

**Tank Characterization Report
for Single-Shell Tank 241-T-107**

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EXECUTIVE SUMMARY

Single shell tank 241-T-107 is a Hanford Site Ferrocyanide Watch List tank most recently sampled in March 1993. Analyses of materials obtained from tank T-107 were conducted to support the Ferrocyanide Unreviewed Safety Question (USQ) and the *Hanford Federal Facility Agreement and Consent Order*¹ (Tri-Party Agreement) Milestone M-10-06 as well as Milestones M-44-05 and M-44-07². Characterization of the tank waste T-107 will support the ferrocyanide safety issue in order to classify the tank as safe, conditionally safe, or unsafe³. This tank characterization report expands on the data found in *Ferrocyanide Safety Program: Data Interpretation Report for Tank 241-T-107 Core Samples* (Sasaki et al. 1994).

Analysis of core samples obtained from tank T-107 strongly indicate the cyanide and oxidizer (nitrate/nitrite) concentrations in the tank waste are not significant enough to support a self-sustaining exothermic reaction. Therefore, the contents of tank T-107 present no imminent threat to the workers at the Hanford Site, the public, or the environment. Because the possibility of an exothermic reaction is remote, the consequences of an accident scenario, as proposed by the General Accounting Office, are not applicable.⁴

Review of the tank waste results have provided valuable insight about the physical and chemical conditions of the waste, and should allow tank T-107 to be categorized as *Safe*. The possibility existed tank 241-T-107 exceeded the previous 1,000 g-mol inventory criterion established for placement on the Ferrocyanide Watch List. Extensive energetic analysis was performed on bulk composites and segment level samples of all three cores, and in nearly all cases, no exotherms were observed. However, a piece of plastic debris found in the tank waste material was analyzed using a differential scanning calorimeter (DSC) and thermogravimetric analysis (TGA). The piece of plastic reacted with the oxygen in the cover gas and burned at approximately 300°C, producing an exothermic response. The analytical results indicate the piece of plastic was stable, not representative of the surrounding waste, and thus, no cause for concern. The final conclusion of the energetic analyses performed was that no observable exotherm attributable to the waste was detected.

¹Ecology, EPA, and DOE, 1992, *Hanford Facility Agreement and Consent Order*, 2 vols., Washington State Department of Ecology, U.S. Environmental Protection Agency, and U.S. Department of Energy, Olympia, Washington.

²Ecology, EPA, and DOE, 1994, *Hanford Federal Facility Agreement and Consent Order*, as amended, Washington State Department of Ecology, U.S. Environmental Protection Agency, and U. S. Department of Energy, Olympia, Washington.

³Postma, A. K., J. E. Meacham, R. J. Cash, G. S. Barney, G. L. Borsheim, M. D. Crippen, D. R. Dickinson, D. W. Jeppson, M. Kummerer, J. L. McLaren, C. S. Simmons, and B. C. Simpson, 1994, *Ferrocyanide Safety Program: Safety Criteria for Ferrocyanide Watchlist Tanks*, WHC-EP-0691, Westinghouse Hanford Company, Richland, Washington.

⁴Peach, J. D., 1990, "Consequences of Explosion of Hanford's Single-Shell Tank are Understated," (Letter B-241479 to C. M. Synar, Chairman of Environment, Energy, and Natural Resources Subcommittee, Committee on Government Operations, House of Representatives), GAO/RCED-91-34, General Accounting Office, Washington, D.C.

Other analytical results further suggest the tank presents a relatively low hazard. Cyanide concentrations, in the samples, are considerably lower than the presently established decision threshold of 8 wt% (80,000 $\mu\text{g/g}$) $\text{Na}_2\text{NiFe}(\text{CN})_6$ ³. A comparison of the core composite fusion and water digestion results indicate most of the cyanide is present in its water soluble form. A comparison of the core composite results for total organic carbon show a large discrepancy between the results for the two analytical methods, the hot persulfate oxidation method performed by 325 Laboratory and the coulometric detection method performed by 222-S Laboratory. Carbon results on the water digestion samples, using the coulometric detection method, are two to six times higher than results on the direct samples using the hot persulfate oxidation method. The results for total organic carbon indicate a general decreasing trend as a function of depth for both Core 51 and Core 52. Core 50 did not have enough data to make the same observation. Total organic carbon results for each segments were well under the accepted safety criteria. Radionuclide concentration results for ^{137}Cs , ^{154}Eu , and ^{241}Am were low, and this result is reflected in the tank's low heat load and temperature. The ^{137}Cs results indicate it is found in a largely water soluble form, while ^{90}Sr remains insoluble within the waste. In addition, an investigation into the concentrations of radionuclides and their potential mechanisms which generate temperatures high enough to be of concern (i.e. cause in-tank self-heating) was performed. No credible mechanisms were postulated that could initiate the generation of such temperature levels⁵ in the tank.

Tank T-107 waste is a complex material made up of water and inert salts. The insoluble solids are a mixture of phosphates, sulfates, and hydroxides in combination with aluminum, calcium, iron, nickel, and uranium. Examining the trends of these cations as a function of depth, suggest layering of the waste within the tank. Disodium nickel ferrocyanide and sodium cesium nickel ferrocyanide may exist, in small quantities, in the tank. The most prevalent soluble analytes found in tank T-107 are sodium, nitrate, and nitrite.

The results of the analyses have been compared to the dangerous waste codes in the Washington Dangerous Waste Regulations (WAC 173-303)⁶. This assessment was conducted by comparing tank analyses against dangerous waste characteristics ("D" waste codes) and against state waste codes. The comparison did not include checking tank analyses against "U", "P", "F", or "K" waste codes since application of these codes is dependent on the source of the waste and not on particular constituent concentrations. The results indicate that the waste in this tank is adequately described in the Dangerous Waste Permit Application for the Single-Shell Tank System⁷; this permit is discussed in the *Tank Characterization Reference Guide*⁸

⁵Dickinson, D. R., J. M. McLaren, G. L. Borsheim, M. D. Crippen, 1993 *Credibility of Drying Out Ferrocyanide Tank Waste Sludge by Hot Spots*, WHC-EP-0648, Westinghouse Hanford Company, Richland, Washington.

Ecology, 1991, WAC 173-303, "Dangerous Waste Regulations," WAC 173-303, *Washington Administrative Code*, as amended, Washington State Department of Ecology, Olympia, Washington.

⁷DOE, Richland Operations Office, 1993, "Dangerous Waste Permit Application for the Single-Shell Tank System," DOE/RL-88-21, Rev. 3, U. S. Department of Energy Field Office, Richland, Washington.

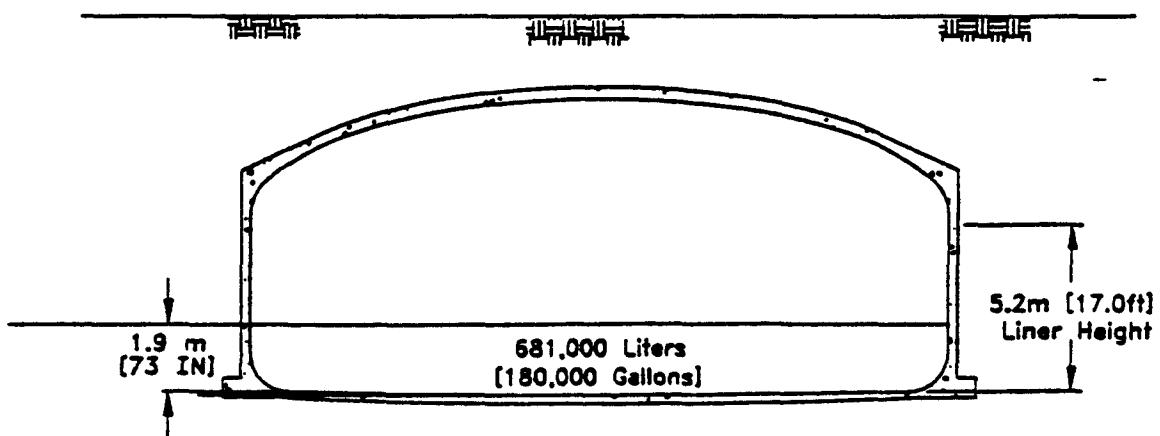
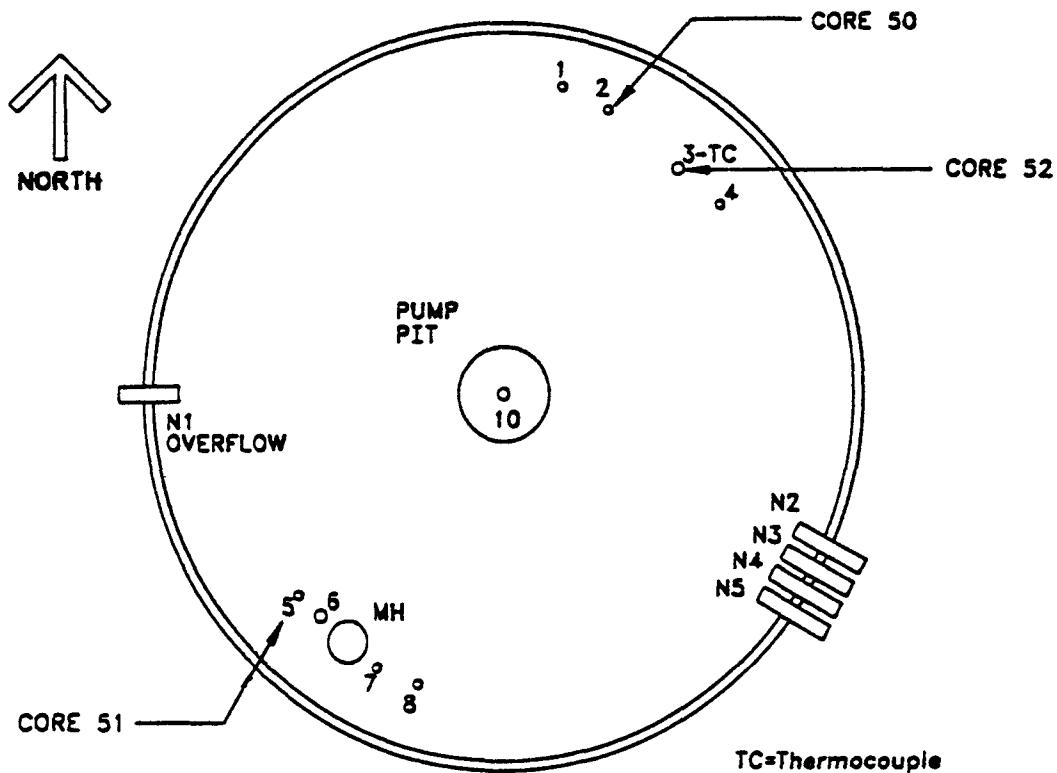
⁸DeLorenzo, D. S., J. H. Rutherford, D. J. Smith, D. B. Hiller, K.W. Johnson, and B. C. Simpson, 1994, *Tank Characterization Reference Guide*, WHC-SD-WM-TI-648, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

Comparison of the calculated inventories for various analytes of concern show tank T-107 are within the established operating safety requirements for heat-load, cyanide, total organic carbon, and moisture content. The analytical results suggest that an exothermic reaction in tank T-107 is unlikely and the tank should be categorized as *Safe*. A summary of the results is found on the following page (Tables ES-1 and ES-2). A summary of the primary and secondary data requirements to classify a ferrocyanide tank as safe, unconditionally safe, or unsafe, as well as the concentrations observed for tank T-107 waste, is found in Table ES-3. A figure showing the current configuration of the tank is shown in Figure ES-1.

Table ES-2	
TANK 241-T-107	
TANK DESCRIPTION	
Type:	Single-Shell
Constructed:	1944
In Service:	1945
Diameter:	75' (22.9 m)
Usable Depth:	17' (5.2 m)
Operating Capacity:	530,000 gal (2,010,000 L)
Bottom Shape:	Dished
Hanford Coordinates:	43,447.5 North 75,637.5 West
Ventilation:	Passive
TANK STATUS: as of May 1994	
Contents:	Non-Complexed Waste
Total Waste:	180,000 gal (681,000 L)
Supernate Volume:	9,000 gal (34,000 L)
Drainable Interstitial Liquid:	13,000 gal (49,200 L)
Manual Tape Surface Level:	73 inches
Liquid Observation Well Level:	None
Integrity Category:	Assumed Leaker
Watch List Status:	Ferrocyanide

Table ES-1		
Single-Shell Tank 241-T-107 Concentrations and Inventories for Critical List Analytes		
Physical Properties		
Density	1.51 g/mL	H ₂ O 56.0% 575,700 kg
Temperature	19°C	Heat Load 800 W (2720 Btu/h)
pH	11.5	Total Waste Mass 1,028,000 kg
Chemical Constituents	Average Concentration (wt%)	Bulk Inventory (kg)
Calcium (Ca)	0.076	780
Chromium (Cr)	0.036	370
Iron (Fe)	2.84	30,000
Manganese (Mn)	0.023	230
Sodium (Na)	13.04	134,000
Bismuth (Bi)	1.20	12,350
Lanthanum (La)	--	< DL
Silicon (Si)	0.60	6,200
Uranium (U)	2.62	27,000
Total Phosphate (PO ₄ ³⁻)	10.6	109,000
Sulfate (SO ₄ ²⁻)	1.0	10,300
Nitrate (NO ₃ ⁻)	7.49	77,000
Nitrite (NO ₂ ⁻)	1.17	12,000
Fluoride (F ⁻)	1.16	12,000
Cyanide (CN ⁻)	0.0069	71
Total Organic Carbon	0.15	1,600
Radionuclides	(μ Ci/g)	(Ci)
Total Plutonium	0.144	148
Am-241	0.0141	14.5
Sr-90	108	111,000
Cs-137	12.0	12,300

Figure ES-1. Tank T-107 Configuration.



Ref: Blue Print File
Number 73550
Drawing 2

ES-3. Primary and Secondary Data Requirements for Ferrocyanide Tanks⁵

Analyte	Analytical Method	Decision Threshold ¹	Results ²
Total Fuel ³	DSC/Adiabatic Calorimetry	8 wt% (.48 MJ/kg or 115 cal/g)	No Exotherms
Moisture Content	Thermogravimetric Analysis	4/3 [Fuel - 8]	46%
Tank Temperature	Thermocouple	90°C	19°C
¹³⁷ Cs	Gamma Energy Analysis	NL	38 µCi/g
⁹⁰ Sr	Beta Radiochemistry	NL	250 µCi/g
Total Cyanide	Direct Assay	NL	187 µg/g
Total Organic Carbon	Direct Persulfate Oxidation	NL	1,500 µg/g
Nickel	Inductively Coupled Plasma	NL	260 µg/g ⁴

¹ Excluding moisture and tank temperature. All decision thresholds reported on a dry basis.

² Results reported are tank averages on a dry basis. Excluding moisture and tank temperature.

³ Calculated on a Na₂NiFe(CN)₆ energy equivalent basis.

⁴ Nickel results are those obtained from acid digestion.

⁵ Postma 1994

NL = Not Limiting.

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TANK CHARACTERIZATION DATA REPORT: TANK 241-T-107

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LIST OF TERMS

AA	Atomic Absorption
ACL	Analytical Chemistry Laboratory
ANOVA	Analysis of Variance
CI	Confidence Interval
CW	Cladding Waste
CVAA	Cold Vapor Atomic Absorption
DL	Detection Limit
DQO	Data Quality Objectives
DSC	differential scanning calorimeter
EOX	extractable organic halides
delta H	change in heat
HLWV	high level waste vitrification
HSD	honestly significant difference
GEA	gamma energy analysis
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
IX	Ion Exchange Waste
NPH	non-paraffin hydrocarbon
PPT	precipitate
PUREX	Plutonium Uranium Redox Plant
REML	restricted maximum likelihood estimation
RPD	relative percent difference
SST	Single Shell Tank
sVOA	semi-volatile organic analysis
TC	Total Carbon
TIC	Total inorganic carbon
TGA	thermogravimetric analysis
TOC	Total organic carbon
TOX	total organic halides
TBP	Tributyl Phosphate Waste
USQ	Unreviewed Safety Question
VOA	Volatile Organic Analysis
1C	First Cycle Decontamination Waste

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TANK CHARACTERIZATION REPORT: TANK 241-T-107**1.0 INTRODUCTION**

Analysis was conducted on materials obtained from tank 241-T-107 to support the resolution of the Ferrocyanide Unreviewed Safety Question (USQ) and Safety Issue. In addition, several of the analytes contributing to the energetic properties of the waste need to be measured as a function of position (e.g., total cyanide, nitrate/nitrite, water content, and the distribution and inventory of ^{137}Cs , and ^{90}Sr in the tank). Other objectives that these measurements and inventory estimates support are as follows:

- Complete *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) Milestone M-10-06 (Ecology et al. 1992) to sample and analyze two cores from each tank.
- Complete the amended *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) Milestones M-44-07 and M-44-05 (Ecology et al. 1994).
- Obtain estimates of both the concentration and total quantity of key analytes relating to other safety issues, such as organics and radionuclides.
- Provide input to risk assessment-based disposal decisions for the waste.
- Measure/determine physical properties, such as rheology, bulk density, and particle size. These measurements are necessary for the design and fabrication of retrieval, pretreatment, and vitrification systems.
- Determine whether tank T-107 can be categorized as *Safe, Conditionally Safe, or Unsafe*.

This report expands on Sasaki and Valenzuela (1994) WHC-EP-0796, extending the characterization of the tank. This assessment includes not only those analytes pertinent to the Ferrocyanide Safety Issue, but all analytes of interest to the various technical programs.

1.1 PURPOSE

The purpose of the tank characterization report is to describe and characterize the waste in single shell tank (SST) 241-T-107, and arrange this information in a format useful for supporting management and technical decisions concerning the waste tank.

1.2 SCOPE

This report presents a broad background of information that was available prior to core sampling, which initially guided the development of the sampling and analysis program. This material includes process stream data, historical information, transfer records, and observations from in-tank photographs. The results of tank 241-T-107 core sample analyses

are summarized and presented, along with a statistical interpretation of the data. The information obtained from historical sources is compared and correlated with the actual waste measurements. As characterization efforts proceed and additional information becomes available, this document will be periodically revised to reflect the new data set. Analytical data will also be compared to programs' established data requirements which have been developed through the data quality objectives (DQO) process.

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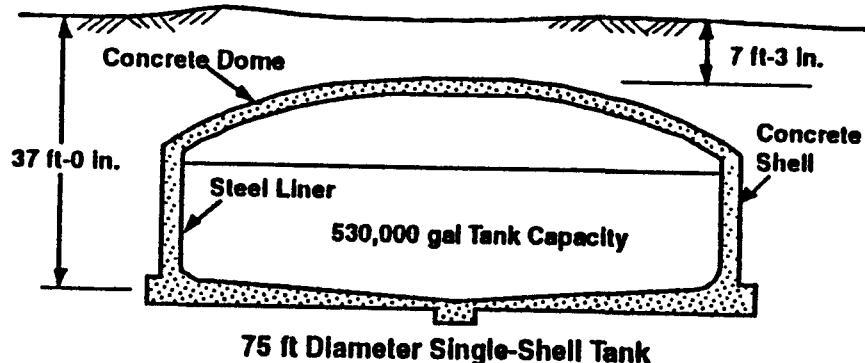
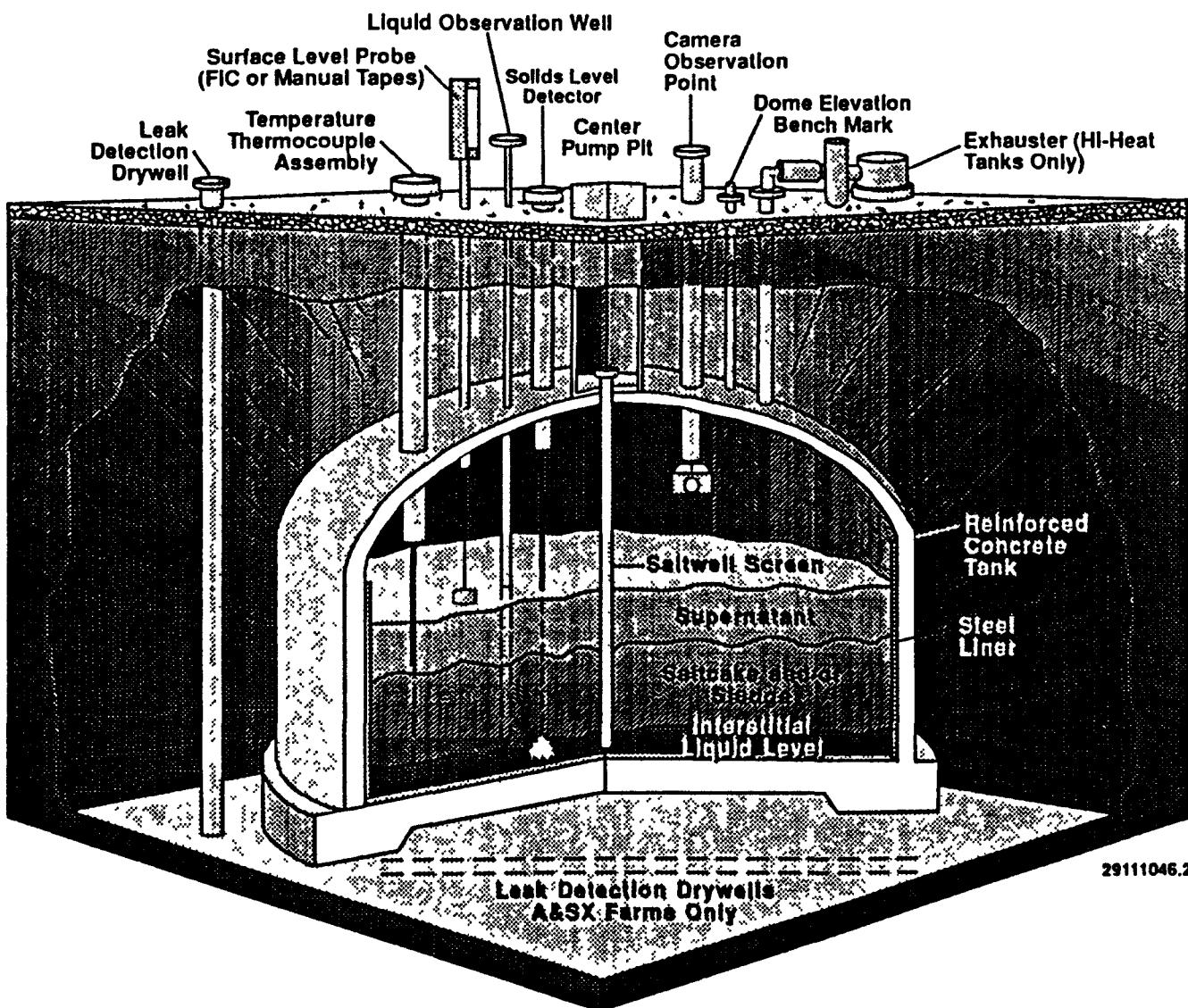
2.0 HISTORICAL INFORMATION AND EVALUATION

Radioactive wastes from defense operations have accumulated at the Hanford Site's underground waste tanks since the late 1940's. The original tank farms (B, C, T, U) were built between 1943-1944; tank 241-T-107 was placed into service in 1945. Groups of waste tanks that were physically located together and built at the same time are called tank farms at the Hanford Site. Tank T-107 has a diameter of 22.9 m (75 ft), an operating depth of 5.2 m (17 ft), and a nominal capacity of 2 million liters (530,000 gal). The basic design of a typical SST is shown in Figure 2-1. The tank was constructed of reinforced concrete with a mild steel liner covering its bottom and sides. The carbon steel liner was designed to receive and contain neutralized, mildly alkaline wastes. The top of the tank is a concrete dome. Tanks such as 241-T-107 were all covered by at least 1.8 m (6 ft) of soil for shielding purposes (Anderson 1990). The SST in the tank farms were connected in groups of three or four and overflowed from one to another (known as a cascade). Tank 241-T-107 is the first tank in a cascade that includes 241-T-108 and 241-T-109. Cascades served several functions in Hanford Site waste management operations. By cascading tanks, fewer connections needed to be made during waste disposal; consequently, all three tanks were usable without having to connect the active waste transfer line directly to each individual tank. This handling method reduced the likelihood for personnel exposure to the waste and diminished the chances for a loss of tank integrity due to overfilling. Another benefit of the cascades was clarification of the wastes. When used in this manner, most of the solids in the waste slurries routed to the tanks settled in the first tank (241-T-107), and the clarified liquids cascaded on to the other tanks in the series (241-T-108 and 241-T-109). Supernate from the final tank in a cascade series was sometimes routed to a disposal trench. Since most radionuclides are insoluble in aqueous alkaline media, clarification reduced the potential amount of radiological contamination to the environment. However, it was observed from historical sources that cascade lines routinely clogged (Anderson 1990). When this occurred, very little could be done to resolve the problem, other than re-routing the effluent stream directly to the disposal tank. Cascading was a common practice in the early process history of the tanks, but became less frequent as time passed, virtually ceasing by the late 1950's.

2.1 BACKGROUND--FERROCYANIDE PROCESS

During the 1950's, additional tank storage space was required to support plutonium production for the United States defense mission. To obtain additional tank storage volume within a short period of time and to minimize construction of additional storage tanks, Hanford Site scientists developed a process to scavenge cesium from tank waste liquids (Sloat 1954; Abrams 1956). Ferrocyanide compounds were used in a carrier-precipitation process to scavenge ^{137}Cs and other soluble radionuclides from the Hanford Site waste tanks. This treatment was used on U Plant waste effluent, bismuth phosphate first-cycle decontamination waste, and selected wastes that had been previously discharged to the tanks. The radionuclides settled in the waste tanks while the decontaminated supernate was discharged to the cribs and trenches. As a result of this process, occupied waste volume in the waste tanks was greatly reduced, while minimizing the amount of long-lived radionuclides discharged to the ground.

Figure 2-1. Typical Single-Shell Tank Diagram.



Note: Tank 241-T-107 has a dished bottom.

In implementing this process, approximately 140 metric tons of ferrocyanide [as $\text{Fe}(\text{CN})_6^{4-}$] were added to the tanks. Ferrocyanide is a stable complex of iron(II) and cyanide, whose compounds are considered nontoxic because they do not appreciably dissociate in aqueous solutions (Burger 1984). In the presence of oxidizing materials such as nitrates and/or nitrites, ferrocyanide compounds can undergo uncontrolled exothermic reactions in the laboratory by heating them to high temperatures (above 280 °C [540 °F]). The reactive nature of ferrocyanide in the presence of an oxidizer has been known for decades, but the conditions under which the compound can undergo rapid exothermic reactions have not been thoroughly studied. Because the scavenging process involved precipitating ferrocyanides from solutions containing nitrate and nitrite, the potential for a reactive mixture of ferrocyanides and nitrates/nitrites in the SSTs must be evaluated.

2.2 TANK 241-T-107 HISTORY

Tank T-107 went into service in 1945. The first type of waste it received was first-cycle decontamination (1C) waste. This type of waste originated from the bismuth phosphate process performed at the B and T Plants. This waste consists of by-product materials co-precipitated from a plutonium containing solution. It contained about 10% of original fission activity and 1% of the plutonium. In addition, 24% of the 1C waste stream that went to T-107 was coating waste from the removal of the aluminum fuel element coating (Svancara 1993). First cycle decontamination waste was continuously added until tank T-107 was full in September 1945 and began to overflow into tank T-108. The cascade was completely full by March 1946 (Anderson 1990).

The tank remained undisturbed until 1952 when tank T-107 was reserved to receive tributyl phosphate (TBP) waste because of a plug in 241-C-110-111-112 cascade. This type of waste comes from the tributyl phosphate uranium extraction process generated at U Plant. It is composed of both concentrated and neutralized aqueous effluents from the primary extraction column and from the solvent wash. Tributyl phosphate waste was added to the tank from the end of 1952 until the middle of 1953. In 1953, the supernatant was removed and sent to TX-118 to feed the 200 West evaporator, leaving tank T-107 slightly less than half-full with 886,000 L (234,000 gal) remaining in the tank. In the last quarter of 1953, T-107 received unconcentrated, ferrocyanide-scavenged, TBP waste from T-101. Tank T-101 was the discharge tank for the In-Plant test of the scavenging process. Tank T-107 continued to receive flushes of TBP waste from T-101 until early 1954. Between 1954 and 1966 no addition or removal of waste was recorded, and the total waste volume remained constant. Tank waste liquids were removed from T-107 in 1966 and moved to tank TX-118 leaving 787,000 L (208,000 gal) of waste in the heel to make room for the cladding waste.

In 1967, tank T-107 received cladding waste (CW) from C-102. Cladding waste was produced at the PUREX plant from the processing of cold uranium. Several tons of waste were generated from the dissolution of aluminum fuel cladding or zircaloy fuel cladding (after 1964). The zircaloy cladding was dissolved by boiling it in an ammonium fluoride/ammonium nitrate solution. Aluminum-clad fuels were declad by boiling them in a solution of sodium nitrate and sodium hydroxide. Approximately 1,040,000 L (275,000 gal) of CW waste, which included mostly liquids and a small amount of solids, was removed from T-107 and transferred to tank TY-103 in 1969 (Anderson 1990). Both zircaloy and aluminum cladding waste are expected in T-107.

In the beginning of 1973, tank T-107 received flush water from tank BX-104. This was ion exchange waste (IX) from the cesium recovery process at B Plant. The feed for this process was PUREX supernate, which included column waste, column wash waste and cesium purification waste. The waste, however, was immediately distributed to tanks T-108 and T-105 in the second quarter of 1973 (Svancara 1993). This type of waste is not expected to contribute substantially to the solids volume of the tank.

In 1976 tank T-107 was removed from service. Tank T-107 no longer received any wastes and was designated inactive. Drainable liquids were moved to T-101 using the salt well pumping method in 1976. Early in 1977, drainable liquids were removed leaving 496,000 L (131,000 gal) in solids. In the last quarter of 1977, the solid level was 568,000 L (150,000 gal) and the liquid level was 106,000 L (28,000 gal). In 1979, the integrity of the tank was questioned either because of an anomalous activity noted in the dry wells, or because of a noticeable drop in waste volume. In February of 1980, a new solids level of 632,000 L (167,000 gal) and a liquid level of 42,000 L (11,000 gal) was recorded. The Anderson (1990) report does note a significant drop in the liquids volume, this may be the cause of the integrity being in question.

Tank T-107 was categorized as a ferrocyanide tank when it received waste from T-101, a settling tank for the In-Plant ferrocyanide scavenging process. It was a possible tank T-107 had greater than 1000 g-mol of ferrocyanide. Analytes that differentiate ferrocyanide waste from other wastes are elevated levels of nickel, calcium, and ^{137}Cs . Over time, additional gravity settling may have compressed the waste layers, increasing the concentration of some of these analytes. However, the effect of radiation and high pH conditions on the waste matrix is largely unknown. Exposure of the waste to these conditions is believed to have degraded the ferrocyanide. However, laboratory results confirming that hypothesis are still pending (Lilga et al. 1992; Babad et al. 1993).

The ferrocyanide scavenging method was performed on U Plant waste effluent, bismuth phosphate first cycle decontamination waste and selected wastes previously discharged to the tanks. This was accomplished either through an In-Farm or In-Plant scavenging process. The In-Plant scavenging process was used in U Plant which contained an in house processing vessel (244-CR vault). Before being routed to the storage tanks, the effluent produced in the plant was sent to the 244-CR vault for ferrocyanide scavenging. The 244-CR vault facility contained stainless steel tanks with chemical addition, agitation, and sampling capabilities. Once the waste was in the vault the pH was adjusted with HNO_3 and/or NaOH to $\text{pH } 9.3 \pm 0.7$. Next, sodium ferrocyanide and nickel were added (generally to 0.005 M each) to precipitate ^{137}Cs in addition to other soluble radionuclides from the waste. Representative samples were routinely taken from the vault for analysis. If laboratory analysis of the feed tank contents indicated additional ^{90}Sr decontamination was necessary, calcium nitrate was also added (Sloat 1955). Scavenging of ^{60}Co with Na_2S was also commonly done. Once the ferrocyanide had been added the waste was routed to a settling tank, as precipitates settled out the supernatant was discharged. The results of the scavenging process produced good results and alleviated two concerns; the rapidly growing need for tank storage space as well as decreasing the amount of long-lived radioactive species discharged to the environment.

2.2.1 Unreviewed Safety Question Declaration

When the sampling of T-107 was performed, an unreviewed safety question (USQ) had been declared. Presently the ferrocyanide issue USQ has been closed. More information on the USQ can be found in DOE (1987); Peach (1990); and Public Law 101-510 (1990).

Using a computer model output (Jungfleisch 1984), process knowledge, and transfer records, 24 waste tanks were identified at the Hanford Site as potentially containing 1,000 g-mol (465 lb) or more of ferrocyanide as the $\text{Fe}(\text{CN})_6^{4-}$ ion (Borsheim and Simpson 1991). On further investigation, six tanks are believed to have received less than 1,000 g-mol of ferrocyanide sludge and are therefore candidates for removal from the Watch List (Cash 1993). Presently, four tanks have been removed from the watch list while two are pending approval to be removed. Tank 241-T-107 is on the Ferrocyanide Watch List because it was a known receiver of ferrocyanide-scavenged waste.

2.3 PROCESS KNOWLEDGE

Process knowledge obtained from historical records can be used to predict the major constituents and some general physical properties of the waste matrix in the waste tanks. Tank 241-T-107 is expected to contain relatively soft sludge, which can be push-mode sampled. This expectation was supported by inspection of in-tank photographs that indicated a moist and pliant waste surface (Figure 2-2). During its operating history, tank 241-T-107 was never subject to any of the various waste volume reduction or in-tank solidification processes; consequently, there was no formation of hard salt cake on top of the sludge, as there was in the BY or TX Tank Farms. The effluents that were added to the tank during waste management operations were slurries consisting primarily of water (Schneider 1951). There was no mixing equipment in tank 241-T-107 to blend the layers of settled solids together and there were distinct compositional differences in the wastes directed to 241-T-107 over its operating life. Because of the lack of agitation or mixing in the tank, evidence of layering is expected to be observed in some of the segment-level analytical results.

As mentioned in Section 2.2, there were four significant types of wastes stored in tank T-107: 1C, TBP, CW and IX (Anderson 1990). Each process has characteristic analytes that can be identified as being strongly associated with a particular waste stream. It is acknowledged that some of the chemicals used historically may have degraded over time. However, there are various key analytes, closely related to a specific waste stream, can be identified which cannot degrade any further. To develop a waste profile and inventory for the tank, characteristic species from each principal waste stream will be selected for review on a segment-level basis (where the data is available), and associated spatially with a location in the tank. The spatial location will then be compared to the filling history of the tank to determine if the data is consistent.

The first type of waste received was first cycle decontamination waste (1C) from the bismuth phosphate (BiPO_4) process. The BiPO_4 process was used to separate plutonium from uranium in irradiated fuel rods. It precipitates out plutonium from uranium solutions. The precipitate is then redissolved and recrystallized for greater purification of the plutonium. To enhance the efficiency of the BiPO_4 process, SiF_6^{2-} was added to solubilize other fission

Figure 2-2. In Tank Photograph of T-107.

241-T-107
7-13-84



products. The use of SiF_6^{2-} was limited due to the corrosive properties of fluoride. Aluminum cladding was removed by dissolving it in a mixture of sodium hydroxide and sodium nitrate. Twenty four percent of this 1C waste was comprised of aluminum cladding waste. Therefore, within this characterization report, when discussing 1C waste it is a mixture of two waste types. The indicator ions representative of these processes are bismuth, phosphate, aluminum, silicon, and fluoride.

The second type of waste received by tank T-107 was TBP waste. This waste was from the tributyl phosphate uranium extraction process at U Plant. This process was designed for the recovery of uranium metal from the waste generated by the BiPO_4 process. The major ions of this process would be phosphates, sulfates, and uranium.

The next type of waste added was produced at the PUREX Plant, specifically cladding waste. This waste was produced by either dissolving the zirconium (after 1964) or aluminum fuel cladding. The zirconium cladding was removed by dissolving it in a solution of sodium nitrate and sodium fluoride, while the aluminum fuel cladding was dissolved in a mixture of sodium hydroxide and sodium nitrate. High concentrations of aluminum and fluoride are expected, with some zirconium possible.

The final waste type stored in T-107 was IX waste. Ion exchange (IX) waste originates from the cesium recovery process at B Plant, and is expected to contain a very low percentage of solids. The major ions used and/or found in this process are nitrates and hydroxides.

Since tank T-107 is the first member of the cascade, a majority of the solids are going to be found here. The majority of the waste found in T-107 should be 1C waste and CW. Figure 2-3 illustrates the addition or removal of the waste in T-107 over time. Within the waste are various analytes such as sodium, nitrates, nitrites, sulfates, and hydroxides that are common to several waste streams, substantial changes in concentration are considered significant. Over the years, the types and amounts of chemical compounds have changed within the processes to improve recovery and waste management practices have further confounded the waste matrix. Therefore, the concentrations found currently may not parallel historical records well.

2.3.1 Principal Waste Stream Compositions

The estimated composition of neutralized 1C, TBP, and CW waste streams (i.e. unsettled) are given in Table 2-1 as determined from Schneider (1951). The records kept previously are neither complete nor comprehensive enough to provide authoritative characterization, but provides a useful starting point for interpretation. Schneider (1951) process stream compositions are for the unsettled wastes being transferred from the separations plants. The estimates using Agnew (1994) provide insight as to the analyte concentrations of the settled sludge. Table 2-1 does not account for IX waste since it will not donate a significant amount of solids. How the waste has settled in T-107 will be discussed further in Section 6.

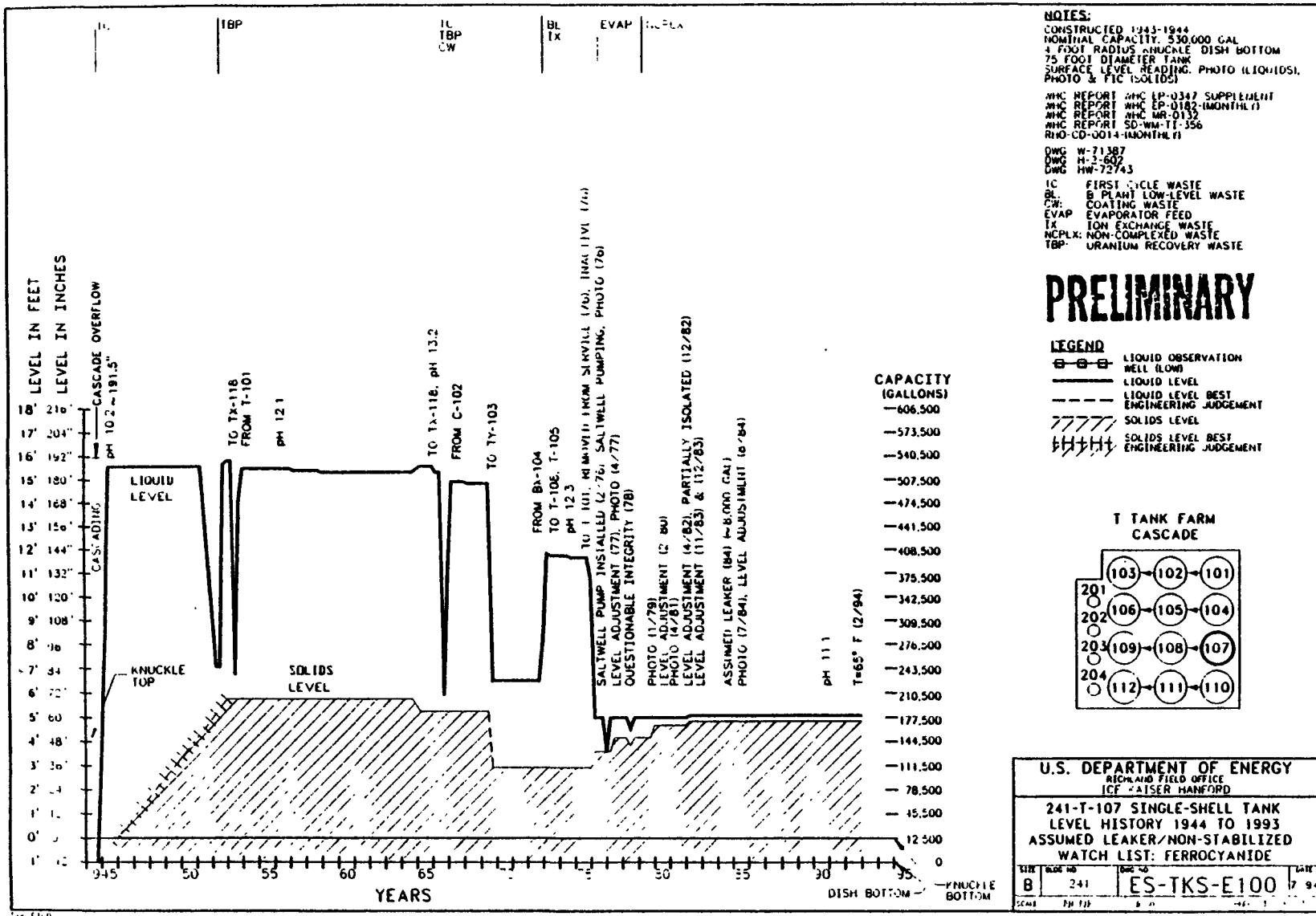


Figure 2-3. Fill and Transfer History of 241-T-107

Table 2-1. Expected Constituents for T-107 Waste Streams.

Analyte	Unsettled 1C BiPO ₄ Waste (Schneider 1951)		Settled 1C BiPO ₄ Waste (Agnew 1994)		Settled TBP Waste (Agnew 1994)		Unsettled CW (Schneider 1951)		Settled CW (Agnew 1994)	
Cation	Wt %	(μ g/g)	Wt %	(μ g/g)	Wt %	(μ g/g)	Wt %	(μ g/g)	Wt %	(μ g/g)
Bi	0.24	2,400	1.39	13,900	0	0	--	--	0	0
Cr	0.02	200	0.0048	47.5	0	0	--	--	0	0
Na	4.3	43,000	8.49	84,900	8.54	84,500	6.6	66,000	21.1	215,000
NH ₄ ⁺	0.18	1,800	--	--	--	--	--	--	--	--
Fe	0.13	1,300	0.83	8,300	8.53	85,300	--	--	0	0
Mn	--	--	0	0	0	0	--	--	0	0
K	--	--	0	0	0	0	--	--	0.34	3400
La	--	--	0	0	0	0	--	--	0	0
Al	--	--	2.69	26,900			--	--	9.5	95,100
Zr(OH) ₂	0.0046	46.1	0.067	670			--	--	13.5	135,000
Anions										
PO ₄ ³⁻	2.4	24,000	9.53	95,300	0.45	4,500	2.0	20,000	0	0
SO ₄ ²⁻	0.44	4,400	0.26	2,600	11.2	112,400	1.9	19,000	0	0
NO ₃ ⁻	8.5	85,000	1.42	14,200	6.5	65,000	0.8	8,000	0	0
F	--	--	0.55	5,500	0	0	--	--	16.7	167,200
SiF ₆ ²⁻	0.40	4,000	--	--	--	--	--	--	--	--
C ₂ O ₄ ²⁻	--	--	--	--	--	--	--	--	--	--
H ₂ O	83.4		65.49	654,900	30.45	304,500	78.2	782,000	55.8	558,000

Analytes in italics are mostly soluble

NH₄⁺ has likely dissipated over time, and is no longer believed to be present

2.4 ANALYTICAL RESULTS FROM SIMULANT STUDIES

In order to simulate the conditions and subsequent activity in tank T-107, as well as other ferrocyanide watch list tanks, a laboratory simulant was created. The simulant was considered to be energetically conservative, but reasonably similar physically and chemically to the waste deposited in the tank. The sludge was centrifuged for 14 hours and for 7 days to simulate the settling, stratification, and segregation caused by gravity over a 3.6 and 30 year period, respectively.

Estimates of tank waste reactivity were performed both theoretically and experimentally. The theoretically derived heats of reaction assume dry, solid reagents at standard temperature and pressure. The heats of reaction were calculated theoretically using bond energies of the reactants with in the reaction. The heat of reaction (ΔH) ranged from -9.6 kJ/g to +19.7 kJ/g. The experimental data derived from the physically and chemically similar sludge indicated the waste contained an average of 51 to 66% water (by weight). The DSC thermograms produced a calculated ΔH per gram between -3.95 kJ/g to -4.71 kJ/g which was considerably less than the theoretical values. The reason proposed for this

observed discrepancy is that the ferrocyanide is believed to have degraded over time (Babad). Furthermore, these sludges contain substantial amount of free water. As water changes from a liquid to a gas a good heat sink is developed before any self-sustained chemical reaction can occur. This statement suggests the tank waste is stable and does not pose a hazard. The results from the simulant studies indicate that the sludge must be dried and the temperature must exceed 180°C before reactions can occur, making the initiation and propagation of a rapid exothermic reaction is unlikely. A more detailed description of the preparation, calculations and equations used to render the results can be found in *Tank Characterization Data Report: Tank 241-C-112* (Simpson et al. 1993).

2.5 SURVEILLANCE DATA

The last recorded observation found in *A History of the 200 Area Tank Farms* (Anderson 1990), which dates 1980, notes the total volume of tank T-107 has remained approximately 634,000 L (178,000 gal) with 42,000 L (11,000 gal) being liquids and 632,000 L (167,000 gal) consisting of solids. In 1984 tank T-107 was declared an assumed leaker. The most recent tank volume observations found in Hanlon (1994), note the total waste volume in tank T-107 as 680,000 L (180,000 gal) with 38,000 L (10,000 gal) liquids and 643,000 L (170,000 gal) solids.

In 1984, tank T-107 was declared an assumed leaker and has yet to be interim stabilized. The total amount of waste was 73 inches with no substantial change noted in Hanlon (1994) or Anderson (1990). The highest temperature recorded in the waste was 67°F (19°C) taken on 3/7/94. It should be noted the tank temperature varies by several degrees over an annual cycle.

3.0 TANK SAMPLING OVERVIEW

3.1 DESCRIPTION OF SAMPLING EVENT

Tank 241-T-107 was push-mode core sampled through three risers during a period from November 5, 1992 to March 15, 1993. Approximately four segments were expected from each core sample (three full segments and one partial segment). Initially, two core samples were scheduled for T-107, but because of poor sample recovery, a third core was taken (Silvers 1993). Core 50 was obtained from riser #2; Core 51 from riser #5; and Core 52 from riser #3. The first core (Core 50) was sampled on November 10, 1993; the second core sample was completed on February 18, 1993 (Core 51) and the third core (Core 52) on March 10, 1993.

