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Ferrocyanide Safety Program: Data Interpretation Report for Tank 241-T-107 Core Samples

Prepared for the U.S. Department of Energy
Office of Environmental Restoration and
Waste Management

Westinghouse
Hanford Company Richland, Washington

Hanford Operations and Engineering Contractor for the
U.S. Department of Energy under Contract DE-AC06-87RL10930

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L. M. Sasaki
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LIST OF TERMS

AEA	Alpha energy analysis
DQO	Data Quality Objectives
DSC	Differential scanning calorimetry
GEA	Gamma energy analysis
IC	Ion chromatography
ICP	Inductively coupled plasma atomic emission spectroscopy
IS	Insufficient sample for analysis
KOH	Potassium hydroxide
PNL	Pacific Northwest Laboratory
RPD	Relative percent difference
TC	Total carbon
TGA	Thermogravimetric analysis
TIC	Total inorganic carbon
TOC	Total organic carbon
WHC	Westinghouse Hanford Company

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1.0 INTRODUCTION

Between November 1992 and March 1993, three core samples were obtained from tank 241-T-107. Analyses were performed on these core samples to support the Ferrocyanide Safety Program and the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1994) Milestone M-10-00.

This document summarizes and evaluates those analytical results that are pertinent to the Ferrocyanide Safety Issue. This document compares the analytical results with the data requirements for ferrocyanide tanks as documented in *Data Requirements of the Ferrocyanide Safety Issue Developed Through the Data Quality Objectives Process* (Meacham et al. 1994) and provides an assessment of the safety condition of the tank. Analytes not listed in the Data Quality Objectives (DQO) document (Meacham et al. 1994) or not pertinent to the Ferrocyanide Safety Issue are not discussed in this report. Complete documentation of the analytical results can be found in the data package for the tank 241-T-107 cores (Svancara and Pool 1993). A more complete evaluation of the analytical results and an estimate of the tank inventory will be provided in a forthcoming tank characterization report for tank 241-T-107.

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2.0 BACKGROUND

2.1 TANK 241-T-107 HISTORY

Single-shell tank 241-T-107, with an operating capacity of 2,006,000 L (530,000 gal), was placed into service in 1944. The tank is on the Ferrocyanide Watch List and is estimated to have contained approximately 5,000 gram moles ferrocyanide [1,060 kg as $Fe(CN)_6^{-4}$] at the end of the ferrocyanide scavenging campaign (Borsheim and Simpson 1991). Tank 241-T-107 is also an assumed leaker. During its process history, it received four main types of waste: first-cycle decontamination waste, tri-butyl phosphate waste (including unconcentrated ferrocyanide scavenged tri-butyl phosphate waste from the U Plant flowsheet used during the scavenging process), cladding waste, and ion-exchange waste. The tank was removed from service in 1976.

2.2 FERROCYANIDE ISSUE DATA QUALITY OBJECTIVES

Data requirements for the Ferrocyanide Safety Issue have been developed using the DQO process (Meacham et al. 1994). This process has resulted in the definition of primary decision rules for Ferrocyanide tanks. Two key decision rules were identified to place Ferrocyanide tanks into one of three categories; *Safe*, *Conditionally Safe*, and *Unsafe* (Postma et al. 1994). These decision rules are defined as follows.

- If the fuel concentration average for all homogenized quarter-segment layers[1] is less than or equal to 8 wt% as disodium nickel ferrocyanide [$Na_2NiFe(CN)_6$] on an energy equivalent basis (i.e., 115 cal/g of dry material), then the tank is categorized as *Safe*. Otherwise, the tank is categorized as *Conditionally Safe* or *Unsafe* (the moisture and temperature conditions of the tank resolve the difference between these two categories).

- If the fuel concentration in any homogenized quarter-segment layer is greater than 8 wt% and if the wt% free water[2] is greater than 4/3 [wt% fuel[3] - 8 wt%], then the

---

[1] Quarter segment (12 cm) layers apply only to sludge samples. For salt cake samples, analyses will be made on homogenized half-segment (24 cm) layers. Based on historical records and inference from physical and chemical principles, most of the ferrocyanide should be in the sludge.

[2] Free water is defined as the water removed from a sample by drying at 120 °C for 18 hours.

[3] Wt% fuel represents the energy value of the sample based on an equivalent wt% $Na_2NiFe(CN)_6$. Fuel content is calculated by measuring the exothermic energy of the sample and dividing by the reaction energy of $Na_2NiFe(CN)_6$.

tank is categorized as *Conditionally Safe*. Otherwise, the tank is categorized as *Unsafe*.

Tanks categorized as *Safe* or *Conditionally Safe* cannot support a propagating exothermic reaction. The temperature of the waste is a secondary data requirement that is used to support the decision whether a tank is categorized as *Conditionally Safe* or *Unsafe*. Temperature is not a core sample data value, but is obtained from instrument tree measurements taken as part of the overall tank surveillance effort.

Furthermore, the DQO process has identified data requirements for the analysis of core samples from Ferrocyanide Watch List tanks. Table 2-1 lists primary and secondary data requirements (those analyses which (1) are necessary to categorize a ferrocyanide tank as *Safe*, *Conditionally Safe*, or *Unsafe*; (2) support the categorization of a Ferrocyanide tank; or (3) are important to the resolution of the Ferrocyanide Safety Issue). Table 2-2 list tertiary data requirements (those analyses necessary for resolving the Ferrocyanide Safety Issue but which do not have the urgency of primary or secondary data requirements).

Table 2-1. Primary and Secondary Data Requirements for Ferrocyanide Tanks.

