically-Separated and Chemically-Separated Radionuclides in Sludge Composite Samples from Tank 241-Z-361

Composite	Plutonium-239 (ug/g)		Plutonium-240 (ug/g)		Plutonium/Americium-241 (ug/g)	
	Result	Detection Limit	Result	Detection Limit	Result	Detection Limit
Core 263						
263-1	331	24.68	25.5	24.68	< 24.68	24.68
	362	0.33	45.41	0.33	0.87	0.33
263-2	388	23.93	< 23.93	23.93	< 23.93	23.93
	357	0.319	40.95	0.319	0.69	0.319
263-3	578	24.6	37.23	24.6	< 24.6	24.6
	551	0.328	60.97	0.328	0.803	0.328
263-4	253	24.67	< 24.67	24.67	< 24.67	24.67
	218	0.329	25.28	0.329	0.366	0.329
263-5	329	24.61	< 24.6	24.61	< 24.6	24.61
	300	0.329	30.15	0.329	0.381	0.329
263-6	328	24.62	< 24.62	24.62	< 24.62	24.62
	331	0.329	31.13	0.329	< 0.329	0.329
263-7	114	24.56	< 24.56	24.56	< 24.56	24.56
	102	0.328	10.19	0.328	< 0.328	0.328
263-8	69.07	23.93	< 23.93	23.93	< 23.93	23.93
	70.84	0.319	3.543	0.319	< 0.319	0.319
Core 264						
264-1	147	24.32	< 24.32	24.32	< 24.32	24.32
	123	0.325	15.21	0.325	< 0.325	0.325
264-2	414	23.6	28.48	23.6	< 23.6	23.6
	372	0.315	44.23	0.315	0.808	0.315
264-3	428	23.42	30.13	23.42	< 23.42	23.42
	320	0.313	33.51	0.313	0.398	0.313
264-4	402	24.39	27.63	24.39	< 24.39	24.39
	384	0.326	36.28	0.326	0.408	0.326
264-5	104	23.82	< 23.82	23.82	< 23.82	23.82
	94.42	0.318	8.459	0.318	< 0.318	0.318

NOTE: First row for each composite contains the nonchemically-separated results; the second row contains the chemically-separated results.

Table C-3. Aroclor Results and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (3 sheets)

Analyte	Detection Limit*	Composite Result*	Duplicate*	LCS Recovery (%)	Surrogate Recovery (%)	
					TCX	DCB
Composite 263-1 (Sample No. S00Z000027)						
Aroclor-1232	1.00e+03	< 1.00e+03	< 1000.0	-	74.0	131
Aroclor-1242	1.00e+03	< 1.00e+03	< 1000.0	-	74.0	131
Aroclor-1248	1.00e+03	5.35e+03	6.34e+03	96.0	74.0	131
Aroclor-1254	1.00e+03	< 1.00e+03	< 1000.0	-	74.0	131
Aroclor-1260	1.00e+03	< 1.00e+03	< 1000.0	-	74.0	131
Composite 263-2 (Sample No. S00Z000028)						
Aroclor-1232	1.00e+03	< 1.00e+03	n/a	-	57.0	112
Aroclor-1242	1.00e+03	< 1.00e+03	n/a	-	57.0	112
Aroclor-1248	1.00e+03	2525.000	n/a	-	57.0	112
Aroclor-1254	1.00e+03	< 1.00e+03	n/a	-	57.0	112
Aroclor-1260	1.00e+03	< 1.00e+03	n/a	-	57.0	112
Composite 263-3 (Sample No. S00Z000029)						
Aroclor-1232	1.00e+03	< 1.00e+03	n/a	-	61.6	102
Aroclor-1242	1.00e+03	< 1.00e+03	n/a	-	61.6	102
Aroclor-1248	1.00e+03	5.98e+03	n/a	96.0	61.6	102
Aroclor-1254	1.00e+03	< 1.00e+03	n/a	-	61.6	102
Aroclor-1260	1.00e+03	< 1.00e+03	n/a	-	61.6	102
Composite 263-4 (Sample No. S00Z000030)						
Aroclor-1232	1.00e+03	< 1.00e+03	n/a	-	57.4	97.7
Aroclor-1242	1.00e+03	< 1.00e+03	n/a	-	57.4	97.7
Aroclor-1248	1.00e+03	2.28e+03	n/a	96.0	57.4	97.7
Aroclor-1254	1.00e+03	< 1.00e+03	n/a	-	57.4	97.7
Aroclor-1260	1.00e+03	< 1.00e+03	n/a	-	57.4	97.7
Composite 263-5 (Sample No. S00Z000031)						
Aroclor-1232	1.00e+03	< 1.00e+03	n/a	-	65.9	114
Aroclor-1242	1.00e+03	< 1.00e+03	n/a	-	65.9	114
Aroclor-1248	1.00e+03	< 1.00e+03	n/a	96.0	65.9	114
Aroclor-1254	1.00e+03	< 1.00e+03	n/a	-	65.9	114
Aroclor-1260	1.00e+03	< 1.00e+03	n/a	-	65.9	114

Table C-3. Aroclor Results and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (3 sheets)

Analyte	Detection Limit*	Composite Result*	Duplicate*	LCS Recovery (%)	Surrogate Recovery (%)	
					TCX	DCB
Composite 263-6 (Sample No. S00Z000032)						
Aroclor-1232	200.0	< 2.00e+02	< 2.00e2	-	71.7	110
Aroclor-1242	200.0	< 2.00e+02	< 2.00e2	-	71.7	110
Aroclor-1248	200.0	< 2.00e+02	< 2.00e2	96.0	71.7	110
Aroclor-1254	200.0	3.22e+02	995.0	-	71.7	110
Aroclor-1260	200.0	< 2.00e+02	< 2.00e2	-	71.7	110
Composite 263-7 (Sample No. S00Z000033)						
Aroclor-1232	200.0	< 2.00e+02	n/a	-	66.8	105
Aroclor-1242	200.0	< 2.00e+02	n/a	-	66.8	105
Aroclor-1248	200.0	< 2.00e+02	n/a	-	66.8	105
Aroclor-1254	200.0	1.24e+03	n/a	112	66.8	105
Aroclor-1260	200.0	< 2.00e+02	n/a	-	66.8	105
Composite 263-8 (Sample No. S00Z000034)						
Aroclor-1232	200.0	< 2.00e+02	n/a	-	69.7	89.3
Aroclor-1242	200.0	< 2.00e+02	n/a	-	69.7	89.3
Aroclor-1248	200.0	< 2.00e+02	n/a	112	69.7	89.3
Aroclor-1254	200.0	5.77e+02	n/a	-	69.7	89.3
Aroclor-1260	200.0	< 2.00e+02	n/a	-	69.7	89.3
Composite 264-1 (Sample No. S00Z000035)						
Aroclor-1232	400.0	< 4.00e+02	n/a	-	66.8	121
Aroclor-1242	400.0	< 4.00e+02	n/a	-	66.8	121
Aroclor-1248	400.0	3.78e+03	n/a	112	66.8	121
Aroclor-1254	400.0	< 4.00e+02	n/a	-	66.8	121
Aroclor-1260	400.0	< 4.00e+02	n/a	-	66.8	121
Composite 264-2 (Sample No. S00Z000036)						
Aroclor-1232	1.00e+03	< 1.00e+03	1.00e+03	-	70.9	125
Aroclor-1242	1.00e+03	< 1.00e+03	1.00e+03	-	70.9	125
Aroclor-1248	1.00e+03	1.18e+04	8.38e+03	104	70.9	125
Aroclor-1254	1.00e+03	< 1.00e+03	1.00e+03	-	70.9	125
Aroclor-1260	1.00e+03	< 1.00e+03	1.00e+03	-	70.9	125

Table C-3. Aroclor Results and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (3 sheets)

Analyte	Detection Limit*	Composite Result*	Duplicate*	LCS Recovery (%)	Surrogate Recovery (%)	
					TCX	DCB
Composite 264-3 (Sample No. S00Z000037)						
Aroclor-1232	4.00e+03	< 4.00e+03	n/a	-	66.8	122
Aroclor-1242	4.00e+03	< 4.00e+03	n/a	-	66.8	122
Aroclor-1248	4.00e+03	5.60e+04	n/a	104	66.8	122
Aroclor-1254	4.00e+03	< 4.00e+03	n/a	-	66.8	122
Aroclor-1260	4.00e+03	< 4.00e+03	n/a	-	66.8	122
Composite 264-4 (Sample No. S00Z000038)						
Aroclor-1232	1.00e+03	< 1.00e+03	n/a	-	45.2	103
Aroclor-1242	1.00e+03	< 1.00e+03	n/a	-	45.2	103
Aroclor-1248	1.00e+03	< 1.00e+03	n/a	104	45.2	103
Aroclor-1254	1.00e+03	< 1.00e+03	n/a	-	45.2	103
Aroclor-1260	1.00e+03	< 1.00e+03	n/a	-	45.2	103
Composite 264-5 (Sample No. S00Z000039)						
Aroclor-1232	200.0	< 2.00e+02	n/a	-	82.2	109
Aroclor-1242	200.0	< 2.00e+02	n/a	-	82.2	109
Aroclor-1248	200.0	2.61e+03	n/a	104	82.2	109
Aroclor-1254	200.0	< 2.00e+02	n/a	-	82.2	109
Aroclor-1260	200.0	< 2.00e+02	n/a	-	82.2	109

* Data are in units of g/kg.

Table C-4. Mercury Results and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361

Composite	Sample No	Detection Limit*	Composite Result*	Duplicate*	Spk Rec (%)	Recovery (%)	
						ICV	CCV
Core 263							
263-1	S99Z000264	3.94	177	166	82.6	108	93.7
263-2	S99Z000265	1.03	63.5	n/a	n/a	97.4	103
263-3	S99Z000239	1.24	20.5	n/a	n/a	97.4	103
263-4	S99Z000240	1.21	63.0	n/a	n/a	97.4	103
263-5	S99Z000241	1.3	21.3	n/a	n/a	97.4	103
263-6	S99Z000266	1.08	18.6	14.12	90.5	107	109
263-7	S99Z000242	0.94	23.4	n/a	n/a	107	109
263-8	S99Z000267	1.38	20.7	n/a	n/a	107	109
Core 264							
264-1	S99Z000275	0.73	65.1	69.59	82.9	107	106.2
264-2	S99Z000276	0.76	83.0	n/a	n/a	107	106
264-3	S99Z000250	0.60	26.8	n/a	n/a	107	106
264-4	S99Z000251	0.69	37.6	n/a	n/a	107	106
264-5	S99Z000252	0.69	67.7	n/a	n/a	107	106

* Data are in units of g/g.

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Aluminum by ICP (Fusion Digest) (µg/g)						
263-1	S99Z000306	10100	1010	n/a	n/a	98
263-2	S99Z000307	26400	978	100	25700	98
263-3	S99Z000308	46500	1000	n/a	n/a	98
263-4	S99Z000309	38200	1010	n/a	n/a	98
263-5	S99Z000310	51200	1000	n/a	n/a	99.2
263-6	S99Z000311	27100	1010	99.2	28400	99.2
263-7	S99Z000312	19900	1000	n/a	n/a	99.2
263-8	S99Z000313	2690	978	n/a	n/a	99.2
264-1	S99Z000314	1390	994	97.7	1780	98.8
264-2	S99Z000315	10300	965	n/a	n/a	98.8
264-3	S99Z000316	39200	956	n/a	n/a	98.8
264-4	S99Z000317	30800	996	n/a	n/a	98.8
264-5	S99Z000318	12200	973	n/a	n/a	98.8
Arsenic by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	< 20.1	20	101.9	< 19.2	87.2
263-2	S99Z000294	< 19.5	19.5	n/a	n/a	87.2
263-3	S99Z000295	< 9.8	9.8	n/a	n/a	87.2
263-4	S99Z000296	< 10.1	10.1	n/a	n/a	87.2
263-5	S99Z000297	< 10.1	10.1	n/a	n/a	87.2
263-6	S99Z000298	< 19.9	19.9	n/a	n/a	87.2
263-7	S99Z000299	< 19.8	19.8	n/a	n/a	87.2
263-8	S99Z000300	< 40.2	40.2	n/a	n/a	86
264-1	S99Z000301	23.4	20.3	83.72	23.4	86
264-2	S99Z000302	< 20.4	20.4	n/a	n/a	86
264-3	S99Z000303	< 19.7	19.7	n/a	n/a	86
264-4	S99Z000304	< 20.7	20.7	n/a	n/a	86
264-5	S99Z000305	< 21.1	21.1	n/a	n/a	86