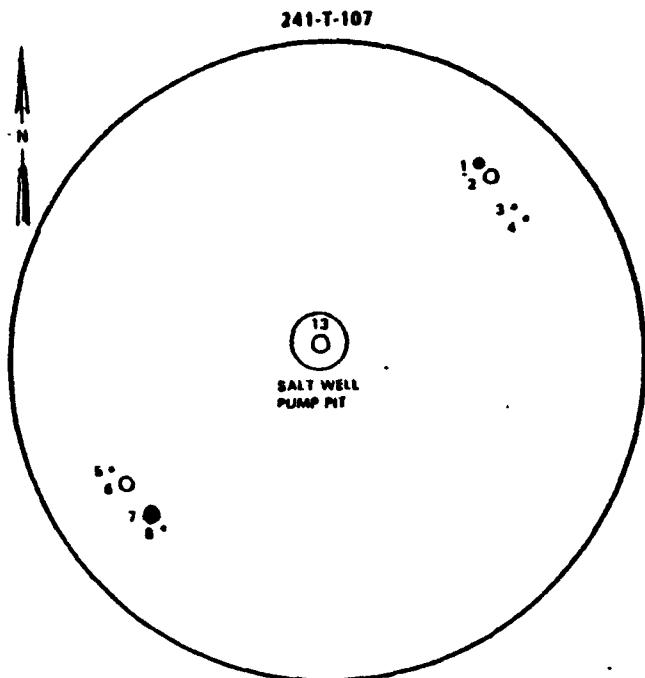
Access to the interior of the tank is provided by various tank risers. The riser configuration for tank 241-T-107 is given in Figure 3-1. A field review of the tank farm operating risers determines which risers can be used in the sampling operation. A riser is opened and the truck is positioned over the riser. The sampler is lowered into the tank through the drill string and pushed into the waste. Core samples from tank T-107 were obtained by using a core sampling truck with sampling equipment mounted on a rotating platform. The sampler advances down in 19-inch increments until the bottom of the tank is reached. The sampler is constructed of a stainless steel column 48-cm (19-in) long with a 2.2-cm (7/8-in) inside diameter, and a volume of 187 mL (0.05 gal). The sampler is placed on the bottom of the drill string and the drill string is lowered into the waste. When a 19 inch segment is removed from the tank it is sealed within a stainless steel liner, and the liner is placed in a shipping cask. The shipping casks are approximately 122-cm (48-in) tall, 13-cm (5-in) in diameter with 2.5-cm (.98-in) lead shielding. This degree of containment protects the workers from exposure to radiation and prevents sample (liquid or solid) from being lost. It is important to note water, not normal paraffin hydrocarbons, was used as the hydrostatic head fluid for the tank T-107 sampling event potentially biasing the sample results. Water is a key component of the tank waste and if the hydrostatic head fluid had leaked into the sample, a water content much higher than actual could be observed. The 222-S Laboratory did not note any contamination of the sample by the hydrostatic head fluid, however this type of contamination could be difficult to detect solely on a visual basis. Refer to *Tank Characterization Reference Guide* (ref.) for more specific information on T-107 sampling event.

The samples were then transported to 222-S Laboratory between November 10, 1992 and March, 15, 1993. This facility is located in the 200 West Area of the Hanford Site and is operated by Westinghouse Hanford Company . Further physical and radiochemical characterization was performed at the 325 Analytical Chemistry Laboratory (ACL). The 325 ACL is operated by Battelle Pacific Northwest Laboratory and is located in the 300 Area of the Hanford Site.

3.2 CHAIN-OF-CUSTODY

A chain-of-custody record was kept during the sampling event for each segment sampled. The chain-of-custody form is a one-page record that is used to ensure (1) the sample is safely and properly transported from the field to the laboratory, and (2) the correct personnel are involved in the sampling operation and transportation of the sample to the laboratory.

Figure 3-1. T-107 Riser Configuration.



C, INSIDE TANK BOTTOM = 635.00

REF DWGS DOME PLAN/AS BLT	H-W-72743
RISER ELEV	H-2-1741, H-2-37909
ISOLATION	H-2-73043
VERIFIED	9/9/86

One of the additional functions of the chain-of-custody records is to provide radiation survey data. This is a record of the radiation dose that is emitted from the shipping cask. The dose rates in mrem/hour are measured from the top, sides, and bottom of the cask. These values are recorded on the chain-of-custody and represent the radiation emission from the sample. The last item recorded under the radiation survey data is the smearable contamination. Smearable contamination represents the radiation from waste material that is not sealed within the shipping cask; values greater than 100 mrem/hour are considered unsafe. Measurements are made both in the field and in the laboratory. In all three cores the smearable contamination was lower than the detection limits.

The chain-of-custody has several important functions: (1) to provide a modest description of the cask, sampler, and the expected contents of the sampler as well as shipment, sample, and cask serial numbers for the specific sampling event; (2) to provide summary information about the requested analytes; and (3) to provide traceability for the integrity on arrival at the laboratory. This information is provided to ensure that each sample can be uniquely identified. A summary of the most pertinent data contained in the chain-of-custody forms for 241-T-107 samples is presented in Tables 3-1, 3-2, and 3-3. Copies of the chain-of-custody forms are available in the full data packages (Svancara 1993). From inspection of the chain of custody records sampling operations had no difficulty retrieving the sample from the tank and/or transporting the sampler to the performing laboratory. The laboratory, does not note any leaking of the sampler into the liner or cask. One difficulty which was encountered during the sampling process of tank T-107 was several segments from each of the cores had insufficient sample for full suite of analyses. Core 50 Segment 1 sat in the riser for more than 48 hours. Each sampling event has a guidelines for how much time the sample has from sampling to delivery to the lab; because this requirement was violated, another sample from Core 50 Segment 1 was retrieved. During extrusion the sampler for Core 50 Segment 2 was under pressure. When it was opened, a small amount of the sample was ejected from the sampler, however, there was a sufficient amount of sample left (194.6 g) to perform all the analyses.

Table 3-1. 241-T-107 Core 50--Chain-of-Custody Summary.

Sample	Core 50				
Place Taken	Riser 2				
Sender	M.C. Jones				
Receiver	R. Akita				
Place Received	222-S Lab				
	Segment 1	Segment 1R	Segment 2	Segment 3	Segment 4
Date Taken	11/5/92	11/9/92	11/10/92	11/10/92	11/10/92
Date Released	11/10/92	11/10/92	11/10/92	11/10/92	11/10/92
Time Released	18:40	18:40	18:40	20:57	20:57
Time Received	18:55	18:55	18:55	21:15	21:15
Smearable Contamination	< DL alpha < DL beta/gamma				
Dose Rate Thru Drill String	25 mr/hr	24 mr/hr	70 mr/hr	5 mr/hr	10 mr/hr

Table 3-2. 241-T-107 Core 51--Chain-of-Custody Summary.

Sample	Core 51			
Place Taken	Riser 5			
Sender	D. Hartley			
Receiver	S. Cobb/ D.B Hardy			
Place Received	222-S Lab			
	Segment 1	Segment 2	Segment 3	Segment 4
Date Taken	2/12/93	2/12/93	2/17/93	2/18/93
Date Released	2/16/93	2/18/93	2/18/93	2/18/93
Time Released	13:00	19:10	19:10	20:30
Time Received	13:15	19:35	19:35	20:55
Smearable Contamination	< DL alpha < DL beta-gamma			
Dose Rate Through Drill String	10 mr/hr	350 mr/hr	80 mr/hr	80 mr/hr

Table 3-3. 241-T-107 Core 52--Chain-of-Custody Summary.

Sample	Core 52			
Place Taken	Riser 3			
Sender	D. Hartley			
Receiver	S. Cobb/ V. Massie			
Place Received	222-S Lab			
	Segment 1	Segment 2	Segment 3	Segment 4
Date Taken	3/10/93	3/10/93	3/10/93	3/10/93
Date Released	3/15/93	3/15/93	3/15/93	3/15/93
Time Released	10:15	10:15	12:45	10:15
Time Received	10:35	10:35	13:00	10:35
Smearable Contamination	< DL alpha < DL beta-gamma			
Dose Rate Through Drill String	20 mr/hr	25 mr/hr	40 mr/hr	20 mr/hr

4.0 SAMPLE HANDLING AND ANALYTICAL SCHEME

The primary objective for these waste analyses was to evaluate the tank waste with regard to the safety issue whether this tank could be categorized as *Safe*, *Conditionally Safe*, or *Unsafe*. The data would aid in evaluating whether constituent concentrations are within safe operating limits by evaluating key components. Other concerns and interests for this data include satisfying the *Hanford Federal Facility Agreement and Consent Order (Tri-Party Agreement)* milestone M-10-06 (Ecology 1992) as well as Milestone M-44-07 and M-44-05 (Ecology 1994). The results of this report aids in quantifying constituent concentrations in order to focus on a waste category defined by Postma (1993).

4.1 SAMPLE BREAKDOWN PROCEDURE

The shipping casks from tank T-107 were transported to 222-S Laboratory for analysis. Each segment remained in the cask until it was extruded from the sampler in the hot cell. The sampler is placed in a horizontal position on the sample extruder and the sample is removed using a piston positioned at the top of the sampler. The piston pushes the sample, bottom end first, onto a metal tray where the solids and any liquids were collected. From T-107, a total of three core samples were taken, averaging approximately three and a quarter segments per core. Assuming there was enough sample, each of the segments was divided into subsegments: upper (U) and lower (L). The drainable liquid from each core was collected and consolidated into a core drainable liquid composite sample. Next, the mass of the segment and the approximate length are recorded. From this information the gross bulk densities can be estimated. The sample volume is determined by measuring the length of the extruded sample and multiplying by 9.85 mL/in (the sampler has a volume of 187 mL for a sample length of 19 inches). The work performed on the samples was done remotely behind 60 cm (2 ft) of lead glass.

The segments for tank T-107 were broken down according to the *Tank Waste Characterization Project Plan* (Winters 1992). At the time of the breakdown procedures, In-Plant ferrocyanide tanks were only required to be divided into half segments. Presently, the *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objectives Process* (Meacham 1994) lists all ferrocyanide tanks be divided into quarter segments for specific analytes. Although the breakdown procedures have changed over time, evaluation of the data obtained and its interpretation is still be of great benefit from a safety standpoint.

4.2 TANK 241-T-107 CORE SAMPLE DESCRIPTIONS

After extrusion from the sampler, the samples were photographed with the jar number and a color comparator chart to have consistency regarding the description of each segment. Visual characteristics of the extruded samples, as well as mass and length, were recorded in a log book. Special attention was placed on the sample volume, liquid/solid ratio, color, consistency, texture, and homogeneity of each segment. These notes would aid in providing qualitative descriptions for the cores. The written descriptions, as seen by operating hot cell technicians, aid in capturing the physical characteristics a photograph cannot depict, such as consistency and texture. Table 4-1 summarizes the log book notes.

Table 4-1. Sampler Recovery for Tank T-107.

Core	Seg	% Sampler Recovery	Sample Obtained		Comments
			% Solid	% Liquid	
50	1	36	72	28	22.87 g of very light to medium brown solids. Dark stripe down one side of the extruded solids. 8.75 g of opaque brown drainable liquid. Not used per AE&R direction.
50	1R	34	70	30	25.58 g of light brown solids homogeneous mixture. 10.89 g of opaque brown drainable liquid.
50	2	94	100	0	194.45 g of solids. Sampler was under pressure. Solids were inhomogeneous and ranged from a light brown section, similar to Segment 1 except darker in color, to medium brown solids, to a dark brown section. No drainable liquids.
50	3	96	5	95	Sample was recovered by holding the sampler vertical and tapping with a hammer. 8.52 g of dark brown solids were recovered. The solids were thick and homogeneous. 165 g of opaque brown drainable liquid; density of 0.97 g/mL.
50	4	67	1	99	1.17 g piece of solids was recovered. 120.42 g of opaque brown drainable liquid; density 0.97 g/mL.
51	1	0	0	0	Sampler was completely empty.
51	2	64	40	60	64.48 g of dark brown solids. 87.30 g of opaque drainable liquid; density 1.26 g/mL.
51	3	100	100	0	215.66 g of dark brown solids. Solids appeared to be homogeneous. No drainable liquids.
51	4	100	100	0	206.15 g of dark brown solids. Top 1 in. and bottom 6 in. appeared to have more fluids. No drainable liquids.
52	1	43	100	0	28.46 g of medium to dark grey solids. One side appeared to be dark grey rest was light grey. No drainable liquids.
52	2	56	100	0	111.23 g of brown solids. Solids appeared wet.
52	3	95	100	0	201.41 g of solids. Color ranged from light brown at bottom to dark brown at top. Solids were lumpy. No drainable liquids.
52	4	60	3	97	4.25 g of light brown solids. 117.34 g of brown turbid drainable liquid; density of 1.12 g/mL.

General characteristics of tank 241-T-107 waste materials are as follows

- Drainable liquids were brown in color and contained a large amount of suspended solids.
- Core samples ranged from light to dark brown, with some medium to dark grey solids in the upper segments.
- The consistency of the solids ranged from a homogeneous slurry to a lumpy sludge. In all cases the waste held its shape fairly well.
- Poor recovery from the first riser prompted the sampling of a third riser.

The next step in the sample preparation process is the distribution of aliquots for the various analytical procedures. The unhomogenized (direct) samples were obtained by pushing a small open metal tube into the segment. These were used for particle size analysis and volatile organic analysis. Subsequent homogenization of the segments was performed. This was done in an apparatus called a stomacher, which is simply machine with paddles. A bag containing the sample is placed in the stomacher and the samples are homogenized by a process similar to kneading bread. A majority of the analyses were performed on the homogenized samples. By mixing equal portions of each homogenized segment together it is believed a representative composite for each core is obtained. When homogenization is completed and aliquots are removed for analysis, the remaining sample is archived and stored at the 222-S Laboratory.

Segments, composite samples, and subsamples were often divided into different aliquots to satisfy sample analysis requirements. The aliquots of a sample were usually not divided into equal amounts, and often a remainder of the sample was left in the original sample jar. Since distribution of the aliquots was routinely done after sample homogenization, the aliquots are assumed to be identical. At times it was necessary to reallocate samples (i.e. use sample from an aliquot slated for an assay different than the one it was intended) usually to satisfy analytical priorities.

4.3 HOLD TIME CONSIDERATIONS

All analytes have a predetermined maximum allowable holding time, set by the Environmental Protection Agency (EPA SW-846 [EPA 1986]), during which the analysis should be completed. Completion of analysis during the maximum allowable holding time enhances the regulatory defensibility of the data. The length of the holding time varies for each analyte. For example, analyses performed on volatile and semivolatile organic compounds, many of which decompose or dissipate quickly, have shorter holding times. On the other hand, persistent analytes such as metals (except mercury), do not readily decompose or dissipate, and therefore have much longer acceptable holding times. Nearly all of the analyses of Cores 50, 51, and 52 exceeded their respective maximum holding times. The only analyses which holding time criteria were met were radiochemistry and metal analyses. Both of these analyses were completed within six months after sampling, which is the maximum hold time for these analyses. With the possible exception of VOA's, exceeding the holding times should not adversely effect the results. Holding times in SW-846 were established with a completely different waste matrix in mind. Because the way the samples are handled and prepped, the

laboratories cannot meet all the holding times. Although exceeding the maximum allowable holding times weakens the defensibility of the analytical results for some uses, it is anticipated that the overall effect on the analytical results for T-107 relative to waste management and disposal information is minimal. Further discussion of holding times can be found in Winters (1990a).

4.4 SAMPLE PREPARATION

Sample preparation procedures are conducted in order to optimize the recovery of each analyte of interest from the tank waste. Water digestion, acid digestion, and potassium hydroxide fusion are commonly used to extract metals and several radioisotopes from solid samples, and in some cases digestions are performed on liquid samples to improve analytical matrices. Many separations are specific to a particular analysis and are described within the corresponding analytical methods referenced in Tables 4-2, 4-3, and 4-4. In order to verify analyte recoveries resulting from separation techniques, laboratory control samples, carriers, spikes, tracers, and surrogates are analyzed concurrently with the environmental samples.

In some cases no sample preparation is necessary or desired. Direct analyses are assays performed on the sample matrix with little or no sample preparation. Several direct analyses were performed relating to the physical or energetic properties of the waste: density, thermogravimetric analysis, differential scanning calorimeter, and gravimetric weight percent water.

Water digestion (leach) analyses are assays performed after the sample matrix has been digested in distilled/deionized water; the water is then analyzed for soluble analytes. The soluble anions are determined by ion chromatography (IC). The primary anions analyzed in this manner are fluoride, chloride, nitrate, nitrite, phosphate, and sulfate. In addition, total organic and inorganic carbon, free cyanide, pH, and ammonia were also analyzed from water digestion samples by various analytical methods. Note that ion chromatography assays use a 1:100 sample:water dilution, where pH measurements use a 1:1 sample:water ratio. Selected radionuclides were measured on some of the water digestion samples to determine the type and number of soluble radionuclides. Inductively coupled plasma (ICP) and atomic absorption (AA) were also performed on some of the water digestion samples. These assays were performed to determine the amount of soluble metal cations (ICP), arsenic, or mercury (AA). Nitrite and Chromium (VI) from water digestions were analyzed by spectrophotometry. A total alpha and total beta count were performed on the water digestion samples as well. Gamma Energy Analysis (GEA) was also performed on water digestion samples to detect water soluble radionuclides. In many cases, these analytes were below the detection limits for the water digestion samples, suggesting that many of the analytes are not water soluble.

For acid digestion preparation, the sample is dissolved in a mixture of hydrochloric and nitric acids. This preparation brings most of the insoluble metals into solution with a minimum amount of dilution, and is usually best for the detection of trace elements and some major metals. Some elements occur in the tank in relatively large quantities and are referred to as major metals. These properties are the reason that acid digestion is generally used as the sample preparation for the homogenization tests in SW-846-based environmental sampling. The analyses performed on this preparation were ICP, GEA, and AA analyses (the AA analysis used nitric acid only). Analyzing an acid digestion solution using ICP analysis detects elemental compositions within the waste, especially trace and major metals.

Experience with Hanford tank waste matrices has shown that acid digestion does not always provide complete solubilization and that a more rigorous dissolution preparation (i.e. fusion) necessary to get adequate quantitation. Analyses that were performed on fusion-prepared samples were ICP and GEA for radionuclides. Fusion dissolution analyses are assays performed on the sample matrix after it has been fused with potassium hydroxide in a nickel crucible and then dissolved in acid. This preparation dissolves the entire sample, whereas other sample preparation procedures may not completely dissolve the sample matrix. However, one significant disadvantage of fusion preparation is that large amounts of potassium hydroxide are required to bring a sample into solution, which means a large dilution is involved. Because of this high dilution factor, trace elements are less likely to be correctly quantified, if they are detected at all. Another limitation of the preparation method is if the sample contains substantial quantities of potassium or nickel, these analytes will not be quantifiable, since the procedure uses potassium hydroxide and a nickel crucible (this limitation can be overcome using alternate preparation methods, if potassium or nickel are analytes critical to interpretation of the data). Elements that occur in abundance (major metals) or are highly insoluble are likely to be detected better by the fusion results than by any other sample preparation.

Generally, fusion dissolution is the preferred method of analyzing radionuclide content, with the exception of ^{14}C , ^{129}I , and ^{3}H (tritium). However, the sample preparation specified in the test instructions for ^{14}C (water digestion) is likely not the best for the high-level waste matrices. Difficulty with dissolving the sample with a water leach, and volatility associated with a fusion preparation, will potentially bias the ^{14}C results low for both sample preparation types, if they are associated with the water insoluble solid materials. Similar difficulties are encountered for the other radionuclides. However, none of these analytes are expected to be significant contributors to the radionuclide content of the waste.

The major analytes that were detected well with fusion ICP analysis for tank T-107 were aluminum, bismuth, iron, and sodium, phosphorous, silicon, and sulfur. In the case of these elements, the fusion result is the preferred method of analysis, since it is believed to provide more complete dissolution of the waste and a more complete quantitation of the analytes. Comparisons of these results with ion chromatography results can provide insight to the solubility characteristics of the waste. Some of the primary radionuclides that are measured using fusion preparation are ^{237}Np , $^{239/240}\text{Pu}$, ^{90}Sr , ^{137}Cs , and ^{99}Tc . A total alpha and total beta count were performed on the fusion dissolution samples as well.

Previously, chemical and radiological analyses were largely done on core composites, and in these characterization efforts, the core composites were built using quantities of segments based on a proportion of the total weight of sample for the core (Winters et al. 1990a; Winters et al. 1990b). This method assumed that the sample obtained is representative of what is in the tank. However, when partially filled segments are obtained, this procedure assumes that the tank does not contain any waste in this area. Incomplete recovery for a segment is more likely the result of sampling problems rather than voids in the waste.

The approach used to generate the composites for tank T-107 was to composite equal quantities of the homogenized subsegment material and assume that whatever is obtained in a partial subsegment is representative of a whole subsegment. Some inaccuracies may be introduced from density differences between subsegments. In general, those deviations are minimal compared to the other errors inherent in core sampling and analysis. If full segments are obtained for the entire core, and the homogenization procedure is satisfactory, there will be little difference between the two approaches.

4.5 ANALYTICAL METHODS

This section briefly describes the analyses used to characterize the waste in tank T-107; the analyses were split between Westinghouse 222-S Laboratory and Battelle's Pacific Northwest Laboratory (PNL). Several of the analytical tests performed on the composites were also done on the segments, but on a much more limited scope. There were some free liquids observed in all three Cores (50, 51, and 52), a liquid core composite was prepared for each of the three cores and analyzed. A summary of the drainable liquid core composite results is found in Section 5.

4.5.1 Physical and Rheological Tests

Physical tests completed at 222-S Laboratory included: particle size analysis, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), specific gravity, and percent water analyses. Duplicates were performed for the percent water analyses. The physical properties measured at PNL included wt% solids, settling behavior, and wt% dissolved solids. Rheological testing on these samples were performed at PNL and included shear strength and shear stress as a function of shear rate. Rheological properties were measured in duplicate. Table 4-2 lists the analytical methods used by 222-S and 325 Laboratory for physical and rheological testing. One segment from Core 50 (Segment 2) was selected to perform the full suite of rheological and physical measurements, in addition to the particle size assay done on each segment. Viscosity, settling properties, fluid behavior, and shear strength were some of the primary characteristics investigated. The samples tested for these properties were not homogenized prior to analysis.

Table 4-2. Analytical Methods for

Analyte	Procedure
Particle Size	T044-A-01712F
TGA	LA-560-112
DSC	LA-514-113
Specific Gravity	LA-510-112
Percent Water	LA-564-101
Rheology	PNL-ALO-501
Physical Properties	N/A

Scanning thermogravimetric analysis (TGA) and differential scanning calorimeter (DSC) are useful in determining the thermal stability or reactivity of a material. TGA measures the mass of a sample while the temperature of the sample is increased at a constant rate. In DSC analysis, the heat absorbed/evolved over and above the usual heat capacity of the substance is measured while the substance is exposed to a constant increase in temperature. The gravimetric weight percent water was determined by drying the sample for 12 to 24 hours in an oven at 103 to 105°C and measuring the difference in the weight of the sample.

4.5.2 Chemical and Radionuclide Constituent Analysis

Most of the chemical and radionuclide analyses were performed at the 222-S Laboratory. The uranium and plutonium isotopic analyses, and total inorganic carbon/total organic carbon/total carbon on direct samples, however, were performed at PNL. Duplicate analyses were performed on every tank sample. Table 4-4 lists the analytical methods used (Winters et al. 1990a; Winters et al. 1990b).

4.5.3 Sample Homogenization

Once the segments have been through the stomacher, homogenization tests are performed. Two portions of the solids were placed into separate sample vials each having a unique serial number. Aliquots from three of the homogenized segments (Core 50-Segment 2, Core 51-Segment 3L, Core 52-Segment 3L) were taken to determine the effectiveness of the process. The sample and its duplicate were prepared through the acid digestion procedure and analyzed via ICP. If the ions analyzed are within an acceptable relative percent difference (RPD) (10%) the samples are considered to be completely homogenized. Because of high RPD's for Cores 50 and 51, the homogenization test samples were digested a second time and analyzed via ICP in the same manner as the first set of samples. The solids used for the second digestion were taken from the same sample vial as the material for the first digest was taken from. The second set of homogenization tests were within acceptance criterion. Core 52 did have sufficiently low RPD, indicating homogenization.

4.5.4 Organic Constituent Analyses

All organic analyses of the samples from tank T-107 were performed at the Pacific Northwest Laboratory (PNL). An organics speciation analysis was performed on the core composites using a U.S. Environmental Protection Agency Contract Laboratory Procedure. No significant levels of organic compounds were found in any of the samples, and they were not expected to contribute to the sample matrix. The organic analyses performed were Volatile Organic Analysis (VOA), Semi-Volatile Organic Analyses (SVOA), Total Organic Halides (TOX), and Extractable Organic Halides (EOX). Duplicates were performed for all of the analyses. The holding times for SVOA's were exceeded; samples were collected 5-19-93, received by the laboratory on 7-13-93, extracted 7-16-93, and analyzed for SVOA's on 8-13-93.

Total organic carbon (TOC) for the solids was determined using the hot persulfate method, which dissolved the sample in a sulfuric acid solution (90°C+) to liberate inorganic carbon (carbonate). The persulfate ($K_2S_2O_8$) is then added, the organic carbon is converted to CO_2 , which is measured coulometrically.

4.5.5 Subsegment-Level Analysis

Core samples obtained from tank T-107 were not only divided into segments but subdivided into half-segments (when the amount of sample permitted). The justification of subsegment analysis is to provide (1) information to the waste energetics, (2) distribution of ^{137}Cs and ^{90}Sr , and (3) distribution of CN^- in the tank waste. Table 4-5 describes the type of analysis performed and the type of matrix.

Table 4-3. Analytical Methods For Organic Analyses.

Analysis	Method	Procedure Number
VOA	Gas Chromatography/Mass Spectrometry	PNL-ALO-335
SVOA	Gas Chromatography/Mass Spectrometry	PNL-ALO-345
EOX	Microcoulometric Titration	PNL-ALO-320.2
TOX	Microcoulometric Titration	PNL-ALO-321

Table 4-4. Analytical Methods for Chemical and Radionuclide Analyses.

Analyte	Method	Procedure Number
Hg	Cold Vapor Atomic Absorption	LA-325-102
F ⁻ , Cl ⁻ , NO ₃ ⁻ , NO ₂ ⁻ , PO ₄ ³⁻ , SO ₄ ²⁻	Ion Chromatography	LA-533-105
CN ⁻	Distillation/Spectrometric Analysis	LA-695-101 LA-695-102
U	Laser Fluorimetry	LA-925-106
Total Alpha Total Beta	Proportional Counting	LA-508-101
²³⁸ Pu, ^{238,240} Pu, ²⁴¹ Am	Alpha Spectrometry	LA-503-156
²³⁷ Np	Alpha Proportional Counting	LA-933-141
Total Metals	Inductively Coupled Plasma	LA-505-151
⁹⁰ Sr	Beta Proportional Counting	LA-220-101
⁹⁹ Tc ⁷⁹ Se	Liquid Scintillation	LA-348-104 LA-218-114
¹²⁹ I	Low Energy Gamma Analysis	LA-548-121
¹⁴ C ³ H	Liquid Scintillation	LA-348-104 LA-218-114
¹⁵⁴ Eu, ¹⁵⁵ Eu, ²⁴¹ Am, ¹³⁷ Cs, ⁶⁰ Co	Gamma Energy Analysis	LA-548-121
NO ₂ ⁻	Spectrophotometry	LA-645-001
H ⁺	pH	LA-212-103
As	Hydride Atomic Absorption	LA-355-131 LA-365-131
Pu Isotopic	Fusion Mass Spectrometry	PNL-ALO-423 PNL-MA-597
U Isotopic	Mass Spectrometry	PNL-MA-597 PNL-ALO-445
TOC	Total Organic Carbon	LA-344-105 PNL-ALO-381
CO ₃ /C	Total Inorganic Carbon	LA-622-102 PNL-ALO-381

Table 4-5. Subsegment Analysis.

Direct	Fusion Dissolution	Water Leach*	Acid Digestion
TOC/TIC	ICP (metals)	pH	ICP
TGA	GEA (¹³⁷ Cs)		AA
DSC	⁹⁰ Sr		
Total CN ⁻			
Wt% H ₂ O			
Bulk Density			

DSC = Differential Scanning Calorimeter

GEA = Gamma Energy Analysis

ICP = Inductively Coupled Plasma/Atomic Emission Spectroscopy

TGA = Thermogravimetric Analysis

TIC = Total Inorganic Carbon

TOC = Total Organic Carbon

* IC (Anions), cyanide (CN⁻), and GEA were performed on the water leaches of the core composites, not on the segments or subsegments

5.0 ANALYTICAL RESULTS: TANK 241-T-107

5.1 CHEMICAL ANALYSES

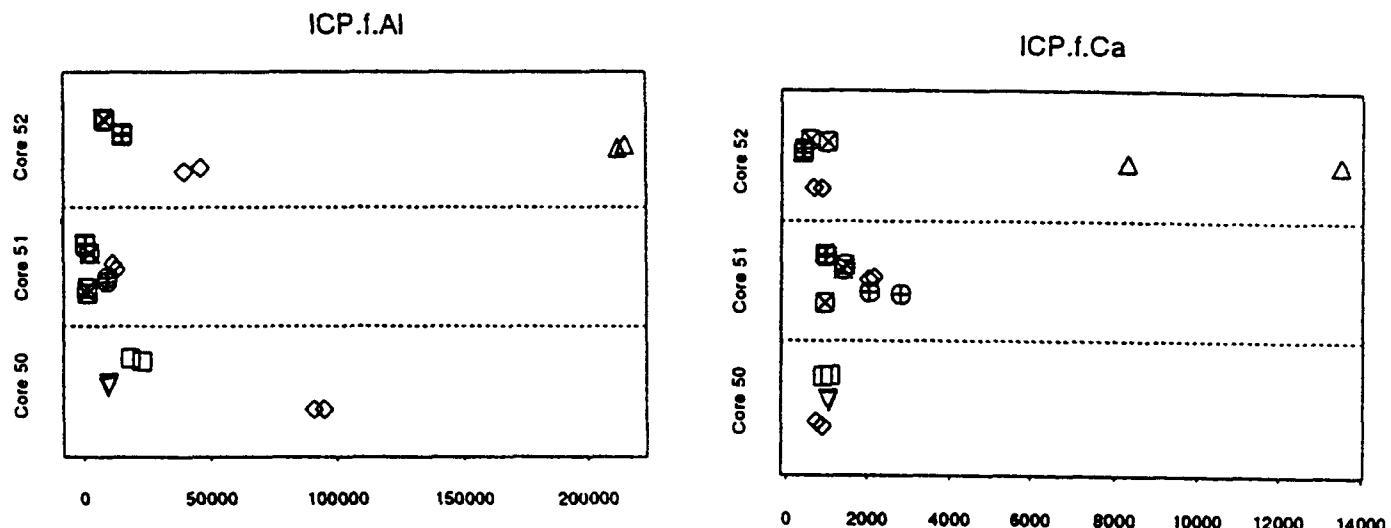
5.1.1 Analytical Results: Inductively Coupled Plasma-Atomic Emission Spectroscopy

Analysis of metals and nonmetals by ICP was performed on water and acid digestions samples, as well as the KOH fusion samples. Online inter-element corrections were performed for matrix interferences. The ICP has a built-in correction capability to adjust for moderate matrix interferences; however, there may be performance degradation on samples containing weight percent quantities of iron, aluminum, or uranium. Corrections were performed to correct for moderate levels of aluminum, calcium, chromium, and magnesium content in the samples. Major elements (greater than 1000 $\mu\text{g/g}$) detected by ICP analysis of the acid digestion of the solid composition were aluminum, bismuth, sodium, phosphorous, sulfur, and iron. The major elements in the water digestion of the solid composites were sodium, phosphorous, sulfur, and silicon. The ICP analysis of the fusion digestion of solid composite samples was used to evaluate the completeness of the acid digestion procedure, and to identify any acid insoluble compounds. The major elements found in the fusion ICP analysis were aluminum, bismuth, iron, sodium, phosphorus, sulfur, and silicon. Figure 5-1 through 5-7 graphically illustrates the ICP results.

Low spike recoveries consistently occurred for several elements in the ICP analysis. Silver recoveries were commonly low due to the precipitation of silver chloride during sample preparation. Poor spike recoveries for iron, magnesium, and calcium accompanied high preparation blank values. When the added spike concentration was insignificant compared to the concentration of the element present in the sample, a failure generally occurred. For this reason, serial dilutions were used for evaluation of the method performance for major constituents. Spike failures are frequently noted for major elements when the spike concentration is insignificant when compared to the analyte concentration present in the waste matrix. Spike and standard results outside the acceptance criterion for these analytes do not necessarily invalidate the sample results for the ICP in general. All of these behaviors could affect, and are considered in the interpretation of the results. The detection limit for each analyte is provided for comparison with the results to aid in interpretation. All ICP analytes are reported in the data tables, however those consistently contributing significant amounts to the composition of the waste matrix are generally relevant to bulk characterization. The acid/fusion ratio for most analytes indicate near total dissolution for the acid digestion assay, with the exception of lithium, samarium, and silicon. The full range of ICP analytical results can be found in the full data packages (HASM 1993). All reported concentration values are based on grams of wet sample, unless otherwise specified. Table 5-1 provides ICP analytes concentrations information on the core composites as a function of the sample preparation.

Homogenization test samples (Core 50 Segment 2; Core 51 Segment 3L; Core 52 Segment 3L) were prepared by first homogenizing the solid segment material in the hot cell. Because of high RPD's all of the homogenization test samples for this tank were digested a second time and analyzed on the ICP in the same manner as the first set of samples. RPD's for a majority of the elements were below the 20% acceptance criterion for acid and fusion results. For the remaining analytes the samples were below their respective detection limits, producing high RPD's which is expected.

Figure 5-1. Graphical Representation of Fusion Digestion Results (1 of 7 pages).



Definition of Plotting Symbols

▽	Segment 1R	⊗	Segment 3U
△	Segment 1	⊕	Segment 3L
◇	Segment 2	⊗	Segment 4U
□	Segment 3	⊕	Segment 4L

Units are ug/g except for radionuclides which are uCi/g

Figure 5-2. Graphical Representation of Fusion Digestion Results (2 of 7 pages).

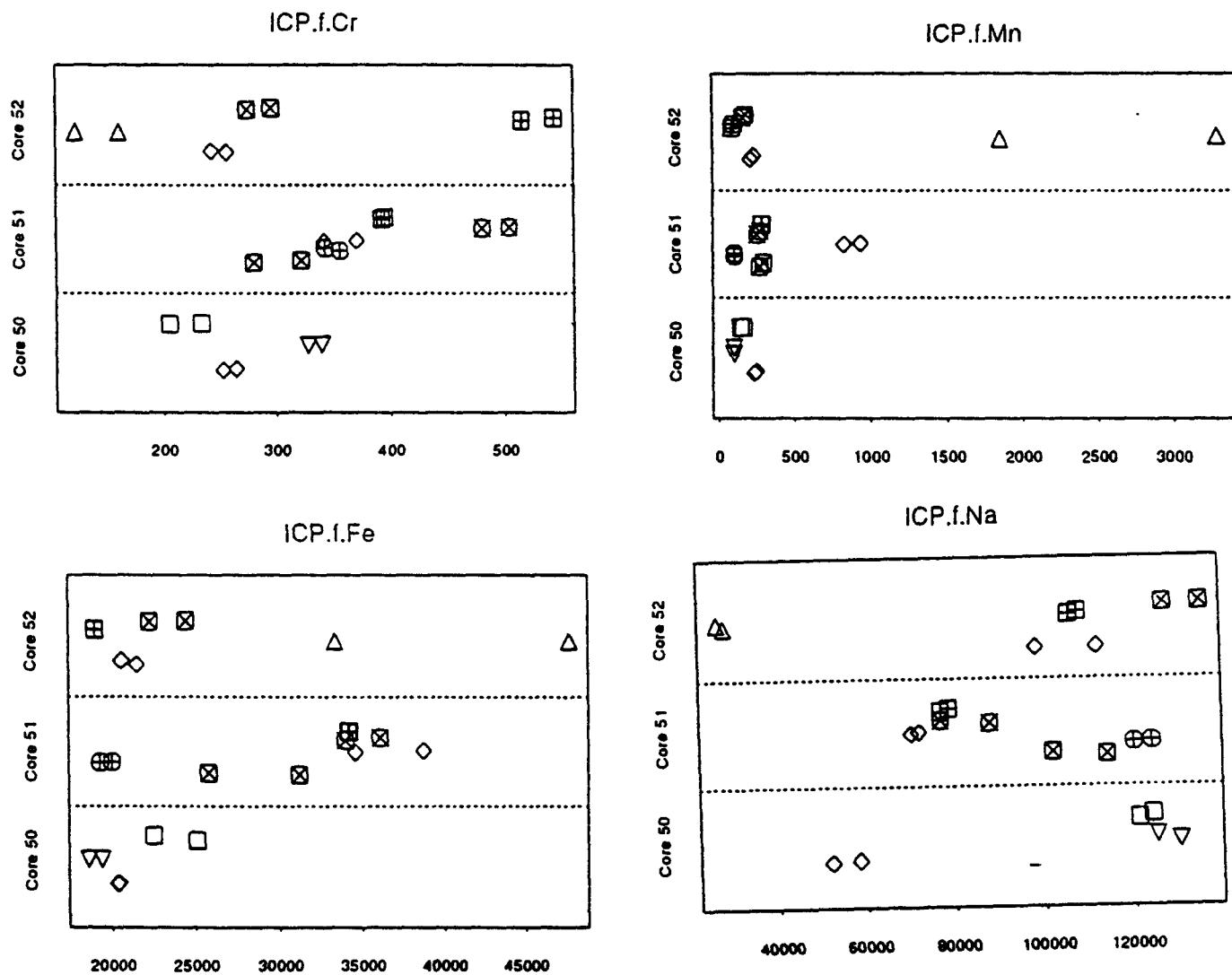


Figure 5-3. Graphical Representation of Fusion Digestion Results (3 of 7 pages).

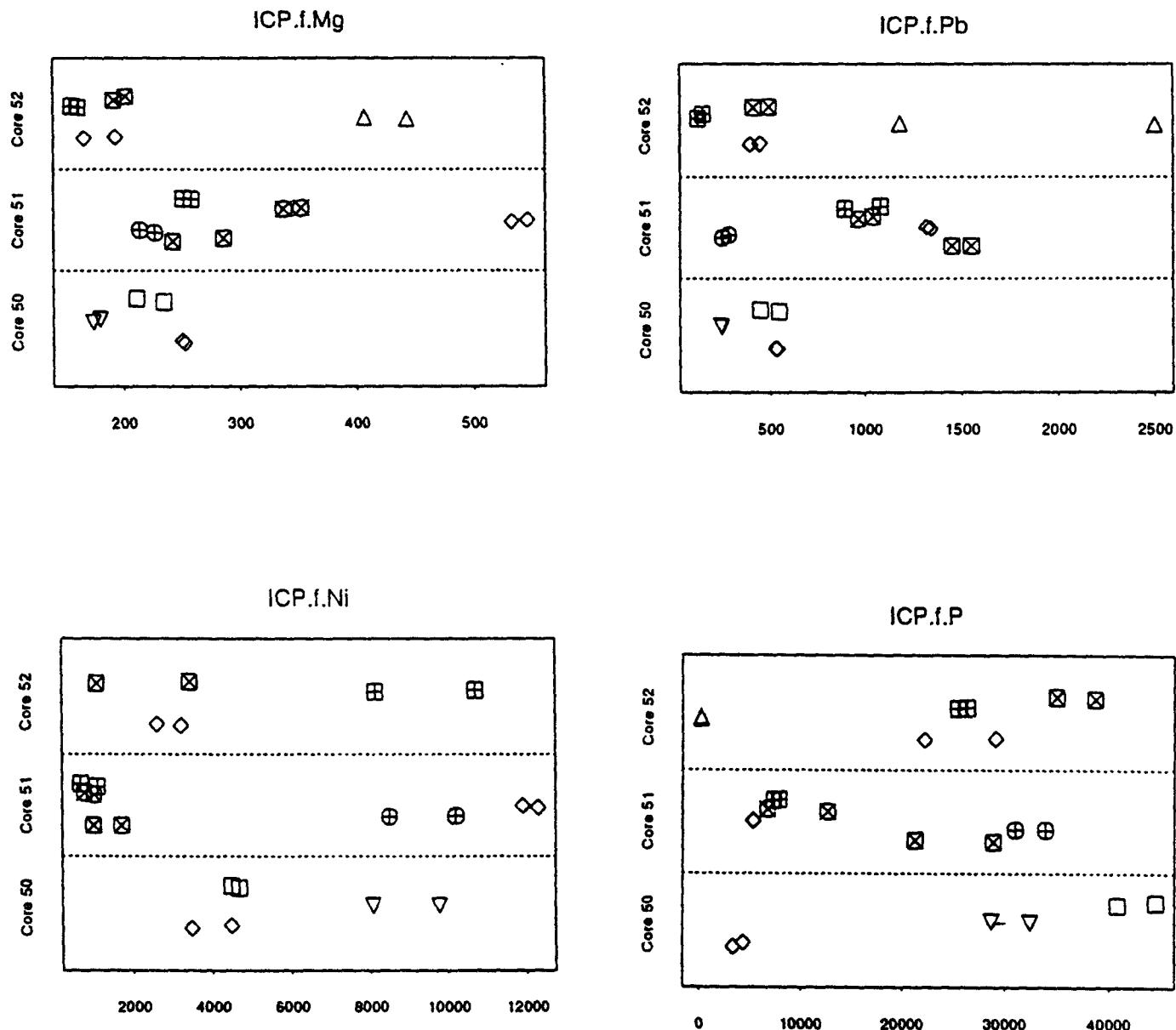


Figure 5-4. Graphical Representation of Fusion Digestion Results (4 of 7 pages).

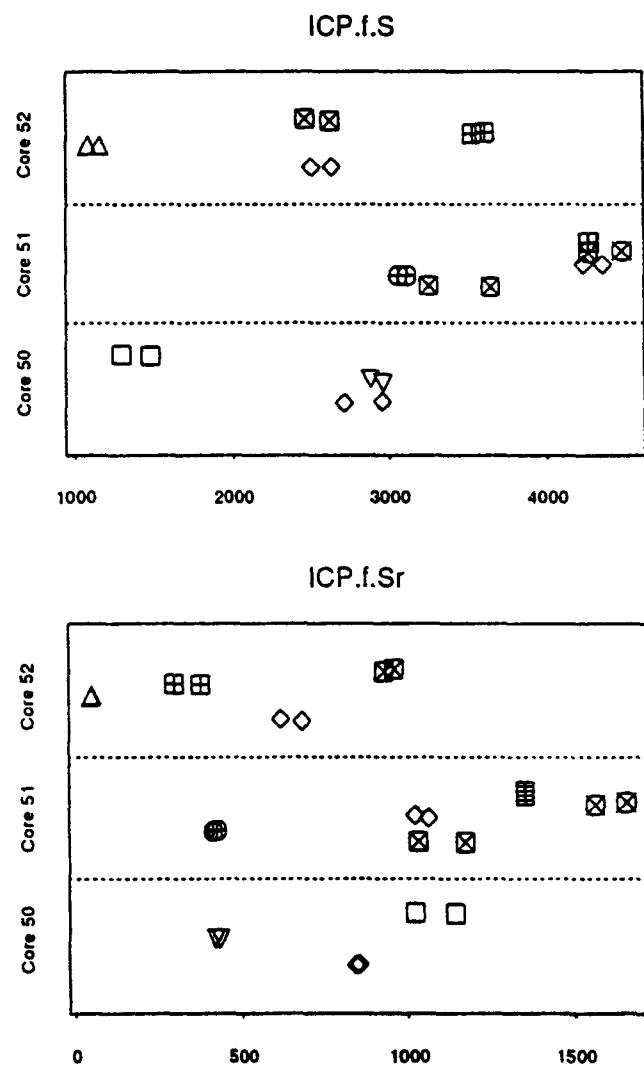


Figure 5-5. Graphical Representation of Fusion Digestion Results (5 of 7 pages).

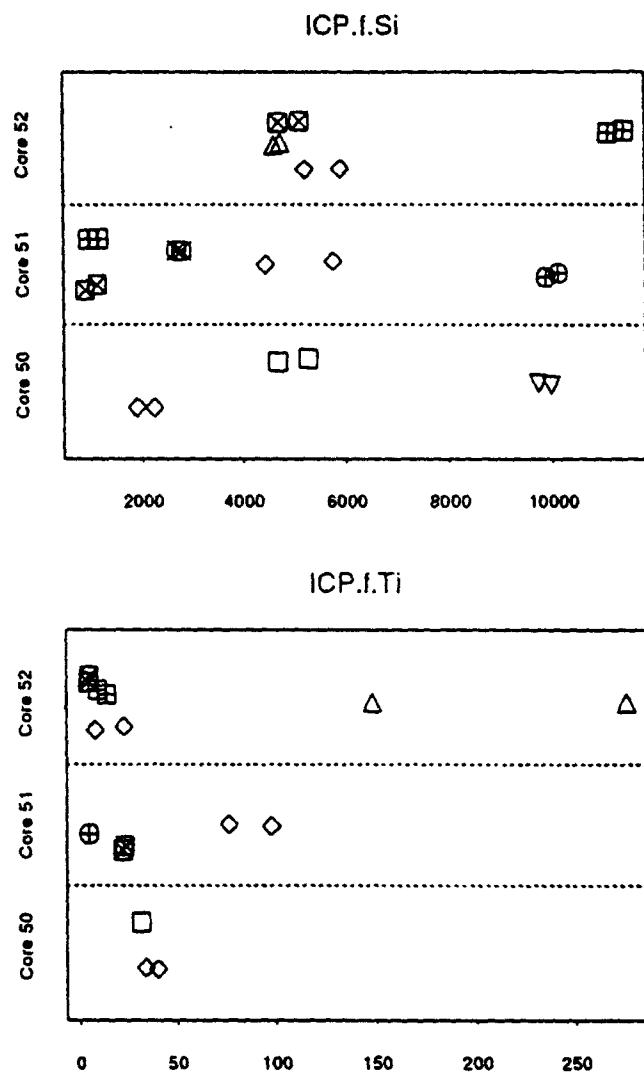


Figure 5-6. Graphical Representation of Fusion Digestion Results (6 of 7 pages).