Analyte	Analytical method[1]	Sample[2]	Decision[3] threshold	Required[4] analytical uncertainty
Total fuel[5]	Differential scanning calorimetry/adiabatic calorimetry	¼-Segment	8 wt% (0.48 MJ/kg or 115 cal/g)	≤10%
Moisture content	Thermogravimetric analysis	¼-Segment	4/3 [Fuel - 8]	≤10%[6]
Tank temperature	Thermocouple	NA[7]	90 °C	≤10%
Cs^{137}	Gamma energy analysis	¼-Segment & liquid	50 μCi/g	≤10%
Sr^{90}	Beta radiochemistry	¼-Segment & liquid	50 μCi/g	≤10%
Total cyanide	Direct assay	¼-Segment	3.9 wt%	≤10%
Total organic carbon	Direct persulfate oxidation	¼-Segment	3 wt%	≤10%
Nickel	Inductively coupled plasma	¼-Segment	1,000 μg/g	≤18%

[1] Other techniques that meet the required uncertainty are also acceptable.
[2] All analyses are conducted on homogenized samples.
[3] Excluding moisture, all decision thresholds reported on a dry basis.
[4] Values that are less than 25% of the decision threshold do not require the specified analytical uncertainty.
[5] Calculated on a $Na_2NiFe(CN)_6$ energy equivalent basis.
[6] Values less than 5 or greater than 35 wt% water do not require the specified uncertainty.
[7] NA = Not applicable.

Table 2-2. Tertiary Data Requirements for Ferrocyanide Tanks.

Analyte	Analytical method[1]	Sample[2]	Required[3] sensitivity	Required[4] analytical uncertainty
Aluminum, calcium, iron, phosphorus, sodium	Inductively coupled plasma	¼-Segment & liquid	500 μg/g	≤25%
Chloride, fluoride, nitrate, nitrite, phosphate	Ion chromatography	¼-Segment & liquid	500 μg/g	≤25%
pH	Ion selective electrode	liquid	4 - 12	±0.5[5]
Total carbon	Coulometric detection	¼-Segment	1,200 μg/g	≤25%
Total inorganic carbon	Coulometric detection	¼-Segment	1,200 μg/g	≤25%
Total alpha	Proportional counting	¼-Segment	2 μCi/g	≤18%
Total beta	Proportional counting	¼-Segment	50 μCi/g	≤18%
Total gamma	High purity germanium detector	¼-Segment	50 μCi/g	≤18%
Pu^{238}	Separation and alpha spectrometry	Composite	0.1 μCi/g	≤25%
$Pu^{239/240}$	Separation and alpha spectrometry	Composite	2 μCi/g	≤25%
Am^{241}	Separation and alpha spectrometry/gamma energy analysis	Composite	2 μCi/g	≤25%
Co^{60}	Gamma energy analysis	Composite	0.1 μCi/g	≤25%
$Eu^{154/155}$	Gamma energy analysis	Composite	5 μCi/g	≤25%
Uranium	Laser induced kinetic phosphorescence	Composite	1,000 μg/g	≤25%
Bulk density	Gravimetric	Composite & liquid	NA[6]	≤10%
Consolidation	Centrifugation	¼-Segment[7]	NA	≤10%
Particle size	Laser	Composite	2 μm[8]	≤18%

[1] Other techniques that meet the required uncertainty are also acceptable.
[2] All analyses are conducted on homogenized samples except for consolidation.
[3] Required sensitivity on a dry basis for solid samples.
[4] Values lower than the desired sensitivity do not require this uncertainty.
[5] Values outside this pH range do not require the specified uncertainty.
[6] NA = Not applicable.
[7] Consolidation tests must be conducted on samples before homogenization.
[8] An estimate of the total number and mass of particles under 2 μm in diameter is required. Determination of particle sizes under 2 μm is not necessary.

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3.0 SAMPLE HANDLING AND ANALYTICAL PROCESS

3.1 TANK SAMPLING

Tank 241-T-107 was push-mode core sampled during a period from November 5, 1992 to March 15, 1993. Initially, two core samples were scheduled for the tank but because of poor sample recovery, a third core sample was taken (Silvers and Noonan 1993). Core 50 was obtained from riser No. 2; Core 51 was obtained from riser No. 5, and Core 52 was obtained from riser No. 3. Each core was composed of four segments. The first segment in Core 50 was resampled because the sampler was left in the tank riser for more than 48 hours, exceeding a requirement that the sample be received at the laboratory within 48 hours of sampling (Silvers and Noonan 1993). Water was used as the hydrostatic head fluid during sampling; normal paraffin hydrocarbon was not used. The samples were transported to the Westinghouse Hanford Company (WHC) 222-S Laboratory for extrusion and analysis. Selected analyses were also performed at the Pacific Northwest Laboratory's (PNL) Analytical Chemistry Laboratory.

3.2 CORE SAMPLE DESCRIPTIONS

The core samples were extruded at the 222-S Laboratory. Table 3-1 provides a listing of the amount of sample recovered and a description of each of the segments. For each core, segments 2 through 4 were expected to be 48-cm (19-in.) long with a total volume of 187 mL/segment. Because of the tank waste level, Segment 1 was expected to be only ¼ full (12 cm [~5 in.] of sample). Table 3-2 summarizes the sample recovery for each segment.

Poor recovery resulted in insufficient sample to meet the half-segment analysis criteria that existed at the time of sampling. The subsequent safety criteria document (Postma et al. 1994) and the Ferrocyanide DQO (Meacham et al. 1994) require analysis at the quarter-segment level. Therefore, it may be necessary to resample tank 241-T-107 to definitively categorize the tank.

Table 3-1. Sample Descriptions.

