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**Sampling and Analysis of Inactive
Radioactive Waste Tanks W-17,
W-18, WC-5, WC-6, WC-8, and
WC-11 Through WC-14
at ORNL**

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Chemical Technology Division

**SAMPLING AND ANALYSIS OF INACTIVE RADIOACTIVE WASTE
TANKS W-17, W-18, WC-5, WC-6, WC-8, AND
WC-11 THROUGH WC-14 AT ORNL**

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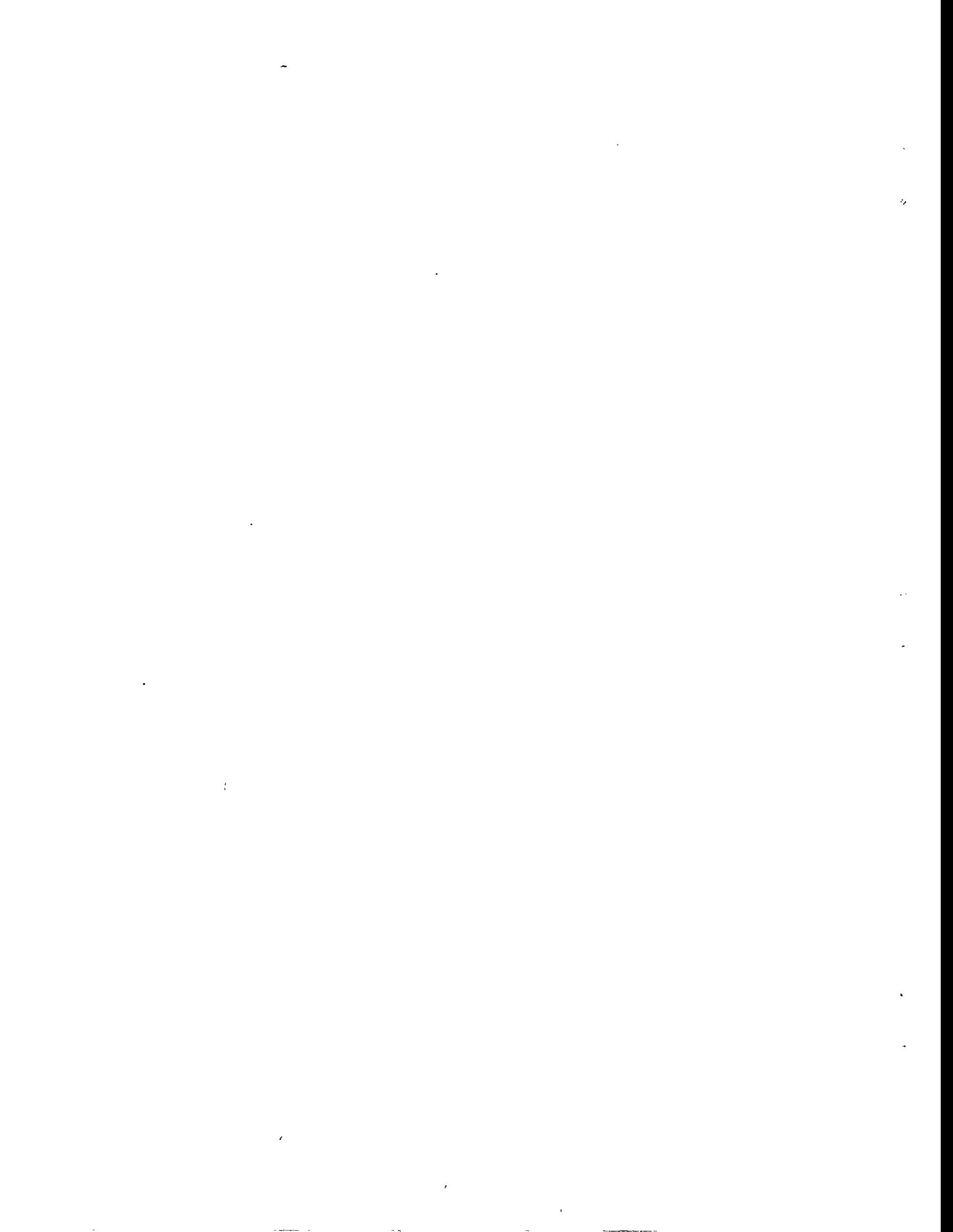
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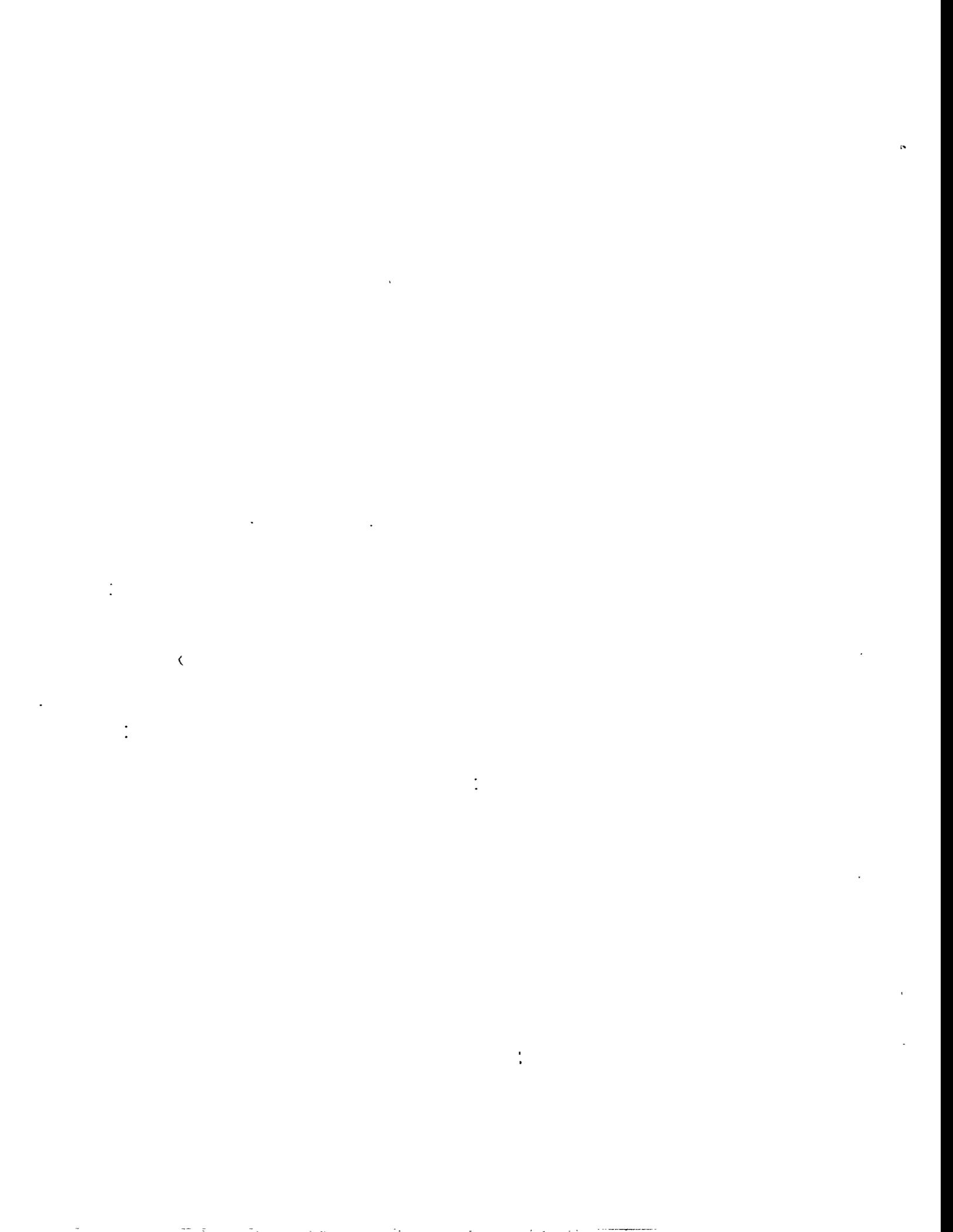
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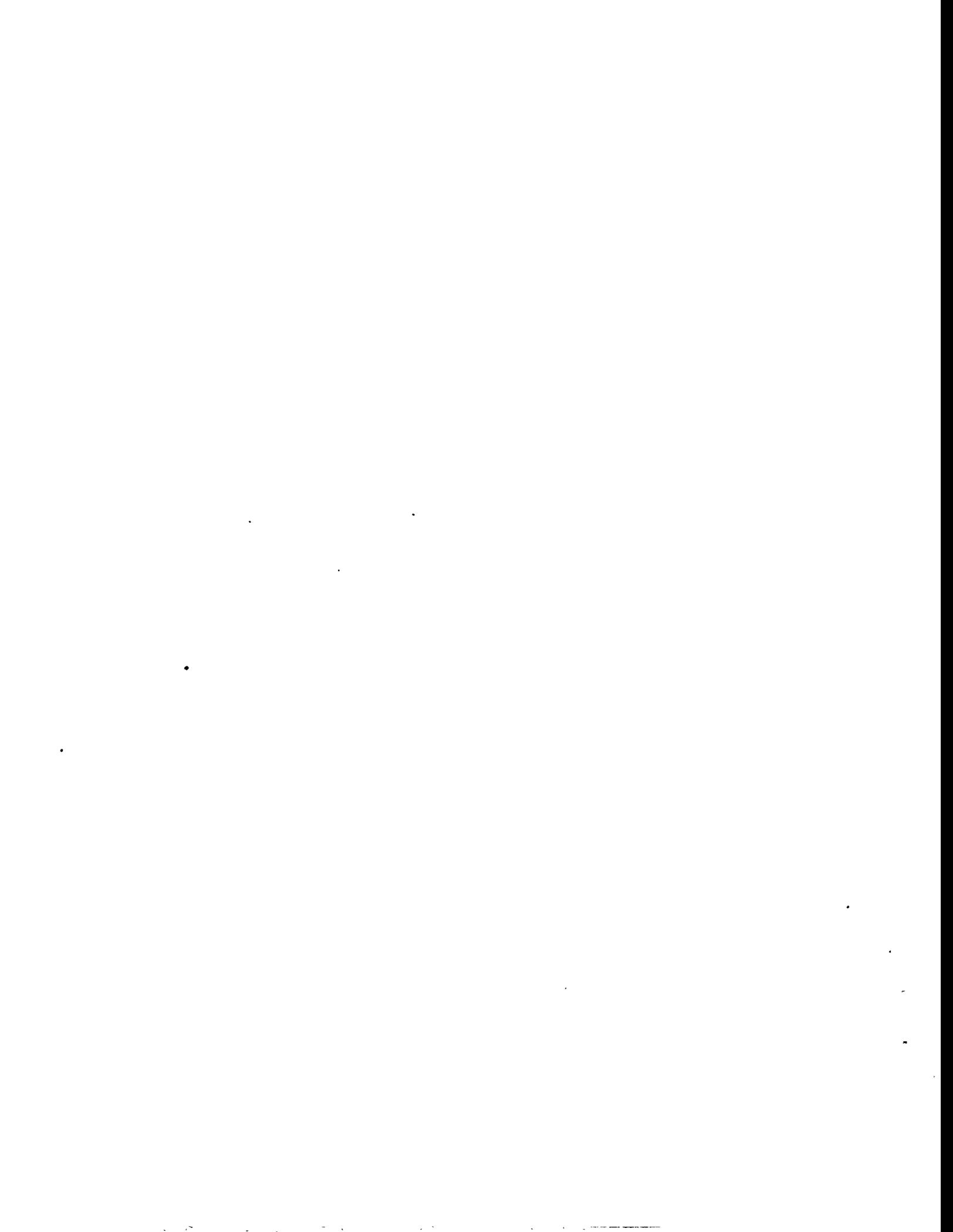
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ACRONYMS AND INITIALISMS

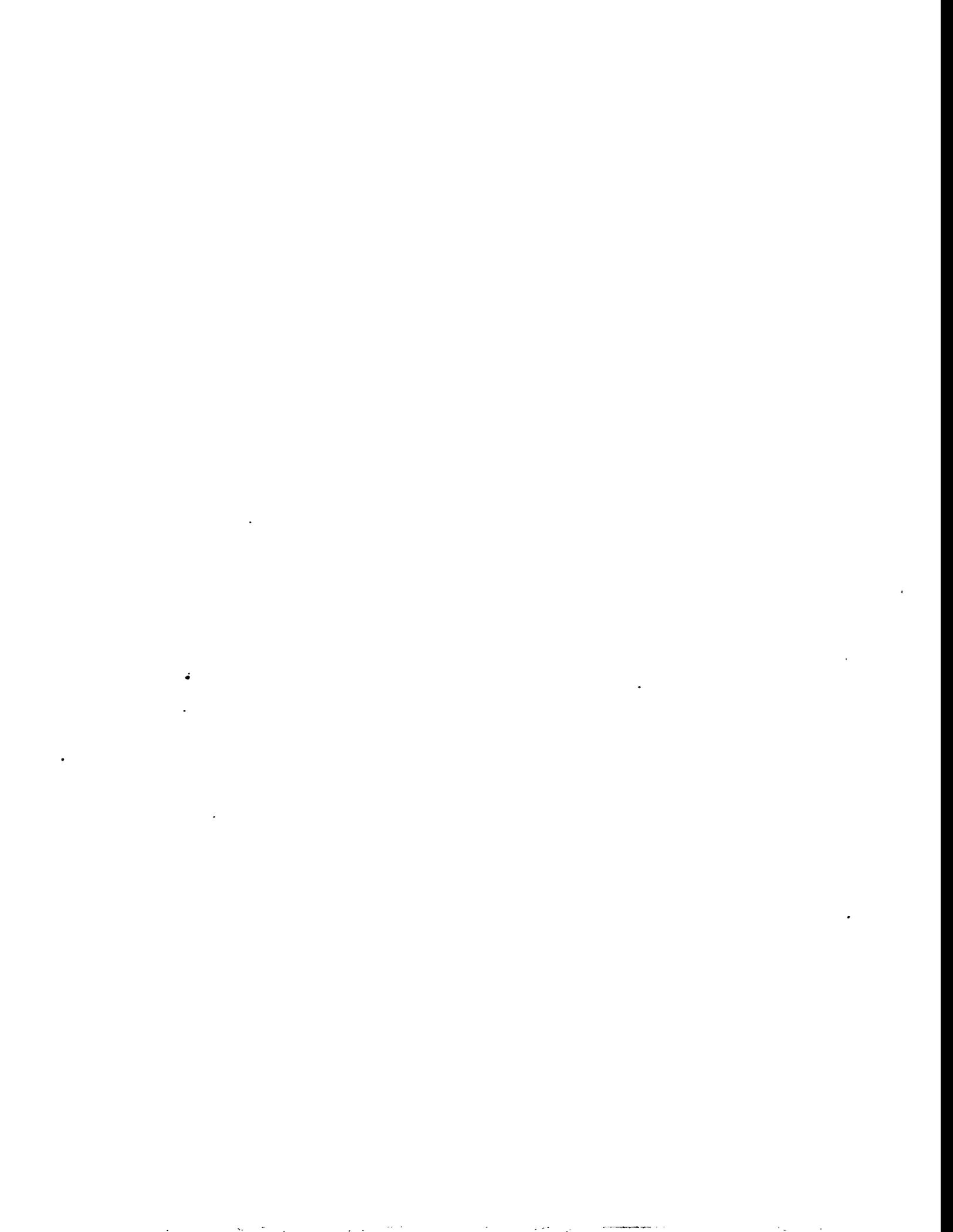
AA	Atomic absorption
ACD	Analytical Chemistry Division
ALARA	As low as reasonably achievable
CLP	Contract Laboratory Program (EPA)
DAI-GC	Direct aqueous injection gas chromatography
DCBP	Decachlorobiphenyl
EPA	U.S. Environmental Protection Agency
FFA	Federal Facility Agreement
GC	Gas chromatography
GC-ECD	Gas chromatography—electron capture detection
GC-FID	Gas chromatography—flame ionization detector
GC-MS	Gas chromatography—mass spectrometry
HEPA	High-efficiency particulate air (filtration system)
HP	Health physicist
IC	Inorganic carbon
ICP	Inductively coupled plasma
ICP-AES	Inductively coupled plasma—atomic emission spectroscopy
ID	Internal diameter
IDL	Instrument detection limit
LLLW	Liquid low-level waste
LLW	Low-level waste
MS	Matrix spike
MSD	Matrix spike duplicate
MVST	Melton Valley Storage Tank
NAA	Neutron activation analysis
NITS	National Institute of Standards and Technology
OD	Outer diameter
ORNL	Oak Ridge National Laboratory
PCB	Polychlorinated biphenyl
PRD	Percent relative difference
PVC	Polyvinyl chloride (polymer)
QA	Quality assurance
QC	Quality control
R&D	Research and development
RCRA	Resource Conservation and Recovery Act
RMAL	Radioactive Materials Analytical Laboratory
RPD	Relative percent difference
SVOA	Semivolatile organic compound analysis
TAL	Transuranium Analytical Laboratory
TC	Total carbon
TCL	Target Compound List
TIC	Tentatively identified compound
TOC	Total organic carbon
TRU	Transuranic
VOA	Volatile organic compound analysis



ABSTRACT

The sampling and analysis of nine inactive liquid low-level waste (LLLW) tanks at the Oak Ridge National Laboratory (ORNL) are described—tanks W-17, W-18, WC-5, WC-6, WC-8, and WC-11 through WC-14. Samples of the waste tank liquids and sludges were analyzed to determine (1) the major chemical constituents, (2) the principal radionuclides, (3) metals listed on the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program Inorganic Target Analyte List, (4) organic compounds, and (5) some physical properties. The organic chemical characterization consisted of determinations of the EPA Contract Laboratory Program Target Compound List volatile and semivolatile compounds, pesticides, and polychlorinated biphenyls (PCBs).

This report provides data (1) to meet requirements under the Federal Facility Agreement (FFA) for the Oak Ridge Reservation to characterize the contents of LLLW tanks which have been removed from service and (2) to support planning for the treatment and disposal of the wastes.



1. INTRODUCTION

This report describes the sampling and analysis of nine inactive liquid low-level waste (LLLW) tanks at the Oak Ridge National Laboratory (ORNL)—tanks W-17, W-18, WC-5, WC-6, WC-8, and WC-11 through WC-14. Objectives include providing data (1) to meet requirements of the Federal Facility Agreement (FFA) for the Oak Ridge Reservation and (2) to support planning for the treatment and disposal of the wastes. These tanks were used for the collection and transfer of LLLW. They were installed in the early 1950s and were removed from programmatic service under the FFA effective January 1, 1992. At the time of this survey, the tanks contained residual waste from past operations and were collecting nonprogrammatic waste, which is pumped periodically to the active LLLW system for treatment. As a result, the analyses in this report should be considered a "snapshot" in time.

Samples of the supernatant liquid were collected from the midpoint of the liquid layer. The air-liquid interface was sampled to determine the presence of an immiscible (i.e., organic) layer floating on the aqueous layer. Then, depending upon the tank contents, either liquid samples or two "cores" of sludge were collected from the bottom of the tank. Samples of the wastes were analyzed to determine (1) major chemical constituents, (2) principal radionuclides, (3) hazardous constituents, (4) organic compounds, and (5) general waste characteristics.

Background information on the tanks is given in Sect. 2, and an overview of the field sampling and analytical activities is given in Sect. 3. Summaries of the waste characterization data are presented in Sect. 4 in a data manual type format. Details of the work are presented in the appendixes including field sampling data (Appendix A), the inorganic and radiochemical characterization (Appendixes B and C), and the organic chemical characterization (Appendixes D and E). These appendixes provide information concerning the analytical methods, quality control (QC), and comprehensive listings of the analytical data.

This is one of several reports on the characterization of radioactive tank wastes at ORNL. The results of the sampling and analysis of 33 inactive tanks have been reported in refs. 1 and 2, and the characterization of the wastes stored in the Melton Valley Storage Tanks (MVSTs) and evaporator facility storage tanks in ref. 3.

1.1 REFERENCES

1. J. W. Autrey et al., *Sampling and Analysis of the Inactive Waste Storage Tank Contents at ORNL*, ORNL/ER-13, Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory, Oak Ridge, Tennessee, September 1990.
2. J. W. Autrey et al., *Sampling and Analysis of the Inactive Waste Tanks TH-2, WC-1, and WC-15*, ORNL/ER-19, Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory, Oak Ridge, Tennessee, February 1992.
3. M. B. Sears et al., *Sampling and Analysis of Radioactive Liquid Wastes and Sludges in the Melton Valley and Evaporator Facility Storage Tanks at ORNL*, ORNL/TM-11652, Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory, Oak Ridge, Tennessee, September 1990.



2. BACKGROUND

The waste tanks characterized in this study were used for the collection and transfer of LLLW. They were installed in the early 1950s and were buried underground to minimize radiation exposure to personnel. Wastes generated by various research and development (R&D) activities were discharged from the source building to the collection tank which served the building. In many instances, waste from hoods, hot cells, and glove boxes drained by gravity through unvalved piping to the tank. The wastes that accumulated in the collection tank were then pumped periodically to the low-level waste (LLW) evaporator facility for treatment, and from there the waste concentrates were placed in the Melton Valley and evaporator facility storage tanks. The waste tanks sampled in this study were removed from programmatic service under the FFA effective January 1, 1992.

The locations of the tanks within the main ORNL plant area are shown in Fig. 2.1. Tanks W-17 and W-18 are in the South Tank Farm, near the corner of Central Avenue and Fourth Street. Tanks WC-5, WC-6, and WC-8 are south of Building 3503 near Southside Drive. Tanks WC-11 through WC-14 are located in the WC-10 tank farm, which lies south of Building 3587 near the corner of White Oak Avenue and Fifth Avenue. The piping access points for sampling the tanks in the WC-10 tank farm are inside a shelter (Building 3618).

General information about the tanks is given in Table 2.1. Most of the tanks in this group are 1000-gal tanks, except tanks WC-11 (4600 gal) and WC-5 (500 gal). The tanks and associated piping are constructed of type 347 stainless steel. A schematic of a typical installation is shown in Fig. 2.2. The tanks are buried underground on a concrete "saucer," which has a sump at its low point and a "dry well" that extends to the surface for monitoring the ground water. These are vertically oriented tanks with dished-bottom heads, except tank WC-11 (4600 gal), which is a horizontal tank. Waste transfers are handled remotely using pumps or steam jets. The tanks are equipped with pneumatic liquid-level measurement instrumentation and with conductivity high-level alarm devices. The signals are monitored at the Waste Operations Control Center in Building 3130. Tanks WC-11 through WC-14 are ventilated by a central hot off-gas system. These tanks are operated under a small negative pressure relative to the atmosphere (typically -1 in. water gauge), and are vented to a scrubber and high-efficiency particulate air (HEPA) filtration system, and then the effluent is released through the 3039 stack. The other tanks are vented through a local HEPA filtration system at the tank farm.

At the time of this survey, the tanks contained residual wastes from past operations and were collecting nonprogrammatic inflow, which is pumped periodically to the active LLLW system for treatment. Because the wastes may contain chlorides or other agents which are corrosive in acid solution, a basic heel is maintained in the collection tanks to neutralize any acid. This practice has been continued since the shutdown of programmatic service. After a tank is pumped, sodium hydroxide (NaOH) solution is added to the tank (e.g., 2 gal of 50 wt % NaOH to a 1000-gal tank).

Six tanks were pumped shortly (1-8 days) before the liquid samples were collected. In these cases (W-17, W-18, WC-5, WC-8, WC-11, and WC-12), each tank was sampled after it was pumped, but before the NaOH solution was added to the tank.

Table 2.1. Description of the waste tanks

Tank	Location	Buildings served	Nominal capacity (gal)	Transfer method	Ventilation system
W-17	South tank farm	3026C	1000	Jet	Local HEPA
W-18	South tank farm	3026C	1000	Jet	Local HEPA
WC-5	South of Bldg. 3503	3503 3508	1000	Pump	Local HEPA
WC-6	South of Bldg. 3503	3508	500	Pump	Local HEPA
WC-8	South of Bldg. 3503	3503 Pump pit	1000	Pump	Local HEPA
WC-11	South of Bldg. 3587	4500N 4505 4507	4600	Submerged pump	Hot off-gas
WC-12	South of Bldg. 3587	4505	1000	Submerged pump	Hot off-gas
WC-13	South of Bldg. 3587	4500N 4500S 4501 4508	1000	Submerged pump	Hot off-gas
WC-14	South of Bldg. 3587	4501 4507	1000	Submerged pump	Hot off-gas

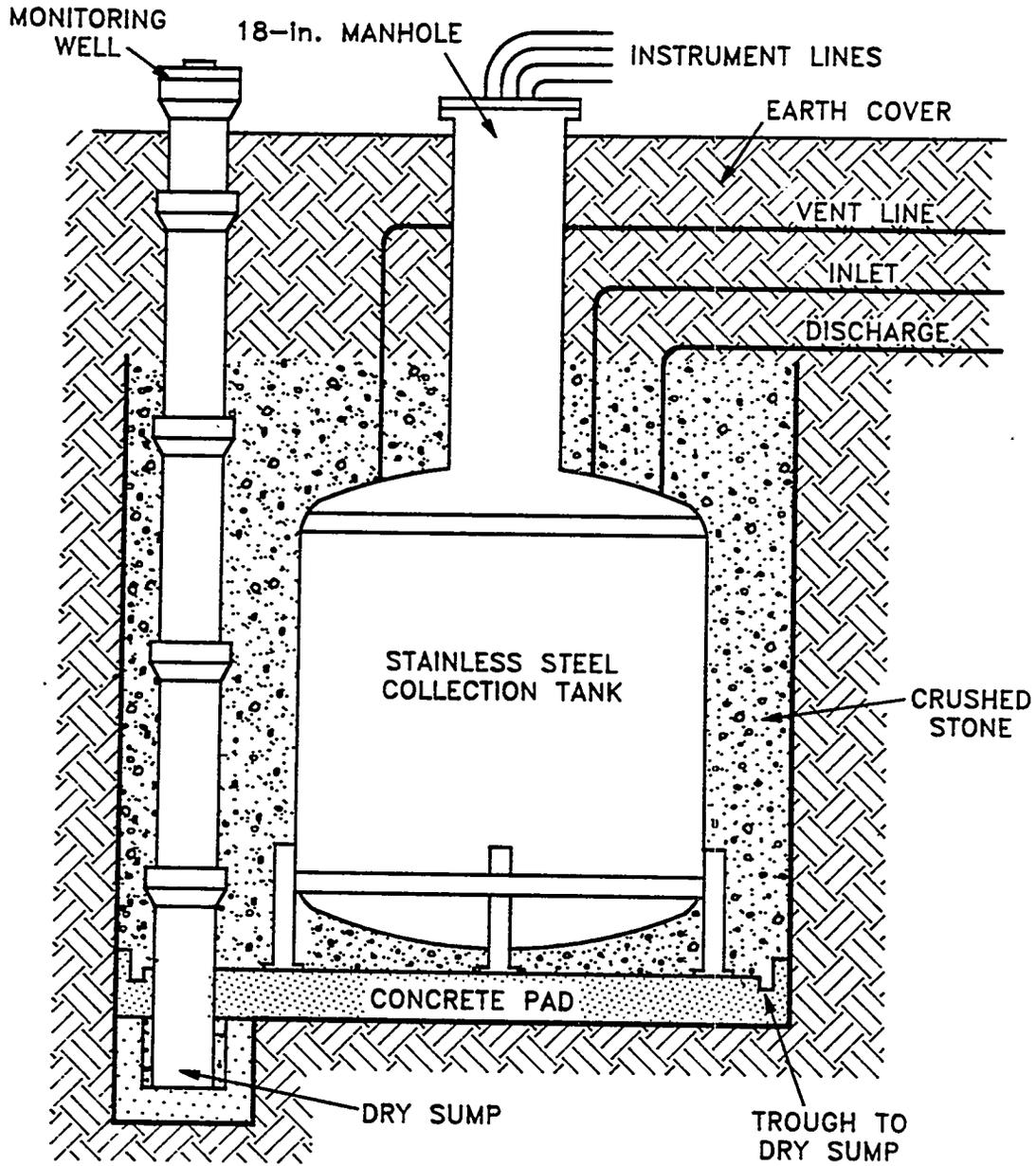


Fig. 2.2. Typical waste collection tank installation.

3. SAMPLING AND ANALYTICAL ACTIVITIES

3.1 SAMPLE COLLECTION

The sampling plan provides information concerning the aqueous liquid layer, the air-liquid interface, and the layer near the bottom of the tank. Most of the tanks had been pumped or jetted before the start of the sampling campaign, and the liquid layer was relatively shallow (≤ 10 -in. deep, except tank WC-5, which was 20-in. deep). Liquid samples were collected first from the midpoint of the liquid layer to minimize the potential for contamination of the aqueous supernate if other phases were present. The air-liquid interface was sampled to check for the presence of an immiscible (i.e., organic) layer floating on the aqueous layer. Then, depending upon the tank contents, either liquid samples or two "cores" of sludge were collected from the bottom of the tank. A sludge-level detector was used to determine the depth of the supernatant liquid and either the location of the liquid-sludge interface or the distance to the tank bottom. Because only the sludge located directly under the access port can be sampled, sludge samples may not be representative of other locations in the tank and, therefore, should be considered merely an indicator of the tank contents.

A general description of the sample collection is presented in this section. The sampling methods are essentially the same as those used in earlier waste tank sampling campaigns.¹⁻³ Sample quantities were limited to keep radiation exposures to personnel as low as reasonably achievable (ALARA).

Field-sampling data, including the profile of the tank contents and a comprehensive listing of the samples collected (date of collection, sampling location, and radiation level), are given in Appendix A. The field data are from the perspective of the sampling crew who were standing over the tank. The distances were measured from the flange on the access pipe down into the tank.

3.1.1 Tank Access

Samples were drawn through the riser pipe, which penetrates the tank from the ground surface (Fig. 3.1). Most of the tanks were sampled by opening the riser cover. The external piping was disconnected, the bolts on the riser cover were removed, and the cover was slid over to allow about a 3-in.-wide opening. Sampling access is very restricted because of the piping in the tank. For tanks WC-11 through WC-14, the obstructions include the submerged pump, waste-discharge piping, electrical conduit for the pump, and metal tubing (4 lines) for the level instrumentation, all of which are attached to the riser cover.

There is a capped pipe opening through the riser cover of all the tanks. Tanks W-17 and W-18 have a 3-in.-diam opening and were sampled through the pipe nipple on the riser cover. The other tanks have only a 2-in.-diam pipe opening, which is too small to use for the initial measurements to locate the phases in the tank or for sludge sampling. However, when tank WC-12 was resampled in March 1993, the liquid samples were drawn through the 2-in.-diam pipe opening because the tank measurements were known.

The tank-bottom samples were collected ~8 in. from the tank centerline (i.e., near the low part of the dished-bottom head) for samples collected by opening the riser cover, and ~6 in. from the centerline for samples collected through the pipe nipple.

3.1.2 Presampling Survey

A presampling survey was conducted before the samples were collected. At the time of the survey, the air-liquid interface and either the liquid-sludge interface or the tank bottom were located using a Markland Model 10 Sludge Gun,TM thus establishing the depth of the supernatant liquid in the tank. This instrument measures the amount of light transmitted across a fixed gap in a probe. As the probe detects a phase change or enters the sludge, the instrument sounds a "beep," and the operator logs the depth from the tank access point (i.e., the flange on the access pipe). This information was then used in making final plans for sample collection.

The presampling survey was conducted 1–3 days before the liquid samples were collected from the tank. This allowed time for any disturbed solids to settle before sampling. The Markland instrument is capable of detecting immiscible liquid layers (e.g., an organic layer over an aqueous layer) if the layers are deep enough to give a reading with the probe—a distance of about 2 in. vertically. No immiscible liquid layers were detected in the tanks by the sampling team using the Markland instrument.

3.1.3 Collection of Liquid Samples with the Vacuum-Pump System

Liquid samples were collected using a vacuum-pump sampling system. Samples were taken from (1) the midpoint of the liquid layer, (2) the air-liquid interface, and (3) the bottom of the tank if the tank did not contain sludge. The exception was tank W-18, in which the waste layer was only ~1-in. deep, and only a tank-bottom sample was collected.

A schematic of the liquid sampling system is shown in Fig. 3.1. The sample was pulled by vacuum from the specified level in the tank through TeflonTM tubing (1/4-in. OD) into the sample jar. The pump was arranged with a safety surge bottle (~4 L) as a backup. A metal tip, a standard stainless steel pipe nozzle 4-in. long by 3/8-in. OD, was attached with a stainless steel Swagelok fitting to the lower end of the Teflon tubing to keep the tubing vertical. Liquid samples were collected in precleaned 250-mL glass sample jars and sealed with Teflon-lined caps. Actual sample volumes were ~180–200 mL with ~1 cm (~15–25 mL) headspace.

The radiation level of the sample was surveyed by the health physicist. Most of the samples did not require shielding. The dose rates for the samples containing solids from the bottom of tank WC-12 were higher than the others (up to 900 mrem/h); these samples were placed in lead pigs.

The usual sampling method was to collect eight bottles of sample material from the midpoint of the liquid layer, one from the air-liquid interface, and five from the tank bottom. If an organic layer was observed, two or three bottles of sample material were pulled from the top surface. However, fewer samples were taken from the bottom of tank WC-12 because of the high-gamma-activity solids.

3.1.3.1 Midpoint of the Liquid Layer

The depth of the liquid phase and the sampling locations of the midpoint samples were determined from the Markland measurements. The Teflon tubing was cut to length, premeasured, and marked with tape to indicate when the end of the tubing had been lowered below the access flange to the appropriate level within the tank liquid. The upper end of the tubing was plugged while the tubing was lowered to restrict the entry of liquid until the desired depth was reached.

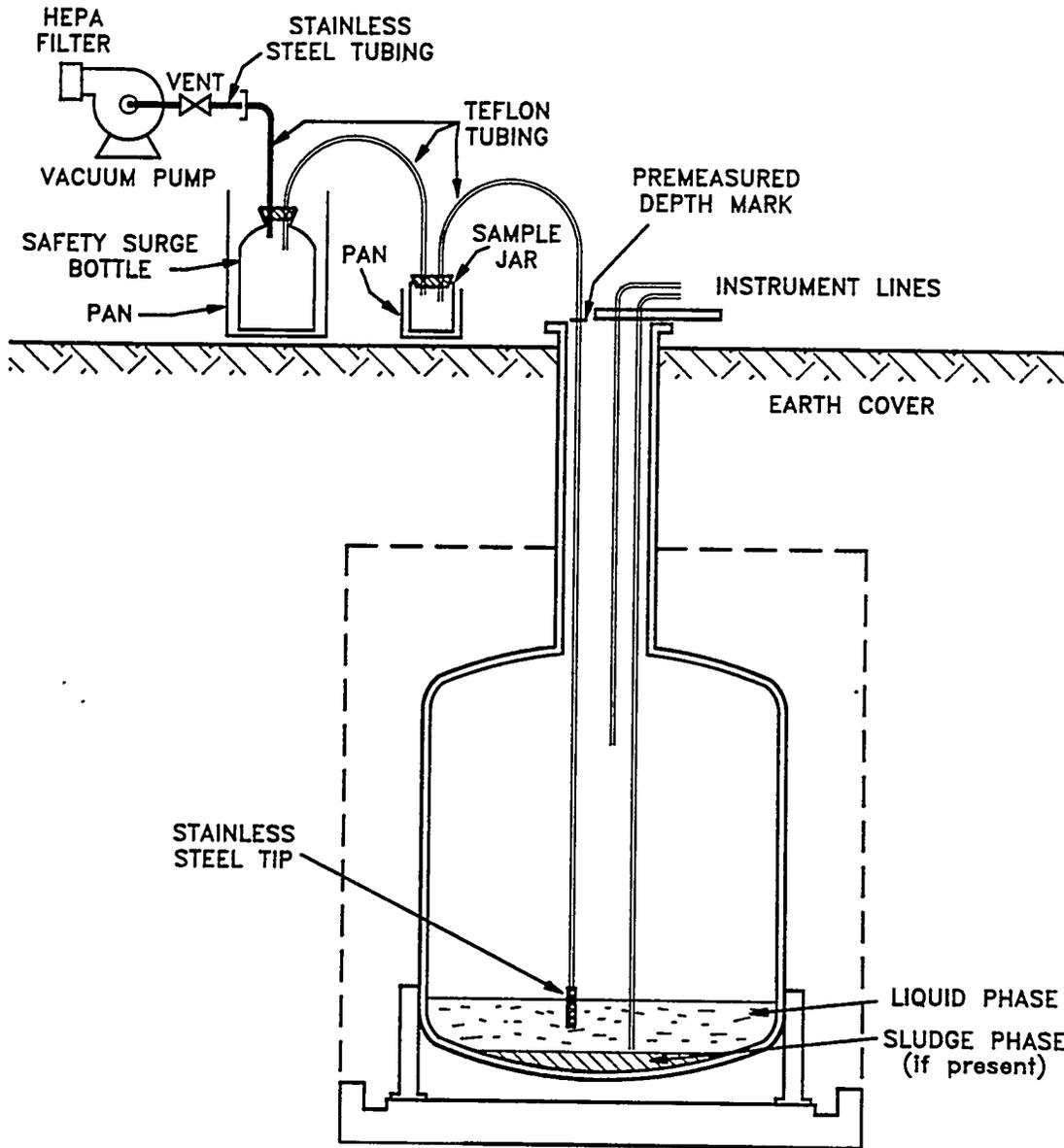


Fig. 3.1. Liquid sample collection system.

3.1.3.2 Air-Liquid Interface

The air-liquid interface was checked for the presence of an immiscible organic layer floating over the aqueous layer. The tubing was positioned above the air-liquid interface. The pump was then turned on, and with the pump running, the tubing was slowly lowered until the tubing barely skimmed the top of the liquid surface. Then a sample was collected. Most of the liquid-surface samples were clear. However, samples pulled from the top of the liquid in tanks WC-11 and WC-13 contained a brown organic layer floating on the aqueous layer. For these samples, the end of the tubing was slowly lowered through the organic layer until the pump was pulling only heavier (i.e., aqueous) liquid and then raised back into the organic layer to obtain a more representative sample of the organic material.

3.1.3.3 Tank Bottom

To collect the tank-bottom samples, the tubing was lowered until the stainless steel tip touched the bottom of the tank, and the sample was then pumped. Several tanks (W-18, WC-5, WC-6, and WC-11) contained a thin layer of sediment on the bottom, while tank WC-12 contained about a 0.5-in.-deep layer of a very thin, watery sludge. Because these layers were too shallow to sample with the soft-sludge sampler (Sect. 3.1.4), they were sampled as liquid samples using the vacuum-pump sampling system to suction material from the tank bottom. This method provides qualitative information about the bottom layer and the sediments. No immiscible organic layer heavier than water was observed in any of the liquid samples which were pumped from the bottom of a tank.

3.1.4 Collection of Sludge Samples

A bottom-opening, soft-sludge sampling device was used to collect sludge samples from tanks WC-13 and WC-14. The device consists of a detachable handle assembly and a hollow probe of polyvinyl chloride (PVC) pipe with a bottom closure that can be controlled from above by the operator (Fig. 3.2). Two "cores" of sludge were collected from each tank—as independently as was possible given the limited tank access. The samples were taken ~8 in. from the tank centerline (i.e., the bottom of the sampler was near the low point of the dished-bottom head). After a sludge sample was collected, the outside of the sampling device was rinsed with deionized water and wiped. The handle was removed, the PVC sample tube was capped, and the vent hole was plugged. The sampler was then wrapped in a custom-made plastic bag and packaged in a can. The radiation level of the sample was surveyed by the health physicist in the field.

The samplers contained both sludge and supernatant tank liquid. Because the sample collector is a clear material, visual measurements of sludge depth can be made, and other properties can be observed.