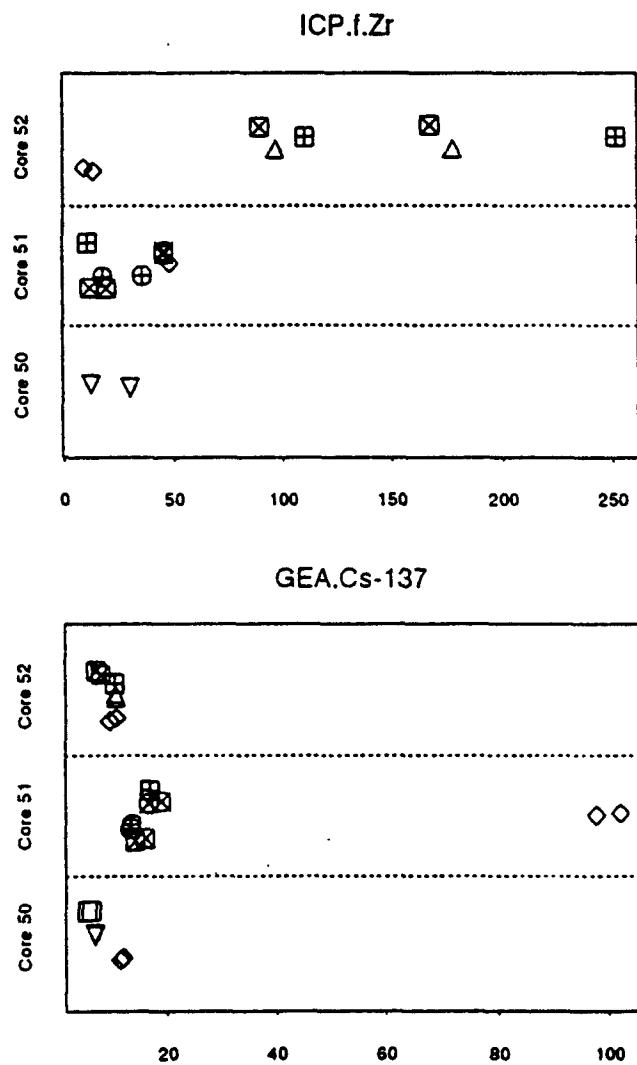


Figure 5-7. Graphical Representation of Fusion Digestion Results (7 of 7 pages).

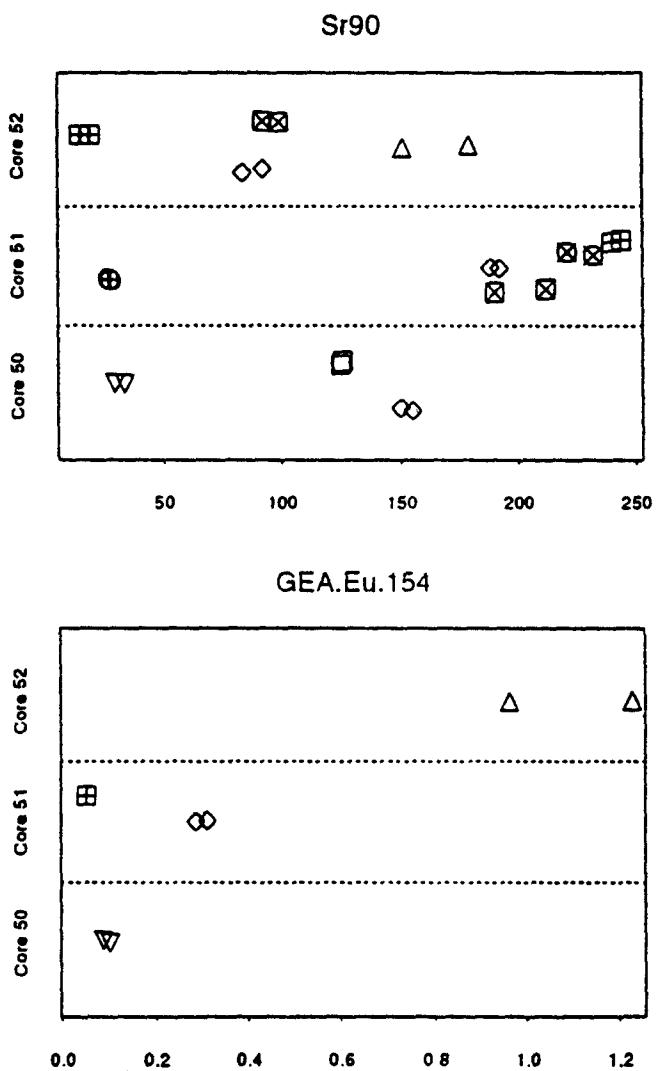


Table 5-1. Analytical Results for T-107. (3 pages)

Analyte	Detection Limit		Prep Type	Core 51 Result 1	Core 51 Result 2	Core 52 Result 1	Core 52 Result 2
Cations	Core 51	Core 52		µg/g	µg/g	µg/g	µg/g
Aluminum (Al)	0.5	0.5	Water	344	627	849	784
	0.5	0.9	Acid	4,030	4,250	23,900	25,300
	4.5	4.5	Fusion	7,260	4,200	26,400	27,500
Antimony (Sb)	21.0	21.0	Water	< DL	< DL	< DL	< DL
	21.0	15.2	Acid	< DL	< DL	37.4	53.6
	76.0	76.0	Fusion	139	104	< DL	< DL
Arsenic (As)	3.0	3.0	Water	< DL	< DL	< DL	< DL
	3.0	3.9	Acid	< DL	< DL	4.63	3.95
	19.5	19.5	Fusion	< DL	< DL	< DL	< DL
Beryllium (Be)	0.30	0.30	Water	< DL	< DL	< DL	< DL
	0.30	0.30	Acid	< DL	< DL	< DL	< DL
	1.50	1.50	Fusion	< DL	< DL	< DL	< DL
Bismuth (Bi)	6.3	6.3	Water	108	81.5	375	409
	6.3	9.2	Acid	7,790	8,130	13,100	14,400
	46.0	46.0	Fusion	8,200	8,490	14,000	17,300
Boron (B)	1.0	1.0	Water	607	666	16.1	1.48
	1.0	1.0	Acid	33.8	13.5	3.59	0.990
	5.0	5.0	Fusion	< DL	< DL	< DL	< DL
Cadmium (Cd)	0.6	0.6	Water	< DL	< DL	< DL	< DL
	0.6	0.7	Acid	7.93	6.50	4.17	7.01
	3.5	3.5	Fusion	6.35	5.95	5.66	9.81
Calcium (Ca)	0.9	0.9	Water	151	800	71.1	59.9
	0.9	0.5	Acid	808	897	542	643
	2.5	2.5	Fusion	765	794	702	781
Chromium (Cr)	0.9	0.9	Water	230	216	213	184
	0.9	1.5	Acid	383	381	309	342
	7.5	7.5	Fusion	351	359	341	389
Cerium (Ce)	6.8	6.8	Water	< DL	< DL	< DL	< DL
	6.8	12.8	Acid	< DL	< DL	180	185
	64.0	64.0	Fusion	87.6	104	132	137
Iron (Fe)	1.5	1.5	Water	315	229	429	449
	1.5	1.6	Acid	33,400	32,900	20,100	39,500
	8.0	8.0	Fusion	26,300	26,600	21,000	42,800

Table 5-1. Analytical Results for T-107. (3 pages)

Analyte	Detection Limit		Prep Type	Core 51 Result 1	Core 51 Result 2	Core 52 Result 1	Core 52 Result 2
Cations	Core 51	Core 52		µg/g	µg/g	µg/g	µg/g
Lanthanum (La)	1.7	1.7	Water	< DL	< DL	< DL	< DL
	1.7	2.0	Acid	< DL	< DL	< DL	< DL
	10.0	10.0	Fusion	< DL	< DL	< DL	< DL
Lead (Pb)	6.2	6.2	Water	20.3	20.1	< DL	< DL
	6.2	7.8	Acid	1,170	1,040	357	618
	39.0	39.0	Fusion	763	690	346	796
Lithium (Li)	0.4	0.4	Water	20.3	20.1	< DL	< DL
	0.4	0.6	Acid	1,170	1,040	357	618
	3.0	3.0	Fusion	4.87	5.07	3.14	4.98
Magnesium (Mg)	0.3	0.3	Water	7.30	12.2	10.0	9.83
	0.3	0.3	Acid	265	259	157	173
	1.50	1.50	Fusion	223	240	190	252
Manganese (Mn)	0.3	0.3	Water	1.78	2.37	< DL	< DL
	0.3	0.3	Acid	236	226	126	298
	1.5	1.5	Fusion	196	183	161	313
Molybdenum (Mo)	0.6	0.6	Water	8.72	8.67	6.96	7.17
	0.6	0.9	Acid	< DL	< DL	5.57	7.26
	4.5	4.5	Fusion	10.5	10.6	7.96	6.63
Neodymium (Nd)	8.2	8.2	Water	< DL	< DL	< DL	< DL
	8.2	7.8	Acid	106	112	35.5	38.2
	39.0	39.0	Fusion	81.1	83.3	55.3	<38.9
Nickel (Ni)	1.3	1.3	Water	5.18	3.79	< DL	< DL
	1.3	1.5	Acid	308	301	274	186
			Fusion	N/A	N/A	N/A	N/A
Phosphorous (P)	4.6	4.6	Water	27,300	33,300	18,400	15,900
	4.6	7.0	Acid	33,900	25,400	33,400	27,300
	35.0	35.0	Fusion	31,500	34,300	33,600	28,900
Potassium (K)	11.6	11.6	Water	641	260	226	135
	11.6	14.8	Acid	267	220	233	217
	74.0	74.0	Fusion	N/A	N/A	N/A	N/A
Samarium (Sm)	9.4	9.4	Water	< DL	< DL	< DL	< DL
	9.4	10.4	Acid	480	448	115	96.3
	52.0	52.0	Fusion	148	208	79.5	63.4

Table 5-1. Analytical Results for T-107. (3 pages)

Analyte	Detection Limit		Prep Type	Core 51 Result 1	Core 51 Result 2	Core 52 Result 1	Core 52 Result 2
Cations	Core 51	Core 52		µg/g	µg/g	µg/g	µg/g
Selenium (Se)	8.7	8.7	Water	58.6	52.1	< DL	89.4
	8.7	14.1	Acid	104	< DL	< DL	< DL
	70.5	70.5	Fusion	< DL	< DL	< DL	< DL
Silicon (Si)	1.6	1.6	Water	3,710	4,180	< DL	< DL
	1.6	3.4	Acid	63.7	156	1,820	1,460
	17.0	17.0	Fusion	4,980	4,750	7,110	7,390
Silver (Ag)	0.5	0.5	Water	< DL	< DL	< DL	< DL
	0.5	0.9	Acid	6.79	7.94	< DL	< DL
	4.5	4.5	Fusion	< DL	< DL	< DL	< DL
Sodium (Na)	6.2	6.2	Water	128,000	140,000	87,100	76,500
	6.2	4.0	Acid	142,000	131,000	131,000	117,000
	20.0	20.0	Fusion	119,000	127,000	115,000	108,000
Strontium (Sr)	0.3	0.3	Water	6.44	4.54	5.06	6.08
	0.3	0.3	Acid	1,250	1,230	704	665
	1.5	1.5	Fusion	934	974	751	854
Sulfur (S)	3.3	3.3	Water	4,100	3,840	3,360	2,860
	3.3	3.9	Acid	3,600	3,520	2,490	2,570
	19.5	19.5	Fusion	3,490	3,640	2,910	3,060
Titanium (Ti)	0.3	0.3	Water	1.25	2.98	< DL	< DL
	0.3	0.7	Acid	< DL	< DL	4.11	1.46
	3.5	3.5	Fusion	< DL	16.3	5.22	7.28
Thallium (Tl)	16.4	16.4	Water	< DL	< DL	< DL	< DL
	16.4	25.0	Acid	< DL	< DL	72	35.4
	125.0	125.0	Fusion	< DL	144	< DL	< DL
Zirconium (Zr)	0.7	0.7	Water	2.10	3.90	12.2	< DL
	0.7	1.2	Acid	117	121	22.5	25.7
	6.0	6.0	Fusion	85.6	66.3	127	93.8
Mercury (Hg)	0.12		Direct/CVAA*	< DL	< DL	0.134	0.144

* CVAA = Cold Vapor Atomic Absorption
 Res. = Result

Several analytical trials were performed by AA on the samples for inorganic metals however the detection for most analytes was in most cases was either too low to be detected or barely above the detection limit (DL).

Cesium analysis was performed on the fusion digestion of the core samples. Determination of cesium was also performed on the Core 51 acid digestion sample because of the larger solid/acid ratio and a lower DL was obtainable. Nevertheless, cesium was not observed.

Mercury analysis was performed on the core solid samples. No mercury was detected in Core 51 composite and in Core 52, an amount just above the detection limit (DL = 0.12 $\mu\text{g/g}$) was observed.

Chromium (VI), analyzed by spectrophotometry, was performed on the water digestion of the core composites. No chromium (VI) was detected in the samples.

Core 50

Because of poor sample recovery, few analyses were performed on Core 50. Inductively coupled plasma fusion results indicate the most abundant analytes found were sodium, aluminum, bismuth, and phosphorus. Since KOH fusion is one of the only preparation techniques performed on Core 50 no inferences can be made regarding the solubility of the analytes within the waste. However, given the process history of the tank, it is unlikely these analytes (except Na) exist in their water soluble forms. From Table 5-2 there appears to be an inconsistency between Segments 1 and 3 in several of the analytes in that there is a drop in concentration for the analytes. This anomaly may be due to the fact Core 50 Segment 3 was not homogenized or it may be evidence of tank heterogeneity (i.e. layering). When the RPD's from the major metals were compared, for the various segments, a majority of the analytes were within the 20% acceptance criterion. This would suggest the drastic change in concentration, toward the middle (vertically) of the tank, seen in Table 5-2 represents the waste configuration. The higher RPD's appear in the less significant contributors, which could be attributed to the fact the analytes are close to the detection limits of the ICP and the high dilution factor of the fusion preparation.

Table 5-2. Tank 241-T-107 Core 50 ICP Selected Analyte Trends as a Function of Depth.
(Fusion prep on samples)

Seg	Al ($\mu\text{g/g}$)	Ca ($\mu\text{g/g}$)	Cr ($\mu\text{g/g}$)	Na ($\mu\text{g/g}$)	Bi ($\mu\text{g/g}$)	Pb ($\mu\text{g/g}$)	P ($\mu\text{g/g}$)	Si ($\mu\text{g/g}$)	S ($\mu\text{g/g}$)	Fe ($\mu\text{g/g}$)
1R	9,810	1,050	333	127,000	19,400	242	30,600	9,850	2,920	19,000
2	92,900	822	258	55,200	2,610	530	3,840	2,060	2,830	20,400
2 ^a	95,600	900	279	71,300	3,250	636	5,240	1,640	3,600	25,700
3	20,700	1,100	219	123,000	11,000	496	42,700	4,960	1,380	23,800
4	IS	IS	IS	IS	IS	IS	IS	IS	IS	IS

^a Average of the acid digestions samples used in homogenization tests

IS = Insufficient Sample

In the fusion assays, some elements can appear to be at high concentrations because of the large dilution factors required for fusion samples. Those analytes may actually only be present in concentrations marginally above the detection limit. For several analytes, higher quantitation was found in the acid digestion results. For purposes of determining inventories and making comparisons, the highest reliable average analytical result will be used between acid and fusion preparations. In reviewing the KOH fusion data most of the samples contain less than $5.0 \times 10^4 \mu\text{g/g}$ of aluminum. Only Core 50 Segment 2 and Core 52 Segment 1 have concentrations of 9.29×10^4 and $2.14 \times 10^5 \mu\text{g/g}$ aluminum respectively. This anomalous concentrations could possibly be caused from a piece of crust or a particle inclusion. Calcium results are considerably uniform except for the Core 52 Segment 1 samples and one of the four core composite results. All cadmium results are less than 10 times the detection limit (DL). Cerium, lithium, and molybdenum results show scattered results as expected for concentrations less than 10 times the DL. Chromium concentrations for both of the cores appear to be fairly homogeneous throughout the tank. Although samples for Core 51 and Core 52 appear to have high iron concentrations, they appear to be consistent throughout the tank. The concentrations of magnesium has good reproducibility between duplicate pairs, however the concentration throughout the tank are slightly scattered. Manganese results appear to be very consistent with the exception of only two samples, Core 51 Segment 2 and Core 52 Segment 1. Sodium results have close agreement between duplicates, but concentrations appear to vary by location in the tank. Composite results for sodium are generally higher than the segment level results. Phosphorous results show a widely different phosphorus content in different areas of the tank. Since phosphorus does not have many contamination pathways vertical and lateral heterogeneity are assumed. Results for lead, sulfur, silicon, and strontium all show fairly consistent pairing but demonstrate variation in concentrations as a function of sampling location. This type of observations are expected, process batches within a given campaign may be responsible. Antimony, samarium, neodymium, titanium, and zirconium are all less than 10 times DL; since these are considered trace analytes this is not surprising. Cores 50 and 52 appear to have similar compositions, while Core 51 differs markedly from those two cores.

Inductively coupled plasma analysis was also performed on the acid digestions of Core 50 Segment 2. This analysis was run several times in order to determine the efficiency of the homogenization procedure. Comparing the results from the KOH fusion with the average of the results obtained from the acid digestion samples show good correlation for Segment 2. This correlation strengthens the observed change in concentration seen in the ICP fusion results. The only analyte to change drastically is nickel, but this behavior is expected because of contamination from the nickel crucibles used in the fusion digestion. The more reliable result for nickel is from the acid digestion. The RPD results for the acid digestion of Core 50 Segment 2 are below the acceptance criterion in most cases. The constant exceptions to this were phosphorus, potassium, boron, and bismuth.

Core 51

The water digestion of the core composite revealed sodium, phosphorous, sulfur, and silicon to be the most abundant analytes. Sulfur, and phosphorous are likely to be present in their water soluble forms, sulfate and phosphate. The high concentrations of silicon in Core 51 is unusual because neither silicon nor any of its compounds dissolve readily in water. The high Si concentrations could be attributed to contamination of the sample, as of present, from an unknown source. The majority of the analytes above the detection limit had high RPD's. A large number of the analytes were below the detection limit, therefore, no relevant RPD's could be calculated.

The results obtained from the acid digestions improved substantially from the water digestions. The major analytes observed were aluminum, bismuth, iron, lithium, phosphorus, sodium, strontium, sulfur, and titanium. The RPD's were very good in general for most of the major analytes (generally between 5% and 10%), well within the 20% acceptance criterion. Duplicate results, for acid digestion agree considerably better than the duplicate results from the water digestion samples. The elements that were the exceptions to this are selenium and silicon (200% and 83.5% respectively). Selenium's high RPD's are not surprising due to the fact that one of the results is below the detection limit. The high silicon RPD's, from the acid digestions, are also not unexpected since the waste has solubility properties that resist acid digestion, making the results marginal, at best. Based on the ratio of water digestion to acid digestion results, a substantial portion of the analytes found in the waste are not water soluble. Comparisons with the acid digestion results indicate that the samples were well dissolved by the acid preparation, but in some cases, Si especially, the fusion dissolution was necessary to obtain reliable, quantitative results for the analyte.

Fusion results for Core 51 are similar to the acid digestions. The exceptions to this were silicon and boron. Since silicon is more readily digestible in the fusion preparation than acid, the silicon results were not surprising. The observed boron concentrations indicate a majority of the boron was brought into solution with acid digestion, while the fusion results are less than the detection limit. These boron results are not expected. It's possible, since fusion digestion samples require a large dilution ratio, the sample concentration may have been too low to detect. The RPD's for the fusion results are generally between 5% and 10%. The exceptions to this were aluminum, nickel, sulfur, titanium, thallium, and zirconium. As noted previously, nickel concentration will be erratic due to the nickel crucible contaminations. The sulfur concentration trends seen in Table 5-3 appear fairly consistent through the core even though it has high RPD's. The concentrations of the analytes in segments 3U, 3L, and 4L appear to be inconsistent with the other segments. Core 51 is similar to Core 50 in all analytes with the exception of bismuth. The bismuth concentrations in Core 50 Segment 1 is considerably lower concentration of analytes toward the middle (vertically) of the tank follows for both Cores 50 and 51. Titanium, thallium, and zirconium concentrations are low and dilution from the fusion preparation makes their results unreliable. Lastly, aluminum concentrations as a function of depth have been noted as being inconsistent.

Core 51 Segment 3L was acid digested in order to perform homogenization test. The average of the acid digestions and the fusion results agree very well except for nickel and aluminum. The aluminum concentrations observed for the acid digestion are lower than the concentrations determined from fusion. This difference can be explained by fusion dissolution being a more vigorous means of bringing samples into solution than acid digestion. This anomaly adds evidence to tank layering or spot heterogeneity. The nickel can be easily explained by contamination from the nickel crucibles. The comparison of the fusion result with the acid digestion results for several analytes suggests the observed change in concentration as a function of depth actually represents the waste configuration, thus the waste is layered in some fashion.

Table 5-3. Tank 241-T-107 Core 51 ICP Selected Analyte Trends as a Function of Depth.
(Fusion prep on samples)

Seg	Al ($\mu\text{g/g}$)	Ca ($\mu\text{g/g}$)	Cr ($\mu\text{g/g}$)	Na ($\mu\text{g/g}$)	Bi ($\mu\text{g/g}$)	Pb ($\mu\text{g/g}$)	P ($\mu\text{g/g}$)	Si ($\mu\text{g/g}$)	S ($\mu\text{g/g}$)	Fe ($\mu\text{g/g}$)
1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
2	12,000	2,090	355	71,100	987	1,330	5,330	5,090	4,290	36,700
3U	1,240	961	301	108,000	936	1,500	25,100	981	3,440	28,500
3L	688	989	393	77,900	1,070	986	7,610	1,030	4,270	34,300
3L ^a	267	848	363	89,400	1,210	1,130	9,320	840	4,360	36,000
4U	2,270	1,430	492	82,500	3,620	1,000	9,700	2,730	4,370	35,100
4L	9,210	2,440	349	122,000	18,500	264	32,400	9,980	3,080	19,700

^a Average of the acid digestions samples used in homogenization tests

NS = No Sample

Core 52

The major analytes revealed from the water digestion of the core composite were phosphorous, sodium, and sulfur. Both sulfur (as sulfate) and phosphorus (as phosphate) are found in their water soluble forms. High water soluble silicon concentrations were not seen in Core 52, giving some support to the contention that concentrations found in Core 51 were anomalous, resulting from either sample contamination or local heterogeneity. Again a high majority of the species analyzed using this sample preparation were close to or below the detection limit. Water digestion analysis produced acceptable RPD's (between 5-15%) for those species above the detection limit. Potassium, selenium, and zirconium had high (over the 20% acceptance criterion) RPD's. Selenium and zirconium having high RPD's is not surprising because fact the waste contains low concentration and these species are not water soluble. Potassium's high RPD (approximately 50%) is possibly due to contamination of the sample. The high RPD's result from having one result below detection limit. A large number of species did not report RPD's because both results were below detection limits. Water digestion analysis does not dissolve many species of the waste into solution and can be dependent on particle size distribution. Therefore low concentrations for water digestions are to be expected, resulting in high RPD's for soluble, or partially soluble analytes; or non-existent RPD's for insoluble or absent analytes.

Acid digestion for Core 52 reveal aluminum, bismuth, iron, phosphorus, silicon, sodium, and sulfur as the major components of the waste. The major components, with the exception of silicon are equivalent to Core 51. Lithium and strontium are found in lower concentrations in Core 52 than 51. Lithium concentrations are considerably lower (almost 2.5 times) for the acid digestion results; this may be due to cross contamination during the analysis. Strontium's concentration drop is not as notable as lithium and the duplicate results agree very well, suggesting the results are valid. This anomaly suggests a slight gradient from one side of the tank to the other and indicates the results are potentially dependent on the sampling location proximity to the waste inlets and outlets. Silicon, aluminum, and bismuth were more abundant in Core 52 than 51. Silicon and aluminum have the greatest difference in concentration between cores (approximately 10 times and 5 times respectively). Again, this may be due to a particle inclusion of an aluminosilicate. The bismuth concentration for Core 52 is two times higher than Core 51. The RPD's for the acid digestion were acceptable with few species having a calculated RPD greater than 20%.

Fusion digestion results were similar to the acid digestion concentrations. The major species detected via this method were aluminum, bismuth, iron, nickel, phosphorous, selenium, silicon, sodium, and sulfur. The high nickel concentrations observed were attributed to the nickel crucibles used in fusion assay procedure. Selenium and silicon concentrations are higher than those obtained from acid digestion. This anomaly is to be expected because the fusion is a more vigorous sample preparation than acid preparation and provides more reliable quantitation for significant analytes. Several analytes produced high RPD's (over the 20% acceptance criterion). Some of these analyte concentrations were very close to the detection limits causing some of the high RPD's. Very few of the analytes significantly above the detection limit produced RPD's within the acceptance criterion.

Examination of the fusion digestion results in the individual segments in Core 52, reveals that the concentrations of aluminum, calcium, lead, sodium, and phosphorous are at least two times higher than Core 51. The aluminum concentrations for Core 52 Segment 1 are significantly higher (approximately 1680%) than the other two cores (see Table 5-4). This high concentration was not surprising when considered in context; Core 52 Segment 1 has a much lower percent water content than core 50 and 51. The low percent water in the segment suggests that the core may have a crust (perhaps of dried CW) forming under riser 3 from where 52 was taken. A salt cake crust based on CW would consist of aluminum salts, usually Al(OH)_x or aluminosilicates, giving rise to the large concentrations of aluminum. Cores 50 and 51 have a high percent water content, implying the cores do not have a substantial salt cake in the segments. Also, from historical data, coating waste was among the last waste types discarded to the tanks and had substantial amounts of solids associated with the waste stream. Coating waste solids are extremely high in aluminum concentration and this waste should be located toward the top of the waste. The difference in concentration for this analyte also suggests a filling effect (i.e. a heterogeneity) resulting from the samples proximity to a waste inlet. The variation of analyte concentration as a function of depth is not as evident in Core 52. By comparing the Al results obtained from the homogenization test on Segment 3U (acid digestion) with the fusion digestion, the values are comparable. The nickel concentration for the acid digestion are more acceptable than the fusion results because of the crucible contamination. Silicon concentrations for acid digestion are lower than fusion for the reason given in the above paragraph. Core 52 (riser 3) is physically close to riser 2 where core 50 was extracted from, therefore it would be logical that a majority of the results should be similar. However, the expected degree of similarity is not observed.

Table 5-4. Tank 241-T-107 Core 52 ICP Selected Analyte Trends as a Function of Depth.
(Fusion prep on samples)

Seg	Al ($\mu\text{g/g}$)	Ca ($\mu\text{g/g}$)	Cr ($\mu\text{g/g}$)	Na ($\mu\text{g/g}$)	Bi ($\mu\text{g/g}$)	Pb ($\mu\text{g/g}$)	P ($\mu\text{g/g}$)	Si ($\mu\text{g/g}$)	S ($\mu\text{g/g}$)	Fe ($\mu\text{g/g}$)
1	214,000	10,900	142	27,300	376	1,840	NA	4,660	1,120	40,500
2	43,000	771	249	105,000	11,100	420	25,600	5,540	2,558	21,000
3U	8,170	800	286	131,000	10,300	452	36,900	4,880	2,550	23,400
3L	15,400	422	531	107,000	22,300	132	26,000	11,300	3,570	19,000
3L ^a	15,800	499	581	113,000	25,200	147	26,600	1,330	3,530	20,600
4	IS	IS	IS	IS	IS	IS	IS	IS	IS	IS

^a Average of the acid digestions samples used in homogenization tests

IS = Insufficient Sample

NA = Not Analyzed

5.1.2 Analytical Results--Anion Assays

Ion Chromatography Results

The detection of anions in the tank was performed using ion chromatography (IC). Ion chromatography was performed on the water digestion samples of the solid core composites. The anions fluoride (F^-), chloride (Cl^-), nitrite (NO_2^-), nitrate (NO_3^-), phosphate (PO_4^{3-}), and sulfate (SO_4^{2-}) were analyzed for by IC.

In general, the concentrations of the anions for Cores 51 and 52 are higher than expected. A majority of the anions in Table 5-5, with the exception of fluoride and phosphate, are usually associated with water soluble compounds. Theoretically, water digestion preparation of solid samples should not contain extremely high concentrations of water soluble compounds if the samples are in a matrix of 56% water. However, this expectation is not the case. One possibility for the results is the waste could have reached a supersaturation point producing crystals for each analyte. These anomalies may also be attributable to spatial variability. The placement of the inlet and outlet risers with respect to the risers where the samples were taken from could substantially influence the samples. The significant difference in the concentrations between the two cores suggests that no microconvection occurred within the tank since convective mixing would tend to even out any concentration gradients among soluble species. If there is no microconvection, little mixing of the waste occurs. Since little mixing has occurred, and there are several potential sources for spatial bias, the significant difference in the results is observed.

Core 51

Ion chromatography revealed high concentrations in all anions except chloride. Although nitrite is presumed to be a minor contributor to the waste, the nitrate concentration is quite large. Because the ion chromatography results for nitrite were considered an estimate, the results were confirmed by spectrographic analysis. The results from the spectrographic analysis compare very well with the ion chromatography results; RPD results between the two methods were under 6%. The RPD's for all anions detected by ion chromatography fell under the 20% acceptance criterion. Comparison of P and S from water digestion ICP results with ion chromatography results for PO_4^{3-} and SO_4^{2-} gives poor agreement for Core 52, however, Core 51 solubility results are very favorable with phosphorus and sulfur appearing to be almost 100% water soluble. The concentrations observed by IC are notably larger than those of the ICP in Core 52. Core 51 IC and ICP results for the samples agree fairly well. Tables 5-5 and 5-6 presents the summary results for the ion chromatography ions as well as a comparison of IC and ICP.

Core 52

Ion chromatography indicate results similar to those observed in Core 51. Chloride is found in very low concentrations, while the other ions are seen in abundance. Nitrite, nitrate, and sulfate concentrations in Core 52 are half of those seen in Core 51. The RPD's for these ions are very good with all analytes having RPD's less than 10%. When IC and ICP results were compared they did not parallel. These results could also be attributed to insufficient water digestion of the samples. Phosphate results for IC were an order of magnitude greater than ICP results; sulfate concentrations for ICP were half of the IC results. The solubility for PO_4^{3-} was approximately 50% for Core 52, this low solubility could be attributed to several

different possibilities. This anomaly could also be due to insufficient water digestion of the sample. Secondly, the low solubility could suggest a change in waste type or particle size. Tables 5-5 and 5-6 give a summary of the IC results and a comparison of IC and ICP, respectively. The difference in concentrations between the two Cores (51 and 52) for several of the anions and cations is suggestive of lateral bias or heterogeneity.

Table 5-5. Ion Chromatography Results for Core Composite Samples. (Water digestion prep on samples)

Analyte	Detection Limit		Core 51 Average	Core 52 Average
	Core 51	Core 52	µg/g	µg/g
Fluoride (F ⁻)	10	4.1	9,200	13,600
Chloride (Cl ⁻)	20	4.2	682	399
Nitrite (NO ₂ ⁻)	100	21	15,300	8,100
Nitrate (NO ₃ ⁻)	100	21	92,800	56,300
Sulfate (SO ₄ ²⁻)	100	41	12,650	7,300
Phosphate (PO ₄ ³⁻)	100	21	94,500	132,500

Table 5-6. Comparison of IC and Water Digestion ICP results for Selected Analytes.

Sample ID	PO ₄ ³⁻ Concentration (µg/g)		PO ₄ ³⁻ RPD	SO ₄ ²⁻ Concentration (µg/g)		SO ₄ ²⁻ RPD	PO ₄ ³⁻ Solubility*		SO ₄ ²⁻ Solubility*	
	(IC)	(ICP.w)		(IC)	(ICP.w)		(IC)	(ICP.w)	(IC)	(ICP.w)
C51	92,000	92,700	-0.75	12,650	11,910	6.03	91.4	92.1	118.3	111.4
C52	132,500	52,479	86.52	7,300	9,330	-24.1	138.6	54.9	81.9	104.2

* Solubility is the ratio of the IC/ICP.w result to the ICP fusion result.

Core 51 PO₄²⁻.f = 100,700 µg/g
 SO₄²⁻.f = 10,700 µg/g

Core 52 PO₄²⁻.f = 95,600 µg/g
 SO₄²⁻.f = 8,900 µg/g

IC = Ion Chromatography

ICP.w = Notation for ICP water digestion result

Core 51 and 52 Other Anion Results:

Nitrite concentrations determined by spectroscopic methods are in good agreement with the respective core duplicates, however, the concentration difference between Core 51 and Core 52 is similar to that seen earlier in the IC results (Core 51 is approximately 2 times higher than Core 52). The pH of the solids is done using a water dilution of a solids aliquot. The results for tank T-107 from this method are consistent, with a pH of 11.6. Ammonia results for the solids were all below the detection limits. Since ammonia is a volatile species, over time it was likely removed through passive ventilation. The results seen for the direct cyanide analysis is Core 51 having higher concentrations than Core 52. The RPD's for each core is good, less than 10%.

Table 5-7. Composite Data (water leach)*.

Analyte	Detection Limit ($\mu\text{g/g}$)	Core 51 ($\mu\text{g/g}$)		Core 52 ($\mu\text{g/g}$)	
Nitrite ¹ (NO_2^-)	380	15,000	13,500	7,710	8,240
Total Organic Carbon (TOC)	550	1,520	1,350	2,000	1,920
Total Inorganic Carbon (TIC)	500	5,640	5,710	2,990	2,560
Free Hydroxide (OH^-)	--	--	--	--	--
pH	NA	11.6	11.6	11.4	11.4
Ammonia (NH_3)	800/4000	<820	<816	<4200	<4260
Direct Cyanide (CN^-)	2	92.9	90.6	44.9	46.9

¹ Nitrite by Spectrophotometry

* Direct Cyanide, NH_3 , pH, TOC, and TIC are not IC analysis, but are reported together for convenience.

NA Not applicable

Carbon Analysis

The total organic carbon (TOC) and total inorganic carbon (TIC) analyses were performed on the direct subsegment or segment samples and core composite samples using the hot persulfate oxidation method. Total organic carbon (TOC) results from 222-S Laboratory and TOC/TIC/TC analyses performed by Pacific Northwest Laboratory (325) are found in Tables 5-8 and 5-9. In both cases the results are consistent between the two cores. The RPD's for the cores are less than 10%. Total inorganic carbon results are consistent for the individual core composites; however, comparing the two cores, Core 51 is 1.4 times greater than Core 52. The TOC and TIC results were derived independently; total carbon (TC) was calculated by adding the corresponding TOC and TIC values.

The TOC and TIC analyses were performed on the liquid samples (drainable liquid composites and water leach of the core solids composites) using coulometric detection. These analyses were performed at the Westinghouse Hanford Company's 222-S Laboratory.

Table 5-8. Total Carbon, Total Inorganic Carbon, and Total Organic Carbon
 Results for Segments and Subsegments.
 Performed by 325 Laboratory.
 Hot Persulfate Method.

Sample	Total carbon (µg/g)	Total inorganic carbon (µg/g)	Total organic carbon (µg/g)	Total organic carbon (µg/g dry sample)
Core 50, Segment 1R	2,260	1,760	505	616
Core 50, Segment 2	3,690	3,040	655	1,120
Core 50, Segment 3	IS	IS	IS	IS
Core 50, Segment 4	IS	IS	IS	IS
Core 51, Segment 2	5,110	4,020	1,100	2,750
Core 51, Segment 3U	4,420	3,150	1,270	2,820
Core 51, Segment 3L	3,530	2,630	905	1,920
Core 51, Segment 4U	3,050	2,780	265	589
Core 51, Segment 4L	1,930	1,670	270	535
Core 52, Segment 1	4,080	2,140	1,950	2,340
Core 52, Segment 2	3,930	2,960	970	1,880
Core 52, Segment 3U	2,040	1,350	685	1,410
Core 52, Segment 3L	1,760	1,490	265	570
Core 52, Segment 4	IS	IS	IS	IS

IS = Insufficient sample for analysis

Table 5-9. Total Carbon, Total Inorganic Carbon, and Total Organic Carbon Results for Drainable Liquids and Core Solids Composites.

Sample	Method	Percent Water	Total carbon ($\mu\text{g/g}$)	Total inorganic carbon ($\mu\text{g/g}$)	Total organic carbon ($\mu\text{g/g}$)	Total organic carbon ($\mu\text{g/g dry sample}$)
Core 50, drainable liquid composite (222-S)	Coulometric detection	95.6	1,660 $\mu\text{g/mL}$	512 $\mu\text{g/mL}$	1,150 $\mu\text{g/mL}$	25,600
Core 51, drainable liquid composite (222-S)	Coulometric detection	75.3	5,600 $\mu\text{g/mL}$	4,540 $\mu\text{g/mL}$	1,060 $\mu\text{g/mL}$	3,580
Core 52, drainable liquids composite (222-S)	Coulometric detection	86.5	693 $\mu\text{g/mL}$	339 $\mu\text{g/mL}$	354 $\mu\text{g/mL}$	2,360
Core 51, core solids composite, water digest (222-S)	Coulometric detection	51.9	7,120	5,680	1,440	2,990
Core 51, core solids composite, direct (325)	Hot persulfate oxidation	51.9	2,480	2,080	400	832
Core 52, core solids composite, water digest (222-S)	Coulometric detection	47.8	4,740	2,780	1,690	3,750
Core 52, core solids composite, direct (325)	Hot persulfate oxidation	47.8	1,640	1,320	320	613

Total organic carbon results for all samples are well below the Ferrocyanide DQO established threshold of 8 wt% (80,000 $\mu\text{g/g dry sample}$) carbon. Only the Core 50 drainable liquid composite sample approached the limit. However, this liquid would exist in the tank as interstitial liquid and a layer of waste with such a TOC concentration would not exist in the tank. The TC and TIC results had RPDs below 25% in almost all cases. The single exception is Core 52, Segment 3U which had a RPD of 31% for TC and 41% for TIC. These high RPDs were attributed to sample inhomogeneity as the sample was observed to contain "unusual hard chunks."

A comparison of the core composite results for the two analytical methods shows a large discrepancy between the results obtained from the hot persulfate oxidation method performed by 325 Laboratory and the coulometric detection method performed by 222-S Laboratory. Carbon results on the water digestion samples using the coulometric method are two to six times higher than results on the direct samples using the hot persulfate oxidation method. The results for the total organic carbon indicate a general decreasing trend as a function of depth for both Core 51 and Core 52. Core 50 did not have enough data to determine the same observation. This decreasing trend could be attributed to a possible

lateral heterogeneity within the tank. The coulometric method may be higher than the hot persulfate method because of potential chemical interferences such as the oxides of sulfur and nitrogen, all of which may be evolved from the addition of acids or their salts.

Cyanide Analysis

Cyanide analysis was performed on (1) segment/subsegment samples; (2) drainable liquid and solid core composite samples; and (3) the water digestion of the solid core composite samples. Cyanide concentrations were found to be greater in Core 51 samples than in the Core 50 and Core 52 samples. In all samples, the ferrocyanide concentration is considerably lower than the established criterion of 8 wt% (80,000 $\mu\text{g/g}$ dry sample) to categorize the tank as Safe. Cyanide concentrations were found to be greater in Core 51 samples than in those of Core 50 and 52. A comparison of the core composites and water digestion results indicated that most of the cyanide is present in water soluble form. Table 5-10 summarizes the cyanide results.

Table 5-10. Cyanide Results for Tank T-107.

Sample	Cyanide ($\mu\text{g/g}$)	Cyanide ($\mu\text{g/g}$ dry sample)	Ferrocyanide equivalent ¹ ($\mu\text{g/g}$ dry sample)
Core 50, Segment 1R	48.5	59.1	80.2
Core 50, Segment 2	64.0	109	148
Core 50, Segment 3	42.7	75.3	102
Core 50, Segment 4	IS	IS	IS
Core 51, Segment 2	95.2	239	324
Core 51, Segment 3U	110	245	333
Core 51, Segment 3L	102	217	295
Core 51, Segment 4U	91.5	203	276
Core 51, Segment 4L	57.3	114	155
Core 52, Segment 1	31.0	37	50.2
Core 52, Segment 2	61.7	120	163
Core 52, Segment 3U	52.1	107	145
Core 52, Segment 3L	43.5	93.5	127
Core 52, Segment 4	IS	IS	IS
Core 50, drainable liquid composite	13.4 $\mu\text{g/mL}$	299	406
Core 51, drainable liquid composite	152 $\mu\text{g/mL}$	513	697
Core 52, drainable liquids composite	39.8 $\mu\text{g/mL}$	266	361
Core 51, core solids composite	95.8	199	270
Core 51, core solids composite, water digest	91.8	191	259
Core 52, core solids composite	56.4	108	147
Core 52, core solids composite, water digest	45.9	87.9	119

¹ Assumes all cyanide is present as ferrocyanide ($\text{Fe}(\text{CN})_6^{4-}$).

IS = Insufficient sample for analysis

5.2 ANALYTICAL RESULTS--RADIOCHEMISTRY

5.2.1 Alpha Emitters

Total alpha was performed on the fusion and water digestion samples. Total alpha results were difficult to obtain because of interference from the high salts resulting from the fusion preparation. Therefore small sample sizes were used to minimize the amount of salts on the mount. Normally, plutonium and americium account for >95% of the total alpha results. This is not the case for the composite fusion digestion samples (the only matrix for which isotopes were measured). The results appear to show a higher total alpha concentration than the sum of the representative isotopes (^{238}U , ^{239}Pu , ^{241}Am). The higher total alpha concentration may be due to: 1) high counting error 2) cross talk from Cs-137 and Sr-90/Y-90 present in the samples (total beta was 1,000 times greater than total alpha for these samples). A small amount of the β -emissions may be confounding the detector (the activity of the samples is so low that the offset used to discriminate between alpha and beta plateaus was not sufficient to provide accurate readings). Presently, this particular issue of cross talk between alpha and beta emitters has been resolved 3) another alpha emitting isotope may be present which is not identified or quantified and/or 4) the uranium isotopic content maybe significantly different than that found in nature. Isotopic determination of the samples was obtained by thermal ionization mass spectroscopy.

By comparing fusion and water digestion results, it appears that most of the alpha-emitting isotopes are not water soluble (less than 1% of the alpha emitters are water soluble). The RPD's for total alpha were small for each distinct core on both types of sample preparations. Core 51 and 52 have similar concentrations for total alpha for the fusion.

Uranium analysis was performed on the fusion of the solid composite samples and Core 51 Segment 4L. Analyses were performed on a laser fluorometer by comparing the signal obtained for the mixture to that of the known amount of uranium added. Chloride and hydrogen ions are known interferences for uranium determination by this method. Acid, hydrogen peroxide and heat were used to destroy the interfering ions, and confirm previous results. The assays show good agreement between the sample and its duplicate for each individual core composite, but there is not good agreement between the Cores 51 and 52 (Table 5-11). Again, this observation is suggestive of layering or heterogeneity in the waste.

Table 5-11. Core Composite Uranium Isotopic Distribution.

Sample	U (222-S) ($\mu\text{g/g}$)	U (325) ($\mu\text{g/g}$)	^{235}U mass percent	^{238}U mass percent
Core 51 Result 1	32,900	NR	0.688	99.303
Core 51 Result 2	31,600	NR	0.695	99.296
Core 52 Result 1	18,200	NR	0.687	99.303
Core 52 Result 2	19,000	NR	0.686	99.304

NR = Not reported

Plutonium-239/240 analysis was performed on the fusion digestions of core composites as well as Core 51 Segment 4L. The RPD's for the analysis were within acceptance criterion ($\pm 20\%$). The concentrations of the isotopes are fairly consistent throughout the tank with Core 52 having a slightly higher concentration than Core 51 (Table 5-12).

Table 5-12. Core Composite Plutonium Concentration and Isotopic Distribution.

Sample	Pu (222-S) ($\mu\text{Ci/g}$)	Pu (325) ($\mu\text{Ci/g}$)	^{238}Pu mass percent	^{239}Pu mass percent	^{240}Pu mass percent	^{241}Pu mass percent	^{242}Pu mass percent
Core 51 R1	0.131	0.208	0.003	98.101	1.871	0.02	0.01
Core 51 R2	0.108	0.264	0.005	98.115	1.850	0.02	0.01
Core 52 R1	0.153	0.190	0.006	98.113	1.853	0.02	0.01
Core 52 R2	0.184	0.183	0.006	98.011	1.940	0.03	0.02

222-S assay date: 3/93

325 assay date: 8/93

R = Result

Americium analysis was performed on the fusion digestion of the solid composite samples. Comparisons between fusion digestion and GEA results for americium are not possible because of a high americium detection limit for GEA (because of a significantly higher concentration of Cs-137 than Am-241 in the samples). Core 52 fusion digestion ^{241}Am results match those obtained from second analysis. The results from the second run were less than ideal because the total alpha count appears to be confounded due to beta cross talk. The americium concentration is calculated using a ratio of the alpha energy analysis peaks results from both analysis runs given in the summary table. The RPD's for the cores are very good, calculated to be less than 10%. The ^{241}Am concentrations between the two cores are similar, with Core 52 having a slightly higher concentration than Core 51.

5.2.2 Beta Emitters

Total beta analysis was performed on the fusion and water digestions of the solid core composite samples. Core 51 and 52 solid composite water digestion sample did not have acid added as a preservation agent. By comparing the fusion and water digestion results in Table 5-14 it can be seen that water digestion does not dissolve all of the beta-emitting isotopes.