Segment	Core 50	Core 51	Core 52
1	Contained 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	Sampler was completely empty.	Contained 28.46 g of medium to dark gray solids. One side appeared to be dark gray, the rest was light gray. No drainable liquids.
1R	Contained 25.58 g of light brown solids, homogeneous mixture. 10.89 g of opaque brown drainable liquid.	NA	NA
2	Contained 194.45 g of solids. Sampler was under pressure. Solids were inhomogeneous and ranged from a light brown section, similar to segment 1 except little darker in color, to medium brown solids, to a dark brown section. No drainable liquid.	Contained 64.48 g of dark brown solids. 87.30 g of opaque drainable liquid; density 1.26 g/mL.	Contained 111.23 g of brown solids. Solids appeared wet.
3	Sample recovered by holding the sampler vertical and tapping with a hammer. 8.53 g of dark brown solids were recovered. The solids were thick and homogeneous. There was 165 g of opaque brown drainable liquid and with a density of 0.96 g/mL.	Contained 215.66 g of dark brown solids. Solids appeared to be homogeneous. No drainable liquids.	Contained 201.41 g of solids. Color ranged from light brown at bottom to dark brown at top. Solids were lumpy. No drainable liquids.
4	A 1.17 g piece of solids that looked like a flat piece of plastic or a piece of gum that had been stepped on was recovered. There was 120.42 g of opaque brown drainable liquid with a density 0.97 g/mL.	Contained 206.16 g of dark brown solids. Top 2.5 cm (1 in.) and bottom 15 cm (6 in.) appeared to have more fluids. No drainable liquids.	Contained 4.25 g of light brown solids. 117.34 g of brown turbid drainable liquid; density 1.12 g/mL

NA = Not applicable.

Table 3-2. Volumetric Sample Recovery.

Core	Segment	% Sample Recovery	Sample obtained		Comments
			% Solids	% Liquids	
50	1	36	72	28	Not used per AE&R direction
50	1R	34	70	30	Single-segment sample
50	2	94	100	0	Single-segment sample
50	3	96	5	95	Single-segment sample
50	4	67	1	99	Single-segment sample
51	1	0	0	0	No sample
51	2	64	40	60	Single-segment sample
51	3	100	100	0	Two half-segment samples
51	4	100	100	0	Two half-segment samples
52	1	43	100	0	Single-segment sample
52	2	56	100	0	Single-segment sample
52	3	95	100	0	Two half-segment samples
52	4	60	3	97	Single-segment sample

AE&R = Analytical Evaluation and Reporting

3.3 SAMPLE PREPARATION

Sample analysis was performed in accordance with the requirements of the following documents.

- *Tank Waste Remediation System Tank Waste Characterization Plan* (Hill 1992)
- *Sampling and Analysis of SST and DST Waste Tanks in Support of TWRS Fiscal Year 1993 Statement of Work* (Rich 1993)
- *Technical Project Plan for 222-S Laboratory in Support of Tank Waste Remediation System Tank Waste Characterization Plan (WHC-SD-WM-PLN-047, Rev. 0) Statement of Work (WHC-SOW-93-0002)* (Winters 1992).

The analyses were performed prior to the development of the Ferrocyanide DQO (Meacham et. al 1994) and the sample breakdown and analysis requirements differed from those of the Ferrocyanide DQO.

Most of the analyses were performed on core composite samples. Selected analyses were to be performed at the half-segment level. Because of limited sample recoveries, a core composite was not prepared for Core 50 and some segments were not split into half segments for analysis. Samples for Core 50 consisted of segment samples 1R, 2, and 3 and a drainable liquid composite. Samples for Core 51 consisted of segment/subsegment samples 2, 3U (upper half of segment 3), 3L (lower half of segment 3), 4U, and 4L; a drainable liquid composite; and a core solids composite. Samples for Core 52 consisted of segment/subsegment samples 1, 2, 3U, and 3L; a drainable liquid composite; and a core solids composite. This sample splitting scheme differs from the Ferrocyanide DQO data requirements, (Meacham et al. 1994) which calls for analysis at the quarter-segment and core composite levels.

Sample matrices were analyzed directly or prepared using water digestion, acid digestion, or potassium hydroxide (KOH) fusion prior to analysis. Acid digestions were performed using a mixture of hydrochloric and nitric acids. The fusions were performed by placing samples in nickel crucibles and fusing them using KOH. Although the fusion method is more likely to dissolve solid components than the acid digestion, it has the disadvantage of diluting the sample more, thereby increasing the detection limit and making trace elements less likely to be detected or analyzed correctly.

4.0 DISCUSSION OF ANALYTICAL RESULTS

This section presents analytical results and compares them to the analytical data requirements of the Ferrocyanide DQO (Meacham et al. 1994). All reported concentrations and results are based on grams of wet sample, unless otherwise specified. When results have been converted to a dry sample basis, the gravimetric weight percent water result for the sample was used to make the conversion. If a gravimetric result was not available, the thermogravimetric analysis (TGA) result was used.

Analytical uncertainties are discussed when the primary or secondary analysis results are at or above 25% of the decision thresholds. For tertiary analyses, analytical uncertainties are discussed if the analytical results are at or above the required sensitivities.

4.1 THERMAL ANALYSIS

4.1.1 Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is used to identify the potential for an exothermic reaction in the waste upon heating. DSC is also used to identify secondary reactions or a change in state that may occur as a result of temperature increases. DSC analysis measures the amount of heat released or absorbed by a sample while it is 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 sample to remove decomposition gases. A graph of the change in heat versus time is plotted. On these particular graphs, an upward peak indicates exothermic behavior while a downward peak indicates endothermic behavior. The computer program on the DSC can calculate the change in heat evolution, whether endothermic or exothermic, by integrating the area under the curve.