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Barium by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	111	10	99.01	111	91.6
263-2	S99Z000294	105	9.74	n/a	n/a	91.6
263-3	S99Z000295	104	4.9	n/a	n/a	91.6
263-4	S99Z000296	111	5.06	n/a	n/a	91.6
263-5	S99Z000297	152	5.06	n/a	n/a	91.6
263-6	S99Z000298	116	9.96	n/a	n/a	91.6
263-7	S99Z000299	131	9.92	n/a	n/a	91.6
263-8	S99Z000300	197	20	n/a	n/a	88.4
264-1	S99Z000301	87.1	10.1	84.34	84.1	88.4
264-2	S99Z000302	93	10.2	n/a	n/a	88.4
264-3	S99Z000303	192	9.86	n/a	n/a	88.4
264-4	S99Z000304	114	10.3	n/a	n/a	88.4
264-5	S99Z000305	144	10.5	n/a	n/a	88.4
Beryllium by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	< 1	1	95.92	< 0.959	90.6
263-2	S99Z000294	< 0.975	0.974	n/a	n/a	90.6
263-3	S99Z000295	< 0.49	0.49	n/a	n/a	90.6
263-4	S99Z000296	< 0.506	0.506	n/a	n/a	90.6
263-5	S99Z000297	< 0.506	0.506	n/a	n/a	90.6
263-6	S99Z000298	< 0.996	0.996	n/a	n/a	90.6
263-7	S99Z000299	< 0.992	0.992	n/a	n/a	90.6
263-8	S99Z000300	< 2.01	2	n/a	n/a	88.6
264-1	S99Z000301	< 1.01	1.01	84.45	< 1.03	88.6
264-2	S99Z000302	< 1.02	1.02	n/a	n/a	88.6
264-3	S99Z000303	< 0.986	0.986	n/a	n/a	88.6
264-4	S99Z000304	< 1.03	1.03	n/a	n/a	88.6
264-5	S99Z000305	< 1.05	1.05	n/a	n/a	88.6

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Cadmium by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	54.7	1	96.95	55.5	85.2
263-2	S99Z000294	56.7	0.974	n/a	n/a	85.2
263-3	S99Z000295	45	0.49	n/a	n/a	85.2
263-4	S99Z000296	32.8	0.506	n/a	n/a	85.2
263-5	S99Z000297	74.7	0.506	n/a	n/a	85.2
263-6	S99Z000298	1.48	0.996	n/a	n/a	85.2
263-7	S99Z000299	2.15	0.992	n/a	n/a	85.2
263-8	S99Z000300	21.4	2	n/a	n/a	86
264-1	S99Z000301	17.8	1.01	80.21	16.7	86
264-2	S99Z000302	58.7	1.02	n/a	n/a	86
264-3	S99Z000303	112	0.986	n/a	n/a	86
264-4	S99Z000304	16	1.03	n/a	n/a	86
264-5	S99Z000305	17.1	1.05	n/a	n/a	86
Calcium by ICP (Fusion Digest) (µg/g)						
263-1	S99Z000306	39300	2020	n/a	n/a	102.2
263-2	S99Z000307	32600	1960	102	31800	102.2
263-3	S99Z000308	41200	2010	n/a	n/a	102.2
263-4	S99Z000309	49500	2020	n/a	n/a	102.2
263-5	S99Z000310	70100	2010	n/a	n/a	104.2
263-6	S99Z000311	60600	2010	102	64600	104.2
263-7	S99Z000312	73100	2010	n/a	n/a	104.2
263-8	S99Z000313	103000	1960	n/a	n/a	104.2
264-1	S99Z000314	28500	1990	96	26000	103.4
264-2	S99Z000315	32800	1930	n/a	n/a	103.4
264-3	S99Z000316	70100	1910	n/a	n/a	103.4
264-4	S99Z000317	62100	1990	n/a	n/a	103.4
264-5	S99Z000318	91600	1950	n/a	n/a	103.4

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Chromium by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	4030	2	54.15	3900	89.2
	S99Z000306	5050	202	n/a	n/a	99.8
263-2	S99Z000294	936	1.95	n/a	n/a	89.2
	S99Z000307	1260	196	101	1190	99.8
263-3	S99Z000295	691	0.98	n/a	n/a	89.2
	S99Z000308	881	201	n/a	n/a	99.8
263-4	S99Z000296	710	1.01	n/a	n/a	89.2
	S99Z000309	815	202	n/a	n/a	99.8
263-5	S99Z000297	926	1.01	n/a	n/a	89.2
	S99Z000310	1420	201	n/a	n/a	100.8
263-6	S99Z000298	1340	1.99	n/a	n/a	89.2
	S99Z000311	1380	201	102	1490	100.8
263-7	S99Z000299	2150	1.98	n/a	n/a	89.2
	S99Z000312	2320	201	n/a	n/a	100.8
263-8	S99Z000300	10000	4.02	n/a	n/a	89
	S99Z000313	8560	196	n/a	n/a	100.8
264-1	S99Z000301	5910	20.3	-8.76	6000	89
	S99Z000314	6220	199	99.4	6280	100.8
264-2	S99Z000302	3090	2.04	n/a	n/a	89
	S99Z000315	3210	193	n/a	n/a	100.8
264-3	S99Z000303	1110	1.97	n/a	n/a	89
	S99Z000316	1270	191	n/a	n/a	100.8
264-4	S99Z000304	1220	2.07	n/a	n/a	89
	S99Z000317	1400	199	n/a	n/a	100.8
264-5	S99Z000305	4260	2.11	n/a	n/a	89
	S99Z000318	5040	195	n/a	n/a	100.8

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Iron by ICP (Fusion Digest) (µg/g)						
263-1	S99Z000306	44800	1010	n/a	n/a	101.2
263-2	S99Z000307	28200	978	103	27200	101.2
263-3	S99Z000308	6970	1000	n/a	n/a	101.2
263-4	S99Z000309	3730	1010	n/a	n/a	101.2
263-5	S99Z000310	3840	1000	n/a	n/a	102.8
263-6	S99Z000311	3850	1010	103	4230	102.8
263-7	S99Z000312	6830	1000	n/a	n/a	102.8
263-8	S99Z000313	20600	978	n/a	n/a	102.8
264-1	S99Z000314	33000	994	101	47400	101.8
264-2	S99Z000315	31800	965	n/a	n/a	101.8
264-3	S99Z000316	4500	956	n/a	n/a	101.8
264-4	S99Z000317	4680	996	n/a	n/a	101.8
264-5	S99Z000318	12700	973	n/a	n/a	101.8
Lead by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	367	20	91.79	341	83
263-2	S99Z000294	120	19.5	n/a	n/a	83
263-3	S99Z000295	75.8	9.8	n/a	n/a	83
263-4	S99Z000296	32	10.1	n/a	n/a	83
263-5	S99Z000297	34.5	10.1	n/a	n/a	83
263-6	S99Z000298	33.8	19.9	n/a	n/a	83
263-7	S99Z000299	91.9	19.8	n/a	n/a	83
263-8	S99Z000300	446	40.2	n/a	n/a	84.6
264-1	S99Z000301	136	20.3	78.97	129	84.6
264-2	S99Z000302	263	20.4	n/a	n/a	84.6
264-3	S99Z000303	53.6	19.7	n/a	n/a	84.6
264-4	S99Z000304	49.4	20.7	n/a	n/a	84.6
264-5	S99Z000305	173	21.1	n/a	n/a	84.6

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Lithium by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	75.1	2	105.7	73.2	97.4
263-2	S99Z000294	222	1.95	n/a	n/a	97.4
263-3	S99Z000295	31.3	0.98	n/a	n/a	97.4
263-4	S99Z000296	95.9	1.01	n/a	n/a	97.4
263-5	S99Z000297	230	1.01	n/a	n/a	97.4
263-6	S99Z000298	72.3	1.99	n/a	n/a	97.4
263-7	S99Z000299	275	1.98	n/a	n/a	97.4
263-8	S99Z000300	101	4.02	n/a	n/a	89.6
264-1	S99Z000301	13.8	2.03	87.65	13.5	89.6
264-2	S99Z000302	374	2.04	n/a	n/a	89.6
264-3	S99Z000303	94.2	1.97	n/a	n/a	89.6
264-4	S99Z000304	237	2.07	n/a	n/a	89.6
264-5	S99Z000305	89.9	2.11	n/a	n/a	89.6
Magnesium by ICP (Fusion Digest) (µg/g)						
263-1	S99Z000306	7290	2020	n/a	n/a	99.6
263-2	S99Z000307	5030	1960	97.3	4620	99.6
263-3	S99Z000308	4910	2010	n/a	n/a	99.6
263-4	S99Z000309	5120	2020	n/a	n/a	99.6
263-5	S99Z000310	6940	2010	n/a	n/a	101
263-6	S99Z000311	10800	2010	99.6	11500	101
263-7	S99Z000312	8810	2010	n/a	n/a	101
263-8	S99Z000313	6950	1960	n/a	n/a	101
264-1	S99Z000314	4890	1990	98.1	4570	100.8
264-2	S99Z000315	6320	1930	n/a	n/a	100.8
264-3	S99Z000316	6350	1910	n/a	n/a	100.8
264-4	S99Z000317	10700	1990	n/a	n/a	100.8
264-5	S99Z000318	8530	1950	n/a	n/a	100.8

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Manganese by ICP (Fusion Digest) (µg/g)						
263-1	S99Z000306	565	202	n/a	n/a	93.8
263-2	S99Z000307	301	196	92.8	288	93.8
263-3	S99Z000308	286	201	n/a	n/a	93.8
263-4	S99Z000309	< 202	202	n/a	n/a	93.8
263-5	S99Z000310	< 201	201	n/a	n/a	95.8
263-6	S99Z000311	< 201	201	93.7	< 202	95.8
263-7	S99Z000312	422	201	n/a	n/a	95.8
263-8	S99Z000313	< 196	196	n/a	n/a	95.8
264-1	S99Z000314	771	199	89.8	777	94
264-2	S99Z000315	635	193	n/a	n/a	94
264-3	S99Z000316	< 191	191	n/a	n/a	94
264-4	S99Z000317	< 199	199	n/a	n/a	94
264-5	S99Z000318	439	195	n/a	n/a	94
Nickel by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	1530	4.02	87.41	1520	87
263-2	S99Z000294	415	3.9	n/a	n/a	87
263-3	S99Z000295	86.7	1.96	n/a	n/a	87
263-4	S99Z000296	56.7	2.03	n/a	n/a	87
263-5	S99Z000297	68.3	2.03	n/a	n/a	87
263-6	S99Z000298	210	3.98	n/a	n/a	87
263-7	S99Z000299	325	3.96	n/a	n/a	87
263-8	S99Z000300	1580	8.04	n/a	n/a	86.8
264-1	S99Z000301	3360	40.5	12.3	3400	86.8
264-2	S99Z000302	1310	4.07	n/a	n/a	86.8
264-3	S99Z000303	91.1	3.94	n/a	n/a	86.8
264-4	S99Z000304	196	4.14	n/a	n/a	86.8
264-5	S99Z000305	680	4.21	n/a	n/a	86.8

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Phosphorus by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	578	40.2	100.6	558	88.2
263-2	S99Z000294	141	39	n/a	n/a	88.2
263-3	S99Z000295	133	19.6	n/a	n/a	88.2
263-4	S99Z000296	173	20.3	n/a	n/a	88.2
263-5	S99Z000297	156	20.3	n/a	n/a	88.2
263-6	S99Z000298	139	39.8	n/a	n/a	88.2
263-7	S99Z000299	169	39.6	n/a	n/a	88.2
263-8	S99Z000300	831	80.4	n/a	n/a	89.8
264-1	S99Z000301	656	40.5	81.45	627	89.8
264-2	S99Z000302	405	40.7	n/a	n/a	89.8
264-3	S99Z000303	201	39.4	n/a	n/a	89.8
264-4	S99Z000304	135	41.4	n/a	n/a	89.8
264-5	S99Z000305	306	42.1	n/a	n/a	89.8
Potassium by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	< 100	100	104.7	< 95.9	84
263-2	S99Z000294	< 97.5	97.4	n/a	n/a	84
263-3	S99Z000295	< 49	49	n/a	n/a	84
263-4	S99Z000296	< 50.6	50.6	n/a	n/a	84
263-5	S99Z000297	< 50.6	50.6	n/a	n/a	84
263-6	S99Z000298	< 99.6	99.6	n/a	n/a	84
263-7	S99Z000299	< 99.2	99.2	n/a	n/a	84
263-8	S99Z000300	270	200	n/a	n/a	83.4
264-1	S99Z000301	< 101	101	84.65	< 103	83.4
264-2	S99Z000302	< 102	102	n/a	n/a	83.4
264-3	S99Z000303	< 98.6	98.6	n/a	n/a	83.4
264-4	S99Z000304	< 103	103	n/a	n/a	83.4
264-5	S99Z000305	< 105	105	n/a	n/a	83.4