3.2 SAMPLE ANALYSIS

The radioactive waste tank liquids and sludges were analyzed to determine (1) the major chemical constituents, (2) the principal radionuclides, (3) metals listed on the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program Inorganic Target Analyte List,⁴ (4) organic compounds, and (5) some physical properties. The organic chemical characterization consisted of determinations of the EPA Contract Laboratory Program Target Compound List (TCL) volatile

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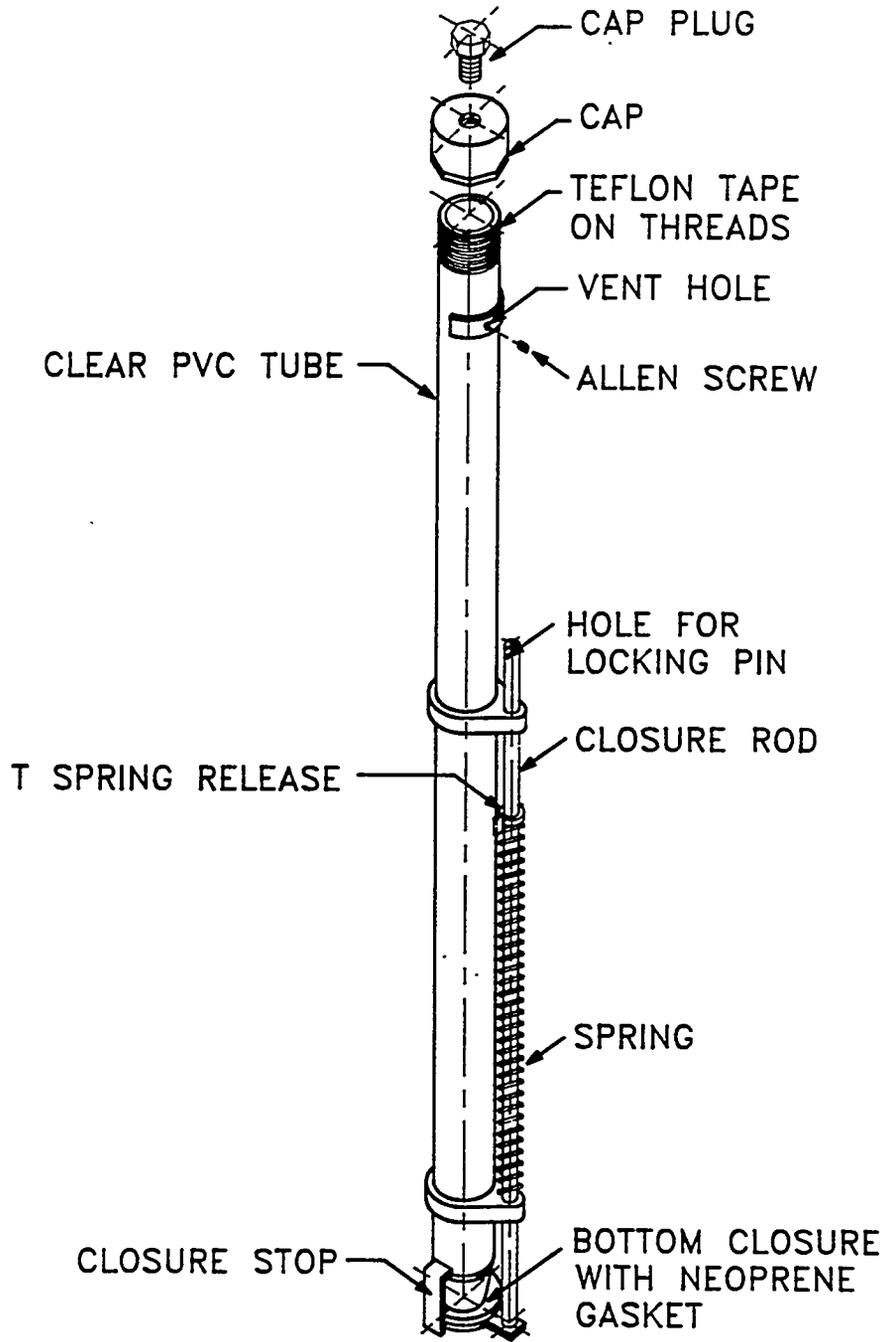


Fig. 3.2. Soft-sludge sampler.

and semivolatile compounds, pesticides, and polychlorinated biphenyls (PCBs).⁵ The overall scheme used for the characterization of the liquid samples is shown in Fig. 3.3. Samples were inspected visually for the presence of immiscible phases. Most of the samples drawn from the midpoint of the supernate were clear, although a few were slightly cloudy. The aqueous samples from the midpoint were analyzed for metals, anions, radioactive materials, organic compounds, and other parameters.

The samples drawn from the air-liquid interface (surface samples) were examined visually for the presence of an immiscible organic layer floating on the aqueous layer. Samples that were clear were photographed for documentation, and the density was determined to check if these were from an aqueous layer. When the sample contained an organic layer, an aliquot of the organic phase was drawn off and analyzed for volatile, semivolatile, and PCB organic compounds.

Several tanks contained a thin layer of sediment on the bottom; these layers were too shallow to sample with the bottom-opening soft-sludge sampler. Therefore, they were sampled as liquid samples using the vacuum-pump sampling system to suction material from the bottom of the tank. The amount of sediment material in these samples was very small (estimated as <0.5 wt % by visual examination). Since the amount of sample material (solids) was limited, the approach used in this study was to analyze one aliquot of the sample including the sediment and a second aliquot (filtered) without the sediment. Radiochemical and a limited metals analyses were performed on both fractions. The difference between the results provides a qualitative indication of the constituents in the solids. Because the samples pulled by suction are probably a mixture of the sediment phase and tank liquid, the results for the samples including the sediment should be considered qualitative information.

Sludge layers were found in tanks WC-13 and WC-14. The overall scheme for the characterization of the sludge samples is shown in Fig. 3.4. Two "cores" were collected from each tank as independently as was possible given the very limited tank access. The samplers contained both sludge and the overlying supernatant tank liquid. At the analytical laboratory, the samples were allowed to stand inside the PVC sampling tubes to allow the sludge solids to settle. The heights of the phases in the tubes were then measured, and the phases were separated. The sludge phases from the two samples were then combined and mixed by sonication to form a composite sample for the tank. Metals, radioactive materials, organic compounds, and other parameters were determined on aliquots of the sludge. The determinations of the metals and radioactive materials were a total analysis after microwave assisted digestion of the sludge in nitric acid. The organic chemical characterization of the sludge was a direct analysis after extraction of the organic compounds with methylene chloride. Tank WC-14 was resampled in August 1993 to confirm the PCB results. The sludge samples from the second set were analyzed as two independent samples. They were mixed by stirring with a spatula.

Details of the analytical work including sample preparation, analytical methods, and quality control are described in Appendixes B and D. The EPA analytical methods were followed, as much as possible, in analyses for constituents listed as hazardous under the Resource Conservation and Recovery Act (RCRA) and for the organic chemical characterization. Generally, modifications of methods from the EPA guide, *Test Methods for Evaluating Solid Waste (SW-846)*, were used.⁶ Some modifications to the standard procedures were necessary because of the radiation levels, salt content, and generally complex chemical matrices in the samples. Methodology was modified or developed on an as-needed basis. Procedures for the other analysis were selected from the ORNL Analytical Chemistry Division methods or modified EPA methods^{6,7} based on the sample characteristics and type of information needed.

The volatile organic analysis by gas chromatography—mass spectrometry (GC-MS) was performed if the sample radionuclide content was low enough to process the sample in a conventional, nonzoned laboratory. If the radioactivity exceeded the guidelines for the nonzoned

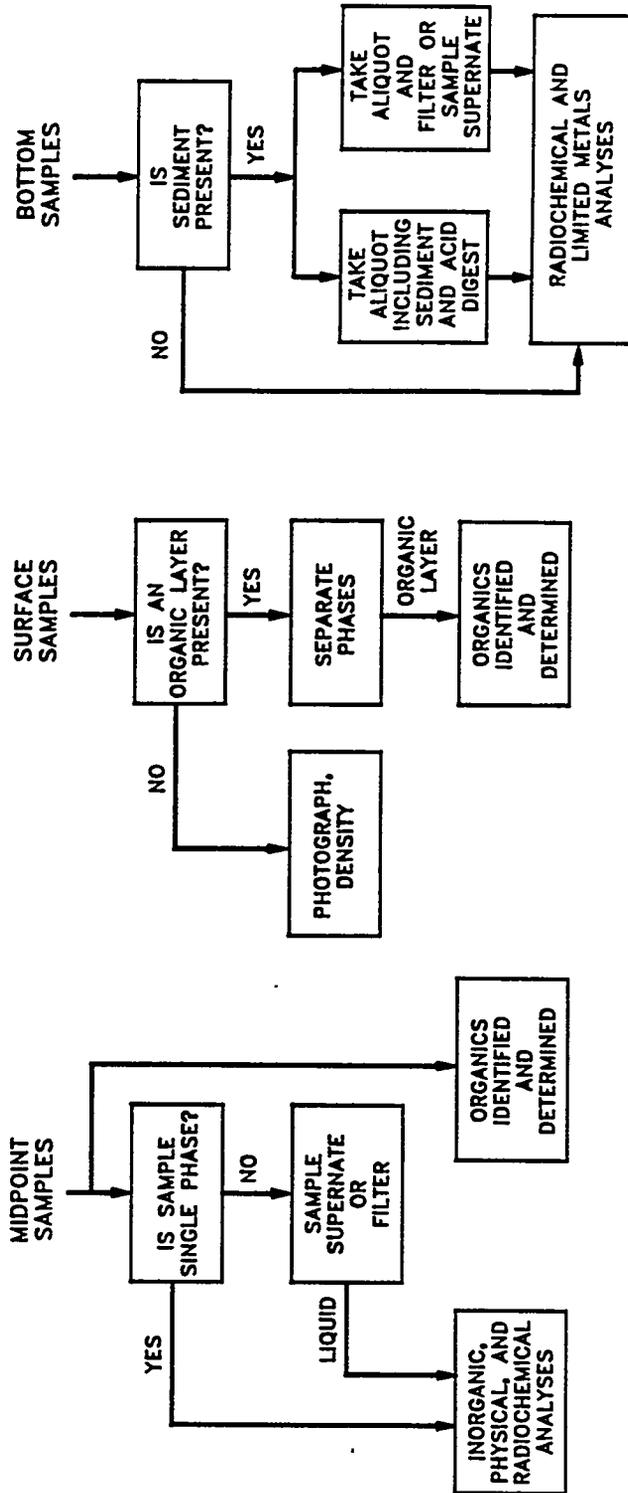


Fig. 3.3. Overall analysis scheme for liquid samples.

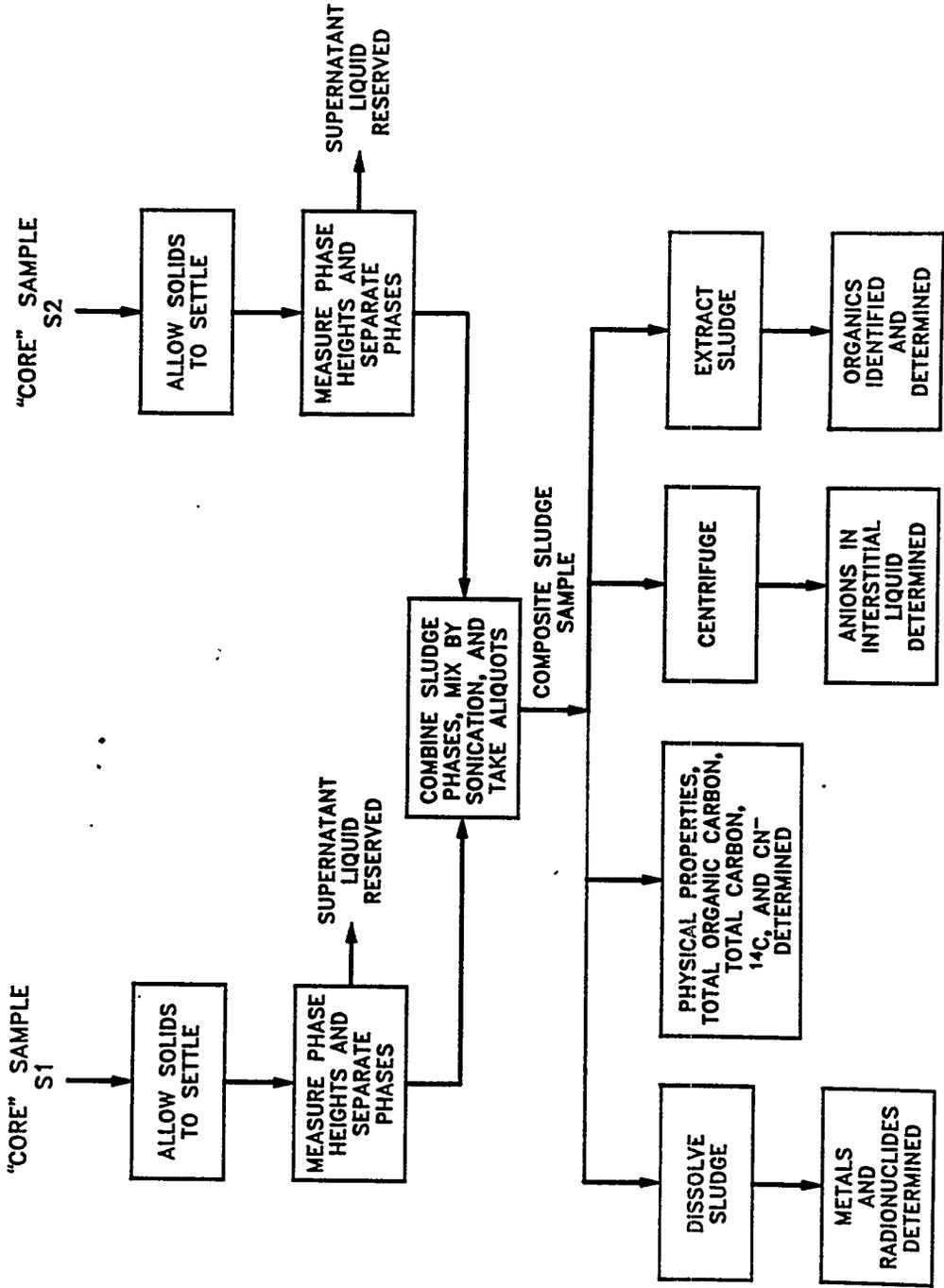


Fig. 3.4. Overall analysis scheme for sludge samples.

laboratory, the volatile organic analysis was not performed. However, water-soluble volatile organics were determined by direct aqueous injection gas chromatography (DAI-GC), which is conducted in a radiochemical laboratory.

3.3 QUALITY ASSURANCE

Field data, such as liquid and sludge depths, sample identification numbers, and field surveys, were recorded on data forms to ensure an accurate account of the sampling activities. Chain-of-custody forms were used to track each sample from its collection point to the analytical laboratory.

Quality assurance (QA) during sample analyses is described in Appendixes B, D, and E, including data for matrix spike recoveries, duplicate samples, laboratory blanks, and surrogate standard recoveries. A sample-integrity and chain-of-custody record form accompanied every sample, subsample, or extract when these were transferred between analytical laboratories.

3.4 REFERENCES

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2. M. B. Sears et al., *Sampling and Analysis of Radioactive Liquid Wastes and Sludges in the Melton Valley and Evaporator Facility Storage Tanks at ORNL*, ORNL/TM-11652, Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory, Oak Ridge, Tennessee, September 1990.
3. J. W. Autrey et al., *Sampling and Analysis of the Inactive Waste Tanks TH-2, WC-1, and WC-15*, ORNL/ER-19, Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory, Oak Ridge, Tennessee, February 1992.
4. U.S. Environmental Protection Agency Contract Laboratory Program, "Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration," SOW No. 787, 1987.
5. U.S. Environmental Protection Agency Contract Laboratory Program, "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration," February 1988.
6. U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, D.C., November 1986; Update I, July 1992; and Proposed Update II, November 1992.
7. U.S. Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, Rev., Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, March 1983.



4. WASTE CHARACTERIZATION SUMMARY

4.1 INTRODUCTION

The purpose of this study was (1) to characterize the tank contents for hazardous and radioactive constituents to meet requirements under the FFA and (2) to provide data to support planning for the treatment and disposal of the wastes. Liquid samples were collected from the midpoint and surface of the liquid layer. Depending upon the tank contents, either liquid samples or two "cores" of sludge were collected from the bottom of the tank. The samples were analyzed to determine (1) the major chemical constituents, (2) the principal radionuclides, (3) metals on the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program Inorganic Target Analyte List,¹ (4) organic compounds, and (5) some physical properties. The organic chemical characterization consisted of determinations of the EPA Contract Laboratory Program Target Compound List (TCL) volatile and semivolatile compounds, pesticides, and polychlorinated biphenyls (PCBs).²

Summaries of the waste characterization data for each tank are presented in Sect. 4.3, and a listing of the phases present in the tanks is given in Table 4.1. Only those organic compounds whose presence was indicated by the analyses are listed in the summary tables. Also while RCRA and proposed RCRA (Ni and Tl) metals are included, in the case of other metal analytes, only those metals present at concentrations ≥ 1 mg/L for liquid samples and ≥ 10 mg/kg for sludge samples are listed in Sect. 4.3. (A complete listing of the analytical data including trace metals below the cutoffs is provided in the appendixes.) Some of the information presented is qualitative, such as the data for samples containing sediment. Because the samples with sediment were collected by suctioning material from the tank bottom, they are probably a mixture of the sediment phase and tank liquid. The results should be considered as merely a qualitative indicator of the constituents in the sediment phase on the tank bottom. These tanks collect nonprogrammatic waste, which is pumped out periodically to the active LLLW system. As a result, the analyses in this report represent a snapshot in time of the tank contents.

The waste characterization is explained more fully and a comprehensive listing of the analytical data are provided in Appendixes A-E. Field-sampling data, including the profile of the tank contents and a complete listing of the samples collected (date of collection, sampling location, and radiation level), are given in Appendix A. The field data are from the perspective of the sampling crew who were standing over the tank. The inorganic and radiochemical analytical support is described in Appendix B. This appendix provides a brief summary of the sample preparation and analytical methods. Information is provided on quality control, matrix spike recovery data, and results for duplicate samples. Comprehensive lists of the physical, inorganic, and radiochemical analytical data for the waste tank liquids and sludges are presented in Appendix C.

The organic chemical characterization is described in Appendix D, and the supporting tables are in Appendix E. The discussion of the organic analysis results in Sect. D.2 is organized by the type of analysis and is more detailed than the summary tank-by-tank presentation in Sect. 4.3. The TCLs (135 organic compounds) and the reporting limits for each compound for conventional analysis of nonradioactive samples are given in Appendix E, Tables E.3-E.6. As discussed in Sect. D.2, the aliquot size and dilution factor affect the reporting limits. These varied somewhat from sample to sample. Conversion factors are given in Tables E.1 and E.2 for calculating the reporting limits for the analyses of these radioactive waste tank samples from the values in Tables E.3-E.6. Results for surrogate standard and matrix spike recoveries are presented in Tables E.11-E.16.

Table 4.1. General characteristics of waste samples

Tank	Aqueous samples: radiation level (mrem/h) (a)	Organic layer	Tank bottom	
			Sediment	Sludge samples: radiation level (mrem/h) (b)
W-17	<0.1	No	No	
W-18	<0.1	No	Yes	
WC-5	<0.1	No	Yes	
WC-6	<0.1	No	Yes	
WC-8	<0.1	No	No	
WC-11	20-25	Yes	Yes	
WC-12	20-24	(c)	(d)	(d)
WC-13	2-3	Yes		28-33
WC-14	22-25 (e)	No		220-500 (f)

(a) Field survey of 200-mL samples in glass jars; samples collected from the midpoint of the liquid layer, except tank W-18, which was from the tank bottom.

(b) Field survey of PVC sampling tubes.

(c) Traces of an oily layer, but too thin to sample for analysis.

(d) Tank WC-12 contained ~0.5-in. deep layer of a thin sludge with high-gamma activity solids. Processed as a liquid sample containing sediment. Dose rates up to ~900 mrem/h with the probe under the sample bottle (i.e., under the sludge layer) and up to ~480 mrem/h with the probe parallel to the sample bottle.

(e) November 1992 sampling.

(f) March 1993 sampling.

4.2 REPORTING CONVENTIONS

An explanation of the reporting conventions is provided herein to clarify the presentation. The headings in the tables refer to sample identification codes. These codes include the tank identification (e.g., W17, W18, WC5, etc.) followed by a hyphen and a tank-phase identification (“-L” and “-Surface” = liquid; “-S” = soft sludge). The term “-Surface” in the code identifies a sample collected from the air-liquid interface. The codes may contain an additional term or terms to indicate the order in which the samples were collected. Usually the “-L1” code sample was collected from the midpoint of the liquid layer, while the “-L2” code sample was collected from the tank bottom. Exceptions are noted in the footnotes to the tables. For example, the waste layer was only ~1 in. deep in tank W-18, and only samples from the tank bottom (W18-L1) were collected from that tank. [A listing of the locations (depths) in the tanks at which the samples were collected is provided in Appendix A.]

Some of the samples contained more than one phase. The terms “filtered” and “including sediment” in the table headings indicate how the sample aliquot was processed, while the terms “oil layer” and “sludge” in the headings indicate the phase assayed (see Sect. 3.2).

Some initialisms and other data reporting conventions used in the tables are as follows:

1. *Inorganic and radiochemical characterization*

Less-than symbol, <	Constituent was not detected, and the limit of detection or the reporting limit is reported.
Asterisk, *	Data are unavailable.
N	Qualitative analysis; N = not detected.
Y	Qualitative analysis; Y = detected.
Parenthesis, ()	Data in parentheses (e.g., Ag) are of questionable quality because of poor spike recovery.

2. *Organic chemical characterization*

Reporting limits	The reporting limits are the concentrations above which the response of the instrument for a calibrated range of concentrations is linear.
B	Data qualifier meaning that the compound also was found in the accompanying laboratory blank sample.
J	Data qualifier meaning that the compound was estimated at a concentration below the reporting limit. Also used to indicate that the concentrations for TICs are estimates.
DAI-GC	Direct aqueous injection gas chromatography.
GC-MS	Gas chromatography—mass spectrometry.

MS	Matrix spike.
MSD	Matrix spike duplicate.
PCB	Polychlorinated biphenyl.
SVOA	Semivolatile organic compound analysis.
TCL	Target Compound List.
TIC	Tentatively identified compound. The identification is based upon the mass spectrum only and the quantitation upon the response factor of the nearest eluting internal standard. All TIC values are estimated and bear the "J" qualifier.

4.3 SUMMARY OF RESULTS

4.3.1 Tank W-17

Tank W-17 contained only a clear, aqueous liquid. No sediment was observed, and there was no indication of any organic layers that are immiscible with the aqueous phase. Dose rates (field survey) for ~200-mL samples were <0.1 mrem/h.

The samples were basic and contained dilute NaOH. Very few inorganic analytes were detected in the waste, and their concentrations (except sodium) were low (Table 4.2). The gross alpha and gross beta activities were <1 and 24 Bq/mL, respectively.

Very little organic material on the TCLs was found in sample W17-L1 (Table 4.3). The only volatile organic compound detected was acetone at a concentration of 39 µg/L, but acetone was found at 17 µg/L in the laboratory blank run with the sample. Therefore, the hit was only slightly higher than the blank and is probably not significant. No semivolatile TCL organic compounds were detected with reporting limits of 50–250 µg/L, nor were pesticides or PCBs with reporting limits of 0.25–5.0 µg/L. Three semivolatile organic TICs were observed (total, ~1938J µg/L). The total organic carbon was 233 mg/L.

Table 4.2. Waste characterization data for tank W-17

Characteristic	W17-L1 (a), (b)	W17-L2 (a), (c)
<i>Physical properties and miscellaneous data</i>		
Total dissolved solids (mg/mL)	3.9	*
Total solids (mg/mL)	3.9	*
Density (g/mL)	1.002	*
Inorganic carbon (mg/L)	22	*
Total organic carbon (mg/L)	233	*
Total carbon (mg/L)	255	*
<i>RCRA metals (mg/L)</i>		
Ag	(<0.006)	(<0.006)
As	0.006	0.003
Ba	<0.001	<0.001
Cd	<0.006	<0.006
Cr	<0.006	0.008
Hg	<0.005	<0.005
Ni	<0.015	<0.015
Pb	0.40	0.38
Se	<0.002	<0.002
Tl	<0.002	0.003
<i>Process metals (mg/L)</i>		
Ca	3.1	4.0
Mg	0.1	0.2
Na	1430	1460
Th	<0.17 (d)	<0.17 (d)
U	3.3	3.1
Zn	10.5	10.7
<i>Anions (mg/L)</i>		
Bromide	12-30 (e)	*
Chloride	30	*
Cyanide	<0.01	*
Fluoride	<0.1	*
Nitrate	<0.1	*
Nitrite (f)	N	*
Phosphate	<0.1	*
Sulfate	<0.1	*
Sulfide (f)	N	*

Table 4.2. Waste characterization data for tank W-17 (continued)

Characteristic	W17-L1 (a), (b)	W17-L2 (a), (c)
<i>Alkalinity</i>		
pH	12.4	12.4
OH ⁻ (M)	0.06	*
CO ₃ ²⁻ (M)	<0.02	*
HCO ₃ ⁻ (M)	(g)	*
<i>Radiochemical screening (Bq/mL)</i>		
Gross alpha	<1.0E+0	<1.0E+0
Gross beta	2.4E+1	2.3E+1
<i>Betalgamma emitters (Bq/mL)</i>		
⁶⁰ Co	<1.6E-1	<1.4E-1
¹³⁷ Cs	1.60E+1	1.59E+1
¹⁵² Eu	<5.4E-1	<5.4E-1
¹⁵⁴ Eu	<2.2E-1	<4.3E-1
¹⁵⁵ Eu	<3.2E-1	<3.2E-1
³ H	1.79E+1	*
⁹⁰ Sr	4.1E+0	*
⁶⁵ Zn	(h)	(h)

(a) Tank sampled October 13, 1992.

(b) Sample collected from the midpoint of the liquid layer, ~4.5 in. above the tank bottom.

(c) Sample collected from the bottom of the tank.

(d) Visual scan.

(e) Some peak broadening due to older column; lower value excludes tail, higher value includes tail.

(f) Qualitative analysis. N= not detected.

(g) Bicarbonate not stable at this pH.

(h) Possibly some ⁶⁵Zn (~2 Bq/mL) was observed, but this observation is suspect given the half-life and the known tank history.

Table 4.3. Summary of organic characterization results for liquid samples from tanks W-17, W-18, WC-5, WC-6, and WC-8

Analysis class and compound hits, $\mu\text{g/L}$ (a)	W17-L1	W18-L1	WC5-L1	WC6-L1	WC8-L1
<i>Volatile organics</i>					
Acetone	39B (b)	37B (b)			
<i>Water-soluble organics</i>					
<i>Semivolatile organics</i>					
Butylbenzylphthalate			2J	2J	
<i>Pesticides/PCBs</i>					
Endosulfan II		0.046J			
α -Chlordane		0.12J			
Aroclor-1254		4J			
Aroclor-1260		1.9J			
<i>Semivolatile organic TICs (c)</i>					
Unknown	1938J (3)	918J (3)	711J (3)	184J (3)	713J (3)
Unknown hydrocarbons				13J (1)	11J (1)

(a) Only compounds whose possible presence was indicated by the analysis are listed. See Tables E.3–E.6 for the target compound lists and Table E.1 for the conversion factors to calculate the reporting limits.

(b) Acetone was found at 17 $\mu\text{g/L}$ in the laboratory blank run with the sample.

(c) Result is the sum of the tentatively identified compounds (TICs). The number of TICs are shown in parenthesis.

4.3.2 Tank W-18

Tank W-18 contained a clear, aqueous liquid with a thin layer of a fine, chalk-white sediment on the bottom. There was no indication of any organic layers that are immiscible with the aqueous phase. Dose rates (field survey) for ~200-mL samples were <0.1 mrem/h. Because the waste layer was only ~1-in. deep when the tank was sampled, samples were collected only from the bottom of the tank.

The results of the inorganic and radiochemical analyses are summarized in Table 4.4. The waste was basic (with sodium the principal metal analyte) and contained some bromide (1,200 mg/L). The concentrations of other inorganic analytes were low. The gross alpha and gross beta activities were <1 and 14 Bq/mL, respectively, for the filtered sample.

The samples contained a trace of sediment (<0.5 wt %). The principal metal analyte in the sediment appeared to be zinc, probably as ZnO. With bromide in the liquid phase, this suggests that zinc bromide waste from a hot-cell shielding window may have been discharged into the tank. The sediment was readily resuspended by shaking the jar and it then settled relatively rapidly.

Sample W18-L1 contained very little organic material (Table 4.3). The only volatile organic compound found was acetone at a concentration of 37 µg/L, but acetone was also detected in the blank at 17 µg/L, so this is probably not significant. No semivolatile TCL organic compounds were detected at reporting limits of 50–250 µg/L. Traces of pesticides and PCBs below the reporting limits were detected. Three semivolatile organic TICs were observed (total, ~918J µg/L). The total organic carbon was low (11 mg/L).

Table 4.4. Waste characterization data for tank W-18

Characteristic	W18-L1 Filtered (a), (b)	W18-L1 Including sediment (a)
<i>Physical properties and miscellaneous data</i>		
Total dissolved solids (mg/mL)	2.1	*
Total solids (mg/mL)	*	*
Density (g/mL)	1.001	*
Inorganic carbon (mg/L)	57	*
Total organic carbon (mg/L)	11	*
Total carbon (mg/L)	68	*
<i>RCRA metals (mg/L)</i>		
Ag	(0.008)	(0.008)
As	<0.002	0.004
Ba	<0.001	<0.001
Cd	<0.006	<0.006
Cr	<0.006	0.081
Hg	<0.005	<0.005
Ni	<0.015	<0.015
Pb	<0.27	1.3
Se	<0.002	<0.002
Tl	0.002	<0.002
<i>Process metals (mg/L)</i>		
Ca	3.1	14.0
Mg	2.1	3.3
Na	550	480
Th	<0.17 (c)	~0.5 (c)
U	0.39	2.4
Zn	1.4	420
<i>Anions (mg/L)</i>		
Bromide	1200	*
Chloride	20	*
Cyanide	<0.01	*
Fluoride	<0.1	*
Nitrate	<0.1	*
Nitrite (d)	N	*
Phosphate	<0.1	*
Sulfate	<0.1	*
Sulfide (d)	N	*

Table 4.4. Waste characterization data for tank W-18 (continued)

Characteristic	W18-L1 Filtered (a), (b)	W18-L1 Including sediment (a)
<i>Alkalinity</i>		
pH	10.2	*
OH ⁻ (M)	<0.01	*
CO ₃ ²⁻ (M)	<0.01	*
HCO ₃ ⁻ (M)	<0.01	*
<i>Radiochemical screening (Bq/mL)</i>		
Gross alpha	<1.0E+0	<1.0E+0
Gross beta	1.4E+1	*
<i>Beta/gamma emitters (Bq/mL)</i>		
⁶⁰ Co	<1.0E-2	6.4E-1
¹³⁷ Cs	1.26E+1	1.55E+1
¹⁵² Eu	<5.0E-1	<4.5E-1
¹⁵⁴ Eu	<2.2E-1	<3.8E-1
¹⁵⁵ Eu	<2.9E-1	<2.6E-1
³ H	9.29E+1	*
⁹⁰ Sr	1.7E+0	*
⁶⁵ Zn	<2.9E-1	(e)

(a) Tank sampled October 13, 1992. Sample was collected from the bottom of the tank.

(b) Analyses for density, carbons, anions, and alkalinity were performed on supernate.

(c) Visual scan.

(d) Qualitative analysis. N = not detected.

(e) Possibly some ⁶⁵Zn (~0.4 Bq/mL) was observed, but this observation is suspect given the half-life and the known tank history.

4.3.3 Tank WC-5

Tank WC-5 contained a clear, aqueous liquid with a trace of a fine, reddish-brown sediment on the bottom. There was no indication of any organic layers that are immiscible with the aqueous phase. Dose rates (field survey) for ~200-mL samples were <0.1 mrem/h.

The results of the inorganic and radiochemical analyses for the aqueous phase, samples WC5-L1 and WC5-L2 filtered, are summarized in Table 4.5. The samples were basic and contained dilute NaOH. The concentrations of other inorganic analytes were low. However, the samples contained mercury at a concentration of 0.22 mg/L, which is slightly over the threshold that classifies a waste as RCRA (i.e., mercury ≥ 0.2 mg/L). The gross alpha and gross beta activities were ~1 Bq/mL each. Results for the sample, WC5-L2 filtered, which was collected from the tank bottom, were similar to those for sample WC5-L1 from the midpoint of the liquid layer.

Two bottles of the sample material collected from the tank bottom contained a trace of sediment (<0.5 wt %). The other three bottles from the tank bottom were clear with no sediment. The tank bottom sample, WC5-L2 including sediment, contained 141 Bq/mL of alpha activity and 0.44 mg/L of mercury. The principal metal analytes in the sediment solids appeared to be Ca, Fe, and Mg. The sediment was readily resuspended, and it then settled relatively rapidly.

Very little organic material was found in sample WC5-L1 (Table 4.3). No volatile organic compounds were detected (reporting limits of 50–100 $\mu\text{g/L}$). In the SVOA (reporting limits of 16–80 $\mu\text{g/L}$), the only TCL constituent detected was a trace of a phthalate at a concentration below the reporting limit. No pesticides or PCBs were detected (reporting limits of 0.08–1.7 $\mu\text{g/L}$). Three semivolatile organic TICs were observed (total, ~711J $\mu\text{g/L}$). The total organic carbon was low (17 mg/L).

Table 4.5. Waste characterization data for tank WC-5

Characteristic	WC5-L1 (a), (b)	WC5-L2 Filtered (a), (c)	WC5-L2 Including sediment (a), (c)
<i>Physical properties and miscellaneous data</i>			
Total dissolved solids (mg/mL)	8.1	*	*
Total solids (mg/mL)	7.9	*	*
Density (g/mL)	1.012	1.011 (d)	*
Inorganic carbon (mg/L)	106	*	*
Total organic carbon (mg/L)	17	*	*
Total carbon (mg/L)	123	*	*
<i>RCRA metals (mg/L)</i>			
Ag	(<0.006)	(<0.006)	(0.009)
As	0.006	*	*
Ba	0.017	0.016	0.080
Cd	<0.006	<0.006	<0.006
Cr	0.29	0.31	0.83
Hg	0.22	0.22	0.44
Ni	<0.015	<0.015	0.12
Pb	0.25	0.16	0.49
Se	0.002	*	*
Tl	<0.002	*	*
<i>Process metals (mg/L)</i>			
Al	0.12	<0.02	1.0
Ca	8.0	5.2	18.8
Fe	0.02	0.09	9.1
K	5.8	6.0	6.2
Mg	0.05	<0.04	8.2
Na	3300	3300	3400
Th	<0.17	<0.17	0.55
U	<0.35	0.46	0.56
<i>Anions (mg/L)</i>			
Bromide	<0.05	*	*
Chloride	70	*	*
Cyanide	<0.01	*	*
Fluoride	<10. (e)	*	*
Nitrate	9.2	*	*
Nitrite (f)	N	*	*
Phosphate	<0.1	*	*
Sulfate	18	*	*
Sulfide (f)	N	*	*

Table 4.5. Waste characterization data for tank WC-5 (continued)

Characteristic	WC5-L1 (a), (b)	WC5-L2 Filtered (a), (c)	WC5-L2 Including sediment (a), (c)
<i>Alkalinity</i>			
pH	13.1	13.0 (d)	*
OH ⁻ (M)	0.15	0.16 (d)	*
CO ₃ ²⁻ (M)	<0.02	*	*
HCO ₃ ⁻ (M)	(g)	*	*
<i>Radiochemical screening (Bq/mL)</i>			
Gross alpha	1.1E+0	<1.0E+0	1.41E+2
Gross beta	1.0E+0	<1.0E+0	*
<i>Beta/gamma emitters (Bq/mL)</i>			
²⁴¹ Am	<3.7E-1	<3.5E-1	6.77E+1
⁶⁰ Co	<1.0E-1	<8.3E-2	<1.2E-1
¹³⁷ Cs	3.5E-1	2.4E-1	4.5E-1
¹⁵² Eu	<2.8E-1	<2.3E-1	<2.0E-1
¹⁵⁴ Eu	<1.9E-1	<2.5E-1	<2.6E-1
¹⁵⁵ Eu	<2.0E-1	<1.7E-1	<1.6E-1
³ H	<4.0E-2	*	*
⁹⁰ Sr	<1.0E+0	<1.0E+0	*

(a) Tank sampled October 29, 1992.

(b) Sample collected from the midpoint of the liquid layer, ~10 in. above the tank bottom.

(c) Sample collected from the bottom of the tank.

(d) Analysis performed on supernate.

(e) Interference with the fluoride analysis. This may result from small aliphatic acids which elute from the AS4A column at about the same time as fluoride.

(f) Qualitative analysis. N = not detected.

(g) Bicarbonate not stable at this pH.

4.3.4 Tank WC-6

Tank WC-6 contained a clear, aqueous liquid with a trace of a fine, reddish-brown sediment on the bottom. There was no indication of any organic layers that are immiscible with the aqueous phase. Dose rates (field survey) for ~200-mL samples were <0.1 mrem/h.

The results of the inorganic and radiochemical analyses of the aqueous phase, samples WC6-L1 and WC6-L2 filtered, are summarized in Table 4.6. The samples were basic and contained dilute NaOH. Except for sodium, very few inorganic analytes were detected in the aqueous phase, and their concentrations were low. The gross alpha and gross beta activities were <1 and 1 Bq/mL, respectively. Results for the sample, WC6-L2 filtered, from the tank bottom were similar to those for sample WC6-L1 from the midpoint of the liquid layer, except that the sodium concentration was higher in the sample from the tank bottom (4100 vs 2500 mg/L). (Because this tank was not pumped prior to sampling, there was no agitation to mix the tank contents.)

Three bottles of the sample material collected from the tank bottom contained a trace of sediment (<0.5 wt %). The other two bottles from the tank bottom were clear with no sediment. The sample, WC6-L2 including sediment, contained some alpha emitters (1670 Bq/mL) and low levels of uranium and thorium (45 and 30 mg/L, respectively). The sediment was readily resuspended, and it then settled relatively rapidly.

Sample WC6-L1 contained very little organic matter (Table 4.3). No volatile organic compounds were detected with a reporting limit of 25–50 µg/L. In the SVOA (reporting limits of 16–80 µg/L), the only TLC constituent detected was a trace of a phthalate at a concentration below the reporting limit. No pesticides or PCBs were detected (reporting limits of 0.08–1.6 µg/L). Four semivolatile organic TICs were observed (total, ~197J µg/L). The total organic carbon was low (20 mg/L).

Table 4.6. Waste characterization data for tank WC-6

Characteristic	WC6-L1 (a), (b)	WC6-L2 Filtered (a), (c)	WC6-L2 Including sediment (a), (c)
<i>Physical properties and miscellaneous data</i>			
Total dissolved solids (mg/mL)	6.2	*	*
Total solids (mg/mL)	5.9	*	*
Density (g/mL)	1.010	1.011 (d)	*
Inorganic carbon (mg/L)	105	*	*
Total organic carbon (mg/L)	20	*	*
Total carbon (mg/L)	125	*	*
<i>RCRA metals (mg/L)</i>			
Ag	<0.006	<0.006	(0.020)
As	<0.002	*	*
Ba	0.012	0.002	0.040
Cd	<0.006	<0.006	0.036
Cr	0.018	0.035	0.082
Hg	<0.001	0.002	0.005
Ni	<0.015	<0.015	0.048
Pb	0.007	0.008	0.032
Se	<0.002	*	*
Tl	<0.002	*	*
<i>Process metals (mg/L)</i>			
Al	0.09	0.25	3.4
Ca	20	10	31
Fe	0.05	0.07	3.9
K	9.4	8.2	9.2
Mg	0.37	<0.04	6.3
Na	2500	4100	4200
Th	<0.17	<0.17	30
U	<0.35	0.55	45
<i>Anions (mg/L)</i>			
Bromide	<0.05	*	*
Chloride	50	*	*
Cyanide	<0.01	*	*
Fluoride	<10. (e)	*	*
Nitrate	16	*	*
Nitrite (f)	N	*	*
Phosphate	<0.1	*	*
Sulfate	13	*	*
Sulfide (f)	N	*	*

Table 4.6. Waste characterization data for tank WC-6 (continued)

Characteristic	WC6-L1 (a), (b)	WC6-L2 Filtered (a), (c)	WC6-L2 Including sediment (a), (c)
<i>Alkalinity</i>			
pH	13.1	13.1 (d)	*
OH ⁻ (M)	0.11	0.19 (d)	*
CO ₃ ²⁻ (M)	<0.02	*	*
HCO ₃ ⁻ (M)	(g)	*	*
<i>Radiochemical screening (Bq/mL)</i>			
Gross alpha	<1.0E+0	<1.0E+0	1.67E+3
Gross beta	1.1E+0	1.0E+0	*
<i>Beta/gamma emitters (Bq/mL)</i>			
²⁴¹ Am	<3.9E-1	<3.4E-1	6.62E+2
⁶⁰ Co	<1.0E-1	<1.4E-1	<1.2E-1
¹³⁷ Cs	3.6E-1	1.2E-1	6.9E-1
¹⁵² Eu	<2.3E-1	<2.0E-1	<3.0E-1
¹⁵⁴ Eu	<2.9E-1	<4.0E-1	<1.4E-1
¹⁵⁵ Eu	<1.9E-1	<1.8E-1	<3.1E-1
³ H	<4.0E-2	*	*
⁹⁰ Sr	<1.0E+0	<1.0E+0	*

(a) Tank sampled October 29, 1992.

(b) Sample collected from the midpoint of the liquid layer, ~4.5 in. above the tank bottom.

(c) Sample collected from the bottom of the tank.

(d) Analysis performed on supernate.

(e) Interference with the fluoride analysis. This may result from small aliphatic acids which elute from the AS4A column at about the same time as fluoride.

(f) Qualitative analysis. N = not detected.

(g) Bicarbonate not stable at this pH.

4.3.5 Tank WC-8

Tank WC-8 contained only a clear, aqueous liquid. No sediment was observed, and there was no indication of any organic layers that are immiscible with the aqueous phase. Dose rates (field survey) for ~200-mL samples were <0.1 mrem/h.

The samples were basic and contained dilute NaOH. The concentrations of other inorganic analytes were low (Table 4.7). The gross alpha and gross beta activities were <1 and 6 Bq/mL, respectively.

Very little organic matter was found in sample WC8-L1 (Table 4.3). No volatile organic compounds were detected with reporting limits of 50–100 µg/L. No TCL semivolatile organic compounds were detected (reporting limits of 17–85 µg/L), nor were pesticides or PCBs (reporting limits of 0.09–1.8 µg/L). Four semivolatile organic TICs were observed (total, ~724J µg/L). The total organic carbon was low (15 mg/L).