DSC was performed on nonhomogenized facies, homogenized segments/subsegments, and drainable liquid composites. Duplicate analyses were performed on most samples, although duplicate analyses were required only when an exothermic reaction was observed. Results of the DSC analysis are provided in Table 4-1. The only sample from tank 241-T-107 to exhibit an exothermic reaction was Core 50, Segment 4. In this sample, observation of the extruded sample noted "a flat piece of plastic or a piece of gum that had been stepped on." This item was specifically placed in a vial for DSC/TGA analysis. The sample and duplicate exhibited an exothermic reaction, beginning at about 300 °C, of 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. DSC measurements with nitrogen as a cover gas were not performed/requested. With the exception of this piece of plastic, which was not representative of the surrounding waste

matrix, no exothermic reactions were observed in the tank 241-T-107 samples. The DSC results suggest that tank 241-T-107 should be categorized as *Safe* (i.e., the fuel concentration is less than 115 cal/g).

Table 4-1. DSC Results.

Sample	Results
Core 50, Segment 1R, nonhomogenized	No exothermic activity
Core 50, Segment 1R, homogenized	No exothermic activity
Core 50, Segment 2, nonhomogenized	No exothermic activity
Core 50, Segment 2, homogenized	No exothermic activity
Core 50, Segment 3, nonhomogenized	No exothermic activity
Core 50, Segment 4, nonhomogenized	306 cal/g beginning at ~300 °C
Core 50, drainable liquid composite	No exothermic activity
Core 51, Segment 2, homogenized	No exothermic activity
Core 51, Segment 3U, homogenized	No exothermic activity
Core 51, Segment 3L, homogenized	No exothermic activity
Core 51, Segment 4U, homogenized	No exothermic activity
Core 51, Segment 4L, homogenized	No exothermic activity
Core 51, drainable liquid composite	No exothermic activity
Core 52, Segment 1, homogenized	No exothermic activity
Core 52, Segment 2, homogenized	No exothermic activity
Core 52, Segment 3U, homogenized	No exothermic activity
Core 52, Segment 3L, homogenized	No exothermic activity
Core 52, Segment 4, homogenized	No exothermic activity
Core 52, drainable liquid composite	No exothermic activity

4.1.2 Thermogravimetric Analysis And Gravimetric Analysis

TGA 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 was air for the TGA measurements. The percent water is calculated by measuring the weight loss at 100 °C. The numbers produced may vary 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 4-2 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 4-2.

Table 4-2. 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.

4.2 CHEMICAL ANALYSIS

4.2.1 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 cyanide concentration is considerably lower than the established decision threshold of 3.9 wt% (39,000 μg/g dry sample) cyanide. A comparison of the core composites and water digestion results indicated that most of the cyanide is present in water soluble form. Table 4-3 summarizes the cyanide results.

Table 4-3. Cyanide Results.

Sample	Cyanide (μg/g)	Cyanide (μg/g dry sample)	Ferrocyanide equivalent[1] (μg/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[2]	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 μg/mL	299	406
Core 51, drainable liquid composite	152 μg/mL	513	697
Core 52, drainable liquids composite	39.8 μg/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 [$Fe(CN)_6^{-4}$].
[2]IS = Insufficient sample for analysis.

4.2.2 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. These analyses were performed by Pacific Northwest Laboratory's Analytical Chemistry Laboratory. 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.

Results for the hot persulfate oxidation and coulometric detection methods are shown in Tables 4-4 and 4-5. TOC results for all samples are well below the Ferrocyanide DQO-established decision threshold of 3 wt% (30,000 μg/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 the Core 52, Segment 3U which had an 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 and the coulometric detection method. 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.

Table 4-4. Total Carbon, Total Inorganic Carbon, and Total Organic Carbon Results for Segments and Subsegments.

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 4-5. Total Carbon, Total Inorganic Carbon, and Total Organic Carbon Results for Drainable Liquids and Core Solids Composites.

Sample	Method	Total carbon (μg/g)	Total inorganic carbon (μg/g)	Total organic carbon (μg/g)	Total organic carbon (μg/g dry sample)
Core 50, drainable liquid composite	Coulometric detection	1,660 μg/mL	512 μg/mL	1,150 μg/mL	25,600
Core 51, drainable liquid composite	Coulometric detection	5,600 μg/mL	4,540 μg/mL	1,060 μg/mL	3,580
Core 52, drainable liquids composite	Coulometric detection	693 μg/mL	339 μg/mL	354 μg/mL	2,360
Core 51, core solids composite, water digest	Coulometric detection	7,120	5,680	1,440	2,990
Core 51, core solids composite, direct	Hot persulfate oxidation	2,480	2,080	400	832
Core 52, core solids composite, water digest	Coulometric detection	4,740	2,780	1,690	3,750
Core 52, core solids composite, direct	Hot persulfate oxidation	1,640	1,320	320	613

4.2.3 Inductively Coupled Plasma Analysis

Analysis for metals was performed by inductively coupled plasma atomic emission spectroscopy (ICP). Analyses were performed on drainable liquid composites, core solids composites, and segments or subsegment samples. Preparation methods used on the core solids composites were water digestion, acid digestion, and KOH fusion. The segment and subsegment samples were prepared by KOH fusion only. Homogenization check samples (one segment/subsegment per core) were prepared by acid digestion and analyzed using ICP.

ICP results for nickel, aluminum, calcium, iron, phosphorus, and sodium are shown in Tables 4-6 and 4-7. Because nickel crucibles are used in the KOH fusion, nickel results for the fusion samples are not usable and are not presented. Although nickel concentration data are available only for the acid digested homogenization segments or subsegments, water and acid digested core composites, and drainable liquid composites, all results are below the 1,000 μg/g (dry basis) decision threshold in the Ferrocyanide DQO.