Total beta is an estimate of the overall activity in the sample. Because each beta isotope has a different energy, each isotope has a different detector efficiency. Total beta results from the 222-S Laboratory are based on the efficiency of the detector for Co-60. Emissions from other isotopes have lower or higher efficiencies based on their energies. Because Co-60 is lower in energy than the isotopes usually present in Hanford Site waste the total beta results are usually biased high (see Table 5-13). The following equation is used to correct for the efficiency of the detector:

$$\text{Isotope equivalent} = (\text{Isotope concentration}) \times \frac{(\text{Isotope efficiency})}{(^{60}\text{Co efficiency})}$$

where

$$\begin{aligned} {}^{60}\text{Co efficiency} &= 0.346 \mu\text{Ci/g} \\ {}^{90}\text{Sr efficiency} &= 0.490 \mu\text{Ci/g} \\ {}^{137}\text{Cs efficiency} &= 0.522 \mu\text{Ci/g} \end{aligned}$$

Strontium-90 analyses were performed on the fusion digestions of the solid composites samples as well as the segments. Most of the Sr-90 batches were counted on multiple detectors; daily calibrations correct for any variation in detector efficiencies. Results for Core 50, Segment 1R; Core 51, Segment 4L; Core 52, Segment 3L appeared unexplainably low. All results were double checked and verified. Table 5-15 gives a summary of the segment results. Core composite results are comparable between duplicates, however there is a definite difference in concentration between the cores. Core 52 has a 34.6% lower concentration than Core 51. Differences in concentration between the water and fusion sample preparations suggest ${}^{90}\text{Sr}$ is in its insoluble form.

Technetium-99, carbon-14, tritium analyses were performed on the fusion digestions or water digestions of the solid core composites. Technetium analyses were limited due to the small amount of sample. Cores 51 and 52 were similar in concentration. Core 51 and 52 fusion digestion results were good with RPD's lower than 10%.

Carbon-14 analysis was performed on the water digestions of the composite samples. A sample vial spiked with ${}^{14}\text{C}$ was used to measure sample recovery; sample results were not corrected for this recovery. All of the samples had RPD's greater than 20% but either the results were less than 10 times the DL or there was insufficient sample to perform a rerun. The concentration for ${}^{14}\text{C}$ in T-107 were low for both Cores 51 and 52. Only one result was reported for the ${}^{14}\text{C}$ analysis on the Core 51 water digestion of the solid composite sample because the other ${}^{14}\text{C}$ analysis were performed on an aliquot to which acid preservation again had been added.

Tritium was performed on the water digestion of the core composite samples. A sample from each sample point was spiked with tritium to measure chemical recovery; sample results were not corrected for this recovery. An interference was present in the sample matrix that caused poor sample recovery when using direct aliquots were taken for analysis. No sample in the batch met all of the QC criteria. An investigation has not been made into the extent or nature of the interference so its effect on the sample results has not been quantified. The RPD's for the analytes in Core 51 were slightly above the 20% acceptance criterion; while Core 52 relative percent difference was 12%. The concentrations between Core 51 and 52 agree fairly well. The results for the previous analytes are found in Table 5-12. The sum of the isotopes compared very well to the total beta results. A comparison of the average total beta and beta-emitting isotope concentrations is found in Table 5-13.

Iodine-129 analysis was only performed on the drainable liquid composite samples and on the core solid composite samples. Attempts were made to analyze the solid samples, but the method did not work on the solid samples of this matrix. Instead of being oxidized to the zero valence state, the iodine in the solid samples was oxidized to a positive valence state, probably formed ionic salts which are water soluble. The chemist did not have time to investigate why the sample matrix behaved the way it did or to develop a new procedure to quantify the ${}^{129}\text{I}$, so analysis of the solid samples could not be completed.

Table 5-13. Comparison of Average Total Beta and Beta-Emitting Isotope Concentrations.

Composite/Fusion and Water	Core 51 (Equivalent)	Core 52 (Equivalent)
	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$
Total Beta	404	257
^{89/90} Sr(+ ⁹⁰ Y in Equiv)	375	243
⁹⁹ Tc	0.0483	0.0527
¹⁴ C	< DL	0.0168
¹³⁷ Cs	21.0	16.0
Sum of Isotopes	396	259

Sum of the Beta-emitting isotopes was calculated as follows:
 $(1.42)(2)(^{90}\text{Sr}_{\text{avg}}) + 1.51 (^{137}\text{Cs}_{\text{avg}})$

5.2.3 Gamma Energy Analysis

Gamma energy analysis was performed on the fusion and water digestions of the solid core composites and segment samples. Table 5-12 gives the summary of the results obtained for the core composites, Tables 5-15 and 5-16 summarizes the results for the core segments. The replicate samples of the core composites for Cores 51 and 52 prepared by caustic fusion agree. The Cs-137 result for Core 51 Segment 2 sample appears to be 5 to 10 times higher than any other segment. Analyses were performed to confirm the previous results and they agreed with the previous values. This concentration could have originated from commingled TBP waste and suggestive of tank layering. The concentrations for cesium-137 in Core 52 contrast with Core 51; this may be lateral heterogeneity or a dependence on sampling location with respect to the inlets and outlets. Gamma energy analysis results did not detect many radionuclides above the detection limit. Potassium-40 has a concentration slightly above the detection limit for Core 52, the same can not be said for Core 51. Cesium-137 results for both water and fusion are considerably above the detection limits. The similarity in the ¹³⁷Cs results suggests it's in its water soluble form. Calculations for ¹³⁷Cs indicate it is 64-86% soluble. Because the ¹³⁷Cs is largely soluble, this behavior suggests little or no $\text{Fe}(\text{CN})_6^{4-}$ is present in the tank.

Table 5-14. Results from Radioanalytical Analyses.

Analyte	Detection Limit		Core 51 Result 1	Core 51 Result 2	Core 52 Result 1	Core 52 Result 2
	Core 51	Core 52	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$
Uranium and Transuranics						
Total Alpha.w	0.0002	0.0003	0.000544	0.000496	0.0055	0.00426
Total Alpha.f	0.0038	0.0045	.475	.471	.379	.411
Total Uranium.f ($\mu\text{g/g}$)	4.7	243	32,900	31,600	18,200	22,900
Plutonium-239/240.f (222-S) (325)	0.0042	0.0033	0.131 0.208	0.108 0.264	0.184 0.190	0.176 0.183
Americium-241.f	0.0049	0.0064	0.0115	0.0111	0.0161	0.0175
Beta Emitters						
Total Beta.w	0.0039	0.0074	16.1	16.9	11.0	9.68
Total Beta.f	0.2	2.7	392	415	240	274
Tritium-3.w	0.00027	0.0002	0.00156	0.00117	0.00104	0.00118
Carbon-14.w	0.00022	0.0002	0.000255	IS	0.000115	0.000173
Strontium-90.f	1.5	15	132	131	92.0	78.9
Technetium-99	0.0048	0.016	0.046	0.0505	0.0512	0.0543
GEA Analytes						
Potassium-40.w	0.032	0.058	< DL	< DL	< DL	< DL
Potassium-40.f	0.725	0.160	< DL	< DL	0.291	0.184
Cobalt-60.w	0.0011	0.0014	< DL	< DL	< DL	< DL
Cobalt-60.f	0.0022	0.0057	< DL	< DL	< DL	< DL
Ruthenium-103.w	0.096	0.0016	< DL	< DL	< DL	< DL
Ruthenium-103.f	0.0048	0.0187	< DL	< DL	< DL	< DL
Ruthenium/Rhodium-106.w	0.02	0.025	< DL	< DL	< DL	< DL
Ruthenium/Rhodium-106.f	0.367	0.10	< DL	< DL	< DL	< DL
Cesium-134.w	0.0013	0.0016	< DL	< DL	< DL	< DL
Cesium-134.f	0.0063	0.0218	< DL	< DL	< DL	< DL
Cesium-137.w	0.0016	0.0014	12.4	11.6	6.39	6.59
Cesium-137.f	0.0298	0.069	13.5	14.2	10.3	10.1
Europium-154.w	0.0036	0.0049	< DL	< DL	< DL	< DL
Europium-154.f	0.071	0.018	< DL	< DL	< DL	0.0688
Europium-155.w	0.0038	0.0026	< DL	< DL	< DL	< DL
Europium-155.f	0.478		< DL	< DL	< DL	< DL
Cerium/Praseodymium-144.w	0.0081	0.014	< DL	< DL	< DL	< DL
Cerium/Praseodymium-144.f	0.021	0.04	< DL	< DL	< DL	< DL
Thorium-228/Lead.w	0.0017	0.0025	< DL	< DL	< DL	< DL
Thorium-228/Lead.f	0.0293	0.0085	0.0513	< DL	< DL	< DL
Americium-241.w	0.0056	0.0055	< DL	< DL	< DL	< DL
Americium-241.f	0.213	0.028	< DL	< DL	< DL	< DL

* performed by direct analysis

IS = Insufficient Sample

Analyte.f = Fusion digestion

Analyte.a = acid digestion

Analyte.w = water digestion

< DL = below detection limit

Table 5-15. Tank T-107 Core 51 Radionuclide Analyte Trending As a Function of Depth.
($\mu\text{Ci/g}$) (Fusion prep on segments).

Segment	^{137}Cs	^{154}Eu	^{155}Eu	^{241}Am	^{60}Co
2	100	0.314	< DL	< DL	< DL
3 Upper	15.3	< DL	< DL	< DL	< DL
3 Lower	17.1	0.044	< DL	< DL	0.0376
4 Upper	17.9	< DL	< DL	< DL	< DL
4 Lower	13.6	< DL	< DL	< DL	< DL

< DL = below detection limit

Table 5-16. Tank T-107 Core 52 Radionuclide Analyte Trending As a Function of Depth ($\mu\text{Ci/g}$) (Fusion prep on segments).

Segment	^{137}Cs	^{154}Eu	^{155}Eu	^{241}Am	^{60}Co
1	10.9	1.08	0.92	0.253	< DL
2	10.3	< DL	< DL	< DL	< DL
3 Upper	7.83	< DL	< DL	< DL	< DL
3 Lower	10.7	< DL	< DL	< DL	< DL
4	IS	IS	IS	IS	IS

< DL = below detection limit

IS = Insufficient Sample

Radionuclides concentrations appear to be fairly low in the analyte trending as a function of depth. Since the concentrations are so low, not much information can be derived from this. No stratification layers are evident, except for the high ^{137}Cs concentration seen in Core 51 Segment 2. This could be indicative of a radiologically different material on top of the waste.

5.3 RHEOLOGICAL/PHYSICAL MEASUREMENTS

5.3.1 Density

As a preliminary assessment, solid bulk density estimates were calculated by dividing the weight of the solids within the segment by the estimated solids volume. For solid samples, the length measured was multiplied by 9.85 mL/in to obtain the volume. Liquid densities were estimated by observing the volume and measuring the weight of the drainable liquid in the hot cell. The volume of the liquid was determined by collecting the liquid in a jar with volume markings (every 25 mL) and estimating the volume of liquid present. Because these densities are approximate, the density measurements performed on homogenized materials are considered more accurate.

Bulk densities were performed on homogenized material from each segment. Density was performed on all segments except those which didn't have enough material (Core 50, Segments 1R and 3; Core 51, Segment 2; Core 52, Segments 1 and 4). This procedure does not apply to bulk density measurements performed in the hot cell. The bulk densities on the homogenized samples were approximately 1.5 g/ml. Several segments (Core 50, Segment 2; Core 51, Segment 3L; and Core 52 composite) produced anomalously low or high values densities which ranged from 1.2 g/mL to 1.7 g/mL. The average density calculated for all segments, including the anomalous points, was $1.51 \text{ g/mL} \pm 0.14$. The densities determined from homogenized material are found in Table 5-17.

Table 5-17. Densities for Tank T-107.

Core	Segment	Bulk Density homogenized sample (g/ml)
50	2	1.71
51	3 Upper	1.49
51	3 Lower	1.70
51	4 Upper	1.48
51	4 Lower	1.53
51	Composite	1.46
52	2	1.55
52	3 Upper	1.50
52	3 Lower	1.52
52	Composite	1.19
	Tank Average	1.56 Std. dev. ± 0.14

Std. Dev. = Standard Deviation

5.3.2 Shear Strength

The shear strength of the waste from tank T-107 was measured on unhomogenized samples. Since only one visually discernable stratum was observed in tank T-107 shear strength measurements were only performed on Core 50 Segment 2. The shear strength measurements were made at ambient temperature using a shear vane connected to a viscometer and rotated at 0.3 rpm. Shear strength (τ_s) is a semiquantitative measurement of the force required to move the sample. The shear strength of the sample was measured at four different locations. The average shear strength was 7,200 dyne/cm²; the standard deviation for these four measurements was 3,700 dyne/cm². This large variance between measurements is largely attributed to the heterogeneity of the sample. The heterogeneity of this sample is evident in the moisture content. There is an observable relationship to the moisture content: Core 50 Segment 2 has 29.8% water for an unhomogenized sample (43% for homogenized sample). The percent water does not compare with adjoining segments which have a significantly higher amount of water present. The torque on the sample was recorded as a function of time and the shear strength was calculated using the following equation.

$$\tau_s = \frac{[\% \tau/100] * S_\tau * 4.9e+05}{\frac{\pi * H_v * D_v^2}{2} + \frac{\pi * D_v^3}{6}}$$

where:

$\% \tau/100$ = the ratio of the total torque to the maximum torque of the viscometer head, measured as a percentage of the full scale on the plot of the shear stress versus time diagram (dimensionless)

S_τ = signal (reading) proportional to the torque

$4.9e+05$ = maximum torque of the viscometer head (dynes•cm)

H_v = shear vane height (1.582 cm)

D_v = shear vane diameter (0.800 cm)

Although relatively low, the shear strength of the material substantially exceeded the baseline value for the measurement system (200 dyne/cm²)

5.3.3 Shear Stress and Viscosity as Functions of Shear Rate

Shear stress measurements, as functions of shear rate were performed on the 1:1 and 3:1 (water:sample) dilution of the sample at ambient hot cell temperatures. Because of drying of the sample on the plate at elevated temperatures the shear stress of the samples as a function of shear rate could not be measured on the as received samples at 95°C.

A rheogram for a material with a yield has two sections. The first section is a straight line beginning at the origin and climbing up the ordinate. This portion of the rheogram records the material as it acts like a solid or gel. When sufficient force is applied to the material to make the gel yield, the rheogram breaks sharply to the right; recording the material's behavior as a fluid. The point on the rheogram at which the sample's behavior transfers from a solid or gel to a fluid is the yield point or yield stress. The minimum shear stress must be exceeded to initiate fluid behavior in the material. The sample is elastic under low shear conditions (less than 50 s^{-1}) and plastic under high shear conditions (greater than 300 s^{-1}). The general behavior exhibited by the waste is best described by a yield pseudoplastic model, however the system was not modeled and empirical model parameters were not determined, because the system was at the limits of detection.

The 1:1 dilution samples have significant yield points at approximately 0.75 Pa; therefore, the 1:1 dilution samples exhibit yield pseudoplastic behavior. The 3:1 dilutions exhibit essentially Newtonian behavior.

The viscosity of this sample (Core 50, Segment 2) ranges from 20 to 9 cP over a shear rate range of approximately 100 to 400 s^{-1} . The viscosity of the sample decreases with increasing shear rate. At 90°C the viscosity of the sample was slightly lower (12 to 7 cP over a shear rate range of 100 to 400 s^{-1}) than at ambient temperature. At shear rates greater than 100 s^{-1} the viscosity of the 3:1 dilution was less than or equal to 5 cP. At 95°C the viscosity of the 3:1 dilution is lower than was observed at ambient temperature (less than or equal to 3 cP at shear rates greater than 100 s^{-1}); thus, it appears that the viscosity of the samples decreases with increasing temperature.

5.3.4 Particle Size

To evaluate which potential waste retrieval method will be done for each tank a particle size analysis is performed. Particle size analysis was directed to be performed only once on tank T-107 because at the time of extrusion and sample breakdown the hot cell chemist only visually observed one stratum.

An important consideration involving the analysis of particle size is the dispersant (the liquid used to disperse and suspend the particles from the solid sample) used. The primary concern involved with the dispersant is dissolving the particles present in the waste. Any particles existing in the tank that are soluble in the dispersant will dissolve or decrease in size during the analysis. Depending on the dispersant, the particle size analysis may not represent the true particle size distribution in the tank. In the case of tank 241-T-107, water was used as the dispersing medium. If a "true" particle size distribution is required, the mother liquor (drainable liquid) of the tank should be used, if possible, because the tank particulates are already in equilibrium with the tank mother liquor.

To perform particle size analysis a small aliquot of waste is placed in a dispersant (water) to separate and suspend the particles. The waste/water matrix is placed in the device, a beam of laser light passes through the dispersant. The diameter of solid particles can be determined by the amount of light that passes through the matrix. There are two distinct ways the analyzer determines particle size; by number distribution range and volume distribution range.

Tank T-107 had only one particle size analysis performed on Core 50, Segment 2. The analysis was performed on the unhomogenized sample. The particle sizes for the Core 50 Segment 2 are a number distribution range .5 to 8 μm with a mean of 1.09 μm , and a majority of the volume distribution range .10 to 150 μm with a median of 32.97 μm . Some particles may have been greater than 150 μm but this number was the upper limit on the analyzer. Refer to Figure 5-8 and 5-9 for a graphical representation.

5.3.5 Settling Behavior

This section analyzes the settling behavior for the as-received, 1:1 and 3:1 (water:sample) dilutions. The physical properties reported here include settling rates and volume percent settled solids, and weight percent centrifuged solids. The experimental procedures used to perform these measurements were reported previously (HASM 1993).

The data from Table 5-19 indicates that the as-received sample did not settle, but a substantial amount of liquid was associated with the sample, as was observed by the vol% and wt% centrifuged solids. This conclusion is supported by the wt% solids data. A two-fold decrease in the vol% settled and centrifuged solids between each dilution and linear decrease in the slurry density as a function of dilutions is expected for insoluble solids. The decrease in the centrifuged supernate density as a function of dilution is also indicative of insoluble solids. These conclusions do not exclude the possibility that some components of the solids are soluble, but these soluble components are not the major components of the solids. The wt% dissolved solids indicate that a significant amount of salts are dissolved in the centrifuged supernate of the as-received sample, but no analysis was performed on the dilutions to correlate the amount of solids dissolved during each dilution.

Table 5-18. Particle Size in Tank T-107.

Core 50 Segment 2	Distribution Range	Mean
Number Distribution	0.5 to 8.0 μm	1.09 μm
Volume Distribution	.10 to 150 μm	32.97 μm

The 1:1 dilution for Core 50 Segment 2 reached a final volume percent settled solids behavior of 65 to 75 percent. Settling was observed throughout the 2-day period, but the majority of the settling was observed in the first 10 hours. The 3:1 dilution reached a final volume percent settled solids of approximately 32 percent. Significant settling for both of these dilutions was observed over the 48 hours, but the settling velocity of these dilutions decreased sharply over the first 8 hours and then remained essentially constant. After the drop in percent settled solids, the remainder of the suspended solids, approximately 25% or so, took up almost the rest of the time settling in a long, gradual decline, before coming to equilibrium.

5.3.6 pH

Sample pH was measured on solid segment samples, drainable liquid core composite samples, and on the field and hot cell blanks. If any pH was greater than 12.5; a hydroxide analysis was to be completed. All sample pH measurements were less than 12.

Figure 5-8. Particle Size Distribution (Volume Distribution).

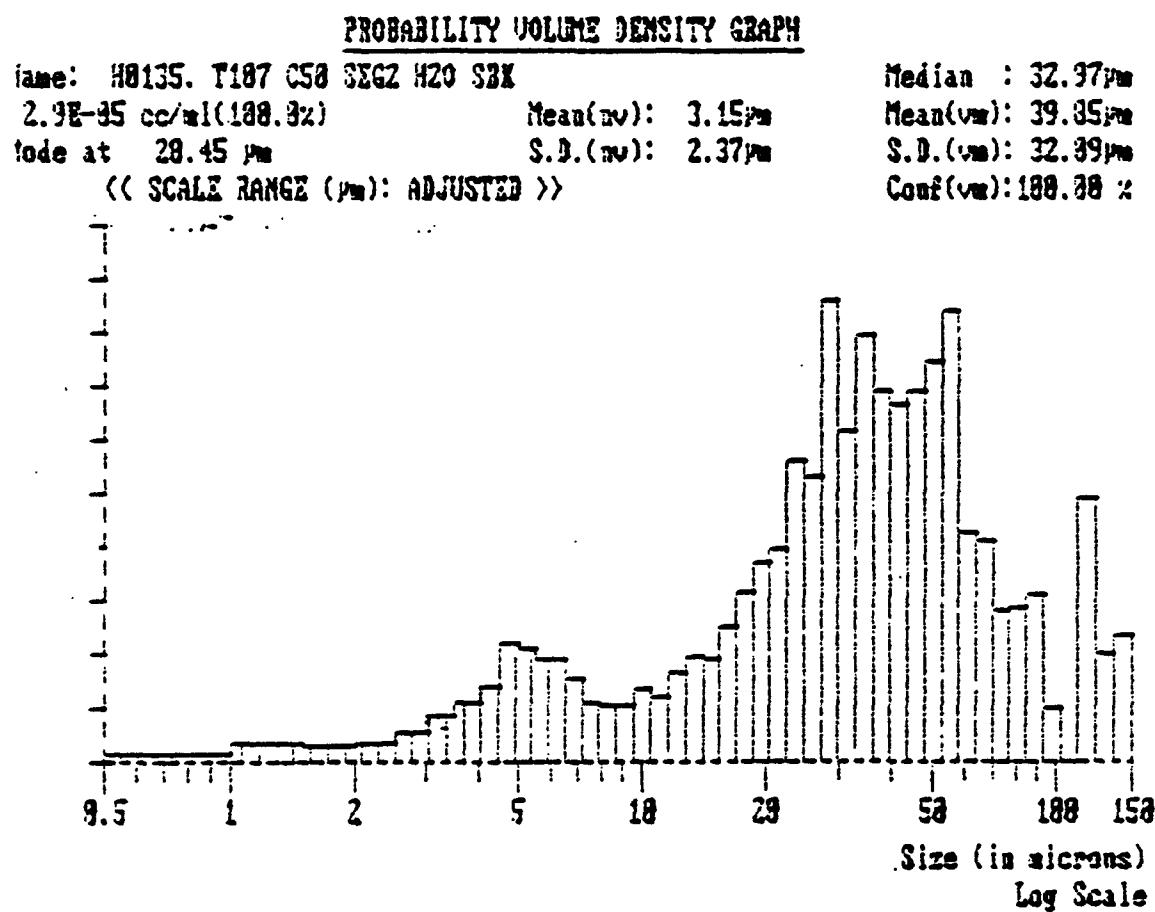


Figure 5-9. Particle Size Distribution (Number Distribution).

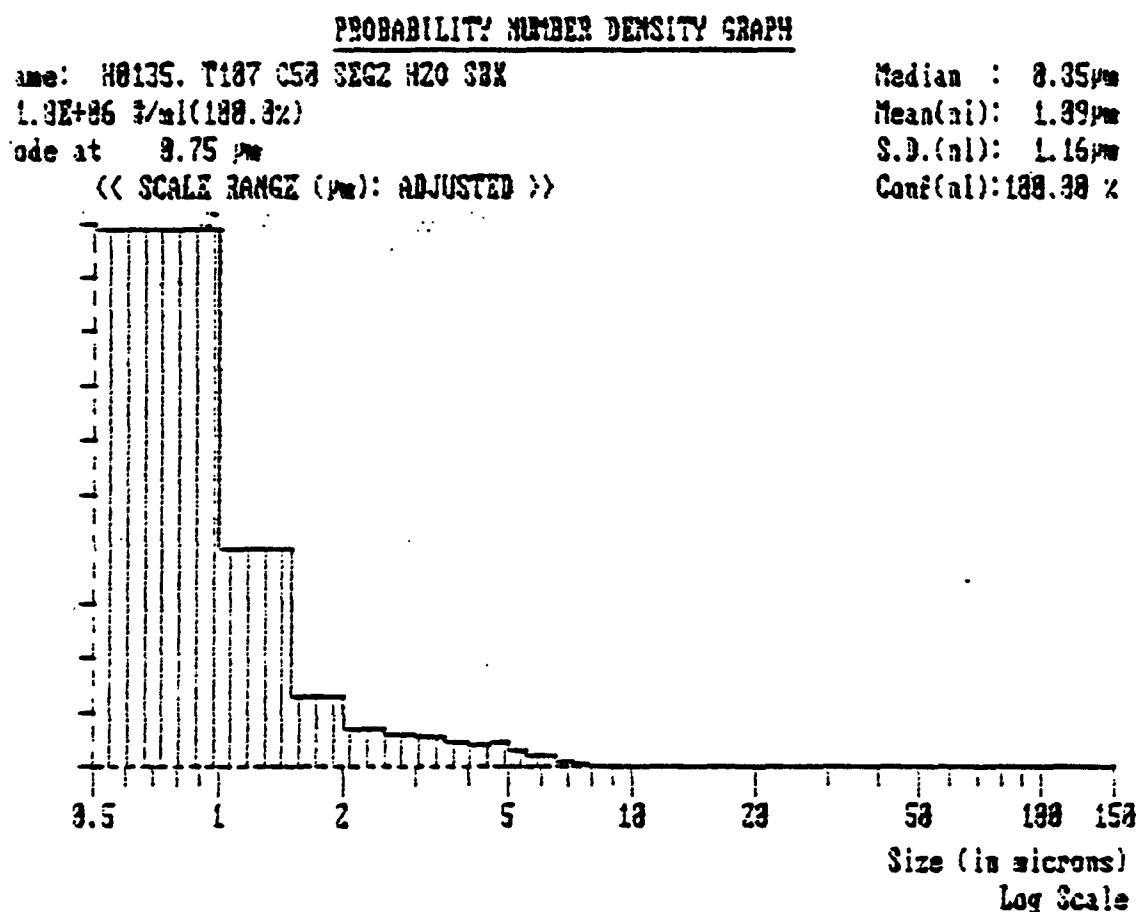


Figure 5-10. Settling Behavior (Settling Velocities) for Core 50 Segment 2.

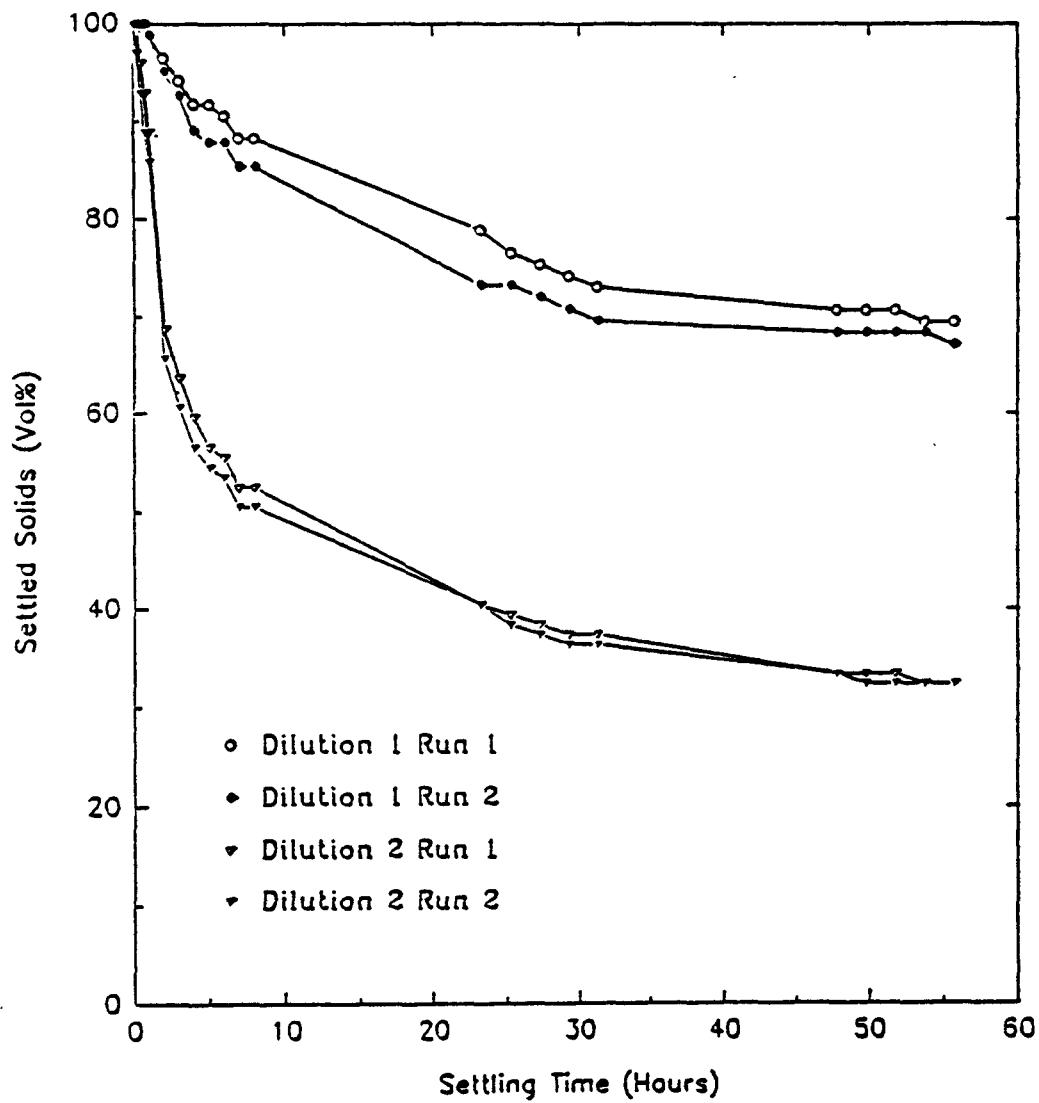


Figure 5-11. Settling Behavior (Settling Velocities) for Core 50 Segment 2.

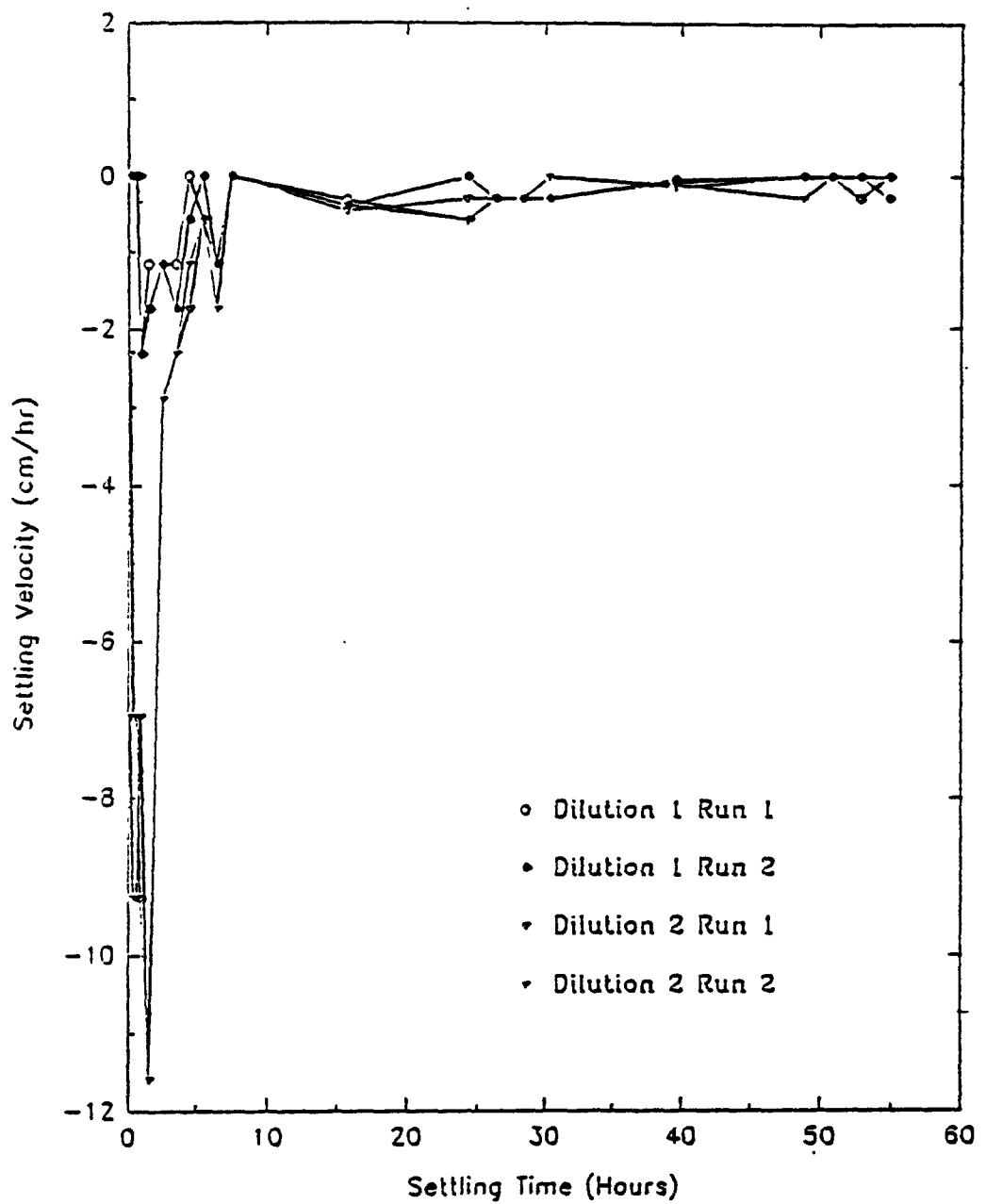


Table 5-19. Physical Properties Summary (Core 50 Segment 2).

Property	As Received	1:1	3:1
Settled Solids (vol%)	100	68	32
Centrifuged Solids (vol%)	74	36	16
Centrifuged Solids (wt%)	79	44	19
Density (g/ml)			
Sample	1.44*	1.22	1.10
Centrifuged Supernate	1.20	1.07	1.03
Centrifuged Solid	1.53	1.44	1.32
Total Solids (wt%)	47	NA	NA
Dissolved Solids (wt%)	22	NA	NA
Undissolved Solids (wt%)	25	NA	NA

* = density results obtained from 325 (PNL)

NA = Not Analyzed

Table 5-20. pH Results for Tank T-107.

Segment	pH
Core 50	
1	NS
1R	10.3
2	11.2
3	11.4
4	IS
Drainable Liquid Composite	9.6
Core 51	
1	NS
2	10.6
3U	11.4
3L	11.4
4U	11.2
4L	11.6

Table 5-20. pH Results for Tank T-107.

Segment	pH
Drainable Liquid Composite	10.7
Core 52	
1	10.5
2	11.4
3U	11.8
3L	10.9
4	IS
Drainable Liquid Composite	10.3

IS = Insufficient Sample

NS = No Sample

5.4 THERMAL ANALYSIS

Thermal analysis performed on tank T-107 waste used a Differential Scanning Calorimeter (DSC) and a Thermogravimetric Analyzer (TGA).

5.4.1 Differential Scanning Calorimeter (DSC)

A DSC is used to identify the potential for an exothermic reaction in the waste upon heating. A second purpose is to identify secondary reactions or a change in state that may occur due to an increase in temperature. DSC analysis measures the amount of heat released or absorbed by a sample while being heated at a constant rate (10 °C/min). The sample is compared to a reference sample and any temperature difference between the two is recorded as an endothermic or exothermic process. During the heating of a sample, a gas (usually air or nitrogen) is passed over the top to remove decomposition gases being released. A graph of the change in heat absorbed/evolved versus time is plotted. On these particular graphs an upward peak indicates an exothermic process while a downward peak is an endothermic process. The computer on the DSC is capable of calculating the change in heat, whether endothermic or exothermic, by integrating the area under the curve. The units are adjusted to give calories and are then divided by the mass of the sample.

If a self-sustaining exothermic reaction should occur in the waste as a result of an elevation in temperature, it would pose a safety concern. DSC assays were performed on each unhomogenized facies, every homogenized segment, homogenized half segments, and drainable liquid composites in addition to those tests done on the field and hot cell blanks (calibration and quality control).

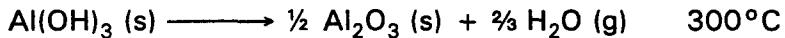
When an exotherm is found in one of the segments a duplicate sample is performed. In tank T-107, a duplicate sample was directed to be performed for each segment regardless. No exotherms attributable to the tank waste was observed. The only sample from tank T-107 to exhibit an exotherm was a separable facies from Core 50 Segment 4. In this sample, the

observation of the extruded sample noted "a flat piece of plastic or a piece of gum that had been stepped on" (Svancara 1993) and was specifically placed in a vial for DSC/TGA analysis. The sample and duplicate exhibited an exotherm beginning at 300°C with the results being 1016.4 J/g and 1541.2 J/g (243 cal/g and 368 cal/g), respectively when analyzed with air as a cover gas. The observations of the chemist stated the plastic debris was stable and not representative of the tank waste.

A couple of the upper segments produced two endotherms. One of the endotherms at approximately 100°C and the other starting at 300°C. Two suggestions are offered to explain these endotherms. The first endotherm could be attributed to the evaporation of water.



The second of the endothermic process could be caused from the dehydration of aluminum hydroxide to alumina and gaseous water.



The magnitude of the endotherms observed correspond well with the proposed mechanisms. From the reported observations there was slight marbling in the waste with light grey streaks. This light grey matter could be aluminum hydroxide which would give the second endotherm, as described above.

5.4.2 Thermogravimetric Analysis And Gravimetric Analysis

Thermogravimetric analysis was performed to determine the weight loss of a sample as a function of increasing temperature. TGA was performed on nonhomogenized facies, homogenized segments or subsegments, and drainable liquid composites. The cover gas used for the TGA measurements was air. The percent water is calculated by measuring the weight loss at 100 °C. The values produced may vary substantially as a result of the small sample size and sample heterogeneity. In Core 50, Segment 4, an anomalous percent water was noted which was attributed to the fact that the plastic material burned with the air cover gas. The TGA was therefore not measuring the water content of this sample. When the cover gas was changed to nitrogen, no loss in weight was noted.

Gravimetrically measuring the amount of solids provides more representative measurements of the water/solids content within a sample. The gravimetric method uses a larger sample aliquot than the TGA (about 1 g versus 10 to 35 mg), reducing variations caused by sample heterogeneity. The samples are heated in an oven at 102 °C until the weight measurements do not change, indicating all free water has been removed. All solid composite and homogenized segments or subsegments (except Core 50, Segments 3 and 4 and Core 52, Segment 4) were analyzed in duplicate by this method. Table 5-20 shows the weight percent water results obtained from both the TGA and gravimetric methods. For each method, the relative percent difference (RPD) between samples and duplicates was under 10% for all samples except for the Core 50, Segment 2, homogenized sample TGA analysis (RPD = 12.79%). The RPDs between the results for the two methods are shown in Table 5-21.

Table 5-21. Percent Water Results From Thermogravimetric Analysis and Gravimetric Analysis.

Sample	Thermogravimetric Analysis	Gravimetric	RPD (%)
Core 50, Segment 1R, nonhomogenized	5.76	NR	NA
Core 50, Segment 1R, homogenized	26.2	18.0	37.1
Core 50, Segment 2, nonhomogenized	29.8	NR	NA
Core 50, Segment 2, homogenized	43.0	41.5	3.6
Core 50, Segment 3, nonhomogenized	43.3	IS	NA
Core 50, Segment 4, nonhomogenized	58.1 w/air 0 w/nitrogen	IS	NA
Core 51, Segment 2, homogenized	59.3	60.2	1.2
Core 51, Segment 3U, homogenized	59.6	55.1	7.9
Core 51, Segment 3L, homogenized	54.2	52.9	2.4
Core 51, Segment 4U, homogenized	54.7	55.0	0.6
Core 51, Segment 4L, homogenized	53.1	49.5	7.0
Core 52, Segment 1, homogenized	15.2	16.7	9.4
Core 52, Segment 2, homogenized	55.5	48.5	13.5
Core 52, Segment 3U, homogenized	54.6	51.4	6.0
Core 52, Segment 3L, homogenized	52.2	53.5	2.5
Core 52, Segment 4, homogenized	53.5	IS	NA
Core 50, drainable liquid composite	95.1	95.6	0.5
Core 51, drainable liquid composite	73.7	75.3	2.1
Core 52, drainable liquid composite	82.9	86.5	4.3
Core 51, core solids composite	NR	51.9	NA
Core 52, core solids composite	NR	47.8	NA

NR = Analysis not required

IS = Insufficient sample for analysis

NA = Not applicable.

5.5 DRAINABLE LIQUID RESULTS

Table 5-21, contains a summary of the results for analyses performed on the drainable liquids for tank T-107. Each core (50, 51, and 52) has a liquid core composite associated with it. No liquid segment analysis was performed. The liquid core composites were analyzed similar to the solid material.

Table 5-22. Summary of Drainable Liquid Results. (3 pages)

Analyte	Detection Limit			Drainable Liquid Core 50	Drainable Liquid Core 51	Drainable Liquid Core 52	Water Digestion (Avg. 51 and 52)
	Core 50	Core 51	Core 52				
Cations	µg/mL	µg/mL	µg/L	µg/L	µg/L	µg/L	µg/g
Aluminum (Al)	0.51	0.51	1.28	4.26	11.5	47.8	651
Antimony (Sb)	1.52	1.52	3.80	1.92	2.52	< DL	< DL
Arsenic (As)	0.39	0.39	0.975	< DL	< DL	< DL	< DL
Beryllium (Be)	0.03	0.03	0.075	< DL	< DL	< DL	< DL
Bismuth (Bi)	0.92	0.92	2.3	< DL	< DL	17.0	243
Boron (B)	0.1	0.1	0.25	9.18	24.6	30.2	322
Cadmium (Cd)	0.07	0.07	0.175	< DL	< DL	< DL	< DL
Calcium (Ca)	0.05	0.05	0.125	4.29	4.98	3.94	271
Chromium (Cr)	0.15	0.15	0.375	45.3	273	188	211
Cerium (Ce)	1.28	1.28	3.2	< DL	< DL	< DL	< DL
Iron (Fe)	0.160	0.160	0.4	5.75	48.0	19.1	356
Lanthanum (La)	0.2	0.2	0.5	< DL	< DL	< DL	< DL
Lead (Pb)	0.78	0.78	1.95	< DL	< DL	< DL	20.2
Lithium (Li)	0.06	0.06	0.15	< DL	< DL	< DL	0.849
Magnesium (Mg)	0.03	0.03	0.075	1.24	0.411	0.393	9.83
Manganese (Mn)	0.03	0.03	0.075	0.0345	0.784	< DL	2.08
Molybdenum (Mo)	0.09	0.09	0.225	2.19	22.4	2.13	7.88
Neodymium (Nd)	0.78	0.78	1.95	1.03	0.918	< DL	< DL
Nickel (Ni)	0.15	0.15	0.375	1.77	1.61	2.79	4.49
Phosphorus (P)	0.7	0.7	1.75	790	2,030	2,590	23,700
Potassium (K)	1.48	1.48	37.3	46.7	367	138	316
Samarium (Sm)	1.04	1.04	2.60	2.07	3.66	< DL	< DL
Selenium (Se)	1.41	1.41	3.52	< DL	< DL	< DL	55.4
Silicon (Si)	0.34	0.34	850	58.5	66.3	87.2	3,950
Silver (Ag)	0.09	0.09	0.225	< DL	0.258	< DL	< DL
Sodium (Na)	0.4	0.4	1.0	14,600	95,500	51,900	108,000
Strontium (Sr)	0.03	0.03	0.075	0.140	0.170	1.81	5.53
Sulfur (S)	0.39	0.39	0.975	825	6,010	3,335	3,540
Titanium (Ti)	0.02	0.02	0.175	< DL	< DL	< DL	2.12
Thallium (Tl)	2.50	2.50	6.25	< DL	3.21	< DL	< DL
Uranium (U)	96	96	NR	95.2	588	40.6	NR
Zirconium (Zr)	0.12	0.12	0.3	0.134	0.649	1.70	6.07

Table 5-22. Summary of Drainable Liquid Results. (3 pages)

Analyte	Detection Limit			Drainable Liquid Core 50	Drainable Liquid Core 51	Drainable Liquid Core 52	Water Digestion (Avg. 51 and 52)
	Core 50	Core 51	Core 52				
Anions				$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$	$\mu\text{g/mL}$
Ammonia (NH_3)	40	80	80	42.3	83.1	221	< DL
Chloride Cl^-	0.2	0.2	1.0	196	1,340	860	540
Cyanide (CN^-)	0.4	0.4	0.4	13.5	152	39.8	68.8
Fluoride (F^-)	0.1	0.1	0.1	174	825	673	11,400
Nitrate (NO_3^-)	1.0	1.0	1.0	21,200	134,000	100,000	74,600
Nitrite (NO_2^-)	1.0	1.0	1.0	2,580	27,650	8,055	11,700
Phosphate (PO_4^{3-})	1.0	1.0	1.0	2,400	6,240	7,630	114,000
Sulfate (SO_4^{2-})	1.0	1.0	1.0	4,650	16,800	9,580	9,975
TOC	5.5	5.5	5.5	1,150	1,070	355	1,700
TIC	5	5	5	512	4,550	339	4,230
Radionuclides	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$
Americium-241	3.0e-05	5.0e-05	5.0e-05	< DL	0.000204	0.000007	< DL
Carbon-14	2.2e-06	2.2e-06	2.2e-06	0.000018	0.00038	0.0000739	< DL
Cesium-134	4.7e-04	1.4e-04	2.7e-04	< DL	< DL	< DL	< DL
Cesium-137	5.8e-04	1.4e-03	1.5e-04	1.72	18.4	5.23	9.25
Cobalt-60	4.1e-04	1.1e-03	1.2e-04	< DL	< DL	< DL	< DL
Cerium/ Praseodymium 144	1.0e-02	4.8e-02	8.5e-03	< DL	< DL	< DL	< DL
Europium-154	1.3e-03	4.0e-03	5.3e-04	< DL	< DL	< DL	< DL
Europium-155	1.3e-03	4.0e-03	2.6e-03	< DL	< DL	< DL	< DL
Iodine-129	7.5e-05	3.7e-05	3.8e-05	< DL	< DL	< DL	--
Plutonium-239/240	6.0e-05	1.3e-04	6.3e-05	< DL	< DL	< DL	0.00
Plutonium-238	9.0e-05	2.3e-04	1.1e-04	0.000032	0.0025	< DL	--
Potassium-40	1.3e-02	3.0e-02	3.4e-02	< DL	< DL	0.00373	< DL
Ruthenium-103	1.5e-03	7.2e-03	1.3e-03	< DL	< DL	< DL	< DL
Ruthenium/Rhodium- 106	2.1e-02	9.3e-02	1.6e-02	< DL	< DL	< DL	< DL
Strontium-90	7.9e-05	3.2e-05	3.2e-05	0.0108	0.118	0.0449	--
Technetium-99	1.6e-05	1.8e-05	1.8e-05	0.00805	0.0858	0.0167	--
Thorium 228/Lead	7.3e-04	6.1e-02	1.9e-04	< DL	< DL	< DL	< DL
Tritium-3 (H^3)	2.6e-05	2.3e-04	2.6e-05	0.00296	0.00695	0.000471	0.00121
Total Alpha	4.1e-04	6.5e-04	3.0e-05	0.000914	0.0166	0.000511	0.0027
Total Beta	8.2e-03	9.5e-03	1.5e-02	2.50	25.2	7.56	13.42

Table 5-22. Summary of Drainable Liquid Results. (3 pages)

Analyte	Detection Limit			Drainable Liquid Core 50	Drainable Liquid Core 51	Drainable Liquid Core 52	Water Digestion (Avg. 51 and 52)
	Core 50	Core 51	Core 52				
Physical Properties							
pH	--	--	--	9.63	10.7	10.3	N/A
Density	--	--	--	1.02	1.21	1.11	N/A
% Water	--	--	--	95.1	73.7	82.9	N/A

-- = Analyte concentration was not calculated

N/A = Not Applicable

NR = Not Reported

5.6 CHEMICAL ANALYSIS SUMMARY

For Table 5-23 the process history is found in Agnew 1994 based on the ppm found in the sludge. The drainable liquid results are an average of all the liquid core composites results. When all the results determined are less than the set detection limits, the highest detection limit is reported with a less than preceding the number. The average of Core 51 and 52 are based on the preparation method that produced the highest average, either acid or fusion digestion. For the core composite average if both results are found to be less than the detection limit a < DL is reported, the detection limits for each of the cores can be found in Tables 5-1, 5-5 and 5-12 in their respective columns. If one result is above the detection limit and one below the latter is reported as the concentration; this strategy is the same for the drainable liquid averages.