Table 4.7. Waste characterization data for tank WC-8

Characteristic	WC8-L1 (a), (b)	WC8-L2 (a), (c)
<i>Physical properties and miscellaneous data</i>		
Total dissolved solids (mg/mL)	12	*
Total solids (mg/mL)	12	*
Density (g/mL)	1.013	1.013
Inorganic carbon (mg/L)	64	*
Total organic carbon (mg/L)	15	*
Total carbon (mg/L)	79	*
<i>RCRA metals (mg/L)</i>		
Ag	<0.006	<0.006
As	<0.002	*
Ba	0.005	0.006
Cd	<0.006	<0.006
Cr	0.022	0.021
Hg	0.002	0.002
Ni	<0.015	<0.015
Pb	0.23	0.22
Se	<0.002	*
Tl	0.002	*
<i>Process metals (mg/L)</i>		
Ca	11.6	11.5
K	7.6	7.6
Na	5500	5500
Th	<0.17	<0.17
U	1.4	3.2
<i>Anions (mg/L)</i>		
Bromide	1.1	*
Chloride	110	*
Cyanide	<0.01	*
Fluoride	<10. (d)	*
Nitrate	27	*
Nitrite (e)	N	*
Phosphate	<0.1	*
Sulfate	15	*
Sulfide (e)	N	*

Table 4.7. Waste characterization data for tank WC-8 (continued)

Characteristic	WC8-L1 (a), (b)	WC8-L2 (a), (c)
<i>Alkalinity</i>		
pH	13.4	13.2
OH ⁻ (M)	0.26	0.26
CO ₃ ²⁻ (M)	<0.02	<0.02
HCO ₃ ⁻ (M)	(f)	(f)
<i>Radiochemical screening (Bq/mL)</i>		
Gross alpha	<1.0E+0	<1.0E+0
Gross alpha	6.3E+0	6.8E+0
<i>Beta/gamma emitters (Bq/mL)</i>		
⁶⁰ Co	<1.5E-1	<7.3E-2
¹³⁷ Cs	2.5E+0	2.8E+0
¹⁵² Eu	<3.4E-1	<1.5E-1
¹⁵⁴ Eu	<3.1E-1	<2.7E-1
¹⁵⁵ Eu	<2.0E-1	<1.5E-1
³ H	2.6E-1	*
⁹⁰ Sr	1.9E+0	*

(a) Tank sampled October 29, 1992.

(b) Sample collected from the midpoint of the liquid layer, ~3.5 in. above the tank bottom.

(c) Sample collected from the bottom of the tank.

(d) Interference with the fluoride analysis. This may result from small aliphatic acids which elute from the AS4A column at about the same time as fluoride.

(e) Qualitative analysis. N = not detected.

(f) Bicarbonate not stable at this pH.

4.3.6 Tank WC-11

Tank WC-11 contained (1) an aqueous liquid layer, (2) a small amount of brown sediment on the bottom, and (3) a dark-brown, oily layer floating over the aqueous layer.

4.3.6.1 Aqueous Liquid Samples

The results of the inorganic and radiochemical analyses for the aqueous phase samples, WC11-L1 and WC11-L2 filtered, are summarized in Table 4.8. The samples were radioactive—with ^{137}Cs the principal contributor. The samples also contained some ^{60}Co . Dose rates (field survey) for ~200 mL of sample were ~20–25 mrem/h for sample WC11-L1. The samples were basic with sodium the principal metal analyte. The concentrations of other inorganic analytes were relatively low.

Tank WC-11 contained a layer of sediment on the tank bottom. The Markland Sludge Gun™ detector sounded at ~1 in. above the tank bottom. This instrument will detect slightly cloudy layers. The samples collected from the tank bottom contained only a small amount of sediment (<0.5 wt %). The tank-bottom sample, WC11-L2 including sediment, contained alpha emitters, europium isotopes, and ^{60}Co in addition to ^{137}Cs . Dose rates for ~200 mL of tank bottom sample were ~40–50 mrem/h. The principal metal analytes in the sediment solids appeared to be Ca, Mg, Fe, and Mn. The sediment solids also contained some U, Th, and RCRA metals.

Very little organic material was found in the aqueous sample WC11-L1 (Table 4.9). No water-soluble volatile organic compounds were detected by DAI-GC (reporting limit of 1000 µg/L). The only TCL constituents detected in the SVOA (reporting limits of 25–125 µg/L) were traces of two phthalates below the reporting limit. No pesticides or PCBs were detected at reporting limits of 0.50–10 µg/L. Several semivolatile organic TICs were observed (total ~1400J µg/L). The volatile organics analysis by GC-MS was not performed because the sample was too radioactive to process in a conventional, nonzoned laboratory. The total organic carbon was low (84 mg/L).

4.3.6.2 Organic Layer in Tank WC-11

Tank WC-11 contained a dark-brown, oily layer, about 0.5 in. deep, floating on the aqueous layer. The results of the organic characterization are summarized in Table 4.9. The volatile organics analysis of a methyl alcohol dilution of the oil did not reveal any volatile compounds beyond traces of methylene chloride and hexane, which also were present in the solvent blank. The reporting limits for the volatile organics analysis were 50–100 mg/L. No TCL constituents were detected in the SVOA (reporting limits of 55–275 mg/L), but the pesticide/PCB analysis (reporting limits of 53–1060 µg/L) indicated Aroclor-1254 at 1700 µg/L. Some pesticide peaks also were observed, but these are believed to be the result of co-eluting components from the Aroclor mixture, and not pesticides. A GC profile of the organic layer suggested that it is vacuum-pump oil or an oil with a distillation range similar to vacuum-pump oil.

Table 4.8. Waste characterization data for tank WC-11

Characteristic	WC11-L1 (a), (b)	WC11-L2 Filtered (a), (c)	WC11-L2 Including sediment (a), (c)
<i>Physical properties and miscellaneous data</i>			
Total dissolved solids (mg/mL)	1.7	*	*
Total solids (mg/mL)	*	*	*
Density (g/mL)	1.006	1.005 (d)	*
Inorganic carbon (mg/L)	98	*	*
Total organic carbon (mg/L)	84	*	*
Total carbon (mg/L)	182	*	*
<i>RCRA metals (mg/L)</i>			
Ag	(<0.006)	(<0.006)	(0.036)
As	<0.002	*	*
Ba	0.024	0.061	20
Cd	<0.006	0.016	0.22
Cr	0.055	0.13	2.6
Hg	0.005	0.018	2.0
Ni	0.13	0.31	6.5
Pb	0.23	0.36	4.6
Se	0.002	*	*
Tl	0.003	*	*
<i>Process metals (mg/L)</i>			
Al	0.31	0.23	10.9
Ca	4.7	5.0	980
Cu	0.06	0.07	1.3
Fe	0.06	0.22	44
K	16	20	22
Mg	0.20	1.8	270
Mn	0.08	0.18	26
Na	500	850	850
Th	<0.17	<0.17	3.4
U	<0.35	0.91	3.8
Zn	0.02	0.06	6.1
<i>Anions (mg/L)</i>			
Bromide	<0.05	*	*
Chloride	15	*	*
Cyanide	0.012	*	*
Fluoride	<0.05	*	*
Nitrate	52	*	*
Nitrite (e)	Y	*	*
Phosphate	<0.08	*	*
Sulfate	20	*	*
Sulfide (e)	N	*	*

Table 4.8. Waste characterization data for tank WC-11 (continued)

Characteristic	WC11-L1(a), (b)	WC11-L2 Filtered (a), (c)	WC11-L2 Including sediment (a), (c)
<i>Alkalinity</i>			
pH	11.4	11.2 (d)	*
OH ⁻ (M)	<0.01	0.02 (d)	*
CO ₃ ²⁻ (M)	<0.01	<0.02 (d)	*
HCO ₃ ⁻ (M)	<0.01	<0.01	*
<i>Radiochemical screening (Bq/mL)</i>			
Gross alpha	<1.0E+0	1.4E+0	3.64E+2
Gross beta	5.0E+4	*	*
<i>Beta/gamma emitters (Bq/mL)</i>			
⁶⁰ Co	1.10E+2	1.50E+2	5.18E+2
¹³⁷ Cs	4.77E+4	5.80E+4	6.33E+4
¹⁵² Eu	<1.6E+2	<2.6E+2	1.66E+3
¹⁵⁴ Eu	<1.4E+1	<1.9E+1	8.25E+2
¹⁵⁵ Eu	<8.4E+1	<1.3E+2	2.49E+2
³ H	3.1E+1	*	*
⁹⁰ Sr	9.3E+0	*	*

(a) Tank sampled November 4, 1992.

(b) Sample collected from the midpoint of the liquid layer, ~5 in. above the tank bottom.

(c) Sample collected from the bottom of the tank.

(d) Analysis performed on supernate.

(e) Qualitative analysis. N = not detected. Y = detected.

Table 4.9. Summary of organic characterization results for tank WC-11

Analysis class and compound hits, $\mu\text{g/L}$ (a)	WC11-L1	WC11-Surface Oil layer
<i>Volatile organics</i>	(b)	(c)
<i>Water-soluble organics</i>		(d)
<i>Semivolatile organics</i>		
Diethylphthalate	7J	
Di-n-octylphthalate	17J	
<i>Pesticides/PCBs</i>		
Aroclor-1254		1700 (e)
<i>Semivolatile organic TICs (f)</i>		
Unknown	1276J (8)	
Unknown hydrocarbons	124J (7)	

(a) Only compounds whose possible presence was indicated by the analysis are listed. See Tables E.3–E.6 for the target compound lists and Table E.1 for the conversion factors to calculate the reporting limits.

(b) Analysis was not run because the sample was too radioactive for nonzoned laboratory.

(c) Traces of methylene chloride and hexane were detected. Their presence in the blank suggests cross-contamination among the diluted oil samples during transport.

(d) Analysis is not appropriate for organic samples.

(e) Some pesticide peaks also were detected, but these are believed to be the result of co-eluting components from the Aroclor mixture, and not pesticides.

(f) Result is the sum of the tentatively identified compounds (TICs). The number of TICs are shown in parenthesis.

4.3.7 Tank WC-12

Tank WC-12 contained (1) an aqueous layer, (2) a shallow layer (~0.5 in. deep) of a thin sludge with highly radioactive solids, and (3) a trace of a yellow, oily layer floating on the aqueous layer. The oily phase was not characterized because the amount of this phase in the WC12-Surface samples was too small to draw-off a 1–2 mL aliquot for organic characterization. The oily phase is probably not significant because the amount of material was so small.

4.3.7.1 Aqueous Liquid Sample WC12-L1

Sample WC12-L1 from the midpoint of the liquid layer was basic and contained dilute NaOH (Table 4.10). The samples were radioactive with ^{137}Cs the principal contributor. Dose rates (field survey) for ~200 mL of sample were ~20–25 mrem/h. The concentrations of inorganic analytes (except sodium) were low.

Very little organic matter was found in sample WC12-L1, which was collected from the midpoint of the liquid layer, ~4 in. above the tank bottom (Table 4.11). No water-soluble volatile organic compounds were detected by DAI-GC (reporting limits of 1000 $\mu\text{g/L}$), and no TCL constituents were found in the SVOA (reporting limits of 25–125 $\mu\text{g/L}$) or the pesticide/PCB analysis (reporting limits of 0.50–10 $\mu\text{g/L}$). The volatile organics analysis by GC-MS was not performed because the sample was too radioactive to process in a conventional, nonzoned laboratory. Several semivolatile organic TICs were observed (total, ~1017J $\mu\text{g/L}$). The total organic carbon in the sample from the midpoint of the liquid layer was very low (<10 mg/L).

4.3.7.2 Sludge Samples WC12-L3-A, -B, and -C

Tank WC-12 contained a shallow layer (~0.5 in. deep) of a thin sludge containing highly radioactive solids. Because this layer was too shallow to sample with the soft-sludge sampler, it was sampled as liquid samples using the vacuum-pump sampling system to suction material from the bottom of the tank. This method provides qualitative information about the bottom layer and solids. The samples were taken ~6 in. from the tank centerline (i.e., near the low part of the dished bottom). Initial surveys indicated radiation fields of up to ~900 mrem/h with the probe under the sample bottle (i.e., under the sludge layer) and up to ~480 mrem/h with the probe parallel to the sample bottle on the bench top. The highly penetrating nature of the radiation indicated the presence of ^{60}Co . The samples were examined visually about 6 h after collection. Samples WC12-L3-A and WC12-L3-C contained a layer on the bottom with black solids (~0.2-in. and ~0.1-in. deep, respectively) with an overlying layer containing suspended finely divided black solids, which made the liquid nearly opaque, similar in appearance to India ink. The bottom layer in the WC12-L3-B sample was ~0.05 in. deep.

Because of the limited quantity of sample material available and the highly radioactive nature of the solids, the analyses of the sample was limited per the ALARA principle. An aliquot of sample WC12-L3-A (including the solids) was treated by microwave assisted digestion in HNO_3 , filtered, and the resulting solution was analyzed for metals and radioactive materials. Sample WC12-L3-B was filtered, and the filtrate was analyzed for metals and radioactive materials. Sample WC12-L3-B was used for the filtered comparison sample (rather than an aliquot of WC12-L3-A) to reduce the radiation exposure to the analyst. Sample WC12-L3-B was collected from the same location in the tank, but it contained fewer solids; consequently the radiation dose rate was

lower than that of WC12-L3-A (~70 vs ~480 mrem/h measured with the survey meter parallel with the sample on the bench top).

The constituents in the sludge that were soluble after microwave digestion included relatively high levels of europium isotopes, ^{60}Co , and ^{137}Cs and some curium and plutonium (Table 4.10, sample WC12-L3-A including solids). The principal metal analytes present in addition to Na were Ca, Fe, Mg, and Mn. Low concentrations of Pb, Hg, Th and U were also detected. The difference between the results for sample WC12-L3-A including solids and the filtered sample WC12-L3-B provides a qualitative indication of the constituents in the solids. The residue from the microwave digestion of WC12-L3-A was not analyzed because of the highly radioactive nature of the solids.

The concentrations of sodium and ^{137}Cs in the liquid associated with the sludge, sample WC12-L3-B filtered, were about 60% of the levels in sample WC12-L1. This finding is probably the result of dilution by nonprogrammatic inflow during the interim period between the collection of WC12-L1 on November 4, 1992, and WC12-L3-B on March 17, 1993. Europium isotopes were detected in sample WC12-L3-B filtered. In basic waste tanks, europium, if it is present, is found usually only in the precipitated solids because of the low solubility of europium hydroxide. The presence of europium isotopes in the WC12-L3-B filtrate suggests that some of the europium may have been in the form of very fine particles which passed through the filter.

Sample WC12-L3-C was filtered, and the filtrate was analyzed for organic compounds (Table 4.11). No pesticides or PCBs were detected (at reporting limits of 3.4–69 $\mu\text{g/L}$). Several phthalates were observed in the SVOA [diethylphthalate, di-n-butylphthalate, bis(2-ethylhexyl) phthalate, and di-n-octylphthalate at 89B, 390, 25J, and 12J $\mu\text{g/L}$, respectively]. The semivolatile organic TICs included tributylphosphate (630J $\mu\text{g/L}$), another isomer of dibutylphthalate (66J $\mu\text{g/L}$), a propanol derivative (64J $\mu\text{g/L}$), and 18 unknown compounds (total of 7540J $\mu\text{g/L}$). Of interest was the identification of 2,6-dibromo-4-nitrophenol (54J $\mu\text{g/L}$), which is a nitration product of the 2,4,6-tribromophenol surrogate standard added to the sample as part of the quality control check. This illustrates the difficulty of preparing some tank waste samples for analysis. The filtrate was not analyzed for volatile organics by GC-MS because the sample was too radioactive to process in a nonzoned analytical laboratory, and the water-soluble organic compound analysis could not be run because of instrument breakdown.

The precipitated solids in sample WC12-L3-C were estimated as <0.5 wt % of the sample based on the quantity of wet residue observed on the filter paper.

Table 4.10. Waste characterization data for tank WC-12

Characteristic	WC12-L1 (a)	WC12-L3-B Filtered (b)	WC12-L3-A Including solids (b), (c)
<i>Physical properties and miscellaneous data</i>			
Total dissolved solids (mg/mL)	6.4	4.9	*
Total solids (mg/mL)	6.4	*	*
Density (g/mL)	1.008	1.006	*
Inorganic carbon (mg/L)	1110	*	*
Total organic carbon (mg/L)	<10	*	*
Total carbon (mg/L)	1050	*	*
<i>RCRA metals (mg/L)</i>			
Ag	(<0.006)	(<0.009)	(0.07)
As	<0.002	<0.006	<0.006
Ba	0.019	0.094	8.92
Cd	0.067	0.058	0.55
Cr	0.25	0.29	5.62
Hg	0.002	0.064	4.32
Ni	0.36	0.74	12.8
Pb	1.92	0.75	26.1
Se	0.003	<0.006	<0.006
Tl	<0.002	<0.4	<0.4
<i>Process metals (mg/L)</i>			
Al	5.8	3.2	69
Ca	2.8	8.0	430
Fe	0.1	2.3	220
K	42	58	62
Mg	0.6	2.2	110
Mn	0.9	2.6	210
Na	2700	2200	2200
Th	<0.17	0.41	21.6
U	2.6	6.1	8.7
<i>Anions (mg/L)</i>			
Bromide	<0.05	*	*
Chloride	35	*	*
Cyanide	0.016	*	*
Fluoride	<0.05	*	*
Nitrate	45	*	*
Nitrite (d)	Y	*	*
Phosphate	<0.08	*	*
Sulfate	9.3	*	*
Sulfide (d)	N	*	*

Table 4.10. Waste characterization data for tank WC-12 (continued)

Characteristic	WC12-L1 (a)	WC12-L3-B Filtered (b)	WC12-L3-A Including solids (b), (c)
<i>Alkalinity</i>			
pH	10.1	9.8	*
OH ⁻ (M)	0.04	*	*
CO ₃ ²⁻ (M)	<0.02	*	*
HCO ₃ ⁻ (M)	<0.02	*	*
<i>Radiochemical screening (Bq/mL)</i>			
Gross alpha	<1.0E+0 (e)	<2.6E+1 (f)	1.8E+3 (e)
Gross beta	5.6E+4	3.7E+4	7.3E+5
<i>Beta/gamma emitters (Bq/mL)</i>			
⁶⁰ Co	2.63E+2	9.25E+2	4.56E+4
¹³⁷ Cs	5.03E+4	2.76E+4	3.59E+4
¹⁵² Eu	<1.7E+2	4.50E+3	3.80E+5
¹⁵⁴ Eu	<1.7E+1	3.42E+3	3.02E+5
¹⁵⁵ Eu	<8.7E+1	8.38E+2	7.04E+4
³ H	2.8E+1	*	*
⁹⁰ Sr	1.1E+1	*	1.43E+3
<i>Alpha emitters (Bq/mL)</i>			
²⁴⁴ Cm	(g)	(g)	1.6E+3
²³⁸ Pu			1.5E+2
²³⁹ Pu/ ²⁴⁰ Pu			2.4E+1
²³³ U/ ²³⁹ Th/ ²³⁷ Np			2.3E+0

(a) Sample collected November 4, 1992, from the midpoint of the liquid layer, ~4 in. above the tank bottom. The sample was filtered before the radiochemical analysis.

(b) Sample collected March 17, 1993, from the bottom of the tank.

(c) Data is for the liquid plus the solids which dissolved during microwave assisted digestion in ~2.6 M HNO₃ acid. The residue was not analyzed.

(d) Qualitative analysis. N = not detected; Y = detected.

(e) Alpha emitters were analyzed after TRU•Spec™ separation.

(f) Alpha emitters were analyzed without a cleanup step, and the dilution factor is included in the reported value.

(g) Gross alpha too low for alpha spectrometry measurements.

Table 4.11. Summary of organic characterization results for tank WC-12

Analysis class and compound hits, µg/L (a)	WC12-L1	WC12-L3-C Filtered
<i>Volatile organics</i>	(b)	(b)
<i>Water-soluble organics</i>		(c)
<i>Semivolatile organics</i>		
Diethylphthalate		89B
Di-n-butylphthalate		390
Bis(2-ethylhexyl)phthalate		25J
Di-n-octylphthalate		12J
<i>Pesticides/PCBs</i>		
<i>Semivolatile organic TICs (d)</i>		
Unknown	943J (10)	7540J (18)
Unknown hydrocarbons	74J (5)	
Tributylphosphate		630J
Propanol derivative		64J
2,6-Dibromo-4-nitrophenol		54J (e)
Dibutylphthalate		66J

(a) Only compounds whose possible presence was indicated by the analysis are listed. See Tables E.3–E.6 for the target compound lists and Table E.1 for the conversion factors to calculate the reporting limits.

(b) Analysis was not run because the sample was too radioactive for nonzoned laboratory.

(c) Analysis was not run because of instrument breakdown.

(d) Result is the sum of the tentatively identified compounds (TICs). The number of TICs are shown in parenthesis.

(e) Compound could result from nitration of surrogate standard.

4.3.8 Tank WC-13

Tank WC-13 contained (1) an aqueous liquid layer, (2) a layer of sludge on the tank bottom, and (3) an oily layer floating on the aqueous layer.

4.3.8.1 Aqueous Liquid Samples WC13-L1 and WC13S-L

The aqueous sample WC13-L1 collected November 1992 from the midpoint of the liquid layer was radioactive—with ^{137}Cs the principal contributor (Table 4.12). Dose rates (field survey) for ~200 mL of sample were ~2–3 mrem/h. The waste was basic (with sodium the principal metal analyte), and it contained some phosphate, sulfate, chloride, and nitrate. There were also low concentrations of other metal analytes including uranium and mercury.

Sample WC13S-L was the supernatant liquid over the sludge samples, a vertical “column” of tank liquid collected March 1993. The results for WC13S-L were similar to those for WC13-L1. (There was nonprogrammatic inflow, and the tank was pumped once between the two samplings.)

The results of the organic characterization are summarized in Table 4.13. The water-soluble volatile organics analysis showed the presence of 9600 $\mu\text{g/L}$ of methyl alcohol, 8900 $\mu\text{g/L}$ of n-butyl alcohol, and 230 $\mu\text{g/L}$ of an unknown compound eluting near the retention time of acetone. The confirmation column showed that the unknown was not acetone. No semivolatile TCL organic compounds were detected at reporting limits of 250–1300 $\mu\text{g/L}$, and no pesticides or PCBs were found at reporting limits of 2.5–50 $\mu\text{g/L}$. Tributyl phosphate (470J $\mu\text{g/L}$) and three unknowns (total ~2480J $\mu\text{g/L}$) were observed in the semivolatile organic TICs. The volatile organics analysis by GC-MS was not performed because the sample was too radioactive to process in a nonzoned laboratory. The total organic carbon in the aqueous phase was 174 mg/L.

4.3.8.2 Sludge Samples from Tank WC-13

Inorganic and Radiochemical Characterization. The tank WC-13 sludge contained relatively high levels of europium isotopes, ^{60}Co , ^{137}Cs , and ^{90}Sr , and some alpha emitters and metals listed as hazardous under RCRA (Table 4.12). The principal metals in the sludge were sodium, uranium, thorium, calcium, and iron. The dose rate (laboratory survey) for ~200 mL of the composite sludge sample WC13-S was ~50 mrem/h.

Organic Chemical Characterization. Results for the composite sludge sample, WC13-S, are summarized in Table 4.13. The only semivolatile TCL compound found was di-n-butylphthalate (31,000 B $\mu\text{g/kg}$). The semivolatile organic TICs included tributylphosphate (47,000J $\mu\text{g/kg}$) and 6 unknowns (total ~377,000J $\mu\text{g/kg}$). The pesticide/PCB analysis showed 1630 $\mu\text{g/kg}$ of Aroclor 1254. Some pesticide peaks also were detected, but these are believed to be the result of co-eluting components from the Aroclor mixture, and not pesticides. This sample was found to contain approximately 6 wt % oil, which appeared to be vacuum-pump oil. Because of the large amount of organic material, the SVOA extracts required dilution before GC-MS. This procedure raised the reporting limits for the SVOA to 92,000–460,000 $\mu\text{g/kg}$.

Water-soluble volatile organics were determined in an interstitial liquid sample from the sludge. Methyl and n-butyl alcohols were found at 5000 and 5200 $\mu\text{g/L}$, respectively. The volatile organics analysis by GC-MS was not conducted because the sample was too radioactive to process in a nonzoned analytical laboratory.

General Characteristics of Tank WC-13 Sludge. The tank WC-13 sludge was a thin watery sludge with ~8.3 wt % precipitated solids (Table 4.14), which tended to stick to the walls of the

container. This sticky characteristic may be the result of the high organic content (~6 wt % oil). The sonicated sludge had the consistency and appearance of a thick, chocolate milk shake.

There are uncertainties in estimating the inventory of sludge in the tank because of the complex tank geometry, lack of as-built drawings, and limited tank access for measurements. The sludge inventory is probably in the range of 70–175 gal. The lower value assumes a 5.4583-ft-ID tank with a standard dished-bottom head where the head is 11.25 in. deep at the low part, and a sludge depth at the low part of 11.25 in.* The high value assumes a 5.4583-ft-ID tank with a flat bottom and a sludge depth of 12 in. The estimated depth of sludge in the tank based on measurements of the heights of the sludge phase in the sampling tubes were 9.0 in. from the WC13-S1 "core" and 12.0 in. from the WC13-S2 "core." The estimated depth based on the first sample collected (i.e., WC13-S1, 9.0 in.) is probably more representative of the tank contents, than the second sample, because the sludge layer is disturbed (pushed aside) in the process of collecting the first sample. These samples were collected ~8 in. from the tank centerline (i.e., near the low part of the dished-bottom head).

Sludge Density and Solids Values. The density and the solids content of the whole sludge and its dissolved and undissolved components were determined by the method developed by Ceo et. al. (see ref. 3 and Appendix B, Sect. B.7). The results for the sludge sample WC13-S are presented in Table 4.14. The undissolved component was 8.3 wt % (i.e., a thin, watery sludge) with a density of 1.8 g/mL. This is similar to densities reported for the undissolved solids in sludges stored at the Melton Valley and evaporator service tank facilities.³

Sludge Dissolution Test. Exploratory tests with sludge samples from the Melton Valley and evaporator facility storage tanks had indicated that some waste tank sludges dissolve relatively easily in 2 M HNO₃ (ref. 4). This suggested that chemical dissolution might be considered as an option for removing sludges from small tanks in preparation for decommissioning.

A qualitative, "test tube" type experiment was conducted to test the dissolution behavior of the tank WC-13 sludge. About 0.5-g aliquots of sample WC13-S were added to 1 M, 2 M, 4 M, 8 M, and 16 M HNO₃ solutions. There was no appreciable dissolution of the sludge even in 16 M HNO₃ after 4 h contact with occasional stirring. This low (or no) reactivity at room temperature may be the result of the oil coating the solids. It was concluded that acid flushing or in-tank acid dissolution is not a practical option for removing the sludge from tank WC-13.

4.3.8.3 Organic Layer in Tank WC-13

Tank WC-13 contained a dark-brown oily layer, about 0.5-in. deep, floating on the aqueous layer. The results of the organic characterization are summarized in Table 4.13. The volatile organics analysis of a methyl alcohol dilution of the oil did not reveal any volatile compounds beyond traces of methylene chloride and hexane, which also were present in the solvent blank. The reporting limits for the volatile organics analysis were 50–100 mg/L. In the SVOA (reporting limits of 55–280 mg/L), the only TCL constituent detected was di-n-butyl-phthalate at 17,000J mg/L. The pesticide/PCB analysis showed 5060 µg/L of Aroclor 1254 (reporting limits were 105–2100 µg/L). Some pesticide peaks also were detected, but these are believed to be the result of co-eluting components from the Aroclor mixture, and not pesticides. A GC profile of the organic layer suggested that it is vacuum-pump oil or an oil with a distillation range similar to vacuum-pump oil.

*Based on the Drawing D-7152, "Area 3507 Liquid Waste System Addt. 100 Gal S.S. Tank Plan & Det.," 4/25/50.

Table 4.12. Waste characterization data for tank WC-13

Characteristic	WC13-L1 Liquid (a)	WC13S-L Liquid (b)	WC13-S Sludge (c), (d)
<i>Physical properties and miscellaneous data</i>			
Total dissolved solids (mg/mL or mg/g)	18.1	*	23
Total solids (mg/mL or mg/g)	18.4	*	106
Density (g/mL)	1.020	*	1.062
Inorganic carbon (mg/L or mg/kg)	168	*	1,700
Total organic carbon (mg/L or mg/kg)	174	*	41,400
Total carbon (mg/L or mg/kg)	342	*	42,000
<i>RCRA metals (mg/L or mg/kg)</i>			
Ag	(<0.006)	(0.016)	(18) (e)
As	0.054	*	<0.006
Ba	0.001	0.038	18.8
Cd	<0.006	<0.006	1.3
Cr	2.7	3.6	280
Hg	2.7	0.33	390
Ni	3.6	2.4	240
Pb	<0.26	<0.28	34.7
Se	0.015	*	<0.006
Tl	0.008	*	<0.22
<i>Process metals (mg/L or mg/kg)</i>			
Al	12	16	360
Ca	1.9	0.8	3,800
Cu	2.8	3.0	84
Fe	0.7	0.5	3,400
K	48	32	50
Mg	<0.04	<0.03	1,100
Mn	<0.01	<0.01	110
Na	8,200	9,600	11,700
Si	*	*	270
Th	0.44	0.36	1,600
U	14.4	10.0	6,700
V	1.6	1.0	7.8
Zn	0.5	9.4	83
<i>Anions (mg/L or mg/kg)</i>			
Bromide	<0.05	*	(f)
Chloride	170	*	*
Cyanide	0.25	*	0.17
Fluoride	(g)	*	*
Nitrate	84	*	*
Nitrite (h)	Y	*	*
Phosphate	2,200	*	*
Sulfate	580	*	*
Sulfide (h)	N	*	N

Table 4.12. Waste characterization data for tank WC-13 (continued)

Characteristic	WC13-L1 Liquid (a)	WC13S-L Liquid (b)	WC13-S Sludge (c), (d)
<i>Alkalinity</i>			
pH	13.2	13.2	<14 (i)
OH ⁻ (M)	0.29	*	NA (j)
CO ₃ ²⁻ (M)	<0.02	*	NA
HCO ₃ ⁻ (M)	(k)	*	NA
<i>Radiochemical screening (Bq/mL or Bq/g)</i>			
Gross alpha	<1.0E+0	<1.0E+0	5.8E+2
Gross beta	5.0E+3	4.9E+3	6.2E+4
<i>Betalgamma emitters (Bq/mL or Bq/g)</i>			
¹⁴ C	*	*	9.7E+1
⁶⁰ Co	1.33E+1	8.57E+1	1.52E+3
¹³⁴ Cs	<1.0E+0	1.19E+1	<6.0E+1
¹³⁷ Cs	5.54E+3	4.58E+3	4.58E+3
¹⁵² Eu	<9.1E+0	<1.8E+1	3.97E+4
¹⁵⁴ Eu	<1.2E+0	<3.5E+0	2.04E+4
¹⁵⁵ Eu	<4.7E+0	<1.1E+1	5.17E+3
³ H	4.2E+0	*	*
⁹⁰ Sr	<2.0E+0	*	2.86E+2
<i>Alpha emitters (Bq/mL or Bq/g) (l)</i>			
²⁴⁴ Cm	(m)	(m)	1.1E+2
²³⁸ Pu			4.9E+1
²³⁹ Pu/ ²⁴⁰ Pu			4.7E+1
²³³ U/ ²³² Th/ ²³⁷ Np			3.0E+2
²³⁵ U			1.1E+1
²³⁸ U			4.9E+1

(a) Sample collected November 12, 1992, from the midpoint of the liquid layer, ~5 in. above the sludge layer.

(b) Aqueous supernate over the sludge, a vertical "column" of tank liquid collected March 2, 1993. Sample was filtered before analysis.

(c) Sludge sample data are reported on a weight basis.

(d) Sample WC13-S was a composite of the sludge in samples WC13-S1 and WC13-S2, collected March 2, 1993.

(e) Silver analyzed by graphite furnace atomic absorption (GFAA). Result is an estimate because of the large dilution factor.

(f) For data on the anions in the interstitial sludge liquid see Appendix C, Table C.10.

(g) Interference with the fluoride analysis from small aliphatic acids which elute from the AS4A column at about the same time as fluoride.

(h) Qualitative analysis. N = not detected. Y = detected.

(i) pH of sludge interstitial liquid by qualitative analysis with pH stick.

(j) NA = not applicable.

(k) Bicarbonate not stable at this pH.

(l) Alpha emitters by alpha spectrometry.

(m) Gross alpha too low for alpha measurements.

Table 4.13. Summary of organic characterization results for tank WC-13

Analysis class and compound hits (a)	WC13-L1 Aqueous layer	WC13-Surface Oil layer	WC13-S Sludge (b)
<i>Volatile organics</i>	(c)	(d)	(c)
<i>Water-soluble organics (µg/L)</i>			
Methyl alcohol	9,600	(e)	5,000 (f)
n-Butyl alcohol	8,900	(e)	5,200 (f)
<i>Semivolatile organics (µg/L or µg/kg)</i>			
Di-n-butylphthalate		17,000,000J	31,000B
<i>Pesticides/PCBs (µg/L or µg/kg)</i>			
Aroclor-1254		5,060 (g)	1,630 (g)
<i>Semivolatile organic TICs (µg/L or µg/kg) (h)</i>			
Unknown	2,480J (3)		377,000J (6)
Tributylphosphate	470J		47,000J
<i>Oil analysis (wt %)</i>			
Vacuum-pump oil range			~6

(a) Only compounds whose possible presence was indicated by the analysis are listed. See Tables E.3-E.6 for the target compound lists and Table E.1 for the conversion factors to calculate the reporting limits.

(b) Sludge sample data are reported on a weight basis.

(c) Analysis was not run because the sample was too radioactive for nonzoned laboratory.

(d) Traces of methylene chloride and hexane were detected. Their presence in the blank suggests cross-contamination among the diluted oil samples during transport.

(e) Analysis is not appropriate for organic samples.

(f) Analysis performed on the interstitial liquid of the sludge. Units are micrograms per liter of interstitial liquid.

(g) Some pesticide peaks also were detected, but these are believed to be the result of co-eluting components from the Aroclor mixture, and not pesticides.

(h) Result is the sum of the tentatively identified compounds (TICs). The number of TICs are shown in parenthesis.

Table 4.14. Density and solids measurements for sludge samples from tanks WC-13 and WC-14

Property	WC13-S Sludge	WC14-S Sludge
<i>Sludge solids (wt %)</i>		
Total solids	10.6	14.7
Dissolved solids	2.3	2.0
Undissolved solids	8.3	12.7
<i>Density (g/mL)</i>		
Bulk sludge	1.062	1.094
Interstitial liquid	1.024	1.026
Undissolved solids	1.80	2.02

4.3.9 Tank WC-14

Tank WC-14 contained (1) an aqueous liquid layer and (2) a layer of sludge on the tank bottom.

4.3.9.1 Aqueous Liquid Samples from Tank WC-14

Sample WC-14 (November 1992) was radioactive with ^{137}Cs the principal contributor (Table 4.15). Dose rates (field survey) for ~200-mL of sample were ~22–25 mrem/h. The principal inorganic constituents were sodium, nitrate, chloride, and sulfate. The liquid was neutral when the tank was sampled in November 1992, but it was basic when the sludge samples were collected in March 1993. This change was the result of the addition of NaOH solution following waste transfers per waste operations procedures.

The supernatant liquid in Tank WC-14 was resampled in December 1994 for a limited analysis to provide information concerning the nature of the inflow to the tank. Results for sample WC14-L2 collected from the midpoint of the liquid layer in the December 1994 are presented in Table 4.15. The concentrations of nitrate and chloride (anions whose compounds are usually soluble), and sulfate were ~20% of the levels in the November 1992 sampling. By comparison, the ^{137}Cs and ^{134}Cs levels, after adjusting for radioactive decay, were ~50% of the levels in the first sampling. This suggests that the inflow is contaminated with cesium isotopes (e.g., a source such as condensate from a ventilation filter house or a pit which is contaminated from past activities).

The water-soluble volatile organics analysis of sample WC14-L1 (November 1992) showed 14,700 $\mu\text{g/L}$ of methyl alcohol and 330 $\mu\text{g/L}$ of an unknown with a retention time near acetone (Table 4.16). The confirmation column showed that the unknown was not acetone. In the SVOA (reporting limits of 250–1300 $\mu\text{g/L}$), the only TCL detected was benzoic acid at a concentration below the reporting limit. Several semivolatile organic TICs were observed, including tributyl phosphate (9700J $\mu\text{g/L}$), diethylbenzene (280J $\mu\text{g/L}$), 10 unknown TICs (total ~2870J $\mu\text{g/L}$), and two unknown hydrocarbons (total ~1770J $\mu\text{g/L}$). The volatile organics analysis by GC-MS was not performed because the sample was too radioactive to process in a nonzoned laboratory.

Sample WC14-L1 (November 1992) contained 278 $\mu\text{g/L}$ of Aroclor-1248. The analysis of the second sampling of this tank (Table 4.17), conducted almost 1 year later, showed only a trace (5–7 $\mu\text{g/L}$) of Aroclor-1248. A third sampling and analysis of WC-14 aqueous liquid (conducted in December 1994) yielded similar results of 10.9 and 13.2 $\mu\text{g/L}$ of Aroclor-1248 for duplicate samples. The first set (taken in November 1992) of liquid samples from tank WC-14 were collected about 2.5 in. above the sludge phase. These samples were slightly cloudy and may have contained a trace of the sludge phase, which could have elevated the apparent PCB concentration (see Sect. 4.3.9.2).

4.3.9.2 Sludge Samples from Tank WC-14

Inorganic and Radiochemical Characterization. The sludge samples from Tank WC-14 contained relatively high levels of alpha emitters, europium isotopes, ^{137}Cs , ^{60}Co , and ^{90}Sr , and some metals listed as hazardous under RCRA (Table 4.15). The principal metals were uranium, thorium, iron, and sodium. The sludge is a transuranic (TRU) waste containing ~ 1000 nCi/g (wet basis) of transuranics with half-lives greater than 20 years. Dose rates (field survey of the PVC tubes) for the March 1993 sampling were 500 and 220 mrem/h for WC14-S1 and WC14-S2, respectively. Dose rates (laboratory survey of samples in glass jars) for the second set of samples (August 1993) were as follows: WC-14S-1, 130 mrem/h unshielded at contact for 90 g of sample; WC-14S-2, 160 mrem/h unshielded at contact for 47 g of sample. (The glass jars provide some shielding from beta radiation.)

Organic Chemical Characterization. Results for the composite sludge sample WC14-S (March 1993 sampling) are given in Table 4.16. The two TCL semivolatile compounds found were bis(2-ethylhexyl) phthalate (14,000J $\mu\text{g}/\text{kg}$) and naphthalene (3,500J $\mu\text{g}/\text{kg}$). The semivolatile organic TICs included tributylphosphate (270,000J $\mu\text{g}/\text{kg}$), two C_4 -benzenes (\sim total of 95,200J $\mu\text{g}/\text{kg}$), seven unknown hydrocarbons (total $\sim 674,200$ J $\mu\text{g}/\text{kg}$), seven unknown compounds (total $\sim 734,000$ J $\mu\text{g}/\text{kg}$), and several chlorinated biphenyls. The pesticide/PCB analysis found 119,000 $\mu\text{g}/\text{kg}$ of Aroclor-1248. The matrix spike sample (which was not spiked with PCBs, but only certain pesticides) was found to contain 128,000 $\mu\text{g}/\text{kg}$ of Aroclor 1248. The average of the two analyses was 124,000 $\mu\text{g}/\text{kg}$. The PCB results were supported by the semivolatile organic TICs, where 49,100J $\mu\text{g}/\text{kg}$ of tri-, tetra-, and pentachlorobiphenyls were estimated. Heptachlor also was reported at 7370 $\mu\text{g}/\text{kg}$ in the sample, but none was found in the matrix spike sample (< 414 $\mu\text{g}/\text{kg}$), so this is not considered to be a confident hit. The volatile organics analysis was not conducted on the sludge because of insufficient sample quantity and high radioactivity. The water-soluble volatile organics analysis was not performed because of insufficient sample to collect the interstitial liquid.

Because the concentrations of PCBs determined in the first sampling of tank WC-14 exceeded 50,000 $\mu\text{g}/\text{kg}$, the tank was resampled to check the results. The PCB analysis (Table 4.17) of the second set of sludge samples collected in August 1993 confirmed the results for the first set. Samples WC-14S-1 and WC-14S-2 were found to contain Aroclor-1248 at concentrations of 105,000 and 241,000 $\mu\text{g}/\text{kg}$, respectively. Results for the matrix spike and matrix spike duplicate of the WC-14S-1 sample, after correcting for the spike, were 126,000 and 163,000 $\mu\text{g}/\text{kg}$, respectively. The result for the three aliquots of WC-14S-1 was $131,000 \pm 29,400$ $\mu\text{g}/\text{kg}$ (22% relative standard deviation). The matrix spike recoveries could not be accurately measured because the Aroclor-1248 was spiked at 20,000 $\mu\text{g}/\text{kg}$, which was "overshadowed" by the native PCB and aliquot-to-aliquot inhomogeneity. The second set of sludge samples were stirred only with a spatula (per recommended SW-846 practice) before aliquots were taken. Sonication before taking aliquots probably would have produced more homogenous aliquots.