The DQO does not provide a decision threshold for aluminum, calcium, iron, phosphorus, or sodium but does specify a required analytical sensitivity of 500 μg/g (dry basis). Although the detection limits frequently exceeded this value, the analytical results were generally well above the detection limits. The exception is the drainable liquids analyses, where results were sometimes near the detection limits. In reviewing the aluminum data, most of the samples contain less than 5.0×10^4 μg/g. Only Core 50, Segment 2 and Core 52, Segment 1 have higher concentrations (9.29×10^4 and 2.14×10^5 μg/g, respectively). Calcium results are quite uniform except for the Core 52, Segment 1 samples and one of the four core composite results. This anomalous calcium concentration could be attributed to glove powder used by personnel in the laboratory. Iron concentrations appear to be fairly consistent throughout the tank. Phosphorus results show widely differing phosphorus content in different areas of the tank. Sodium results have matched duplicate pairs but appear to vary by location in the tank. Composite results for sodium are generally higher than segment results.

Table 4-6. ICP Results for Nickel.

Sample	Nickel (μg/g)	Nickel (μg/g dry sample)
Core 50, Segment 2, acid digestion	148	252
Core 51, Segment 3L, acid digestion	216	458
Core 52, Segment 3L, acid digestion	35.2	75.6
Core 50, drainable liquid composite	1.77 μg/mL	39.4
Core 51, drainable liquid composite	16.0 μg/mL	54.0
Core 52, drainable liquid composite	2.79 μg/mL	18.6
Core 51, core solids composite, water digestion	4.49	9.33
Core 51, core solids composite, acid digestion	304	632
Core 52, core solids composite, water digestion	<13.2	<25.3
Core 52, core solids composite, acid digestion	279	534

Table 4-7. ICP Results for Aluminum, Calcium, Iron, Sodium, and Phosphorus.

Sample	Aluminum (μg/g)	Calcium (μg/g)	Iron (μg/g)	Sodium (μg/g)	Phosphorus (μg/g)
Core 50, Segment 1R, fusion	9,810	1,050	19,000	127,000	30,600
Core 50, Segment 2, fusion	92,900	822	20,400	55,200	3,840
Core 50, Segment 2, acid digestion	95,600	900	24,100	71,300	5,240
Core 50, Segment 3, fusion	20,700	1,100	23,800	123,000	42,700
Core 50, Segment 4, fusion	IS	IS	IS	IS	IS
Core 51, Segment 2, fusion	12,000	2,090	36,700	71,100	5,330
Core 51, Segment 3U, fusion	1,240	961	28,500	108,000	25,100
Core 51, Segment 3L, fusion	688	989	34,300	77,900	7,610
Core 51, Segment 3L, acid digestion	267	848	40,700	89,400	9,320
Core 51, Segment 4U, fusion	2,270	1,430	35,100	82,500	9,700
Core 51, Segment 4L, fusion	9,210	2,440	19,700	122,000	32,400
Core 52, Segment 1, fusion	214,000	10,900	40,500	27,300	<226
Core 52, Segment 2, fusion	43,000	771	21,000	105,000	25,600
Core 52, Segment 3U, fusion	8,170	800	23,400	131,000	36,900
Core 52, Segment 3L, fusion	15,400	422	19,000	107,000	26,000
Core 52, Segment 3L, acid digestion	15,800	499	20,200	113,000	26,600
Core 52, Segment 4, fusion	IS	IS	IS	IS	IS

Table 4-7. ICP Results for Aluminum, Calcium, Iron, Sodium, and Phosphorus.

Sample	Aluminum (μg/g)	Calcium (μg/g)	Iron (μg/g)	Sodium (μg/g)	Phosphorus (μg/g)
Core 50, drainable liquid composite	4.25 μg/mL	4.28 μg/mL	5.75 μg/mL	14,600 μg/mL	790 μg/mL
Core 51, drainable liquid composite	11.5 μg/mL	4.97 μg/mL	4.8 μg/mL	95,500 μg/mL	2,020 μg/mL
Core 52, drainable liquid composite	47.8 μg/mL	3.94 μg/mL	19.0 μg/mL	51,900 μg/mL	2,590 μg/mL
Core 51, core solids composite, water digestion	485	476	272	134,000	30,300
Core 51, core solids composite, acid digestion	4,140	853	33,200	137,000	29,600
Core 51, core solids composite, fusion	5,730	779	26,500	123,000	32,900
Core 52, core solids composite, water digestion	816	65.5	439	81,800	17,100
Core 52, core solids composite, acid digestion	24,600	592	29,800	124,000	30,400
Core 52, core solids composite, fusion	26,900	742	31,900	112,000	31,300

IS = Insufficient sample for analysis

4.2.4 Anion Analysis

Chloride, fluoride, nitrate, nitrite, and phosphate analyses was performed using ion chromatography (IC). Because the nitrite results from IC analysis are considered estimates, nitrite analyses were also performed using spectrophotometric methods. These analyses were performed on the drainable liquid composites and on the water digestions of the solid core composites.

Results of the analyses are shown in Table 4-8. The IC revealed relatively high concentrations of all anions in the core samples except chloride. A comparison of the nitrite results from both IC the spectrophotometry show good agreement between the two methods. With the exception of the Core 51 drainable liquid composite, RPDs between the two

methods are less than 10%. The phosphate results compare quite well with the ICP phosphorus results. If it is assumed that all the phosphorus exists as phosphate, the RPDs between the two methods is less than 5% for the drainable liquids and the Core 51 core solids composite samples. There is a discrepancy between the IC and ICP results for the Core 52 core solids composite samples; the phosphate results are more than twice as high as the phosphorus. The detection limits did not always meet the DQO's required sensitivity of 500 μg/g dry sample, but IC results for the water digestion samples and all the spectrophotometric nitrite results were well above the detection limits. IC results for the drainable liquids were often near the detection limits.