Table 5-23. Overall Data Summary and Inventory Estimates. (3 pages)

Analyte	Process History (Agnew 1994)	TRAC (Jungfleisch 1984)	Drainable Liquid Results (Avg.)	Average Core 51 and 52	Total Tank Inventory
Cations	µg/g	µg/g	µg/L	µg/g	kg
Aluminum (Al)	41,700	21.0	21.2	16,300	16,750
Antimony (Sb)	--	--	2.22	121	125
Arsenic (As)	--	--	< .975	< DL	--
Beryllium (Be)	--	--	< 0.075	< DL	--
Bismuth (Bi)	11,800	14,200	16.9	12,000	12,350
Boron (B)	--	--	21.3	13.0	13.4
Cadmium (Cd)	--	--	< 0.175	6.94	7.13
Calcium (Ca)	0	0	4.40	760	780
Chromium (Cr)	420	505	169	360	370
Cerium (Ce)	--	0	< 3.2	115	118

Table 5-23. Overall Data Summary and Inventory Estimates. (3 pages)

Analyte	Process History (Agnew 1994)	TRAC (Jungfleisch 1984)	Drainable Liquid Results (Avg.)	Average Core 51 and 52	Total Tank Inventory
Cations (continued)	µg/g	µg/g	µg/L	µg/g	kg
Iron (Fe)	10,000	11,478	24.3	29,200	30,000
Lanthanum (La)	0	0	< 0.5	< DL	--
Lead (Pb)	0	1.40e-11	< 1.95	649	667
Lithium (Li)	--	--	< 0.15	4.52	4.65
Magnesium (Mg)	--	--	0.681	226	230
Manganese (Mn)	0	0	0.064	213	230
Mercury (Hg)	--	--	--	--	--
Molybdenum (Mo)	--	--	8.9	8.92	9.17
Neodymium (Nd)	--	--	0.977	73.3	75.1
Nickel (Ni)	0	0	6.87	267*	275
Phosphorus (P)	--	--	1,800	32,100	33,000
Potassium (K)	0	0	184	234*	240
Samarium (Sm)	--	--	3.13	285	293
Selenium (Se)	--	--	< 3.52	< DL	--
Silicon (Si)	4,300	0	70.7	6,050	6,200
Silver (Ag)	--	0	0.258	< DL	--
Sodium (Na)	95,000	179	54,000	130,250*	134,000
Strontium (Sr)	--	0	0.707	878	900
Sulfur (S)	--	--	3,390	3,275	3,400
Titanium (Ti)	--	--	< 0.175	0	--
Thallium (Tl)	--	--	3.21	< DL	--
Uranium (U)	21,000	--	241	26,400	27,000
Zirconium (Zr)	1,500	1,800	0.826	93.2	95.8
Anions			µg/mL		
Ammonia (NH ₃)	--	--	122	< DL	--
Chloride Cl ⁻	7.0	0	799	540	560
Cyanide (CN ⁻)	--	0	68.4	69	70
Fluoride (F ⁻)	38,231	0	557	11,400	12,000
Nitrate (NO ₃ ⁻)	12,500	1.2e-08	85,233	74,500	77,000

Table 5-23. Overall Data Summary and Inventory Estimates. (3 pages)

Analyte	Process History (Agnew 1994)	TRAC (Jungfleisch 1984)	Drainable Liquid Results (Avg.)	Average Core 51 and 52	Total Tank Inventory
Anions (continued)			µg/mL		
Nitrite (NO ₂ ⁻)	4,000	0	12,700	11,700	12,000
Phosphate (PO ₄ ³⁻)	520,000	--	5,423	113,500	117,000
Sulfate (SO ₄ ²⁻)	25,800	0.28	10,300	9,980	10,250
TOC	--	--	855	963	990
TIC	103	350	1,800	2,970	3,050
Radionuclides	µCi/g	µCi/g	µCi/mL	µCi/g	Ci
Americium-241	--	0.0019	0.000115	0.0141	0.0145
Carbon-14	--	0	0.000113	0.000181	0.00
Cesium-134	--	--	< 0.00027	< DL	--
Cesium-137	4.58	0	8.4	12.0	12.3
Cobalt-60	--	--	< 0.0012	< DL	--
Cerium/Praseodymium 144	--	--	< 0.00484	< DL	--
Europium-154	--	--	< 0.00411	< DL	--
Europium-155	--	--	< 0.0149	< DL	--
Iodine-129	--	0	< 0.000079	NA	--
Plutonium-239/240	0.40	0.053	< 0.0114	0.131	0.13
Plutonium-238	--	8.8e-05	< 0.0025	0.144	0.15
Potassium-40	0	--	0.00373	0.238	0.24
Ruthenium-103	--	--	< 7.26	< DL	--
Ruthenium/Rhodium-106	--	5.8e-09	< 0.0942	< DL	--
Strontium-90	6.27	38.9	0.038	108	110
Technetium-99	--	0	0.0225	--	--
Thorium 228/Lead	--	--	< 0.0021	< DL	--
Tritium-3 (H ³)	--	--	0.00257	0.00124	0.00
Total Alpha	--	--	0.006	0.434	0.45
Total Beta	--	--	11.75	330	340

Table 5-23. Overall Data Summary and Inventory Estimates. (3 pages)

Analyte	Process History (Agnew 1994)	TRAC (Jungfleisch 1984)	Drainable Liquid Results (Avg.)	Average Core 51 and 52	Total Tank Inventory
Physical Properties	Process	TRAC	Drainable	Average	
pH	12.0		10.2	11.4	N/A
Density	1.59	--	1.09	1.51	N/A
% Water	60.0	--	84	56.0	N/A

-- = Value was not determined

N/A = not applicable

6.0 INTERPRETATION OF ANALYTICAL RESULTS

6.1 TANK 241-T-107 WASTE PROFILE

From the time tank T-107 went into service in 1944 it received four major waste types. The waste types, in chronological order, were as follows:

- Bismuth Phosphate First Cycle Decontamination Waste (1C)
- Tributyl Phosphate Waste (TBP)
- Cladding Waste (CW)
- Ion Exchange Waste (IX)

By taking into account the types of waste and the order in which they were added a quantitative waste profile and quantitative tank inventory can be developed.

The approach taken to identify the waste profile was to examine the available segment level assays for analytes or characteristics distinct to the waste types that were disposed in the tank. That information was combined with what is known regarding the tanks process history. The first waste type placed in the tank by a riser inlet was bismuth phosphate first cycle decontamination waste. This process waste stream has been recorded as having high concentrations of bismuth, phosphate, aluminum, and fluoride. Aluminum and fluoride are due to SiF_6^- being added to enhance the efficiency of the BiPO_4 process and 1C waste was comprised of 24% aluminum cladding waste.

Tank T-107 then received TBP waste. The waste is from the tributyl phosphate uranium extraction process at U Plant. This process was designed for the recovery of uranium metal waste generated by BiPO_4 process.

Cladding waste produced at the PUREX plant was then added to tank T-107. This waste was produced by the dissolution of zircaloy or aluminum fuel cladding waste. The predominant constituents are aluminum, zirconium, fluoride, nitrates, phosphates, sulfates, and some uranium.

The last waste type was ion exchange waste from the cesium recovery process at B Plant. The major analytes were nitrate and hydroxides. Ion exchange waste was expected to be mostly dilute aqueous solutions and was not expected to precipitate solids.

Nonradioactive chemicals have been added to the tank while varying amounts of waste and heat-producing radionuclides have been removed at various times during its active processing history. During the 1950's ferrocyanide compounds were used to scavenge ^{137}Cs and other soluble radionuclides. Tank T-107 contains a small amount of waste produced from the U-plant scavenging process. The U-plant scavenging process waste has a lower ferrocyanide concentration than would be found in an In-Farm scavenged tank.

6.1.1 Review of the Analyte Profile

The following conclusions are drawn from review of the available composite and segment analyses presented in Section 5 and historical data in Section 2.

Core 50

Interpretation of the results suggested several distinct types of materials. Because of a limited amount of sample and poor sample recovery no core composite was prepared for this core, and another core was pulled to compensate for Core 50. The analyses on the individual segments reveal high concentrations of aluminum, bismuth, and phosphorus. These results are expected, and the analytes observed generally resemble the composition of CW, TBP, and 1C wastes. The DSC traces for Segment 4 show an exotherm beginning around 300°C. This exothermic region was attributed to a plastic artifact that was commingled with the waste in the last segment of Core 50. Further analysis of the plastic inclusion with different carrier gases show the plastic piece to be anomalous and not representative tank waste. After examining the trends of the segments, all analytes show a slight drop in concentration toward the middle of core (vertically), with the exception of Segment 2, where the core was high in aluminum. This observation is likely because of the high aluminum concentration found in the cladding waste added to the tank late in its service life. The last segment of the core was not recovered, therefore a conclusion cannot be reached whether the indicator analytes of the 1C waste were present, as found in the other two cores. The change in analyte concentration as a function of depth of the core observed in the fusion results is confirmed with the separate acid analysis results from the homogenization tests.

Core 51

Core 51 contained sufficient sample to prepare a core composite. The trends from the composite again indicate high bulk concentrations of bismuth, phosphorus, and aluminum. This behavior is expected from the 1C/CW and CW effluent streams. The solubility of aluminum is lower than expected because of anticipated presence of Al(OH)_3 . The other major constituents that are found in T-107 do not behave as expected. Several trace analytes such as boron, lead, lithium, molybdenum, titanium, and thallium produce erratic results with respect to the three different preparation methods. The process history for the waste streams do not indicate a large amount of these analytes present. Therefore, the concentrations would be low and erratic results were not unexpected.

Following the trending for bismuth and phosphorus, the concentrations increase toward the bottom of the tank. Segment 4L contains the highest concentration of both bismuth and phosphate. These high concentrations are expected at the bottom of the tank, since the first waste type added to tank T-107 was 1C waste from the early bismuth phosphate process. Although, the aluminum concentration at the bottom of the tank is not the highest observed, the concentration generally increased toward the bottom of the tank. This can also be attributed to the 1C waste stream which contained 24% aluminum cladding waste. The high concentrations seen in Segment 2 can be attributed to the CW produced from the dissolution of aluminum cladding added late in tank T-107's service life.

The results from anion analysis reveal high overall concentrations of fluoride, nitrate, nitrite, phosphate, and sulfate. The results indicate low concentrations of chloride in the waste. This is not surprising since chloride is not found in any of the waste streams added to T-107. The presence of the anions indicates notable quantities of water soluble compounds.

Assay results related to the safety concerns for total organic carbon and cyanide/ferrocyanide can be addressed. Total organic carbon (TOC) values for Core 51 are low, and fall below the established safety criteria. Comparing the results obtained from both laboratories, the 222-S results are approximately 3 times higher than those from 325 Laboratory. Even so, by taking the higher result as a basis, the dry weight organics percentage is 0.3%, which is well below the safety criterion. Total cyanide results (from water leach of the sample) for Core 51 are higher than Core 52. Converting the total cyanide to ferrocyanide and calculating the dry weight percent for that analyte gives a result that is again below the safety limits. The results for Core 51 are .07 wt% ferrocyanide (dry); substantially lower than the established safety criteria (8 wt%, dry) for ferrocyanide concentration.

Analyses for radionuclides were performed on the core composite and segments. The results indicate all of the radionuclides analyzed by GEA, except ^{137}Cs , are below the detection limit. Cesium-137 prepared by water digestion produced an average of 9.2 $\mu\text{Ci/g}$ of activity, while the fusion digestion produced an average of 12.0 $\mu\text{Ci/g}$. Examining the GEA results of the segments again only ^{137}Cs produced any significant amount of activity, the remaining analytes are below the detection limit of the instrument. Strontium concentrations range between 250 and 400 $\mu\text{Ci/g}$. Comparisons of the results between water and fusion digestion results indicate mostly soluble cesium and mostly insoluble strontium compounds.

Core 52

The overall high concentrations of aluminum, bismuth, phosphorus, sodium, and silicon are again in good agreement with the historical records. Aluminum, Bi, and P are found in abundance in the waste matrix, and the concentrations are strongly indicative of 1C and CW discharges. Concentrations for core composite analysis are in general higher for Core 52 than Core 51. Aluminum concentrations for Core 52 are higher than Core 51 for all three preparation types. A majority of the duplicates for Core 52 are not similar and produce high RPD's. The addition of the different types of waste onto one another can be observed by the changing concentration over the depth of the tank. By inspecting the trending of the analytes by depth, again toward the upper portion of the waste, high concentrations of aluminum were found and attributed to the TBP/CW waste added to the tank late in its service life. The aluminum concentration drops slightly toward the middle of the core only to increase toward the bottom. The first type of waste added, 1C waste with 24% aluminum cladding, could be responsible for this increase in concentration. Bismuth concentration slowly increases as one goes further down into the waste and this can also be attributed to the 1C waste. Phosphorus concentrations vary as a function of depth for both Cores 50 and 51, this trend does fit well historically. Core 52, however does not have the same variations in the tank, this could be due to the location of the sampling riser with respect to the inlet of the tank.

Examining anion concentrations provides considerable information. High concentrations of fluoride are noted; however, this observation is not surprising due to the presence of ammonium fluoride used in cladding waste and SiF_6^{2-} from 1C waste. Low chloride concentrations are also to be expected because of the lack of this anion in all Hanford waste streams. Nitrate and nitrite were reported in all types of waste therefore not considered significant indicator ions (although substantial changes between segments or cores can be suggestive). Cyanide concentrations are low for core 52 (average 45.9 $\mu\text{g/g}$). By taking this concentration of cyanide and converting it to ferrocyanide ($\text{Fe}(\text{CN}_6)^{4-}$) on a dry basis this converts to .028 wt%, dry. This value is two orders of magnitude below the established safety criterion of 8 wt% on a zero free water basis (dry). Examination of the segment analysis results for cyanide reveals Segment 2 as containing the highest concentration of cyanide, 0.047 wt% dry. This concentration of ferrocyanide is extremely low, and is far below the threshold of concern established in the Ferrocyanide DQO (Meacham et al. 1994)

The first segment of the core had very little water (15.2%). The percentage of water suggests a formation of a crust. The high concentrations of aluminum seen in the first segments as well as the DSC scans showing an endothermic region around 100°C and 300°C further suggests the formation of a crust or regional anomaly on top of the waste under riser 3. The total organic carbon analysis indicate low (small) amounts of residual organics in the waste, producing a dry weight percent of 0.38. This two observations affirm the lack of an observable exotherm representative of the tank waste.

The only radionuclide found to be routinely over the detection limit throughout the tank was cesium-137 found in both the water and fusion digestion results. Segment one actually registered concentrations higher than the detection limits for several of the analytes. Cesium-137 concentrations appear to be consistent throughout the tank with the exception of the significant drop in concentration seen between Core 51 Segment 2 and Segment 3U. Tank T-107 has slightly lower ^{137}Cs concentrations toward the bottom of the tank. Comparing the water digestion results with the fusion results indicates most of the Cesium-137 is in water soluble forms. Cesium-137 in ferrocyanide sludges is highly insoluble, because Cesium-137 is found to be largely water soluble, the cyanide inference correct. Americium-241 can only be detected in low quantities in Segment 1. These observations parallel the historical records of the waste containing low concentrations of heat producing radionuclides. The strontium compounds found in the waste are not water soluble.

6.1.2 Entrance, Exit, and Mixing Effects on Analyte Distribution

The configuration of the waste can have a substantial impact on the distribution of waste in the tank. However, the waste entrance and exit points for the tank over its service life are not well documented, thus the spatial relationship and proximity to the sample risers is not known. There were very limited transfer lines within T farm or with the tank farms as a whole, so no highly enriched layer of radioactive material is expected to lie on top of the waste as has been observed in other tanks sampled. However, the concentrations of radionuclides is observed to be generally higher in the upper portions of the tank

As new wastes entered the tank and distributed themselves across the tank, the material under and around the tank pumpout could have been disturbed (and occasionally solids transferred) in accordance to the last in-first out principle. It is believed that the material beneath the waste inlets, the cascades and perhaps a riser, would have been disturbed initially but, over time, large stratified layers resistant to mixing would have

eventually built up. No deliberate mixing of the wastes was performed, thus where segment-level data is available, distinctions between waste types can be made. The larger particulate materials initially settled in the tank, not being as flocculent or as easily suspended as some of the other solids may have settled out initially near the inlet, providing a slight degree of separation. Perhaps this accounts for the observed lateral heterogeneity between Cores 51 and 52. It must be noted that this lateral heterogeneity is very slight, and that there may be several other factors contributing to this observation especially a highly localized heterogeneity, biasing the observations. However, because of its persistence over the broad range of analytes, it is believed to exist and is not the result of an analytical artifact. The influence of the waste inlet and outlet locations can provide potential insight to the analyte distributions and waste profiles between Cores 51 and 52.

6.2 TWRS PROGRAM ELEMENT CHARACTERIZATION SYNOPSIS

This section provides selected results obtained from core sampling for some of the most pertinent analytes for the various TWRS program elements, including Vitrification, Retrieval, Pretreatment, and Waste Tank Safety. Analytes of interest will be reported on a level of resolution commensurate with the available data and program direction. Watch List tanks will have appropriate segment or subsegment level analyses reported, while Non-Watch List tanks are analyzed on a core composite basis. Analytes of interest to multiple programs will generally only be reported in one section. Further detail can be found in the body of the report or in the data package.

6.2.1 Waste Tank Safety Program Characterization Data Summary

Criticality Safety

The criticality safety program has indicated that Pu and U isotopic analyses on each core composite and the bottom most six inches of each core is required to alleviate the concern for the potential of tank criticality. The analyses will indicate whether the fissile species have settled in a concentrated layer at the bottom of the tank.

Ferrocyanide Tanks

The characterization objectives in support of resolution of the Ferrocyanide Safety Issue are as follows:

- Determine the overall waste energetics and properties governing waste reactivity behavior in the tanks.
- Determine the spatial distribution of ^{137}Cs and ^{90}Sr .
- Determine the concentration of total organic carbon content and the speciation of organics present in the waste.
- Determine the concentrations of the ferrocyanide and nitrate/nitrite content present in the waste.

Table 6-1 shows the primary and secondary analytes required by the Ferrocyanide DQO (Meacham 1994) and their respective concentrations.

Table 6-1. Primary and Secondary Data Requirements for Ferrocyanide Tanks.

Analyte	Analytical Method ¹	Decision Threshold ²	Results ³
Total Fuel ⁴	DSC/Adiabatic Calorimetry	8 wt% (.48 MJ/kg or 115 cal/g)	No Exotherms
Moisture Content	Thermogravimetric Analysis	4/3 [Fuel - 8]	46%
Tank Temperature	Thermocouple	90°C	19°C
Cs ¹³⁷	Gamma Energy Analysis	NL	38 μ Ci/g
Sr ⁹⁰	Beta Radiochemistry	NL	250 μ Ci/g
Total Cyanide	Direct Assay	NL	187 μ g/g
Total Organic Carbon	Direct Persulfate Oxidation	NL	1,500 μ g/g
Nickel	Inductively Coupled Plasma	NL	260 μ g/g ⁵

¹ Other techniques that meet the required uncertainty are also acceptable.

² Excluding moisture and tank temperature. all decision thresholds reported on a dry basis.

³ Results reported are tank averages on a dry basis.

⁴ Calculated on a Na₂NiFe(CN)₆ energy equivalent basis.

⁵ Nickel results are those obtained from acid digestion.

6.2.2 Retrieval Program Data Summary: Physical Properties

A major objective of the characterization program is to measure the physical properties of the waste to support waste retrieval technology development. The analytical methods to determine the physical properties of the waste as it actually exists in the tank require a substantial amount of unhomogenized sample (50 to 100 g). In some cases, the limited amount of sample recovered constrains the number of analyses that can be performed. At the time of the sampling and analysis of 241-T-107, no data quality objective existed to define the scope of the analyses. However, several analytes relating specifically to physical properties were determined to be of interest to the program and are summarized here. The physical characteristics of tank waste are required to develop, and to provide a basis for validation of equipment testing using design criteria and simulated waste.

Performing the rheological/physical measurements once for each stratum of waste in a tank is believed to be sufficient to characterize the entire tank contents. Selected rheological and physical properties are presented in Table 6-2; further information regarding these analytes can be found in Section 5.3.

Table 6-2. Physical Properties Summary (Core 50 Segment 2)
Analysis performed by 325 Laboratory (PNL).

Property	As Received
Settled Solids (vol%)	100
Centrifuged Solids (vol%)	74
(wt%)	79
Density (g/ml)	
Sample	1.44
Centrifuged Supernate	1.20
Centrifuged Solid	1.53
Total Solids (wt%)	47
Dissolved Solids (wt%)	22
Undissolved Solids (wt%)	25
Shear Strength (dynes/cm ²)	7200 ± 3700
Viscosity (mPa/s)--1:1 dilution @ 29°C	20 to 9 cP
Particle Size	
--number distribution	1.09 µm ±
--volume distribution	32.97 µm ±

6.2.3 Pretreatment Program Data Summary: Bulk constituent Inventories

Programmatic decisions pertaining to the design of pretreatment and final disposal systems shall be based upon the average characteristics of the tank waste. Therefore, the majority of the laboratory analyses shall be conducted on representative core composites. However, as noted in other documentation (Bell 1993), segment, subsegment, and additional analyses will be performed, when directed. The constituent concentrations and inventories shall be calculated by either treating the core samples as random samples and averaging the results, or by using a spatial model. The calculated values will include an estimated total quantity of each selected analyte and its corresponding confidence interval based upon analytical and sampling variability. Again, no data quality objective existed to define the scope of the analyses. However, several analytes relating specifically to the most significant chemical and radiological contributors and their solubility properties were determined to be of interest to the program and are summarized here. Chemical analytes of interest are presented in Table 6-3. Trace analytes and more comprehensive (chemical and radiological) characterization information can be found in Section 5.

Table 6-3. Results for Select Analytes for Tank T-107.

Analyte	Average Result (Water Digestion) µg/g	Average Result (Fusion Digestion) µg/g	% Solubility
Calcium	270	760	35.6
Chromium	210	360	58.5
Iron	355	29,175	1.2
Manganese	2.1	213	0.97
Sodium	107,900	117,250	92.0
Bismuth	243	11,997	2.0
Lanthanum	< DL	< DL	--
Silicon	3,945	6,057	65.1
Uranium	Not Measured	25,425	--
Zirconium	5.37	93.2	5.8
Phosphate	23,725	32,075	73.9
Sulfate	3,540	3,275	108.1
Nitrate	74,550	--	--
Fluoride	11,400	--	--
Total Organic Carbon	1,698*	--	--
Radionuclides	µCi/g	µCi/g	
⁹⁰ Sr	--	108	--
¹³⁷ Cs	9.25	12.03	76.9
^{239/240} Pu	--	0.181	--
²⁴¹ Am	--	--	--

% Solubility = (Water/Fusion) x 100

* results obtained from Coulometric method

6.2.4 Waste Vitrification Program Data Summary

The final disposal option for Hanford-Site wastes has been determined to be vitrification after partitioning into low-level and high-level fractions. This program has characterization needs in addition to those described for core sampling. The vitrification process will be performed after the solids have been pretreated. Therefore, the core sample information will provide preliminary bounding design conditions for the vitrification plant. Further characterization for technology development and regulatory compliance will be necessary on the pretreated waste that will be fed to the vitrification plant. Although the data requirements for this option are not formally defined, the analytical requirements for the previous Hanford Waste Vitrification program generally are applicable and are identified in the *Hanford Waste Vitrification Plant Feed Characterization Requirements* Revision 4, (Wagner 1992). These requirements are quite similar to the pretreatment program requirements, and therefore are presented together in this section (see Table 6-2). For more specific information on a particular analyte not giving in this table, consult the data package (Svancara 1993) or the appropriate table in Section 5.

The analytical program for vitrification not only entails determining whether a waste type is suitable for disposal as glass, but also includes determining the physical and chemical characteristics of the glass for process control purposes and to ensure regulatory compliance (see Table 6-4). Sampling and analysis plans will be developed on an individual basis for each tank or process batch. The characterization needs for these efforts include analyses for metals, water-soluble anions, radionuclides, semi-volatile organics, and rheological, and physical testing for both the feed and vitrified product. Tank T-107 Two selected groups of analytes are presented in this summary; one provides a set of analytes of interest to the vitrification process stream, the other are analytes of interest to the regulatory permitting of such a facility. Further characterization efforts in support of design of retrieval, pretreatment, and final disposal systems requiring a data quality objective for early feed tanks are as follows:

- Provide extensive characterization of the chemical and radiological contents of the waste (solids and supernate) as it currently exists in the tanks to support processibility assessments and to verify whether the composition variability study envelope coverage for key analytes is adequate.
- Estimate of the waste fraction that will remain after sludge wash pretreatment and estimate the feeds for the low-level and high-level streams for vitrification.
- Simulate sludge washing pretreatment on the waste material. This will provide a detailed understanding of the sludge wash process and obtain empirical data on soluble species removal.
- Determine the physical and rheological properties of the waste before and after simulated sludge washing to support the design of a waste retrieval system.
- Provide a supply of sludge washed material to be used as feed material for a laboratory scale vitrification.
- Satisfy the general characterization requirements for physical, chemical, and radiological analytes.

Table 6-4. Waste Vitrification Process Stream
Analytes of Concern.

Analyte	Core 51 Composite ($\mu\text{g/g}$)	Core 52 Composite ($\mu\text{g/g}$)
Phosphate (PO_4^{3-})	94,500	132,500
Fluoride (F^-)	9,200	13,600
Chloride (Cl^-)	682	399
TOC	1,435	1,960
Total Oxides	NA	NA

NA = Not Analyzed

Table 6-5. Waste Vitrification Regulatory Operation
Analytes of Concern.

Analyte	Core 51 Composite ($\mu\text{g/g}$)	Core 52 Composite ($\mu\text{g/g}$)
Mercury (Hg)	< DL	< DL
Lead (Pb)	1,105*	488*
Chromium (Cr(VI))	< DL	< DL
Analyte	Core 51 Composite ($\mu\text{Ci/g}$)	Core 52 Composite ($\mu\text{Ci/g}$)
Carbon-14 (^{14}C)	0.000255	0.000662
Technetium-99 (^{99}Tc)	0.483	0.0528
Tritium (^{3}H)	0.00137	0.00111

* Acid Results

7.0 QUANTITATIVE/STATISTICAL INTERPRETATION OF THE DATA SINGLE-SHELL TANK 241-T-107

This section contains the results of the statistical analysis of data from three core samples obtained from single-shell tank (SST) 241-T-107 (T-107). Four specific topics are addressed in this report and are summarized below.

Section 7.1 contains mean concentration estimates of analytes found in T-107. The estimates of "error" associated with the concentration estimates are given as 95% confidence intervals (CI) on the mean. The results given are based upon three types of samples; e.g. core composite samples, core segment samples and drainable liquid samples.

Section 7.2 contains estimates of the spatial variability (variability between cores and between segments) and the analytical error (variability between a sample and duplicate analyses). Statistical tests were performed to test the hypothesis that the between cores and between segments spatial variability is zero (i.e., T-107 waste is homogeneous). The results of the tests indicate that, based upon the core composite data the tank waste is horizontally homogeneous, based upon the core segment data the waste is vertically heterogeneous and horizontally homogeneous, and based upon the drainable liquid data the liquid is heterogeneous.

Section 7.3 contains the results of the application of multiple comparison methods to core composite and segment data. These comparisons are based upon the analytical error only. This section contains graphical comparisons between the mean concentrations of analytes between the core samples and between segments within the core samples. For the drainable liquid data there is also a graphical comparison between core samples.

Section 7.4 contains the results of a statistical test conducted to determine the Process and Analytical Laboratories (222-S) ability to homogenize solid core segments. For 17 out of 18 analytes, the variability between sub-samples taken from different locations could not be distinguished from zero. Based on the results of this statistical test, it is generally concluded that the 222-S Laboratories can satisfactorily homogenize core segments.

Appendix A contains the analyte concentration data given in the data package (Svancara, 1993). The core composite sample results are contained in Table A-1. The corresponding segment data for Cores 50, 51, and 52 are given in Tables A-2, A-3, and A-4 respectively. The drainable liquid data is in Table A-5. The data from the homogenization test is given in Table A-6. The ratios of the mean of each sample and duplicate divided by the detection limit for that pair are also included in Tables A-1 to A-6.

Summary statistics were calculated for analytes with concentrations greater than 10 times their detection limits (DL). Analytical Evaluation and Reporting personnel, within the TWRS Information Management Systems, identified a list of critical analytes that are exception to this rule. Table 7-1 contains this critical list of analytes. Summary statistics were calculated for the analytes from this special list if the concentrations were greater than 3 times their DL. Analyte sample results with concentrations less than the DL were not used.

For a number of analytes, the concentrations in some samples were greater than a particular limit (3 or 10 times the DL) while other samples were less than that limit. In these cases, the summary statistics were calculated using all of the data whether it was above or below the particular limit (3 or 10 DL). The above rules do not apply to alpha or beta/gamma counting methods. The ratios (Mean/DL) reported in Tables B-1 to B-6 are provided to show the magnitude of the analyte concentrations relative to the DL.

Table 7-1. Special Analyte List.

Aluminum	Nitrate
Bismuth	Nitrite
Calcium	Phosphate
Chromium	Carbonate
Iron	Fluoride
Silicon	Chloride
Sodium	Total Organic Carbon
Zirconium	Cyanide

7.1 MEAN CONCENTRATION ESTIMATES

One of the tasks outlined in the Tank Waste Characterization Plan (Bell 1993), is to estimate the constituent inventories in the waste. The inventories are estimated by computing mean concentrations and 95% CIs on the mean concentrations for each constituent. The estimate of the inventory and CI on the inventory of an analyte in the tank can be calculated by multiplying the corresponding mean concentration estimates and CI by the volume of waste in the tank.

Three types of constituent inventories are given in this section. The first inventory, given in Table 7-2, is based upon the core composite data. Table B-1 contains the core composite data used to compute the mean concentration estimates and the CIs. The second inventory, given in Table 7-3, is based upon the core segment data. Table B-2 contains the data used to compute these concentration estimates. The third inventory, given in Table 7-4, is based upon the results from a chemical analysis of the drainable liquid. Table B-3 contains the drainable liquid data.

7.1.1 Statistical Methods

The concentration estimates of the analytes in the waste are given in the form of 95% CIs on the mean concentration. It is assumed that each sample and its duplicate are analyzed independently. The two analytical results are used to estimate the analytical measurement error. Due to the hierarchical structure of the data, the analytical measurement error alone is not the appropriate error term to use in computing the CIs. A linear combination of the analytical measurement variance and the spatial variance is the appropriate variance of the mean for the CIs.

Table 7-2. Core Composite Data Concentration Estimate Statistics. (3 pages)
 Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$

Analyte	\bar{y}	$\sigma^2(\bar{y})$	df	95% LL	95% UL
ICP.a.Ag	7.37E+00	NA	1	NA	NA
ICP.a.Al	1.44E+04	4.19E+08	1	0.00	2.74E+05
ICP.a.B*	1.70E+01	2.68E+02	1	0.00	2.25E+02
ICP.a.Bi	1.09E+04	3.35E+07	1	0.00	8.44E+04
ICP.a.Ca	7.23E+02	6.76E+04	1	0.00	4.03E+03
ICP.a.Cd*	6.40E+00	2.64E+00	1	0.00	2.70E+01
ICP.a.Cr	3.54E+02	3.19E+03	1	0.00	1.07E+03
ICP.a.Fe	3.15E+04	1.12E+07	1	0.00	7.40E+04
ICP.a.K	2.34E+02	3.42E+02	1	0.00	4.69E+02
ICP.a.Li*	5.48E+00	8.67E+00	1	0.00	4.29E+01
ICP.a.Mg	2.14E+02	9.41E+03	1	0.00	1.45E+03
ICP.a.Mn	2.22E+02	3.61E+02	1	0.00	4.63E+02
ICP.a.Na	1.30E+05	1.56E+08	1	0.00	2.89E+05
ICP.a.Nd*	7.29E+01	5.21E+03	1	0.00	9.90E+02
ICP.a.Ni	2.92E+02	6.25E+02	1	0.00	6.10E+02
ICP.a.P	3.00E+04	4.90E+05	1	2.11E+04	3.89E+04
ICP.a.Pb	7.96E+02	3.81E+05	1	0.00	8.64E+03
ICP.a.S	3.05E+03	1.06E+06	1	0.00	1.61E+04
ICP.a.Si	8.75E+02	2.34E+06	1	0.00	2.03E+04
ICP.a.Sm	2.85E+02	1.28E+05	1	0.00	4.84E+03
ICP.a.Sr	9.62E+02	3.09E+05	1	0.00	8.02E+03
ICP.a.Zr	7.16E+01	9.01E+03	1	0.00	1.28E+03
ICP.w.Al	6.51E+02	1.10E+05	1	0.00	4.86E+03
ICP.w.B	3.26E+02	3.86E+05	1	0.00	8.22E+03
ICP.w.Bi	2.43E+02	8.84E+04	1	0.00	4.02E+03
ICP.w.Ca	2.71E+02	1.68E+05	1	0.00	5.48E+03
ICP.w.Cr	2.11E+02	6.00E+02	1	0.00	5.22E+02
ICP.w.Fe	3.56E+02	2.79E+04	1	0.00	2.48E+03
ICP.w.K	3.16E+02	7.29E+04	1	0.00	3.75E+03
ICP.w.Mg	9.83E+00	2.72E-02	1	7.74E+00	1.19E+01
ICP.w.Mo*	7.88E+00	2.66E+00	1	0.00	2.86E+01
ICP.w.Na	1.08E+05	2.72E+09	1	0.00	7.71E+05
ICP.w.S	3.54E+03	7.40E+05	1	0.00	1.45E+04
ICP.w.Se*	6.67E+01	7.73E+02	1	0.00	4.20E+02
ICP.w.Sr*	5.53E+00	6.40E-03	1	4.51E+00	6.55E+00
ICP.w.Zr*	6.07E+00	5.64E+01	1	0.00	1.02E+02

Table 7-2. Core Composite Data Concentration Estimate Statistics. (3 pages)
 Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$

Analyte	\bar{y}	$\sigma^2(\bar{y})$	df	95% LL	95% UL
ICP.f.Al	1.63E+04	4.50E+08	1	0.00	2.86E+05
ICP.f.Bi	1.20E+04	5.34E+07	1	0.00	1.05E+05
ICP.f.Ca	7.61E+02	1.44E+03	1	2.78E+02	1.24E+03
ICP.f.Cr	3.60E+02	1.00E+02	1	2.33E+02	4.87E+02
ICP.f.Fe	2.92E+04	2.97E+07	1	0.00	9.84E+04
ICP.f.Mg	2.26E+02	1.10E+02	1	9.28E+01	3.60E+02
ICP.f.Mn	2.13E+02	2.26E+03	1	0.00	8.17E+02
ICP.f.Na	1.17E+05	1.32E+08	1	0.00	2.63E+05
ICP.f.Ni	2.64E+03	1.11E+06	1	0.00	1.60E+04
ICP.f.P	3.21E+04	2.72E+06	1	1.11E+04	5.30E+04
ICP.f.Pb	6.49E+02	2.42E+04	1	0.00	2.62E+03
ICP.f.S	3.28E+03	3.36E+05	1	0.00	1.06E+04
ICP.f.Si	6.06E+03	5.69E+06	1	0.00	3.64E+04
ICP.f.Sr	8.78E+02	2.30E+04	1	0.00	2.80E+03
ICP.f.Zr	9.32E+01	1.19E+03	1	0.00	5.31E+02
TDSOLID(wt%)	3.95E-01	1.00E-04	1	2.68E-01	5.22E-01
RSWT%	2.87E+01	3.08E+01	1	0.00	9.92E+01
CN ⁻	6.88E+01	2.10E+03	1	0.00	6.51E+02
IC.F ⁻	1.14E+04	1.91E+07	1	0.00	6.70E+04
IC.Cl ⁻	5.41E+02	8.01E+04	1	0.00	4.14E+03
IC.NO ₂ ⁻	1.17E+04	5.13E+07	1	0.00	1.03E+05
IC.NO ₃ ⁻	7.45E+04	1.34E+09	1	0.00	5.39E+05
IC.PO ₄ ³⁻	1.14E+05	1.44E+09	1	0.00	5.96E+05
IC.SO ₄ ²⁻	9.89E+03	3.06E+07	1	0.00	8.01E+04
NO ₂ ⁻	1.11E+04	3.94E+07	1	0.00	9.08E+04
TIC	4.23E+03	8.41E+06	1	0.00	4.11E+04
TOC	1.70E+03	2.76E+05	1	0.00	8.37E+03
AT	2.70E-03	1.90E-05	1	0.00	5.81E-02
TB	1.34E+01	3.79E+01	1	0.00	9.17E+01
¹⁴ C	1.81E-04	8.21E-09	1	0.00	1.33E-03
³ H	1.24E-03	6.50E-08	1	0.00	4.48E-03
GEA.Cs-137	9.25E+00	3.04E+01	1	0.00	7.93E+01
RS* GEA.Cs-137	1.20E+01	1.18E+02	1	0.00	1.50E+02
RS GEA.Co-60	1.85E-02	4.41E-04	1	0.00	2.85E-01
RS GEA.Eu-154	1.33E-01	9.00E-06	1	9.49E-02	1.71E-01
RS GEA.K-40	3.76E-02	4.27E-07	1	2.93E-02	4.59E-02

Table 7-2. Core Composite Data Concentration Estimate Statistics. (3 pages)
 Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$

Analyte	\bar{y}	$\sigma^2(\bar{y})$	df	95% LL	95% UL
% SOLIDS	5.02E+01	1.72E+01	1	0.00	1.03E+02
pH	1.15E+01	4.00E-02	1	8.96E+00	1.40E+01
CN.dir	7.61E+01	1.56E+03	1	0.00	5.77E+02

★: Analytes with a portion of the data below 10 times the DL

NA: Not available

*RS: Residual solids from water digestion.

Table 7-3. Core Segment Data Concentration Estimate Statistics. (Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$\sigma^2(\bar{y})$	df	95% LL	95% UL
ICP.f.Al	3.88E+04	4.34E+08	2	0.00	1.28E+05
ICP.f.Bi	9.01E+03	5.71E+06	2	0.00	1.93E+04
ICP.f.Ca	1.92E+03	7.16E+05	2	0.00	5.56E+03
ICP.f.Cd★●	9.87E+00	6.79E+00	2	0.00	2.11E+01
ICP.f.Cr	3.17E+02	1.15E+03	2	1.71E+02	4.63E+02
ICP.f.Fe	2.60E+04	7.86E+06	2	1.39E+04	3.80E+04
ICP.f.Mg	2.61E+02	1.25E+03	2	1.09E+02	4.12E+02
ICP.f.Mn	4.27E+02	4.27E+04	2	0.00	1.32E+03
ICP.f.Na	9.55E+04	9.06E+07	2	5.46E+04	1.36E+05
ICP.f.Ni	5.19E+03	1.59E+06	2	0.00	1.06E+04
ICP.f.P	2.13E+04	1.80E+07	2	3.04E+03	3.96E+04
ICP.f.Pb★	7.17E+02	2.96E+04	2	0.00	1.46E+03
ICP.f.S	2.91E+03	2.55E+05	2	7.36E+02	5.08E+03
ICP.f.Si	5.39E+03	1.05E+06	2	9.82E+02	9.80E+03
ICP.f.Sr	7.93E+02	3.47E+04	2	0.00	1.59E+03
ICP.f.Ti	4.68E+01	5.92E+02	2	0.00	1.51E+02
ICP.f.Zr★●	5.52E+01	1.09E+03	2	0.00	1.97E+02
Sr-90	1.24E+02	8.42E+02	2	0.00	2.49E+02
GEA.Cs-137	1.70E+01	7.35E+01	2	0.00	5.38E+01
GEA.Eu-154	4.66E-01	9.54E-02	2	0.00	1.79E+00
TGA.H2O	4.60E+01	3.07E+01	2	2.22E+01	6.98E+01
%SOLIDS	5.77E+01	4.82E+01	2	2.79E+01	8.76E+01
pH	1.11E+01	1.94E-02	2	1.05E+01	1.17E+01
CN($\mu\text{g/g}$)	6.33E+01	2.02E+02	2	2.24E+00	1.24E+02

★: Analytes with a portion of the data below 10 times the DL.

●: Analytes with a portion of the data below 3 times the DL.

Appendix B contains a description of the statistical models and formulas used to calculate estimates of the mean, variance of the mean, and the confidence interval on the mean. The statistical models for the core composite data and the drainable liquid data are identical. The statistical model for the core and segment data is more complicated.

The summary statistics are as follows:

\bar{y} mean of the concentration data

$\sigma^2(\bar{y})$ estimated variance of \bar{y}

df degrees of freedom

95% LL lower limit to the 95% CI on the mean

95% UL upper limit to the 95% CI on the mean.

For some analytes the lower confidence limit (95% LL) was negative. Since concentrations are greater than or equal to zero, any negative 95% LL values were set equal to zero.

7.1.2 Statistical Results: Core Composite Data

Table 7-2 contains the summary statistics, by analyte, for ICP acid digestion, ICP water leach, ICP fusion dissolution, IC analyses and select radiochemistry. These values are based upon the core composite data. Since there were only two cores composite samples, Core 51 and 52, taken from T-107, the CIs on the mean concentration are very wide relative to the range of the data. The CIs were based upon only one degree of freedom.

7.1.3 Statistical Results: Core Segment Data

Table 7-3 contains the summary statistics, by analyte, for ICP fusion dissolution, radiochemistry, and some other selected analyses. These values are based upon an assay of the segments from each core. There were segment data from the three core samples taken from T-107. The CIs presented here were computed based on two degrees of freedom. The CIs in the previous section were based on one degree of freedom.

7.1.4 Statistical Results: Drainable Liquid Data

Table 7-4 contains the summary statistics, by analyte, for ICP and IC analyses of acidified drainable liquid samples. These values are based upon a chemical analysis of a drainable liquid sample from each core.

Data was available for drainable liquid samples from the three cores samples taken from T-107. The CIs were computed based on two degrees of freedom.

Table 7-4. Drainable Liquid Composite Data Concentration Estimate Statistics. (2 pages)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$\sigma^2(\bar{y})$	df	95% LL	95% UL
Al*	2.12E+01	1.82E+02	2	0.00	7.92E+01
B	2.13E+01	3.94E+01	2	0.00	4.83E+01
Ca	4.40E+00	9.26E-02	2	3.09E+00	5.71E+00
Cr	1.69E+02	4.41E+03	2	0.00	4.55E+02
Fe	2.43E+01	1.56E+02	2	0.00	7.79E+01
K	1.84E+02	9.08E+03	2	0.00	5.94E+02
Mg	6.81E-01	7.81E-02	2	0.00	1.88E+00
Mo	8.90E+00	4.55E+01	2	0.00	3.79E+01
Na	5.40E+04	5.47E+08	2	0.00	1.55E+05
Ni	6.87E+00	2.12E+01	2	0.00	2.67E+01
P	1.80E+03	2.82E+05	2	0.00	4.09E+03
S	3.39E+03	2.24E+06	2	0.00	9.83E+03
Si	7.07E+01	7.31E+01	2	3.39E+01	1.07E+02
Sr*	7.07E-01	3.04E-01	2	0.00	3.08E+00
Zr*	8.26E-01	2.11E-01	2	0.00	2.80E+00
CN ⁻	6.84E+01	1.80E+03	2	0.00	2.51E+02
IC.F ⁻	5.57E+02	3.87E+04	2	0.00	1.40E+03
IC.Cl ⁻	7.99E+02	1.10E+05	2	0.00	2.23E+03
IC.NO ₂ ⁻	1.28E+04	5.79E+07	2	0.00	4.55E+04
IC.PO ₄ ³⁻	5.42E+03	2.45E+06	2	0.00	1.22E+04
IC.SO ₄ ²⁻	1.03E+04	1.25E+07	2	0.00	2.55E+04
IC.NO ₃ ⁻	8.52E+04	1.12E+09	2	0.00	2.30E+05
NO ₂ ⁻	7.57E+03	8.04E+06	2	0.00	1.98E+04
TIC	1.80E+03	1.89E+06	2	0.00	7.71E+03
TOC	8.57E+02	6.36E+04	2	0.00	1.94E+03
AT	6.01E-03	2.81E-05	2	0.00	2.88E-02
U	2.41E+02	3.03E+04	2	0.00	9.90E+02
U-238	8.10E-05	3.42E-09	2	0.00	3.33E-04
Am-241	1.15E-04	1.99E-09	1	0.00	6.82E-04
TB	1.18E+01	4.74E+01	2	0.00	4.14E+01
Sr-90	5.94E-02	1.09E-03	2	0.00	2.02E-01

Table 7-4. Drainable Liquid Composite Data Concentration Estimate Statistics. (2 pages)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$\sigma^2(\bar{y})$	df	95% LL	95% UL
Tc-99	2.70E-02	3.66E-04	2	0.00	1.09E-01
C-14	2.03E-04	2.50E-08	2	0.00	8.84E-04
H-3	2.57E-03	4.79E-06	2	0.00	1.20E-02
GEA.Cs-137	8.43E+00	2.56E+01	2	0.00	3.02E+01

7.2 COMPARISON OF THE VARIANCE COMPONENT ESTIMATES

Using the hierarchical structure of the core composite data and the drainable liquid data, the spatial variability between cores and segments, and the analytical measurement variability can be separated from each other. These two spatial variances are measures of the variability between cores and between segments as a function of location. The analytical measurement variance includes the segment homogenization error, the sample handling error, and the chemical analysis error. This variance is a measure of the difference between the analytical results from the sample and duplicate samples.