General Characteristics of Tank WC-14 Sludge. The tank WC-14 sludge was a thin, watery sludge with ~ 12.7 wt % precipitated solids (Table 4.14), which tended to stick to the walls of the container. This sticky characteristic may be the result of the organic constituents (~ 1 wt % total organic carbon). The sonicated sludge had a consistency similar to creamed spinach baby food or pond sludge and was brown with black particles.

There are uncertainties in estimating the inventory of sludge in the tank because of the complex tank geometry, lack of as-built drawings, and limited tank access for measurements. The sludge inventory is probably in the range of 70 (or little less) to 130 gal. The lower value assumes a 5.4583-ft-ID tank with a standard dished-bottom head 11.25-in. deep at the low part, and a sludge depth at the low part of 11.25 in.* The high value assumes a 5.4583-ft-ID tank with a flat bottom and a sludge depth of 9.1 in. The estimates of sludge depth in the tank based on measurements of the heights of the sludge phase in the sampling tubes for the first "core" collected were 7.3 and 9.1 in. for the March 1993 and August 1993 samplings, respectively (see Appendix B, Sect. B.2). These samples were collected ~8 in. from the tank centerline (i.e., near the low part of the dished-bottom head).

Sludge Density and Solids Values. The density and the solids content of the whole sludge and its dissolved and undissolved components were determined by the method developed by Ceo et. al. (see ref. 3 and Appendix B, Sect. B.7). The results for the sludge sample WC14-S are presented in Table 4.14. The undissolved component was 12.7 wt % (i.e., a thin, watery sludge) with a density of 2.0 g/mL, a density similar to those reported for the undissolved solids in sludges stored at the Melton Valley and evaporator service tank facilities.³

*Based on Drawing D-7152, "Area 3507 Liquid Waste System Addt. 1000 Gal S. S. Tank Plan & Det.," 4/25/50.

Table 4.15. Waste characterization data for tank WC-14

Characteristic	WC14-L1 Liquid (a)	WC14-L2 Liquid (b)	WC14-S Sludge (c), (d)
<i>Physical properties and miscellaneous data</i>			
Total dissolved solids (mg/mL or mg/g)	14.0	*	20
Total solids (mg/mL or mg/g)	14.4	*	147
Density (g/mL)	1.013	*	1.094
Inorganic carbon (mg/L or mg/kg)	12	*	4,500
Total organic carbon (mg/L or mg/kg)	487	*	10,800
Total carbon (mg/L or mg/kg)	499	*	15,300
<i>RCRA metals (mg/L or mg/kg)</i>			
Ag	(0.032)	(0.199)	(<0.009)
As	<0.002	*	<0.006
Ba	0.006	0.094	64
Cd	0.15	<0.075	140
Cr	0.44	0.775	360
Hg	0.032	*	8.5
Ni	7.8	1.00	1,330
Pb	<0.26	<4.2	580
Se	<0.002	*	<0.006
Tl	<0.002	<1.5	(e)
<i>Process metals (mg/L or mg/kg)</i>			
Al	1.4	<0.25	650
Ca	52	11.6	2,600
Co	0.19	0.23	58
Cu	1.7	1.25	68
Fe	0.3	<0.04	6,200
K	32	63.3	180
Mg	42	2.89	780
Mn	1.1	0.17	74
Na	4,300	3,190	13,000
Th	1.5	39.2	12,000
U	48	1,370	50,000
Zn	1.0	0.25	140
<i>Anions (mg/L or mg/kg)</i>			
Bromide	8.9	6.0	(f)
Chloride	2,000	460	*
Cyanide	0.06	*	0.26
Fluoride	35. (g)	40	*
Nitrate	5,300	1,020	*
Nitrite (h)	Y	Y	*
Phosphate	<0.08	40	*
Sulfate	1,800	324	*
Sulfide (h)	N	*	N

Table 4.15. Waste characterization data for tank WC-14 (continued)

Characteristic	WC14-L1 Liquid (a)	WC14-L2 Liquid (b)	WC14-S Sludge (c), (d)
<i>Alkalinity</i>			
pH	7.4	10.0	-13-14 (i)
OH ⁻ (M)	NA (j)	*	NA
CO ₃ ²⁻ (M)	NA	*	NA
HCO ₃ ⁻ (M)	NA	*	NA
<i>Radiochemical screening (Bq/mL or Bq/g)</i>			
Gross alpha	1.52E+1	9.0E+1	3.6E+4
Gross beta	5.5E+4	3.3E+4	5.2E+5
<i>Beta/gamma emitters (Bq/mL or Bq/g)</i>			
¹⁴ C	*	*	<3.0E+1
⁶⁰ Co	3.10E+1	3.4E+1	1.27E+4
¹³⁴ Cs	1.13E+3	2.9E+2	2.72E+3
¹³⁷ Cs	5.48E+4	2.7E+4	1.58E+5
¹⁵² Eu	<1.3E+2	<1.3E+2	2.64E+5
¹⁵⁴ Eu	<1.3E+1	<1.9E+1	8.48E+4
¹⁵⁵ Eu	<6.8E+1	<7.5E+1	2.11E+4
³ H	3.1E+1	*	*
⁹⁰ Sr	6.64E+2	9.2E+1	2.24E+4
<i>Alpha emitters (Bq/mL or Bq/g) (k)</i>			
²⁴¹ Am	3.8E+0		1.4E+4
²⁴⁴ Cm	2.9E+0		1.3E+3
²³⁷ Np	4.5E+0		
²³⁸ Pu			2.3E+3
²³⁹ Pu/ ²⁴⁰ Pu	2.3E+0		2.2E+4
²²⁸ Th	3.4E-1		
²³³ U/ ²²⁹ Th/ ²³⁷ Np			2.6E+2
²³³ U/ ²³⁴ U	9.3E-1		
²³⁸ U	4.3E-1		3.8E+2

(a) Sample collected November 12, 1992, from the midpoint of the liquid layer, ~2.5 in. above the sludge layer.

(b) Sample collected December 1, 1994, from the midpoint of the liquid layer, ~19 in. above the sludge layer.

(c) Sludge sample data are reported on a weight basis.

(d) Sample WC14-S was a composite of the sludge in samples WC14-S1 and WC14-S2, collected March 24, 1993.

(e) Analysis not run because of spectral interference from uranium.

(f) For data on the anions in the interstitial sludge liquid see Appendix C, Table C.10.

(g) Interference with the fluoride analysis from small aliphatic acids, which elute from the AS4A column at about the same time as fluoride.

(h) Qualitative analysis. N = not detected. Y = detected.

(i) pH of sludge interstitial liquid by qualitative analysis with pH stick.

(j) NA = not applicable.

(k) Alpha emitters by alpha spectrometry.

Table 4.16. Summary of organic characterization results for tank WC-14

Analysis class and compound hits (a)	WC14-L1 Liquid (b)	WC14-S Sludge (c), (d)
<i>Volatile organics</i>	(e)	(f)
<i>Water-soluble organics (µg/L)</i>		
Methyl alcohol	14,700	(g)
<i>Semivolatile organics (µg/L or µg/kg)</i>		
Benzoic acid	100J	
Bis(2-ethylhexyl)phthalate		14,000J
Naphthalene		3,500J
<i>Pesticides/PCBs (µg/L or µg/kg)</i>		
Aroclor-1248	278 (h), (i)	119,000 (i)
Heptaclor		7,370
<i>Semivolatile organic TICs (µg/L or µg/kg) (j)</i>		
Unknown	2,870J (10)	734,000J (7)
Unknown hydrocarbons	1,770J (2)	674,200J (7)
C ₄ -benzene		95,200J (2)
Diethylbenzene	280J	
Tributylphosphate	9,700J	270,000J
Trichlorobiphenyl		25,500J (2)
Tetrachlorobiphenyl		15,600J (2)
Pentachlorobiphenyl		8,000J

(a) Only compounds whose possible presence was indicated by the analysis are listed. See Tables E.3–E.6 for the target compound lists and Table E.1 for the conversion factors to calculate the reporting limits.

(b) Sample collected November 12, 1992.

(c) Sludge sample data are reported on a weight basis.

(d) Sample collected March 24, 1993.

(e) Analysis was not run because the sample was too radioactive for nonzoned laboratory.

(f) Analysis was not run because of insufficient sample quantity and high radioactivity.

(g) Analysis was not run because insufficient sample to collect interstitial liquid.

(h) Some pesticide peaks also were detected, but these are believed to be the result of co-eluting components from the Aroclor mixture, and not pesticides.

(i) The tank was resampled for PCB analysis. See Table 4.7 for the PCB results of the second and third sets of samples.

(j) Result is the sum of the tentatively identified compounds (TICs). The number of TICs are shown in parenthesis.

Table 4.17. PCB results for the second and third sets of samples from tank WC-14

Phrase	Sample	Aroclor-1248
Liquid	WC14-L1 (a)	4.7 µg/L
	WC14-Surface (a)	6.9 µg/L
	WC14-L2-B (b)	13.2 µg/L
	WC14-L2-C (b)	10.9 µg/L
Sludge	WC-14S-1 (a)	105,000 µg/kg
	WC-14S-1-MS (a)	126,000 µg/kg (c)
	WC-14S-1-MSD (a)	163,000 µg/kg (c)
	WC-14S-2 (a)	241,000 µg/kg

(a) Second set of samples collected in August 1993.

(b) Third set of samples collected in December 1994.

(c) "Native" Aroclor-1248 in the aliquot after correction for that added by the matrix spike.

4.3.10 Addendum: Isotopic Data for the Sludge Samples

Just before the completion of this report, some additional data were obtained which had been requested to support the criticality safety evaluation of the LLLW system. Sufficient time was not available to integrate this information into the main body of the report or to include a brief summary of the analytical methods in Appendix B.

The results of the uranium and plutonium isotopic analyses of the sludge samples by mass spectrometry are shown in Tables 4.18 and 4.19, respectively, as the fraction (atom %) of the total present. The principle nuclear materials in the sludge (micrograms per gram of sludge) are presented in Table 4.20. The results for the gross fissile analysis by neutron activation analysis with delayed neutron counting are in good agreement with the values from the mass spectrometry data. The gross fissile content is reported as a ^{235}U equivalent, but it is actually a measure of the total fissile content. The denature ratios for uranium and plutonium are >100 , which is in accordance with administrative requirements for wastes discharged to the LLLW system. Table 4.21 provides a listing of the actinide concentrations (becquerels per gram of sludge) which incorporates the mass spectrometry data for uranium and plutonium.

Table 4.18. Uranium mass spectrometry data for sludge samples

Isotope	WC13-S (atom %)	WC14-S (atom %)
^{233}U	<0.01	<0.01
^{234}U	<0.01	<0.01
^{235}U	0.50	0.22
^{236}U	<0.01	<0.01
^{238}U	99.50	99.77

Table 4.19. Plutonium mass spectrometry data for sludge samples

Isotope	WC13-S (atom %)	WC14-S (atom %)
^{238}Pu	2.03	0.06
^{239}Pu	87.71	89.35
^{240}Pu	8.60	8.42
^{241}Pu	0.31	0.42
^{242}Pu	1.22	1.75
^{244}Pu	0.14	<0.01

Table 4.20. Principal nuclear materials in the sludge samples

Characteristic	WC13-S	WC14-S
<i>Isotope concentration in waste (µg/g)</i>		
²³⁵ U	3.30E+1	1.10E+2
²³⁸ Pu	1.2E-4	4.6E-3
²³⁹ Pu	5.42E+0	6.91E+0
²⁴¹ Pu	1.9E-5	3.3E-2
<i>Gross fissile (µg/g)</i>		
²³⁵ U equivalent (a)	3.0E+1	8.8E+1
<i>Denature ratio</i>		
²³⁸ U/(²³³ U + ²³⁵ U)	2.02E+2	4.59E+2
²³² Th/(²³⁹ Pu + ²⁴¹ Pu)	3.04E+5	1.77E+3

(a) The gross fissile data are based upon neutron activation followed by delayed neutron counting. The equivalent values are relative to known ²³⁵U standards.

Table 4.21. Actinide concentrations in the sludge samples

Isotope	Decay mode	Concentration in waste (Bq/g)		Comment
		WC13-S	WC14-S	
²⁴¹ Am	Alpha		1.4E+4	(a)
²⁴⁴ Cm	Alpha	1.1E+2	1.3E+3	(a)
²³⁸ Pu	Alpha	7.9E+1	2.9E+3	(b)
²³⁹ Pu	Alpha	1.2E+1	1.6E+4	(b)
²⁴⁰ Pu	Alpha	4.5E+0	5.5E+3	(b)
²⁴¹ Pu	Beta	7.4E+1	1.2E+5	(b)
²⁴² Pu	Alpha	1.1E-1	2.0E+2	(b)
²²⁹ Th/ ²³⁷ Np	Alpha	3.0E+2	2.6E+2	(a)
²³² Th	Alpha	6.8E+0	5.0E+1	(c)
²³⁵ U	Alpha	2.6E+0	8.7E+0	(d)
²³⁸ U	Alpha	8.4E+1	6.4E+2	(d)

(a) By alpha spectrometry.

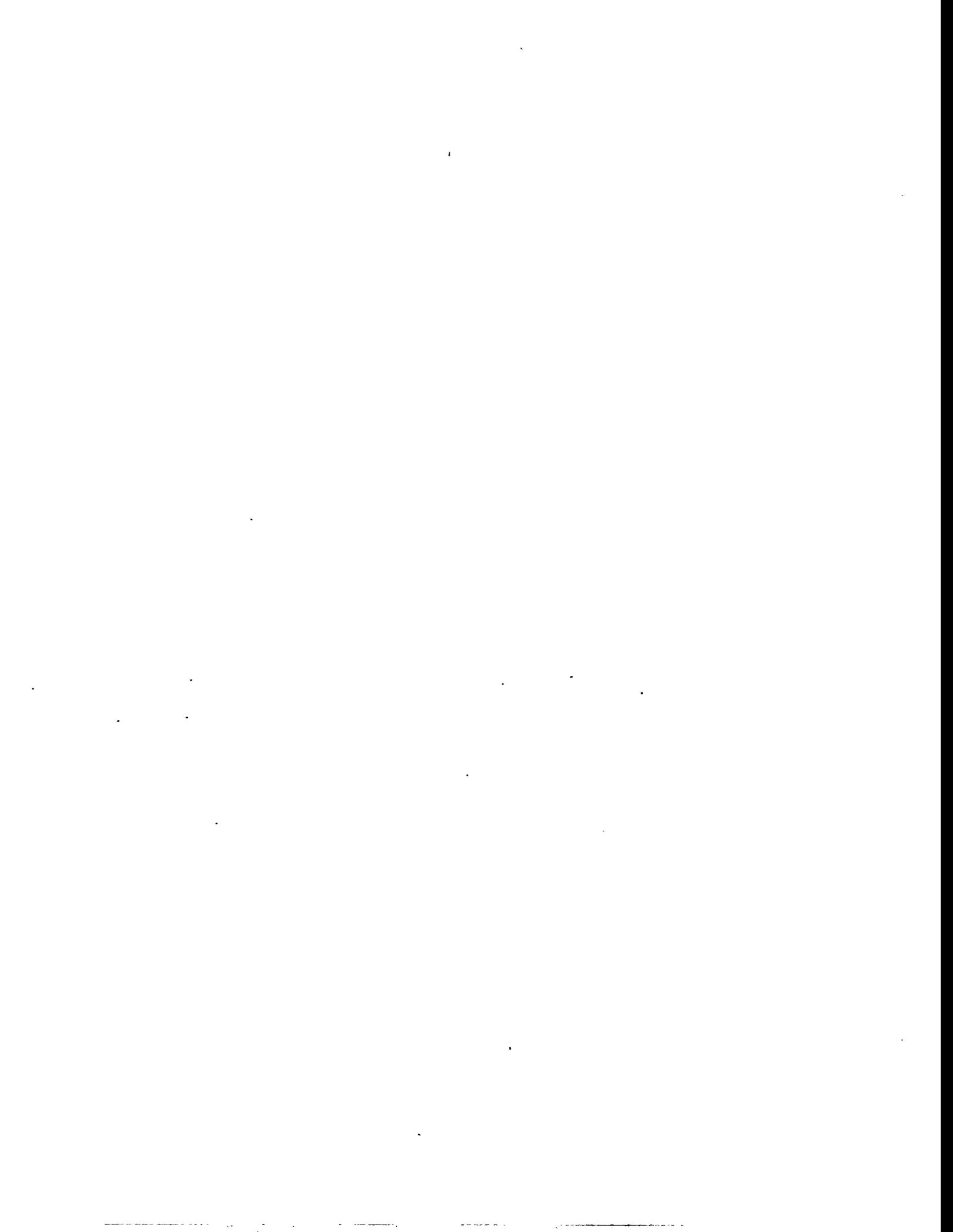
(b) By mass spectrometry; total plutonium alpha by a radiochemical method.

(c) Assumes thorium >99.9 wt% ²³²Th; total thorium by inductively coupled plasma (ICP)

(d) By mass spectrometry; total uranium by ICP.

4.4 REFERENCES

1. U.S. Environmental Protection Agency Contract Laboratory Program, "Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration," SOW No. 787, 1987.
2. U.S. Environmental Protection Agency Contract Laboratory Program, "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration," February 1988.
3. R. N. Ceo, M. B. Sears, and J. T. Shor, *Physical Characterization of Radioactive Sludges in Selected Melton Valley and Evaporator Facility Storage Tanks*, ORNL/TM-11653, Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory, Oak Ridge, Tennessee, October 1990.
4. M. B. Sears, J. L. Botts, and J. M. Keller, *Exploratory Tests of Washing Radioactive Sludge Samples from the Melton Valley and Evaporator Facility Storage Tanks at ORNL*, ORNL/M-1528, Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory, Oak Ridge, Tennessee, September 1991.



Appendix A
FIELD SAMPLING DATA



Table A.1. Profile of tank contents

Tank	Date	Waste inventory (b) (gal)	Access	Depth from tank access point (a)			Depth of liquid layer (in.)	Total depth of waste (in.)
				Top of liquid	Midpoint of liquid layer	Top of sludge layer (c)		
W-17	10/12/92	0	3-in. Pipe	17 ft 3 in.	17 ft 7.5 in.	N (d)	18 ft 0 in.	9
W-18	10/12/92	0	3-in. Pipe	17 ft 11 in.		N	18 ft 0 in.	1
WC-5	10/26/92	240	Riser	13 ft 11 in.	14 ft 9 in.	N	15 ft 7 in.	20
WC-6	10/26/92	54	Riser	14 ft 10 in.	15 ft 2.5 in.	N	15 ft 7 in.	9
WC-8	10/26/92	51	Riser	15 ft 1 in.	15 ft 4.5 in.	N	15 ft 8 in.	7
WC-11	11/3/92	182	Riser	17 ft 4 in.	17 ft 9 in.	(e)	18 ft 2 in.	10
WC-12	11/3/92	67	Riser	17 ft 7 in.	17 ft 11 in.	(f)	18 ft 2 in.	7
WC-13	11/11/92 3/1/93	206 140	Riser Riser	16 ft 10 in.	17 ft 3 in.	17 ft 8 in.	(g)	10
WC-14	11/11/92 3/24/93	172 230	Riser Riser	17 ft 1 in.	17 ft 3.5 in.	17 ft 6 in.	18 ft 4 in.	5
	8/2/93 8/10/93		Riser Riser	16 ft 4 in.	17 ft 1 in.	17 ft 10 in.	18 ft 4 in.	18

(a) Depth measured from the top of the flange on the riser pipe—except for tanks W-17 and W-18, from which measurements are taken from the top of the pipe nipple.

(b) Based on tank level detector readings at the Waste Operations Control Center.

(c) Located with Markland Model 10 Sludge Gun.™

(d) N = not detected.

(e) Detected "dirty" water ~1 in. from tank bottom.

(f) Sludge level detector barely alarmed at tank bottom (~0.5-in. deep layer of a thin sludge).

(g) Not measured. Same configuration as for tanks WC-12 and WC-14.

(h) Based on height of waste in PVC sampling tubes; measurements made at the analytical laboratory.

Table A.2. Field information for samples

Tank	Date collected	Sample	Bottles	Sampling location		Radiation level of ~200-mL sample (mrem/h) (b)
				Liquid level sampled	Sampling depth from tank access point (a)	
W-17	10/13/92	W17-Surface		Surface	17 ft 3 in.	<0.1
		W17-L1	A-H	Midpoint	17 ft 7.5 in.	<0.1
		W17-L2	A-E	Tank bottom	18 ft 0 in.	<0.1
W-18	10/13/92	W18-L1 (c)	A-H	Tank bottom	18 ft 0 in.	<0.1
WC-5	10/29/92	WC5-Surface	1, 2	Surface	13 ft 11 in.	<0.1
		WC5-L1	A-H	Midpoint	14 ft 9 in.	<0.1
		WC5-L2	A-E	Tank bottom	15 ft 7 in.	<0.1
WC-6	10/29/92	WC6-Surface		Surface	14 ft 10 in.	<0.1
		WC6-L1	A-H	Midpoint	15 ft 2.5 in.	<0.1
		WC6-L2	A-E	Tank bottom	15 ft 7 in.	<0.1
WC-8	10/29/92	WC8-Surface		Surface	15 ft 1 in.	<0.1
		WC8-L1	A-H	Midpoint	15 ft 4.5 in.	<0.1
		WC8-L2	A-E	Tank bottom	15 ft 8 in.	<0.1
WC-11	11/4/92	WC11-Surface	1-3	Surface	17 ft 4 in.	11-15
		WC11-L1	A-H	Midpoint	17 ft 9 in.	20-25
		WC11-L2	A-E	Tank bottom	18 ft 2 in.	40-50
WC-12	11/4/92	WC12-Surface	1, 2	Surface	17 ft 7 in.	24-25
		WC12-L1	A-H	Midpoint	17 ft 11 in.	20-24
		WC12-L2	A-C	Tank bottom	18 ft 2 in.	240-700 (d)
	3/17/93	WC12-L3-A		Tank bottom	18 ft 2 in.	500
		WC12-L3-B		Tank bottom	18 ft 2 in.	70
		WC12-L3-C		Tank bottom	18 ft 2 in.	900
		WC12-L4-D		~1 in. above tank bottom	18 ft 1 in.	17

Table A.2. Field information for samples (continued)

Tank	Date collected	Sample	Bottles	Sampling location		Radiation level of ~200-mL sample (mrem/h) (b)
				Liquid level sampled	Sampling depth from tank access point (a)	
WC-13	11/12/92	WC13-Surface	1, 2	Surface	16 ft 10 in.	3
		WC13-L1	A-H	Midpoint	17 ft 3 in.	2-3
	3/2/92	WC13-S1			Core (e)	28
		WC13-S2			Core (e)	33
WC-14	11/12/92	WC14-Surface	1, 2	Surface	17 ft 1 in.	18-21
		WC14-L1	A-H	Midpoint	17 ft 3.5 in.	22-25
	3/24/93	WC14-S1			Core (e)	500
		WC14-S2			Core (e)	220
	8/3/93	WC14-Surface		Surface	16 ft 4 in.	8
		WC14-L1		Midpoint	17 ft 1 in.	8
	8/10/93	WC-14-S1			Core (e)	260
		WC-14-S2			Core (e)	110
	12/1/94	WC14-L1		(f)	14 ft 8 in.	11
		WC14-L2	A-H	Midpoint	16 ft 3 in.	7-12
		WC14-L3			17 ft 4 in.	12
		WC14-L4			17 ft 6 in.	12

(a) Depth measured from the top of the flange on the riser pipe—except for tanks W-17 and W-18 from which measurements are taken from the top of the pipe nipple.

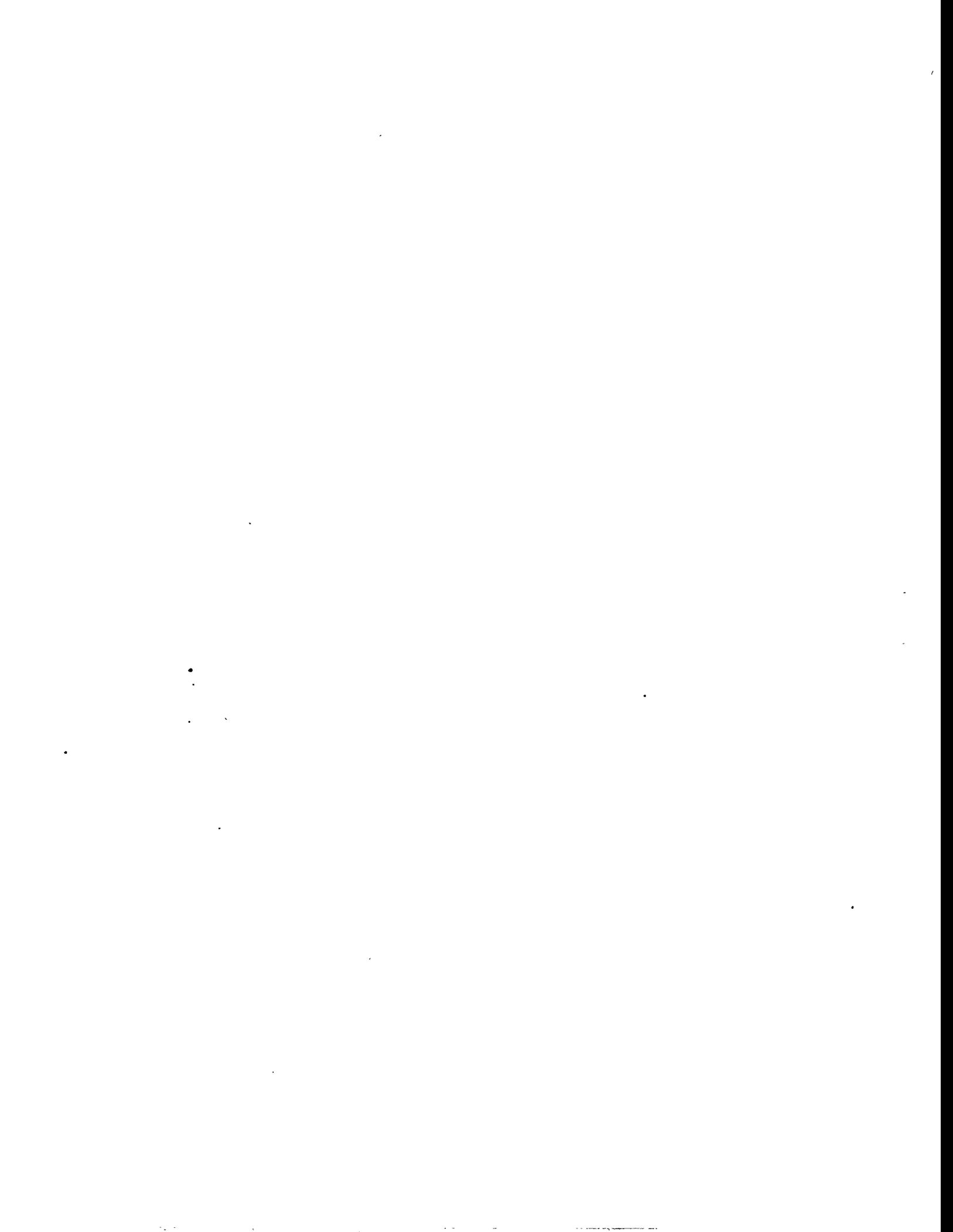
(b) Field survey.

(c) Liquid layer ~1 in. deep. Therefore, only tank bottom sampled.

(d) Solids content of samples varied.

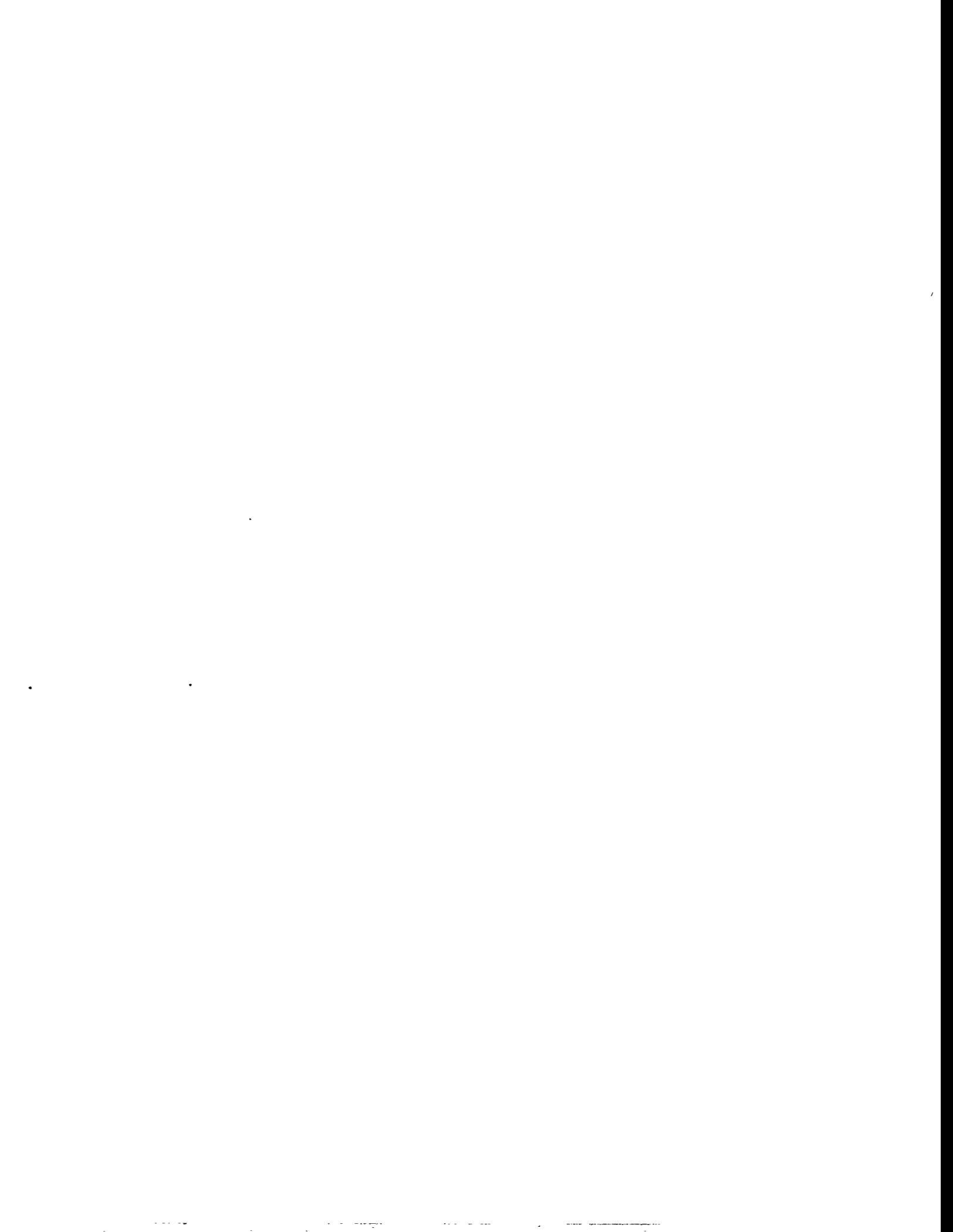
(e) Samplers contained a core of sludge plus supernatant tank liquid. Phases were separated at the analytical laboratory before analysis.

(f) Sample collected ~0.25 in. below top surface of liquid.



Appendix B

INORGANIC AND RADIOCHEMICAL ANALYTICAL SUPPORT



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B.1 INTRODUCTION

The inorganic and radiochemical characterization of the liquid wastes and sludges consisted of the determination of metals, radioactive materials, total organic carbon, inorganic carbon, cyanide, anions in the liquid phase, and some physical measurements. Metals analyses included process metals and metals on the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program Inorganic Target Analyte List.¹

This project has involved the cooperation and participation of several analytical groups. The organization, sample distribution, and most of the inorganic and radiochemical analyses were performed by the Radioactive Materials Analytical Laboratory (RMAL). The organic characterization was conducted by the Separations and Synthesis and Organic Analysis groups.* The Transuranium Analytical Laboratory (TAL) provided alpha spectrometry and duplicated some gross alpha analyses.

Analytical methods from the EPA guides, *Test Methods for Evaluating Solid Waste (SW-846)* and *Methods for Chemical Analysis of Water and Wastes (EPA-600/4-79-020)*, were followed as much as possible in analyses for constituents listed as hazardous under the Resource Conservation and Recovery Act (RCRA).^{2,3} Frequently, modifications to the EPA methods were necessary to (1) reduce radiation exposure to personnel in keeping with the as low as reasonably achievable (ALARA) principle and (2) handle the complex nature of the sample composition to obtain valid data. Methods for the radiochemical analyses and physical measurements were selected from the Analytical Chemistry Division (ACD) methods or modified EPA methods, based on the sample characteristics and type of information needed. Methodology was modified or developed on an as-needed basis for the waste tank samples. Deviations from standard procedures were documented in the raw data files.

A brief description of the inorganic, radiochemical, and physical analytical work, including sample preparation, analytical methods, and quality control, is presented in this appendix.

B.2 INITIAL SAMPLE HANDLING

Samples were received and logged in by RMAL (located in Building 2026), which handled the organization and sample distribution. The ACD request numbers and sample codes for the samples from this project are summarized in Tables B.1 and B.2. This information is useful for the retrieval of data from the ACD data management system. Some of the samples received contained more than one phase. For those samples, a laboratory sample code was sometimes assigned to each phase according to how the assay was to be performed. The terms "SHAKEN" or "UNFILT" as part of the sample code indicate that the sample was shaken to resuspend the particulates before the aliquot was taken and the assay included the sediment. The term "FILT" indicates that the aliquot was filtered before its analysis. The terms "SUPER" or "SUPERNATE" refers to the supernate over settled solids. Tables B.1 and B.2 include also the laboratory sample codes for the organic compound characterization (see Appendix D). For the organic analyses, laboratory codes were assigned to the sample, and to the matrix spike (MS) and the matrix spike duplicate (MSD) if the sample was spiked.

The waste samples were inspected by a health physics (HP) representative, and a field survey of the radiation level was performed on each sample before it was packaged and delivered to

*Toward the end of this characterization campaign, the Organic Analysis group was transferred to the new Analytical Services Organization.

Table B.1. Analytical laboratory sample codes (request number/sample code)

Computer sample code	Radioactive Materials Analytical Laboratory (2026)	Separations and Synthesis (2026) and Organic Analysis (4500S) laboratories	Transuranium Analytical Laboratory (7920)
W17-SURFACE	IPA6906/921013-123		
W17-L1	IPA6906/921013-124		
W-17L1		OAL96477/921014-041	
W-17L1MS		OAL96477/921020-039	
W-17L1MSD		OAL96477/921020-040	
W17-L2	IPA6906/921013-125		
W18-L1(FILT)	IPA6906/921013-126		
W18-L1(UNFILT)	IPA6906/921013-127		
W-18L1		OAL96477/921014-042	
W-18L1MS		OAL96477/921020-041	
W-18L1MSD		OAL96477/921020-042	
WC5 SURFACE	IPA6923/921029-019		
WC5-L1	IPA6923/921029-017		
WC5L1 (E,F,G)		OAL96559/921030-062	
WC5-L2(FILT)	IPA6923/921029-018		
WC5-L2(SHAKEN)	IPA6923/921029-026		
WC5-L2(SUPER)	IPA6923/921029-040		
WC6 SURFACE	IPA6923/921029-022		
WC6-L1	IPA6923/921029-020		
WC6L1 (E,F,G)		OAL96559/921030-063	
WC6-L2(FILT)	IPA6923/921029-021		
WC6-L2(SHAKEN)	IPA6923/921029-027		
WC6-L2(SUPER)	IPA6923/921029-041		
WC8-SURFACE	IPA6923/921029-025		
WC8-L1	IPA6923/921029-023		
WC8L1 (E,F,G)		OAL96559/921030-064	
WC8-L2	IPA6923/921029-024		
WC11 SURFACE	IPA6926/921105-017		
WC11SURFACE 3		OAL96594/921109-017 (a)	
WC11-L1	IPA6926/921105-016		
WC11L1 (E,F,G)		OAL96594/921109-015	

Table B.1. Analytical laboratory sample codes (request number/sample code) (continued)

Computer sample code	Radioactive Materials Analytical Laboratory (2026)	Separations and Synthesis (2026) and Organic Analysis (4500S) laboratories	Transuranium Analytical Laboratory (7920)
MS		OAL96595/921109-019 (b)	
MSD		OAL96595/921109-020 (c)	
WC11-L2(FILT)	IPA6926/921105-018		
WC11-L2(SHAKEN)	IPA6926/921105-019		
WC11-L2(SUPER)	IPA6926/921105-020		
WC12 SURFACE	IPA6926-921105-022		
WC12-L1	IPA6926-921105-021		
WC12L1 (E,F,G)		OAL96594/921109-016	
WC12-L2	(Not analyzed)		
WC12-L3-A	IPA6982/930317-085		
WC12L3A			TRU12642/930510-231
WC12-L3-B	IPA6982/930317-086		
WC12L3-C		OAL97136/930324-038	
WC12-L3-D	(Not analyzed)		
WC13 SURFACE	IPA6929/921112-021		
WC13 SURFACE 2		OAL96658/921117-027 (a)	
WC13-L1	IPA6929/921112-020		
WC13L1 (E,F,G)		OAL96658/921117-025	
WC13-S	IPA6979/930303-068		TRU12642/930510-232
WC13-S	IPA7254/941108-109 (d)		
WC-13-S		OAL97063/930304-107	
WC-13-S MS		OAL97063/930304-108	
WC-13-S MSD		OAL97063/930311-080	
WS-13-S		OAL97063/930304-109 (e)	
WS-13-S MS		OAL97063/930304-110 (e)	
WS-13-S MSD		OAL97063/930304-111 (e)	
WC13S-SUPERNATE	IPA6981/930310-096		
WC14 SURFACE	IPA6929/921112-023		
WC14-L1	IPA6929/921112-022		
WC14L1 (E,F,G)		OAL96658/921117-026	

Table B.1. Analytical laboratory sample codes (request number/sample code) (continued)

Computer sample code	Radioactive Materials Analytical Laboratory (2026)	Separations and Synthesis (2026) and Organic Analysis (4500S) laboratories	Transuranium Analytical Laboratory (7920)
WC14-S	IPA6984/930324-069		TRU12642/930510-233
WC14-S	IPA7254/941108-108 (d)		
WC14S		OAL97161/930330-051	
WC14S-MS		OAL97161/930330-052	
WC14S-SUPERNATE	IPA6984/930324-070		

(a) Organic layer.

(b) Matrix spike of sample WC11L1.

(c) Matrix spike duplicate of sample WC11L1.

(d) Request number/sample code for uranium and plutonium isotopic analyses by mass spectrometry and gross fissile estimate by neutron activation analysis.

(e) Interstitial liquid in sludge.

Table B.2. Analytical laboratory sample codes (request number/sample code) for samples from tank WC-14 collected in August 1993 and December 1994

Sample code	Radioactive Materials Analytical Laboratory (2026)	Separations and Synthesis (2026) and Analytical Services Organization (4500S) laboratories (a), (b)
<u>Samples collected in August 1993</u>		
WC14-SURFACE		OAL97694/930805-046
		SMO00319/931013-192 (c)
WC14-L1		OAL97694/930805-045
		SMO00319/931013-191 (c)
WC-14S-1		OAL97749/930813-035
WC-14S-1 MS		OAL97749/930816-012
WC-14S-1 MSD		OAL97749/930816-013
WC-14S-2		OAL97749/930813-036
<u>Samples collected in December 1994</u>		
WC-14-L1	IPA7264/941201-114	
WC-14-L2	IPA7264/941201-115	
WC-14-L2 (B)		SMO05028/941215-120 (d)
WC-14-L2 (C)		SMO05028/941215-121 (e)
WC-14-L3	(Not analyzed)	
WC-14-L4	IPA7264/941201-116	

(a) In the summer of 1993, the Organic Analysis Laboratory (OAL) was transferred to the Analytical Services Organization (ASO). The OAL request numbers/sample codes were used by both the Separations and Synthesis Laboratory and the OAL. Since the reorganization, the Separations and Synthesis Laboratory has been using the same laboratory sample code as the Radioactive Materials Analytical Laboratory.

(b) ASO results reports show only the analytical laboratory sample code and do *not* include the customer sample code.

(c) Request OAL97694 was relogged as SMO00319 by ASO.

(d) "B" bottle of sample WC-14-L2. Also laboratory sample code 941201-115B.

(e) "C" bottle of sample WC-14-L2. Also laboratory sample code 941201-115C.

Building 2026. Samples were processed in radiochemical hoods in laboratories classified as radiation/contamination zones. After the radiation dose rate and the gross alpha and gross beta activity had been measured, the samples whose radionuclide content was low were later transferred to nonzoned laboratories for the organic compound characterization (see Appendix D). These were liquid samples from tanks W-17, W-18, WC-5, WC-6, and WC-8.

B.2.1 Liquid Samples

Liquid samples, which were designated with an “-L” or “-Surface” in the sample identification code, were decanted into 250-mL, wide-mouth, glass I-CHEM™ sample jars at the sampling point and were capped using lids with Teflon™ seals. Actual sample volumes as measured at the laboratory were ~180–200 mL with ~1 cm (~15–25 mL) headspace. Several bottles of sample material were collected from the same location in the tank. These were usually processed as aliquots of one sample.