Table 4-8. Anion Results.

Sample	F^- (μg/g)	Cl^- (μg/g)	PO_4^{-3} (μg/g)	NO_3^- (μg/g)	NO_2^- (IC) (μg/g)	NO_2^- (spec) (μg/g)
Core 50, drainable liquid composite	174 μg/mL	196 μg/mL	2,400 μg/mL	21,200 μg/mL	2,580 μg/mL	2,730 μg/mL
Core 51, drainable liquid composite	825 μg/mL	1,340 μg/mL	6,240 μg/mL	134,000 μg/mL	27,600 μg/mL	12,500 μg/mL
Core 52, drainable liquid composite	673 μg/mL	860 μg/mL	7,630 μg/mL	100,000 μg/mL	8,060 μg/mL	7,420 μg/mL
Core 51, core solids composite, water digestion	9,200	682	94,500	92,800	15,300	14,200
Core 52, core solids composite, water digestion	13,700	412	133,000	58,000	8,360	7,980

4.2.5 pH Analysis

Analyses for pH were performed on the direct segments/subsegments, core composites, and drainable liquid composite samples. Results are given in Table 4-9. The pH results ranged from 9.6 to 11.8. Duplicate analyses were run for fourteen of the samples; sample and duplicate results differed by no more than 0.1 pH unit.

Table 4-9. pH Results.

Sample	pH
Core 50, Segment 1R	10.3
Core 50, Segment 2	11.2
Core 50, Segment 3	11.4
Core 50, Segment 4	IS
Core 51, Segment 2	10.5
Core 51, Segment 3U	11.4
Core 51, Segment 3L	11.4
Core 51, Segment 4U	11.2
Core 51, Segment 4L	11.6
Core 52, Segment 1	10.5
Core 52, Segment 2	11.4
Core 52, Segment 3U	11.8
Core 52, Segment 3L	10.9
Core 52, Segment 4	IS
Core 50 drainable liquid composite	9.6
Core 51, drainable liquid composite	10.7
Core 52, drainable liquid composite	10.3
Core 51, core solids composite	11.6
Core 52, core solids composite	11.4

IS = Insufficient sample for analysis

4.3 RADIOCHEMICAL ANALYSIS

4.3.1 Gamma Energy Analysis

Gamma energy analysis (GEA) was performed on the drainable liquid composites, the fusion and water digestions of the solid core composites, and the fusion of the segment/subsegment samples. Cesium-137, Co^{60}, Eu^{154}, and Eu^{155} results are shown in Table 4-10. Americium-241 results are presented and discussed in Section 4.3.3, along with the alpha energy analysis (AEA) results for Am^{241}. With one exception, all Cs^{137} results were below

the decision threshold of 200 μCi/g dry sample. Segment 2 of Core 51 had a Cs^{137} concentration of 99.9 μCi/g (251 μg/g dry sample), five to ten times higher than any other sample. A confirmation analysis was performed and the results proved similar to previous numbers. The Core 51 solids composite results were not correspondingly high. The discrepancy may be due to sample inhomogeneity.

Cobalt-60, Eu^{154}, and Eu^{155} results were generally below detection limits. The detection limits (below 0.2 μCi/g) and the results were well below the DQO's required sensitivity.

Table 4-10. GEA Results.

Sample	Co^{60} (μCi/g)	Eu^{154} (μCi/g)	Eu^{155} (μCi/g)	Cs^{137} (μCi/g)	Cs^{137} (μCi/g dry sample)
Core 50, Segment 1R	$<6.8\times10^{-3}$	0.0885	0.0859	7.03	8.6
Core 50, Segment 2	$<1.27\times10^{-2}$	$<3.14\times10^{-2}$	$<7.01\times10^{-2}$	11.8	20.2
Core 50, Segment 3	$<5.94\times10^{-3}$	$<1.78\times10^{-2}$	$<4.11\times10^{-2}$	6.04	10.7
Core 50, Segment 4	IS	IS	IS	IS	IS
Core 51, Segment 2	$<1.05\times10^{-2}$	0.314	$<1.41\times10^{-1}$	99.9	251
Core 51, Segment 3U	$<3.93\times10^{-3}$	$<2.11\times10^{-2}$	$<8.60\times10^{-2}$	15.3	34.1
Core 51, Segment 3L	0.0376	0.0437	$<9.49\times10^{-2}$	17.1	36.3
Core 51, Segment 4U	0.0264	$<2.52\times10^{-2}$	$<9.49\times10^{-2}$	17.9	39.8
Core 51, Segment 4L	$<8.92\times10^{-3}$	$<2.12\times10^{-2}$	$<5.35\times10^{-2}$	13.6	26.9
Core 52, Segment 1	$<6.39\times10^{-3}$	1.08	0.919	10.9	13.1
Core 52, Segment 2	$<2.51\times10^{-2}$	$<7.37\times10^{-2}$	$<8.27\times10^{-2}$	10.3	20.0
Core 52, Segment 3U	$<2.13\times10^{-2}$	$<7.74\times10^{-2}$	$<8.11\times10^{-2}$	7.83	16.1
Core 52, Segment 3L	$<2.54\times10^{-2}$	$<6.74\times10^{-2}$	$<6.86\times10^{-2}$	10.6	22.8
Core 52, Segment 4	IS	IS	IS	IS	IS
Core 50, drainable liquid composite	$<5.28\times10^{-4}$ μCi/mL	$<1.44\times10^{-3}$ μCi/mL	$<3.14\times10^{-3}$ μCi/mL	1.72 μCi/mL	38.3
Core 51, drainable liquid composite	$<1.35\times10^{-3}$ μCi/mL	$<4.23\times10^{-3}$ μCi/mL	$<1.50\times10^{-2}$ μCi/mL	18.4 μCi/mL	62.1
Core 52, drainable liquid composite	$<1.24\times10^{-4}$ μCi/mL	$<5.50\times10^{-4}$ μCi/mL	$<2.65\times10^{-3}$ μCi/mL	5.23 μCi/mL	34.9
Core 51, core solids composite, water digestion	$<1.45\times10^{-3}$	$<3.91\times10^{-3}$	$<1.25\times10^{-2}$	12.0	24.9
Core 51, core solids composite, fusion	$<2.39\times10^{-2}$	$<6.44\times10^{-2}$	$<9.21\times10^{-2}$	13.9	28.9
Core 52, core solids composite, water digestion	$<2.08\times10^{-3}$	$<5.82\times10^{-3}$	$<1.51\times10^{-2}$	9.49	18.2
Core 52, core solids composite, fusion	$<6.58\times10^{-3}$	0.0688	$<4.07\times10^{-2}$	10.6	20.3