The estimate of the variance of the mean is a linear function of the spatial and analytical measurement variances. To evaluate the magnitude of these variance components, explicit estimates of each variance component are given.

7.2.1 Statistical Methods

Estimates of the spatial variances ($\sigma^2(C)$), between cores and $\sigma^2(S)$, between segments), and analytical measurement variance ($\sigma^2(A)$) were obtained for each analyte using Restricted Maximum Likelihood Estimation (REML) methods. This method is discussed by Harville (1977).

An analysis of variance (ANOVA) was computed for the hierarchical statistical models used to describe the data. Appendix B contains a description of the models. An F-test, from the ANOVA, was used to test the hypothesis that $\sigma^2(C)$ is equal to zero (i.e. there is no difference between core means). When applicable, another F-test, from the ANOVA, was used to test the hypothesis that there is not a significant difference between segment means ($\sigma^2(S) = 0$). If $\sigma^2(S)$ is significantly different from zero, then it is concluded that the waste in T-107 is heterogeneous.

The p-values associated with the various F-test are reported in the following tables. If a p-value is less than 0.05 the variance components are significantly different from zero. The p-values were computed using the ANOVA results, not from the restricted maximum likelihood estimation (REML) results.

7.2.2 Statistical Results: Core Composite Data

Table 7-5 lists the REML estimates and the p-values for the spatial variability (between cores) and analytical variability for the core composite data. The spatial variability is significantly different from zero for 34 out of 74 analytes, 46% of the cases. This means that for a majority of the analytes, 54%, the mean concentrations from Cores 51 and 52 cannot be distinguished from each other.

Table 7-5. Core Composite Data Variance Component Estimates. (3 pages)

Analyte	$\sigma^2(C)$	Test: $\sigma^2(C) = 0$ p-value	$\sigma^2(A)$
ICP.a.Al	2.09E+08	0.001	5.02E+05
ICP.a.B*	4.67E+01	0.458	2.06E+02
ICP.a.Bi	1.65E+07	0.013	4.51E+05
ICP.a.Ca	3.15E+04	0.061	4.53E+03
ICP.a.Cd*	5.65E-02	0.414	2.53E+00
ICP.a.Cr	1.46E+03	0.076	2.73E+02
ICP.a.Fe	1.79E-31	0.763	6.65E+07
ICP.a.K	1.71E-13	0.534	5.25E+02
ICP.a.Li*	4.13E+00	0.044	4.05E-01
ICP.a.Mg	4.67E+03	0.008	7.30E+01
ICP.a.Mn	2.31E-20	0.846	5.07E+03
ICP.a.Na	3.85E+07	0.295	7.93E+07
ICP.a.Nd*	2.60E+03	0.002	1.08E+01
ICP.a.Ni	2.91E+02	0.062	4.25E+01
ICP.a.P	2.47E-25	0.906	1.84E+07
ICP.a.Pb	1.80E+05	0.051	2.13E+04
ICP.a.S	5.29E+05	0.003	3.20E+03
ICP.a.Si	1.15E+06	0.014	3.45E+04
ICP.a.Sm	6.40E+04	0.003	3.43E+02
ICP.a.Sr	1.54E+05	0.002	4.80E+02
ICP.a.Zr	4.50E+03	0.001	6.56E+00
ICP.w.Al	4.42E+04	0.150	2.11E+04
ICP.w.B	1.92E+05	0.002	8.71E+02
ICP.w.Bi	4.39E+04	0.005	4.65E+02
ICP.w.Ca	3.14E+04	0.334	1.05E+05
ICP.w.Cr	1.71E+02	0.268	2.59E+02

Table 7-5. Core Composite Data Variance
Component Estimates. (3 pages)

Analyte	$\sigma^2(C)$	Test: $\sigma^2(C) = 0$ p-value	$\sigma^2(A)$
ICP.w.Fe	1.30E+04	0.063	1.95E+03
ICP.w.K	1.73E+04	0.302	3.84E+04
ICP.w.Mg	5.88E-32	0.952	4.02E+00
ICP.w.Mo*	1.32E+00	0.004	1.17E-02
ICP.w.Na	1.33E+09	0.023	6.41E+07
ICP.w.S	3.30E+05	0.093	7.94E+04
ICP.w.Se*	5.64E+02	0.104	2.11E+01
ICP.w.Sr*	1.82E-42	0.948	7.77E-01
ICP.w.Zr*	4.11E+01	0.107	1.62E+00
ICP.f.Al	2.24E+08	0.006	2.64E+06
ICP.f.Bi	2.53E+07	0.048	2.74E+06
ICP.f.Ca	3.55E-21	0.462	1.66E+03
ICP.f.Cr	6.95E-23	0.721	4.28E+02
ICP.f.Fe	1.60E-17	0.667	8.91E+07
ICP.f.Mg	1.11E-20	0.775	7.26E+02
ICP.f.Mn	2.95E-19	0.597	4.63E+03
ICP.f.Na	5.20E+07	0.163	2.83E+07
ICP.f.Ni	8.11E+04	0.392	9.51E+05
ICP.f.P	2.22E-19	0.608	5.90E+06
ICP.f.Pb	8.77E-18	0.566	4.27E+04
ICP.f.S	1.63E+05	0.032	1.13E+04
ICP.f.Si	2.83E+06	0.006	3.28E+04
ICP.f.Sr	9.95E+03	0.111	3.05E+03
ICP.f.Zr	4.09E+02	0.215	3.69E+02
TD SOLID(wt%)	1.75E-28	0.553	1.67E-04
R S WT%	1.36E+01	0.099	3.55E+00
CN	1.05E+03	0.001	2.32E+00
IC.F-	9.25E+06	0.032	6.43E+05
IC.Cl-	3.87E+04	0.031	2.60E+03
IC.NO ₂ ⁻	2.56E+07	0.004	1.84E+05
IC.NO ₃ ⁻	6.60E+08	0.011	1.51E+07
IC.PO ₄ ³⁻	6.66E+08	0.070	1.13E+08
IC.SO ₄ ²⁻	1.52E+07	0.005	1.42E+05

Table 7-5. Core Composite Data Variance Component Estimates. (3 pages)

Analyte	$\sigma^2(C)$	Test: $\sigma^2(C) = 0$ p-value	$\sigma^2(A)$
NO ₂	1.94E+07	0.016	6.33E+05
TIC	4.18E+06	0.006	4.75E+04
TOC	1.33E+05	0.031	8.83E+03
AT	9.31E-06	0.020	3.85E-07
TB	1.87E+01	0.015	5.96E-01
¹⁴ C	4.90E-09	0.271	1.68E-09
³ H	1.11E-08	0.344	4.29E-08
GEA.Cs-137	1.51E+01	0.006	1.70E-01
RS* GEA.Cs-137	5.84E+01	0.007	8.64E-01
RS GEA.Co-60	2.20E-04	0.003	1.36E-06
RS GEA.Eu-154	1.20E-42	0.868	1.71E-04
RS GEA.K-40	8.00E-08	0.454	3.20E-07
%SOLIDS	8.49E+00	0.014	2.53E-01
pH	2.00E-02	0.000	4.99E-11
CN.dir	7.65E+02	0.016	2.53E+01

★: Analytes with a portion of the data below 10 times the DL.

*RS: Residual solids from water digestion.

7.2.3 Statistical Results: Core Segment Data

Table 7-6 lists the REML estimates and p-values for the spatial variability ($\sigma^2(C)$) between cores and $\sigma^2(S)$ between segments) and analytical variability for the core segment data. The between segments spatial variability is significantly different from zero for 22 out of 24 analytes, 92% of the cases. This variability is not significantly different from zero for ICP.f.Zr and Eu-154. On the other hand, the between cores variability is significantly different from zero for only 4 out of 24 analytes, 17% of the cases. This indicates that the waste in T-107 is vertically heterogeneous but horizontally homogeneous.

Table 7-6. Core Segment Data Variance Component Estimates.

Analyte	$\sigma^2(C)$	Test: $\sigma^2(C) = 0$ p-value	$\sigma^2(S)$	Test: $\sigma^2(S) = 0$ p-value	$\sigma^2(A)$
ICP.f.Al	3.82E+08	0.314	3.52E+09	0.000	3.76E+06
ICP.f.Bi	1.88E-02	0.496	6.53E+07	0.000	5.26E+05
ICP.f.Ca	8.15E-20	0.587	7.65E+06	0.000	1.16E+06
ICP.f.Cd★●	3.42E-23	0.697	6.57E+01	0.001	2.45E+01
ICP.f.Cr	3.57E+02	0.389	1.17E+04	0.000	3.03E+02
ICP.f.Fe	9.10E+06	0.245	4.98E+07	0.000	1.12E+07
ICP.f.Mg	4.48E+02	0.388	1.25E+04	0.000	2.21E+02
ICP.f.Mn	3.26E-17	0.547	4.48E+05	0.000	8.58E+04
ICP.f.Na	2.79E-18	0.926	1.03E+09	0.000	2.56E+07
ICP.f.Ni	2.46E-28	0.954	1.60E+07	0.000	9.30E+05
ICP.f.P	2.44E-10	0.675	2.03E+08	0.000	8.03E+06
ICP.f.Pb★	1.02E+04	0.365	2.63E+05	0.001	7.55E+04
ICP.f.S	5.93E+05	0.038	6.48E+05	0.000	1.51E+04
ICP.f.Si	3.78E-16	0.565	1.20E+07	0.000	1.31E+05
ICP.f.Sr	6.19E+04	0.133	1.60E+05	0.000	2.33E+03
ICP.f.Ti	4.46E-31	0.927	3.90E+03	0.005	1.18E+03
ICP.f.Zr★●	2.03E+03	0.012	1.50E+03	0.134	1.97E+03
Sr-90	1.13E+03	0.223	5.30E+03	0.000	6.68E+01
GEA.Cs-137	5.89E+01	0.323	6.19E+02	0.000	1.27E+00
GEA.Eu-154	2.57E-01	0.005	2.77E-02	0.136	1.19E-02
TGA.H2O	5.24E+01	0.150	1.50E+02	0.000	2.81E+00
%SOLIDS	9.51E+01	0.105	1.56E+02	0.000	3.74E-01
pH	1.40E-22	0.845	2.21E-01	0.000	1.67E-03
CN(µg/g)	5.37E+02	0.005	2.60E+02	0.000	8.37E+00

★: Analytes with a portion of the data below 10 times the DL.

●: Analytes with a portion of the data below 3 times the DL.

7.2.4 Statistical Results: Drainable Liquid Data

Table 7-7 lists the REML estimates and p-values for the spatial variability (between cores) and analytical variability for the drainable liquid data. The spatial variability is significantly different from zero for 36 out of 39 analytes, 92% of the cases. This means that for a majority of the analytes, 92%, the drainable liquid mean concentrations from Cores 50, 51 and 52 are significantly different from each other. The concentrations of Ca, Si, and H³ cannot be distinguished from each other. These results are consistent with similar results given in section 7.3.

Table 7-7. Drainable Liquid Composite Data Variance Component Estimates. (2 pages)

Analyte	$\delta^2(C)$	Test:	
		$\sigma^2(C) = 0$	p-value
Al*	5.45E+02	0.000	3.47E-01
B	1.17E+02	0.002	2.40E+00
Ca	5.41E-27	0.844	2.08E+00
Cr	1.32E+04	0.000	5.39E+00
Fe	4.67E+02	0.000	1.85E-01
K	2.72E+04	0.000	3.33E+01
Mg	2.29E-01	0.006	1.10E-02
Mo	1.37E+02	0.000	2.88E-02
Na	1.64E+09	0.000	5.33E+05
Ni	6.35E+01	0.000	1.61E-02
P	8.47E+05	0.000	1.10E+03
S	6.72E+06	0.000	4.45E+02
Si	1.91E+02	0.066	5.71E+01
Sr*	9.12E-01	0.000	7.28E-05
Zr*	6.31E-01	0.000	2.85E-03
TGA	1.15E+02	0.001	1.43E+00
SPG	8.07E-03	0.001	8.33E-05
TD.SOLID	1.03E+02	0.000	4.22E-01
pH	2.95E-01	0.000	1.67E-05
CN	5.41E+03	0.000	2.83E-02
IC.F ⁻	1.16E+05	0.000	2.55E+01
IC.Cl ⁻	3.30E+05	0.000	6.00E+01
IC.NO ₂ ⁻	1.74E+08	0.000	1.79E+04
IC.PO ₄ ³⁻	7.33E+06	0.000	8.60E+03
IC.SO ₄ ²⁻	3.74E+07	0.000	1.67E+01
IC.NO ₃ ⁻	3.37E+09	0.000	1.67E+05
NO ₂	2.40E+07	0.001	2.23E+05
TIC	5.66E+06	0.000	8.60E+02
TOC	1.90E+05	0.001	1.96E+03
AT	8.41E-05	0.000	1.70E-07
U	9.07E+04	0.000	3.24E+02
U-238	1.02E-08	0.000	3.76E-11
Am-241	8.96E-09	0.018	9.25E-12

Table 7-7. Drainable Liquid Composite Data Variance Component Estimates. (2 pages)

Analyte	$\delta^2(C)$	Test: $\sigma^2(C) = 0$ p-value	$\delta^2(A)$
TB	1.42E+02	0.000	1.70E-01
Sr-90	3.27E-03	0.000	1.38E-05
Tc-99	1.82E-03	0.000	2.16E-07
C-14	6.87E-08	0.038	1.28E-08
H-3	1.14E-05	0.115	5.94E-06
GEA.Cs-137	7.68E+01	0.000	1.01E-01

*: Analytes with a portion of the data below 10 times DL.

7.3 MULTIPLE COMPARISONS

A group of statistical methods known as multiple comparisons can be used to determine whether or not there are significant differences between core composite samples and between sub-segment samples. These differences will help determine the heterogeneity or existence of layers within the waste. In addition, if significant differences exist between core composite samples or between the sub-segment samples, this will help explain the extreme width of the CIs (i.e., will help explain the large spatial variability observed). The multiple comparison procedure known as Tukey's Honestly Significant Difference (HSD) was used. The HSD procedure determines if there are significant differences between core composite samples and subsegment samples. The core composite samples and sub-samples that are not significantly different from each other can then be grouped together.

7.3.1 Comparison Between Core Composite Samples

Table 7-8 gives a visual comparison of core composite means between Cores 51 and 52. Since there are only two means, the multiple comparison procedures are equivalent to an ordinary one-way ANOVA. The means are represented by letters A and B. If the two means are significantly different from each other the two letters are different (the letter A denotes the smaller of the two means). Otherwise the two letters are the same. The two core means are significantly different in 13 out of 27 comparisons.

7.3.2 Comparison Between Core Segment Samples

Due to the incomplete core recovery, data is available for only some sub-segments, locations within a core. Table 7-9 gives the relative locations of the sub-segments within Cores 50, 51, and 52.

Table 7-8. Core Composite Data, Multiple Comparison of Means.

Analyte	Core 51	Core 52	Analyte	Core 51	Core 52
ICP.a.Al	A	B	ICP.f.P	A	A
ICP.a.Ca	A	A	ICP.f.Pb	A	A
ICP.a.Fe	A	A	CN	B	A
ICP.a.Na	A	A	IC.F-	A	B
ICP.a.P	A	A	IC.CI-	B	A
ICP.a.Pb	A	A	IC.NO2-	B	A
ICP.w.Al	A	A	IC.NO3-	B	A
ICP.w.Ca	A	A	IC.PO4-	A	A
ICP.w.Fe	A	A	IC.SO4-	B	A
ICP.w.Na	B	A	NO2	B	A
ICP.f.Al	A	B	GEA.Cs-137	B	A
ICP.f.Ca	A	A	RS GEA.Cs-137	B	A
ICP.f.Fe	A	A	CN.dir	B	A
ICP.f.Na	A	A			

Table 7-9. Position of Segments Within Cores.

Core	50	51	52
Segment	1R	-	1
	2	2	2
	3	3U	3U
	-	3L	3L
	-	4U	-
	-	4L	-

-: Missing value

U: Upper half segment

L: Lower half segment

For each analyte, comparisons were made between the means of the sub-segment samples. These comparisons are given in Table 7-10. The letters A, B, etc. are used to represent groupings. The means with letters in common cannot be distinguished from each other. Sub-segment means with different letters are significantly different from each other. The means for each analyte are also given. The letter A is used for the smallest mean, B the next smallest, etc. One relative standard deviation is also given. The HSD comparisons do not incorporate the spatial variability. They are a function of the analytical measurement variance.

Table 7-10. Core Segment Data, Tukey's HSD Comparisons.

Core	Group			Mean (µg/g)			RSD
	50	51	52	50	51	52	
ICP.f.Al	BC	-	G	9810	-	213500	5.43%
ICP.f.Ca	A	-	B	1050	-	10900	54.6%
	A	A	A	822	2095	771	
ICP.f.Fe	A	-	D	19000	-	40550	12.5 %
	A	CD	AB	20350	36650	21050	
	ABC	ABC	ABC	23800	28500	23400	
	-	D	A	-	34250	19000	
	-	BCD	-	-	35050	-	
	-	CD	-	-	19650	-	
		A					
ICP.f.Na	EF	-	A	127500	-	27250	5.4%
	B	CB	D	55200	71100	105250	
	DEF	ED	F	122500	108000	131000	
	-	C	D	-	77900	107000	
	-	C	-	-	82450	-	
	-	DEF	-	-	122000	-	
ICP.f.Ni	CD	-	<	8925	-	LT	19.7%
	AB	D	AB	3970	12100	2915	
	-	-	-	-	-	-	
	-	A	D	-	853.5	9415	
	-	A	-	-	860.5	-	
	-	D	-	-	9345	-	
ICP.f.P	CE	-	<	30550	-	LT	13.9%
	A	A	C	3840	5330	25650	
	E	CD	C	42650	25100	36850	
	-	A	C	-	7615	25950	
	-	AB	-	-	9715	-	
	-	EC	-	-	32450	-	
ICP.f.Pb	AB	-	D	241.5	-	1840	35.8 %
	ABC	BCD	ABC	530	1330	419.5	
	ABC	CD	ABC	496	1500	451.5	
	-	ABC	A	-	987.5	131.5	
	-	D	-	-	1002.5	-	
	-	ABC	-	-	263.5	-	
		D					
		AB					

-: Missing value

<: Less than value

LT: Less than value

The comparisons between means can be complicated, except for Ca. For this analyte, all sub-samples (except for Core 52 Segment 1) cannot be distinguished from each other. For some of the analytes there is evidence of significant differences between cores and between sub-segments; i.e., the waste is heterogenous.

7.3.3 Comparison Between Drainable Liquid Samples

The HSD comparisons between the results from the drainable liquid samples from the three cores is given in Table 7-11. The comparisons given here are easy to interpret. For 15 out of 16 comparisons the three core means have three different letters (A, B, and C). This says that, for 15 analytes, the three core means are significantly different from each other. The three core means cannot be distinguished for Ca. The comparisons do not incorporate the spatial variability. They are a function of the analytical measurement variance.

Table 7-11. Drainable Liquid, Tukey's HSD Comparison.

Analyte	Core 50	Core 51	Core 52
Al	A	B	C
Ca	A	A	A
Fe	A	C	B
Na	A	C	B
P	A	B	C
CN	A	C	B
IC.F ⁻	A	C	B
IC.Cl ⁻	A	C	B
IC.NO ₂ ⁻	A	C	B
IC.PO ₄ ³⁻	A	B	C
IC.SO ₄ ²⁻	A	C	B
IC.NO ₃ ²⁻	A	C	B
NO ₂	A	C	B
U	A	B	A
Sr90	A	C	B
GEA.Cs-137	A	C	B

7.4 HOMOGENIZATION TEST DESCRIPTION

A task in complying with the Tank Waste Characterization Plan (Bell, 1993) was to evaluate the Process and Analytical Laboratories ability to homogenize segments.

Two homogenization tests were done on samples taken from Cores 50, 51, and 52. In the first test, analytical difficulties with the samples were encountered. This data was not statistically analyzed. In the second test, data from Core 51 was incomplete. Consequently, only the data from Cores 50 Segment 2 and Core 52 Segment 3L were included in the statistical analysis.

In the homogenization tests, samples from cores were homogenized and arbitrarily divided into two parts. One subsample was obtained from each part. Two aliquots were taken from each subsample and prepared for chemical analysis. ICP acid digestion was conducted on the samples. The data from the homogenization tests are given in Table B-6.

7.4.1 Statistical Methods and Results

Because the nested structure (aliquots within subsamples, subsamples within segments) of the data, a hierarchical statistical model was fit to the data. Snedecor (1980), contains a description of this type of model. Such a model is used to estimate different components of variability in the data. The model used is explicitly described in Jensen and Remund (1993). The total variability in the data is decomposed into two components: one the variability between samples taken from different locations, $\sigma^2(L)$, and one arising from the analytical measurement error, $\sigma^2(A)$. The analytical measurement error accounts for the differences between aliquots taken from the same location.

To quantify the contribution of $\sigma^2(L)$ the component of variability due to differences in location or lack of homogenization, the ANOVA corresponding to the hierarchical model was used. The hypothesis tested is that the variability due to differences in location, $\sigma^2(L)$, is not significantly different from zero was then tested by comparing $\sigma^2(L)$ with $\sigma^2(A)$ using an F-test. For a given analyte, if $\sigma^2(L)$ is relatively small compared to $\sigma^2(A)$, then we conclude that any variability due to lack of homogenization was not significant for that analyte. If $\sigma^2(L)$ is relatively large compared to $\sigma^2(A)$, then we conclude that samples taken from separate locations may be different due to lack of homogenization.

The F-test is used to determine whether or not $\sigma^2(L)$ is significantly different from zero. The p-values from these tests are given in Table 6-1. P-values less than 0.05 indicate that $\sigma^2(L)$ was significantly different from zero at the 0.05 significance level. In this test, ICP.a.Bi was the only analyte, out of a total of 18, with a p-value less than 0.05.

For 17 out of 18 analytes the variability due to differences in location was significantly less than the analytical variability. Hence, relative to the analytical variability, the Process and Analytical Laboratories were able to adequately homogenize core segments in this experiment.

Table 7-12. Homogenization Test
Statistical Results.

Analyte	Test: $\sigma^2(L) = 0$ p-value
ICP.a.Al	0.980
ICP.a.Bi	0.011
ICP.a.Ca	0.642
ICP.a.Cd*	0.745
ICP.a.Cr	0.273
ICP.a.Fe	0.496
ICP.a.K	0.899
ICP.a.Li*	0.846
ICP.a.Mg	0.551
ICP.a.Mn	0.365
ICP.a.Mo	0.963
ICP.a.Na	0.466
ICP.a.Ni	0.499
ICP.a.P	0.703
ICP.a.Pb	0.823
ICP.a.S	0.143
ICP.a.Sm	0.709
ICP.a.Sr	0.583

*: Analytes with a portion of
the data below 10 times the DL.

7.5 ANALYTICAL RESULTS - POTENTIAL WASTE CONSTITUENTS

7.5.1 Suggested Components of Waste Matrix

The actual composition of the waste matrix is quite complex, and trace amounts of several various compounds probably exist in the tank. However, with some simple assumptions regarding how the anions and cations will combine, a list of the most probable compounds that exist in the waste matrix and contribute significantly to its overall makeup can be developed.

Table 7-13 is a condensed version of a more general chart found on page D-147 in the *Handbook of Chemistry and Physics 64th Ed.* (Weast 1984). It provides solubility data on some of the most common anions and cations.

Table 7-13. Probable Solids in the Waste Matrix.

	NO_3^-	PO_4^{3-}	SO_4^{2-}	OH^-	Si (as SiO_3^-)	CO_3^{2-}	F ⁻
Al^{+3}		PPT		PPT			
Bi^{+3}		PPT		PPT			
Ca^{+2}		PPT	PPT			PPT	
Cr^{+3}				PPT			
$\text{Fe}^{+2,+3}$	PPT			PPT	PPT	PPT	
Na^+							
La^{+3}							
Mn^{+4}		PPT		PPT	PPT	PPT	PPT
U^{+6}	NL	PPT	PPT	PPT			

PPT = Precipitate forms

NL = Precipitate formation not likely under tank conditions.

From the earlier tables and process information, chloride and ferrocyanide will not be significant mass contributors to the waste matrix. However, sulfide and cyanide precipitates are significant because they provide a potential fuel source. Sodium, NO_2^- , and NO_3^- are highly soluble, and thus probably do not contribute much to the insoluble solids. However, they contribute significantly to the overall solids content of the waste (dissolved + insoluble solids). In addition, they represent three of the four most prevalent analytes, after water, in the waste. No analytical measurement of hydroxide was made, but it is known that in the process history of tank 241-T-107, basic solutions were added routinely to the tank.

7.6 MASS CHARGE BALANCE

Mass and charge balance determinations provide a method of accountability for analytical results and can be used as a measure of accuracy for the sum of the determinations. The mass and charge balance can only be used if the analyte species are correctly characterized as to their true chemical nature. If, for example, it is determined through inductively coupled plasma spectroscopy that substantial quantities of iron (Fe) and chromium (Cr) exist in a sample, a charge and mass balance would not determine the accuracy of the Fe and Cr determinations until the chemical nature of the two species were determined.

Iron (Fe) could exist as Fe^{3+} (aq) or Fe_2O_3 ; chromium (Cr) could exist as Cr^{3+} or CrO_4^{2-} ; and each of these could exist in a number of other likely forms. Thus, the charge and mass of the analyte species depends on the actual chemical nature of the analyte itself. Therefore, the following assumptions were made for this tank waste formulation.

- The "oxide model" was assumed as the standard chemical nature for most of the chemical species determined. Where there was a choice of several possible oxide candidates for the chemical species, that oxide which possessed the lowest (most negative) Gibbs Free Energy was selected.
- Where analytical results were obtained from both ICP and IC methods, ICP results were used only.
- Hydroxide (OH^-) values were derived from pH measurements.
- Chemical species whose determinations for the most part fell below 100 $\mu g/g$ were not included in the mass or charge balances and are not listed in the spreadsheet tables.
- Calcium, potassium, sodium, and strontium were the only cations considered in the charge balance. The elements: Al, Sb, Bi, Cr, Fe, Pb, Mg, Mn, Mo, Ni, Se, U, and Zr were all considered insoluble oxides of various chemical formulas.
- Ionic species considered were: $B_4P_7^{2-}$, Cl^- , F^- , CN^- , NO_3^- , NO_2^- , OH^- , HPO_4^{2-} , SO_4^{2-} , SiO_3^{2-} , CO_3^{2-} , and CH_3COO^- .
- Total organic carbon (TOC) was assumed to be acetate, CH_3COO^- . Total inorganic carbon (TIC) was assumed to be carbonate, CO_3^{2-} .

Table 7-14 lists the results of the charge and mass balance determinations.

Table 7-14. Charge and Mass Balance Determinations.

Sample	Charge Balance	Mass Balance
Core 50, Drainable Liquid	0.97	100.47
Core 51, Drainable Liquid	1.00	105.84
Core 51, Water Leach Analyses	1.00	96.67
Core 51, Acid Digest Analyses	1.10	102.81
Core 51, Fusion Digest Analyses	0.89	103.81
Core 52, Drainable Liquid	1.02	102.82
Core 52, Water Leach Analyses	0.97	74.72
Core 52, Acid Digest Analyses	1.18	95.20
Core 52, Fusion Digest Analyses	0.97	100.84

For the most part the quality of analysis for these tank waste samples appeared to be thorough and adequate. There are, however, some anomalies with the data which require attention. The complete tables and numbers used to calculate the mass and charge balance are found in Appendix C.

- The low mass determined from Core 51 and Core 52 Water Leach analyses was most probably due to the fact that only water soluble species were determined. This would exclude major components such as oxides of aluminum and iron.
- Core 52, water leach analyses (excluding Wt% Solids) appear to be a factor of 1.5 low. This includes all analytes except fluoride and TOC which appear to be too high. Evidence for the low values is seen in the mass balance for the water leach analyses and also in the charge and mass balances of the corresponding acid digest analyses.

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8.0 CONCLUSIONS AND RECOMMENDATIONS

Analysis of waste retrieved from tank T-107 reveal a small number of analytes which make up a large portion of waste. Water is the major contributor to the waste resulting in approximately percent of the solid mass; this tank does contain a small amount of drainable liquid (less than percent of the total mass). Elements (aluminum, bismuth, calcium, chromium, iron, phosphorus, silicon, sodium, sulfur, titanium) constitute approximately 32 percent of the solids mass; they also represent 95% of the total cations. Four anions (fluoride, phosphate, nitrate, nitrite) make up 20 percent of the solid mass. The fraction of the total anions cannot be adequately determined because the analytical methods measure only soluble anions, and it is known that there are insoluble hydroxides that are not assayed at this time. The total organic carbon content was measured and found to be 0.15 percent (dry basis) for the tank as a whole.

The only significant gamma emitter found was ^{137}Cs . Cesium-137 was found throughout the core sample but at very low concentrations. Reviewing the analyte trending table, an anomalous concentration of $100 \mu\text{Ci/g}$ was found in Segment 2 of Core 51. This point was confirmed by additional analyses. The major source of radiological activity in the tank was strontium-90. Examining the results from the core composite analyses, concentrations are constant throughout the waste. Concentrations for the strontium are moderate as expected from the fill history. The bulk waste temperature in the tank was reported to be 67°F . The radiological activity of tank T-107 was relatively low (0.020 to 0.3 R/hr, measured through the drill string). A slight amount of radiological activity was found in the drainable liquid in the tank and the water digestions of the samples. Further analysis shows that ^{137}Cs is found in its water soluble form, and ^{90}Sr is largely insoluble.

The segment trending does show evidence of 1C, TBP, and CW wastes. By examining the distribution of analytes on a segment level basis, the waste shows definite layering within the tank. The tank received ferrocyanide scavenged TBP waste during 1953-1954 from another ferrocyanide tank and because of this tank T-107 is considered a Ferrocyanide Watch List tank. However, because of the relatively small amount of waste and large analytical horizon, evidence of ferrocyanide solids was not expected to be observed, and the low concentrations of cyanide observed in the tank aid in confirming that supposition. The overall analytical results indicate that continued storage of waste in the tank does not pose a threat to the public/worker safety or health. DSC thermograms of the waste do not display any exothermic regions at any temperature, and thus confirm the general evidence indicating the concentration of ferrocyanide in tank T-107 is very low. Results of aging studies now underway on flowsheet simulants may demonstrate that radiolytic, hydrolytic, and thermal processes in the tanks, over the last 35 years, have combined to dissolve, dilute, and destroy the reactive ferrocyanide compounds. The data from tank 241-T-107 strongly indicate that the waste lacks the fuel concentration to sustain any propagating exothermic behavior, and a heat source intense enough to trigger a reaction is absent.

The analytical results should aid in categorizing tank T-107 as *Safe*.

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APPENDIX A

CORE COMPOSITE AND HOMOGENIZATION TEST DATA

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Table A-1. Core Composite Data. (sheet 1 of 5)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
ICP.a.Ag	6.79E+00	7.94E+00	1.47E+01	<8.98E-01	<8.91E-01	NA
ICP.a.Al	4.03E+03	4.25E+03	1.12E+03	2.39E+04	2.53E+04	4.82E+03
ICP.a.As*	<2.99E+01	<2.98E+01	NA	4.63E+00	3.95E+00	1.10E+00
ICP.a.B*	3.38E+01	1.35E+01	2.37E+01	3.59E+00	<9.90E-01	3.59E+00
ICP.a.Be	<2.99E+00	<2.98E+00	NA	<2.99E-01	<2.97E-01	NA
ICP.a.Bi	7.79E+03	8.13E+03	1.26E+03	1.31E+04	1.44E+04	1.50E+03
ICP.a.Ca	8.08E+02	8.97E+02	9.48E+02	5.42E+02	6.43E+02	1.18E+03
ICP.a.Cd*	7.93E+00	6.50E+00	1.20E+01	4.17E+00	7.01E+00	7.99E+00
ICP.a.Ce	<6.78E+01	<6.75E+01	NA	1.80E+02	1.85E+02	1.43E+01
ICP.a.Cr	3.83E+02	3.81E+02	4.24E+02	3.09E+02	3.42E+02	2.17E+02
ICP.a.Fe	3.34E+04	3.29E+04	2.21E+04	2.01E+04	3.95E+04	1.86E+04
ICP.a.K	2.67E+02	2.20E+02	2.10E+01	2.33E+02	2.17E+02	1.52E+01
ICP.a.La	<1.69E+01	<1.69E+01	NA	<2.00E+00	<1.98E+00	NA
ICP.a.Li*	7.59E+00	6.32E+00	1.74E+01	3.97E+00	4.05E+00	6.68E+00
ICP.a.Mg	2.65E+02	2.59E+02	8.73E+02	1.57E+02	1.73E+02	5.50E+02
ICP.a.Mn	2.36E+02	2.26E+02	7.70E+02	1.26E+02	2.98E+02	7.07E+02
ICP.a.Mo*	<5.98E+00	<5.95E+00	NA	5.57E+00	7.26E+00	7.12E+00
ICP.a.Na	1.42E+05	1.31E+05	2.21E+04	1.31E+05	1.17E+05	3.10E+04
ICP.a.Nd*	1.06E+02	1.12E+02	1.33E+01	3.55E+01	3.82E+01	4.72E+00
ICP.a.Ni	3.08E+02	3.01E+02	2.34E+02	2.74E+02	2.85E+02	1.86E+02
ICP.a.P	3.39E+04	2.54E+04	6.43E+03	3.34E+04	2.73E+04	4.34E+03
ICP.a.Pb	1.17E+03	1.04E+03	1.77E+02	3.57E+02	6.18E+02	6.24E+01
ICP.a.S	3.60E+03	3.52E+03	1.08E+03	2.49E+03	2.57E+03	6.49E+02
ICP.a.Sb*	<2.09E+02	<2.08E+02	NA	3.74E+01	5.36E+01	2.99E+00
ICP.a.Se*	1.04E+02	<8.63E+01	1.20E+01	<1.41E+01	<1.40E+01	NA
ICP.a.Si	6.37E+01	1.56E+02	6.88E+01	1.82E+03	1.46E+03	4.82E+02
ICP.a.Sm	4.80E+02	4.48E+02	4.94E+01	1.15E+02	9.63E+01	1.02E+01
ICP.a.Sr	1.25E+03	1.23E+03	4.13E+03	7.04E+02	6.65E+02	2.28E+03
ICP.a.Ti	<2.99E+00	<2.98E+00	NA	4.11E+00	1.46E+02	1.07E+02
ICP.a.Ti*	<1.64E+02	<1.63E+02	NA	7.20E+01	3.54E+01	2.15E+00
ICP.a.Zr	1.17E+02	1.21E+02	1.70E+02	2.25E+01	2.57E+01	2.01E+01

*: Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data. (sheet 2 of 5)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
ICP.w.Ag	<5.26E-01	<5.26E-01	NA	<5.06E+00	<5.07E+00	NA
ICP.w.Al	3.44E+02	6.27E+02	1.31E+02	8.49E+02	7.84E+02	2.21E+02
ICP.w.As	<3.16E+00	<3.16E+00	NA	<3.04E+01	<3.04E+01	NA
ICP.w.B	6.07E+02	6.66E+02	6.36E+02	1.61E+01	1.48E+01	1.55E+01
ICP.w.Be	<3.16E-01	<3.16E-01	NA	<3.04E+00	<3.04E+00	NA
ICP.w.Bi	1.08E+02	8.15E+01	1.50E+01	3.75E+02	4.09E+02	6.22E+01
ICP.w.Ca	1.51E+02	8.00E+02	5.29E+02	7.11E+01	5.99E+01	7.28E+01
ICP.w.Cd	<6.32E-01	<6.32E-01	NA	<6.07E+00	<6.08E+00	NA
ICP.w.Ce	<7.16E+00	<7.16E+00	NA	<6.88E+01	<6.89E+01	NA
ICP.w.Cr	2.30E+02	2.16E+02	2.48E+02	2.13E+02	1.84E+02	2.20E+02
ICP.w.Fe	3.15E+02	2.29E+02	1.81E+02	4.29E+02	4.49E+02	2.93E+02
ICP.w.K	6.41E+02	2.60E+02	3.88E+01	2.26E+02	1.35E+02	1.55E+01
ICP.w.La	<1.79E+00	<1.79E+00	NA	<1.72E+01	<1.72E+01	NA
ICP.w.Li*	9.04E-01	7.93E-01	2.12E+00	<4.05E+00	<4.05E+00	NA
ICP.w.Mg	7.30E+00	1.22E+01	3.25E+01	1.00E+01	9.83E+00	3.31E+01
ICP.w.Mn*	1.78E+00	2.37E+00	6.90E+00	<3.04E+00	<3.04E+00	NA
ICP.w.Mo*	8.72E+00	8.67E+00	1.45E+01	6.96E+00	7.17E+00	1.18E+01
ICP.w.Na	1.28E+05	1.40E+05	2.16E+04	8.71E+04	7.65E+04	1.32E+04
ICP.w.Nd	<8.63E+00	<8.63E+00	NA	<8.30E+01	<8.31E+01	NA
ICP.w.Ni*	5.18E+00	3.79E+00	3.45E+00	<1.32E+01	<1.32E+01	NA
ICP.w.P	2.73E+04*	3.33E+04*	6.59E+03	1.84E+04	1.59E+04	3.72E+03
ICP.w.Pb*	2.03E+01	2.01E+01	3.26E+00	<6.28E+01	<6.28E+01	NA
ICP.w.S	4.10E+03	3.84E+03	1.20E+03	3.36E+03	2.86E+03	9.42E+02
ICP.w.Sb	<2.21E+01	<2.21E+01	NA	<2.13E+02	<2.13E+02	NA
ICP.w.Se*	5.86E+01	5.21E+01	6.37E+00	<8.81E+01	8.94E+01	1.03E+01
ICP.w.Si	3.71E+03	4.18E+03	2.47E+03	<1.62E+01	<1.62E+01	NA
ICP.w.Sm	<9.89E+00	<9.89E+00	NA	<9.51E+01	<9.52E+01	NA
ICP.w.Sr*	6.44E+00	4.54E+00	1.83E+01	5.06E+00	6.08E+00	1.86E+01
ICP.w.Ti*	1.25E+00	2.98E+00	7.07E+00	<3.04E+00	<3.04E+00	NA
ICP.w.Tl	<1.73E+01	<1.73E+01	NA	<1.66E+02	<1.66E+02	NA
ICP.w.Zr*	2.10E+00	3.90E+00	4.29E+00	1.22E+01	<7.09E+00	1.75E+01

*: Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data. (sheet 3 of 5)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
ICP.f.Ag	<4.48E+00	<4.46E+00	NA	<4.46E+00	<4.49E+00	NA
ICP.f.Al	7.26E+03	4.20E+03	2.25E+02	2.64E+04	2.75E+04	1.05E+03
ICP.f.As	<1.94E+01	<1.93E+01	NA	<1.93E+01	<1.95E+01	NA
ICP.f.B	<4.98E+00	<4.95E+00	NA	<4.96E+00	<4.99E+00	NA
ICP.f.Be	<1.49E+00	<1.49E+00	NA	<1.49E+00	<1.50E+00	NA
ICP.f.Bi	8.20E+03	8.49E+03	1.82E+02	1.40E+04	1.73E+04	3.39E+02
ICP.f.Ca	7.65E+02	7.94E+02	3.12E+02	7.02E+02	7.81E+02	2.97E+02
ICP.f.Cd*	6.35E+00	5.95E+00	1.76E+00	5.66E+00	9.81E+00	2.21E+00
ICP.f.Ce*	8.76E+01	1.04E+02	1.50E+00	1.32E+02	1.37E+02	2.09E+00
ICP.f.Cr	3.51E+02	3.59E+02	4.73E+01	3.41E+02	3.89E+02	4.87E+01
ICP.f.Fe	2.63E+04	2.66E+04	3.31E+03	2.10E+04	4.28E+04	3.99E+03
ICP.f.K	NA	NA	NA	NA	NA	NA
ICP.f.La	<9.96E+00	<9.90E+00	NA	<9.92E+00	<9.98E+00	NA
ICP.f.Li*	4.87E+00	5.07E+00	1.66E+00	3.14E+00	4.98E+00	1.35E+00
ICP.f.Mg	2.23E+02	2.40E+02	1.55E+02	1.90E+02	2.52E+02	1.47E+02
ICP.f.Mn	1.96E+02	1.83E+02	1.26E+02	1.61E+02	3.13E+02	1.58E+02
ICP.f.Mo*	1.05E+01	1.06E+01	2.36E+00	7.96E+00	6.63E+00	1.62E+00
ICP.f.Na	1.19E+05	1.27E+05	6.15E+03	1.15E+05	1.08E+05	5.60E+03
ICP.f.Nd*	8.11E+01	8.33E+01	2.11E+00	5.53E+01	<3.89E+01	1.42E+00
ICP.f.Ni	2.85E+03	1.37E+03	2.81E+02	3.80E+03	2.53E+03	4.21E+02
ICP.f.P	3.15E+04	3.43E+04	9.40E+02	3.36E+04	2.89E+04	8.94E+02
ICP.f.Pb	7.63E+02	6.90E+02	1.86E+01	3.46E+02	7.96E+02	1.46E+01
ICP.f.S	3.49E+03	3.64E+03	1.83E+02	2.91E+03	3.06E+03	1.53E+02
ICP.f.Sb*	1.39E+02	1.04E+02	1.61E+00	<7.54E+01	<7.58E+01	NA
ICP.f.Se	<7.02E+01	<6.98E+01	NA	<6.99E+01	<7.04E+01	NA
ICP.f.Si	4.98E+03	4.75E+03	2.86E+02	7.11E+03	7.39E+03	4.26E+02
ICP.f.Sm*	1.48E+02	2.08E+02	3.42E+00	7.95E+01	6.34E+01	1.37E+00
ICP.f.Sr	9.34E+02	9.74E+02	6.36E+02	7.51E+02	8.54E+02	5.35E+02
ICP.f.Ti*	<3.49E+00	1.63E+01	4.65E+00	5.22E+00	7.28E+00	1.79E+00
ICP.f.Ti*	<1.25E+02	1.44E+02	1.15E+00	<1.24E+02	<1.25E+02	NA
ICP.f.Zr	8.56E+01	6.63E+01	1.27E+01	1.27E+02	9.38E+01	1.83E+01

*: Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data. (sheet 4 of 5)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
TD SOLID(wt%)	3.78E-01	3.96E-01	NA	4.12E-01	3.92E-01	NA
R S WT%	2.77E+01	2.42E+01	NA	3.08E+01	3.22E+01	NA
Cr(VI)	<1.85E+01	<1.84E+01	NA	<1.92E+01	<1.95E+01	NA
NH4	<8.20E+02	<8.16E+02	NA	<4.20E+03	<4.26E+03	NA
CN	9.29E+01	9.06E+01	4.59E+01	4.49E+01	4.69E+01	2.30E+01
IC.F-	8.53E+03	9.92E+03	9.23E+02	1.40E+04	1.32E+04	3.32E+03
IC.Cl-	7.32E+02	6.32E+02	3.41E+01	3.89E+02	4.09E+02	9.50E+01
IC.NO2-	1.57E+04	1.49E+04	1.53E+02	7.98E+03	8.29E+03	3.87E+02
IC.NO3-	9.64E+04	8.92E+04	9.28E+02	5.48E+04	5.77E+04	2.68E+03
IC.PO4-	8.70E+04	1.02E+05	9.45E+02	1.40E+05	1.25E+05	3.23E+03
NO2	1.50E+04	1.35E+04	3.75E+01	7.71E+03	8.24E+03	2.10E+01
TIC	5.64E+03	5.71E+03	1.14E+01	2.99E+03	2.56E+03	5.55E+00
TOC	1.52E+03	1.35E+03	2.61E+00	2.00E+03	1.92E+03	3.56E+00

*: Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data. (sheet 5 of 5)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
AT	5.44E-04	4.96E-04	2.60E+00	5.50E-03	4.26E-03	1.74E+01
TB	1.61E+01	1.69E+01	4.23E+03	1.10E+01	9.68E+00	1.40E+03
C14	2.55E-04	<2.22E-04	5.80E-01	1.15E-04	1.73E-04	6.26E+00
H3	1.56E-03	1.17E-03	5.06E+00	1.04E-03	1.18E-03	4.11E+00
GEA.Cs-137	1.24E+01	1.16E+01	4.43E+03	6.39E+00	6.59E+00	4.06E+03
GEA.Cs-134	<1.27E-03	<1.29E-03	NA	<2.04E-03	<1.64E-03	NA
GEA.Am-241	<2.81E-02	<2.67E-02	NA	<2.22E-02	<2.28E-02	NA
GEA.Co-60	<1.20E-03	<1.45E-03	NA	<1.99E-03	<2.08E-03	NA
GEA.Eu-154	<3.91E-03	<3.40E-03	NA	<4.36E-03	<5.82E-03	NA
GEA.Eu-155	<1.25E-02	<1.23E-02	NA	<1.49E-02	<1.51E-02	NA
GEA.K-40	<3.24E-02	<3.27E-02	NA	<6.49E-02	<6.53E-02	NA
GEA.CePr-144	<4.09E-02	<3.95E-02	NA	<5.79E-02	<5.79E-02	NA
GEA.Ru-103	<6.19E-03	<5.93E-03	NA	<7.11E-03	<7.21E-03	NA
GEA.RuRh-106	<7.91E-02	<7.57E-02	NA	<7.52E-02	<7.49E-02	NA
GEA.Th-228/Pb	<8.73E-03	<8.44E-03	NA	<1.03E-02	<1.07E-02	NA
RS* GEA.Cs-137	1.71E+01	1.77E+01	NA	5.67E+00	7.43E+00	NA
RS GEA.Cs-134	<1.21E-02	<1.39E-02	NA	<7.66E-03	<7.50E-03	NA
RS GEA.Am-241	<3.49E-01	<3.85E-01	NA	<1.92E-01	<1.92E-01	NA
RS GEA.Co-60	2.84E-02	2.96E-02	1.21E+01	7.00E-03	9.00E-03	4.21E+00
RS GEA.Eu-154	1.20E-01	1.49E-01	3.06E+01	1.25E-01	1.38E-01	2.80E+01
RS GEA.Eu-155	<1.59E-01	<2.06E-01	NA	1.07E-01	1.39E-01	1.23E+01
RS GEA.K-40	3.71E-02	<3.38E-02	5.98E-01	3.83E-02	3.75E-02	NA
RS GEA.CePr-144	<2.71E-01	<3.05E-01	NA	<1.50E-01	<1.58E-01	NA
RS GEA.Ru-103	<2.56E-02	<2.78E-02	NA	<1.42E-02	<1.55E-02	NA
RS GEA.RuRh-106	<1.61E-01	<1.81E-01	NA	<9.57E-02	<9.86E-02	NA
RS GEA.Th-228/Pb	<5.18E-02	<5.79E-02	NA	<2.95E-02	<3.10E-02	NA
% SOLIDS	48.61	47.61	NA	5.22E+01	5.23E+01	NA
BULK DEN	1.46E+00	NR	NA	1.19E+00	NR	NA
pH	1.16E+01	1.16E+01	NA	1.14E+01	1.14E+01	NA
Hg	<1.25E-01	<1.25E-01	NA	1.53E-01	1.34E-01	NA
CN.dir	9.49E+01	9.67E+01	NA	5.14E+01	6.13E+01	NA

*: Analytes with a portion of the data below 10 times the DL

*RS: Residual solids from water digestion.