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Silicon by ICP (Fusion Digest) (µg/g)						
263-1	S99Z000306	2100	1010	n/a	n/a	105.4
263-2	S99Z000307	1640	978	104	1640	105.4
263-3	S99Z000308	1540	1000	n/a	n/a	105.4
263-4	S99Z000309	1180	1010	n/a	n/a	105.4
263-5	S99Z000310	2170	1000	n/a	n/a	103
263-6	S99Z000311	1560	1010	105	1660	103
263-7	S99Z000312	2470	1000	n/a	n/a	103
263-8	S99Z000313	< 978	978	n/a	n/a	103
264-1	S99Z000314	2050	994	100	2390	101.6
264-2	S99Z000315	1780	965	n/a	n/a	101.6
264-3	S99Z000316	1480	956	n/a	n/a	101.6
264-4	S99Z000317	1590	996	n/a	n/a	101.6
264-5	S99Z000318	1950	973	n/a	n/a	101.6
Silver by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	34.6	2	99.27	32.9	88.7
263-2	S99Z000294	15.6	1.95	n/a	n/a	88.7
263-3	S99Z000295	24.4	0.98	n/a	n/a	88.7
263-4	S99Z000296	59.3	1.01	n/a	n/a	88.7
263-5	S99Z000297	40.6	1.01	n/a	n/a	88.7
263-6	S99Z000298	17.2	1.99	n/a	n/a	88.7
263-7	S99Z000299	29.1	1.98	n/a	n/a	88.7
263-8	S99Z000300	182	4.02	n/a	n/a	88.1
264-1	S99Z000301	17.8	2.03	82.69	16.1	88.1
264-2	S99Z000302	22.4	2.04	n/a	n/a	88.1
264-3	S99Z000303	60.3	1.97	n/a	n/a	88.1
264-4	S99Z000304	17.2	2.07	n/a	n/a	88.1
264-5	S99Z000305	128	2.11	n/a	n/a	88.1

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Sodium by ICP (Fusion Digest) (µg/g)						
263-1	S99Z000306	11000	2020	n/a	n/a	100.2
263-2	S99Z000307	39200	1960	101	37100	100.2
263-3	S99Z000308	17200	2010	n/a	n/a	100.2
263-4	S99Z000309	7830	2020	n/a	n/a	100.2
263-5	S99Z000310	9610	2010	n/a	n/a	103.2
263-6	S99Z000311	8490	2010	94.9	7760	103.2
263-7	S99Z000312	5860	2010	n/a	n/a	103.2
263-8	S99Z000313	9240	1960	n/a	n/a	103.2
264-1	S99Z000314	3290	1990	102	2920	101.4
264-2	S99Z000315	23800	1930	n/a	n/a	101.4
264-3	S99Z000316	10700	1910	n/a	n/a	101.4
264-4	S99Z000317	7040	1990	n/a	n/a	101.4
264-5	S99Z000318	6620	1950	n/a	n/a	101.4
Sulfur by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	748	20	96.18	750	85.8
263-2	S99Z000294	441	19.5	n/a	n/a	85.8
263-3	S99Z000295	448	9.8	n/a	n/a	85.8
263-4	S99Z000296	499	10.1	n/a	n/a	85.8
263-5	S99Z000297	408	10.1	n/a	n/a	85.8
263-6	S99Z000298	433	19.9	n/a	n/a	85.8
263-7	S99Z000299	423	19.8	n/a	n/a	85.8
263-8	S99Z000300	1090	40.2	n/a	n/a	89
264-1	S99Z000301	683	20.3	81.65	678	89
264-2	S99Z000302	500	20.4	n/a	n/a	89
264-3	S99Z000303	471	19.7	n/a	n/a	89
264-4	S99Z000304	362	20.7	n/a	n/a	89
264-5	S99Z000305	529	21.1	n/a	n/a	89

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Titanium by ICP (Fusion Digest) (µg/g)						
263-1	S99Z000306	276	202	n/a	n/a	102.6
263-2	S99Z000307	< 196	196	100	< 197	102.6
263-3	S99Z000308	< 201	201	n/a	n/a	102.6
263-4	S99Z000309	< 202	202	n/a	n/a	102.6
263-5	S99Z000310	< 201	201	n/a	n/a	102.2
263-6	S99Z000311	< 201	201	103	< 202	102.2
263-7	S99Z000312	< 201	201	n/a	n/a	102.2
263-8	S99Z000313	< 196	196	n/a	n/a	102.2
264-1	S99Z000314	< 199	199	105	< 200	102.2
264-2	S99Z000315	216	193	n/a	n/a	102.2
264-3	S99Z000316	< 191	191	n/a	n/a	102.2
264-4	S99Z000317	< 199	199	n/a	n/a	102.2
264-5	S99Z000318	< 195	195	n/a	n/a	102.2
Zinc by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	393	2	93.6	383	84.4
263-2	S99Z000294	265	1.95	n/a	n/a	84.4
263-3	S99Z000295	124	0.98	n/a	n/a	84.4
263-4	S99Z000296	58.5	1.01	n/a	n/a	84.4
263-5	S99Z000297	88.9	1.01	n/a	n/a	84.4
263-6	S99Z000298	177	1.99	n/a	n/a	84.4
263-7	S99Z000299	303	1.98	n/a	n/a	84.4
263-8	S99Z000300	622	4.02	n/a	n/a	85
264-1	S99Z000301	263	2.03	79.17	251	85
264-2	S99Z000302	446	2.04	n/a	n/a	85
264-3	S99Z000303	109	1.97	n/a	n/a	85
264-4	S99Z000304	180	2.07	n/a	n/a	85
264-5	S99Z000305	367	2.11	n/a	n/a	85

Table C-5. Metals Concentration and Associated Quality Control Data for Sludge Composite Samples from Tank 241-Z-361 (12 sheets)

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Zirconium by ICP (Acid Digest) (µg/g)						
263-1	S99Z000293	75.7	2	103.4	75.7	93.4
263-2	S99Z000294	18	1.95	n/a	n/a	93.4
263-3	S99Z000295	3.89	0.98	n/a	n/a	93.4
263-4	S99Z000296	5.69	1.01	n/a	n/a	93.4
263-5	S99Z000297	4.36	1.01	n/a	n/a	93.4
263-6	S99Z000298	< 1.99	1.99	n/a	n/a	93.4
263-7	S99Z000299	3.84	1.98	n/a	n/a	93.4
263-8	S99Z000300	36.3	4.02	n/a	n/a	92.8
264-1	S99Z000301	193	2.03	86.31	185	92.8
264-2	S99Z000302	48	2.04	n/a	n/a	92.8
264-3	S99Z000303	8.7	1.97	n/a	n/a	92.8
264-4	S99Z000304	3.31	2.07	n/a	n/a	92.8
264-5	S99Z000305	14	2.11	n/a	n/a	92.8

Table C-6. Ammonia/Ammonium by ISE-Std Additions (Ion-selective Electrode) for Sludge Composite Samples from Tank 241-Z-361

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Ammonia/Ammonium by ISE-Std Additions (Ion-selective Electrode) (µg/g)						
263-1	S99Z000280	< 493	493	90.6	< 487	115
263-2	S99Z000281	< 482	482	n/a	n/a	105.2
263-3	S99Z000282	< 498	498	n/a	n/a	105.2
263-4	S99Z000283	< 486	486	n/a	n/a	105.2
263-5	S99Z000284	< 496	496	n/a	n/a	105.2
263-6	S00Z000040	656	523	96.69	595	116
263-7	S00Z000041	< 512	512	n/a	n/a	116
263-8	S00Z000042	< 513	513	n/a	n/a	105.2
264-1	S99Z000288	< 498	498	98.77	< 504	106.8
264-2	S99Z000289	< 491	491	n/a	n/a	106.8
264-3	S99Z000290	< 494	494	n/a	n/a	106.8
264-4	S99Z000291	< 490	490	n/a	n/a	106.8
264-5	S99Z000292	649	510	n/a	n/a	106.8

Table C-7. Cyanide by EDTA Distillation/Spectrometry for Sludge Composite Samples from Tank 241-Z-361

Composite	Sample No.	Composite Result	Detection Limit	Spk Rec (%)	Duplicate	LCS Recovery (%)
Cyanide by EDTA Distillation/Spectrophotometry (µg/g)						
263-1	S99Z000264	< 0.738	0.738	108	< 0.739	99.29
263-2	S99Z000265	< 0.9	0.9	n/a	n/a	99.29
263-3	S99Z000239	< 0.787	0.787	n/a	n/a	99.29
263-4	S99Z000240	< 0.883	0.883	n/a	n/a	99.29
263-5	S99Z000241	< 0.799	0.799	n/a	n/a	99.29
263-6	S99Z000266	1.41	0.861	101	1.02	107.8
263-7	S99Z000242	1.01	0.838	n/a	n/a	107.8
263-8	S99Z000267	1.34	0.77	n/a	n/a	107.8
264-1	S99Z000275	< 0.695	0.695	101	< 0.676	104
264-2	S99Z000276	< 0.875	0.875	n/a	n/a	104
264-3	S99Z000250	< 0.766	0.766	n/a	n/a	104
264-4	S99Z000251	< 0.796	0.796	n/a	n/a	104
264-5	S99Z000252	< 0.65	0.65	n/a	n/a	104

Table C-8. Anions by Ion Chromatography of Water Extract for Sludge Composite Samples from Tank 241-Z-361 (4 sheets)

Composite	Sample No.	Detection Limit	Composite Result
Bromide (µg/g)			
263-1	S99Z000280	147.9	1040
263-2	S99Z000281	144.7	2710
263-3	S99Z000282	149.5	638
263-4	S99Z000283	145.7	1050
263-5	S99Z000284	148.8	2630
263-6	S00Z000024	152.8	1660
263-7	S00Z000025	149.5	1930
263-8	S00Z000026	149.9	1770
264-1	S99Z000288	149.5	280
264-2	S99Z000289	147.2	1150
264-3	S99Z000290	148.2	5370
264-4	S99Z000291	147.1	3110
264-5	S99Z000292	153.1	1370
Chloride (µg/g)			
263-1	S99Z000280	20.11	647
263-2	S99Z000281	19.68	510
263-3	S99Z000282	20.33	532
263-4	S99Z000283	19.81	606
263-5	S99Z000284	20.24	482
263-6	S00Z000024	20.78	907
263-7	S00Z000025	20.33	858
263-8	S00Z000026	20.38	835
264-1	S99Z000288	20.33	718
264-2	S99Z000289	20.02	572
264-3	S99Z000290	20.15	565
264-4	S99Z000291	20	612
264-5	S99Z000292	20.82	562

Table C-8. Anions by Ion Chromatography of Water Extract for Sludge Composite Samples from Tank 241-Z-361 (4 sheets)

Composite	Sample No.	Detection Limit	Composite Result
Fluoride (µg/g)			
263-1	S99Z000280	14.2	4430
263-2	S99Z000281	13.89	10800
263-3	S99Z000282	14.35	7310
263-4	S99Z000283	13.99	2370
263-5	S99Z000284	14.29	2980
263-6	S00Z000024	14.67	3840
263-7	S00Z000025	14.35	5770
263-8	S00Z000026	14.39	5760
264-1	S99Z000288	14.35	1070
264-2	S99Z000289	14.13	3820
264-3	S99Z000290	14.23	8300
264-4	S99Z000291	14.12	2850
264-5	S99Z000292	14.69	3820
Nitrate (µg/g)			
263-1	S99Z000280	164.5	1000
263-2	S99Z000281	160.9	435
263-3	S99Z000282	166.3	314
263-4	S99Z000283	162	209
263-5	S99Z000284	165.5	< 166
263-6	S00Z000024	170	346
263-7	S00Z000025	166.3	< 166
263-8	S00Z000026	166.7	< 167
264-1	S99Z000288	166.3	1230
264-2	S99Z000289	163.7	< 164
264-3	S99Z000290	164.8	603
264-4	S99Z000291	163.6	< 164
264-5	S99Z000292	170.2	< 170

Table C-8. Anions by Ion Chromatography of Water Extract for Sludge Composite Samples from Tank 241-Z-361 (4 sheets)

Composite	Sample No.	Detection Limit	Composite Result
Nitrite (µg/g)			
263-1	S99Z000280	127.8	519
263-2	S99Z000281	125	737
263-3	S99Z000282	129.2	909
263-4	S99Z000283	125.9	1020
263-5	S99Z000284	128.6	863
263-6	S00Z000024	132.1	1370
263-7	S00Z000025	129.2	1540
263-8	S00Z000026	129.5	1530
264-1	S99Z000288	129.2	362
264-2	S99Z000289	127.2	867
264-3	S99Z000290	128	527
264-4	S99Z000291	127.1	986
264-5	S99Z000292	132.3	971
Phosphate (µg/g)			
263-1	S99Z000280	142	< 142
263-2	S99Z000281	138.9	< 139
263-3	S99Z000282	143.5	< 144
263-4	S99Z000283	139.9	< 140
263-5	S99Z000284	142.9	< 143
263-6	S00Z000024	146.7	< 147
263-7	S00Z000025	143.5	< 144
263-8	S00Z000026	143.9	< 144
264-1	S99Z000288	143.5	< 144
264-2	S99Z000289	141.3	< 141
264-3	S99Z000290	142.3	< 142
264-4	S99Z000291	141.2	< 141
264-5	S99Z000292	146.9	< 147

Table C-8. Anions by Ion Chromatography of Water Extract for Sludge Composite Samples from Tank 241-Z-361 (4 sheets)