Liquid samples were examined visually, and representative samples were photographed on the benchtop using ambient light. Summary descriptions of the physical appearance of the samples and a listing of the ORNL photo numbers are given in Table B.3. The samples collected from the midpoint of the liquid layer (“-L1” code) from tanks W-17, WC-5, WC-6, WC-8 and WC-13 were clear, single-phase liquids. The midpoint samples WC11-L1 and WC12-L1 contained a slight trace of sediment, while the November 1992 sample WC14-L1 was slightly cloudy. The December 1994 samples from tank WC-14 were clear. The samples from the top of liquid layers (“-Surface” code) in tanks WC-11 and WC-13 contained a dark-brown oily layer floating on the aqueous layer. There also were some brown particles suspended in the oil layers; these particles settled slowly to the bottom of the jars. A trace of a yellow oily layer was observed in the surface samples from tank WC-12. The amount of oily material was too small to draw off a sample with a pipette for organic compound analysis, and it is probably not significant. No immiscible organic layers were observed in the surface samples from tanks W-17, W-18, WC-5, WC-6, WC-8, or WC-14. No heavier-than-water immiscible organic phases were observed in any of the liquid samples collected from the tank bottoms (“-L2” code samples and W18-L1).

Some of the samples collected from the tank bottoms contained a small amount (<0.5 wt %, visual estimate) of a fine sediment (samples W18-L1, WC5-L2, WC6-L2, and WC11-L2). Because the amount of sediment was very small, it was not practical to separate the solids from the liquid phase for analysis. The decision was then made to analyze one aliquot including the sediment and a second aliquot (filtered) without the sediment. The difference between the results of the two assays provides a qualitative indication of the constituents in the solids. The bottle containing the most sediment was used for the assay which included the sediment.

Aliquots of the liquid samples were transferred to glass bottles for the total organic carbon, total carbon, and inorganic carbon analyses and to polyethylene bottles for the anion analyses and then stored at 4°C. The EPA sample preservation requirement for acidifying samples for total organic carbon was not performed because it would result in the loss of the inorganic carbon (as CO₂ gas). Samples were (1) tested for pH with a pH stick, (2) for free Cl₂ with KI starch paper, and (3) for sulfide with lead acetate paper as part of the preservation procedure specified in SW-846 Method 9010A (ref. 2). All tests for free Cl₂ and sulfide were negative. Aliquots for cyanide analysis were preserved to a pH >12 with 1 M NaOH, if necessary, except sample WC14-L1, which was not preserved because it was analyzed immediately (within 3 h of receipt). Samples for cyanide assay were stored at 4°C. Liquid samples were preserved/refrigerated the day they were received except the IPA6926 samples, which were preserved/refrigerated 7 days after their receipt.

Table B.3. Visual examination of liquid samples

Sample	ORNL photo number	Comments
W17-Surface	9517-92	Clear, colorless liquid
W17-L1	9516-92	Clear, colorless liquid
W17-L2	9518-92	Clear, colorless liquid
W18-L1 (settled solids)	9513-92	Clear liquid with <0.5% chalk-white sediment
W18-L1 (resuspended solids)	9515-92	
WC5-Surface	9951-92	Clear, colorless liquid
WC5-L1	9952-92	Clear, colorless liquid
WC5-L2 (settled solids)	9953-92	A and B jars: clear liquid with a trace (<0.5%) of a reddish-brown sediment; C to E jars: clear liquid, no sediment
WC5-L2 (resuspended solids)	9950-92	
WC6-Surface	9949-92	Clear, colorless liquid
WC6-L1	9948-92	Clear, colorless liquid
WC6-L2 (settled solids)	9942-92	A to C jars: clear liquid with a trace (<0.5%) of a reddish-brown sediment; D and E jars: clear liquid, no sediment
WC6-L2 (resuspended solids)	9944-92	
WC8-Surface	9947-92	Clear, colorless liquid
WC8-L1	9946-92	Clear, colorless liquid
WC8-L2	9945-92	Clear, colorless liquid
WC11-Surface	10239-92	Dark-brown oily layer floating on the aqueous layer; brown particulates (<0.5%) settled to the bottom with a few suspended particulates
WC11-L1	10238-92	Slightly colored; over time trace (<0.5%) of brown sediment settled
WC11-L2 (settled solids)	10241-92	Slightly colored with <0.5% of a brown sediment
WC11-L2 (resuspended solids)	10240-92	

Table B.3. Visual examination of liquid samples (continued)

Sample	ORNL photo number	Comments
WC12-Surface	10236-92	Trace of a yellow oily layer, quantity too small to sample for analysis; aqueous phase slightly cloudy
WC12-L1	10237-92	Slightly cloudy, very pale-yellow color
WC12-L3-A	*	Thin sludge layer with black solids on the bottom of the jars; liquid layer dark with suspended black fines, very slow to settle
WC12-L3-B	*	
WC12-L3-C	*	
WC12-L3-D	*	Slightly cloudy with a few black particulates
WC13-Surface	10476-92	Dark-brown oily layer floating on the aqueous layer; orange-yellow aqueous phase; dark-brown sediment (<0.5%) settled to the bottom of the jar
WC13-L1	10475-92	Clear, orange-yellow color
WC14-Surface	10478-92	Slightly cloudy, pale-yellow color
WC14-L1	10477-92	Slightly cloudy, pale-yellow color

B.2.2 Samples WC12-L3-A, -B, and -C

Samples WC12-L3-A, -B, and -C were a mixture of a thin sludge and tank liquid collected from the bottom of tank WC-12 with the vacuum-pump sampling system. Initial surveys indicated radiation fields of up to ~900 mrem/h with the probe under the bottle (i.e., under the sludge layer), and up to 480 mrem/h with the probe parallel to the sample bottle on the bench top. The highly penetrating nature of the radiation indicated the presence of ^{60}Co . The samples were examined visually about 6 h after collection. Samples WC12-L3-A and WC12-L3-C contained a layer on the bottom of the sample bottle with black solids (~0.2 and ~0.1 in. deep, respectively) with an overlying layer containing suspended finely divided black solids that made the liquid opaque. The bottom layer in the WC12-L3-B sample was ~0.05 in. deep.

Because of the limited quantity of sample material available and the highly radioactive nature of the solids, the analyses of these samples were limited per the ALARA principal. The samples were stored in lead pigs, and lead-brick shielding units were assembled to use when working with the samples. The samples were processed as a sediment sample using the "A" bottle for the assay including the solids and the "B" bottle for the filtered comparison sample. [Sample WC12-L3-C was signed over to the Separations and Synthesis group for organic analysis (Appendix D).]

B.2.3 Sludge Samples

Sludge samples were delivered to Building 2026 in the field samplers, which contained both the sludge phase and the overlying supernatant tank liquid (Sect. 3, Fig. 3.2). The samples were allowed to stand in the polyvinyl chloride (PVC) sampling tubes to allow the solids to settle by gravity (Sect. 3, Fig. 3.4). The heights of the sludge (solids) and liquid phases in the sampling tubes were then measured, and the phases were separated. The cap was removed from the top of the sampler, and the liquid was removed from on top of the sludge. After removing the free liquid, the bottom closure was opened, and the sludge was allowed to flow into a wide-mouth glass sample jar, which was capped with a Teflon-lined lid. The sludge phase from the second sample was drained into the jar with the first sample. The combined sludge phases were transferred to a Teflon beaker and mixed by sonication to form a composite sample for the tank. The sample was divided into aliquots for the various analyses immediately after it was sonicated.

Tank WC-14 was resampled in August 1993 to confirm the PCB results. The second set of sludge samples were analyzed as two independent samples. They were mixed by stirring with a spatula. The sample preparation for the second set of samples from tank WC-14 was performed by the Separations and Synthesis group, but a description of it is also included in this section of the report for completeness (see Appendix D, Sect. D.2.5.3.2).

The dimensional characteristics of the "core" samples are listed in Table B.4. For estimating the depth of sludge in the tanks, ~0.5 in. should be added to the laboratory measurements of the phases in the sampling tubes to allow for the bottom closure on the sampler and operating the mechanism. The estimated depth based on the first sample collected ("-S1" code) is probably more representative of the tank contents, than the second sample ("-S2" code), because the sludge layer is disturbed (pushed aside) in the process of collecting the first sample.

Aliquots of the sonicated sludge sample for the total organic carbon, total carbon, inorganic carbon, and anion analyses were stored at 4°C. The aliquots for total organic carbon and metals were preserved to pH <2 with H_2SO_4 and HNO_3 , respectively. Off-gassing was noted when sample WC14-S was acidified. There was also a decrease in the viscosity when the tank WC-14 sludge was acidified. Samples were tested for pH with a pH stick, for free Cl_2 with KI starch

Table B.4. Dimensional characteristics of core samples

Date collected	Sample	Phase height in sampler (in.)			
		Sludge	Aqueous liquid	Organic layer	Total
3/02/92	WC13-S1	8.5 (a), (b)	8.0	0.5	16.5
	WC13-S2	11.5 (b)	4.2	0.2	15.9
3/24/93	WC14-S1	6.8 (c)	12.2		19.0
	WC14-S2	5.0 (c)	14.5		19.5
8/10/94	WC14-S1	8.6 (d)	10.9		19.5
	WC14-S2	4.1 (d)	15.2		19.4

(a) Includes 1-in. correction for ~15 mL of sludge which leaked into plastic bag.

(b) Settling time ~1 h after setup on stands and ~2 h after receiving at the laboratory.

(c) Settling time of ~4 h after setup.

(d) Allowed to stand overnight.

Note: For estimating the depth of sludge in the tank, add ~0.5 in. to the above measurements to allow for the bottom closure on the sampler and operating the mechanism.

paper, and for sulfide with lead acetate paper as part of the procedure specified in SW-846 Method 9010A for cyanide. Tests for free Cl_2 and sulfide were negative as far as could be determined. There is some uncertainty about the applicability of these tests to sludge samples. Aliquots for cyanide analysis were also stored at 4°C. Sludge samples were preserved/refrigerated after they were sonicated, which was conducted 1 and 2 days after sample receipt for WC14-S and WC13-S, respectively.

B.2.3.1 Comments on Tank WC-13 "Core" Samples

The tank WC-13 samples, WC13-S1 and WC13-S2, were removed from the PVC tubes shortly after they were received because the samplers were leaking. The settling time was ~1 h after the samples had been set up on stands and ~2 h after they had been received at the laboratory. (Normal protocol is to allow sludge samples to stand overnight before separating the phases.) The sludge solids settled a little more while standing overnight in the sample jar. About 2 mL of the free liquid over the sludge was drawn off with a pipette (sample WS13-S) for the water-soluble organic compounds analysis (see Appendix D). The combined sludge phases (including the free liquid in the jar) were then mixed by sonication to form the composite sludge sample WC13-S. The free liquid was included because it was part of the sludge phase when the phase-height measurements were made.

The original, natural sludge was a thin, watery sludge with predominantly brown solids as well as some red/fuchsia solids which settled in a layer on the bottom. The sludge tended to stick to the walls of the container. The sonicated sludge sample WC13-S had the consistency and appearance of a thick, chocolate milk shake.

The aqueous liquids from the two "core" samples were combined to form the sample WC13S-L, which was filtered and analyzed for metals and some radioactive materials. This sample represented a complete vertical "column" of the aqueous tank liquid. (The oil phase from the core samples was not analyzed.)

B.2.3.2 Comments on "Core" Samples from Tank WC-14, March 1993

The samples from tank WC-14, WC14-S1 and WC14-S2 (March 1993), were removed from the PVC tubes after a settling time of ~4 h, because the samplers were beginning to leak. The phase heights were measured, and the phases were separated and removed from the samplers. No appreciable solids settling was observed after the samples had stood overnight in the sample jars. The combined sludge phases were then mixed by sonication to form the composite sludge sample WC14-S.

The natural sludge from tank WC-14 was thicker than the sludge from tank WC-13. It also tended to stick to the walls of the container. The sonicated sludge WC14-S had a consistency similar to creamed spinach baby food or pond sludge and was brown with black particles.

Except for measuring the pH, the supernatant liquid (sample WC14S-L) was not analyzed because of budget constraints.

B.3 ANALYTICAL METHODOLOGY

The analytical methods for the inorganic, radiochemical, and physical analyses are listed in Table B.5, which includes a brief summary for each procedure and detection limits for most measurements. The detection limit is not adjusted for dilution or interferences. In the following sections additional information is provided on selected procedures and techniques including documentation of matrix spike recovery data, the results for duplicate samples, and deviations from procedures or problems.

B.4 METAL MEASUREMENTS

B.4.1 Sample Preparation for Metal Analysis

Samples were prepared for metal analysis by microwave digestion with a Floyd RMS-150PCM microwave digestion system. The digestion procedures followed the proposed SW-846 Method 3015, "Microwave Assisted Digestion of Aqueous Samples and Extracts," and Method 3051, "Microwave Assisted Digestion of Sediments, Sludges, Soils, and Oils," for metal analysis by inductively coupled plasma (ICP) and graphite furnace atomic absorption (GFAA).² Mercury analysis was also performed on the microwave prepared samples.

Liquid samples were prepared by pipetting 50 mL of sample into the Teflon microwave vessels with 5 mL of 16 M HNO₃. Samples collected from the bottoms of tanks W-18, WC-5, WC-6, and WC-11 contained a small amount of a fine sediment, which was estimated visually as <0.5 wt %. The bottle which contained the most sediment was used for the assay that included the sediment. The sample including the sediment was well mixed by shaking to resuspend the particulates before the aliquot was taken. A second aliquot was filtered through a 0.45- μ m filter before the acid-digestion step.

The liquid samples from the bottom of tank WC-12 contained a small amount of a thin sludge mixed with tank liquid. Because of the limited quantity and highly radioactive nature of the solids, this sample was handled as a sediment sample. Aliquots of 25 mL were pipetted into the Teflon vessels with 5 mL of 16 M HNO₃. Sample WC12-L3-A was shaken to resuspend the particulates before the aliquots were taken. The solids did not dissolve completely during the microwave digestion in ~2.6 M HNO₃. After digestion, the sample was filtered through a No. 541 Whatman filter, and the filtrate was analyzed. The residue from the microwave digestion was not analyzed because of the high beta-gamma activity. Sample WC12-L3-B was used for the filtered comparison sample (rather than an aliquot of WC12-L3-A) to reduce radiation exposure to the analyst. Sample WC12-L3-B was collected from the same location in the tank, but it contained fewer solids; consequently, the radiation dose rate was lower than that of WC12-L3-A (~70 vs ~480 mrem/h measured with the survey meter parallel with the sample on the bench top). Sample WC12-L3-B was filtered through a No. 541 Whatman filter before it was microwave acid digested.

For the WC13-S sludge sample, about 3 g of the sample were microwave-digested with 40 mL of 8 M HNO₃. With sample WC14-S, about 10 g of sludge were microwaved with 35 mL of 4.5 M HNO₃. After they were microwaved, the samples were filtered through a No. 541 Whatman filter. There was a slight discoloration of the filter paper, but no appreciable residue remained after the acid digestion of either WC13-S or WC14-S.

Table B.5. Analytical methods

Parameter	Reference	Method summary	Approximate detection limit
<i>Radiochemical procedures</i>			
Gross alpha	ACD 9 002301, EPA-600-900.0 ACD 9 0733005	Sample is dissolved, if necessary; then samples are diluted, if needed, to reduce dissolved solids. A small volume of sample (usually 0.1 mL) is evaporated on a stainless steel planchet that is alpha-counted with a gas-flow proportional counter in 2-pi geometry.	2 pCi/L
Gross beta	ACD 9 0733005 ACD 9 002302	Sample is dissolved, if necessary; then samples are diluted, if needed, to reduce dissolved solids. A small volume of sample (usually 0.1 mL) is evaporated on a stainless steel planchet that is beta-counted with a gas-flow proportional counter.	4 pCi/L
Gamma spectroscopy	ACD 2 21996 ACD 2 00376 EPA-600-901.1	Sample is dissolved, if necessary, and a 10 mL aliquot is analyzed by gamma spectroscopy. If the dead time exceeds 5%, the sample is diluted or mounted in a less efficient geometry and reanalyzed.	Varies with energy
Alpha spectrometry (Americium-241; Curium-244; Uranium-232, -233, -235, 238; Plutonium-238, -239, -240)	ACD 2 0973 ACD 1 003115	Sample is dissolved, if necessary, and passed through an EIChroM TRU•Spec column to separate actinides from complex matrix. A small volume of sample is then evaporated on a stainless steel plate that is counted with a surface barrier detector.	50 pCi/mL
Strontium-90	EPA-600-905.0 ACD 2 21807	Sample is dissolved, if necessary; strontium carrier is added, and the sample is processed through several purification steps. Final purification is made by precipitation of the strontium as the oxalate. The precipitate is mounted for beta counting with a gas-flow proportional counter.	4 pCi/L

Table B.5. Analytical methods (continued)

Parameter	Reference	Method summary	Approximate detection limit
Tritium	ACD 2 0950	Tritium is distilled from aqueous samples, and a portion of the distillate is analyzed by liquid scintillation counting.	5 pCi/mL
Carbon-14		Samples were heated to 900°C to volatilize organic species and decompose carbonates. Organics were converted to CO ₂ in an oxidizing bed. Off-gases were collected in a basic solution and counted by liquid scintillation.	5 pCi/mL
<i>Inorganic analytical procedures</i>			
<u>Metals by ICP</u>	SW-846-6010A EPA-600-200.7	Liquid samples or dissolved sludges are nebulized, and the aerosol that is produced is transported to an argon plasma torch. Characteristic atomic-line spectra are produced by an inductively coupled plasma (ICP); the intensity of the emission lines from the excited elements are measured and compared to stored calibration curves for quantitation.	
		<u>Wavelength (nm)</u>	
Aluminum (Al)		308.215, 396.152	0.026 mg/L
Antimony (Sb)		217.581	0.062 mg/L
Barium (Ba)		455.404	0.0008 mg/L
Beryllium (Be)		313.042	0.0005 mg/L
Cadmium (Cd)		226.502	0.004 mg/L
Calcium (Ca)		317.933	0.014 mg/L
Chromium (Cr)		267.716	0.005 mg/L
Cobalt (Co)		228.616	0.007 mg/L
Copper (Cu)		324.754	0.004 mg/L
Iron (Fe)		259.94	0.013 mg/L
Lead (Pb)		220.353, 283.306	0.28 mg/L
Magnesium (Mg)		279.079	0.030 mg/L
Manganese (Mn)		257.61	0.0006 mg/L
Mercury (Hg)		253.652	0.20 mg/L
Nickel (Ni)		231.603	0.006 mg/L
Potassium (K)		766.49	0.109 mg/L
Silicon (Si)		251.611	0.006 mg/L
Silver (Ag)		328.068	0.009 mg/L
Sodium (Na)		588.955	0.302 mg/L
Thallium (Tl)		276.787	0.40 mg/L
Thorium (Th)		438.186	0.17 mg/L
Uranium (U)		424.167	0.35 mg/L
Vanadium (V)		292.402	0.004 mg/L
Zinc (Zn)		213.856	0.008 mg/L

Table B.5. Analytical methods (continued)

Parameter	Reference	Method summary	Approximate detection limit
<u>Metals by GFAA</u>	SW-846-7000A	Graphic furnace atomic absorption (GFAA) is based upon the attenuation of characteristic radiation from a hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL) by ground-state atoms in a vapor phase. An aliquot of sample is placed in a graphite tube in the furnace, evaporated to dryness, charred, and atomized. A monochromator and a photomultiplier tube (PMT) are used to measure the attenuated transmitted radiation as it passes through the vapor containing ground-state atoms of the element to be measured. The absorption of the characteristic radiation increases in proportion to the amount of ground-state element in the vapor. The measured absorption is compared to stored calibration curves for quantitation.	
		<u>Wavelength (nm)</u> <u>Pyrolysis</u> <u>Atomization</u>	
Arsenic (As)	SW-846-7060	193.7 1300 2150	0.002 mg/L
Lead (Pb)	SW-846-7421	283.3 850 1800	0.002 mg/L
Selenium (Se)	SW-846-7740	196.0 1300 2050	0.002 mg/L
Silver (Ag)	SW-846-7761	328.1 1000 1800	0.0002 mg/L
Thallium (Tl)	SW-846-7841	276.8 1000 1600	0.002 mg/L
Mercury (Hg) by cold vapor technique	SW-846-7470 SW-846-7471	The flameless atomic absorption (AA) procedure is a physical method based upon the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state with stannous chloride and aerated from the solution. The mercury vapor passes through a cell positioned in the light path of an AA spectrometer. The absorbance (peak height) is measured as a function of concentration.	1–2 ng/mL for a 1 mL sample size
<u>Anions by ion chromatography</u>		Samples, diluted if necessary, are injected directly into an ion chromatograph, and the resultant chromatogram is analyzed to identify anions and determine concentrations.	

Table B.5. Analytical methods (continued)

Parameter	Reference	Method summary	Approximate detection limit
Bromide (Br ⁻)	EPA-600-300.0		0.1 mg/L
Fluoride (F ⁻)	EPA-600-300.0		0.05 mg/L
Chloride (Cl ⁻)	EPA-600-300.0		0.05 mg/L
Phosphate (PO ₄ ³⁻)	EPA-600-300.0		0.2 mg/L
Nitrate (NO ₃ ⁻)	EPA-600-300.0		0.1 mg/L
Sulfate (SO ₄ ²⁻)	EPA-600-300.0		0.1 mg/L
pH	SW-846-9040	The pH of the sample is determined electrometrically using combination electrodes. The measuring device is calibrated using a series of standard solutions of known pH.	Not applicable
Hydroxide (OH ⁻) Acid (H ⁺) Carbonate (CO ₃ ²⁻) Bicarbonate (HCO ₃ ⁻)	American Public Health Association Method 2320	The OH ⁻ , CO ₃ ²⁻ , and HCO ₃ ⁻ are determined by potentiometric titration with 0.1 N HCl; the H ⁺ is determined by titration with 0.1 N NaOH. The change in potential as a function of reagent consumption is recorded with an automatic potentiograph.	0.02 N
Cyanide (CN ⁻)	SW-846-9010A (modified)	The cyanide is released by refluxing the sample with a strong acid and distillation of the HCN into an absorber-scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by ion chromatography (see text, Sect. B.5.3)	0.01 mg/L
Sulfide (S ²⁻)	SW-846-9030A (modified)	Test for sulfide using lead acetate paper before pH adjustment as part of the sample preservation procedure specified in SW-846-9010A. If the screening test is positive, sulfide is to be determined in the cyanide distillate by ion chromatography.	Not applicable
Total organic carbon (TOC)	SW-846-9060	See text, Sect. B.5.2.	1 mg/L
Total carbon (TC)		See text, Sect. B.5.2.	
Inorganic carbon (IC)		See text, Sect. B.5.2.	

Table B.5. Analytical methods (continued)

Parameter	Reference	Method summary	Approximate detection limit
<i>Physical measurement procedures</i>			
Total solids	EPA-600-160.3	A well-mixed aliquot of the sample is quantitatively transferred to a preweighed evaporating dish, evaporated, and dried at 103–105°C to a constant weight.	10 mg/L
Dissolved solids	EPA-600-160.2	A well-mixed sample is filtered, and the filtrate is quantitatively transferred to a preweighed evaporating dish, evaporated, and dried at 103–105°C to a constant weight.	4 mg/L
Density (liquids)	ACD 1 1011	A measured volume of sample is weighed at ambient temperature.	
Density (sludge)	ORNL/ TM-11653	See text, Sect. B.7.	

B.4.2 Metal Analysis by Inductively Coupled Plasma Emission Spectroscopy (ICP-AES)

A Spectroflame model FAO-05 ICP system was modified for use with radioactive samples. The ICP torch box was isolated in a stainless steel enclosure to contain radioactivity. The enclosure is provided with sliding-glass doors and ports that may be gloved if higher levels of alpha radioactivity must be handled. The optic system for the ICP contained three polychromators and a monochromator. The three polychromators allowed for the simultaneous measurement of up to 18 metals (1992–1993 campaign), and the monochromator may be used for sequential measurement of analytes with emission wavelengths between 210–800 nm.* Because this system uses quartz fiber optics to carry light emitted from the plasma to the spectrometer, the system is particularly well suited for radioactive sample analysis. A Meinhard Type K nebulizer was used for all sample analyses.

B.4.3 Mercury Analysis by Cold Vapor Technique

The instrumentation for the mercury cold vapor technique was an MHS-20 (mercury-hydride system) attachment for the Perkin-Elmer model 5100 atomic absorption (AA) system. The MHS-20 included (1) a heated 10-cm quartz cell that is placed in the flame AA light path of the 5100 system, (2) a sample handling system, and (3) a system controller for programming the addition of reductant and reaction times. The signal from the spectrometer is then processed by the 5100 system computer, and peak heights are used for quantitation.

Potassium permanganate was used to test the reducing power of the waste tank samples before they were analyzed for mercury. The samples were tested by adding several drops of 5% potassium permanganate and observing the decolorization. If the potassium permanganate was reduced, a serial dilution was performed, and the test was repeated until the permanganate was no longer reduced.

B.4.4 Metal Analysis by Graphite Furnace Atomic Absorption (GFAA)

The instrumentation used for the GFAA measurements was a Perkin-Elmer 5100 AA system with the HGA-600 furnace option and Zeeman-effect background correction. The spectrometer was placed in a stainless steel enclosure (similar to that of the ICP system) to allow measurements on radioactive samples. The technique used for all GFAA samples was the stabilized temperature platform furnace (STPF) approach,⁴ which was developed by the Perkin-Elmer Corporation. The furnace temperature program included a cool-down step to ambient temperature before the atomization step.

B.4.5 Cleanup of Sample WC14-S by Extraction Chromatography

The sludge sample from tank WC-14 (sample WC14-S) contained high levels of uranium and thorium, which caused spectral interferences in the analysis of several metals (aluminum, antimony, beryllium, copper, magnesium, silver, and vanadium). The uranium and thorium were

*The polychromators were expanded to 22 metals before the 1994 sampling.

removed using a recently developed technique based on extraction chromatography—specifically the use of a commercially available EIChroM* TRU•Spec™ column.⁵

The water was drained from the TRU•Spec columns, and the columns were then pre-conditioned by passing 20 mL of 4 M nitric acid through them. A sample, duplicate, and two spikes were prepared. High and low spikes were prepared because some metals were expected to be between 20–200 ppm, while others were expected to be less than 5 ppm. Aliquots (8 mL) of the dissolved sample were pipetted into plastic beakers. The sample and duplicate were brought to a total volume of 10 mL using 2 mL of 5% nitric acid. The two spikes were brought to a total volume of 10 mL using 2 mL of the appropriate spiking solution. These samples were then passed through the columns and were next washed with 20-mL of 4 M nitric acid. All the eluate was collected and brought to a final volume of 50 mL using 4 M nitric acid. The samples at this point had a pale-yellow tint. It was determined that the columns had been overloaded and that the samples still contained a significant concentration of uranium. New TRU•Spec columns were conditioned as previously described. Aliquots of 10 mL of the previously filtered samples were loaded onto the columns, followed by a wash with 15 mL of 4 M nitric acid. All the eluate was collected and brought to a final volume of 25 mL. The samples were then analyzed by ICP.

The results for the TRU•Spec cleanup of sample WC14-S are presented in Table B.6. Spike recoveries were very good, except for silver, which is precipitated by the chloride in the matrix. Elements which were not affected by spectral interference were analyzed before and after the TRU•Spec cleanup, and good agreement was found.

B.4.6 Data Management and Comments for Metal Analyses

Most of the metal analyses were performed by ICP. Mercury was analyzed by the cold vapor technique, except for sample WC13-S, in which the mercury concentration was high enough to be determined by ICP. Arsenic and selenium were always determined by GFAA. Lead was determined by ICP, except in the IPA6923 group of samples for which GFAA was used. Thallium was analyzed by GFAA for most liquid samples and by ICP for samples WC12-L3-A including solids, WC12-L3-B filtered, WC13-S, and the December 1994 sample WC14-L2. The lead analyses were easier to run by ICP than by GFAA.

High levels of uranium and thorium may cause spectral interferences in the determination of several elements on the EPA Target Analyte List.¹ For sample WC13-S, the only element that was affected significantly was silver, which was analyzed by GFAA and was detected in the sample. The reported result is only an estimate because of the large dilution that was required to bring the concentration within the range of the calibration curve. Also, the normal quality control (QC) samples were not analyzed for silver because of the large dilution factor that would have been required to bring them within the range of the calibration curve.

The high uranium concentration in WC14-S caused spectral interferences in the determination of several metals. The results reported for the following elements were obtained after TRU•Spec cleanup to reduce the uranium concentration: aluminum, antimony, beryllium, copper, magnesium, silver, and vanadium (see Sect. B.4.5). Thallium was not determined on sample WC14-S because of the spectral interference.

Uranium also caused spectral interference in the ICP determination of thallium in WC14-L2 (IPA7264). A less-than value for thallium was estimated based on a visual scan and a comparison with 1000-ppm-uranium and 10-ppm-thallium standards.

*EIChroM Industries, Inc., P.O. Box 2399, Darien, IL 60559.

Table B.6. TRU•Spec cleanup of sludge sample WC14-S

Element	Result (mg/kg)		Spike recovery with TRU•Spec cleanup (%)	
	Before cleanup	After TRU•Spec cleanup	Low spike	High spike
Aluminum	(a)	648	(b)	101
Antimony	(a)	<0.062	99	100
Barium	64.2	65.9	97	100
Beryllium	(a)	3.10	102	100
Cadmium	142	164	90	103
Calcium	2,560	2,210	(b)	98
Chromium	360	370	77	103
Cobalt	58.4	61.2	100	103
Copper	(a)	68.5	96	100
Iron	6,170	5,540	(b)	103
Magnesium	(a)	784	(b)	101
Manganese	74.0	75.2	99	102
Nickel	1,330	1,440	(b)	98
Potassium	175	183	102	102
Silver	(a)	<0.009	3 (c)	(d)
Sodium	13,000	12,300	(b)	121
Vanadium	(a)	6.89	104	102
Zinc	144	158	92	100

(a) Not analyzed on the original sample due to spectral interference from uranium and thorium.

(b) The spike amount was very small compared to the level in the sample.

(c) Silver spike recovery was low due to precipitation by the matrix (e.g. chloride).

(d) High spike for silver was not run.

Metal analysis was conducted using a similar procedure for all methods. All standards are traceable to the National Institute of Standards and Technology (NIST). A calibration curve was generated using a blank and three standards for each method. Immediately after the generation of the calibration curve, a series of checks were performed to verify the curve. The high-standard check used the standard that generated the data point at the high end of the calibration curve. The result from the high-standard was required to be within $\pm 5\%$ of the known value. A calibration check made from a different source than the standards used in the generation of the calibration curve was then analyzed. The acceptance criteria for the calibration check was $\pm 10\%$ of the known value. A mid-check was run every 10 samples, and a check was run at the end of the sample run. The mid-check was the same sample used to generate the data point in the middle of the calibration curve and was required to be within $\pm 10\%$ of the known value. A duplicate and a spike sample were run with each group of samples. The Percent Relative Difference (PRD) for the duplicate samples was calculated by subtracting the duplicate sample result from the actual result, dividing by the average of the two results, and multiplying by 100. The PRD was required to be within $\pm 20\%$ for analytes at 10 times the instrument detection limit (IDL) or higher. Also, ACD blind quality control samples were analyzed with each batch of waste tank samples.

Spike recoveries and the PRDs for duplicates are presented in Tables B.7 and B.8. Spike recoveries were good, generally within the range of 75–125%, except for silver, which was consistently low (–1 to 32%), and aluminum (0%) in IPA7264. The waste tank samples contain chloride, which precipitates silver from the spike. A laboratory spike of sodium prepared from a 1000-ppm high-purity standard was used for IPA6979, 6981, 6982, and 6984 because the normal QC spike was too low for these waste tank samples. No spike was prepared for uranium and thorium for the 1992–1993 sampling campaign. For the 1994 sample WC14-L2, the thorium spike recovery was 111%. A uranium spike was also prepared for WC14-L2, but the spike amount was too low relative to the actual amount of uranium in the sample. The PRDs for duplicates were good (within the $\pm 20\%$ range; see Table B.8). The SW-846 holding times were met except the mercury recheck in IPA6906.

B.5 ANION MEASUREMENTS AND CARBON ANALYSES

B.5.1 Common Anions by Ion Chromatography Analysis

Liquid samples and the interstitial liquid of the sludge samples were analyzed for bromide, chloride, fluoride, nitrate, phosphate, and sulfate (with a qualitative check for nitrite) following EPA-600 Method 300.0, "The Determination of Inorganic Anions by Ion Chromatography" (ref. 3). A Dionex series 4500i ion chromatograph configured with a pulsed electrochemical detector operating in the conductivity mode was used. A carbonate eluant was used with a Dionex IonPac AS4A anion-exchange column for the anion separation. Fluoride was determined in some samples using a weakly eluting sodium hydroxide eluant with a Dionex IonPac AS10 column to separate the fluoride ions from the acetate and formate ions that were present. Sludge samples were centrifuged, and the separated interstitial liquid drawn off for the anion analyses.

The first samples analyzed (W17-L1 and W18-L1) were run on an older AS4A column, and some peak broadening was seen because of decreased column performance. This was only a factor in the analysis of bromide in sample W17-L1, in which the level was low such that the excessive peak tail was causing artificially high results. A range was reported with the lower value excluding the tail and the higher value including it.

Table B.7. Spike recovery for metals

Metal (RCRA metals in bold)	IPA6906 (W18-L1 Filtered) %	IPA6923, 6926 (WC5-L1 Liquid) %	IPA6929 (WC14-L1 Liquid) %	IPA6979 (WC13-S Sludge) %	IPA6981 (WC13S-L Supernate) %	IPA6982 (WC12-L3-A With solids) %	IPA6982 (WC12-L3-B Filtered) %	IPA6984 (WC14-S Sludge) %	IPA7264 [WC14-L2 Liquid (a)] %
Aluminum	98	94	81	(b)	66 (b)	(b)	92	101	0
Antimony	94	99	101	69	91	93	101	99	135
Arsenic	107	109	92	88	*	100	105	83 (c)	*
Barium	98	92	90	76	90	89	106	97	109
Beryllium	98	91	100	90	85	96	100	102	108
Cadmium	100	100	99	77	96	95	104	90	122
Calcium	142	(b)	(b)	(b)	94	(b)	90	98	83
Chromium	101	96	98	(b)	139	75	105	103	113
Cobalt	99	96	114	94	89	95	103	100	113
Copper	96	90	83	86	83	96	104	96	105
Iron	101	100	76	(b)	85	(b)	102	103	116
Lead	106	112	98	100	117	(b)	110	(b)	(d)
Magnesium	96	91	(b)	(b)	85	(b)	105	101	115
Manganese	99	99	111	71	92	(b)	111	99	118
Mercury	81 (e)	75	78	(b)	84	(b)	83	(a)	*
Nickel	102	99	90	(b)	128	64	111	98	123
Potassium	99	99	86	80	97	81	93	102	101
Selenium	85	97	95	104	*	98	103	81 (c)	*
Silicon	*	*	*	(b)	*	*	*	*	*
Silver	3 (f)	5 (f)	15 (f)	(g)	32 (f)	-1 (f)	2 (f)	3 (f)	2 (f)
Sodium	(b)	(b)	(b)	97	86	108	103	121	(b)
Thallium	108	87	111	77	*	83	97	*	(g)
Vanadium	100	95	107	94	87	95	101	104	115
Zinc	136	96	107	85	39	89	110	92	119

(a) December 1994 sample.

(b) The spike amount was small relative to the actual amount of the element in the sample.

(c) For IPA 6984: Arsenic and selenium spike recoveries are from the original sample prior to cleanup with TRU-Spec to remove uranium. The rest of the spike recoveries were obtained after cleanup.

(d) Spike amount was too low for sample.

(e) The first mercury spike recovery was low, but the analysis was repeated, and the result then was acceptable (81%).

(f) Silver spike recovery was low due to fallout.

(g) See Sect. B.4.6.

Table B.8. Percent relative difference (PRD) for duplicate samples

Metal (RCRA metals in bold)	IPA6906 (W17-L1 Liquid) %	IPA6923, 6926 (WC8-L1 Liquid) %	IPA6929 (WC13-L1 Liquid) %	IPA6979 (WC13-S Sludge) %	IPA6981 (WC13S-L Supernate) %	IPA6982 (WC12-L3-A With solids) %	IPA6982 (WC12-L3-B Filtered) %	IPA6984 (WC14-S Sludge) %	IPA7264 [WC14-L2 Liquid (a)] %
Aluminum	IDL (b)	11	1	-8	0	2	-3	3	IDL
Antimony	IDL	IDL	IDL	-11	IDL	IDL	IDL	IDL	IDL
Arsenic	24 (c)	IDL	9	6	*	IDL	IDL	IDL	*
Barium	IDL	2	IDL	0	-2	1	-4	-1	0
Beryllium	IDL	IDL	IDL	-2	5	1	IDL	2	IDL
Cadmium	IDL	IDL	IDL	5	0	1	8	-1	IDL
Calcium	2	-1	7	-1	-28	2	-4	3	-6
Chromium	IDL	7	4	-4	-8	1	0	-1	0
Cobalt	IDL	IDL	IDL	-5	3	1	IDL	-1	5
Copper	7	-7	2	0	0	2	-2	3	-1
Iron	IDL	-31 (c)	-12	0	8	4	-2	3	IDL
Lead	-10	4	IDL	9	IDL	1	0	0	IDL
Magnesium	-3	IDL	IDL	-1	IDL	1	-2	3	13
Manganese	IDL	IDL	-6	-1	6	0	-4	-1	-1
Mercury	IDL	IDL	-1	-9	3	-3	13	15	*
Nickel	IDL	7	5	-4	-8	3	-4	-1	-5
Potassium	-7	-2	1	2	0	0	0	1	-1
Selenium	IDL	IDL	IDL	IDL	*	IDL	IDL	IDL	*
Silicon	*	*	*	18	*	*	*	*	*
Silver	IDL	IDL	IDL	*	12	9	IDL	IDL	-2
Sodium	-1	-1	-1	5	1	1	0	4	-2
Thallium	IDL	IDL	IDL	IDL	*	IDL	IDL	*	IDL
Thorium	(d)	IDL	-4	0	-4	3	-11	4	-2
Uranium	6	3	1	-1	-1	0	3	5	-2
Vanadium	IDL	IDL	1	0	4	4	IDL	1	IDL
Zinc	3	-1	4	1	4	2	-1	-1	IDL

(a) December 1994 sample.

(b) IDL (Instrument Detection Limit) indicates that the results were at or near the detection limit.

(c) Analyte at trace level; see Sect. B.4.6.

(d) Visual scan only on ICP.

The presence of small aliphatic acids (formate and acetate) hampered the determination of fluoride in samples WC5-L1, WC6-L1, WC8-L1, WC13-L1, WC13-S, and WC14-S. These acids are weakly retained on the AS4A column and elute at about the same time as fluoride, thus resulting in overlapping peaks. A less-than value for fluoride was estimated for samples WC5-L1, WC6-L1 and WC8-L1 based on an 0.86-ppm fluoride spike (recovery 85%) in sample WC5-L1. For sample WC13-L1, the peak overlap was such that a reasonable less-than value could not be estimated. Fluoride was determined in samples WC13-S and WC14-S using a newly purchased AS10 column. The acetate and formate anions were not quantified. These anions were considered minor constituents in all samples based on their peak heights and areas compared to the other anions.

An unknown anion was observed in the chromatograms of samples containing formate and acetate. It eluted from the AS4A column with a retention time of about 3.6 min compared with 3.9 min for nitrate. This peak was not identified. It is considered a minor constituent based on its height.

Nitrite peaks were identified in samples WC11-L1, WC12-L1, WC13-L1, WC14-L1 (November 1992), WC14-L2 (December 1994), WC13-S, and WC14-S. The amount of nitrite in the samples varied in direct proportion to the amount of nitrate present.

B.5.2 Total Carbon, Inorganic Carbon, and Total Organic Carbon Analyses

Samples were analyzed for carbon content using a Dohrmann model DC-90 carbon analyzer following SW-846 Method 9060, "Total Organic Carbon" (ref. 2). For liquid samples, total carbon (TC) and total inorganic carbon (IC) were measured directly. Total organic carbon (TOC) results were obtained by taking the difference between TC and IC. For TC, the sample was injected into a 0.2% phosphoric acid carrier stream and then transported to a combustion tube packed with cobalt oxide which was heated to a temperature of 900°C. The furnace was continually sparged with oxygen. All carbon in the sample was converted to carbon dioxide and transported through a cleanup system for the removal of moisture and chloride to a nondispersive infrared (IR) detector. Liquids were assayed for IC by sample injection into a phosphoric acid bath that converted the carbonates in the samples to carbon dioxide gas. The phosphoric acid bath was continually sparged with oxygen, which transported the evolved carbon dioxide through the cleanup system to the IR detector.

For sludge sample WC13-S, the solid particles after sonication were small enough such that the sample was diluted with Type II water and injected directly into the carbon analyzer. The TOC was measured on a portion of the sludge that had been preserved with H₂SO₄ to a pH <2. The H₂SO₄ treatment should convert any inorganic carbonates to CO₂ gas which escapes from the sample. As a check, the preserved sample was also assayed for IC, and none was found. TC and IC, were determined directly on an unpreserved portion of the sludge. The value for TOC obtained by taking the difference between the TC and IC for the unpreserved sample was in good agreement with the TOC result for the preserved sample (4.04 vs 4.14%, respectively).

With sludge sample WC14-S, the preservative (H₂SO₄ to pH <2) partially dissolved the sample and reduced the viscosity. For TOC measurements, the preserved sample was diluted and injected directly into the carbon analyzer. IC was also run on the preserved sample, and none was found.