Table A-2. Core 50 Segment Data. (sheet 1 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Segment	50 1R	Mean/DL	50 2	Mean/DL	50 3	Mean/DL
ICP.f.Ag	<4.46E+00 <4.45E+00	NA	<4.50E+00 <4.47E+00	NA	<4.46E+00 <4.46E+00	NA
ICP.f.Al	9.90E+03 9.72E+03	3.85E+02	9.09E+04 9.49E+04	3.64E+03	1.85E+04 2.30E+04	8.12E+02
ICP.f.As	<1.93E+01 <1.93E+01	NA	2.23E+01 <1.94E+01	1.14E+00	<1.93E+01 <1.93E+01	NA
ICP.f.B	6.98E+00 6.92E+00	1.39E+00	<5.00E+00 <4.97E+00	NA	<4.95E+00 <4.95E+00	NA
ICP.f.Be	<1.49E+00 <1.48E+00	NA	<1.50E+00 <1.49E+00	NA	<1.49E+00 <1.49E+00	NA
ICP.f.Bi	2.05E+04 1.83E+04	4.22E+02	2.71E+03 2.52E+03	5.67E+01	1.06E+04 1.14E+04	2.39E+02
ICP.f.Ca	1.06E+03 1.04E+03	4.20E+02	8.95E+02 7.49E+02	3.29E+02	1.10E+03 9.10E+02	4.04E+02
ICP.f.Cd	4.68E+00 6.83E+00	1.65E+00	6.58E+00 6.41E+00	1.86E+00	6.65E+00 6.49E+00	1.88E+00
ICP.f.Ce	1.33E+02 1.13E+02	1.92E+00	<6.40E+01 <6.36E+01	NA	<6.34E+01 7.87E+01	1.23E+00
ICP.f.Cr	3.39E+02 3.27E+02	4.44E+01	2.64E+02 2.52E+02	3.44E+01	2.05E+02 2.33E+02	2.92E+01
ICP.f.Fe	1.94E+04 1.86E+04	2.38E+03	2.03E+04 2.04E+04	2.55E+03	2.25E+04 2.51E+04	2.98E+03
ICP.f.K	NA NA	NA	NA NA	NA	NA NA	NA
ICP.f.La	<9.90E+00 <9.88E+00	NA	<1.00E+01 <9.94E+00	NA	<9.90E+00 <9.90E+00	NA
ICP.f.Li	<2.97E+00 <2.96E+00	NA	4.40E+00 4.34E+00	1.46E+00	3.31E+00 4.69E+00	1.33E+00

IS: Insufficient sample

NR: Not required

NA: Not available

Table A-2. Core 50 Segment Data. (sheet 2 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Segment	50 1R	Mean/DL	50 2	Mean/DL	50 3	Mean/DL
ICP.f.Mg	1.80E+02 1.74E+02	1.18E+02	2.52E+02 2.50E+02	1.67E+02	2.11E+02 2.34E+02	1.49E+02
ICP.f.Mn	9.55E+01 9.52E+01	6.36E+01	2.42E+02 2.29E+02	1.57E+02	1.39E+02 1.59E+02	9.93E+01
ICP.f.Mo	4.80E+00 <4.45E+00	1.07E+00	1.29E+01 1.28E+01	2.87E+00	<4.46E+00 4.49E+00	9.97E-01
ICP.f.Na	1.25E+05 1.30E+05	6.35E+03	5.83E+04 5.21E+04	2.76E+03	1.24E+05 1.21E+05	6.15E+03
ICP.f.Nd	<3.86E+01 <3.85E+01	NA	4.02E+01 7.35E+01	1.46E+00	7.69E+01 8.43E+01	2.07E+00
ICP.f.Ni	9.78E+03 8.07E+03	1.19E+03	4.47E+03 3.47E+03	5.29E+02	4.48E+03 4.67E+03	6.09E+02
ICP.f.P	2.87E+04 3.24 E+04*	8.72E+02	4.34E+03 3.34E+03	1.10E+02	4.45E+04 4.08E+04	1.22E+03
ICP.f.Pb	2.39E+02 2.44E+02	6.21E+00	5.35E+02 5.25E+02	1.36E+01	4.48E+02 5.44E+02	1.27E+01
ICP.f.S	2.96E+03 2.88E+03	1.50E+02	2.95E+03 2.71E+03	1.45E+02	1.29E+03 1.47E+03	7.08E+01
ICP.f.Sb	<7.52E+01 <7.51E+01	NA	7.83E+01 1.90E+02	1.76E+00	1.28E+02 1.18E+02	1.62E+00
ICP.f.Se	<6.98E+01 <6.97E+01	NA	<7.05E+01 <7.01E+01	NA	<6.98E+01 <6.98E+01	NA
ICP.f.Si	9.97E+03 9.72E+03*	5.79E+02	2.23E+03 1.88E+03	1.21E+02	4.67E+03 5.25E+03	2.92E+02
ICP.f.Sm	6.95E+01 6.99E+01	1.34E+00	1.95E+02 1.94E+02	3.73E+00	2.02E+02 1.36E+02	3.25E+00
ICP.f.Sr	4.29E+02 4.14E+02	2.81E+02	8.53E+02 8.41E+02	5.65E+02	1.02E+03 1.14E+03	7.20E+02

IS: Insufficient sample

NR: Not required

NA: Not available

Table A-2. Core 50 Segment Data. (sheet 3 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Segment	50 1R	Mean/DL	50 2	Mean/DL	50 3	Mean/DL
ICP.f.Ti	<3.47E+00 <3.46E+00	NA	3.98E+01 3.37E+01	1.05E+01	<3.47E+00 3.14E+01	8.97E+00
ICP.f.Ti	<1.24E+02 <1.24E+02	NA	<1.25E+02 <1.24E+02	NA	2.03E+02 <1.24E+02	1.62E+00
ICP.f.Zr	1.24E+01 3.02E+01	3.55E+00	<6.00E+00 <5.96E+00	NA	<5.94E+00 <5.94E+00	NA
Sr90	3.37E+01 2.96E+01	7.03E+03	1.55E+02 1.50E+02	2.54E+04	1.26E+02 1.25E+02	2.09E+04
GEA.Cs -137	7.08E+00 6.97E+00	9.76E+02	1.21E+01 1.15E+01	8.25E+02	5.65E+00 6.43E+00	NA
GEA.Cs -134	<6.10E-03 <5.63E-03	NA	<1.33E-02 <1.43E-02	NA	<6.88E-03 <6.73E-03	NA
GEA.Am -241	<6.69E-02 <6.57E-02	NA	<1.51E-01 <1.54E-01	NA	<8.42E-02 <8.81E-02	NA
GEA.Co -60	<6.24E-03 <6.87E-03	NA	<1.14E-02 <1.27E-02	NA	<5.15E-03 <5.94E-03	NA
GEA.Eu -154	1.08E-01 8.90E-02	4.93E+00	<2.96E-02 <3.14E-02	NA	<1.69E-02 <1.78E-02	NA
GEA.Eu -155	8.92E-02 8.26E-02	6.82E+00	<6.89E-02 <7.01E-02	NA	<3.89E-02 <4.11E-02	NA
GEA.K -40	<1.54E-01 <1.54E-01	NA	<3.14E-01 <3.12E-01	NA	<1.55E-01 <1.55E-01	NA
GEA.CePr -144	<8.82E-02 <8.75E-02	NA	<2.02E-01 <1.98E-01	NA	<1.10E-01 <1.18E-01	NA
GEA.Ru -103	<1.12E-02 <1.13E-02	NA	<2.16E-02 <2.06E-02	NA	<1.04E-02 <1.12E-02	NA
GEA.RuRh -106	<1.63E-01 <1.53E-01	NA	<3.26E-01 <3.11E-01	NA	<1.60E-01 <1.66E-01	NA

IS: Insufficient sample

NR: Not required

NA: Not available

Table A-2. Core 50 Segment Data. (sheet 4 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Segment	50 1R	Mean/DL	50 2	Mean/DL	50 3	Mean/DL
GEA.Th -228/Pb	<1.67E-02 <1.67E-02	NA	<3.49E-02 <3.33E-02	NA	<1.79E-02 <1.93E-02	NA
DSC	NO EXOTH NO EXOTH	NA	NO EXOTH NO EXOTH	NA	NO EXOTH NO EXOTH	NA
TGA(%H ₂ O)	2.70E+01 2.54E+01	NA	4.58E+01 4.03E+01	NA	4.44E+01 4.21E+01	NA
% SOLIDS	8.25E+01 8.15E+01	NA	5.83E+01 5.86E+01	NA	IS IS	NA
BULK DEN	NA IS	NA	1.71E+00 NR	NA	NA NA	NA
CN($\mu\text{g/g}$)	4.43E+01 5.26E+01	1.21E+01	6.56E+01 6.25E+01	1.60E+01	3.92E+01 4.62E+01	1.07E+01
pH	10.3 NA	NA NA	11.2 NA	NA NA	11.4 NA	NA NA

IS: Insufficient sample

NR: Not required

NA: Not available

Table A-3. Core 51 Segment Data. (sheet 1 of 4)
 (Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Seg	51 2	Mean/DL	51 3U	Mean/DL	51 3L	Mean/DL	51 4U	Mean/DL	51 4L	Mean/DL
ICP.f.Ag	4.57E+00 <4.48E+00	1.01E+00	<4.23E+00 <4.16E+00	NA	<4.39E+00 <4.50E+00	NA	6.21E+00 <4.38E+00	1.38E+00	<4.34E+00 <4.32E+00	NA
ICP.f.Al	1.13E+04 1.27E+04	4.71E+02	1.34E+03 1.15E+03	4.86E+01	5.86E+02 7.90E+02	2.70E+01	2.22E+03 2.32E+03	8.90E+01	9.27E+03 9.16E+03	3.61E+02
ICP.f.As	<1.94E+01 <1.94E+01	NA	<1.83E+01 <1.80E+01	NA	<1.90E+01 <1.95E+01	NA	<1.88E+01 <1.90E+01	NA	<1.88E+01 <1.87E+01	NA
ICP.f.B	<4.98E+00 <4.98E+00	NA	<4.70E+00 <4.62E+00	NA	<4.87E+00 <5.00E+00	NA	<4.81E+00 <4.86E+00	NA	8.64E+00 <4.80E+00	1.73E+00
ICP.f.Be	<1.49E+00 <1.49E+00	NA	<1.41E+00 <1.39E+00	NA	<1.46E+00 <1.50E+00	NA	<1.44E+00 <1.46E+00	NA	<1.45E+00 <1.44E+00	NA
ICP.f.Bi	9.86E+02 9.87E+02	2.15E+01	1.00E+03 8.66E+02	2.03E+01	1.09E+03 1.04E+03	2.33E+01	3.51E+03 3.73E+03	7.87E+01	1.96E+04 1.75E+04	4.02E+02
ICP.f.Ca	2.16E+03 2.03E+03	8.36E+02	9.63E+02 9.59E+02	3.84E+02	9.56E+02 1.02E+03	3.96E+02	1.40E+03 1.46E+03	5.72E+02	2.06E+03 2.81E+03	9.76E+02
ICP.f.Cd	4.06E+01 1.67E+01	8.17E+00	7.84E+00 7.60E+00	2.21E+00	7.52E+00 6.53E+00	2.01E+00	8.27E+00 1.04E+01	2.67E+00	5.57E+00 7.51E+00	1.87E+00
ICP.f.Ce	<6.37E+01 <6.37E+01	NA	<6.02E+01 <5.91E+01	NA	<6.24E+01 <6.40E+01	NA	6.79E+01 <6.23E+01	1.06E+00	2.12E+02 2.26E+02	3.42E+00
ICP.f.Cr	3.70E+02 3.41E+02	4.73E+01	3.21E+02 2.80E+02	4.01E+01	3.92E+02 3.95E+02	5.24E+01	4.80E+02 5.04E+02	6.56E+01	3.55E+02 3.42E+02	4.65E+01
ICP.f.Fe	3.46E+04 3.87E+04	4.59E+03	3.12E+04 2.58E+04	3.56E+03	3.42E+04 3.43E+04	4.29E+03	3.40E+04 3.61E+04	4.39E+03	2.00E+04 1.93E+04	2.46E+03
ICP.f.K	NA NA	NA								
ICP.f.La	<9.96E+00 <9.96E+00	NA	<9.40E+00 <9.24E+00	NA	<9.75E+00 <1.00E+01	NA	<9.62E+00 <9.73E+00	NA	<9.65E+00 <9.60E+00	NA
ICP.f.Li	8.79E+00 8.75E+00	2.92E+00	7.74E+00 6.86E+00	2.43E+00	8.64E+00 7.03E+00	2.61E+00	8.95E+00 8.50E+00	2.91E+00	<2.90E+00 2.89E+00	9.63E-01

IS: Insufficient Sample

NA: Not Available

NR: Not Required

Table A-3. Core 51 Segment Data. (sheet 2 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Seg	51 2	Mean/DL	51 3U	Mean/DL	51 3L	Mean/DL	51 4U	Mean/DL	51 4L	Mean/DL
ICP.f. Mg	5.47E+02 5.33E+02	3.60E+02	2.86E+02 2.42E+02	1.76E+02	2.58E+02 2.51E+02	1.69E+02	3.37E+02 3.52E+02	2.29E+02	2.14E+02 2.27E+02	1.47E+02
ICP.f. Mn	8.24E+02 9.33E+02	5.86E+02	2.92E+02 2.67E+02	1.87E+02	2.78E+02 2.85E+02	1.88E+02	2.50E+02 2.68E+02	1.73E+02	9.89E+01 9.96E+01	6.61E+01
ICP.f. Mo	2.06E+01 1.82E+01	4.31E+00	1.22E+01 1.25E+01	2.76E+00	9.53E+00 1.17E+01	2.36E+00	7.13E+00 6.93E+00	1.56E+00	5.27E+00 7.30E+00	1.40E+00
ICP.f. Na	7.20E+04 7.02E+04	3.56E+03	1.02E+05 1.14E+05	5.40E+03	7.89E+04 7.69E+04	3.90E+03	8.80E+04 7.69E+04	4.12E+03	1.20E+05 1.24E+05	6.10E+03
ICP.f. Nd	1.07E+02 7.91E+01	2.38E+00	1.17E+02 1.20E+02	3.03E+00	1.98E+02 1.40E+02	4.33E+00	1.47E+02 1.36E+02	3.64E+00	<3.76E+01 7.12E+01	1.83E+00
ICP.f. Ni	1.19E+04 1.23E+04	1.61E+03	1.70E+03 9.68E+02	1.77E+02	1.07E+03 6.37E+02	1.14E+02	9.83E+02 7.38E+02	1.15E+02	1.02E+04 8.49E+03	1.25E+03
ICP.f. P	5.35E+03 5.31E+03	1.52E+02	2.13E+04 2.89E+04*	7.17E+02	7.88E+03 7.35E+03	2.17E+02	1.27E+04 6.73E+03	2.77E+02	3.10E+04* 3.39E+04*	9.26E+02
ICP.f. Pb	1.34E+03 1.32E+03	3.41E+01	1.55E+03 1.45E+03	3.85E+01	8.95E+02 1.08E+03	2.53E+01	9.65E+02 1.04E+03	2.56E+01	2.78E+02 2.49E+02	6.77E+00
ICP.f. S	4.35E+03 4.23E+03	2.20E+02	3.64E+03 3.25E+03	1.76E+02	4.26E+03 4.27E+03	2.19E+02	4.26E+03 4.47E+03	2.24E+02	3.11E+03 3.06E+03	1.58E+02
ICP.f. Sb	1.08E+02 7.74E+01	1.22E+00	1.15E+02 1.10E+02	1.47E+00	2.30E+02 1.60E+02	2.57E+00	1.81E+02 1.83E+02	2.39E+00	<7.34E+01 9.42E+01	1.24E+00
ICP.f. Se	<7.02E+01 <7.02E+01	NA	<6.63E+01 <6.52E+01	NA	<6.87E+01 <7.05E+01	NA	<6.78E+01 <6.86E+01	NA	<6.81E+01 <6.77E+01	NA
ICP.f. Si	5.74E+03 4.44E+03	2.99E+02	1.10E+03 8.63E+02	5.77E+01	9.25E+02 1.14E+03	6.06E+01	2.68E+03 2.78E+03	1.61E+02	1.01E+04* 9.87E+03*	5.87E+02
ICP.f. Sm	1.35E+02 9.74E+01	2.23E+00	2.39E+02 1.03E+02	3.29E+00	3.05E+02 <5.20E+01	5.87E+00	1.94E+02 2.35E+02	4.12E+00	<5.02E+01 6.24E+01	1.20E+00
ICP.f. Sr	1.06E+03 1.02E+03	6.93E+02	1.17E+03 1.03E+03	7.33E+02	1.35E+03 1.35E+03	9.00E+02	1.56E+03 1.65E+03	1.07E+03	4.21E+02 4.09E+02	2.77E+02

* Outside instrument calibration range

IS: Insufficient Sample

NA: Not Available

NR: Not Required

Table A-3. Core 51 Segment Data. (sheet 3 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Seg	51 2	Mean/DL	51 3U	Mean/DL	51 3L	Mean/DL	51 4U	Mean/DL	51 4L	Mean/DL
ICP.f.Ti	9.75E+01 7.61E+01	2.48E+01	2.30E+01 2.20E+01	6.43E+00	<3.41E+00 <3.50E+00	NA	<3.37E+00 <3.40E+00	NA	<3.38E+00 4.51E+00	1.29E+00
ICP.f.Ti	<1.25E+02 <1.25E+02	NA	1.67E+02 <1.16E+02	1.34E+00	<1.22E+02 <1.25E+02	NA	<1.20E+02 <1.22E+02	NA	<1.21E+02 <1.20E+02	NA
ICP.f.Zr	<5.98E+00 4.82E+01	8.04E+00	1.94E+01 1.19E+01	2.62E+00	1.10E+01 <6.00E+00	1.83E+00	4.58E+01 4.56E+01	7.62E+00	1.79E+01 3.59E+01	4.48E+00
Sr90	1.88E+02 1.92E+02	6.79E+04	2.12E+02 1.90E+02	1.06E+04	2.44E+02 2.40E+02	1.27E+04	2.21E+02 2.32E+02	1.19E+04	2.69E+01 2.80E+01	1.31E+03
GEA.Cs -137	9.77E+01 1.02E+02	1.26E+04	1.62E+01 1.44E+01	1.94E+03	1.71E+01 1.70E+01	2.16E+02	1.67E+01 1.90E+01	2.26E+03	1.37E+01 1.35E+01	1.72E+03
GEA.Cs -134	<1.39E-02 <1.51E-02	NA	<8.79E-03 <9.19E-03	NA	<1.14E-02 <1.02E-02	NA	<1.05E-02 <1.00E-02	NA	<8.49E-03 <9.06E-03	NA
GEA.Am -241	<2.13E-01 <2.16E-01	NA	<1.36E-01 <1.26E-01	NA	<1.48E-01 <1.51E-01	NA	<1.39E-01 <1.48E-01	NA	<8.58E-02 <8.34E-02	NA
GEA.Co -60	<7.10E-03 <1.05E-02	NA	<8.93E-03 <8.78E-03	NA	3.76E-02 <8.00E-03	2.65E+00	<8.89E-03 2.64E-02	1.86E+00	<8.92E-03 <7.72E-03	NA
GEA.Eu -154	3.10E-01 3.17E-01	1.31E+01	<2.07E-02 <2.11E-02	NA	4.37E-02 <2.59E-02	8.92E-01	<1.85E-02 <2.52E-02	NA	<2.12E-02 <2.11E-02	NA
GEA.Eu -155	<1.40E-01 <1.41E-01	NA	<8.60E-02 <8.09E-02	NA	<9.35E-02 <9.49E-02	NA	<8.79E-02 <9.49E-02	NA	<5.35E-02 <5.32E-02	NA
GEA.K-40	<2.86E-01 <2.93E-01	NA	<2.76E-01 <2.72E-01	NA	<2.82E-01 <2.93E-01	NA	<2.82E-01 <2.83E-01	NA	<2.87E-01 <2.78E-01	NA
GEA.CeP r-144	<5.10E-01 <5.13E-01	NA	<2.92E-01 <2.71E-01	NA	<3.17E-01 <3.10E-01	NA	<2.97E-01 <3.16E-01	NA	<1.98E-01 <1.97E-01	NA
GEA.Ru -103	<6.13E-02 <6.28E-02	NA	<2.49E-02 <2.36E-02	NA	<2.64E-02 <2.71E-02	NA	<2.71E-02 <2.74E-02	NA	<2.33E-02 <2.26E-02	NA
GEA.RuR h-106	<6.18E-01 <6.53E-01	NA	<2.88E-01 <2.68E-01	NA	<2.97E-01 <2.88E-01	NA	<2.85E-01 <3.03E-01	NA	<2.57E-01 <2.53E-01	NA

IS: Insufficient Sample

NA: Not Available

NR: Not Required

Table A-3. Core 51 Segment Data. (sheet 4 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Seg	51 2	Mean/DL	51 3U	Mean/DL	51 3L	Mean/DL	51 4U	Mean/DL	51 4L	Mean/DL
GEA.Th -228/Pb	<8.80E-02 <8.92E-02	NA	<4.21E-02 <3.85E-02	NA	<4.30E-02 <4.50E-02	NA	<4.23E-02 <4.42E-02	NA	<3.46E-02 <3.32E-02	NA
DSC	NO EXOTH NO EXOTH	NA	NO EXOTH NO EXOTH	NA						
TGA(%H 2O)	6.01E+01 5.85E+01	NA	5.94E+01 5.99E+01	NA	5.43E+01 5.42E+01	NA	5.48E+01 5.46E+01	NA	5.28E+01 5.34E+01	NA
% SOLIDS	4.03E+01 3.92E+01	NA	4.40E+01 4.57E+01	NA	4.67E+01 4.75E+01	NA	4.45E+01 4.56E+01	NA	5.01E+01 5.08E+01	NA
BULK DEN	IS NR	NA	1.49E+00 NR	NA	1.70E+00 NR	NA	1.48E+00 NR	NA	1.53E+00 NR	NA
CN ($\mu\text{g/g}$)	9.57E+01 9.48E+01	2.38E+01	1.10E+02 1.09E+02	2.74E+01	1.03E+02 1.02E+02	2.56E+01	9.42E+01 8.88E+01	2.29E+01	5.74E+01 5.72E+01	NA
pH	10.6 NA	NA	11.4 NA	NA	11.4 NA	NA	11.2 NA	NA	11.6 NA	NA

IS: Insufficient Sample

NA: Not Available

NR: Not Required

Table A-4. Core 52 Segment Data. (sheet 1 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Segment	52 1	Mean/DL	52 2	Mean/DL	52 3U	Mean/DL	52 3L	Mean/DL
ICP.f.Ag	<2.46E+01 <2.46E+01	NA	<4.48E+00 <4.48E+00	NA	4.72E+00 5.45E+00	1.13E+00	5.31E+00 5.09E+00	1.16E+00
ICP.f.Al	2.12E+05 2.15E+05	1.16E+04	4.63E+04 3.98E+04	1.69E+03	7.81E+03 8.53E+03	3.20E+02	1.52E+04 1.56E+04	6.04E+02
ICP.f.As	<1.48E+02 <1.47E+02	NA	2.35E+01 <1.94E+01	1.20E+00	<1.93E+01 <1.92E+01	NA	<1.94E+01 <1.92E+01	NA
ICP.f.B	<4.92E+01 <4.91E+01	NA	<4.98E+00 <4.98E+00	NA	<4.96E+00 <4.92E+00	NA	8.02E+00 1.18E+01	1.98E+00
ICP.f.Be	<1.48E+01 <1.47E+01	NA	<1.49E+00 <1.49E+00	NA	<1.49E+00 <1.48E+00	NA	<1.49E+00 <1.48E+00	NA
ICP.f.Bi	<3.10E+02 3.76E+02	1.19E+01	1.15E+04 1.07E+04	2.41E+02	9.58E+03 1.09E+04	2.24E+02	2.21E+04 2.26E+04	4.85E+02
ICP.f.Ca	1.35E+04 8.30E+03	2.42E+03	6.71E+02 8.71E+02	3.08E+02	5.83E+02 1.02E+03	3.20E+02	4.37E+02 4.07E+02	1.69E+02
ICP.f.Cd	<2.95E+01 <2.95E+01	NA	4.59E+00 5.19E+00	1.40E+00	6.22E+00 4.78E+00	1.57E+00	5.92E+00 6.08E+00	1.71E+00
ICP.f.Ce	<3.35E+02 <3.34E+02	NA	2.02E+02 1.58E+02	2.81E+00	2.07E+02 2.35E+02	3.45E+00	3.76E+02 3.08E+02	5.34E+00
ICP.f.Cr	1.23E+02 1.61E+02	3.16E+01	2.56E+02 2.42E+02	3.32E+01	2.75E+02 2.96E+02	3.81E+01	5.45E+02 5.16E+02	7.08E+01
ICP.f.Fe	4.77E+04 3.34E+04	5.40E+03	2.06E+04 2.15E+04	2.62E+03	2.45E+04 2.23E+04	2.92E+03	1.90E+04 1.90E+04	2.38E+03
ICP.f.K	NA NA	NA	NA NA	NA	NA NA	NA	NA NA	NA
ICP.f.La	<8.37E+01 <8.35E+01	NA	<9.96E+00 <9.96E+00	NA	<9.92E+00 <9.84E+00	NA	<9.94E+00 <9.86E+00	NA
ICP.f.Li	<1.97E+01 <1.96E+01	NA	6.21E+00 5.80E+00	2.00E+00	7.88E+00 8.64E+00	2.75E+00	5.55E+00 4.57E+00	1.69E+00

IS: Insufficient Sample

NR: Not Required

NA: Not Available

Table A-4. Core 52 Segment Data. (sheet 2 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Segment	52 1	Mean/DL	52 2	Mean/DL	52 3U	Mean/DL	52 3L	Mean/DL
ICP.f. Mg	4.07E+02 4.43E+02	2.83E+02	1.93E+02 1.66E+02	1.20E+02	2.02E+02 1.92E+02	1.31E+02	1.56E+02 1.61E+02	1.06E+02
ICP.f.Mn	1.85E+03 3.28E+03	1.71E+03	2.26E+02 2.06E+02	1.44E+02	1.75E+02 1.58E+02	1.11E+02	9.94E+01 8.57E+01	6.17E+01
ICP.f. Mo	<2.95E+01 <2.95E+01	NA	1.37E+01 1.31E+01	2.98E+00	9.82E+00 5.74E+00	1.73E+00	9.77E+00 8.21E+00	2.00E+00
ICP.f.Na	2.65E+04 2.80E+04	8.81E+02	9.85E+04 1.12E+05	5.25E+03	1.35E+05 1.27E+05	6.55E+03	1.08E+05 1.06E+05	5.35E+03
ICP.f.Nd	<4.04E+02 <4.03E+02	NA	7.68E+01 <3.88E+01	1.97E+00	5.97E+01 9.63E+01	2.00E+00	7.07E+01 4.94E+01	1.54E+00
ICP.f.Ni	NA NA	NA	2.61E+03 3.22E+03	3.89E+02	3.44E+03 1.06E+03	3.00E+02	8.13E+03 1.07E+04	1.26E+03
ICP.f.P	<2.26E+02 <2.26E+02	NA	2.22E+04* 2.91E+04*	7.31E+02	3.87E+04 3.50E+04*	1.05E+03	2.55E+04 2.64E+04*	7.43E+02
ICP.f.Pb	1.18E+03 2.50E+03	5.94E+01	4.44E+02 3.95E+02	1.08E+01	4.91E+02 4.12E+02	1.16E+01	1.44E+02 1.19E+02	3.38E+00
ICP.f.S	1.08E+03 1.15E+03	6.79E+01	2.64E+03 2.51E+03	1.32E+02	2.47E+03 2.63E+03	1.31E+02	3.61E+03 3.53E+03	1.83E+02
ICP.f.Sb	<1.03E+03 <1.03E+03	NA	8.28E+01 <7.57E+01	1.09E+00	1.13E+02 1.27E+02	1.58E+00	1.42E+02 <7.50E+01	1.86E+00
ICP.f.Se	<4.28E+02 <4.27E+02	NA	<7.02E+01 <7.02E+01	NA	<6.99E+01 <6.94E+01	NA	<7.01E+01 <6.95E+01	NA
ICP.f.Si	4.60E+03 4.72E+03	5.82E+02	5.89E+03 5.19E+03	3.26E+02	4.68E+03 5.09E+03	2.87E+02	1.14E+04 1.11E+04*	6.65E+02
ICP.f. Sm	<4.63E+02 <4.62E+02	NA	1.10E+02 9.71E+01	2.00E+00	1.06E+02 1.02E+02	2.00E+00	<5.17E+01 <5.13E+01	NA
ICP.f.Sr	4.67E+01 5.06E+01	3.24E+01	6.81E+02 6.16E+02	4.32E+02	9.60E+02 9.30E+02	6.30E+02	3.72E+02 2.91E+02	2.21E+02

IS: Insufficient Sample

NR: Not Required

NA: Not Available

Table A-4. Core 52 Segment Data. (sheet 3 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Segment	52 1	Mean/DL	52 2	Mean/DL	52 3U	Mean/DL	52 3L	Mean/DL
ICP.f.Ti	1.48E+02 2.75E+02	1.41E+02	2.24E+01 7.89E+00	4.31E+00	4.89E+00 4.30E+00	1.31E+00	8.96E+00 1.39E+01	3.26E+00
ICP.f.TI	<8.07E+02 <8.06E+02	NA	<1.25E+02 <1.25E+02	NA	<1.24E+02 <1.23E+02	NA	<1.24E+02 <1.23E+02	NA
ICP.f.Zr	9.72E+01 1.78E+02	3.91E+01	9.29E+00 1.36E+01	1.92E+00	1.68E+02 9.00E+01	2.15E+01	1.11E+02 2.52E+02	3.02E+01
Sr90	1.79E+02 1.51E+02	1.65E+02	9.22E+01 8.37E+01	3.26E+02	9.25E+01 9.92E+01	3.55E+02	1.96E+01 1.51E+01	6.43E+01
GEA.Cs -137	1.08E+01 1.09E+01	1.57E+03	1.09E+01 9.66E+00	3.45E+02	7.39E+00 8.26E+00	2.63E+02	1.06E+01 1.07E+01	3.57E+02
GEA.Cs -134	<9.00E-03 <8.14E-03	NA	<2.46E-02 <2.25E-02	NA	<2.09E-02 <2.40E-02	NA	<2.19E-02 <2.54E-02	NA
GEA.Am -241	2.53E-01 <9.49E-02	4.52E+00	<5.28E-02 <4.98E-02	NA	<5.21E-02 <5.06E-02	NA	<3.61E-02 <3.58E-02	NA
GEA.Co -60	<6.39E-03 <6.39E-03	NA	<2.51E-02 <2.07E-02	NA	<2.13E-02 <1.90E-02	NA	<2.20E-02 <2.54E-02	NA
GEA.Eu -154	1.21E+00 9.44E-01	5.98E+01	<6.48E-02 <7.37E-02	NA	<7.74E-02 <7.67E-02	NA	<6.76E-02 <6.42E-02	NA
GEA.Eu -155	1.07E+00 7.69E-01	7.07E+01	<8.27E-02 <8.07E-02	NA	<7.89E-02 <8.11E-02	NA	<6.86E-02 <6.66E-02	NA
GEA.K-40	1.70E-01 <1.55E-01	5.31E-01	<7.17E-01 <7.32E-01	NA	<7.24E-01 <7.08E-01	NA	<7.20E-01 <7.20E-01	NA
GEA.Ce Pr-144	<1.47E-01 <1.33E-01	NA	<3.19E-01 <3.13E-01	NA	<3.05E-01 <3.14E-01	NA	<2.84E-01 <2.82E-01	NA
GEA.Ru -103	<1.46E-02 <1.42E-02	NA	<3.83E-02 <3.40E-02	NA	<3.37E-02 <3.41E-02	NA	<3.63E-02 <3.56E-02	NA
GEA.Ru Rh-106	<2.19E-01 <2.10E-01	NA	<5.03E-01 <5.03E-01	NA	<4.39E-01 <4.28E-01	NA	<4.74E-01 <4.79E-01	NA

IS: Insufficient Sample

NR: Not Required

NA: Not Available

Table A-4. Core 52 Segment Data. (sheet 4 of 4)
 (Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Segment	52 1	Mean/DL	52 2	Mean/DL	52 3U	Mean/DL	52 3L	Mean/DL
DSC	NO EXOTH NO EXOTH	NA	NO EXOTH NR	NA	NO EXOTH NR	NA	NO EXOTH NO EXOTH	NA
TGA (%H ₂ O)	1.52E+01 1.53E+01	NA	5.56E+01 5.54E+01	NA	5.46E+01 5.46E+01	NA	5.48E+01 4.97E+01	NA
% SOLIDS	8.34E+01 8.33E+01	NA	5.11E+01 5.19E+01	NA	4.85E+01 4.87E+01	NA	4.66E+01 4.65E+01	NA
BULK DEN	IS NR	NA	1.55E+00 NR	NA	1.50E+00 NR	NA	1.52E+00 NR	NA
CN ($\mu\text{g/g}$)	3.15E+01 3.05E+01	7.75E+00	6.34E+01 6.00E+01	1.54E+01	5.05E+01 5.37E+01	1.30E+01	4.14E+01 4.57E+01	NA
pH	10.5 NA	NA	11.4 NA	NA	11.8 NA	NA	10.9 NA	NA

IS: Insufficient Sample

NR: Not Required

NA: Not Available

Table A-5. Drainable Liquid Core Composite Data, Acidified Samples. (sheet 1 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	50 1	50 2	Mean/DL	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
Ag	<9.00E-02	<9.00E-02	NA	2.54E-01	2.62E-01	2.87E+00	<2.25E-01	<2.25E-01	NA
Al*	4.45E+00	4.06E+00	8.33E+00	1.11E+01	1.18E+01	2.25E+01	4.72E+01	4.84E+01	3.75E+01
As	<3.90E-01	<3.90E-01	NA	<3.90E-01	<3.90E-01	NA	<9.75E-01	<9.75E-01	NA
B	9.33E+00	9.03E+00	9.18E+01	2.42E+01	2.50E+01	2.46E+02	2.83E+01	3.20E+01	1.21E+02
Be	<3.00E-02	<3.00E-02	NA	<3.00E-02	<3.00E-02	NA	<7.50E-02	<7.50E-02	NA
Bi	<9.20E-01	<9.20E-01	NA	<9.20E-01	<9.20E-01	NA	1.69E+01	1.70E+01	7.40E+00
Ca	4.87E+00	3.70E+00	8.56E+01	7.00E+00	2.95E+00	9.94E+01	3.48E+00	4.40E+00	3.15E+01
Cd	<7.00E-02	<7.00E-02	NA	<7.00E-02	<7.00E-02	NA	<1.75E-01	<1.75E-01	NA
Ce	<1.28E+00	<1.28E+00	NA	<1.28E+00	<1.28E+00	NA	<3.20E+00	<3.20E+00	NA
Cr	4.56E+01	4.50E+01	3.02E+02	2.71E+02	2.75E+02	1.82E+03	1.86E+02	1.90E+02	5.00E+02
Fe	5.82E+00	5.67E+00	3.59E+01	4.75E+01	4.85E+01	3.00E+02	1.89E+01	1.92E+01	4.76E+01
K	4.68E+01	4.66E+01	3.16E+01	3.60E+02	3.74E+02	2.48E+02	1.37E+02	1.39E+02	3.73E+01
La	<2.00E-01	<2.00E-01	NA	<2.00E-01	<2.00E-01	NA	<5.00E-01	<5.00E-01	NA
Li	<6.00E-02	<6.00E-02	NA	<6.00E-02	<6.00E-02	NA	<1.50E-01	<1.50E-01	NA
Mg	1.32E+00	1.16E+00	4.13E+01	5.11E-01	3.10E-01	1.37E+00	3.90E-01	3.96E-01	5.24E+00
Mn	<3.00E-02	3.45E-02	1.15E+00	7.56E-02	8.11E-02	2.61E+00	<7.50E-02	<7.50E-02	NA
Mo	2.23E+00	2.14E+00	2.43E+01	2.22E+01	2.26E+01	2.49E+02	2.09E+00	2.16E+00	9.48E+00
Na	1.46E+04	1.46E+04	3.65E+04	9.63E+04	9.47E+04	2.39E+05	5.23E+04	5.15E+04	5.20E+04
Nd*	9.49E-01	1.12E+00	1.32E+00	7.95E-01	1.04E+00	1.18E+00	<1.95E+00	<1.95E+00	NA
Ni	1.79E+00	1.75E+00	1.18E+01	1.59E+01	1.62E+01	1.07E+02	2.75E+00	2.82E+00	7.44E+00
P	7.85E+02	7.95E+02	1.13E+03	2.02E+03	2.03E+03	2.89E+03	2.63E+03	2.55E+03	1.48E+03

*: Analytes with a portion of the data below 10 times the DL

NA: Not available

NR: Not required

IS: Insufficient sample

Table A-5. Drainable Liquid Core Composite Data, Acidified Samples. (sheet 2 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	50 1	50 2	Mean/DL	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
Pb	< 7.80E-01	< 7.80E-01	NA	< 7.80E-01	< 7.80E-01	NA	< 1.95E+00	< 1.95E+00	NA
S	8.31E+02	8.18E+02	2.11E+03	6.03E+03	5.99E+03	1.54E+04	3.35E+03	3.32E+03	3.42E+03
Sb*	1.57E+00	2.27E+00	1.26E+00	2.52E+00	2.52E+00	1.66E+00	<3.80E+00	<3.80E+00	NA
Se	<1.41E+00	<1.41E+00	NA	<1.41E+00	<1.41E+00	NA	<3.52E+00	<3.52E+00	NA
Si	6.08E+01	5.62E+01	1.72E+02	6.58E+01	6.68E+01	1.95E+02	7.82E+01	9.61E+01	1.02E+02
Sm*	2.07E+00	<1.04E+00	1.99E+00	3.40E+00	3.92E+00	3.52E+00	<2.60E+00	<2.60E+00	NA
Sr*	1.40E-01	1.39E-01	4.67E+00	1.75E-01	1.69E-01	5.73E+00	1.80E+00	1.82E+00	2.41E+01
Ti	<7.00E-02	<7.00E-02	NA	<7.00E-02	<7.00E-02	NA	<1.75E-01	<1.75E-01	NA
Tl	<2.50E+00	<2.50E+00	NA	<2.50E+00	3.21E+00	1.28E+00	<6.25E+00	<6.25E+00	NA
Zr*	1.34E-01	1.34E-01	1.12E+00	6.56E-01	6.41E-01	5.41E+00	1.76E+00	1.63E+00	5.68E+00
DSC	NO EXOT	NO EXOT	NA	NO EXOT	NO EXOT	NA	NO EXOT	NO EXOT	NA
TGA (%H ₂ O)	96.5	93.7	NA	73.4	74	NA	83.2	82.6	NA
SPG	1.02	1.02	NA	1.21	1.19	NA	1.10	1.11	NA
TD SOLID	4.1	4.67	NA	24	25.4	NA	13.2	13.7	NA
pH	9.63	9.62	NA	10.7	10.7	NA	10.3	10.3	NA
NH ₄	4.16E+01	4.30E+01	1.06E+00	8.31E+01	8.03E+01	5.19E-01	2.17E+02	2.25E+02	2.76E+00
CN	1.35E+01	1.34E+01	3.36E+01	1.52E+02	1.52E+02	3.80E+02	4.00E+01	3.96E+01	9.95E+01
IC.F-	1.70E+02	1.77E+02	1.74E+03	8.24E+02	8.26E+02	8.25E+03	6.68E+02	6.78E+02	6.73E+03
IC.Cl-	1.99E+02	1.93E+02	9.80E+02	1.34E+03	1.34E+03	6.70E+03	8.51E+02	8.69E+02	4.30E+03
IC.NO ₂ -	2.59E+03	2.57E+03	2.58E+03	2.75E+04	2.78E+04	2.77E+04	7.99E+03	8.12E+03	8.06E+03

*: Analytes with a portion of the data below 10 times the DL

NA: Not available

NR: Not required

IS: Insufficient sample

Table A-5. Drainable Liquid Core Composite Data, Acidified Samples (sheet 3 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	50 1	50 2	Mean/DL	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
IC.PO4-	2.30E+03	2.50E+03	2.40E+03	6.29E+03	6.19E+03	6.24E+03	7.65E+03	7.61E+03	7.63E+03
IC.SO4-	4.64E+03	4.65E+03	4.65E+03	1.68E+04	1.68E+04	1.68E+04	9.58E+03	9.58E+03	9.58E+03
IC.NO3-	2.12E+04	2.12E+04	2.12E+04	1.34E+05	1.35E+05	1.35E+05	1.00E+05	1.00E+05	1.00E+05
NO2	2.83E+03	2.63E+03	1.37E+01	1.31E+04	1.20E+04	1.53E+01	7.57E+03	7.27E+03	1.81E+01
TIC	5.16E+02	5.08E+02	1.02E+02	4.58E+03	4.51E+03	9.09E+02	3.46E+02	3.32E+02	6.78E+01
TOC	1.10E+03	1.20E+03	2.09E+02	1.05E+03	1.08E+03	1.94E+02	3.40E+02	3.69E+02	6.45E+01
AT	9.74E-04	8.54E-04	2.23E+00	1.71E-02	1.61E-02	2.55E+01	5.42E-04	4.80E-04	1.70E+01
U	9.44E+01	9.61E+01	1.02E+01	6.10E+02	5.66E+02	6.13E+00	3.97E+01	4.15E+01	9.23E+00
U-238	3.17E-05	3.23E-05	1.03E+01	2.05E-04	1.90E-04	6.17E+00	1.33E-05	1.39E-05	9.07E+00
Pu239/40	<6.57E-05	<6.60E-05	NA	1.11E-02	1.17E-02	8.51E+01	<6.70E-05	<6.28E-05	NA
Pu-238	<9.01E-05	<9.01E-05	NA	2.44E-03	2.56E-03	1.10E+01	<1.71E-04	<1.14E-04	NA
Am-241	<4.03E-05	<2.67E-05	NA	2.04E-04	NA	1.40E+01	6.79E-05	7.22E-05	1.40E+00
TB	2.54E+00	2.45E+00	3.04E+02	2.57E+01	2.47E+01	2.65E+03	7.50E+00	7.61E+00	5.04E+02
Sr90	1.05E-02	1.11E-02	1.37E+02	1.18E-01	NA	2.00E-01	4.55E-02	4.43E-02	1.40E+03
Tc99	7.93E-03	8.16E-03	5.03E+02	8.58E-02	IS	2.38E+03	1.71E-02	1.62E-02	9.25E+02
C14	1.99E-05	1.60E-05	8.16E+00	3.80E-04	6.56E-04	8.64E+01	6.22E-05	8.55E-05	3.36E+01
H3	2.48E-04	3.43E-04	1.14E+01	9.93E-03	3.96E-03	3.02E+01	4.89E-04	4.52E-04	1.81E+01
GEA.Cs-137	1.70E+00	1.73E+00	2.95E+03	1.80E+01	1.87E+01	1.31E+04	5.06E+00	5.40E+00	3.51E+04
GEA.Cs-134	<4.66E-04	<5.14E-04	NA	<1.40E-03	<1.35E-03	NA	<2.66E-04	<2.72E-04	NA

*: Analytes with a portion of the data below 10 times the DL

NA: Not available

NR: Not required

IS: Insufficient sample

Table A-5. Drainable Liquid Core Composite Data, Acidified Samples. (sheet 4 of 4)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Core Result	50 1	50 2	Mean/DL	51 1	51 2	Mean/DL	52 1	52 2	Mean/DL
GEA.Am-241	<7.33E-03	<7.33E-03	NA	<3.23E-02	<3.30E-02	NA	<5.68E-03	<5.90E-03	NA
GEA.Co-60	<4.06E-04	<5.28E-04	NA	<1.35E-03	<1.10E-03	NA	<1.24E-04	<1.24E-04	NA
GEA.Eu-154	<1.31E-03	<1.44E-03	NA	<4.23E-03	<3.99E-03	NA	<5.50E-04	<5.29E-04	NA
GEA.Eu-155	<3.07E-03	<3.14E-03	NA	<1.47E-02	<1.50E-02	NA	<2.57E-03	<2.65E-03	NA
GEA.K-40	<1.30E-02	<1.29E-02	NA	<3.04E-02	<3.04E-02	NA	3.73E-03	<3.35E-03	NA
GEA.Ce Pr-144	<9.99E-03	<1.01E-02	NA	<4.78E-02	<4.88E-02	NA	<8.34E-03	<8.61E-03	NA
GEA.Ru-103	<1.50E-03	<1.52E-03	NA	<7.17E-03	<7.34E-03	NA	<1.27E-03	<1.30E-03	NA
GEA.RuR h-106	<2.08E-02	<2.07E-02	NA	<9.32E-02	<9.51E-02	NA	<1.63E-02	<1.68E-02	NA
GEA.Th-228/Pb	<2.08E-03	<2.10E-03	NA	<1.02E-02	<1.03E-02	NA	<1.78E-03	<1.84E-03	NA
I-129	<7.48E-05	<8.26E-05	NA	<3.85E-05	<3.72E-05	NA	<3.79E-05	<3.84E-05	NA

*: Analytes with a portion of the data below 10 times the DL

NA: Not available

NR: Not required

IS: Insufficient sample

Table A-6. Homogenization Test Data. (sheet 1 of 3) (Units $\mu\text{g/g}$)

Analyte	Core 50 Seg. 2 Loc. 1 Aliquot 2	Mean/DL	Core 50 Seg. 2 Loc. 2 Aliquot 2	Mean/DL
ICP.a.Al	1.32E+05 7.90E+04	2.86E+04	9.58E+04 1.22E+05	2.95E+04
ICP.a.Bi	4.03E+03 2.80E+03	5.43E+02	3.87E+03 3.61E+03	5.94E+02
ICP.a.Ca	1.52E+03 1.47E+03	1.67E+03	4.69E+03 9.42E+02	3.13E+03
ICP.a.Cd	7.52E+00 5.21E+00	1.06E+01	6.94E+00 6.84E+00	1.15E+01
ICP.a.Cr	3.29E+02 2.33E+02	3.12E+02	3.14E+02 2.89E+02	3.34E+02
ICP.a.Fe	3.18E+04 2.10E+04	1.76E+04	3.15E+04 2.93E+04	2.03E+04
ICP.a.K	2.81E+02 1.77E+02	1.97E+01	2.51E+02 2.27E+02	2.06E+01
ICP.a.Li	7.47E+00 5.09E+00	1.57E+01	7.09E+00 6.46E+00	1.69E+01
ICP.a.Mg	2.82E+02 1.94E+02	7.93E+02	3.27E+02 2.46E+02	9.57E+02
ICP.a.Mn	3.16E+02 2.25E+02	9.03E+02	3.25E+02 3.19E+02	1.07E+03
ICP.a.Mo	1.51E+01 1.07E+01	2.15E+01	1.35E+01 1.30E+01	2.20E+01
ICP.a.Na	8.93E+04 6.32E+04	1.23E+04	8.26E+04 7.68E+04	1.29E+04
ICP.a.Ni	1.77E+02 1.26E+02	1.16E+02	1.77E+02 1.72E+02	1.34E+02
ICP.a.P	8.17E+03 4.05E+03	1.33E+03	4.12E+03 5.79E+03	1.08E+03
ICP.a.Pb	7.63E+02 5.34E+02	1.05E+02	7.33E+02 6.65E+02	1.13E+02
ICP.a.S	4.32E+03 3.42E+03	1.17E+03	4.49E+03 4.86E+03	1.42E+03
ICP.a.Sm	3.76E+02 2.03E+02	3.07E+01	3.37E+01 3.34E+02	1.96E+01
ICP.a.Sr	1.21E+03 8.32E+02	3.40E+03	1.20E+03 1.14E+03	3.90E+03

*: Analytes with a portion of the data below 10 times the DL.