Composite	Sample No.	Detection Limit	Composite Result
Sulfate (µg/g)			
263-1	S99Z000280	163.3	1780
263-2	S99Z000281	159.8	1320
263-3	S99Z000282	165.1	1310
263-4	S99Z000283	160.9	1240
263-5	S99Z000284	164.3	1100
263-6	S00Z000024	168.7	1480
263-7	S00Z000025	165.1	1980
263-8	S00Z000026	165.5	1950
264-1	S99Z000288	165.1	1560
264-2	S99Z000289	162.5	1130
264-3	S99Z000290	163.6	1400
264-4	S99Z000291	162.4	974
264-5	S99Z000292	169	1040
Phthalate by Ion Chromatography (µg/g)			
263-1	S99Z000280	4420	< 4420
263-2	S99Z000281	4320	< 4320
263-3	S99Z000282	4460	< 4460
263-4	S99Z000283	4350	< 4350
263-5	S99Z000284	4440	< 4440
263-6	S00Z000024	1140	< 1140
263-7	S00Z000025	1120	< 1120
263-8	S00Z000026	1120	< 1120
264-1	S99Z000288	1120	< 1120
264-2	S99Z000289	1100	< 1100
264-3	S99Z000290	1110	< 1110
264-4	S99Z000291	1100	< 1100
264-5	S99Z000292	1140	< 1140

Table C-9. Hydroxide by Potentiometric Titration for Sludge Composite Samples from Tank 241-Z-361

Composite	Sample No.	Detection Limit	Result (g/g)
263-1	S99Z000280	8220	< 8220
263-2	S99Z000281	8040	< 8040
263-3	S99Z000282	8300	< 8300
263-4	S99Z000283	8090	< 8090
263-5	S99Z000284	8270	< 8270
263-6	S99Z000285	8480	< 8480
263-7	S99Z000286	8010	< 8010
263-8	S99Z000287	8340	< 8340
264-1	S99Z000288	8300	< 8310
264-2	S99Z000289	8180	< 8180
264-3	S99Z000290	8230	< 8230
264-4	S99Z000291	8170	< 8170
264-5	S99Z000292	8510	< 8510

Table C-10. Metals Concentration in Drainable Liquid from Core 264-Segment 2 from Tank 241-Z-361 (Sample No. S00Z000002)

Analyte	Composite Result*	Detection Limit*
Arsenic	<29.3	29.3
Barium	88.5	14.6
Beryllium	<1.46	1.46
Cadmium	13.1	1.46
Chromium	3950	29.3
Lead	513	29.3
Lithium	1260	2.93
Nickel	1410	58.5
Potassium	151	146
Silver	46.3	2.93
Sulfur	731	293
Uranium	150	146
Zinc	254	2.93
Zirconium	83.2	2.93

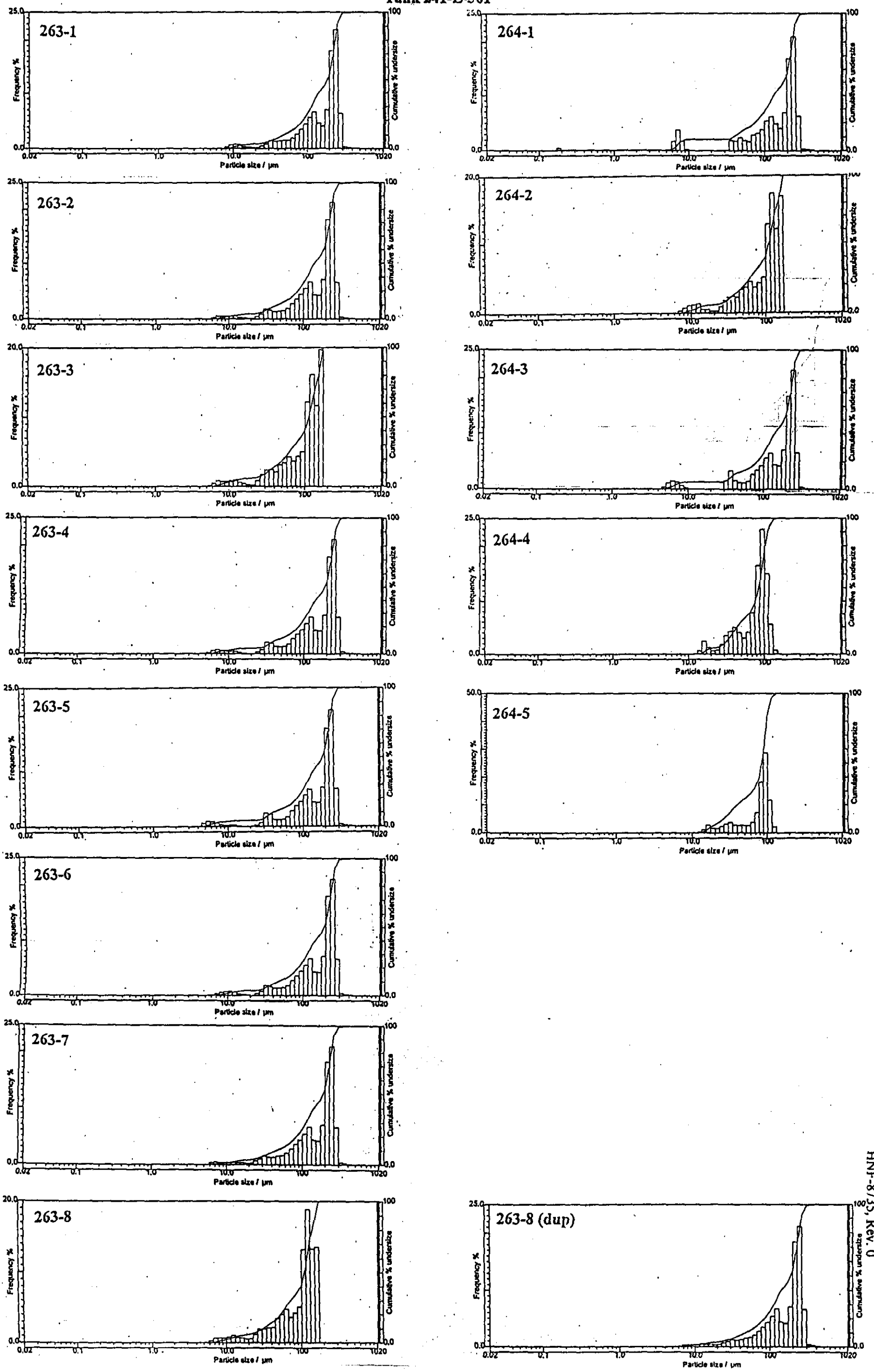
*All results in μg/mL

Table C-11. Particle Size Distribution for Sludge Composite Samples from Tank 241-Z-361

	Frequency (%) of Fraction ≥ Size Indicated																
Size (um)	0.20	0.23	0.26	0.30	3.90	4.47	5.12	5.87	6.72	7.70	8.82	10.10	11.56	13.25	15.17	17.38	19.90
Composite																	
263-1	-	-	-	-	-	-	-	-	-	-	0.34	0.71	0.82	0.79	0.34	0.31	0.22
263-2	-	-	-	-	-	-	-	-	0.23	0.56	0.58	0.55	0.56	0.56	0.30	0.23	0.14
263-3	-	-	-	-	-	-	-	0.13	0.52	0.88	0.75	0.73	0.85	1.01	0.62	0.44	0.28
263-4	-	-	-	-	-	-	-	0.22	0.58	0.76	0.58	0.49	0.49	0.45	0.25	0.12	-
263-5	-	-	-	-	-	-	0.60	0.97	0.89	0.60	0.32	0.25	0.26	0.25	0.16	-	-
263-6	-	-	-	-	-	-	-	-	-	0.33	0.61	0.74	0.75	0.70	0.27	0.20	0.11
263-7	-	-	-	-	-	-	-	-	0.36	0.52	0.40	0.35	0.38	0.47	0.38	0.36	0.28
263-8	-	-	-	-	-	-	-	-	0.30	0.64	0.66	0.68	0.81	1.03	0.68	0.70	0.56
263-8 dup	-	-	-	-	-	-	-	-	0.11	0.31	0.37	0.40	0.46	0.56	0.42	0.46	0.37
264-1	0.48	0.15	-	-	-	-	-	0.23	1.72	3.76	1.79	0.25	-	-	-	-	-
264-2	-	-	-	-	-	-	-	-	-	0.35	0.74	1.03	1.22	1.35	0.54	0.51	0.32
264-3	-	-	-	-	-	-	0.39	1.11	1.53	1.31	0.64	0.29	0.12	-	-	-	-
264-4	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.63	2.46	1.28
264-5	-	-	-	-	-	-	-	-	-	-	-	-	-	0.36	1.20	2.82	1.76

	Frequency (%) of Fraction ≥ Size Indicated (cont.)																						
Size (um)	22.80	26.11	29.91	34.25	39.23	44.94	51.47	58.95	67.52	77.34	88.58	101.50	116.20	133.10	152.50	174.60	200.00	229.10	262.40	300.50	344.20	394.20	451.60
Composite																							
263-1	0.27	0.53	0.85	1.48	1.39	1.47	1.57	1.54	1.96	2.68	3.51	4.42	5.62	6.60	4.59	4.05	6.95	18.08	21.92	6.29	0.37	0.20	0.11
263-2	0.14	0.49	0.88	1.78	1.61	1.34	1.41	1.48	1.88	2.76	3.59	4.42	5.51	6.66	4.34	4.21	7.10	18.09	21.29	6.57	0.39	0.22	0.12
263-3	0.24	0.86	1.75	2.43	2.49	2.13	3.12	3.48	4.35	3.71	4.40	5.08	12.23	16.17	11.68	19.68	-	-	-	-	-	-	-
263-4	-	0.34	0.75	2.08	1.91	1.32	1.31	1.37	1.79	2.77	3.63	4.40	5.54	6.69	4.33	4.22	7.03	18.02	21.10	6.71	0.39	0.22	0.12
263-5	-	0.27	0.69	2.37	2.16	1.26	1.18	1.24	1.68	2.79	3.68	4.40	5.56	6.63	4.31	4.23	7.00	17.70	21.07	6.74	0.40	0.22	0.12
263-6	-	0.41	0.80	1.83	1.67	1.37	1.42	1.47	1.86	2.76	3.60	4.43	5.53	6.70	4.28	4.23	7.16	18.22	21.21	6.61	0.40	0.22	0.12
263-7	0.33	0.68	0.98	1.51	1.32	1.27	1.47	1.62	2.03	2.78	3.57	4.37	5.42	6.71	4.36	4.20	7.08	18.28	21.13	6.65	0.39	0.22	0.12
263-8	0.56	1.17	1.94	1.96	2.10	2.14	3.47	4.02	4.77	3.59	4.21	5.10	13.23	18.82	13.30	13.56	-	-	-	-	-	-	-
263-8 dup	0.46	0.69	0.94	1.30	1.18	1.29	1.57	1.72	2.11	2.75	3.52	4.35	5.39	6.73	4.39	4.18	7.07	18.42	21.11	6.63	0.39	0.22	0.12
264-1	-	-	-	-	1.88	1.75	2.39	1.82	1.70	2.91	3.16	3.82	5.42	6.15	4.87	4.02	6.87	16.97	20.92	6.27	0.37	0.21	0.12
264-2	0.29	0.89	1.77	2.27	2.39	2.22	3.34	3.69	4.50	3.64	4.33	5.11	12.86	17.46	12.13	17.07	-	-	-	-	-	-	-
264-3	-	-	-	1.60	3.35	1.56	1.26	1.17	1.45	2.72	3.53	4.32	5.65	6.54	4.47	4.26	7.09	16.83	21.50	6.58	0.40	0.22	0.12
264-4	0.63	0.82	1.84	3.43	4.16	4.96	4.03	2.97	4.05	7.78	16.57	22.98	15.05	5.58	0.78	-	-	-	-	-	-	-	-
264-5	1.63	2.31	3.16	3.82	2.85	2.70	2.83	2.70	3.86	7.26	18.16	28.75	11.65	2.19	-	-	-	-	-	-	-	-	-

Figure C-1. Particle Size Distribution Curves for Sludge Composite Samples from Tank 241-Z-361



APPENDIX D

DETAILED STATISTICAL FORMULAS AND CALCULATIONS

D1.0 INTRODUCTION

Choosing a method for the analysis of the results for the composite samples is difficult due to the limited sample size and different sample supports associated with each of the composite samples. Logistical considerations dominated the determination of the number of samples as well as the compositing scheme. A detailed explanation of logic behind the decision to combine certain layers of the sludge for analysis can be found in the *Tank 241-Z-361 Sludge Sample Compositing Plan* (EQM 1999). The extreme heterogeneity of the sludge is reflected in the lack of correspondence between the number, depth and consistency of layers found in the cores that were taken in the sludge only a few feet apart from one another. The numerous distinct layers found in each core, coupled with lack of consistency between the cores, show that there are not homogeneous layers of uniform thickness across the horizontal extent of the tank in the sludge. From the results of the two core samples, it appears that there are numerous layers of varying thickness that do not necessarily span the horizontal extent of the tank although the presence of several such layers is not entirely out of the question.

Because of this extreme heterogeneity, it was decided that any attempts to account for the differences in the sample support between composite samples would be of little utility. Due to the small sample size, quantitative assessment of the concentrations is somewhat questionable as well. The majority of the presentation of the data from the composite samples lies in the qualitative realm. Core plots are used to represent the spatial distribution of the data. Exploratory data analysis (EDA) plots are used to examine the distribution of the results of the composite sampling for each analyte. Explanations of these graphical representations of the data can be found in the main text.