IS = Insufficient sample for analysis

4.3.2 Strontium-90 Analysis

Strontium-90 analyses were performed on drainable liquid composites and fusion preparations of solid core composites and segment/subsegment samples. Results are presented in Table 4-11. RPDs for twelve of the seventeen sets of analyses were below 10%. RPDs that exceeded the 10% criterion ranged from 11% to 28%. Strontium results vary widely, but are generally near or above the 200 μCi/g dry sample decision threshold. The highest results (up to 504.4 μCi/g dry sample) are seen in Core 51.

Table 4-11. Strontium-90 Results.

Sample	Sr^{90} (μCi/g)	Sr^{90} (μCi/g dry sample)
Core 50, Segment 1R	31.6	38.5
Core 50, Segment 2	153	262
Core 50, Segment 3	125	221
Core 50, Segment 4	IS	IS
Core 51, Segment 2	189	475
Core 51, Segment 3U	201	448
Core 51, Segment 3L	242	514
Core 51, Segment 4U	227	504
Core 51, Segment 4L	27.5	54.5
Core 52, Segment 1	165	198
Core 52, Segment 2	88.0	171
Core 52, Segment 3U	95.9	197
Core 52, Segment 3L	18.1	38.9
Core 52, Segment 4	IS	IS
Core 50, drainable liquid composite	0.0108 μCi/mL	0.241
Core 51, drainable liquid composite	0.123 μCi/mL	0.415
Core 52, drainable liquid composite	0.0449 μCi/mL	0.300
Core 51, core solids composite	132	274
Core 52, core solids composite	84.1	161

IS = Insufficient sample for analysis

4.3.3 Total Beta Analysis

Total beta analysis was performed on the fusion and water digestions of the core solids composite samples and on the drainable liquid composites. Total beta results are shown in Table 4-12. Total beta results from the 222-S Laboratory are based on the efficiency of the detector for Co^{60}. Beta emissions from other isotopes have lower or higher efficiencies depending on their energies. Because Co^{60} is lower in energy than the isotopes usually present in Hanford Site wastes, the total beta results are usually biased high. Cesium-137 and Sr^{90} are the major beta emitters in the tank waste. Total beta results were compared to the sum of the Cs^{137} and Sr^{90} results. Total beta results were higher with the ratio of total beta to cesium and strontium ranging from about 1.3 for drainable liquid and water digestion samples to about 2.7 for fusion samples. The RPDs for the total beta results were 14% or lower.

Table 4-12. Total Beta Results.

Sample	Total beta (μCi/g)
Core 50, drainable liquid composite	2.5 μCi/mL
Core 51, drainable liquid composite	25.1 μCi/mL
Core 52, drainable liquid composite	7.55 μCi/mL
Core 51, core solids composite, water digestion	16.5
Core 51, core solids composite, fusion	404
Core 52, core solids composite, water digestion	10.3
Core 52, core solids composite, fusion	257

4.3.4 Alpha Energy Analysis

AEA was performed on fusion preparations for one subsegment and core composites. Drainable liquid composites were also analyzed by AEA. AEA results for Pu^{238}, $Pu^{239/240}$, and Am^{241} are shown in Table 4-13. Americium-241 results from GEA analysis are also shown for comparison. All the results are below the required sensitivity of 50 μCi/g (dry sample). AEA detection limits were below 0.02 μCi/g for Pu^{238}, 10^{-4} μCi/g for $Pu^{239/240}$ and Am^{241}. Americium-241 detection limits were below 0.1 μCi/g for the GEA.

Table 4-13. Plutonium^{238}, Plutonium$^{239/240}$, and Americium^{241} Results.