Table A-6. Homogenization Test Data. (sheet 2 of 3) (Units $\mu\text{g/g}$)

Analyte	Core 51 Seg. 3L Loc. 1 Aliquot 2	Mean/DL	Core 51 Seg. 3L Loc. 2 Aliquot 2	Mean/DL
ICP.a.Al	9.15E+02 IS	2.47E+02	4.38E+02 3.85E+02	1.11E+02
ICP.a.Bi	1.92E+03 IS	3.05E+02	1.53E+03 1.39E+03	2.32E+02
ICP.a.Ca	1.09E+03 IS	1.21E+03	9.06E+02 8.43E+02	9.72E+02
ICP.a.Cd*	1.23E+01 IS	2.05E+01	7.47E+00 6.62E+00	1.18E+01
ICP.a.Cr	4.94E+02 IS	5.49E+02	4.15E+02 3.87E+02	4.46E+02
ICP.a.Fe	6.91E+04 IS	4.61E+04	4.25E+04 4.00E+04	2.75E+04
ICP.a.K	4.35E+02 IS	3.75E+01	3.53E+02 3.37E+02	2.97E+01
ICP.a.Li	1.30E+01 IS	3.25E+01	9.79E+00 9.10E+00	2.36E+01
ICP.a.Mg	3.23E+02 IS	1.08E+03	2.73E+02 2.55E+02	8.80E+02
ICP.a.Mn	4.34E+02 IS	1.45E+03	3.23E+02 3.00E+02	1.04E+03
ICP.a.Mo*	1.29E+01 IS	2.15E+01	9.93E+00 1.01E+01	1.67E+01
ICP.a.Na	1.13E+05 IS	1.82E+04	9.57E+04 9.89E+04	1.57E+04
ICP.a.Ni	2.99E+02 IS	2.30E+02	2.44E+02 2.26E+02	1.81E+02
ICP.a.P	6.33E+03 IS	1.38E+03	7.46E+03 9.84E+03	1.88E+03
ICP.a.Pb	1.66E+03 IS	2.68E+02	1.34E+03 1.23E+03	2.08E+02
ICP.a.S	5.92E+03 IS	1.79E+03	4.92E+03 4.51E+03	1.43E+03
ICP.a.Sm	1.12E+03 IS	1.19E+02	9.23E+02 8.86E+02	9.63E+01
ICP.a.Sr	2.03E+03 IS	6.77E+03	1.71E+03 1.60E+03	5.50E+03

IS: Insufficient Sample

*: Analytes with a portion of the data below 10 times the DL.

Table A-6. Homogenization Test Data. (sheet 3 of 3) (Units $\mu\text{g/g}$)

Ana-lyte	Core 52 Seg. 3L Loc. 1 Aliquot 2	Mean/DL	Core 52 Seg. 3L Loc. 2 Aliquot 2	Mean/DL
ICP.a. Al	1.60E+04 1.64E+04	4.38E+03	1.85E+04 1.87E+04	5.03E+03
ICP.a. Bi	2.37E+04 2.49E+04	3.86E+03	2.78E+04 2.80E+04	4.43E+03
ICP.a. Ca	3.75E+02 4.05E+02	4.33E+02	4.20E+02 4.09E+02	4.60E+02
ICP.a. Cd	3.99E+00 3.10E+00	5.90E+00	3.94E+00 4.07E+00	6.67E+00
ICP.a. Cr	5.32E+02 5.63E+02	6.09E+02	6.23E+02 6.08E+02	6.84E+02
ICP.a. Fe	1.88E+04 1.96E+04	1.28E+04	2.23E+04 2.23E+04	1.49E+04
ICP.a. K	2.18E+02 2.39E+02	1.97E+01	2.10E+02 2.17E+02	1.84E+01
ICP.a. Li*	1.39E+00 1.48E+00	3.60E+00	1.58E+00 1.57E+00	3.92E+00
ICP.a. Mg	1.49E+02 1.50E+02	4.97E+02	1.63E+02 1.59E+02	5.37E+02
ICP.a. Mn	5.64E+01 6.01E+01	1.94E+02	6.83E+01 6.55E+01	2.23E+02
ICP.a. Mo*	3.61E+00 3.81E+00	6.18E+00	4.05E+00 3.87E+00	6.60E+00
ICP.a. Na	1.16E+05 1.08E+05	1.81E+04	1.25E+05 1.25E+05	2.02E+04
ICP.a. Ni	3.36E+01 3.39E+01	2.60E+01	3.83E+01 3.76E+01	2.92E+01
ICP.a. P	2.80E+04* 2.42E+04*	5.67E+03	2.76E+04* 2.74E+04*	5.98E+03
ICP.a. Pb	1.44E+02 1.46E+02	2.34E+01	1.66E+02 1.62E+02	2.65E+01
ICP.a. S	3.43E+03 3.51E+03	1.05E+03	3.84E+03 3.84E+03	1.16E+03
ICP.a. Sm*	2.85E+01 2.75E+01	2.98E+00	3.64E+01 3.79E+01	3.95E+00
ICP.a. Sr	3.90E+02 3.29E+02	1.20E+03	3.35E+02 3.19E+02	1.09E+03

*: Sample absorbance outside linear range of instrument.

*: Analytes with a portion of the data below 10 times the DL.

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APPENDIX B
MEAN CONCENTRATION CALCULATION METHODS

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This appendix contains a description of the statistical models that describes the structure of the data from core samples taken from tank 241-T-107. Equations are also presented for estimates of the mean concentration, the variance for the mean concentration, and the CI on the mean concentration.

The statistical model that describes the structure of the core composite data or the drainable liquid data is

$$y_{ij} = \mu + C_i + A_{ij}, \quad i = 1, \dots, a, \quad j = 1, \dots, n_i, \quad (1)$$

where

- y_{ij} = laboratory results from the j^{th} duplicate of the i^{th} core composite sample or drainable liquid sample from the tank,
- μ = the grand mean of all the data,
- C_i = the effect of the i^{th} core (spatial effect),
- A_{ij} = the analytical error associated with the j^{th} duplicate from the i^{th} core,
- a = the number of cores,
- n_i = the number of analytical results from the i^{th} core.

This is an unbalanced one-way random effects analysis of variance model. The C_i variable is assumed to be a random effects. It is assumed that C_i and A_{ij} are each distributed normally with mean zero and variances of $\sigma^2(C)$, and $\sigma^2(A)$, respectively. Estimates of $\sigma^2(C)$ and $\sigma^2(A)$ were obtained using Restricted Maximum Likelihood Estimation (REML). This method applied to variance component estimation is described by Harville (1977).

An estimate of the true unknown mean concentration μ is the mean of the core means; i.e., each core is weighted equally

$$\hat{\mu} = \bar{y} = \frac{1}{a} \sum_{i=1}^a \bar{y}_{i+}, \quad \text{where} \quad \bar{y}_{i+} = \sum_{j=1}^{n_i} \frac{y_{ij}}{n_i}. \quad (2)$$

The variance of \bar{y} (Snedecor, Chapter 13) is

$$\sigma^2(\bar{y}) = \frac{\sigma^2(C)}{a} + \frac{\sigma^2(A)}{a^2} \sum_{i=1}^a \frac{1}{n_i}. \quad (3)$$

The estimated variance of \bar{y} is obtained by substituting the REML estimators of the two variance components into equation (3). The degrees of freedom associated with the estimates variance is approximately the number of cores with data minus 1.

The 95% confidence interval on the mean concentration μ is (LL, UL) where

$$95\% \text{ LL} = \bar{y} - t_{.975} \sqrt{\hat{\sigma}^2(\bar{y})}, \text{ and } 95\% \text{ UL} = \bar{y} + t_{.975} \sqrt{\hat{\sigma}^2(\bar{y})}, \quad (4)$$

and $t_{.975}$ is the 0.975 percentile point from Student's t distribution with approximated degrees of freedom $a-1$.

The statistical model that describes the structure of the core segment data is

$$y_{ijk} = \mu + C_i + S_{ij} + A_{ijk}, \quad i = 1, \dots, a, \quad j = 1, \dots, b_i, \quad k = 1, \dots, n_{ij}, \quad (5)$$

where

- y_{ijk} = laboratory results from the k^{th} duplicate of the j^{th} segment of the i^{th} core from the tank,
- μ = the grand mean of all the data,
- C_i = the effect of the i^{th} core (spatial effect),
- S_{ij} = the effect of the j^{th} segment sample from the i^{th} core (spatial effect),
- A_{ijk} = the analytical error associated with the k^{th} duplicate in the j^{th} composite from the i^{th} core,
- a = the number of cores,
- b_i = the number of composite samples in the i^{th} core, and
- n_{ij} = the number of analytical results from the j^{th} composite sample in the i^{th} core.

For cores 50, 51 and 52 there were data from three, five, and four subsegments (i.e., $b_1 = 3$, $b_2 = 5$ and $b_3 = 4$).

The variables C_i and S_{ij} are treated as random effects. It is assumed that C_i , S_{ij} , and A_{ijk} are each distributed normally with mean zero and variances of $\sigma^2(C)$, $\sigma^2(S)$, and $\sigma^2(A)$, respectively. Estimates of $\sigma^2(C)$, $\sigma^2(S)$, and $\sigma^2(A)$ were obtained using Restricted Maximum Likelihood Estimation (REML). This method applied to variance component estimation is described by Harville (1977).

The mean concentration of each analyte of interest in the tank was calculated using the following equation:

where

This mean gives the results from each core the same weight regardless of the unbalance that may exist for a particular analyte.

$$\bar{y} = \frac{1}{a} \sum_{i=1}^a \bar{y}_{i++} = \frac{1}{a} \sum_{i=1}^a \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} y_{ijk}}{n_{i+}} = \frac{1}{a} \sum_{i=1}^a \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} (\mu + C_i + S_{ij} + A_{ijk})}{n_{i+}}. \quad (6)$$

$$\bar{y}_{i++} = \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} y_{ijk}}{n_{i+}} \quad \text{and} \quad n_{i+} = \sum_{j=1}^{b_i} n_{ij}. \quad (7)$$

The variance of \bar{y} is

$$V(\bar{y}) = C_1 \sigma^2(C) + C_2 \sigma^2(S) + C_3 \sigma^2(A) \quad (8)$$

where

$$C_1 = \frac{1}{a}, \quad C_2 = \frac{1}{a^2} \sum_{i=1}^a \left[\frac{1}{n_{i+}} \right]^2 \left[\sum_{j=1}^{b_i} n_{ij}^2 \right], \quad C_3 = \frac{1}{a^2} \sum_{i=1}^a \left[\frac{1}{n_{i+}} \right]. \quad (9)$$

Using $\hat{\sigma}^2(C)$, $\hat{\sigma}^2(S)$, and $\hat{\sigma}^2(A)$ (REML variance component estimates), an estimated variance of \bar{y} is

$$\hat{\sigma}^2(\bar{y}) = C_1 \hat{\sigma}^2(C) + C_2 \hat{\sigma}^2(S) + C_3 \hat{\sigma}^2(A). \quad (10)$$

The approximate degrees of freedom used for $\hat{\sigma}^2(\bar{y})$ is the number of cores with data minus one.

The lower and upper 95% limits (95% LL and 95% UL respectively) on the mean concentration are

$$95\% \text{ LL} = \bar{y} - t_{.975} \sqrt{\hat{\sigma}^2(\bar{y})} \quad \text{and} \quad 95\% \text{ UL} = \bar{y} + t_{.975} \sqrt{\hat{\sigma}^2(\bar{y})} \quad (11)$$

where $t_{.975}$ is the 0.975 percentile from Student's t-distribution with the degrees of freedom associated with $\hat{\sigma}^2(\bar{y})$.

APPENDIX C
CHARGE AND MASS BALANCES

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Sample: Core 50 Drainable Liquid Composite Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	4.10	4.67	% (g/g)	Solids			
Water*	95.9	95.33	% (g/g)	H ₂ O	9.56e-01		
Aluminum	4450	4060	µg/L	Al(OH) ₄ ⁻	9.27e-06		
Antimony	1570	2270	µg/L	Sb(OH) ₃	2.67e-06		
Bismuth	920	920	µg/L	Bi(OH) ₃	1.12e-06		
Chromium	45600	45000	µg/L	Cr(OH) ₃	8.80e-05		
Iron	5820	5670	µg/L	FeO(OH)	8.96e-06		
Lead	780	780	µg/L	PbO ₂	8.83e-07		
Magnesium	1320	1160	µg/L	Mg(OH) ₂	2.92e-06		
Manganese	30	34.5	µg/L	Mn(OH) ₂	5.12e-08		
Molybdenum	2230	2140	µg/L	MoO ₂ (OH)	3.62e-06		
Nickel	1790	1750	µg/L	Ni(OH) ₂	2.74e-06		
Selenium	1410	1410	µg/L	Se(OH) ₄	2.57e-06		
Total U	94.4	96.1	µg/g	UO ₂ (OH) ₂	1.19e-04		
Zirconium	134	134	µg/L	Zr(OH) ₄	2.29e-07		
Boron	9330	9030	µg/L	B ₄ O ₇ ²⁻	3.23e-05	2.08e-07	4.16e-07
Chloride	199	193	µg/mL	Cl ⁻	1.92e-04	5.42e-06	5.42e-08
Cyanide	13.5	13.4	µg/mL	CN ⁻	1.32e-04	5.07e-07	5.07e-07
Fluoride	170	177	µg/mL	F ⁻	1.70e-04	8.95e-06	8.95e-06
Nitrate	21200	21200	µg/mL	NO ₃ ⁻	2.08e-02	3.35e-04	3.35e-04
Nitrite	2590	2570	µg/mL	NO ₂ ⁻	2.53e-03	5.50e-05	5.50e-05
pH	9.63	9.62	pH	OH ⁻	7.03e-07	4.13e-08	4.13e-08
Phosphate	2300	2500	µg/mL	HPO ₄ ²⁻			
Phosphorus	785000	795000	µg/L	HPO ₄ ²⁻	2.40e-03	2.50e-05	5.00e-05
Silicon	60800	56200	µg/L	SiO ₃ ²⁻	1.63e-04	2.20e-06	4.40e-06
Sulfate	4640	4650	µg/mL	SO ₄ ²⁻			
Sulfur	831000	818000	µg/L	SO ₄ ²⁻	2.42e-03	2.52e-05	5.04e-05
TIC	516	508	µg(C)/mL	CO ₃ ²⁻	2.51e-03	4.18e-05	8.36e-05
TOC	1100	1200	µg(C)/mL	Acetate	2.77e-03	4.69e-05	4.69e-05
Calcium	4870	3700	µg/L	Ca ²⁺	4.20e-06	1.05e-07	2.10e-07
Potassium	46800	46600	µg/L	K ⁺	4.58e-05	1.17e-06	1.17e-06
Sodium	14600000	14600000	µg/L	Na ⁺	1.43e-02	6.23e-04	6.23e-04
Strontium	140	139	µg/L	Sr ²⁺	1.37e-07	1.56e-09	3.12e-09
Mass Balance: (%) →					100.47		0.97
Charge Balance (Cation/Anion) ↑							

Specific Gravity	1.02
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* Water determination is made by subtracting "Solids" determination from 100

Sample: Core 51 Drainable Liquid Composite Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	24.0	25.4	% (g/g)	Solids			
Water*	76.0	74.6	% (g/g)	H ₂ O	7.53e-01		
Aluminum	11100	11800	µg/L	Al(OH) ₄	2.12e-05		
Antimony	2520	2520	µg/L	Sb(OH) ₃	2.98e-06		
Bismuth	920	920	µg/L	Bi(OH) ₃	9.54e-07		
Chromium	271000	275000	µg/L	Cr(OH) ₃	4.51e-04		
Iron	47500	48500	µg/L	FeO(OH)	6.36e-05		
Lead	780	780	µg/L	PbO ₂	7.50e-07		
Magnesium	511	310	µg/L	Mg(OH) ₂	8.21e-07		
Manganese	75.6	81.1	µg/L	Mn(OH) ₂	1.06e-07		
Molybdenum	22200	22600	µg/L	MoO ₂ (O)	3.15e-05		
Nickel	15900	16200	µg/L	Ni(OH) ₂	2.11e-05		
Selenium	1410	1410	µg/L	Se(OH) ₄	2.19e-06		
Total U	610	566	µg/g	UO ₂ (OH)	6.26e-04		
Zirconium	656	641	µg/L	Zr(OH) ₄	1.11e-06		
Boron	24200	25000	µg/L	B ₄ O ₇ ²⁻	8.66e-05	5.58e-07	1.12e-06
Chloride	1340	1340	µg/mL	Cl ⁻	1.31e-03	3.71e-05	3.71e-05
Cyanide	152	152	µg/mL	CN ⁻	1.49e-04	5.73e-06	5.73e-06
Fluoride	824	826	µg/mL	F ⁻	8.09e-04	4.26e-05	4.26e-05
Nitrate	134000	135000	µg/mL	NO ₃ ⁻	1.32e-01	2.13e-03	2.13e-03
Nitrite	27500	27800	µg/mL	NO ₂ ⁻	2.71e-02	5.89e-04	5.89e-04
pH	10.7	10.7	pH	OH ⁻	8.36e-06	4.91e-07	4.91e-07
Phosphate	6290	6190	µg/mL	HPO ₄ ²⁻			
Phosphorus	2020000	2030000	µg/L	HPO ₄ ²⁻	6.15e-03	6.41e-05	1.28e-04
Silicon	65800	66800	µg/L	SiO ₃ ²⁻	1.85e-04	2.49e-06	4.98e-06
Sulfate	16800	16800	µg/mL	SO ₄ ²⁻			
Sulfur	6030000	5990000	µg/L	SO ₄ ²⁻	1.77e-02	1.84e-04	3.68e-04
TIC	4580	4510	µg(C)/mL	CO ₃ ²⁻	2.23e-02	3.71e-04	7.42e-04
TOC	1050	1080	µg(C)/mL	Acetate	2.57e-03	4.35e-05	4.35e-05
Calcium	7000	2950	µg/L	Ca ²⁺	4.88e-06	1.22e-07	2.43e-07
Potassium	360000	374000	µg/L	K ⁺	3.60e-04	9.20e-06	9.20e-06
Sodium	96300000	94700000	µg/L	Na ⁺	9.36e-02	4.07e-03	4.07e-03
Strontium	175	169	µg/L	Sr ²⁺	1.69e-07	1.92e-09	3.85e-09
Mass Balance:(%) →					105.84		1.00
Charge Balance (Cation/Anion) ↑							

Specific Gravity	1.20
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* Water determination is made by subtracting "Solids" determination from 100

Sample: Core 51 Composite (Water Leach) Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	48.6	47.6	% (g/g)	Solids			
Water*	51.4	52.4	% (g/g)	H ₂ O	5.19e-01		
Aluminum	344	627	µg/g	Al(OH)	1.08e-03		
Antimony	22.1	22.1	µg/g	Sb(OH) ₃	3.14e-05		
Bismuth	108	81.5	µg/g	Bi(OH) ₃	1.18e-04		
Chromium	230	216	µg/g	Cr(OH) ₃	4.42e-04		
Iron	315	229	µg/g	FeO(OH)	4.33e-04		
Lead	20.3	20.1	µg/g	PbO ₂	2.33e-05		
Magnesium	7.30	12.2	µg/g	Mg(OH) ₂	2.34e-05		
Manganese	1.78	2.37	µg/g	Mn(OH) ₂	3.36e-06		
Molybdenum	8.72	8.67	µg/g	MoO ₂ (OH)	1.47e-05		
Nickel	5.18	3.79	µg/g	Ni(OH) ₂	7.08e-06		
Selenium	58.6	52.1	µg/g	Se(OH) ₄	1.03e-04		
Total U	32900	31600	µg/g	UO ₂ (OH) ₂	4.12e-02		
Zirconium	2.10	3.90	µg/g	Zr(OH) ₄	5.13e-09		
Boron	607	666	µg/g	B ₄ O ₇ ²⁻	2.29e-09	1.47e-05	2.94e-05
Chloride	732	632	µg/g	Cl ⁻	6.82e-04	1.92e-05	1.92e-05
Cyanide	94.9	96.7	µg/g	CN ⁻	9.58e-05	3.68e-06	3.68e-06
Fluoride	8530	9920	µg/g	F ⁻	9.23e-03	4.86e-04	4.86e-04
Nitrate	96400	89200	µg/g	NO ₃ ⁻	9.28e-02	1.50e-03	1.50e-03
Nitrite	15700	14900	µg/g	NO ₂ ⁻	1.53e-02	3.33e-04	3.33e-04
pH	11.6	11.6	pH	OH ⁻	6.77e-05	3.98e-06	3.98e-06
Phosphate	87000	102000	µg/g	HPO ₄ ²⁻			
Phosphorus	27300	33300	µg/g	HPO ₄ ²⁻	9.39e-02	9.78e-04	1.96e-03
Silicon	3710	4180	µg/g	SiO ₃ ²⁻	1.12e-02	1.51e-04	3.02e-04
Sulfate	13000	12300	µg/g	SO ₄ ²⁻			
Sulfur	4100	3840	µg/g	SO ₄ ²⁻	1.19e-02	1.24e-04	2.48e-04
TIC	5640	5710	µg(C)/g	CO ₃ ²⁻	2.84e-02	4.72e-04	9.45e-04
TOC	1520	1350	µg(C)/g	Acetate	3.53e-03	5.97e-05	5.97e-05
Calcium	151	800	µg/g	Ca ⁺	4.76e-04	1.19e-05	2.37e-05
Potassium	641	260	µg/g	K ⁺	4.51e-04	1.15e-05	1.15e-05
Sodium	128000	140000	µg/g	Na ⁺	1.34e-01	5.83e-03	5.83e-03
Strontium	6.44	4.54	µg/g	Sr ²⁺	5.49e-06	6.27e-08	1.25e-07
Mass Balance:(%) →					96.67		1.00

Charge Balance (Cation/Anion) ↑

* Water determination is made by subtracting "Solids" determination from 100

Sample: Core 51 Composite (Acid Digest) Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	48.6	47.6	% (g/g)	Solids			
Water*	51.4	52.4	% (g/g)	H ₂ O	5.19e-01		
Aluminum	4030	4250	µg/g	Al(OH)	9.20e-03		
Antimony	209	208	µg/g	Sb(OH) ₃	2.96e-04		
Bismuth	7790	8130	µg/g	Bi(OH) ₃	9.90e-03		
Chromium	383	381	µg/g	Cr(OH) ₃	7.57e-04		
Iron	33400	32900	µg/g	FeO(OH)	5.27e-02		
Lead	1170	1040	µg/g	PbO ₂	1.28e-03		
Magnesium	265	259	µg/g	Mg(OH) ₂	6.29e-04		
Manganese	236	226	µg/g	Mn(OH) ₂	3.74e-04		
Molybdenum	5.98	5.95	µg/g	MoO ₂ (OH)	1.01e-05		
Nickel	308	301	µg/g	Ni(OH) ₂	4.81e-04		
Selenium	104	86.3	µg/g	Se(OH) ₄	1.77e-02		
Total U	32900	31600	µg/g	UO ₂ (OH) ₂	4.12e-02		
Zirconium	117	121	µg/g	Zr(OH) ₄	2.04e-07		
Boron	33.8	13.5	µg/g	B ₄ O ₇ ²⁻	8.49e-05	5.47e-07	1.09e-06
Chloride	732	632	µg/g	Cl ⁻	6.82e-04	1.92e-05	1.92e-05
Cyanide	94.9	96.7	µg/g	CN ⁻	9.58e-05	3.68e-06	3.68e-06
Fluoride	8530	9920	µg/g	F ⁻	9.23e-03	4.86e-04	4.86e-04
Nitrate	96400	89200	µg/g	NO ₃ ⁻	9.28e-02	1.50e-03	1.50e-03
Nitrite	15700	14900	µg/g	NO ₂ ⁻	1.53e-02	3.33e-04	3.33e-04
pH	11.6	11.6	pH	OH ⁻	6.77e-05	3.98e-06	3.98e-06
Phosphate	87000	102000	µg/g	HPO ₄ ²⁻			
Phosphorus	33900	25400	µg/g	HPO ₄ ²⁻	9.19e-02	9.57e-04	1.91e-03
Silicon	63.7	156	µg/g	SiO ₃ ²⁻	3.12e-04	4.21e-06	8.42e-06
Sulfate	13000	12300	µg/g	SO ₄ ²⁻			
Sulfur	3600	3520	µg/g	SO ₄ ²⁻	1.07e-02	1.11e-04	2.22e-04
TIC	5640	5710	µg(C)/g	CO ₃ ²⁻	2.84e-02	4.72e-04	9.45e-04
TOC	1520	1350	µg(C)/g	Acetate	3.53e-03	5.97e-05	5.97e-05
Calcium	808	897	µg/g	Ca ⁺	8.53e-04	2.13e-05	4.25e-05
Potassium	267	220	µg/g	K ⁺	2.44e-04	6.23e-06	6.23e-06
Sodium	142000	131000	µg/g	Na ⁺	1.37e-01	5.94e-03	5.94e-03
Strontium	1250	1230	µg/g	Sr ²⁺	1.24e-03	1.42e-05	2.83e-05
Mass Balance:(%) →					102.79		1.10

Charge Balance (Cation/Anion) ↑

* Water determination is made by subtracting "Solids" determination from 100

Sample: Core 51 Composite (Fusion Digest) Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	48.6	47.6	% (g/g)	Solids			
Water*	51.4	52.4	% (g/g)	H ₂ O	5.19e-01		
Aluminum	7260	4200	µg/g	Al(OH) ₃	1.27e-02		
Antimony	139	104	µg/g	Sb(OH) ₃	1.72e-04		
Bismuth	8200	8490	µg/g	Bi(OH) ₃	1.04e-02		
Chromium	351	359	µg/g	Cr(OH) ₃	7.03e-04		
Iron	26300	2660	µg/g	FeO(OH)	4.21e-02		
Lead	763	690	µg/g	PbO ₂	8.39e-04		
Magnesium	223	240	µg/g	Mg(OH) ₂	5.55e-04		
Manganese	196	183	µg/g	Mn(OH) ₂	3.07e-04		
Molybdenum	10.5	10.6	µg/g	MoO ₂ (OH)	1.78e-05		
Nickel	2850	1370	µg/g	Ni(OH) ₂	3.33e-03		
Selenium	70.2	69.8	µg/g	Se(OH) ₄	1.30e-04		
Total U	32900	31600	µg/g	UO ₂ (OH) ₂	4.12e-02		
Zirconium	85.6	66.3	µg/g	Zr(OH) ₂	1.30e-07		
Boron	4.98	4.95	µg/g	B ₄ O ₇ ²⁻	1.78e-05	1.15e-07	2.30e-07
Chloride	732	632	µg/g	Cl ⁻	6.82e-04	1.92e-05	1.92e-05
Cyanide	94.9	96.7	µg/g	CN ⁻	9.58e-05	3.68e-06	3.68e-06
Fluoride	8530	9920	µg/g	F ⁻	9.23e-03	4.86e-04	4.86e-04
Nitrate	964000	89200	µg/g	NO ₃ ⁻	9.28e-02	1.50e-03	1.50e-03
Nitrite	15700	14900	µg/g	NO ₂ ⁻	1.53e-02	3.33e-04	3.33e-04
pH	11.6	11.6	pH	OH ⁻	6.77e-05	3.98e-06	3.98e-06
Phosphate	87000	102000	µg/g	HPO ₄ ²⁻			
Phosphorus	31500	34300	µg/g	HPO ₄ ²⁻	1.02e-01	1.06e-03	2.12e-03
Silicon	4980	4750	µg/g	SiO ₃ ²⁻	1.38e-02	1.87e-04	3.73e-04
Sulfate	13000	12300	µg/g	SO ₄ ²⁻			
Sulfur	3490	3640	µg/g	SO ₄ ²⁻	1.07e-02	1.11e-04	2.22e-04
TIC	5640	5710	µg(C)/g	CO ₃ ²⁻	2.84e-02	4.72e-04	9.45e-04
TOC	1520	1350	µg(C)/g	Acetate	3.53e-03	5.97e-05	5.97e-05
Calcium	765	794	µg/g	Ca ²⁺	7.80e-04	1.94e-05	3.89e-05
Potassium			µg/g	K ⁺			
Sodium	119000	127000	µg/g	Na ⁺	1.23e-01	5.35e-03	5.35e-03
Strontium	934	974	µg/g	Sr ²⁺	9.54e-04	1.09e-05	2.18e-05
Mass Balance:(%) →					103.27		0.89

Charge Balance (Cation/Anion) ↑

* Water determination is made by subtracting "Solids" determination from 100

Sample: Core 52 Drainable Liquid Composite Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	13.2	13.7	% (g/g)	Solids			
Water*	86.8	86.3	% (g/g)	H ₂ O	8.66e-01		
Aluminum	47200	48400	µg/L	Al(OH) ₃	9.66e-05		
Antimony	3800	3800	µg/L	Sb(OH) ₃	4.90e-06		
Bismuth	16900	17000	µg/L	Bi(OH) ₃	1.92e-05		
Chromium	186000	190000	µg/L	Cr(OH) ₃	3.39e-04		
Iron	18900	19200	µg/L	FeO(OH)	2.76e-05		
Lead	1950	1950	µg/L	PbO ₂	2.05e-06		
Magnesium	390	390	µg/L	Mg(OH) ₂	8.57e-07		
Manganese	75.0	75.0	µg/L	Mn(OH) ₂	1.10e-07		
Molybdenum	2090	2160	µg/L	MoO ₂ (OH)	3.26e-06		
Nickel	2750	2820	µg/L	Ni(OH) ₂	4.00e-06		
Selenium	3520	3520	µg/L	Se(OH) ₄	5.96e-06		
Total U	39.7	41.5	µg/g	UO ₂ (OH) ₂	4.71e-04		
Zirconium	1760	1630	µg/L	Zr(OH) ₄	2.90e-06		
Boron	28300	32000	µg/L	B ₄ O ₇ ²⁻	3.94e-04	2.54e-06	5.07e-06
Chloride	851	869	µg/mL	Cl ⁻	7.82e-04	2.21e-05	2.21e-05
Cyanide	40.0	39.6	µg/mL	CN ⁻	3.62e-05	1.39e-06	1.39e-06
Fluoride	668	678	µg/mL	F ⁻	6.12e-04	3.22e-05	3.22e-05
Nitrate	100000	100000	µg/mL	NO ₃ ⁻	9.09e-02	1.47e-03	1.47e-03
Nitrite	7990	8120	µg/mL	NO ₂ ⁻	7.32e-03	1.59e-04	1.59e-04
pH	10.3	10.3	pH	OH ⁻	3.08e-06	1.81e-07	1.81e-07
Phosphate	7650	7610	µg/mL	HPO ₄ ²⁻			
Phosphorus	2630000	2550000	µg/L	HPO ₄ ²⁻	2.38e-03	2.48e-05	4.96e-05
Silicon	78200	96100	µg/L	SiO ₃ ²⁻	2.25e-04	3.04e-06	6.07e-06
Sulfate	9580	9580	µg/mL	SO ₄ ²⁻			
Sulfur	3350000	3320000	µg/L	SO ₄ ²⁻	9.08e-03	9.46e-05	1.89e-04
TIC	346	332	µg(C)/mL	CO ₃ ²⁻	1.54e-03	2.57e-05	5.13e-05
TOC	340	369	µg(C)/mL	Acetate	1.58e-03	2.68e-05	2.68e-05
Calcium	3480	4400	µg/L	Ca ²⁺	3.58e-06	8.94e-08	1.79e-07
Potassium	137000	139000	µg/L	K ⁺	1.25e-04	3.21e-06	3.21e-06
Sodium	52300000	51500000	µg/L	Na ⁺	4.72e-02	2.05e-03	2.05e-03
Strontium	1800	1820	µg/L	Sr ²⁺	1.65e-06	1.88e-08	3.76e-08
Mass Balance:(%) →					102.82		1.02

Charge Balance (Cation/Anion) ↑

* Water determination is made by subtracting "Solids" determination from 100

Sample: Core 52 Composite (Water Leach) Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	52.2	52.3	% (g/g)	Solids			
Water*	47.8	47.7	% (g/g)	H ₂ O	4.78e-01		
Aluminum	849	784	µg/g	Al(OH)	1.82e-03		
Antimony	213	213	µg/g	Sb(OH) ₃	3.02e-04		
Bismuth	375	409	µg/g	Bi(OH) ₃	4.88e-04		
Chromium	213	184	µg/g	Cr(OH) ₃	3.93e-04		
Iron	429	449	µg/g	FeO(OH)	6.98e-04		
Lead	62.8	62.8	µg/g	PbO ₂	7.25e-05		
Magnesium	10.0	98.3	µg/g	Mg(OH) ₂	1.30e-04		
Manganese	3.04	3.04	µg/g	Mn(OH) ₂	4.92e-06		
Molybdenum	6.96	7.17	µg/g	MoO ₂ (OH)	1.19e-05		
Nickel	13.2	13.2	µg/g	Ni(OH) ₂	2.08e-05		
Selenium	88.1	98.4	µg/g	Se(OH) ₄	1.74e-04		
Total U	18200	19000	µg/g	UO ₂ (OH) ₂	2.38e-02		
Zirconium	12.2	7.09	µg/g	Zr(OH) ₄	1.65e-08		
Boron	16.1	14.8	µg/g	B ₄ O ₇ ²⁻	5.55e-05	3.57e-07	7.15e-07
Chloride	389	409	µg/g	Cl ⁻	3.99e-04	1.13e-05	1.13e-05
Cyanide	51.4	61.3	µg/g	CN ⁻	5.64e-05	2.17e-06	2.17e-06
Fluoride	14000	13200	µg/g	F ⁻	1.36e-02	7.16e-04	7.16e-04
Nitrate	54800	57700	µg/g	NO ₃ ⁻	5.63e-02	9.07e-04	9.07e-04
Nitrite	7980	8290	µg/g	NO ₂ ⁻	8.14e-03	1.77e-04	1.77e-04
pH	11.4	11.4	pH	OH ⁻	4.27e-05	2.51e-06	2.51e-06
Phosphate	140000	125000	µg/g	HPO ₄ ²⁻			
Phosphorus	18400	15900	µg/g	HPO ₄ ²⁻	5.31e-02	5.54e-04	1.11e-03
Silicon	16.2	16.2	µg/g	SiO ₃ ²⁻	4.60e-05	6.21e-07	1.24e-06
Sulfate	6980	7260	µg/g	SO ₄ ²⁻			
Sulfur	3360	2860	µg/g	SO ₄ ²⁻	9.32e-03	9.70e-05	1.94e-04
TIC	2990	2560	µg(C)/g	CO ₃ ²⁻	1.39e-02	2.31e-04	4.62e-04
TOC	2000	1920	µg(C)/g	Acetate	4.82e-03	8.16e-05	8.16e-05
Calcium	71.1	59.9	µg/g	Ca ⁺	6.55e-05	1.63e-06	3.27e-06
Potassium	226	135	µg/g	K ⁺	1.81e-04	4.62e-06	4.62e-06
Sodium	87100	76500	µg/g	Na ⁺	8.18e-02	3.56e-03	3.56e-03
Strontium	5.06	6.08	µg/g	Sr ²⁺	5.57e-06	6.36e-08	1.27e-07
Mass Balance:(%) →					74.71		0.97

Charge Balance (Cation/Anion) ↑

* Water determination is made by subtracting "Solids" determination from 100

Sample: Core 52 Composite (Acid Digest) Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	52.2	52.3	% (g/g)	Solids			
Water*	47.8	47.7	% (g/g)	H ₂ O	4.78e-01		
Aluminum	23900	25300	µg/g	AlO(OH)	5.47e-02		
Antimony	37.4	53.6	µg/g	Sb(OH) ₃	6.46e-05		
Bismuth	13100	14400	µg/g	Bi(OH) ₃	1.71e-02		
Chromium	309	342	µg/g	Cr(OH) ₃	6.45e-04		
Iron	20100	39500	µg/g	FeO(OH)	4.74e-02		
Lead	357	618	µg/g	PbO ₂	5.63e-04		
Magnesium	157	173	µg/g	Mg(OH) ₂	3.96e-04		
Manganese	126	298	µg/g	Mn(OH) ₂	3.43e-04		
Molybdenum	5.57	7.26	µg/g	MoO ₂ (OH)	1.08e-05		
Nickel	274	285	µg/g	Ni(OH) ₂	4.41e-04		
Selenium	14.1	14.0	µg/g	Se(OH) ₄	2.62e-05		
Total U	18200	19000	µg/g	UO ₂ (OH) ₂	2.38e-02		
Zirconium	22.5	25.7	µg/g	Zr(OH) ₄	4.12e-08		
Boron	3.59	0.990	µg/g	B ₄ O ₇ ²⁻	8.22e-06	5.30e-08	1.06e-07
Chloride	389	409	µg/g	Cl ⁻	3.99e-04	1.13e-05	1.13e-05
Cyanide	51.4	61.3	µg/g	CN ⁻	5.64e-05	2.17e-06	2.17e-06
Fluoride	14000	13200	µg/g	F ⁻	1.36e-02	7.16e-04	7.16e-04
Nitrate	54800	57700	µg/g	NO ₃ ⁻	5.63e-02	9.07e-04	9.07e-04
Nitrite	7980	8290	µg/g	NO ₂ ⁻	8.14e-03	1.77e-04	1.77e-04
pH	11.4	11.4	pH	OH ⁻	4.27e-05	2.51e-06	2.51e-06
Phosphate	140000	125000	µg/g	HPO ₄ ²⁻			
Phosphorus	33400	27300	µg/g	HPO ₄ ²⁻	9.40e-02	9.80e-04	1.96e-03
Silicon	1820	1460	µg/g	SiO ₃ ²⁻	4.66e-03	6.29e-05	1.26e-04
Sulfate	6980	7260	µg/g	SO ₄ ²⁻			
Sulfur	2490	2570	µg/g	SO ₄ ²⁻	7.58e-03	7.89e-05	1.58e-04
TIC	2990	2560	µg(C)/g	CO ₃ ²⁻	1.39e-02	2.31e-04	4.62e-04
TOC	2000	1920	µg(C)/g	Acetate	4.82e-03	8.16e-05	8.16e-05
Calcium	542	643	µg/g	Ca ⁺	5.93e-04	1.48e-05	2.96e-05
Potassium	233	217	µg/g	K ⁺	2.25e-04	5.61e-06	1.12e-05
Sodium	131000	117000	µg/g	Na ⁺	1.24e-01	5.39e-03	5.39e-03
Strontium	704	665	µg/g	Sr ²⁺	6.85e-04	7.81e-06	1.56e-05
Mass Balance:(%) →					95.19		1.18

Charge Balance (Cation/Anion) ↑

* Water determination is made by subtracting "Solids" determination from 100

Sample: Core 52 Composite (Fusion Digest) Analytical Results

Analyte	Result #1	Result #2	Units	Suspect	g/g	Moles/g	Eq/g
Solids	52.2	52.3	% (g/g)	Solids			
Water*	47.8	47.7	% (g/g)	H ₂ O	4.78e-01		
Aluminum	26400	27500	µg/g	AlO(OH)	5.99e-02		
Antimony	75.4	75.8	µg/g	Sb(OH) ₃	1.07e-04		
Bismuth	14000	17300	µg/g	Bi(OH) ₃	1.95e-02		
Chromium	341	389	µg/g	Cr(OH) ₃	7.23e-04		
Iron	21000	42800	µg/g	FeO(OH)	5.08e-02		
Lead	346	796	µg/g	PbO ₂	6.59e-04		
Magnesium	190	252	µg/g	Mg(OH) ₂	5.30e-04		
Manganese	161	313	µg/g	Mn(OH) ₂	3.83e-04		
Molybdenum	7.96	6.63	µg/g	MoO ₂ (OH)	1.23e-05		
Nickel	3800	2530	µg/g	Ni(OH) ₂	5.00e-03		
Selenium	69.9	70.4	µg/g	Se(OH) ₄	1.31e-04		
Total U	18200	19000	µg/g	UO ₂ (OH) ₂	2.38e-02		
Zirconium	127	93.8	µg/g	Zr(OH) ₄	1.89e-07		
Boron	4.96	4.99	µg/g	B ₄ O ₇ ²⁻	1.79e-05	1.15e-07	2.30e-07
Chloride	389	409	µg/g	Cl ⁻	3.99e-04	1.13e-05	1.13e-05
Cyanide	51.4	61.3	µg/g	CN ⁻	5.64e-05	2.17e-06	2.17e-06
Fluoride	14000	13200	µg/g	F ⁻	1.36e-02	7.16e-04	7.16e-04
Nitrate	54800	57700	µg/g	NO ₃ ⁻	5.63e-02	9.07e-04	9.07e-04
Nitrite	7980	8290	µg/g	NO ₂ ⁻	8.14e-03	1.77e-04	1.77e-04
pH	11.4	11.4	pH	OH ⁻	4.27e-05	2.51e-06	2.51e-06
Phosphate	140000	125000	µg/g	HPO ₄ ²⁻			
Phosphorus	33600	28900	µg/g	HPO ₄ ²⁻	9.68e-02	1.01e-03	2.02e-03
Silicon	7110	7390	µg/g	SiO ₃ ²⁻	2.06e-02	2.78e-04	5.56e-04
Sulfate	6980	7260	µg/g	SO ₄ ²⁻			
Sulfur	2910	3060	µg/g	SO ₄ ²⁻	8.94e-03	9.31e-05	1.86e-04
TIC	2990	2560	µg(C)/g	CO ₃ ²⁻	1.39e-02	2.31e-04	4.62e-04
TOC	2000	1920	µg(C)/g	Acetate	4.82e-03	8.16e-05	8.16e-05
Calcium	702	781	µg/g	Ca ⁺	7.42e-04	1.85e-05	3.70e-05
Potassium			µg/g	K ⁺			
Sodium	115000	108000	µg/g	Na ⁺	1.12e-01	4.85e-03	4.85e-03
Strontium	751	854	µg/g	Sr ²⁺	8.03e-04	9.16e-06	1.83e-05
					97.55		0.96

Charge Balance (Cation/Anion)↑

* Water determination is made by subtracting "Solids" determination from 100

APPENDIX D
MISCELLANEOUS INFORMATION

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To ensure every sample has its own unique identification number a numbering system has been employed. Each number set tells the individual working with the sample; the core number, segment, and subsegment. The customer identification numbers for T-107 were assigned according to the following system.

The first three characters represent the core number.

The fourth character represents the sample type according to the following key:

C = Core
D = Drainable Liquid
F = Field Blank
H = Hot Cell Blank
L = Liner liquid
S = Segment
T = Strata

The fifth and sixth characters represent the number of the sample type.

The seventh character represents the portion of the segment according to the following key:

U = Upper half of segment
L = Lower half of segment
F = Facie
W = Whole segment

The eighth character represents either a number or a sample type according to the key:

D = Direct
H = Homogenized
R = Repeat

For example, the customer identification number for a homogenized solid sample from tank T-107, Core 51, Segment 3, upper half would be as follows:

Sample Origin T-107 Customer ID 051S03UH