For both the metals and radionuclide suites, nonparametric upper confidence limits (UCLs) for the mean were calculated. Depending on the distribution of the underlying population and the sample size, asymptotic results from the central limit theorem regarding the distribution of sample mean can be invoked to justify the assumption of normality needed for the calculation of parametric confidence intervals. A nonparametric method for calculating the UCL was used due to the small sample size and corresponding potential lack of normality for the distribution of the sample means. These UCLs were calculated using a re-sampling technique that is essentially a bootstrap estimate of the sampling distribution for the mean of each analyte based on the results of the composite sampling. In this nonparametric method, the data are sampled, with replacement, to create a new data set of the same size as the original data set. This process is repeated 5,000 times, so that there are 5,000 new data sets of the same size as the original. Then the 90th or 95th percentile of the 5,000 sample means is calculated. These UCLs can be considered equivalent to the critical values for a one-sided hypothesis tests on the mean at the alpha levels of 0.10 and 0.05, respectively. Decisions based on UCLs calculated with such small samples sizes regarding a population with extreme inherent variability are questionable. Despite this, the results of the UCLs can be incorporated with logistical considerations and used as a component of the decision analysis for each of the analytes of interest in the sludge.

No further quantitative analyses for radionuclides were performed, since this was a component of the criticality assessment. However, hypothesis tests were used to compare metals to the Toxicity Characteristic Leachate Procedure action limits (where available). The parametric test

used to compare the center (mean) of a distribution to a threshold such as an action limit is the one-sample t-test. Although the one-sample t-test is somewhat robust with respect to departures from normality, EDA indicated that the lack of normality in the distributions of the composite samples was extreme enough in some cases that it indicated the need to consider an alternative to parametric hypothesis tests. The fact that multiple detection limits were encountered for numerous analytes also factored into the decision not to use the one-sample t-test. Currently there does not exist a modification to the one-sample t-test that allows for the accommodation of multiple detection limits in the data set. The Wilcoxon Signed Rank (WSR) test is the nonparametric analog of the one-sample t-test. The Gehan modification of the WSR test deals with the presence of multiple detection limits. Consider the WSR test of the following hypotheses,

$$Ho: \mu \geq AL \quad \text{vs.} \quad Ha: \mu < AL \tag{D-1}$$

where:

μ	=	the mean of the concentrations for the analyte of interest in the sludge and
AL	=	the corresponding action limit for the analyte of interest.

Because of the multiple detection limits and the departure from normality for most analytes, WSR was used to ensure a more uniform evaluation of the analytes to the corresponding action limits.

D1.1 WILCOXON SIGNED RANK TEST

Many common nonparametric tests are based on the relative ordering of the data. Placing the data in order, relative to one another, is called ranking. The first step in performing the WSR test is to shift the data according to the action limit in the hypothesis. This is done by taking each of the data points and subtracting the corresponding action limit. This gives us an idea of how the data are distributed relative to the action limit stated in the null hypothesis. The next step is to rank the absolute values of the shifted data. The ranking is performed in such a manner that the lowest absolute value is assigned a 1 and the highest absolute value is assigned a number equal to the number of samples. If nondetected values must be dealt with, this is where the Gehan ranking scheme (discussed below) is utilized. The absolute values are ranked because we are interested in determining if the relative magnitudes of the deviations from the hypothesized value are greater for those observations above the hypothesized value than for those observations below the hypothesized value. The third step is to compute the positive signed rank of the data.

The positive signed rank is nothing more than the sum of the corresponding ranks of all the values that are greater than the action limit in the data set. The factors that lend weight to the rejection of the null hypothesis are:

1. How many of the observations are greater/less than the hypothesized value? and
2. Are the observations that are greater than the hypothesized value farther away than those observations that are less than the hypothesized value?

These two pieces of information are summarized in equation (D-2), the test statistic.

$$W^{+} = \sum_{i=1}^{m} R^{+} \tag{D-2}$$

This is just the sum of the positive signed ranks. Combinatorial calculations are used to calculate critical values for the WSR test. Table D-1 presents the critical values for the WSR test at a significance level of 0.05.

Table D-1. Critical Values for WSR Test at $\alpha = 0.05$

Number of Subsets (n)	Two-Sided	One-Sided
6	0	2
7	2	3
8	3	5
9	5	8
10	8	10
11	10	13
12	13	17
13	17	21
14	21	25
15	25	30
16	29	35
17	34	41
18	40	47
19	46	53
20	52	60
21	58	67
22	65	75
23	73	83
24	81	91
25	89	100

If sample sizes are large enough, W+ has an approximate normal distribution with the mean and standard deviation defined as follows:

$$\mu = \left(\frac{n(n+1)}{4}\right) \qquad \sigma = \left(\frac{n(n+1)(2n+1)}{24}\right) \tag{D-3}$$

Using this information, the following statistic has an approximate standard normal distribution,

$$\frac{W^{+} - \mu}{\sigma} \tag{D-4}$$

This can be used to estimate critical values and p-values for the WSR test when sample sizes are large enough.

D2.0 GEHAN RANKING

Now a ranking method should be considered to be used in conjunction with the WSR test that is appropriate when data are censored at different detection limits. The method used to deal with this situation is called the Gehan ranking scheme. The best way to become familiar with the Gehan ranking scheme is to consider a small example. This example ranks the data directly, without first shifting the data by subtracting the action limit. For the WSR tests in this report, the data were first shifted appropriately.

Suppose we have a sample with the following values. The "<" symbol in front of a value denotes a nondetect.

Sample = {1, <4, 5, 7, <12, 15, 2, <4, 17, 8, 24}

Given this information, multiple detection limits to deal with in this data set can be seen.

First consider the initial ranks of the data set as if there were no detection limit issues. These values will be called the initial ranks.

{1, 2, <4, <4, 5, 7, 8, <12, 15, 17}

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank										

A Gehan rank is now assigned to each one of these data values. The Gehan rank could then be used to perform the WSR test.

It is easiest to start with the largest values when doing the Gehan ranking. Here are some guiding principles for the Gehan ranking scheme.

1. First rank the data in order as if the detection status were irrelevant.
2. Start with the highest value and work your way to the smallest value.
3. Determine the lowest and highest possible ranks that each datum can have.
4. Nondetects are assumed to be tied with all of the values below them.

In this example, the value of 17 receives a rank of 10 because is larger than all of the other values, even in the presence of nondetects.

The value of 15 receives a rank of 9 because is larger than all of the other values below it even in the presence of nondetects.

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank									9	10

The <12 value is a nondetect and therefore it is assumed to be tied with all of the values below it.

The averages of all of the ranks up to and including the rank of <12 are now calculated.

(1+2+3+4+5+6+7+8)/8 = 36/8 = 4.5.

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank								4.5	9	10

The value of 8 is certainly greater the six values below it regardless of the detection status of the values below it.

Therefore the rank of 8 must be at least 7.

The value of 8 is treated as a tie with <12 but it is certainly less than 15 and 17.

Therefore ,the rank of 8 can be at most 8.

As such we take the Gehan rank of 8 to be the average of 7and 8 which is 7.5.

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank							7.5	4.5	9	10

The logic is identical to the previous example. The value of 7 is certainly greater the five values below it regardless of the detection status of the values below it.

Therefore the rank of 7 must be at least 6.

The value of 7 is treated as a tie with <12 but it is certainly less than 8, 15 and 17.

Therefore the rank of 7 can be at most 7.

As such we take the Gehan rank of 7 to be the average of 6 and 7 which is 6.5.

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank						6.5	7.5	4.5	9	10

The logic is again identical. The value of 5 is certainly greater the four values below it regardless of the detection status of the values below it.

Therefore the rank of 5 must be at least 5.

The value of 5 is treated as a tie with <12 but it is certainly less than 7, 8, 15 and 17.

Therefore the rank of 5 can be at most 6.

As such we take the Gehan rank of 5 to be the average of 5 and 6 which is 5.5.

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank					5.5	6.5	7.5	4.5	9	10

The two values of <4 are certainly less than 5, 7, 8, 15 and 17.

Therefore the rank of <4 is at most 5.

The value of <4 are taken to be tied with the value of <12 as well as the values 1 and 2.

Therefore the values of <4 receive the Gehan rank that is the average of the initial ranks of the values with which it is tied.

Therefore, the Gehan rank of the value <4 is the average of 1, 2, 3, 4 and 5.

So the rank of <4 = (1+2+3+4+5)/5 = 15/5 = 3.

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank			3	3	5.5	6.5	7.5	4.5	9	10

The value of 2 is certainly greater than the value of 1.

Therefore, the Gehan rank of 2 is at least 2.

The value of 2 is treated as tied with <4, <4, and <12.

Therefore the Gehan rank of the value of 2 is the average of 2, 3, 4, and 5.

So the Gehan rank of 2 is (2+3+4+5)/4 = 3.5.

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank		3.5	3	3	5.5	6.5	7.5	4.5	9	10

Finally, the value of 1 certainly less than all of the detected values.

Therefore the Gehan rank of 1 is at most 4.

The value of 1 is treated as tied with the values <4, <4 and <12.

Therefore the Gehan rank of 1 is the average of the initial ranks 1, 2, 3 and 4.

Therefore, the Gehan rank of the value 1 is (1+2+3+4)/4 = (10/4) = 2.5.

In conclusion, we have the following Gehan ranks that can now be used in the WSR test.

Value	1	2	<4	<4	5	7	8	<12	15	17
Initial Rank	1	2	3	4	5	6	7	8	9	10
Gehan Rank	2.5	3.5	3	3	5.5	6.5	7.5	4.5	9	10

D3.0 BIBLIOGRAPHY AND REFERENCE

D3.1 BIBLIOGRAPHY

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Hollander, M., and Wolfe, D.A., 1999, *Nonparametric Statistical Methods,* John Wiley and Sons, New York.

Lehmann, E.L., 1975, *Nonparametrics: Statistical Methods Based on Ranks*, Holden-Day, Inc. California.

D3.2 REFERENCE

EQM, 1999, *Tank 241-Z-361 Sludge Sample Compositing Plan*, Rev. 0, December 9, 1999, Prepared by Environmental Quality Management for Fluor Daniel Hanford, Inc., Richland, Washington.

APPENDIX E

DRY WELL LOGGING DATA

Am r1 dep	Am r1 conc	Am r1 unc	Am r1 mda	Am r2 dep	Am r2 conc	Am r2 unc	Am r2 mda	Am r3 dep	Am r3 con	Am r3 unc	Am r3 mda
13.920000	1694140.000000	7200.000000	9034.000000	19.670000	565550.000000	3234.000000	4193.000000	17.170000	1332453.000000	3345.000000	4661.000000
14.420000	2984511.000000	11924.000000	15920.000000	19.420000	762934.000000	3973.000000	5259.000000	16.920000	2938665.000000	6676.000000	9485.000000
14.920000	6313750.000000	26000.000000	38581.000000	19.170000	1136350.000000	5365.000000	7323.000000	16.670000	2764644.000000	6417.000000	9416.000000
15.420000	2837692.000000	11741.000000	16573.000000	18.920000	1316757.000000	6017.000000	8201.000000	16.420000	2826484.000000	6489.000000	9376.000000
15.920000	4140884.000000	16918.000000	24366.000000	18.670000	791030.000000	4183.000000	6082.000000	16.170000	4963924.000000	11094.000000	16130.000000
16.420000	3148394.000000	13168.000000	19074.000000	18.420000	1018353.000000	4987.000000	7007.000000	15.920000	4064977.000000	9104.000000	13143.000000
16.920000	2457874.000000	10426.000000	14864.000000	18.170000	1410017.000000	6310.000000	8515.000000	15.670000	3296800.000000	7370.000000	10386.000000
17.420000	1063229.000000	5224.000000	7364.000000	17.920000	1536866.000000	6790.000000	9251.000000	15.420000	2454793.000000	5616.000000	7874.000000
17.920000	1556045.000000	6873.000000	9189.000000	17.670000	1494099.000000	6544.000000	8593.000000	15.170000	3916450.000000	8876.000000	13031.000000
18.420000	999544.000000	4969.000000	6985.000000	17.420000	972003.000000	4758.000000	6486.000000				
18.670000	1175478.000000	5589.000000	7663.000000	17.170000	1350031.000000	6168.000000	8568.000000				
				16.920000	2921090.000000	12142.000000	17319.000000				
				16.670000	2794445.000000	11833.000000	17354.000000				
				16.420000	2858454.000000	11968.000000	17301.000000				
				16.170000	5013282.000000	20397.000000	29556.000000				
				15.920000	4089756.000000	16699.000000	24089.000000				
				15.670000	3343389.000000	13601.000000	19088.000000				
				15.420000	2487866.000000	10364.000000	14509.000000				
				15.170000	3949005.000000	16333.000000	24004.000000				
				14.920000	8702607.000000	35879.000000	53647.000000				
				14.670000	4214788.000000	17029.000000	24232.000000				
				14.420000	2732199.000000	10930.000000	14359.000000				
				14.170000	2590232.000000	10306.000000	13219.000000				
				13.920000	1394936.000000	6018.000000	7247.000000				
				13.670000	2438003.000000	9364.000000	10758.000000				