The unpreserved WC14-S sludge material was not suitable for direct injection. TC was measured following the procedure for solids analysis using the solids adapter for the Dohrmann DC-90. An aliquot of the unpreserved sample was weighed into a platinum boat and then heated to 800°C in a furnace to oxidize the organic species and decompose the carbonates. The furnace was continually sparged with oxygen, which passed through a heated combustion bed packed with

cobalt oxide for conversion of carbon-bearing species to carbon dioxide. The combustion gases were directed to the IR detector via the cleanup system. IC was determined by difference. TOC was also determined on the unpreserved sample after its pretreatment with nitric acid. An aliquot was weighed into the platinum boat, acidified with 1 M HNO₃, and then dried at 80°C for 20 min to drive off the inorganic carbon before placement in the furnace. The TOC measurement on the unpreserved sample using the furnace was in good agreement with the result for direct injection of the preserved sample (1.04 vs 1.08%).

Samples WC11-L1 and WC12-L1 had fine sediments remaining on the bottom of the sample jar. The liquid portions of these samples were used in the carbon analysis.

Samples WC12-L1, WC13-L1 and WC13-S exhibited extremely wide peaks (resulting in "time out" errors) when analyzed for total carbon. This phenomenon was attributed to the sample's sticking to the instrument's tubing and slowly being swept off by the carrier fluid. To resolve this problem, the samples were successively diluted until the "time out" error did not occur. These are the TC results that are reported. The variances between the serial dilutions and straight samples were less than 5%.

B.5.3 Cyanides

Samples were analyzed for cyanide using a microdistillation system developed by Lachat Instruments to liberate the cyanide anion.* The subsequent trapping solution containing free cyanide was analyzed using a Dionex series 4500i ion chromatograph, which was configured with a pulsed electrochemical detector operating in the amperometry mode. A 0.5 M sodium acetate/0.1 M sodium hydroxide/0.5% ethylenediamine eluant was used with a Dionex IonPac AS7 anion-exchange column. The QC requirements of SW-846 Method 9010A, "Total and Amenable Cyanide," were followed.²

Five or 6 mL of liquid sample or 1 g of solid sample was heated to reflux for 30 min under acidic conditions. The off-gases containing HCN pass through a hydrophobic filter into a 1 M NaOH scrubber solution, in which the cyanide anion is trapped. The scrubber solution is cooled, adjusted to volume, and injected into the ion chromatograph for analysis. The procedure is free from interferences such as sulfides, halides, and thiocyanates, which could affect the colorimetric procedure. According to the vendor's literature, the procedure has a detection limit of 1 µg/mL. This detection limit was not confirmed, and a conservative value of 10 µ/L was used as the reporting limit.

Samples WC11-L1 and WC12-L1 had fine sediments on the bottom of the sample jar. The liquid portions of these samples were used in the cyanide analysis.

Samples WC13-S and WC14-S contained surfactants that damaged the hydrophobic membranes in the distillation tubes during the 30-min distillation. The samples were redistilled with smaller portions to prevent the surfactants from coming into contact with the membranes. Sample WC13-S was redistilled in duplicate, and the results were averaged for reporting, but no spike was redistilled because of the limited quantity of sample material. A postdistillation spike was analyzed. Because of the limited sample portion only one replicate of WC14-S could be redistilled with no duplicate or spike. An interfering peak was observed in the chromatogram of WC14-S. A qualitative spike was added to the remainder of the distillate to confirm that the peak being analyzed was the cyanide peak. The cyanide result for WC14-S should be considered a

*Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218.

minimum value. (Note: For future tank waste analyses, it is recommended that the new Lachat cyanide antisurfactant distillation tubes be used rather than the regular tubes.)

The SW-846 holding time requirement of 14 days for cyanide analysis was met, except for samples W17-L1 and W18-L1, which were held for 23 days.

Any sulfide in the samples would have been volatilized as H_2S during the distillation step, and the sulfide anion would have been trapped in the scrubber solution. Using the AS7 column, sulfide would have been detected as a peak before cyanide at about 4 min. No peaks corresponding to sulfide were detected on the chromatograms. This supports the lead-acetate screening-test results for sulfide (see Sect. B.5.4).

B.5.4 Sulfides

When each sample was received at the laboratory, it was tested before pH adjustment for sulfide with the use of lead acetate paper as part of the sample preservation procedure specified in SW-846 Method 9010A (ref. 2). No sulfides were detected in any of the samples undergoing this test. This was confirmed by the absence of sulfide peaks on the chromatograms during the cyanide analysis.

B.5.5 Hydroxide, Carbonate, and Bicarbonate Analysis

Liquid samples were titrated for alkalinity with 0.1 *N* HCl using the Brinkman model 686 titroprocessor connected to a computer with extended graphing capabilities according to American Public Health Association Method 2320, *Alkalinity*, (ref. 6, pp. 2-25 through 2-28). A preliminary titration was made using a 1-mL aliquot to determine proper aliquot size. The samples were pipetted into beakers with phenolphthalein and 40 mL of sodium fluoride/potassium oxalate solution (0.26 *M* NaF/0.07 *M* KOX) to reduce uranium interference. Samples were titrated past the endpoint to a pH range of 3.5 to 4.0. Only hydroxide endpoints were observed; no hydroxide/carbonate or carbonate/bicarbonate endpoints were detected. (Note: Bicarbonate ions are not stable at the pH of these samples; they react with hydroxide ions to form carbonate and water.)

B.5.6 Data Management and Comments for Anion and Carbon Analyses

The calibration curves for anion and cyanide analyses were constructed from a minimum of one blank and three calibration standards traceable to NIST standards. To check calibration of the ion chromatograph, an independently prepared calibration check and a blind QC sample were analyzed. Acceptance criteria for the calibration check was $\pm 10\%$, and the results for the blind QC sample had to be accepted before the analysis was performed. The calibration check and blind QC sample for the cyanide analysis were analyzed before and after distillation. The Dohrmann carbon analyzer was calibrated using a 1-point calibration curve per the manufacturer's instructions. The calibration standard was prepared using reagent grade potassium hydrogen phthalate.

Spiked samples were analyzed with each anion and TOC sample batch (Table B.9). The percent recovery acceptance range was 75–125.

Two replicate measurements were analyzed for the anions and four for carbons. The errors reported in Appendix C are equal to twice the standard deviation of the instrumental measurements.

Table B.9. Spike recovery for anions and total organic carbon

Parameter	IPA6906 (Liquid) %	IPA6923, 6926, 6929 (Liquid) %	IPA6979 (Sludge) %	IPA6984 (Sludge) %	IPA7264 [Liquid (a)] %
Bromide	(b)	89	102	73	(c)
Chloride	90	105	93	106	96
Cyanide	85	85	97 (d)	(e)	*
Fluoride	(b)	85	84	79	84
Nitrate	(b)	95	88	106	90
Phosphate	(b)	103	64 (f)	137	(c)
Sulfate	(b)	104	88	98	(c)
Total organic carbon	107	77	114	105	*

(a) December 1994 sample.

(b) No spike prepared.

(c) Spike diluted out at 500x.

(d) Postdistillation spike; see Sect. B.5.3.

(e) No spike recovery due to membrane-breakdown.

(f) Spike level low compared to sample level.

For cyanide, a duplicate sample was distilled and analyzed with each batch of liquid samples. The PRDs for duplicates were within $\pm 20\%$. As discussed in Sect. B.5.3, the sludge samples contained surfactants which damaged the membranes.

For the alkalinity titrations, the HCl titrant was standardized using reagent grade 0.1 N NaOH. The titrations were run in duplicate and the results averaged.

B.6 RADIOCHEMICAL MEASUREMENTS

The radiochemical characterization of the waste tanks included gross alpha, gross beta, gamma spectrometry, and ^{90}Sr analyses. Tritium was determined on one liquid sample from each tank, and sludge samples were analyzed for ^{14}C . Tank samples that exhibited significant alpha emission were also analyzed by alpha spectrometry. The radiochemical methods are summarized in Table B.5 along with typical detection limits which are not adjusted for dilution or interferences. Most of the radiochemical data are from the RMAL. The Transuranium Analytical Laboratory (TAL) performed the alpha spectrometry and duplicated some gross alpha analyses after cleanup of the samples by extraction chromatography using a TRU•Spec column. The results provided by TAL are identified in the Appendix C tables.

B.6.1 Sample Preparation (by RMAL)

Most liquid samples and all sludge samples were prepared for the radiochemical analyses by microwave-assisted acid digestion following the method described for metals in Sect. B.4.1. Digested samples were used for most of the gross alpha and gross beta measurements, alpha and gamma spectrometry, and for the ^{90}Sr analyses. Four samples were not digested because of insufficient sample material (W18-L1 including sediment, WC5-L2 including sediment, WC6-L2 including sediment, and WC11-L2 including sediment). The liquid samples transferred to TAL were the original sample material (see Sect. B.6.3). The December 1994 liquid samples from tank WC-14 (IPA7264) also were processed without acid digestion.

Original sample material (i.e., without acid digestion) was used for the tritium and ^{14}C analyses.

B.6.2 Gross Alpha and Beta Measurements

A small volume of sample was pipetted onto a stainless steel planchet and slowly evaporated to dryness. After evaporation, the planchets were heated to a dull-red glow over a meeker burner to fix the contamination. The plates were allowed to cool and were then counted. The gross alpha and gross beta measurements at RMAL were performed using a Tennelec LB4000 equipped with 2-pi gas-flow proportional detectors with the alpha counted in the alpha-only mode. Gross alpha counts at TAL were performed with a Nuclear Measurement Corporation PCC 11T counter using a 2-pi gas-flow proportional detector.

For the December 1994 samples from tank WC-14 (IPA7264), the gross beta was determined by beta-scintillation counting. A small volume of sample was pipetted into a 15-mL Ultima Gold™ scintillation cocktail in a scintillation vial and counted on a Packard 2500TR liquid scintillation counter.

B.6.3 Gross Alpha and Alpha Spectrometry (by TAL)

Several samples were transferred to TAL for TRU•Spec cleanup and alpha measurements. A high solids content on the plates from the various salts present caused self-absorption of the alpha and poor alpha spectrometry for isotopic determination, while the high beta in some samples limited the number of alpha counts that could be put onto a plate, thereby leading to poor counting statistics. A reduction in the solids content and the gross beta activity allows for improvement in the energy resolution in alpha spectrometry and for more accurate gross alpha measurements. The cleanup method is based on extraction chromatography using a commercially available EIChrom TRU•Spec column. This column absorbs uranium, thorium, and transuranics at high nitric acid concentrations while allowing the unwanted solids and most of the beta-emitting ^{137}Cs and ^{90}Sr to pass through. The transuranics, uranium, and thorium are then eluted from the column.

The TRU•Spec cleanup was performed according to the ACD Method 2 0973, "Sample 'Clean-up' For Alpha Counting." The TRU•Spec column was drained and then conditioned by passing 20 mL of 4 M nitric acid through the column. The sample (either liquid sample or dissolved sludge sample) was adjusted to about 4 M nitric acid and loaded onto the column. The column was washed with 4 M nitric acid, thus keeping the total wash volume under 65 mL. The actinides and lanthanides were stripped off the column using at least 20 mL of 0.05 M nitric acid, and the eluate was then diluted to a known volume. The column was then stripped using 20 mL of 0.1 M ammonium oxalate to remove any uranium, thorium, or plutonium retained by the column. A new column was used for each sample.

The nitric acid and ammonium oxalate eluates were analyzed separately, and the results were summed. Gross alpha was determined according to ACD Method 9 002301, "Gross Alpha Measurement in Aqueous/Organic Solutions." Samples containing significant alpha activity were assayed for isotopes following ACD Method 1 003115, "Alpha Spectrometry with the ND9900 Data Acquisition System." Alpha spectrometry was performed on a Tennelec TC256 alpha spectrometer with PIP detector, and the spectrometry data was then processed with the ND9900 Data Acquisition system. Gross alpha was counted with a Nuclear Measurement Corporation PCC 11T counter using a 2-pi gas-flow proportional detector.

No matrix spike samples were prepared by TRU•Spec because of a limited amount of sample material and budget constraints.

B.6.4 Gamma Spectrometry

Up to 10 mL of sample were pipetted into a plastic, narrow-mouth scintillation vial and brought to a total volume of 10 mL. The sample amount used was determined by the estimated activity of that sample and the observed "dead" time of the instrument. These vials were then counted 3 cm from the germanium detector for 1 h. A Tennelec high-purity germanium detector (HPGe) with a nominal 25% efficiency was used to determine all gamma spectrometry measurements. Canberra's AccuSpec™ system was used to analyze the gamma spectra.

B.6.5 Strontium-90 Analysis

The sample was dissolved if necessary (Sect. B.6.1). A stable strontium carrier was added, and the sample was next processed through several precipitation steps to remove radium and yttrium which interfere with the measurement of the ^{90}Sr activity. The strontium was then precipitated and

beta-counted using a 2-pi gas-flow proportional detector. This method measures the total radioactive strontium (i.e. ^{89}Sr and ^{90}Sr in combination). The term " ^{90}Sr " as used elsewhere in this report actually refers to the total radioactive strontium.

B.6.6 Carbon-14 Analysis

The combustion tube option of the Dohrmann DC-90 carbon analyzer was adapted for the analysis of ^{14}C . Samples were heated to 900°C to volatilize organic compounds and decompose carbonates. The furnace was continually sparged with oxygen, and the off-gases then passed through an oxidizing bed of cobalt oxide to convert organic species to carbon dioxide. The gases were then directed to gas-washing bottles filled with a carbon-dioxide-absorbent solution. After they were collected, the aliquots of the carbon-dioxide-absorber solution were pipetted into a compatible liquid scintillation cocktail and counted using a Packard 2500TR liquid scintillation counter.

Samples WC13-S and WC14-S were analyzed as one batch. QC consisted of analyzing both samples in duplicate, one standard, a blind QC sample in duplicate, a matrix spike, and a reagent blank. The spike recovery was 116%.

B.6.7 Tritium Analysis

The samples were pipetted into flasks with two drops of phenolphthalein and made basic, when necessary, by adding sodium hydroxide a drop at a time. Most of the tank samples were already basic. The samples were then distilled twice to separate the tritium from the sample matrix. Up to 2 mL of the distilled samples were pipetted into scintillation vials containing Ultima Gold scintillation cocktail and counted using a Packard 2500TR liquid scintillation counter.

B.6.8 Quality Assurance

Blind QC samples supplied by ACD's quality assurance group were run with each batch of samples for all sample analyses except alpha spectrometry. Amersham AMR.43, a NIST traceable standard containing the isotopes ^{241}Am , ^{239}Pu , and ^{244}Cm , was used as the QC standard for alpha spectrometry. All controls were reported to the ACD computer network (ANALis), which gives immediate response to the analyst for out of control results.

Samples were run in duplicate at RMAL to measure gross alpha, gross beta, ^{90}Sr , ^{14}C , and tritium, and the results averaged for reporting the data. Gross alpha screening was performed by two separate laboratories for the following samples: WC11-L1, WC12-L1, WC12-L3A, WC13-L1, WC14-L1 (November 1992), WC13-S, and WC14-S. Results from the two laboratories are presented in Appendix C, Tables C.6-C.10. The TAL laboratory used a TRU•Spec cleanup before the alpha measurements, and the results are considered to be more nearly accurate than the results for the direct measurements without cleanup (see discussion in Sect. B.6.3).

B.6.9 Comments on Radiochemical Measurements

During the gross beta analysis there was a problem initially with the apparent loss of ^{137}Cs . For the liquid samples from tanks WC-11 through WC-14, the gross beta counts were only about

half the gamma spectrometry results for ^{137}Cs . While the exact chemical mechanism by which the ^{137}Cs was lost was and is not understood, it became evident that the loss occurred while flaming the planchets to fix the contamination. In the initial work, a "raw" liquid sample was pipetted directly onto the planchets. There were difficulties with rewetting of the plates from sorbtion of moisture during flaming which may have led to overheating of the plates and cesium volatilization. There were also problems with a nonuniform distribution of solids on the plates. The decision was then made to use microwave-assisted acid digestion to prepare the liquid samples for gross alpha and gross beta analyses and to flame the plates to barely a dull-red glow. Most of the reported gross alpha and gross beta data are for the digested samples. However, four samples could not be rerun because of insufficient sample material: W18-L1 including sediment, WC5-L2 including sediment, WC6-L2 including sediment, and WC11-L2 including sediment. No gross beta values are reported for these samples because of the problem with the analysis.

For the December 1994 liquid samples from tank WC-14, the gross beta was determined by beta scintillation counting. This method avoids the problem of ^{137}Cs volatilization.

The RMAL instrument was designed for low-level radioactive samples which limited the combined alpha plus beta activity to a maximum of 40,000 counts. Because of the high beta activity in the samples from tanks WC-11 through WC-14 the number of alpha counts which could be put on a planchet was low. For samples in which the alpha count was low, this limitation resulted in poor counting statistics and a loss of sensitivity. The samples also had a relatively high solids content, which may result in self-absorption of alpha. A cleanup step was needed to remove unwanted solids and to reduce the beta activity. A method based on the precipitation of a hydrous ferric oxide (commonly called ferric hydroxide) had been used to prepare other waste tank samples (e.g., samples from the Melton Valley storage tanks) for alpha measurements.⁷ Attempts to prepare the liquid samples from tanks WC-11 through WC-14 using the ferric-ion-precipitation method were unsuccessful because of difficulty in forming the precipitate. While the exact mechanism is not understood, the implication is that these samples contained an agent that complexes ferric ions. The decision was then made to transfer the midlevel liquid samples ("-L1" code) from tanks WC-11 through WC-14 and the sludge samples to TAL for cleanup by TRU•Spec before alpha measurements were taken.

B.7 SLUDGE DENSITY AND SOLIDS MEASUREMENT

B.7.1 Method for Determining Sludge Density and Solids Values

In principle, density and solids content are simple measurements to make; in this study, however, accurate values—particularly the percentages of dissolved and undissolved solids—were difficult to obtain. The liquids in these tanks have moderately high salt contents, and most have high pH levels. If a sludge were simply vacuum filtered, the filter cake would retain some interstitial liquid. After they were dried, the solids would contain an indeterminate mass of salts that had been dissolved in the original sample. If the filter cake were rinsed with water to remove the salts, some material that was insoluble in the original matrix might dissolve. The method developed by Ceo et al. provides an indirect means of measuring the density and the solids content of the whole sludge and its dissolved and undissolved components.⁸

Seven measurements were performed on each sludge sample to determine the density and the solids content of the whole sludge, its component interstitial liquid, and its component undissolved solids. Two portions of a sludge sample were required for these measurements.

The first portion of sludge (~6 g) was placed into a tared, graduated 15-mL glass centrifuge tube. Care was taken so that no sludge was smeared onto the inner wall of the centrifuge tube

above the bulk of the sample. The centrifuge tube was then sealed with its screw cap and spun at a high rate (>4000 G) for 15 min to compact the solids and to displace any entrained air from the sludge. After centrifugation, a layer of clear liquid was visible above the compacted solids; the total volume of the sample was taken as the sum of the liquid and compacted solids volumes. The centrifuge tube was reweighed to determine the bulk sludge mass. The density of the bulk sludge was calculated by dividing the mass of the bulk sludge by the sample volume.

The separated interstitial liquid was withdrawn from the centrifuge tube and filtered through a 0.45- μm syringe filter. Then, 1.00 mL of the filtered liquid was pipetted into a tared glass beaker. This beaker was weighed to determine the liquid mass and subsequently dried at 110°C for 16 h and weighed to determine the mass of the residual dried salts. These measurements give the density (in grams per milliliter) of the interstitial liquid, the quantity of dissolved solids per milliliter of liquid, and the water loss per milliliter of liquid upon drying.

The second portion of sludge was used to determine the percentage of total solids in the bulk sludge. About 1 g of the sludge was placed into a tared beaker. The beaker containing the sample was reweighed to determine the wet sludge mass and then dried at 110°C for 16 h. The beaker containing the dried solids was weighed.

B.7.2 Calculational Model

Two portions of each sludge sample were required for a set of measurements. The first portion (about 6 g) was placed into a tared, graduated 15-mL centrifuge tube and weighed. The sludge was centrifuged for 15 min at high speed before the total volume was read and the bulk density was calculated:

$$\text{Bulk density} = \frac{\text{wet mass } 1}{\text{wet volume } 1} .$$

The separated interstitial liquid was drawn off from the centrifuged sample using a transfer pipet, then filtered through a 0.45- μm filter. Then 1 mL of the filtered solution was weighed to determine the interstitial liquid density:

$$\text{Liquid density} = \frac{\text{solution mass}}{1 \text{ mL}} .$$

After being reweighed, the 1-mL sample was dried at 110°C for 16 h and then reweighed to determine loss of water and other volatiles:

$$\text{Water loss (1 mL liquid)} = \text{solution mass} - \text{residue mass} .$$

A second portion (about 1 g) of the original sludge was taken. This portion was placed into a tared beaker and weighed. The sludge was dried for 16 h at 110°C and then reweighed to determine the total solids content and loss of water and other volatiles:

$$\text{Total solids} = \frac{\text{dry mass 2}}{\text{wet mass 2}},$$

$$\text{Water loss (sludge)} = \text{wet mass 2} - \text{dry mass 2}.$$

The dissolved solids content was calculated from the solution residue mass, the water losses from sludge and solution, and the wet sludge mass:

$$\text{Dissolved solids} = \frac{\text{residue mass}}{\text{wet mass 2}} \times \frac{\text{water loss (sludge)}}{\text{water loss (1 mL liquid)}}.$$

The mass of the interstitial liquid present in the second sludge portion is calculated from the liquid density and the ratio of water lost in drying the sludge to that lost in drying the liquid:

$$\text{Liquid mass} = \text{liquid density} \times \frac{\text{water loss (sludge)}}{\text{water loss (1 mL liquid)}}.$$

The undissolved solids content was calculated by difference, as follows:

$$\text{Undissolved solids} = \text{total solids} - \text{dissolved solids}.$$

The undissolved solids density was also calculated as the ratio of differences in mass and volume:

$$\text{Floc density} = \frac{\text{wet mass 2} - \text{liquid mass}}{\left(\frac{\text{wet mass 2}}{\text{bulk density}} \right) - \left[\frac{\text{water loss (sludge)}}{(\text{water loss}/1 \text{ mL liquid})} \right]}.$$

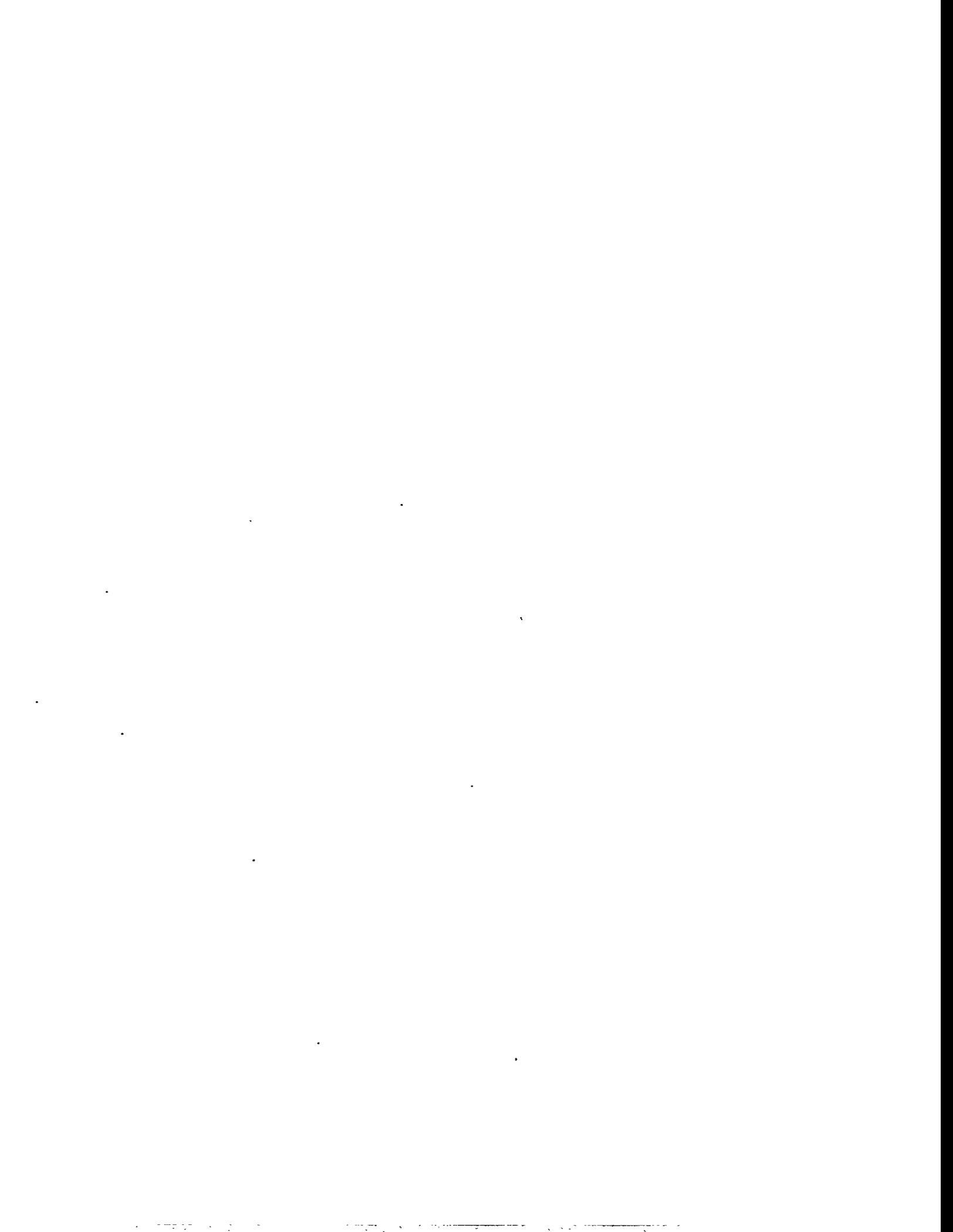
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Appendix C

**LISTING OF PHYSICAL, INORGANIC, AND
RADIOCHEMICAL ANALYTICAL DATA**



The analytical data for the physical, inorganic, and radiochemical analyses are presented in this appendix. These tables and the tank contents to which each refers are organized as follows:

Tank W-17	Table C.1
Tank W-18	Table C.2
Tank WC-5	Tables C.3a and C.3b
Tank WC-6	Tables C.4a and C.4b
Tank WC-8	Table C.5
Tank WC-11	Tables C.6a and C.6b
Tank WC-12	Table C.7
Tank WC-13 (liquid)	Table C.8
Tank WC-13 (sludge)	Table C.10
Tank WC-14 (liquid)	Tables C.9 and C.11
Tank WC-14 (sludge)	Table C.10

Table C.1. Analytical data for tank W-17

Parameter measured	Units	W17-Surface		W17-L1		W17-L2	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	*		3.88		*	
Total solids	mg/mL	*		3.94		*	
Density	g/mL	1.005		1.002		*	
Inorganic carbon	mg/L	*		22.1	2.2	*	
Total organic carbon	mg/L	*		233	23	*	
Total carbon	mg/L	*		255	26	*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	*		<0.021		<0.021	
Antimony	mg/L	*		<0.091		<0.091	
Arsenic	mg/L	*		0.006	0.002	0.003	0.007
Barium	mg/L	*		<0.0004		<0.0004	
Beryllium	mg/L	*		<0.0004		<0.0004	
Cadmium	mg/L	*		<0.006		<0.006	
Calcium	mg/L	*		3.12	0.01	3.92	0.07
Chromium	mg/L	*		<0.006		0.008	0.003
Cobalt	mg/L	*		<0.009		<0.009	
Copper	mg/L	*		0.005	0.001	0.006	0.001
Iron	mg/L	*		<0.003		0.029	0.002
Lead (a)	mg/L	*		0.4	0.103	0.384	0.104
Magnesium	mg/L	*		0.111	0.037	0.254	0.013
Manganese	mg/L	*		<0.0006		<0.0006	
Mercury	mg/L	*		<0.005		<0.005	
Nickel	mg/L	*		<0.015		<0.015	
Potassium	mg/L	*		0.441	0.056	0.54	0.005
Selenium	mg/L	*		<0.002		<0.002	
Silver	mg/L	*		(<0.006)		(<0.006)	
Sodium	mg/L	*		1430	16	1460	22
Thallium (b)	mg/L	*		<0.002		0.003	0.004
Vanadium	mg/L	*		<0.004		<0.004	
Zinc	mg/L	*		10.5	0.1	10.7	0.1
<i>Process metals</i>							
Thorium (c)	mg/L	*		<0.17		<0.17	
Uranium	mg/L	*		3.26	0.18	3.10	0.12

Table C.1. Analytical data for tank W-17 (continued)

Parameter measured	Units	W17-Surface		W17-L1		W17-L2	
		Result	+/-	Result	+/-	Result	+/-
Anions							
Bromide (d)	mg/L	*		12.5-30.2		*	
Chloride	mg/L	*		30	1.9	*	
Cyanide	mg/L	*		<0.01		*	
Fluoride	mg/L	*		<0.1		*	
Nitrate	mg/L	*		<0.1		*	
Nitrite (e)	mg/L	*		N		*	
Phosphate	mg/L	*		<0.1		*	
Sulfate	mg/L	*		<0.1		*	
Sulfide (e)	mg/L	*		N		*	
Alkalinity							
pH		*		12.4		12.4	
Acid (H ⁺)	mol/L	*					
Base (OH ⁻)	mol/L	*		0.056	0.001	*	
Carbonate	mol/L	*		<0.02		*	
Bicarbonate	mol/L	*				*	
Radiochemical screening							
Gross alpha	Bq/mL	<1		<1		<1	
Gross beta	Bq/mL	23	0.4	24	0.4	23	0.4
Beta/gamma emitters							
Am-241	Bq/mL	<0.56		<0.67		<0.69	
Co-60	Bq/mL	<0.12		<0.16		<0.14	
Cs-134	Bq/mL	<0.15		<0.14		<0.13	
Cs-137	Bq/mL	13.0	0.3	16.0	0.3	15.9	0.3
Eu-152	Bq/mL	<0.051		<0.54		<0.54	
Eu-154	Bq/mL	<0.27		<0.22		<0.43	
Eu-155	Bq/mL	<0.30		<0.32		<0.32	
H-3	Bq/mL	*		17.9	0.24	*	
Sr-90	Bq/mL	*		4.13	0.55	*	
Zn-65	Bq/mL	(f)		(f)		(f)	

(a) Analyzed by inductively coupled plasma (ICP).

(b) Analyzed by graphite furnace atomic absorption (GFAA).

(c) Visual scan on ICP.

(d) Excessive peak tailing due to degrading column performance; lower value excludes tail, higher value includes tail.

(e) Qualitative analysis. N = not detected.

(f) Possibly some Zn-65 (~2 Bq/mL) was observed, but this observation is suspect given the half-life and the known tank history.

Table C.2. Analytical data for tank W-18

Parameter measured	Units	W18-L1 Filtered		W18-L1 Supernate		W18-L1 Including sediment	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	2.07		*		*	
Total solids	mg/mL	*		*		*	
Density	g/mL	*		1.001		*	
Inorganic carbon	mg/L	*		57	5.7	*	
Total organic carbon	mg/L	*		11	1.1	*	
Total carbon	mg/L	*		68	6.8	*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	<0.021		*		<0.021	
Antimony	mg/L	<0.091		*		<0.091	
Arsenic	mg/L	<0.002		*		0.004	0.002
Barium	mg/L	<0.0004		*		<0.0004	
Beryllium	mg/L	<0.0004		*		<0.0004	
Cadmium	mg/L	<0.006		*		<0.006	
Calcium	mg/L	3.12	0.03	*		14	0.2
Chromium	mg/L	<0.006		*		0.081	0.006
Cobalt	mg/L	<0.009		*		<0.009	
Copper	mg/L	0.007	0.002	*		0.029	0.002
Iron	mg/L	<0.003		*		0.819	0.011
Lead (a)	mg/L	<0.27		*		1.32	0.02
Magnesium	mg/L	2.06	0.05	*		3.31	0.01
Manganese	mg/L	<0.0006		*		0.016	0.001
Mercury	mg/L	<0.005		*		<0.005	
Nickel	mg/L	<0.015		*		<0.015	
Potassium	mg/L	0.42	0.054	*		0.505	0.025
Selenium	mg/L	<0.002		*		<0.002	
Silver	mg/L	(0.008)	0.002	*		(0.008)	0.003
Sodium	mg/L	553	3	*		481	6
Thallium (b)	mg/L	<0.002		*		<0.002	
Vanadium	mg/L	<0.004		*		<0.004	
Zinc	mg/L	1.38	0.02	*		423	1
<i>Process metals</i>							
Thorium (c)	mg/L	<0.17		*		-0.5 (d)	
Uranium	mg/L	0.39		*		2.37	

Table C.2. Analytical data for tank W-18 (continued)

Parameter measured	Units	W18-L1 Filtered		W18-L1 Supernate		W18-L1 Including sediment	
		Result	+/-	Result	+/-	Result	+/-
<i>Anions</i>							
Bromide	mg/L	*		1200	74	*	
Chloride	mg/L	*		20.2	0.7	*	
Cyanide	mg/L	*		<0.01		*	
Fluoride	mg/L	*		<0.1		*	
Nitrate	mg/L	*		<0.1		*	
Nitrite (e)	mg/L	*		N		*	
Phosphate	mg/L	*		<0.1		*	
Sulfate	mg/L	*		<0.1		*	
Sulfide (e)	mg/L	*		N		*	
<i>Alkalinity</i>							
pH		*		10.2		*	
Acid (H+)	mol/L	*				*	
Base (OH-)	mol/L	*		0.0016	0.0001	*	
Carbonate	mol/L	*		<0.02		*	
Bicarbonate	mol/L	*				*	
<i>Radiochemical screening</i>							
Gross alpha	Bq/mL	<1		<1		<1	
Gross beta	Bq/mL	14		20	1	*	
<i>Beta/gamma emitters</i>							
Am-241	Bq/mL	<0.63		<0.61		<0.53	
Co-60	Bq/mL	<0.01		<0.14		0.64	0.07
Cs-134	Bq/mL	<0.11		<0.10		<0.11	
Cs-137	Bq/mL	12.6	0.3	13.1	0.3	15.5	0.3
Eu-152	Bq/mL	<0.050		<0.50		<0.45	
Eu-154	Bq/mL	<0.22		<1		<0.38	
Eu-155	Bq/mL	<0.29		<0.23		<0.26	
H-3	Bq/mL	*		92.9	5.4	*	
Sr-90	Bq/mL	1.7	0.39	*		*	
Zn-65	Bq/mL	<0.29		<0.12		(f)	

(a) Analyzed by ICP.

(b) Analyzed by GFAA.

(c) Visual scan on ICP.

(d) Estimated concentration based on a visual peak comparison with a 0.500-mg/L thorium standard.

(e) Qualitative analysis. N = not detected.

(f) Possibly some Zn-65 (~0.4 Bq/mL) was observed, but this observation is suspect given the half-life and the known tank history.

Table C.3a. Analytical data for tank WC-5—Part 1

Parameter measured	Units	WC5-Surface		WC5-L1	
		Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>					
Total dissolved solids	mg/mL	*		8.06	
Total solids	mg/mL	*		7.93	
Density	g/mL	1.011	0.001	1.012	0.001
Inorganic carbon	mg/L	*		106	16
Total organic carbon	mg/L	*		17	3
Total carbon	mg/L	*		123	18
<i>EPA target analyte elements (RCRA metals in bold)</i>					
Aluminum	mg/L	*		0.118	0.041
Antimony	mg/L	*		<0.091	
Arsenic	mg/L	*		0.006	0.001
Barium	mg/L	*		0.0173	0.0004
Beryllium	mg/L	*		<0.0004	
Cadmium	mg/L	*		<0.006	
Calcium	mg/L	*		8.05	0.35
Chromium	mg/L	*		0.294	0.018
Cobalt	mg/L	*		<0.009	
Copper	mg/L	*		0.011	0.001
Iron	mg/L	*		0.019	0.001
Lead (a)	mg/L	*		0.250	0.001
Magnesium	mg/L	*		0.048	0.014
Manganese	mg/L	*		<0.0006	
Mercury	mg/L	*		0.218	0.001
Nickel	mg/L	*		<0.015	
Potassium	mg/L	*		5.79	0.31
Selenium	mg/L	*		0.0024	0.0002
Silver	mg/L	*		(<0.006)	
Sodium	mg/L	*		3340	27
Thallium (a)	mg/L	*		<0.002	
Vanadium	mg/L	*		<0.004	
Zinc	mg/L	*		0.138	0.004
<i>Process metals</i>					
Thorium	mg/L	*		<0.17	
Uranium	mg/L	*		<0.35	

Table C.3a. Analytical data for tank WC-5—Part 1 (continued)

Parameter measured	Units	WC5-Surface		WC5-L1	
		Result	+/-	Result	+/-
<i>Anions</i>					
Bromide	mg/L	*		<0.05	
Chloride	mg/L	*		69.7	2.3
Cyanide	mg/L	*		<0.01	
Fluoride (b)	mg/L	*		<10	
Nitrate	mg/L	*		9.17	0.04
Nitrite (c)	mg/L	*		N	
Phosphate	mg/L	*		<0.1	
Sulfate	mg/L	*		17.5	0.2
Sulfide (c)	mg/L	*		N	
<i>Alkalinity</i>					
pH		*		13.1	
Acid (H+)	mol/L	*			
Base (OH-)	mol/L	*		0.15	0.01
Carbonate	mol/L	*		<0.02	
Bicarbonate	mol/L	*			
<i>Radiochemical screening</i>					
Gross alpha	Bq/mL	<1		1.1	0.1
Gross beta	Bq/mL	<1		0.96	0.08
<i>Beta/gamma emitters</i>					
Am-241	Bq/mL	<0.36		<0.37	
Co-60	Bq/mL	<0.13		<0.10	
Cs-134	Bq/mL	<0.11		<0.10	
Cs-137	Bq/mL	0.27	0.01	0.35	0.07
Eu-152	Bq/mL	<0.24		<0.28	
Eu-154	Bq/mL	<0.29		<0.19	
Eu-155	Bq/mL	<0.16		<0.20	
H-3	Bq/mL	*		0.014	0.028
Sr-90	Bq/mL	<1 (d)		<1 (d)	

(a) Analyzed by GFAA.

(b) Peak interference. Estimated concentration based on spike recovery.

(c) Qualitative analysis. N = not detected.

(d) Sr-90 estimated from gross beta analysis.

Table C.3b. Analytical data for tank WC-5—Part 2

Parameter measured	Units	WC5-L2 Supernatant		WC5-L2 Filtered		WC5-L2 Including sediment	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	*		*		*	
Total solids	mg/mL	*		*		*	
Density	g/mL	1.01	0.001	*		*	
Inorganic carbon	mg/L	*		*		*	
Total organic carbon	mg/L	*		*		*	
Total carbon	mg/L	*		*		*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	*		<0.021		1.05	0.03
Antimony	mg/L	*		<0.091		<0.091	
Arsenic	mg/L	*		*		*	
Barium	mg/L	*		0.0156	0.0002	0.080	0.001
Beryllium	mg/L	*		<0.0004		<0.0004	
Cadmium	mg/L	*		<0.006		<0.006	
Calcium	mg/L	*		5.16	0.13	18.8	0.1
Chromium	mg/L	*		0.309	0.005	0.829	0.011
Cobalt	mg/L	*		<0.009		<0.009	
Copper	mg/L	*		0.010	0.003	0.055	0.001
Iron	mg/L	*		0.091	0.004	9.13	0.02
Lead (a)	mg/L	*		0.156	0.001	0.486	0.034
Magnesium	mg/L	*		<0.043		8.19	0.06
Manganese	mg/L	*		<0.0006		0.035	0.002
Mercury	mg/L	*		0.223	0.012	0.441	0.036
Nickel	mg/L	*		<0.015		0.115	0.010
Potassium	mg/L	*		5.95	0.14	6.24	0.06
Selenium	mg/L	*		*		*	
Silver	mg/L	*		(<0.006)		(0.009)	0.004
Sodium	mg/L	*		3320	8	3400	10
Thallium	mg/L	*		*		*	
Vanadium	mg/L	*		<0.004		<0.004	
Zinc	mg/L	*		0.093	0.002	0.220	0.004
<i>Process metals</i>							
Thorium	mg/L	*		<0.17		0.548	0.076
Uranium	mg/L	*		0.46	0.22	0.56	0.30

Table C.3b. Analytical data for tank WC-5—Part 2 (continued)

Parameter measured	Units	WC5-L2 Supernate		WC5-L2 Filtered		WC5-L2 Including sediment	
		Result	+/-	Result	+/-	Result	+/-
<i>Anions</i>							
Bromide	mg/L	*		*		*	
Chloride	mg/L	*		*		*	
Cyanide	mg/L	*		*		*	
Fluoride	mg/L	*		*		*	
Nitrate	mg/L	*		*		*	
Nitrite	mg/L	*		*		*	
Phosphate	mg/L	*		*		*	
Sulfate	mg/L	*		*		*	
Sulfide	mg/L	*		*		*	
<i>Alkalinity</i>							
pH		13.0		*		*	
Acid (H ⁺)	mol/L			*		*	
Base (OH ⁻)	mol/L	0.16		*		*	
Carbonate	mol/L	*		*		*	
Bicarbonate	mol/L	*		*		*	
<i>Radiochemical screening</i>							
Gross alpha	Bq/mL	<1		<1		141	2
Gross beta	Bq/mL	1.7	0.1	<1		*	
<i>Beta/gamma emitters</i>							
Am-241	Bq/mL	<0.29		<0.35		67.7	1.0
Co-60	Bq/mL	<0.10		<0.08		<0.12	
Cs-134	Bq/mL	<0.09		<0.13		<0.10	
Cs-137	Bq/mL	0.42	0.06	0.24	0.05	0.45	0.05
Eu-152	Bq/mL	<0.26		<0.23		<0.20	
Eu-154	Bq/mL	<0.11		<0.25		<0.26	
Eu-155	Bq/mL	<0.17		<0.17		<0.16	
H-3	Bq/mL	*		*		*	
Sr-90	Bq/mL	<1 (b)		<1 (b)		*	

(a) Analyzed by GFAA.