Sample	Pu^{238} (μCi/g)	$Pu^{239/240}$ (μCi/g)	Am^{241} (AEA) (μCi/g)	Am^{241} (GEA) (μCi/g)
Core 51, Segment 4L	$<1.70\times10^{-2}$	0.173	NR	$<8.58\times10^{-2}$
Core 50, drainable liquid composite	$<9.01\times10^{-5}$ μCi/mL	6.57×10^{-5} μCi/mL	4.03×10^{-5} μCi/mL	7.33×10^{-3} μCi/mL
Core 51, drainable liquid composite	0.0250 μCi/mL	0.0114 μCi/mL	0.000204 μCi/mL	$<3.30\times10^{-2}$ μCi/mL
Core 52, drainable liquid composite	$<1.71\times10^{-4}$ μCi/mL	$<6.70\times10^{-5}$ μCi/mL	0.00007 μCi/mL	$<5.90\times10^{-3}$ μCi/mL
Core 51, core solids composite	0.0166	0.117	0.0113	$<6.14\times10^{-2}$
Core 52, core solids composite	$<1.65\times10^{-2}$	0.183	0.0168	$<8.73\times10^{-2}$

DL = Detection limit
NR = Analysis not required

4.3.5 Uranium Analysis

Uranium analysis was performed on the drainable liquid composites and fusion digestions of the core solids composites and Core 51, Segment 4L. Analyses were performed using a laser fluorometer; results are shown in Table 4-14. The RPDs were under 25% for all samples except the replicate analyses for the Core 51 core composite, which had an RPD of 88%.

Table 4-14. Uranium Results.

Sample	Uranium (μg/g)
Core 50, Segment 4L	7,440
Core 50, drainable liquid composite	95.3 μg/mL
Core 51, drainable liquid composite	588 μg/mL
Core 52, drainable liquid composite	40.6 μg/mL
Core 51, core solids composite	26,300
Core 52, core solids composite	18,900

4.3.6 Total Alpha Analysis

Total alpha analysis was performed on the drainable liquid composites and the fusion and water digestions of the core solids composites. Total alpha results are shown in Table 4-15. The total alpha concentration frequently tends to be somewhat lower than the sum of the individual alpha emitters; the difference is likely due to absorption by the salt residue on the counting mounts. However, in this case, the total alpha results are higher than the sum of the alpha emitters (U^{238}, $Pu^{239/240}$, and Am^{241}). The ratio of total alpha to the sum of the alpha emitters ranges from 1.4 to 6.6 and does not appear to follow any pattern among the samples. The higher total alpha concentration may be due to (1) high counting error; (2) cross talk from Cs^{137} and Sr^{90}/Y^{90}; or (3) another isotope may be present which was not measured. All total alpha results are below the 50 μCi/g dry sample required sensitivity of the Ferrocyanide DQO (Meacham et al. 1994).

Table 4-15. Total Alpha Results.

Sample	Total alpha (μCi/g)
Core 50, drainable liquid composite	0.000914 μCi/mL
Core 51, drainable liquid composite	0.0166 μCi/mL
Core 52, drainable liquid composite	0.000511 μCi/mL
Core 51, core solids composite, water digestion	0.000520
Core 51, core solids composite, fusion	0.473
Core 52, core solids composite, water digestion	0.00743
Core 52, core solids composite, fusion	0.395

4.4 PHYSICAL ANALYSIS

Bulk density measurements were performed on segments or subsegments, drainable liquid composites, and core solids composite samples; results are shown in Table 4-16. The bulk density measurement on the homogenized solids samples were around 1.5 g/mL. Several segments (Core 50, Segment 2; Core 51, Segment 3L; and Core 52, core solids composite) produced anomalous densities which were much lower (1.2 g/mL) or higher (1.7 g/mL) than the other solids samples. However, an average density for all solid segments, including the anomalous points, was 1.51 g/mL. Duplicate analyses were performed on the drainable liquid samples only; RPDs for these samples were less than 2%.

Table 4-16. Bulk Density Results.

Sample	Density (g/mL)
Core 50, Segment 1R	IS
Core 50, Segment 2	1.71
Core 50, Segment 3	IS
Core 50, Segment 4	IS
Core 51, Segment 2	IS
Core 51, Segment 3U	1.49
Core 51, Segment 3L	1.70
Core 51, Segment 4U	1.48
Core 51, Segment 4L	1.53
Core 52, Segment 1	IS
Core 52, Segment 2	1.55
Core 52, Segment 3U	1.50
Core 52, Segment 3L	1.52
Core 52, Segment 4	IS
Core 50, drainable liquid composite	1.02
Core 51, drainable liquid composite	1.20
Core 52, drainable liquid composite	1.11
Core 51, core solids composite	1.46
Core 52, core solids composite	1.19

IS = Insufficient sample for analysis

Only one particle size analysis was performed on the tank 241-T-107 core samples as only one stratum was visually observed in the waste. An aliquot from Core 50, Segment 2, was analyzed. The number distribution range was 0.5 to 8 μm with a median of 0.85 μm: about 90% of the particles appear to be 2 μm or smaller. The volume distribution range was 0.10 to 150 μm with a median of 32.97 μm: less than 5% of the volume was made up of particles 2 μm or smaller. Some particles may have been greater than 150 μm but this number was the upper limit on the analyzer.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The results of the analysis of the three core samples from tank 241-T-107 have been compared to the primary, secondary, and tertiary data requirements of the Ferrocyanide Safety Issue DQO. The analytical results indicate that the fuel concentration in the tank will not support a self-sustaining (i. e., propagating) reaction.

With the exception of a piece of plastic recovered from Core 50, Segment 4, the DSC results for all waste samples exhibited no exothermic reactions. The plastic is not representative of the surrounding waste and was found not to react in the absence of air. The stability of the waste is also supported by the low cyanide and TOC concentrations observed in the waste. The analytical results suggest that an exothermic reaction in tank 241-T-107 is unlikely and the tank should be categorized as *Safe*. However, core sampling yielded insufficient recovery to meet the quarter-segment analysis requirement defined in the Ferrocyanide DQO (Meacham et al. 1994) and safety criteria document (Postma et al. 1994). A decision on the need to resample tank 241-T-107 will be made after more samples are taken from the remaining Ferrocyanide Watch List tanks.

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