Gross dep	Gross cps	Gross r2 dep	Gross r2 cps	Gross r3 dep	Gross r3 cps
1.420000	35.000000	19.670000	13218.000000	17.170000	35507.000000
1.920000	224.000000	19.420000	18541.000000	16.920000	85616.000000
2.420000	2459.000000	19.170000	29353.000000	16.670000	83657.000000
2.920000	3807.000000	18.920000	34339.000000	16.420000	83643.000000
3.420000	4307.000000	18.670000	21949.000000	16.170000	155360.000000
3.920000	4821.000000	18.420000	27305.000000	15.920000	123558.000000
4.420000	5406.000000	18.170000	36055.000000	15.670000	95162.000000
4.920000	6155.000000	17.920000	39976.000000	15.420000	68637.000000
5.420000	7156.000000	17.670000	36993.000000	15.170000	121539.000000
5.920000	8274.000000	17.420000	24671.000000		
6.420000	9534.000000	17.170000	35903.000000		
6.920000	11002.000000	16.920000	85299.000000		
7.420000	12673.000000	16.670000	84447.000000		
7.920000	14374.000000	16.420000	84491.000000		
8.420000	16462.000000	16.170000	156230.000000		
8.920000	18775.000000	15.920000	123935.000000		
9.430000	21432.000000	15.670000	95972.000000		
9.930000	24553.000000	15.420000	69381.000000		
10.420000	27846.000000	15.170000	122627.000000		
11.420000	36394.000000	14.920000	295870.000000		
11.920000	41880.000000	14.670000	125295.000000		
12.420000	48781.000000	14.420000	70387.000000		
12.920000	57829.000000	14.170000	64485.000000		
13.420000	61244.000000	13.920000	30841.000000		
13.920000	40294.000000	13.670000	53390.000000		
14.420000	79063.000000	13.420000	63623.000000		
14.920000	206536.000000	13.170000	63453.000000		
15.420000	80933.000000	12.920000	59232.000000		
15.920000	125344.000000	12.670000	55026.000000		
16.420000	94531.000000	12.420000	50467.000000		
16.920000	70933.000000	12.170000	46753.000000		
17.420000	28628.000000	11.920000	43292.000000		
17.920000	39630.000000	11.670000	40240.000000		
18.420000	26845.000000	11.420000	37529.000000		
18.670000	30736.000000	11.170000	35333.000000		

Am r1 cps dep	Am r1 cps	Am r1 cps unc	Am r1 cps mda	Am r2 cps dep	Am r2 cps	Am r2 cps unc	Am r2 cps mda	Am r3 cps dep	Am r3 cps	Am r3 cps unc	Am r3 cps mda
1.420000	5.000000	0.820000	2.380000	19.670000	5387.000000	30.810000	39.940000	17.170000	12692.000000	36.920000	44.400000
1.920000	146.000000	4.450000	4.690000	19.420000	7267.000000	37.850000	50.100000	16.920000	27993.000000	51.500000	90.350000
2.420000	938.000000	11.500000	7.040000	19.170000	10824.000000	51.110000	69.750000	16.670000	26335.000000	51.160000	89.690000
2.920000	786.000000	10.720000	5.670000	18.920000	12543.000000	57.310000	78.120000	16.420000	26924.000000	51.160000	89.310000
3.420000	979.000000	12.000000	2.390000	18.670000	7535.000000	39.850000	57.930000	16.170000	47284.000000	59.080000	153.640000
3.920000	1231.000000	13.450000	34.840000	18.420000	9700.000000	47.500000	66.750000	15.920000	38721.000000	56.420000	125.200000
4.420000	1617.000000	15.510000	36.770000	18.170000	13431.000000	60.110000	81.110000	15.670000	31404.000000	53.000000	98.930000
4.920000	1982.000000	17.420000	40.570000	17.920000	14640.000000	64.680000	88.120000	15.420000	23383.000000	48.160000	75.000000
5.420000	2905.000000	21.250000	28.430000	17.670000	14232.000000	62.340000	81.860000	15.170000	37307.000000	56.220000	124.120000
5.920000	3329.000000	23.020000	28.390000	17.420000	9259.000000	45.320000	61.780000				
6.420000	3952.000000	25.390000	33.140000	17.170000	12860.000000	58.760000	81.610000				
6.920000	4631.000000	28.000000	35.700000	16.920000	27825.000000	115.660000	164.970000				
7.420000	5331.000000	30.680000	38.990000	16.670000	26619.000000	112.710000	165.310000				
7.920000	6121.000000	33.410000	41.580000	16.420000	27229.000000	114.000000	164.800000				
8.420000	7044.000000	36.670000	45.130000	16.170000	47755.000000	194.290000	281.540000				
8.920000	8096.000000	40.100000	48.810000	15.920000	38957.000000	159.070000	229.460000				
9.430000	9368.000000	44.500000	53.070000	15.670000	31848.000000	129.560000	181.820000				
9.930000	10732.000000	49.150000	58.230000	15.420000	23698.000000	98.730000	138.210000				
10.420000	12234.000000	54.100000	63.250000	15.170000	37617.000000	155.580000	228.650000				
11.420000	15822.000000	65.800000	76.850000	14.920000	82898.000000	341.770000	511.020000				
11.920000	18120.000000	73.310000	85.350000	14.670000	40148.000000	162.210000	230.820000				
12.420000	20757.000000	82.330000	96.260000	14.420000	26026.000000	104.110000	136.780000				
12.920000	24237.000000	94.310000	110.510000	14.170000	24674.000000	98.180000	125.920000				
13.420000	26343.000000	99.690000	114.430000	13.920000	13288.000000	57.330000	69.030000				
13.920000	16138.000000	68.590000	86.050000	13.670000	23223.000000	89.200000	102.480000				
14.420000	28429.000000	113.590000	151.650000	13.420000	27178.000000	102.430000	118.130000				
14.920000	60142.000000	247.670000	367.500000	13.170000	27225.000000	102.510000	117.950000				
15.420000	27031.000000	111.840000	157.860000	12.920000	25482.000000	96.790000	111.450000				
15.920000	39445.000000	161.160000	232.100000	12.670000	23728.000000	91.120000	105.350000				
16.420000	29990.000000	125.430000	181.690000	12.420000	21978.000000	85.200000	97.960000				
16.920000	23413.000000	99.310000	141.590000	12.170000	20524.000000	80.320000	92.250000				
17.420000	10128.000000	49.760000	70.150000	11.920000	19164.000000	75.790000	86.880000				
17.920000	14822.000000	65.470000	87.530000	11.670000	17896.000000	71.590000	81.980000				
18.420000	9521.000000	47.330000	66.540000	11.420000	16778.000000	67.970000	77.890000				
18.670000	11197.000000	53.240000	72.990000	11.170000	15822.000000	64.910000	74.620000				

Np depth	Np conc	Np unc	Np mda	Pu 375 dep	Pu 375 con	Pu 375 unc	Pu 375 mda	Pu 414 dep	Pu 414 con	Pu 414 unc	Pu 414 mda
13.920000	1669.000000	889.000000	1544.000000	16.670000	44945987.000000	9982280.000000	29855875.000000	16.670000	37476826.000000	10123863.000000	26323248.000000
14.420000	6238.000000	2232.000000	2764.000000								
15.420000	2768.000000	806.000000	2026.000000								
15.670000	6037.000000	2470.000000	3814.000000								
15.670000	4298.000000	1137.000000	2270.000000								
15.920000	3802.000000	1190.000000	3129.000000								
16.170000	4511.000000	1429.000000	3960.000000								

Depth (ft)	Deadtime (%)	Count Rate Error	Corrected Peak MDA (cps)	Corrected Peak Count Rate (cps	Corrected Gross Count Rate (cps	Calculated Conc. Error	Calculated Conc. MDA	Calculated Conc. (pCi/g)
1.420000	0.000000	0.820000	2.380000	5.000000	35.000000	86.000000	249.000000	520.000000
1.920000	0.410000	4.450000	4.690000	146.000000	224.000000	468.000000	492.000000	15327.000000
2.420000	4.700000	11.500000	7.040000	938.000000	2459.000000	1207.000000	739.000000	98461.000000
2.920000	7.000000	10.720000	5.670000	786.000000	3807.000000	1125.000000	595.000000	82472.000000
3.420000	7.920000	12.000000	2.390000	979.000000	4307.000000	1259.000000	251.000000	102744.000000
3.920000	8.720000	13.450000	34.840000	1231.000000	4821.000000	1412.000000	3658.000000	129230.000000
4.420000	9.770000	15.510000	36.770000	1617.000000	5406.000000	1628.000000	3861.000000	169780.000000
4.920000	10.910000	17.420000	40.570000	1982.000000	6155.000000	1828.000000	4260.000000	208041.000000
5.420000	12.420000	21.250000	28.430000	2905.000000	7156.000000	2230.000000	2984.000000	304979.000000
5.920000	13.900000	23.020000	28.390000	3329.000000	8274.000000	2416.000000	2981.000000	349435.000000
6.420000	15.640000	25.390000	33.140000	3952.000000	9534.000000	2666.000000	3479.000000	414852.000000
6.920000	17.510000	28.000000	35.700000	4631.000000	11002.000000	2939.000000	3747.000000	486110.000000
7.420000	19.430000	30.680000	38.990000	5331.000000	12673.000000	3221.000000	4093.000000	559694.000000
7.920000	21.350000	33.410000	41.580000	6121.000000	14374.000000	3507.000000	4365.000000	642622.000000
8.420000	23.520000	36.670000	45.130000	7044.000000	16462.000000	3850.000000	4738.000000	739479.000000
8.920000	25.640000	40.100000	48.810000	8096.000000	18775.000000	4210.000000	5124.000000	849952.000000
9.430000	27.910000	44.500000	53.070000	9368.000000	21432.000000	4672.000000	5571.000000	983477.000000
9.930000	30.230000	49.150000	58.230000	10732.000000	24553.000000	5160.000000	6113.000000	1126684.000000
10.420000	32.540000	54.100000	63.250000	12234.000000	27846.000000	5680.000000	6640.000000	1284367.000000
11.420000	37.340000	65.800000	76.850000	15822.000000	36394.000000	6908.000000	8068.000000	1661033.000000
11.920000	39.810000	73.310000	85.350000	18120.000000	41880.000000	7696.000000	8960.000000	1902278.000000
12.420000	42.510000	82.330000	96.260000	20757.000000	48781.000000	8643.000000	10105.000000	2179031.000000
12.920000	45.400000	94.310000	110.510000	24237.000000	57829.000000	9900.000000	11601.000000	2544421.000000
13.420000	46.360000	99.690000	114.430000	26343.000000	61244.000000	10466.000000	12013.000000	2765441.000000
13.920000	39.200000	68.590000	86.050000	16138.000000	40294.000000	7200.000000	9034.000000	1694140.000000
14.420000	50.330000	113.590000	151.650000	28429.000000	79063.000000	11924.000000	15920.000000	2984511.000000
14.920000	61.950000	247.670000	367.500000	60142.000000	206536.000000	26000.000000	38581.000000	6313750.000000
15.420000	50.700000	111.840000	157.860000	27031.000000	80933.000000	11741.000000	16573.000000	2837692.000000
15.920000	56.600000	161.160000	232.100000	39445.000000	125344.000000	16918.000000	24366.000000	4140884.000000
16.420000	52.920000	125.430000	181.690000	29990.000000	94531.000000	13168.000000	19074.000000	3148394.000000
16.920000	48.730000	99.310000	141.590000	23413.000000	70933.000000	10426.000000	14864.000000	2457874.000000
17.420000	33.060000	49.760000	70.150000	10128.000000	28628.000000	5224.000000	7364.000000	1063229.000000
17.920000	38.860000	65.470000	87.530000	14822.000000	39630.000000	6873.000000	9189.000000	1556045.000000
18.420000	31.930000	47.330000	66.540000	9521.000000	26845.000000	4969.000000	6985.000000	999544.000000
18.670000	34.360000	53.240000	72.990000	11197.000000	30736.000000	5589.000000	7663.000000	1175478.000000
19.670000	20.130000	30.810000	39.940000	5387.000000	13218.000000	3234.000000	4193.000000	565550.000000
19.420000	25.470000	37.850000	50.100000	7267.000000	18541.000000	3973.000000	5259.000000	762934.000000
19.170000	33.530000	51.110000	69.750000	10824.000000	29353.000000	5365.000000	7323.000000	1136350.000000
18.920000	36.330000	57.310000	78.120000	12543.000000	34339.000000	6017.000000	8201.000000	1316757.000000
18.670000	28.330000	39.850000	57.930000	7535.000000	21949.000000	4183.000000	6082.000000	791030.000000
18.420000	32.200000	47.500000	66.750000	9700.000000	27305.000000	4987.000000	7007.000000	1018353.000000
18.170000	37.200000	60.110000	81.110000	13431.000000	36055.000000	6310.000000	8515.000000	1410017.000000
17.920000	39.070000	64.680000	88.120000	14640.000000	39976.000000	6790.000000	9251.000000	1536866.000000
17.670000	37.670000	62.340000	81.860000	14232.000000	36993.000000	6544.000000	8593.000000	1494099.000000
17.420000	30.400000	45.320000	61.780000	9259.000000	24671.000000	4758.000000	6486.000000	972003.000000
17.170000	37.130000	58.760000	81.610000	12860.000000	35903.000000	6168.000000	8568.000000	1350031.000000
16.920000	51.470000	115.660000	164.970000	27825.000000	85299.000000	12142.000000	17319.000000	2921090.000000
16.670000	51.330000	112.710000	165.310000	26619.000000	84447.000000	11833.000000	17354.000000	2794445.000000
16.420000	51.330000	114.000000	164.800000	27229.000000	84491.000000	11968.000000	17301.000000	2858454.000000
16.170000	59.130000	194.290000	281.540000	47755.000000	156230.000000	20397.000000	29556.000000	5013282.000000
15.920000	56.470000	159.070000	229.460000	38957.000000	123935.000000	16699.000000	24089.000000	4089756.000000
15.670000	53.130000	129.560000	181.820000	31848.000000	95972.000000	13601.000000	19088.000000	3343389.000000
15.420000	48.330000	98.730000	138.210000	23698.000000	69381.000000	10364.000000	14509.000000	2487866.000000
15.170000	56.330000	155.580000	228.650000	37617.000000	122627.000000	16333.000000	24004.000000	3949005.000000
14.920000	64.930000	341.770000	511.020000	82898.000000	295870.000000	35879.000000	53647.000000	8702607.000000