(b) Sr-90 estimated from gross beta analysis.

Table C.4a. Analytical data for tank WC-6—Part 1

Parameter measured	Units	WC6-Surface		WC6-L1	
		Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>					
Total dissolved solids	mg/mL	*		6.19	
Total solids	mg/mL	*		5.88	
Density	g/mL	1.008	0.001	1.010	0.001
Inorganic carbon	mg/L	*		105	16
Total organic carbon	mg/L	*		20	3
Total carbon	mg/L	*		125	19
<i>EPA target analyte elements (RCRA metals in bold)</i>					
Aluminum	mg/L	*		0.0888	0.0345
Antimony	mg/L	*		<0.091	
Arsenic	mg/L	*		<0.002	
Barium	mg/L	*		0.0116	0.0009
Beryllium	mg/L	*		<0.0004	
Cadmium	mg/L	*		<0.006	
Calcium	mg/L	*		20.4	0.6
Chromium	mg/L	*		0.018	0.005
Cobalt	mg/L	*		<0.009	
Copper	mg/L	*		0.0154	0.0026
Iron	mg/L	*		0.0532	0.0031
Lead (a)	mg/L	*		0.0072	0.0001
Magnesium	mg/L	*		0.369	0.004
Manganese	mg/L	*		0.0109	0.0007
Mercury	mg/L	*		<0.0002	
Nickel	mg/L	*		<0.015	
Potassium	mg/L	*		9.42	0.33
Selenium	mg/L	*		<0.002	
Silver	mg/L	*		(<0.006)	
Sodium	mg/L	*		2520	24
Thallium (a)	mg/L	*		<0.002	
Vanadium	mg/L	*		<0.004	
Zinc	mg/L	*		0.192	0.022
<i>Process metals</i>					
Thorium	mg/L	*		<0.17	
Uranium	mg/L	*		<0.35	

Table C.4a. Analytical data for tank WC-6—Part 1 (continued)

Parameter measured	Units	WC6-Surface		WC6-L1	
		Result	+/-	Result	+/-
<i>Anions</i>					
Bromide	mg/L	*		<0.05	
Chloride	mg/L	*		49.7	0.1
Cyanide	mg/L	*		<0.01	
Fluoride (b)	mg/L	*		<10	
Nitrate	mg/L	*		15.7	0.5
Nitrite (c)	mg/L	*		N	
Phosphate	mg/L	*		<0.1	
Sulfate	mg/L	*		13.4	0.5
Sulfide (c)	mg/L	*		N	
<i>Alkalinity</i>					
pH		*		13.1	
Acid (H+)	mol/L	*			
Base (OH-)	mol/L	*		0.11	0.02
Carbonate	mol/L	*		<0.02	
Bicarbonate	mol/L	*			
<i>Radiochemical screening</i>					
Gross alpha	Bq/mL	<1		<1	
Gross beta	Bq/mL	<1		<1 (d)	
<i>Beta/gamma emitters</i>					
Am-241	Bq/mL	<0.34		<0.39	
Co-60	Bq/mL	<0.13		<0.10	
Cs-134	Bq/mL	<0.12		<0.08	
Cs-137	Bq/mL	0.14	0.04	0.36	0.06
Eu-152	Bq/mL	<0.24		<0.23	
Eu-154	Bq/mL	<0.27		<0.29	
Eu-155	Bq/mL	<0.16		<0.19	
H-3	Bq/mL	*		0.011	0.028
Sr-90	Bq/mL	<1 (e)		<1 (e)	

(a) Analyzed by GFAA.

(b) Peak interference. Estimated concentration based on spike recovery.

(c) Qualitative analysis. N = not detected.

(d) Independent gross beta check of 1.1 Bq/mL.

(e) Sr-90 estimated from gross beta analysis.

Table C.4b. Analytical data for tank WC-6—Part 2

Parameter measured	Units	WC6-L2 Supernate		WC6-L2 Filtered		WC6-L2 Including sediment	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	*		*		*	
Total solids	mg/mL	*		*		*	
Density	g/mL	1.011	0.001	*		*	
Inorganic carbon	mg/L	*		*		*	
Total organic carbon	mg/L	*		*		*	
Total carbon	mg/L	*		*		*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	*		0.246	0.018	3.36	0.25
Antimony	mg/L	*		<0.091		<0.091	
Arsenic	mg/L	*		*		*	
Barium	mg/L	*		0.0015	0.0004	0.0405	0.0004
Beryllium	mg/L	*		<0.0004		0.0068	0.0002
Cadmium	mg/L	*		<0.006		0.036	0.005
Calcium	mg/L	*		9.95	0.81	30.9	0.8
Chromium	mg/L	*		0.0349	0.0024	0.0824	0.0055
Cobalt	mg/L	*		<0.009		<0.009	
Copper	mg/L	*		0.0209	0.0007	0.0129	0.004
Iron	mg/L	*		0.0669	0.0031	3.94	0.08
Lead (a)	mg/L	*		0.0079	0.0004	0.0321	0.0002
Magnesium	mg/L	*		<0.043		6.33	0.19
Manganese	mg/L	*		0.0073	0.0009	0.897	0.024
Mercury	mg/L	*		0.0015	0.0026	0.0047	0.0022
Nickel	mg/L	*		<0.015		0.048	0.004
Potassium	mg/L	*		8.24	0.25	9.25	0.19
Selenium	mg/L	*		*		*	
Silver	mg/L	*		(<0.006)		(0.0197)	0.0026
Sodium	mg/L	*		4130	27	4230	13
Thallium	mg/L	*		*		*	
Vanadium	mg/L	*		<0.004		<0.004	
Zinc	mg/L	*		0.34	0.026	0.974	0.024
<i>Process metals</i>							
Thorium	mg/L	*		<0.17		29.6	0.5
Uranium	mg/L	*		0.55	0.862	45.1	0.1

Table C.4b. Analytical data for tank WC-6—Part 2 (continued)

Parameter measured	Units	WC6-L2 Supernate		WC6-L2 Filtered		WC6-L2 Including sediment	
		Result	+/-	Result	+/-	Result	+/-
<i>Anions</i>							
Bromide	mg/L	*		*		*	
Chloride	mg/L	*		*		*	
Cyanide	mg/L	*		*		*	
Fluoride	mg/L	*		*		*	
Nitrate	mg/L	*		*		*	
Nitrite	mg/L	*		*		*	
Phosphate	mg/L	*		*		*	
Sulfate	mg/L	*		*		*	
Sulfide	mg/L	*		*		*	
<i>Alkalinity</i>							
pH		13.1		*		*	
Acid (H+)	mol/L			*		*	
Base (OH-)	mol/L	0.19		*		*	
Carbonate	mol/L	*		*		*	
Bicarbonate	mol/L	*		*		*	
<i>Radiochemical screening</i>							
Gross alpha	Bq/mL	<1		<1		1670	17.1
Gross beta	Bq/mL	<1		<1		*	
<i>Beta/gamma emitters</i>							
Am-241	Bq/mL	<0.35		<0.34		662	3
Co-60	Bq/mL	<0.13		<0.14		<0.12	
Cs-134	Bq/mL	<0.94		<0.074		<0.082	
Cs-137	Bq/mL	0.097	0.05	0.116	0.045	0.69	0.08
Eu-152	Bq/mL	<0.23		<0.20		<0.30	
Eu-154	Bq/mL	<0.19		<0.40		<0.14	
Eu-155	Bq/mL	<0.18		<0.18		<0.31	
H-3	Bq/mL	*		*		*	
Sr-90	Bq/mL	<1 (b)		<1 (b)		*	

(a) Analyzed by GFAA.

(b) Sr-90 estimated from gross beta analysis.

Table C.5. Analytical data for tank WC-8

Parameter measured	Units	WC8-Surface		WC8-L1		WC8-L2	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	*		12.2		*	
Total solids	mg/mL	*		12.1		*	
Density	g/mL	1.013	0.002	1.013	0.001	1.013	0.001
Inorganic carbon	mg/L	*		63.6	9.5	*	
Total organic carbon	mg/L	*		15.4	2.3	*	
Total carbon	mg/L	*		79.0	12	*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	*		0.128	0.032	<0.021	
Antimony	mg/L	*		<0.091		<0.091	
Arsenic	mg/L	*		<0.002		*	
Barium	mg/L	*		0.0053	0.0002	0.0064	0.0007
Beryllium	mg/L	*		<0.0004		<0.0004	
Cadmium	mg/L	*		<0.006		<0.006	
Calcium	mg/L	*		11.6	0.2	11.5	0.2
Chromium	mg/L	*		0.0223	0.0031	0.0207	0.0042
Cobalt	mg/L	*		<0.009		<0.009	0.001
Copper	mg/L	*		0.0222	0.0035	0.0201	0.0013
Iron	mg/L	*		0.0326	0.0011	0.0673	0.0029
Lead (a)	mg/L	*		0.229	0.001	0.215	0.003
Magnesium	mg/L	*		<0.043		<0.043	
Manganese	mg/L	*		<0.0006		<0.0006	
Mercury	mg/L	*		0.0018	0.0001	0.0022	0.0001
Nickel	mg/L	*		<0.015		<0.015	
Potassium	mg/L	*		7.57	0.21	7.60	0.27
Selenium	mg/L	*		<0.002		*	
Silver	mg/L	*		(<0.006)		(<0.006)	
Sodium	mg/L	*		5490	32	5460	37
Thallium (a)	mg/L	*		<0.002		*	
Vanadium	mg/L	*		<0.004		<0.004	
Zinc	mg/L	*		0.280	0.015	0.305	0.008
<i>Process metals</i>							
Thorium	mg/L	*		<0.17		<0.17	
Uranium	mg/L	*		1.37	0.33	3.17	0.15

Table C.5. Analytical data for tank WC-8 (continued)

Parameter measured	Units	WC8-Surface		WC8-L1		WC8-L2	
		Result	+/-	Result	+/-	Result	+/-
Anions							
Bromide	mg/L	*		1.07		*	
Chloride	mg/L	*		107	1	*	
Cyanide	mg/L	*		<0.01		*	
Fluoride (b)	mg/L	*		<10		*	
Nitrate	mg/L	*		26.9	1.2	*	
Nitrite (c)	mg/L	*		N		*	
Phosphate	mg/L	*		<0.1		*	
Sulfate	mg/L	*		14.9	1.2	*	
Sulfide (c)	mg/L	*		N		*	
Alkalinity							
pH		*		13.4		13.2	
Acid (H+)	mol/L	*					
Base (OH-)	mol/L	*		0.26	0.01	0.26	0.01
Carbonate	mol/L	*		<0.02		<0.02	
Bicarbonate	mol/L	*					
Radiochemical screening							
Gross alpha	Bq/mL	<1		<1		<1	
Gross beta	Bq/mL	6.4	0.2	6.3	0.2	6.8	0.2
Beta/gamma emitters							
Am-241	Bq/mL	<0.45		<0.45		<0.37	
Co-60	Bq/mL	<0.13		<0.15		<0.073	
Cs-134	Bq/mL	<0.094		<0.11		<0.064	
Cs-137	Bq/mL	2.83	0.12	2.50	0.13	2.81	0.14
Eu-152	Bq/mL	<0.30		<0.34		<0.15	
Eu-154	Bq/mL	<0.27		<0.31		<0.27	
Eu-155	Bq/mL	<0.20		<0.20		<0.15	
H-3	Bq/mL	*		0.256	0.034	*	
Sr-90	Bq/mL	*		1.9	0.4	*	

(a) Analyzed by GFAA.

(b) Peak interference. Estimated concentration based on spike recovery.

(c) Qualitative analysis. N = not detected.

Table C.6a. Analytical data for tank WC-11—Part 1

Parameter measured	Units	WC11-L1	
		Result	+/-
<i>Physical properties and miscellaneous data</i>			
Total dissolved solids	mg/mL	1.68	
Total solids	mg/mL	*	
Density	g/mL	1.006	0.001
Inorganic carbon	mg/L	97.6	
Total organic carbon	mg/L	182	
Total carbon	mg/L	84.4	
<i>EPA target analyte elements (RCRA metals in bold)</i>			
Aluminum	mg/L	0.314	0.026
Antimony	mg/L	<0.091	
Arsenic	mg/L	<0.002	
Barium	mg/L	0.0243	0.0002
Beryllium	mg/L	0.0007	0.0001
Cadmium	mg/L	<0.006	
Calcium	mg/L	4.72	0.06
Chromium	mg/L	0.055	0.013
Cobalt	mg/L	<0.009	
Copper	mg/L	0.063	0.004
Iron	mg/L	0.057	0.001
Lead (a)	mg/L	0.234	0.117
Magnesium	mg/L	0.198	0.012
Manganese	mg/L	0.079	0.002
Mercury	mg/L	0.0046	0.0018
Nickel	mg/L	0.129	0.004
Potassium	mg/L	15.9	0.2
Selenium	mg/L	0.002	0.01
Silver	mg/L	(<0.006)	
Sodium	mg/L	495	2
Thallium (b)	mg/L	0.002	0.001
Vanadium	mg/L	<0.004	
Zinc	mg/L	0.0233	0.0015
<i>Process metals</i>			
Thorium	mg/L	<0.17	
Uranium	mg/L	<0.35	

Table C.6a. Analytical data for tank WC-11—Part 1 (continued)

Parameter measured	Units	WC11-L1	
		Result	+/-
<i>Anions</i>			
Bromide	mg/L	<0.05	
Chloride	mg/L	14.7	0.8
Cyanide	mg/L	0.012	0.002
Fluoride	mg/L	<0.05	
Nitrate	mg/L	51.7	
Nitrite (c)	mg/L	Y	
Phosphate	mg/L	<0.08	
Sulfate	mg/L	19.7	3.8
Sulfide (c)	mg/L	N	
<i>Alkalinity</i>			
pH		11.4	
Acid (H+)	mol/L		
Base (OH-)	mol/L	0.008	0.002
Carbonate	mol/L	<0.01	
Bicarbonate	mol/L		
<i>Radiochemical screening</i>			
Gross alpha (RMAL)	Bq/mL	<121 (d)	
Gross alpha (TAL)	Bq/mL	0.32 (e)	0.05
Gross beta	Bq/mL	50,000	282
<i>Beta/gamma emitters</i>			
Am-241	Bq/mL	<189	
Co-60	Bq/mL	110	7
Cs-134	Bq/mL	<12	
Cs-137	Bq/mL	47,700	104
Eu-152	Bq/mL	<160	
Eu-154	Bq/mL	<14	
Eu-155	Bq/mL	<84	
H-3	Bq/mL	30.8	0.2
Sr-90	Bq/mL	9.3	2.5

(a) Analyzed by ICP.

(b) Analyzed by GFAA.

(c) Qualitative analysis. N = not detected; Y = detected.

(d) Gross alpha was analyzed without a cleanup step, and the dilution factor is included in the reported value.

(e) Analyzed at Transuranium Analytical Laboratory (TAL) after TRU-Spec separation.

Table C.6b. Analytical data for tank WC-11—Part 2

Parameter measured	Units	WC11-L2 Supernate		WC11-L2 Filtered		WC11-L2 Including sediment	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	*		*		*	
Total solids	mg/mL	*		*		*	
Density	g/mL	1.005	0.001	*		*	
Inorganic carbon	mg/L	*		*		*	
Total organic carbon	mg/L	*		*		*	
Total carbon	mg/L	*		*		*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	*		0.234	0.043	10.9	0.1
Antimony	mg/L	*		<0.091		<0.091	
Arsenic	mg/L	*		*		*	
Barium	mg/L	*		0.061	0.001	19.9	0.2
Beryllium	mg/L	*		<0.0004		0.122	0.001
Cadmium	mg/L	*		0.016	0.005	0.216	0.003
Calcium	mg/L	*		5.02	0.13	976	10
Chromium	mg/L	*		0.129	0.004	2.57	0.03
Cobalt	mg/L	*		<0.009		0.108	0.018
Copper	mg/L	*		0.071	0.002	1.28	0.01
Iron	mg/L	*		0.225	0.006	44.4	0.5
Lead (a)	mg/L	*		0.357	0.085	4.63	0.11
Magnesium	mg/L	*		1.81	0.05	270	4
Manganese	mg/L	*		0.183	0.004	26.1	0.4
Mercury	mg/L	*		0.018	0.017	2.01	0.04
Nickel	mg/L	*		0.314	0.016	6.52	0.08
Potassium	mg/L	*		20.4	0.5	21.80	0.3
Selenium	mg/L	*		*		*	
Silver	mg/L	*		(<0.006)		(0.036)	0.002
Sodium	mg/L	*		849	6	846	3
Thallium	mg/L	*		*		*	
Vanadium	mg/L	*		<0.004		0.018	0.001
Zinc	mg/L	*		0.057	0.005	6.10	0.05
<i>Process metals</i>							
Thorium	mg/L	*		<0.17		3.37	0.17
Uranium	mg/L	*		0.91	0.20	3.81	0.22

Table C.6b. Analytical data for tank WC-11—Part 2 (continued)

Parameter measured	Units	WC11-L2 Supernate		WC11-L2 Filtered		WC11-L2 Including sediment	
		Result	+/-	Result	+/-	Result	+/-
<i>Anions</i>							
Bromide	mg/L	*		*		*	
Chloride	mg/L	*		*		*	
Cyanide	mg/L	*		*		*	
Fluoride	mg/L	*		*		*	
Nitrate	mg/L	*		*		*	
Nitrite	mg/L	*		*		*	
Phosphate	mg/L	*		*		*	
Sulfate	mg/L	*		*		*	
Sulfide	mg/L	*		*		*	
<i>Alkalinity</i>							
pH		11.2		*		*	
Acid (H+)	mol/L	.		*		*	
Base (OH-)	mol/L	0.017		*		*	
Carbonate	mol/L	<0.02		*		*	
Bicarbonate	mol/L			*		*	
<i>Radiochemical screening</i>							
Gross alpha	Bq/mL	12	1.5	1.45	0.53	364	8
Gross beta	Bq/mL	*		*		*	
<i>Beta/gamma emitters</i>							
Am-241	Bq/mL	<263		<284		<300	
Co-60	Bq/mL	125	11	150	12	518	25
Cs-134	Bq/mL	*		*		<32	
Cs-137	Bq/mL	55,000	168	58,000	180	63,300	180
Eu-152	Bq/mL	<240		<260		1,660	111
Eu-154	Bq/mL	<35		<18.7		825	49
Eu-155	Bq/mL	<117		<130		249	64
H-3	Bq/mL	*		*		*	
Sr-90	Bq/mL	*		*		*	

(a) Analyzed by ICP.

Table C.7. Analytical data for tank WC-12

Parameter measured	Units	WC12-L1 (a)		WC12-L3-B Filtered (b)		WC12-L3-A Including solids (b), (c)	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	6.44		4.93		*	
Total solids	mg/mL	6.45		*		*	
Density	g/mL	1.008	0.001	1.006	0.001	*	
Inorganic carbon	mg/L	1110	166	*		*	
Total organic carbon	mg/L	<10		*		*	
Total carbon	mg/L	1050	158	*		*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	5.78	0.29	3.21	0.08	69.4	1.1
Antimony	mg/L	<0.091		<0.062		<0.062	
Arsenic	mg/L	<0.002		<0.006		<0.006	
Barium	mg/L	0.0187	0.0007	0.094	0.001	8.92	0.09
Beryllium	mg/L	<0.0004		<0.0005		0.263	0.002
Cadmium	mg/L	0.067	0.004	0.058	0.003	0.551	0.002
Calcium	mg/L	2.82	0.06	8.02	0.03	431	2
Chromium	mg/L	0.252	0.004	0.291	0.005	5.62	0.06
Cobalt	mg/L	<0.009		0.0089	0.0094	0.209	0.018
Copper	mg/L	0.384	0.007	0.362	0.005	3.99	0.05
Iron	mg/L	0.137	0.006	2.27	0.04	224	1
Lead (d)	mg/L	1.92	0.11	0.75	0.48	26.1	0.6
Magnesium	mg/L	0.557	0.015	2.16	0.08	108	1
Manganese	mg/L	0.865	0.018	2.57	0.06	214	3
Mercury	mg/L	0.0018	0.0009	0.064	0.010	4.32	0.18
Nickel	mg/L	0.363	0.014	0.74	0.04	12.8	0.1
Potassium	mg/L	41.5	0.7	58.4	1.5	62.0	0.7
Selenium	mg/L	0.003	0.006	<0.006		<0.006	
Silver	mg/L	(<0.006)		(<0.009)		(0.070)	0.001
Sodium	mg/L	2,660	17	2,170	8	2,250	28
Thallium	mg/L	<0.002 (e)		<0.40 (d)		<0.40 (d)	
Vanadium	mg/L	0.013	0.003	<0.005		0.103	0.004
Zinc	mg/L	0.235	0.010	0.184	0.003	8.83	0.10
<i>Process metals</i>							
Thorium	mg/L	<0.17		0.406	0.006	21.6	0.3
Uranium	mg/L	2.60	0.28	6.10	0.30	8.69	0.38

Table C.7. Analytical data for tank WC-12 (continued)

Parameter measured	Units	WC12-L1 (a)		WC12-L3-B Filtered (b)		WC12-L3-A Including solids (b), (c)	
		Result	+/-	Result	+/-	Result	+/-
Anions							
Bromide	mg/L	<0.05		*		*	
Chloride	mg/L	35.1	2.6	*		*	
Cyanide	mg/L	0.016	0.002	*		*	
Fluoride	mg/L	<0.05		*		*	
Nitrate	mg/L	44.7	0.2	*		*	
Nitrite (f)	mg/L	Y		*		*	
Phosphate	mg/L	<0.08		*		*	
Sulfate	mg/L	9.29	3.03	*		*	
Sulfide (f)	mg/L	N		*		*	
Alkalinity							
pH		10.1		9.8		*	
Acid (H+)	mol/L			*		*	
Base (OH-)	mol/L	0.039	0.001	*		*	
Carbonate	mol/L	<0.02		*		*	
Bicarbonate	mol/L			*		*	
Radiochemical screening							
Gross alpha (RMAL)	Bq/mL	<121 (g)		<26 (g)		<13,000 (g)	
Gross alpha (TAL)	Bq/mL	0.63 (h)	0.07	*		1,800 (h)	180
Gross beta	Bq/mL	56,000	297	37,000	113	726,000	11,200
Beta/gamma emitters							
Am-241	Bq/mL	<190		*		<301	
Co-60	Bq/mL	263	10	925	39	45,600	2,670
Cs-134	Bq/mL	<13		*		*	
Cs-137	Bq/mL	50,300	106	27,600	133	35,900	2,980
Eu-152	Bq/mL	<170		4,500	142	380,000	9,310
Eu-154	Bq/mL	<17		3,420	97	302,000	10,100
Eu-155	Bq/mL	<87		838	80	70,400	6,820
H-3	Bq/mL	28.4	0.2	*		*	
Sr-90	Bq/mL	10.6	2.6	*		1,430	277

Table C.7. Analytical data for tank WC-12 (continued)

Parameter measured	Units	WC12-L1 (a)		WC12-L3-B Filtered (b)		WC12-L3-A Including solids (b), (c)	
		Result	+/-	Result	+/-	Result	+/-
<i>Alpha emitters (by alpha spectrometry) (h)</i>							
Cm-244	Bq/mL					1,600	160
Pu-238	Bq/mL					150	15
Pu-239/Pu-240	Bq/mL					240	24
U-233/Th-229/Np-237	Bq/mL					2.3	0.2

(a) Sample collected November 4, 1992.

(b) Sample collected March 17, 1993.

(c) The data is for the liquid plus the solids which dissolved during microwave assisted digestion in ~2.6M HNO₃. The residue was not analyzed.

(d) Analyzed by ICP.

(e) Analyzed by GFAA.

(f) Qualitative analysis. N = not detected; Y = detected.

(g) Gross alpha was analyzed without a cleanup step, and the dilution factor is included in the reported value.

(h) Analyzed at Transuranium Analytical Laboratory (TAL) after TRU•Spec separation.

Table C.8. Analytical data for liquid samples from tank WC-13

Parameter measured	Units	WC13-Surface Aqueous (a), (b)		WC13-L1 (a)		WC13S-L Supernate (c)	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	*		18.1		*	
Total solids	mg/mL	*		18.4		*	
Density	g/mL	*		1.020	0.001	*	
Inorganic carbon	mg/L	*		168	25	*	
Total organic carbon	mg/L	*		174	26	*	
Total carbon	mg/L	*		342	51	*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	*		11.6	0.4	15.8	0.4
Antimony	mg/L	*		<0.091		<0.091	
Arsenic	mg/L	*		0.0536	0.0062	*	
Barium	mg/L	*		0.00121	0.00374	0.0384	0.0007
Beryllium	mg/L	*		<0.0004		0.00655	0.00045
Cadmium	mg/L	*		<0.006		<0.006	
Calcium	mg/L	*		1.89	0.09	0.832	0.082
Chromium	mg/L	*		2.74	0.09	3.62	0.16
Cobalt	mg/L	*		<0.009		0.0184	0.0093
Copper	mg/L	*		2.82	0.13	2.95	0.07
Iron	mg/L	*		0.70	0.07	0.52	0.02
Lead (d)	mg/L	*		<0.26		<0.27	
Magnesium	mg/L	*		<0.043		<0.030	
Manganese	mg/L	*		0.00902	0.00154	0.0083	0.0011
Mercury	mg/L	*		2.73	0.53	0.327	0.007
Nickel	mg/L	*		3.58	0.09	2.42	0.10
Potassium	mg/L	*		48.1	0.5	31.7	1.1
Selenium	mg/L	*		0.0151	0.0240	*	
Silver	mg/L	*		(<0.006)		(0.016)	0.005
Sodium	mg/L	*		8160	48	9560	180
Thallium (e)	mg/L	*		0.0079	0.0048	*	
Vanadium	mg/L	*		1.57	0.07	1.01	0.07
Zinc	mg/L	*		0.454	0.010	9.41	0.60
<i>Process metals</i>							
Thorium	mg/L	*		0.440	0.036	0.359	0.043
Uranium	mg/L	*		14.4	0.3	9.96	0.08

Table C.8. Analytical data for liquid samples from tank WC-13 (continued)

Parameter measured	Units	WC13-Surface Aqueous (a), (b)		WC13-L1 (a)		WC13S-L Supernate (c)	
		Result	+/-	Result	+/-	Result	+/-
<i>Anions</i>							
Bromide	mg/L	*		<0.05		*	
Chloride	mg/L	*		171	3	*	
Cyanide	mg/L	*		0.248	0.002	*	
Fluoride	mg/L	*		(g)		*	
Nitrate	mg/L	*		84.2	8.9	*	
Nitrite (f)	mg/L	*		Y		*	
Phosphate	mg/L	*		2230	10	*	
Sulfate	mg/L	*		584	8	*	
Sulfide (f)	mg/L	*		N		*	
<i>Alkalinity</i>							
pH		*		13.2		13.2	
Acid (H+)	mol/L	*				*	
Base (OH-)	mol/L	*		0.29	0.01	*	
Carbonate	mol/L	*		<0.02		*	
Bicarbonate	mol/L	*				*	
<i>Radiochemical screening</i>							
Gross alpha (RMAL)	Bq/mL	5.20	1.47	<1		<1	
Gross alpha (TAL)	Bq/mL	*		0.41 (h)	0.04	*	
Gross beta	Bq/mL	4800	37	4980	38	4900	18
<i>Betalgamma emitters</i>							
Am-241	Bq/mL	<10.0		<10.1		<24	
Co-60	Bq/mL	15.9	0.5	13.3	0.4	85.7	2.8
Cs-134	Bq/mL	<1.1		<1.0		11.9	1.2
Cs-137	Bq/mL	5360	6	5540	6	4580	15
Eu-152	Bq/mL	54.2	3.6	<9.1		<18.4	
Eu-154	Bq/mL	34.3	1.2	<1.2		<3.53	
Eu-155	Bq/mL	<4.8		<4.7		<10.6	
H-3	Bq/mL	*		4.21	0.86	*	
Sr-90	Bq/mL	*		<2		*	

(a) Sample collected November 12, 1992.

(b) WC13-Surface had an organic phase and an aqueous phase. The aqueous phase was analyzed.

(c) Sample WC13S-L was collected March 2, 1993. This sample was the aqueous supernate over the sludge, a vertical column of tank liquid. The sample was filtered before analysis.

(d) Analyzed by ICP.

(e) Analyzed by GFAA.

(f) Qualitative analysis. N = not detected; Y = detected.

(g) Peak interference, results inconclusive.

(h) Analyzed after TRU•Spec separation.

Table C.9. Analytical data for liquid samples from tank WC-14

Parameter measured	Units	WC14-Surface (a)		WC14-L1 (a)		WC14S-L Supernate (b)	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	*		14.0		*	
Total solids	mg/mL	*		14.4		*	
Density	g/mL	1.014	0.001	1.013	0.001	*	
Inorganic carbon	mg/L	*		12.1	1.8	*	
Total organic carbon	mg/L	*		487	73	*	
Total carbon	mg/L	*		499	75	*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	*		1.45	0.04	*	
Antimony	mg/L	*		<0.091		*	
Arsenic	mg/L	*		<0.002		*	
Barium	mg/L	*		0.00616	0.00066	*	
Beryllium	mg/L	*		0.0573	0.0013	*	
Cadmium	mg/L	*		0.147	0.003	*	
Calcium	mg/L	*		51.7	0.6	*	
Chromium	mg/L	*		0.438	0.010	*	
Cobalt	mg/L	*		0.187	0.007	*	
Copper	mg/L	*		1.71	0.03	*	
Iron	mg/L	*		0.25	0.006	*	
Lead (c)	mg/L	*		<0.26		*	
Magnesium	mg/L	*		42.5	0.4	*	
Manganese	mg/L	*		1.07	0.02	*	
Mercury	mg/L	*		0.0325	0.0077	*	
Nickel	mg/L	*		7.82	0.15	*	
Potassium	mg/L	*		31.9	0.9	*	
Selenium	mg/L	*		<0.002		*	
Silver	mg/L	*		(0.0321)	0.0051	*	
Sodium	mg/L	*		4,340	14	*	
Thallium (d)	mg/L	*		<0.002		*	
Vanadium	mg/L	*		<0.004		*	
Zinc	mg/L	*		0.968	0.026	*	
<i>Process metals</i>							
Thorium	mg/L	*		1.46	0.06	*	
Uranium	mg/L	*		48.5	1.1	*	

Table C.9. Analytical data for liquid samples from tank WC-14 (continued)

Parameter measured	Units	WC14-Surface (a)		WC14-L1 (a)		WC14S-L Supernate (b)	
		Result	+/-	Result	+/-	Result	+/-
<i>Anions</i>							
Bromide	mg/L	*		8.89	1.01	*	
Chloride	mg/L	*		1,960	1	*	
Cyanide	mg/L	*		0.057	0.002	*	
Fluoride	mg/L	*		34.9	0.4	*	
Nitrate	mg/L	*		5,310	4	*	
Nitrite (e)	mg/L	*		Y		*	
Phosphate	mg/L	*		<0.08		*	
Sulfate	mg/L	*		1,770	4	*	
Sulfide (e)	mg/L	*		N		*	
<i>Alkalinity</i>							
pH		*		7.4		12.6	
Acid (H+)	mol/L	*				*	
Base (OH-)	mol/L	*				*	
Carbonate	mol/L	*				*	
Bicarbonate	mol/L	*				*	
<i>Radiochemical screening</i>							
Gross alpha (RMAL)	Bq/mL	29	16	20	4	*	
Gross alpha (TAL)	Bq/mL	*		15.2 (f)	1.5	*	
Gross beta	Bq/mL	58,000	2,121	55,100	417	*	
<i>Beta/gamma emitters</i>							
Am-241	Bq/mL	<140		<140		*	
Co-60	Bq/mL	26.5	3.1	31.0	3.0	*	
Cs-134	Bq/mL	1,090	13	1,130	13	*	
Cs-137	Bq/mL	53,500	78	54,800	79	*	
Eu-152	Bq/mL	<128		<132		*	
Eu-154	Bq/mL	<12.8		<12.8		*	
Eu-155	Bq/mL	<66.6		<67.9		*	
H-3	Bq/mL	*		30.6	0.2	*	
Sr-90	Bq/mL	*		664	16	*	

Table C.9. Analytical data for liquid samples from tank WC-14 (continued)

Parameter measured	Units	WC14-Surface (a)		WC14-L1 (a)		WC14S-L Supernate (b)	
		Result	+/-	Result	+/-	Result	+/-
<i>Alpha emitters (by alpha spectrometry) (d)</i>							
Am-241	Bq/mL	*		3.8	0.4	*	
Cm-244	Bq/mL	*		2.9	0.3	*	
Np-237	Bq/mL	*		4.5	0.4	*	
Pu-239/Pu-240	Bq/mL	*		2.3	0.2	*	
Th-228	Bq/mL	*		0.34	0.03	*	
U-233/U-234	Bq/mL	*		0.93	0.09	*	
U-238	Bq/mL	*		0.43	0.04	*	

(a) Sample collected November 12, 1992.

(b) Sample collected March 24, 1993.

(c) Analyzed by ICP.

(d) Analyzed by GFAA.

(e) Qualitative analysis. N = not detected; Y = detected.

(f) Alpha emitters were analyzed after TRU•Spec separation.

Table C.10. Analytical data for sludge samples from tanks WC-13 and WC-14

Parameter measured	Units	WC13-S Sludge (a)		WC14-S Sludge (b)	
		Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>					
Bulk density	g/mL	1.062		1.094	
Liquid density	g/mL	1.024		1.026	
Total solids	%	10.6		14.7	
Dissolved solids	%	2.3		2.0	
Liquid mass	g/g	0.917		0.873	
Undissolved solids	%	8.3		12.7	
Floc density	g/mL	1.805		2.020	
Inorganic carbon	%	0.17	0.02	0.45	0.20
Total organic carbon	%	4.14	0.40	1.08	0.05
Total carbon	%	4.20	0.34	1.53	0.25
<i>EPA target analyte elements (RCRA metals in bold)</i>					
Aluminum	mg/kg	355	6	648 (d)	3
Antimony	mg/kg	2.84	0.73	<.062 (d)	
Arsenic	mg/kg	<0.006		<0.006	
Barium	mg/kg	18.8	0.6	64.2	0.5
Beryllium	mg/kg	0.804	0.072	3.1 (d)	0.01
Cadmium	mg/kg	1.30	0.07	142	1
Calcium	mg/kg	3,830	30	2,560	13
Chromium	mg/kg	285	4	360	2
Cobalt	mg/kg	4.95	0.33	58.4	0.7
Copper	mg/kg	84.4	1.4	68.5 (d)	0.2
Iron	mg/kg	3,420	27	6,170	12
Lead (c)	mg/kg	34.7	2.4	575	18
Magnesium	mg/kg	1,140	19	784 (d)	5
Manganese	mg/kg	111	2.5	74.0	1.4
Mercury	mg/kg	393 (c)	1	8.48	0.56
Nickel	mg/kg	243	7	1,330	7
Potassium	mg/kg	50.1	1.4	175	2
Selenium	mg/kg	<0.006		<0.006	
Silver	mg/kg	(18.1) (e)	1.6	(<.009) (d)	
Sodium	mg/kg	11,700	410	13,000	68
Thallium (c)	mg/kg	<0.22		(f)	
Vanadium	mg/kg	7.84	0.26	6.89 (d)	0.23
Zinc	mg/kg	83.4	1.2	144	1
<i>Process metals</i>					
Silicon	mg/kg	268	12	*	*
Thorium	mg/kg	1,650	37	12,300	562
Uranium	mg/kg	6,680	100	50,500	943

Table C.10. Analytical data for sludge samples from tanks WC-13 and WC-14 (continued)

Parameter measured	Units	WC13-S Sludge (a)		WC14-S Sludge (b)	
		Result	+/-	Result	+/-
<i>Anions in bulk sludge</i>					
Cyanide	mg/kg	0.173	0.028	0.257	0.066
Sulfide (g)	mg/kg	N		N	
<i>Anions in interstitial liquid (h)</i>					
Bromide	mg/L	3.28	0.18	7.20	2.20
Chloride	mg/L	151	1	1,310	22
Fluoride	mg/L	10.0	0.2	56.1	4.2
Nitrate	mg/L	103	26	3,260	26
Nitrite (g)	mg/L	Y		Y	
Phosphate	mg/L	2,880	1	236	10
Sulfate	mg/L	616	8	803	11
<i>Radiochemical screening</i>					
Gross alpha (RMAL)	Bq/g	575	58	42,200	793
Gross alpha (TAL)	Bq/g	580 (i)	58	36,000 (i)	3,600
Gross beta	Bq/g	62,300	208	520,000	2,000
Pu (total)	Bq/g	150	20	24,000	1,000
<i>Beta/gamma emitters</i>					
Am-241	Bq/g	<7.81		16,600	1,030
C-14	Bq/g	97.1	16	13.4	15.4
Co-60	Bq/g	1,520	32	12,700	290
Cs-134	Bq/g	<60		2,720	288
Cs-137	Bq/g	4,580	42	158,000	700
Eu-152	Bq/g	39,700	134	264,000	1,370
Eu-154	Bq/g	20,400	126	84,800	999
Eu-155	Bq/g	5,170	77	21,100	591
H-3	Bq/g	*		*	
Sr-90	Bq/g	286	21	22,400	780
<i>Alpha emitters (by alpha spectrometry) (i)</i>					
Am-241	Bq/g	-		14,000	1,400
Cm-244	Bq/g	110	11	1,300	130
Pu-238	Bq/g	49	5	2,300	230
Pu-239/Pu-240	Bq/g	47	5	22,000	2,200
U-233/Th-229/Np-237	Bq/g	300	30	260	26
U-235	Bq/g	11.0	1.1		
U-238	Bq/g	49	5	380	38

Table C.10. Analytical data for sludge samples from tanks WC-13 and WC-14 (continued)

Parameter measured	Units	WC13-S Sludge (a)		WC14-S Sludge (b)	
		Result	+/-	Result	+/-
<i>Gross fissile screening</i>					
U-235 equivalent	µg/g	30	1	88	1
<i>Uranium isotopes (by mass spectrometry)</i>					
U-233	atom %	<0.01		<0.01	
U-234	atom %	<0.01		<0.01	
U-235	atom %	0.50	0.001	0.22	0.01
U-236	atom %	<0.01		<0.01	
U-238	atom %	99.50		99.77	0.01
<i>Plutonium isotopes (by mass spectrometry)</i>					
Pu-238	atom %	2.03	0.41	0.06	0.006
Pu-239	atom %	87.71	0.25	89.35	2.16
Pu-240	atom %	8.60	0.11	8.42	0.21
Pu-241	atom %	0.31	0.01	0.42	0.02
Pu-242	atom %	1.22	0.04	1.75	0.2
Pu-244	atom %	0.14	0.09	<0.01	

(a) Sample collected March 2, 1993.

(b) Sample collected March 24, 1993.

(c) Analyzed by ICP.

(d) Results were obtained after a TRU•Spec column was used to remove uranium.

(e) Silver was analyzed by GFAA. The reported result is an estimate due to the large dilution factor and no duplicate or spike was run.

(f) Thallium was not determined due to spectral interference from uranium.

(g) Qualitative analysis. N = not detected; Y = detected.

(h) Analysis performed on supernate from a centrifuged portion of the sludge.

(i) Alpha emitters were analyzed after TRU•Spec separation.