Depth (ft)	Deadtime (%)	Count Rate Error	Corrected Peak MDA (cps)	Corrected Peak Count Rate (cps	Corrected Gross Count Rate (cps	Calculated Conc. Error	Calculated Conc. MDA	Calculated Conc. (pCi/g)
14.670000	56.600000	162.210000	230.820000	40148.000000	125295.000000	17029.000000	24232.000000	4214788.000000
14.420000	48.600000	104.110000	136.780000	26026.000000	70387.000000	10930.000000	14359.000000	2732199.000000
14.170000	47.200000	98.180000	125.920000	24674.000000	64485.000000	10306.000000	13219.000000	2590232.000000
13.920000	34.400000	57.330000	69.030000	13288.000000	30841.000000	6018.000000	7247.000000	1394936.000000
13.670000	44.070000	89.200000	102.480000	23223.000000	53390.000000	9364.000000	10758.000000	2438003.000000
13.420000	46.930000	102.430000	118.130000	27178.000000	63623.000000	10753.000000	12401.000000	2853194.000000
13.170000	46.930000	102.510000	117.950000	27225.000000	63453.000000	10762.000000	12382.000000	2858032.000000
12.920000	45.800000	96.790000	111.450000	25482.000000	59232.000000	10161.000000	11700.000000	2675090.000000
12.670000	44.600000	91.120000	105.350000	23728.000000	55026.000000	9566.000000	11060.000000	2490952.000000
12.420000	43.130000	85.200000	97.960000	21978.000000	50467.000000	8945.000000	10283.000000	2307218.000000
12.170000	41.800000	80.320000	92.250000	20524.000000	46753.000000	8432.000000	9684.000000	2154610.000000
11.920000	40.470000	75.790000	86.880000	19164.000000	43292.000000	7956.000000	9121.000000	2011838.000000
11.670000	39.130000	71.590000	81.980000	17896.000000	40240.000000	7515.000000	8606.000000	1878773.000000
11.420000	37.930000	67.970000	77.890000	16778.000000	37529.000000	7135.000000	8177.000000	1761318.000000
11.170000	36.870000	64.910000	74.620000	15822.000000	35333.000000	6814.000000	7834.000000	1660991.000000
17.170000	36.920000	31.870000	44.400000	12692.000000	35507.000000	3345.000000	4661.000000	1332453.000000
16.920000	51.500000	63.590000	90.350000	27993.000000	85616.000000	6676.000000	9485.000000	2938665.000000
16.670000	51.160000	61.130000	89.690000	26335.000000	83657.000000	6417.000000	9416.000000	2764644.000000
16.420000	51.160000	61.810000	89.310000	26924.000000	83643.000000	6489.000000	9376.000000	2826484.000000
16.170000	59.080000	105.680000	153.640000	47284.000000	155360.000000	11094.000000	16130.000000	4963924.000000
15.920000	56.420000	86.720000	125.200000	38721.000000	123558.000000	9104.000000	13143.000000	4064977.000000
15.670000	53.000000	70.200000	98.930000	31404.000000	95162.000000	7370.000000	10386.000000	3296800.000000
15.420000	48.160000	53.500000	75.000000	23383.000000	68637.000000	5616.000000	7874.000000	2454793.000000
15.170000	56.220000	84.550000	124.120000	37307.000000	121539.000000	8876.000000	13031.000000	3916450.000000
16.670000	51.400000	30.430000	44.860000	26522.000000	85093.000000	3195.000000	4709.000000	2784298.000000
Np-137 312keV								
Depth (ft)	Deadtime (%)	Count Rate Error	Corrected Peak MDA (cps)	Corrected Peak Count Rate (cps	Corrected Gross Count Rate (cps	Calculated Conc. Error	Calculated Conc. MDA	Calculated Conc. (pCi/g)
11.670000	39.130000	0.610000	0.850000	1.300000	40240.000000	1169.000000	1626.000000	2489.000000
11.420000	37.930000	0.560000	0.800000	1.120000	37529.000000	1067.000000	1532.000000	2156.000000
12.920000	45.400000	0.840000	1.310000	1.910000	57829.000000	1605.000000	2507.000000	3664.000000
13.420000	46.360000	0.880000	1.450000	2.040000	61244.000000	1682.000000	2773.000000	3911.000000
13.670000	44.070000	0.680000	1.070000	1.330000	53390.000000	1295.000000	2056.000000	2552.000000
13.920000	34.400000	0.460000	0.810000	0.870000	30841.000000	889.000000	1544.000000	1669.000000
14.420000	48.600000	1.160000	1.440000	3.250000	70387.000000	2232.000000	2764.000000	6238.000000
15.420000	48.160000	0.420000	1.060000	1.440000	68637.000000	806.000000	2026.000000	2768.000000
15.670000	53.130000	1.290000	1.990000	3.150000	95972.000000	2470.000000	3814.000000	6037.000000
15.670000	53.000000	0.590000	1.180000	2.240000	95162.000000	1137.000000	2270.000000	4298.000000
15.920000	56.420000	0.620000	1.630000	1.980000	123558.000000	1190.000000	3129.000000	3802.000000
16.170000	59.080000	0.750000	2.070000	2.350000	155360.000000	1429.000000	3960.000000	4511.000000
Depth (ft)	Deadtime (%)	Count Rate Error	Corrected Peak MDA (cps)	Corrected Peak Count Rate (cps	Corrected Gross Count Rate (cps	Calculated Conc. Error	Calculated Conc. MDA	Calculated Conc. (pCi/g)
PU-239 375 keV								
depth	deadtime							
16.670000	51.400000	0.160000	0.470000	0.710000	85093.000000	9982280.000000	29855875.000000	44945987.000000
PU-239 414 keV								
16.670000	51.400000	0.130000	0.340000	0.480000	85093.000000	10123863.000000	26323248.000000	37476826.000000

Np cps dep	Np cps DT%	Np cps	Np cps unc	Np cps mda	Pu375 cps dep	Pu375 cps DT%	Pu375 cps	Pu375 cps unc	Pu375 cps mda	Pu414 cps dep	Pu414 cps DT%	Pu414 cps	Pu414 cps unc	Pu414 cps mda
13.920000	34.400000	0.715400	69.480000	0.662000	16.670000	51.400000	0.405464	44.320000	0.269334	16.670000	51.400000	0.274071	48.020000	0.192504
14.420000	48.600000	2.027000	41.820000	0.898200										
15.420000	48.160000	0.910300	50.290000	0.666300										
15.670000	53.130000	1.700000	51.630000	1.074000										
15.670000	53.000000	1.216000	39.430000	0.642100										
15.920000	56.420000	0.937400	55.630000	0.771500										
16.170000	59.080000	0.974100	58.540000	0.855000										

Riser B M	Riser B M	Riser B M	Riser B M	Riser B PN	Riser B PN	Riser B TG	Riser B TG	Riser G M	Riser G M	Riser G M	Riser G M	Riser G PN	Riser G PN	Riser G TG	Riser G TG
Depth	cps	Corr dep	Corr cps	Depth	cps	Depth	cps	Depth	cps	Corr depth	Corr cps	Depth	cps	Depth	cps
-0.41	7.67	6.210000	9.879000	-0.41	0.76	0.96	53.75	19.3	1656.07	8.17	8.417	18.8	112.94	0.42	157.22
-0.160000	13.120000	6.460000	8.770000	-0.16	0.34	1.46	90.22	19.05	1678.2	8.42	7.122	18.55	134.93	0.92	333.66
0.090000	30.750000	6.710000	8.486000	0.09	0.33	1.96	253.18	18.8	1739.88	8.67	8.151	18.3	150.6	1.19	517.34
0.340000	63.840000	6.960000	10.280000	0.34	0.2	2.46	1475.47	18.55	1754.5	8.92	7.455	18.05	179.87	1.42	1005.82
0.590000	93.110000	7.210000	11.670000	0.59	0.53	2.96	3087.05	18.3	1749.48	9.17	4.004	17.8	203.64	1.92	6868.95
0.840000	118.260000	7.460000	9.077000	0.84	0.6	3.46	3411.18	18.05	1751.73	9.42	1.922	17.55	213.27	2.17	16775.97
1.090000	139.500000	7.710000	10.920000	1.09	0.89	3.96	3535.6	17.8	1732.73	9.67	4.654	17.3	203.9	2.42	30963.9
1.340000	139.500000	7.960000	10.730000	1.34	2.15	4.46	3775.02	17.55	1735.8	9.92	7.806	17.05	212.7	2.92	43770.38
1.590000	139.500000	8.210000	11.110000	1.59	2.87	4.96	4102.77	17.3	1780.47	10.17	9.056	16.8	233.48	3.18	45660.67
1.840000	139.500000	8.460000	10.720000	1.84	5.44	5.46	4460.62	17.05	1792.23	10.42	5.644	16.55	260.24	3.42	49256.75
2.090000	139.500000	8.710000	11.330000	2.09	7.53	5.96	4874.58	16.8	1836.96	10.67	7.3	16.3	270.13	3.92	55007.33
2.340000	139.500000	8.960000	11.870000	2.34	17.23	6.46	5331.79	16.55	1794.18	10.92	8.884	16.05	262.71	4.42	62635.28
2.590000	139.500000	9.210000	13.770000	2.59	29.06	6.96	5654.57	16.3	1730.92	11.17	8.62	15.8	208.72	4.92	72190.88
2.840000	139.500000	9.460000	13.030000	2.84	37.33	7.46	5964.25	16.05	1739.75	11.42	10.91	15.55	162.54	5.42	84517.46
3.090000	139.500000	9.710000	14.540000	3.09	40.5	7.96	6250.08	15.8	1699.35	11.67	8.747	15.3	131.01	5.92	99004.77
3.340000	139.500000	9.960000	10.480000	3.34	42.52	8.46	6587.94	15.55	1626.03	11.92	5.683	15.05	122.55	6.42	118504
3.590000	139.500000	10.210000	9.719000	3.59	40.21	8.96	6856.57	15.3	1584.85	12.17	7.68	14.8	117.91	6.92	139718.9
3.840000	139.500000	10.460000	9.680000	3.84	41.44	9.46	7185.87	15.05	1622.17	12.42	11.5	14.55	112.43	7.42	166101.3
4.090000	139.500000	10.710000	8.016000	4.09	43.18	9.96	7493.21	14.8	1628.17	12.67	13.31	14.3	113.72	7.92	198861.5
4.340000	139.500000	10.960000	11.160000	4.34	40.11	10.46	7906.55	14.55	1608.76	12.92	15.32	14.05	107.34	8.42	238896.1
4.590000	139.500000	11.210000	11.380000	4.59	42.38	10.96	8520.16	14.3	1507.98	13.17	20.02	13.8	85.16	8.92	292899.2
4.840000	139.500000	11.460000	8.086000	4.84	40.14	11.46	9171.36	14.05	1110.77	13.42	38.86	13.55	80.73	9.42	369026.2
5.090000	139.500000	11.710000	7.584000	5.09	45.6	11.96	9549.42	13.8	403.28	13.67	182.2	13.3	77.42	9.92	477864.2
5.340000	139.500000	11.960000	8.630000	5.34	45.14	12.46	9489.69	13.55	135.81	13.92	651.5	13.05	76.79	10.42	658466.5
5.590000	139.500000	12.210000	9.806000	5.59	48.21	12.96	9198.87	13.3	100.04	14.17	1192	12.8	78.45	10.92	926399.3
5.840000	139.500000	12.460000	10.440000	5.84	46.59	13.46	8368.49	13.05	94.16	14.42	1443	12.55	76.19	11.42	999999
6.090000	139.500000	12.710000	15.810000	6.09	47.13	13.96	6847.06	12.8	92.51	14.67	1504	12.3	76.46	11.92	999999
6.340000	139.500000	12.960000	18.340000	6.34	50.86	14.46	12955.64	12.55	87.94	14.92	1506	12.05	78.53	12.42	999999
6.590000	139.500000	13.210000	19.390000	6.59	51.93	14.96	30488.06	12.3	87.84	15.17	1479	11.8	79.55	12.67	999999
6.840000	139.500000	13.460000	34.850000	6.84	53.43	15.46	34147.17	12.05	83.59	15.42	1465	11.55	76.11		
7.090000	139.500000	13.710000	138.700000	7.09	52.01	15.96	41597.19	11.8	86.2	15.67	1486	11.3	76.46		
7.340000	139.500000	13.960000	484.300000	7.34	57.57	16.46	46723.2	11.55	85.47	15.92	1495	11.05	78.29		
7.590000	139.500000	14.210000	867.100000	7.59	54.49	16.96	47885.87	11.3	88.93	16.17	1471	10.8	75.41		
7.840000	139.500000	14.460000	980.500000	7.84	54.78	17.46	36452.74	11.05	84.06	16.42	1494	10.55	77.55		
8.090000	139.500000	14.710000	950.400000	8.09	55.97	17.71	25393.71	10.8	86.06	16.67	1562	10.3	78.43		
8.340000	139.500000	14.960000	924.800000	8.34	59.05	17.81	28427.42	10.55	82.58	16.92	1588	10.05	76.56		
8.590000	139.500000	15.210000	929.300000	8.59	55.27			10.3	84.98	17.17	1576	9.8	78.35		
8.840000	139.500000	15.460000	941.700000	8.84	58.77	16.96	47889.97	10.05	87.21	17.42	1553	9.55	77.59		
9.090000	139.500000	15.710000	967.900000	9.09	57.04	16.46	46928.86	9.8	84.22	17.67	1524	9.3	80.28		
9.340000	139.500000	15.960000	993.500000	9.34	56.82	15.96	41490.71	9.55	80.83	17.92	1545	9.05	78.79		
9.590000	139.500000	16.210000	994.100000	9.59	55.98	15.46	34199.81	9.3	82.02	18.17	1579	8.8	75.51		
9.840000	139.500000	16.460000	979.600000	9.84	58.9	14.96	29960.7	9.05	84.31	18.42	1606	8.55	74.5		
10.090000	139.500000	16.710000	981.000000	10.09	57.84	14.46	12538.17	8.8	83.49	18.67	1619	8.42	75.96		
10.340000	139.500000	16.960000	1003.000000	10.34	56.01	13.96	6773.48	8.55	82.42	18.92	1603				
10.590000	139.500000	17.210000	1023.000000	10.59	56.84	13.46	8364.29	8.3	83.68			7.92	75.89		
10.840000	139.500000	17.460000	1036.000000	10.84	55.71	12.96	9235.14					7.42	70.06		
11.090000	139.500000	17.710000	1038.000000	11.09	55.14	12.46	9552.3					6.92	72.53		
11.340000	139.500000	17.960000	1013.000000	11.34	56.47	11.96	9565.86					6.42	74.66		
11.590000	139.500000	18.210000	993.900000	11.59	57.24	11.46	9262.06					5.92	71.24		
11.840000	139.500000	18.460000	1008.000000	11.84	55.7	11.21	8910.75					5.42	72.17		
12.090000	139.500000	18.710000	1031.000000	12.09	50.95							4.92	66.62		
12.340000	139.500000	18.960000	1045.000000	12.34	51.92	18.96	23597.08					4.42	66.31		
12.590000	139.500000	19.210000	1041.000000	12.59	50.99	18.46	26940.2					3.92	69.2		
12.840000	139.500000	19.460000	1033.000000	12.84	50.33	17.96	24675.73					3.42	65.78		
13.090000	139.500000	19.710000	1033.000000	13.09	49.93							2.92	60.39		
13.340000	139.500000	19.960000	1034.000000	13.34	52.41							2.42	49.1		

Riser B M	Riser B M	Riser B M	Riser B M	Riser B PN	Riser B PN	Riser B TG	Riser B TG	Riser G M	Riser G M	Riser G M	Riser G M	Riser G PN	Riser G PN	Riser G TG	Riser G TG
Depth	cps	Corr dep	Corr cps	Depth	cps	Depth	cps	Depth	cps	Corr depth	Corr cps	Depth	cps	Depth	cps
13.590000	139.500000	20.210000	1030.000000	13.59	47.57							1.92	23.23		
13.840000	139.500000	20.460000	1021.000000	13.84	41.15							1.42	7.48		
14.090000	139.500000			14.09	32.85							0.92	3.6		
14.340000	139.500000			14.34	38.48							0.42	1.82		
14.590000	139.500000			14.59	48.98							0.17	0		
Riser B M	Riser B M	Riser B M	Riser B M	Riser B PN	Riser B PN	Riser B TG	Riser B TG	Riser G M	Riser G M	Riser G M	Riser G M	Riser G PN	Riser G PN	Riser G TG	Riser G TG
Depth	cps	Corr dep	Corr cps	Depth	cps	Depth	cps	Depth	cps	Corr depth	Corr cps	Depth	cps	Depth	cps
14.84	154.815377	20.710000	1222.441525	14.84	66.20959016	19.12871401	29606.72151	8.05	-384.4134545	19.17	1887.889081	1.685710328	12.06535437	12.98716749	830125.6202
15.090000	155.579252	20.960000	1248.172825	15.09	67.04036489	19.40822401	30109.86443	7.8	-439.0136627	19.42	1940.133884	1.392855346	9.166337425	13.43985222	866719.2833
15.340000	156.343127	21.210000	1273.904126	15.34	67.87113961	19.68773401	30613.00734	7.55	-493.6138709	19.67	1992.378687	1.100000365	6.26732048	13.89253695	903312.9463
15.590000	157.107003	21.460000	1299.635426	15.59	68.70191433	19.96724401	31116.15026	7.3	-548.2140791	19.92	2044.623489	0.807145383	3.368303535	14.34522167	939906.6093
15.840000	157.870878	21.710000	1325.366727	15.84	69.53268905	20.24675401	31619.29318	7.05	-602.8142872	20.17	2096.868292	0.514290402	0.46928659	14.7979064	976500.2723
16.090000	158.634753	21.960000	1351.098028	16.09	70.36346378	20.52626401	32122.4361	6.8	-657.4144954	20.42	2149.113094	0.22143542	-2.429730355	15.25059113	1013093.935
16.340000	159.398628	22.210000	1376.829328	16.34	71.1942385	20.80577401	32625.57901	6.55	-712.0147036	20.67	2201.357897	-0.071419561	-5.328747299	15.70327586	1049687.598
16.590000	160.162503	22.460000	1402.560629	16.59	72.02501322	21.08528401	33128.72193	6.3	-766.6149117	20.92	2253.6027	-0.364274543	-8.227764244	16.15596059	1086281.261
16.840000	160.926379	22.710000	1428.291930	16.84	72.85578794	21.364794	33631.86485	6.05	-821.2151199	21.17	2305.847502	-0.657129524	-11.12678119	16.60864532	1122874.924
17.090000	161.690254	22.960000	1454.023230	17.09	73.68656267	21.644304	34135.00777	5.8	-875.8153281	21.42	2358.092305	-0.949984506	-14.02579813	17.06133005	1159468.587
17.340000	162.454129	23.210000	1479.754531	17.34	74.51733739	21.923814	34638.15068	5.55	-930.4155362	21.67	2410.337107	-1.242839487	-16.92481508	17.51401478	1196062.25
17.590000	163.218004	23.460000	1505.485832	17.59	75.34811211	22.203324	35141.2936	5.3	-985.0157444	21.92	2462.58191	-1.535694469	-19.82383202	17.96669951	1232655.913
17.840000	163.981879	23.710000	1531.217132	17.84	76.17888683	22.482834	35644.43652	5.05	-1039.615953	22.17	2514.826713	-1.82854945	-22.72284897	18.41938424	1269249.576
18.090000	164.745755	23.960000	1556.948433	18.09	77.00966155	22.762344	36147.57944	4.8	-1094.216161	22.42	2567.071515	-2.121404432	-25.62186591	18.87206897	1305843.239
18.340000	165.509630	24.210000	1582.679734	18.34	77.84043628	23.041854	36650.72236	4.55	-1148.816369	22.67	2619.316318	-2.414259413	-28.52088286	19.32475369	1342436.902
18.590000	166.273505	24.460000	1608.411034	18.59	78.671211	23.321364	37153.86527	4.3	-1203.416577	22.92	2671.561121	-2.707114395	-31.4198998	19.77743842	1379030.565
18.840000	167.037380	24.710000	1634.142335	18.84	79.50198572	23.600874	37657.00819	4.05	-1258.016785	23.17	2723.805923	-2.999969376	-34.31891675	20.23012315	1415624.228
19.090000	167.801255	24.960000	1659.873636	19.09	80.33276044	23.880384	38160.15111	3.8	-1312.616993	23.42	2776.050726	-3.292824358	-37.21793369	20.68280788	1452217.891
19.340000	168.565131	25.210000	1685.604936	19.34	81.16353517	24.159894	38663.29403	3.55	-1367.217202	23.67	2828.295528	-3.585679339	-40.11695064	21.13549261	1488811.554
19.590000	169.329006	25.460000	1711.336237	19.59	81.99430989	24.43940399	39166.43694	3.3	-1421.81741	23.92	2880.540331	-3.878534321	-43.01596758	21.58817734	1525405.217
19.840000	170.092881	25.710000	1737.067538	19.84	82.82508461	24.71891399	39669.57986	3.05	-1476.417618	24.17	2932.785134	-4.171389302	-45.91498453	22.04086207	1561998.88
20.090000	170.856756	25.960000	1762.798838	20.09	83.65585933	24.99842399	40172.72278	2.8	-1531.017826	24.42	2985.029936	-4.464244284	-48.81400147	22.4935468	1598592.543
20.340000	171.620631	26.210000	1788.530139	20.34	84.48663406	25.27793399	40675.8657	2.55	-1585.618034	24.67	3037.274739	-4.757099265	-51.71301842	22.94623153	1635186.206
20.590000	172.384507	26.460000	1814.261440	20.59	85.31740878	25.55744399	41179.00861	2.3	-1640.218242	24.92	3089.519541	-5.049954247	-54.61203536	23.39891626	1671779.869
20.840000	173.148382	26.710000	1839.992740	20.84	86.1481835	25.83695399	41682.15153	2.05	-1694.818451	25.17	3141.764344	-5.342809228	-57.51105231	23.85160099	1708373.532
21.090000	173.912257	26.960000	1865.724041	21.09	86.97895822	26.11646399	42185.29445	1.8	-1749.418659	25.42	3194.009147	-5.63566421	-60.41006925	24.30428571	1744967.195
21.340000	174.676132	27.210000	1891.455342	21.34	87.80973295	26.39597399	42688.43737	1.55	-1804.018867	25.67	3246.253949	-5.928519191	-63.3090862	24.75697044	1781560.858
21.590000	175.440007	27.460000	1917.186642	21.59	88.64050767	26.67548399	43191.58028	1.3	-1858.619075	25.92	3298.498752	-6.221374173	-66.20810314	25.20965517	1818154.521
21.840000	176.203883	27.710000	1942.917943	21.84	89.47128239	26.95499399	43694.7232	1.05	-1913.219283	26.17	3350.743554	-6.514229154	-69.10712009	25.6623399	1854748.184
22.090000	176.967758	27.960000	1968.649244	22.09	90.30205711	27.23450398	44197.86612	0.8	-1967.819491	26.42	3402.988357	-6.807084136	-72.00613703		
22.340000	177.731633	28.210000	1994.380544	22.34	91.13283184	27.51401398	44701.00904	0.55	-2022.4197	26.67	3455.23316	-7.099939117	-74.90515398		
22.590000	178.495508	28.460000	2020.111845	22.59	91.96360656	27.79352398	45204.15195	0.3	-2077.019908	26.92	3507.477962	-7.392794099	-77.80417092		
22.840000	179.259383	28.710000	2045.843145	22.84	92.79438128	28.07303398	45707.29487	0.05	-2131.620116	27.17	3559.722765	-7.68564908	-80.70318787		
23.090000	180.023259	28.960000	2071.574446	23.09	93.625156	28.35254398	46210.43779	-0.2	-2186.220324	27.42	3611.967567	-7.978504062	-83.60220481		
23.340000	180.787134	29.210000	2097.305747	23.34	94.45593072	28.63205398	46713.58071	-0.45	-2240.820532	27.67	3664.21237	-8.271359043	-86.50122176		
23.590000	181.551009	29.460000	2123.037047	23.59	95.28670545	28.91156398	47216.72362	-0.7	-2295.42074	27.92	3716.457173	-8.564214025	-89.4002387		
23.840000	182.314884	29.710000	2148.768348	23.84	96.11748017	29.19107398	47719.86654	-0.95	-2350.020949	28.17	3768.701975	-8.857069006	-92.29925565		
24.090000	183.078759	29.960000	2174.499649	24.09	96.94825489	29.47058398	48223.00946	-1.2	-2404.621157	28.42	3820.946778	-9.149923988	-95.19827259		
24.340000	183.842635	30.210000	2200.230949	24.34	97.77902961	29.75009398	48726.15238	-1.45	-2459.221365	28.67	3873.19158	-9.442778969	-98.09728954		