Table C.11. Analytical data for liquid samples from tank WC-14 collected in December 1994

Parameter measured	Units	WC14-L1		WC14-L2		WC14-L4	
		Result	+/-	Result	+/-	Result	+/-
<i>Physical properties and miscellaneous data</i>							
Total dissolved solids	mg/mL	*		*		*	
Total solids	mg/mL	*		*		*	
Density	g/mL	*		*		*	
Inorganic carbon	mg/L	*		*		*	
Total organic carbon	mg/L	*		*		*	
Total carbon	mg/L	*		*		*	
<i>EPA target analyte elements (RCRA metals in bold)</i>							
Aluminum	mg/L	*		<0.25		*	
Antimony	mg/L	*		<1.63		*	
Arsenic	mg/L	*		*		*	
Barium	mg/L	*		0.094	0.005	*	
Beryllium	mg/L	*		<0.011		*	
Cadmium	mg/L	*		<0.075		*	
Calcium	mg/L	*		11.6	0.3	*	
Chromium	mg/L	*		0.775	0.018	*	
Cobalt	mg/L	*		0.230	0.098	*	
Copper	mg/L	*		1.25	0.05	*	
Iron	mg/L	*		<0.038		*	
Lead (a)	mg/L	*		<4.25		*	
Magnesium	mg/L	*		2.89	0.53	*	
Manganese	mg/L	*		0.170	0.005	*	
Mercury	mg/L	*		*		*	
Nickel	mg/L	*		1.00	0.22	*	
Potassium	mg/L	*		63.3	0.3	*	
Selenium	mg/L	*		*		*	
Silver	mg/L	*		(0.199)	0.062	*	
Sodium	mg/L	*		3,190	11	*	
Thallium (a), (b)	mg/L	*		<1.50		*	
Vanadium	mg/L	*		<0.25		*	
Zinc	mg/L	*		<0.25		*	
<i>Process metals</i>							
Thorium	mg/L	*		39.2	2.9	*	
Uranium	mg/L	*		1,370	18	*	

Table C.11. Analytical data for liquid samples from tank WC-14 collected in December 1994 (continued)

Parameter measured	Units	WC14-L1		WC14-L2		WC14-L4	
		Result	+/-	Result	+/-	Result	+/-
<i>Anions</i>							
Bromide	mg/L	*		6.0	3.5	*	
Chloride	mg/L	*		459	10	*	
Cyanide	mg/L	*		*		*	
Fluoride	mg/L	*		40	1	*	
Nitrate	mg/L	*		1,021	20	*	
Nitrite (c)	mg/L	*		Y		*	
Phosphate	mg/L	*		43	18	*	
Sulfate	mg/L	*		324	5	*	
Sulfide	mg/L	*		*		*	
<i>Alkalinity</i>							
pH		*		10.0		*	
Acid (H+)	mol/L	*		*		*	
Base (OH-)	mol/L	*		*		*	
Carbonate	mol/L	*		*		*	
Bicarbonate	mol/L	*		*		*	
<i>Radiochemical screening</i>							
Gross alpha	Bq/mL	92	18	90	18	87	17
Gross beta	Bq/mL	33,000	1,000	33,000	1,000	31,000	1,000
<i>Beta/gamma emitters</i>							
Co-60	Bq/mL	33	1	34	8	39	9
Cs-134	Bq/mL	270	40	290	30	290	30
Cs-137	Bq/mL	27,000	1,000	27,000	1,000	27,000	1,000
Eu-152	Bq/mL	<130		<130		<130	
Eu-154	Bq/mL	<20		<19		<19	
Eu-155	Bq/mL	<74		<75		<75	
H-3	Bq/mL	*		*		*	
Sr-90	Bq/mL	84	4	92	4	89	4

(a) Analyzed by ICP.

(b) Special interference from uranium.

(c) Qualitative analysis. Y = detected.

Appendix D

ORGANIC CHEMICAL CHARACTERIZATION



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D.1 INTRODUCTION

The organic chemical characterization of the radioactive waste tank liquids and sludges consisted of determinations of the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) Target Compound List (TCL) volatile and semivolatile organic compounds, pesticides, and polychlorinated biphenyls (PCBs).¹ The approach involved three Analytical Chemistry Division groups: Separations and Synthesis, Organic Analysis, and Radioactive Materials Analysis. Toward the end of this characterization campaign, the Organic Analysis Group was transferred to the new central Analytical Services Organization.

The Radioactive Materials Analysis Group received and logged in the samples, prepared the composite sludge samples, and transferred liquid samples and aliquots of the sludge to the Separations and Synthesis Group for the organic chemical characterization. The radiation dose rate and the gross alpha and gross beta activity of the samples were measured. The decision was then made concerning where the samples would be prepared. If the radionuclide content was low, the sample was transferred to the Organic Analysis Group for preparation and analysis. The limit for the nonzoned Organic Analysis Group laboratories in Building 4500-South is 0.1 μCi of Class 1 radionuclides (e.g., ⁹⁰Sr, plutonium isotopes) per sample.² In practice, levels were usually kept below one-third this limit to allow multiple samples or extracts to be handled together in a single batch. Samples containing higher levels of radioactive materials were extracted by the Separations and Synthesis Group at the Radioactive Materials Analytical Laboratory (Building 2026), and the decontaminated organic extracts were transferred to the Organic Analysis Group. Most quantitative measurements were made by the Organic Analysis Group; however, direct aqueous injection gas chromatography (DAI-GC) was run by the Separations and Synthesis Group. Sample transfers were accompanied by chain-of-custody documentation.

The EPA SW-846 methods were followed as much as possible.³ Some modifications, such as reducing the sample size, were necessary to minimize radiation exposure to both the field sampling crew and the analyst in keeping with the ALARA (as low as reasonably achievable) principle.^{4,5} Sample preparation in the Radioactive Materials Analytical Laboratory was conducted in radiochemical hoods using methods similar to those employed in other waste tank characterization campaigns.⁶⁻⁸ Also, organic solvent usage was limited because of the perchlorate contamination of the radiochemical hood exhaust ductwork. Sample preparation and analysis by the Organic Analysis Group were conducted by conventional SW-846 methods. Deviations from the SW-846 methodology required by the radioactive nature or other characteristics of the samples were documented.

Results of the organic chemical characterization are summarized in Sect. 4. A more detailed discussion of the results and brief descriptions of the methods are presented in this appendix, and a tabular listing of the organic analytical data in Appendix E.

D.2 METHODS

D.2.1 Volatile Organic Compound Analysis

D.2.1.1 Analysis of Aqueous Liquid Samples for Volatile Organic Compounds

For the aqueous liquid samples the volatile organic compound analysis (VOA) was performed only if the radionuclide content of the original natural sample was below the limits for the Organic Analysis Group laboratories.² The VOA was conducted with 0.5–5.0-mL aliquots by purge-and-trap gas chromatography-mass spectrometry (GC-MS) following SW-846 Method 8260. The VOA

surrogate standards were added to all samples before the analysis. A matrix spike (MS) and matrix spike duplicate (MSD) were prepared from one sample. Laboratory blanks were run each day of the analysis. Some samples were analyzed using a purge-and-trap GC-flame ionization detector (GC-FID) screen conducted according to SW-846 Method 8000A.

Aqueous liquid samples which exceeded the radionuclide limits for the Organic Analysis Group were not analyzed for volatile organic compounds because of the lack of a purge-and-trap gas chromatograph-mass spectrometer in the Radioactive Materials Analytical Laboratory.

D.2.1.2 Analysis of Oil Samples for Volatile Organic Compounds

Oil layers were found in two tanks (WC-11 and WC-13). The oil samples were prepared at the Radioactive Materials Analytical Laboratory for VOA by diluting 0.2 mL of the oil to 2.0 mL with methyl alcohol and adding VOA surrogate standards per SW-846 Method 3580A. A solvent blank was also prepared. The dilutions were analyzed for gross alpha and gross beta activities. They were low in radioactivity and were transferred to the Organic Analysis Group for quantitation. Aliquots of the dilutions were analyzed by the purge-and-trap GC-FID screen.

D.2.2 Major Water-Soluble Volatile Organic Compounds by DAI-GC

The lower alcohol and ketone homologs, which are polar and highly water soluble, are either not detected or are poorly measured using purge-and-trap methods. A DAI-GC method based upon SW-846 Method 8015A was used to determine methyl, ethyl, *i*-propyl, allyl/*n*-propyl, *i*-butyl, and *n*-butyl alcohols and acetone, methylethyl ketone (2-butanone), and methyl-*i*-butyl ketone (4-methyl-2-pentanone). This instrument is located in the Radioactive Materials Analytical Laboratory. A small aliquot (~1.5 mL) of aqueous liquid sample was transferred to a radiochemical hood. Injections of 3 μ L were made into a gas chromatograph equipped with a 3.2-mm-OD by 3-m stainless steel column packed with 1% SP-1000 coated on 60/80 mesh Carbowax B™, a flame ionization detector, and a reporting integrator. The column packing is the same as that used for the detection of volatiles by GC-MS in SW-846 Method 8240. The flow rate of the helium carrier gas was 30 mL/min. The initial column oven temperature of 70°C was maintained for 2 min, followed by a programmed 16°C/min increase to 220°C and a final isothermal hold at 220°C for 16 min. The inlet and detector temperatures were 150° and 250°C, respectively. The instrument was calibrated weekly with four standard concentrations ranging from 4 to 40 mg/L, and calibration was verified each day of use by analyzing a midpoint standard. Components in the samples were quantified using the method of external standards. One sample was spiked in duplicate with matrix spike standards. Laboratory blanks of Milli-Q™ reagent water also were analyzed. The detection limits were 1 mg/L.

Because this analysis does not include a mass spectrum or other confirming evidence, it cannot be considered as unambiguous in its compound identifications. To reduce the possibility of erroneous identifications, those samples which yielded "hits" were also run on a second column with a different stationary phase (a 3.2-mm-OD by 2.2-m glass column packed with a mixture of 60/80 and 35/60 mesh Tenax™) under the same temperature and flow rate conditions. This second analysis was conducted after most of the samples had been collected and analyzed on the primary column. It was a qualitative confirmation only. Quantitation was not conducted because holding times could not be met.

D.2.3 Semivolatile Organic Compound Analysis and Pesticide/PCB Analysis of Aqueous Liquids

Aqueous liquid samples were prepared for the semivolatile organic compound analysis (SVOA) and pesticide/PCB analysis in the Radioactive Materials Analytical Laboratory or by the Organic Analysis Group, depending upon the radionuclide content. The preparation procedures were slightly different, but both were based upon SW-846 Method 3510A. The SVOA quantitation was conducted using SW-846 Method 8270A. The pesticide/PCB determinations were accomplished by SW-846 Method 8080 using a capillary column. Usually, ~200-mL aliquots of samples were processed. Laboratory blanks of Milli-Q reagent water and matrix-spiked samples were prepared and analyzed.

D.2.3.1 Samples Prepared in the Radioactive Materials Analytical Laboratory

At the Radioactive Materials Analytical Laboratory, preparation of aqueous liquid samples consisted of adjusting the sample pH to >11 (if the sample were initially alkaline) and adding SVOA and pesticide/PCB surrogate standards (and matrix spikes if a spiked sample). Most samples were alkaline. The sample was then extracted three times with 10 mL of methylene chloride to recover the base/neutral fraction. The acid fraction was then extracted by adjusting the pH to <2 and partitioning three times with 10 mL of methylene chloride. The reverse order was used if the sample was initially acidic. The first extraction was conducted in this manner to prevent precipitation problems typically encountered with radioactive tank wastes when their pH is radically changed. The extracts were combined and screened for gross alpha and beta activity before their transfer to the Organic Analysis Group. In all cases the extracts were essentially uncontaminated by radioactivity.

Extracts transferred to the Organic Analysis Group were concentrated to 10 mL. Nine of the 10 mL were further concentrated to 0.9 mL and were analyzed for semivolatile organic compounds by SW-846 Method 8270A. One mL of the 10-mL extract was solvent-exchanged to hexane, subjected to florisil cleanup (per SW-846 Method 3620A), and concentrated into a final volume of 1.0 mL for pesticide/PCB analysis. For the December 1994 aqueous liquid samples collected from tank WC-14, the entire solvent extracts, after purification, were concentrated to 1.0 mL and used for PCB analysis. This procedure lowered the detection limit of the PCB analysis (in effect, 0.1 of the normal protocol). Pesticides and PCBs were determined by GC-electron capture detection (GC-ECD) according to SW-846 Method 8080, except that a capillary column was used in place of the packed column. [This differed from the proposed Method 8081 because of the slightly different quality control (QC) measures.]

D.2.3.2 Samples Prepared in the Organic Analysis Group Laboratories

The liquid samples from five tanks (W-17, W-18, WC-5, WC-6, and WC-8) were low in activity such that the original, natural sample could be handled in the nonzoned laboratories of the Organic Analysis Group. There were some minor differences between the preparation of these samples and that of the radioactive samples (Sect. D.2.3.1). In the Organic Analysis Group, the acid/neutral fraction of environmental samples is routinely extracted before the basic fraction because of the lability of phenols in alkali. This routine practice was continued with the waste tank samples. About 200 mL of sample were made up to 1 L with Milli-Q reagent water, and surrogate standards (and matrix spike if a spiked sample) were added. The pH was adjusted to 1.5,

and an acid/neutral fraction was extracted three times with 60 mL of methylene chloride. The acid/neutral fraction was concentrated to 1.0 mL, and 0.1 mL was taken for pesticide/PCB analysis. The remainder was reserved for the SVOA. The pH of the sample was then adjusted to >11, and the base fraction was extracted three times with 60 mL of methylene chloride. The base fraction was concentrated to 1.0 mL, and combined with the 0.9 mL of acid/neutral fraction for a final volume of 1.9 mL. The combined extracts were analyzed for semivolatile organic compounds by GC-MS per the SW-846 Method 8270A. The 0.1 mL of the acid/neutral fraction was purified by florisil column chromatography following SW-846 Method 3620A and was then concentrated to 1 mL for pesticide/PCB determinations, as described in Sect. D.2.3.1.

D.2.4 Semivolatile Organic Compound Analysis and Pesticide/PCB Analysis of Oil Samples

Oil samples were prepared for the SVOA per SW-846 Method 3580A by diluting 0.2 mL of oil to 2.0 mL with methylene chloride and then adding the SVOA surrogate standards. For the pesticide/PCB analysis, 0.2 mL of oil was diluted to 2.0 mL with hexane, pesticide/PCB surrogate standard was added, and then 1.1 mL was washed with 40- μ L aliquots of concentrated sulfuric acid until the washings were clear. The sulfuric acid washings were extracted once with hexane, and that hexane was then added to the purified hexane extract. The combined hexane layer was washed with distilled water, and the final volume was reduced to 1.1 mL. This procedure was patterned after the proposed SW-846 Method 3665. All oil sample dilutions were checked for gross alpha and gross beta activity to confirm their low radioactivity before they were transferred to the Organic Analysis Group for quantitative measurements by SW-846 Method 8080. Solvent blanks were prepared and analyzed with the samples.

D.2.5 Semivolatile Organic Compound Analysis and Pesticide/PCB Analysis of Sludge Samples and Special Cases

The preparation of some samples required special attention because of their radioactivity or other properties.

D.2.5.1 Sample WC12-L3-C

Sample WC12-L3-C was a mixture of a thin sludge and tank liquid collected from the bottom of tank WC-12 with the vacuum-pump sampling system. Because of the limited quantity of sample material available and the highly radioactive nature of the solids, a single aliquot (~150 mL) of the sample was filtered, and the filtrate was analyzed. Initial surveys indicated radiation fields of ~900 mrem/h with the probe under the bottle (i.e., under the sludge layer) and ~480 mrem/h with the probe parallel to the sample bottle on the bench top. The highly penetrating nature of the radiation indicated the presence of ^{60}Co . A lead-brick shielding unit was constructed in a radiochemical hood, and all equipment necessary for sample preparation was assembled in advance. A survey at 18 in. indicated a dose rate of ~380 mrem/h (through the lead bricks). The sample (as six 30-mL aliquots) was filtered through a medium-porosity sintered glass filter topped by a 0.7- μ m-porosity fiberglass filter. The filtration took approximately 25 min. The dose rate of the residual solids on the filter, which were estimated as <0.5% of the sample, was ~800 mrem/h. The solid material was discarded. Most of the radioactivity was associated with the solid material. The dose rate for liquid samples (free of solids) from this tank were ~20–25 mR/h. The filtrate

was extracted with methylene chloride/acetone by the SW-846 Method 3510A (separatory funnel), as described in Sect. D.2.3.1. After extraction of the acid/neutral and base fractions, the combined organic extract volume ~75 mL. The extract was treated with sodium sulfate to remove all water, and the resulting extract was analyzed for gross alpha and gross beta. This extract was essentially radionuclide-free. A sample of Milli-Q water was extracted along with the sample to serve as the laboratory blank.

The extracts were transferred to the Organic Analysis Group for further concentration and solvent exchange to hexane prior to analysis for semivolatile organic compounds and pesticides/PCBs. The 10 mL of hexane extract was split. One mL was taken for pesticide/PCB preparation, was purified using the florisil column (Method 3620A), and diluted to 10 mL before analysis. The remaining 9 mL of extract were concentrated to 1 mL for the SVOA.

D.2.5.2 Sludge Sample WC13-S

Two "cores" of sludge were collected from tank WC-13 on March 2, 1993, and were received by the Radioactive Materials Analysis Group. The samplers contained both sludge and supernatant tank liquid. The phases were separated and removed from the polyvinyl chloride (PVC) tubes shortly after the samples were received because the samplers were leaking. The sludge (solids) phases from the two samples, WC13-S1 and WC13-S2, were combined and allowed to stand overnight. The sludge solids settled slightly while standing. A small aliquot of the free liquid over the sludge was removed for water-soluble volatiles analysis by DAI-GC. The combined sludge phases were then mixed by sonication to form the composite sludge sample WC13-S. Aliquots of the composite sludge and the liquid were transferred to the Separations and Synthesis Group for the organic chemical characterization. (For additional information on sample handling, see Appendix B, Sect. B.2).

Because of the high levels of radioactivity and limited quantity of sample material, the sludge samples were extracted for semivolatile organics, pesticides, and PCBs using a modification of the SW-846 Method 3550. A sample, matrix spike, matrix spike duplicate, and sand blank were prepared. About 10 g of sludge slurry for each aliquot were weighed into a 250-mL beaker. Surrogate standards (and matrix spike if a spiked sample) were added to the samples. Because of the limited amount of the sample, the moisture content was not determined. The sludge slurry was stirred, and anhydrous sodium sulfate was slowly mixed into the sample. About 70 g of sodium sulfate were required to dry each aliquot to a finely granular material. The dried sludge mixture was extracted three times with 50-mL aliquots of methylene chloride/acetone (1:1 vol:vol) using a 350-W ultrasonicator fitted with a 1.5-cm-OD horn. Each aliquot was extracted at a 50% duty cycle and a power level of 5 for 3 to 5 min. After each extraction, the supernatant liquid was decanted into a medium-porosity sintered glass funnel, and the extracts were filtered and collected as a single sample. The organic solvent from the first extraction was a fluorescent pink-orange color. The second and third extracts were a "normal" tan color. The extracts were concentrated using dry flowing nitrogen (nitrogen blow-down) to 10 mL. A gross alpha and a gross beta screen were performed to confirm that the samples were low in activity.

The extracts were transferred to the Organic Analysis Group for final preparation and analysis. An attempt was made by the Organic Analysis Group to further concentrate the extracts. However, the extracts could not be concentrated below ~6 mL by nitrogen blow-down. At this point the vials contained two phases—a fluorescent red phase on the top and an oily orange-yellow phase on the bottom. The extracts were diluted back to 10 mL to obtain a homogeneous mixture. A GC-FID screen was performed to find the dilution factor for the SVOA. The screen showed the presence of oil, probably vacuum-pump oil. For the SVOA, 1 mL of the 10-mL dilution was

diluted to 10 mL (in effect, a 1:100 dilution over normal protocol) to prevent the gas chromatograph-mass spectrometer from being overloaded and contaminated with organic material. The semivolatile organic matrix spikes were not analyzed because the spikes were effectively diluted out. For the pesticide/PCB analysis, 1 mL of the 10-mL extract was cleaned up using the florisil column (SW-846 Method 3620A), which removed the color from the extracts. The sample, matrix spike, and matrix spike duplicate were analyzed for pesticides/PCBs. (The samples were spiked with certain pesticides, but not with PCBs).

D.2.5.3 Sludge Samples from Tank WC-14

The sludge phase in tank WC-14 was sampled in March 1993 and analyzed for semivolatile organic and pesticide/PCB analytes (and also inorganic analytes and radioactive materials). After the first analysis showed PCB concentrations in excess of 50,000 $\mu\text{g}/\text{kg}$ (>50 ppm), the decision was made to resample the tank only for PCB analysis. There were some differences in the behavior of the two sets of samples during sample preparation.

D.2.5.3.1 Composite Sludge Sample WC14-S

Two "cores" of sludge were collected from tank WC-14 on March 24, 1993, as independently as was possible given the limited tank access. The cores were received by the Radioactive Materials Analysis Group. The samplers contained both sludge and supernatant tank liquid. The solids were allowed to settle, and the phases were separated. The sludge (solids) phases from the two samples, WC14-S1 and WC14-S2, were then combined and mixed by sonication to form the composite sludge sample WC14-S (see also Appendix B, Sect. B.2). Aliquots of the composite sludge were transferred to the Separations and Synthesis Group for organic chemical characterization.

Because of the limited amount of material, only two aliquots of the sludge sample could be used for extraction and analysis of organic analytes. Each aliquot was ~6 g; one was used for a matrix spike. A sand blank was also processed. The samples were extracted by the method described for sludges in Sect. D.2.5.2. The samples and blank were dried by the addition of ~8 g of sodium sulfate. The dried sample was then split. A small portion (~0.9 g) was removed, sonicated with methylene chloride, and screened by GC-FID to determine the spike concentrations. The remainder of the sample, the matrix spike, and the blank were then sonicated in methylene chloride/acetone. There was considerable foaming of the liquid during sonication and extraction. As the combined extracts for the sample were concentrated by nitrogen blow-down, two layers formed in the extraction fluid—a brownish oily liquid and a pink layer. Both layers appeared to be organic. When the samples were cooled, the layers reversed position. Because it was possible that the organic material contained dissolved water, the solution was saturated with sodium sulfate and allowed to stand under refrigeration over the weekend, after which only one layer was observed.

The formation of organic layers during the extraction of some waste tank samples had been observed previously at ORNL and Pacific Northwest Laboratory. In some cases the layering effect is not resolved by the addition of sodium sulfate. It is not clear why the layers form, but it may be because of the presence of azeotropes. It has been observed that the layering is enhanced by the presence of alcohol even at very low concentrations. It also seems to be enhanced by extraction at pH levels other than near-neutral. Further work is needed to overcome this extraction problem.

The extracts, after being screened for gross alpha and gross beta activities, were transferred to the Organic Analysis Group for final preparation, and semivolatile organic compound and pesticide/PCB analyses. As for the WC13-S sludge, 1 mL of each extract was purified using the florisil column (Method 3620A), and was diluted to 10 mL for pesticide/PCB analysis. The sample, matrix spike, and the blank were analyzed. The remainders of the extracts were taken "as is" (in effect a 1:10 dilution over normal protocol) for the SVOA analysis to prevent the gas chromatograph-mass spectrometer from being overloaded and contaminated with organic material.

D.2.5.3.2 Second Set of Sludge Samples from Tank WC-14

Because the concentrations of PCBs determined in the first sampling of tank WC-14 sludge exceeded 50,000 µg/kg, the tank was resampled to check the results. Two "cores" of sludge were collected on August 10, 1993, and signed over to the Separations and Synthesis Group at the Radioactive Materials Analysis Laboratory. The solids were allowed to settle, and the supernatant liquid was removed. The sludge phases were removed from the samplers and analyzed as two independent samples, WC-14S-1 and WC-14S-2. The samples were mixed by stirring with a spatula before the aliquots were taken.

Two sludge samples [from different "cores" (~10 g each)], a matrix spike, a matrix spike duplicate, and a blank were prepared for PCB analysis per the methods described above for sludges. Layering was not evident after concentrating the extract to a 10-mL volume. (As noted previously, layering was evident in the extracts of the March 1993 samples.) The analysis of these extracts after florisil cleanup by the Organic Analysis Group indicated high levels of organic material which caused interference with the GC-ECD. It was determined that the extracts should be cleaned up using sulfuric acid treatment and subsequent passage through florisil columns. With the exception of the blank, all of the extracts exhibited a yellow color. After overnight storage at 4°C, the sample extracts appeared to contain some water and emulsified compounds.

The extracts were transferred back to the Separations and Synthesis Group from the Organic Analysis Group. Two-mL aliquots of each of the extracts and the hexane (solvent control) were placed in 10-mL concentrator tubes and were reduced in volume to ~0.5 mL by blow-down with dry nitrogen. Two mL of hexane were added to each of the tubes. This solvent exchange was repeated three times, and the volume was returned to 2 mL. During the solvent exchange procedure, the sample extracts exhibited layering. In addition to a clear layer, a pink, oily layer and an emulsified layer were prominent. The emulsified compounds formed droplets on the glass surface of the concentrator tubes. A hard precipitate was observed in the bottom of the sample tubes. The precipitate later proved to be insoluble in either water or organic solvent (methylene chloride or hexane). The hexane extracts were removed to 15-mL vials, and the concentrator tubes were washed 4 times with 2 mL of hexane (total 8-mL wash) to remove the residual material. The hexane wash solution was added to the appropriate sample aliquot prior to treatment with sulfuric acid.

One mL of concentrated sulfuric acid was added to each of the sample aliquots. A brown-to-black precipitate formed in each of the extracts except the blank and the hexane solvent control. After the layers had separated, the sulfuric acid was removed. This sulfuric acid treatment was repeated four more times before the sulfuric acid remained clear, indicating that no more organics were being destroyed. After treatment with sulfuric acid, the hexane extracts were washed with 1 mL of water to remove residual acid. A small amount of fine white precipitate formed in the hexane extract after treatment with acid and overnight storage at 4°C. The precipitate was insoluble in water.

The florisil columns were washed with three bed volumes of hexane prior to loading the extracts. Each extract was passed through a column using gravity flow and was followed by two bed volumes of hexane. The extracts were then reduced to 2-mL volumes by nitrogen blow-down. The 2-mL aliquots were split, and 1 mL was placed in each of two crimp-top vials prior to transfer to the Organic Analysis Group for PCB analysis by SW-846 Method 8080.

D.2.5.4 Second Set of Liquid Samples from Tank WC-14

The supernatant liquid in tank WC-14 was resampled on August 3, 1993, for PCB analysis only (a week before the second set of sludge samples were taken). One ~200-mL sample was collected from the midpoint of the liquid layer and one from the top surface. The samples were prepared for PCB analysis per the methods described in Sect. D.2.3.1. A blank was also processed. (Because the amount of sample was limited no matrix spike was prepared.) The analysis of the sample extracts after florisil cleanup by the Organic Analysis Group indicated high levels of organic material which caused interference with the GC-ECD determination. (No interferences were observed with the first liquid sample from tank WC-14, which had been collected about 9 months earlier.) It was determined that the extracts should be cleaned up by the sulfuric acid treatment followed by florisil cleanup method used to repurify the tank WC-14 sludge samples (see Sect. D.2.5.3).

The extracts were returned to the Separations and Synthesis Group. They were diluted to 2 mL with hexane and treated with 1 mL of concentrated sulfuric acid. The sulfuric acid was removed after the layers had separated, and the treatment was repeated two more times to remove extraneous organics. The sulfuric acid was washed twice with 3 mL of hexane, and the washings were combined with the appropriate aliquot. After treatment with sulfuric acid, the hexane aliquots were washed two times with 2 mL of water to remove any residual acid. They were then purified by florisil cleanup and transferred to the Organic Analysis Group for reanalysis for PCBs. The final chromatograms exhibited "clean" peaks.

D.2.6 Tentative Identification of Oil Layers and Estimation of Oil in Sludge Sample WC13-S

Tanks WC-11 and WC-13 contained an oil layer. The tentative identification of the oil type was conducted by comparing their capillary column GC-FID profiles with those of known oils. The oil samples were diluted 1:10 or 1:100 (vol:vol) in methylene chloride, and 3.3 μL were injected into a 30-m by 0.53-mm-ID fused silica capillary column with a 1.5- μm bonded film of DB-5. The initial column oven temperature of 35°C was maintained for 5 min, followed by a programmed 5°C/min increase to 310°C, and then maintained isothermally at 310°C for 25 min. The inlet temperature was 270°C and the detector was set at 300°C. The helium-carrier-gas flow rate through the column was 6 mL/min, which was made up to 30 mL/min at the detector with auxiliary helium flow. The GC profiles of the samples were compared with those of known oils that had been prepared and analyzed in the same manner. This was only a tentative qualitative identification.

The oil content in the sludge WC13-S was estimated by analyzing the SVOA extract by GC-FID as described in the preceding paragraph and by comparing the height of the "hump" in the chromatogram with that of a known concentration of vacuum-pump oil diluted in methylene chloride.

D.3 DISCUSSION OF RESULTS

The results of the organic chemical characterization are discussed in this section, with the analytical data presented in the tables in Appendix E. These tables are organized in Appendix E as follows:

1. Organic compound lists and conversion factors for calculating reporting limits	Tables E.1–E.6
2. Summary of results	Tables E.7–E.10
3. Surrogate standard recoveries	Tables E.11–E.14
4. Matrix spike recoveries	Tables E.15–E.16

D.3.1 Target Compound Lists and Reporting Limits for Organic Analyses

The reporting limits varied somewhat from sample to sample.* Except for the DAI-GC of major water-soluble volatile organic compounds, the aliquot volumes processed for the analyses were not the same for each sample. There were differences in the dilution factors between samples prepared in the Radioactive Materials Analytical Laboratory and those prepared in the Organic Analysis Group laboratories. Also, it was necessary to dilute several samples (over normal protocol) for the SVOA to prevent the gas chromatograph-mass spectrometer from being overloaded and contaminated with organic material, and for the PCB analyses to minimize interferences. Table E.1 identifies the samples, the sample bottle letter, the aliquot volumes or masses processed, and the conversion factors for calculating the reporting limits for the analyses. Table E.2 is for the second set of samples collected from tank WC-14 for PCB analysis. The EPA Target Compound Lists (TCLs)¹ and the reporting limits for conventional analyses of nonradioactive samples using 5 mL or 5 g for the VOA and 1 L or 30 g for the SVOA and pesticide/PCB analyses are given in Tables E.3–E.5. The compound list for water-soluble volatile organic compounds by DAI-GC is given in Table E.6. The reporting limits in Tables E.3–E.5 should be multiplied by the conversion factors in Tables E.1 and E.2 to determine the actual reporting limits for each sample analysis. The one exception is the DAI-GC of major water-soluble volatile organic compounds. Because this is a direct analysis, the reporting limits (1 mg/L) were the same for all samples.

D.3.2 Volatile Organic Compounds

The results of the VOA for aqueous liquids are summarized in Tables E.7 and E.8. No volatile organic compounds on the EPA TCL were detected in the aqueous liquids from tanks W-17 or W-18, except a trace of acetone: 39B µg/L in W17-L1 and 37B µg/L in W18-L1. The "B" flag indicates that acetone was found also in the laboratory blank run with the samples. The blank concentration of acetone was 17 µg/L; therefore, the hits for these two samples were only slightly

*The reporting limits are the concentrations above which the response of the instrument for a calibrated range of concentrations is linear.

higher than the blank and are probably not significant. Although the holding times were missed on the purge-and-trap GC-MS of these two samples, the purge-and-trap GC-FID screens were carried out within holding times. The GC-FID screens also detected only acetone, estimated as 29 and 8 µg/L for W17-L1 and W18-L1, respectively. This finding confirms the results of the GC-MS analysis. No volatile organics were detected in the purge-and-trap GC-FID screen of samples WC5-L1, WC6-L1, and WC8-L1, but holding times were missed by 39 days. No tentatively identified compounds (TICs) were detected in these analyses.

No volatile organics were detected in the diluted oil samples, WC11-Surface and WC13-Surface. This finding is consistent with the tentative identification of these oils as vacuum-pump oils (see Sect. D.3.5). Such high-boiling oils normally do not contain volatile constituents unless contaminated during use.

The aqueous liquids from tanks WC-11, WC-12, WC-13, and WC-14 and the sludge samples could not be analyzed for volatile organics because the samples were too radioactive to transfer to the nonzoned Organic Analysis Group laboratories.

Recoveries of the VOA surrogate standards (Table E.11) and matrix spikes (Table E.15) were all within the QC acceptance limits for groundwater, but the relevance of these limits to radioactive wastes has not been established. Laboratory blanks were clean, except for traces of acetone.

The results of both the VOA and the water-soluble organics analyses may be biased low because of the method of sample collection. Drawing liquid samples by vacuum through Teflon™ tubing into the sample collection jar may result in the loss of some volatile organic compounds. However, this sampling method is the state of the art for radioactive liquids in underground waste tanks with limited access. Therefore, the results for the volatile organic compounds should be viewed as minimum values.

D.3.3 Water-Soluble Volatile Organic Compounds

The results for the DAI-GC analysis of the aqueous liquids were consistent with the VOA in that most samples did not contain any detectable compounds (Tables E.7 and E.8). Only two samples showed the presence of alcohols, which would not be detected in the VOA. However, these samples were too radioactive to conduct the VOA. Sample WC13-L1 contained 9,600 µg/L of methyl alcohol, 8,900 µg/L of n-butyl alcohol, and 230 µg/L of an unidentified compound eluting near the retention time of acetone. The confirmation column showed that the unknown was not acetone. Sample WC14-L1 contained 14,700 µg/L of methyl alcohol and 330 µg/L of the unknown eluting near acetone.

The water-soluble volatile organic compounds in the interstitial liquid of one sludge, WC13-S, were determined (Table E.9). Methyl and n-butyl alcohol were present at 5,000 and 5,200 µg/L, respectively.

Holding times were met, and laboratory blanks were clean. The matrix spike recoveries were good (Tables E.15 and E.16).

D.3.4 Semivolatile Organic Compounds, Pesticides, and PCBs

D.3.4.1 Results for Aqueous Liquid Samples

Very few analytes on the TCLs were detected in the SVOA and pesticide/PCB analyses of the aqueous liquids (Tables E.7 and E.8). The SVOA hits included traces of phthalates: 2J µg/L of

butylbenzylphthalate in both WC5-L1 and WC6-L1; 17J $\mu\text{g/L}$ of di-n-octylphthalate and 7J μg of diethylphthalate in WC11-L1, and 100J $\mu\text{g/L}$ of benzoic acid in WC14-L1. The "J" flag indicates that the result is an estimate because it was measured at a concentration below the reporting limit of the instrument. Traces of pesticides or PCBs were detected in two samples. W18-L1 contained 0.046J $\mu\text{g/L}$ of endosulfan II, 0.12J $\mu\text{g/L}$ of α -chlordane, 4.0J of Aroclor-1254, and 1.9J $\mu\text{g/L}$ of Aroclor-1260. Sample WC14-L1 (November 1992) contained 278 $\mu\text{g/L}$ of Aroclor-1248. The analysis of the second sampling of this tank (Table E.10), conducted almost 1 year later, showed only a trace (5–7 $\mu\text{g/L}$) of Aroclor-1248. A third sampling and analysis of tank WC-14 aqueous liquid which was conducted in December 1994 yielded similar results of 10.9 and 13.2 $\mu\text{g/L}$ of Aroclor-1248 for duplicate samples. The first set (taken in November 1992) of liquid samples from tank WC-14 were collected about 2.5 in. above the sludge phase. These samples were slightly cloudy and may have contained a trace of the sludge phase, which could have elevated the apparent PCB concentration (see Sect. D.3.4.5).

The main organic compounds detected in the aqueous samples were found in the TICs. Only two compounds could be identified with any confidence from their mass spectra and use at ORNL. These were (1) tributylphosphate, which was found at 470J $\mu\text{g/L}$ in WC13-L1 and 9700J $\mu\text{g/L}$ in WC14-L1, and (2) diethylbenzene (or other C_4 -substituted benzene), which was estimated at 280J $\mu\text{g/L}$ in the latter sample. A summary of the semivolatle organic analysis TICs is included in Tables E.7 and E.8.

The blanks were clean, except for the 4J $\mu\text{g/L}$ of di-n-butylphthalate in the laboratory blank processed with WC11-L1 and WC12-L1. The surrogate standard and matrix spike recoveries for the aqueous liquid analyses were good (Tables E.11, E.12, E.14, and E.15), except for some of the pesticide spike recoveries in the WC11-L1 MS/MSD. Because the pesticide surrogate standard recoveries were acceptable, a matrix effect rather than an error in method execution is suggested.

D.3.4.2 Results for Oil Layer Samples

The SVOA of the diluted oil layers did not reveal any significant hits other than 17,000,000J $\mu\text{g/L}$ of di-n-butylphthalate in the WC13-Surface oil (Table E.8). The SVOA surrogate standard recoveries were generally low (Table E.12). Because the SVOA surrogate standard recoveries for the solvent blank were generally high, the low recoveries for the oil dilutions probably were the result of a matrix interference upon the GC-MS analysis.

The oil layer samples contained Aroclor-1254 at 1700 $\mu\text{g/L}$ (WC11-Surface) and 5060 $\mu\text{g/L}$ (WC13-Surface). The GC-ECD profiles from both the primary and confirmatory columns suggested the presence of pesticides (40 $\mu\text{g/L}$ of γ -chlordane, 280 $\mu\text{g/L}$ of α -chlordane, and 90 $\mu\text{g/L}$ of 4,4'-DDE in WC11-Surface and 0.86 mg/L of γ -chlordane, 0.74 mg/L of α -chlordane, and 0.34 mg/L of aldrin in WC13-Surface, but these were not confident hits. The peaks are believed to be co-eluting chlorinated biphenyls from the complex mixture of the Aroclor-1254. Also, the oil extracts were extensively purified by acid washing—which could destroy some pesticides. Further work would be necessary to confirm the pesticides if they are present at all. The pesticide surrogate standard recoveries were generally high for the oils (Table E.12).

D.3.4.3 Results for Sample WC12-L3-C

The SVOA results for the WC12-L3-C sludge filtrate (Table E.9) were different from those for the WC12-L1 sample, which had been collected from the midpoint of the supernatant liquid. The filtrate from WC12-L3-C was found to contain several phthalates (diethylphthalate,

di-n-butylphthalate, bis(2-ethylhexyl)phthalate, and di-n-octylphthalate at 89B, 390, 25J, and 12J $\mu\text{g/L}$, respectively). The extra sample preparation did not contribute these compounds. The blank contained only diethylphthalate at 12J $\mu\text{g/L}$. The TICs in WC12-L3-C included tributylphosphate (630J $\mu\text{g/L}$), another isomer of dibutylphthalate (66J $\mu\text{g/L}$), a propanol derivative (64J $\mu\text{g/L}$), and 18 unknown compounds (total of 7540 $\mu\text{g/L}$). Of interest was the identification of 2,6-dibromo-4-nitrophenol (54J $\mu\text{g/L}$), which undoubtedly was formed by the nitration of the tribromophenol SVOA surrogate standard during the extraction. This artifactual nitration has been observed in the preparation of other radioactive waste tank samples at ORNL and at other U.S. Department of Energy (DOE) sites. The nitrated bromophenol does not account for all the missing tribromophenol surrogate standard (Table E.13), suggesting either incomplete extraction of this standard during sample preparation or its partial conversion to other products which were either not detected or not identified. These observations illustrate the reactive nature of radioactive wastes.

The SVOA surrogate standard recoveries for the WC12-L3-C filtrate (Table E.13) were good for the base/neutral standards, but low for the acidic phenol standards. The recoveries for the blank were good, suggesting a matrix effect on the phenol standards for the sample results. The recoveries of the more stable pesticide surrogate standards were also good.

D.3.4.4 Results for Sludge Sample WC13-S

Results for the sludge sample WC13-S are shown in Table E.9. The sludge samples presented considerable difficulties in SVOA preparation, as described in the methods section of this appendix (Sect.D.2.5). Also, because of the large amount of organic material, the SVOA extracts could not be concentrated below about 10 mL, and they required further dilution before GC-MS. This procedure raised the reporting limits of the SVOA analyses and diluted the SVOA surrogate standards and matrix spikes, preventing their quantitation. WC13-S contained 31,000B $\mu\text{g/kg}$ of di-n-butylphthalate. The TICs included 47,000J $\mu\text{g/kg}$ of tributylphosphate and 6 unknown compounds which totalled 377,000J $\mu\text{g/kg}$.

The PCB/pesticide analysis of WC13-S was simplified by the purification afforded by the florisil cleanup method. The florisil column removed the color from the crude extract. The GC-ECD profile was a very good match with Aroclor-1254. The result of the analysis was 1630J $\mu\text{g/kg}$ of Aroclor-1254. The results for the matrix spike sample WC13-S-MS and matrix spike duplicate WC13-S-MSD (which were not spiked with PCBs, but only with certain pesticides) were 1510J and 1610J $\mu\text{g/kg}$ of Aroclor-1254, respectively. This adds considerably to the confidence in the analysis (average \pm standard deviation for all three analyses is 1580 ± 64 $\mu\text{g/kg}$). The analysis suggested also the presence of aldrin, heptachlor epoxide, and α -chlordane at 51, 71, and 121 $\mu\text{g/kg}$, respectively, but these tentative identifications are not considered as accurate because of the chromatographic interference of the components of the Aroclor mixture. It is highly likely that the peaks really are co-eluting chlorinated biphenyls. Further work would be necessary to confirm the pesticides—if they are present at all.

The blank contained only 4100J $\mu\text{g/kg}$ of di-n-butylphthalate. The pesticide surrogate standard recoveries were good (Table E.13). Because of the dilution of the SVOA extract, the SVOA surrogate standards and matrix spikes could not be measured.

The oil content of the WC13-S sludge was estimated to be ~6 wt. % and it appeared to be vacuum-pump oil (or an oil with the same distillation range). This result is consistent with the total organic carbon content of 4.14%, which includes only carbon, but not hydrogen or other elements present in the oil.

D.3.4.5 Results for Sludge Samples from Tank WC-14

Results for the March 1993 sampling of tank WC-14 are given in Table E.9. The two SVOA hits were bis(2-ethylhexyl)phthalate (14,000J $\mu\text{g}/\text{kg}$) and naphthalene (3,500J $\mu\text{g}/\text{kg}$). The SVOA TICs included tributylphosphate (270,000J $\mu\text{g}/\text{kg}$); two C_4 -benzenes (total of 95,200J $\mu\text{g}/\text{kg}$), which were probably isomers of diethylbenzene used in processing irradiated targets; seven unknown hydrocarbons (total 674,200J $\mu\text{g}/\text{kg}$); seven unknown compounds (total 734,000J $\mu\text{g}/\text{kg}$); and several chlorinated biphenyls. The latter consisted of two trichlorobiphenyls (total 25,500J $\mu\text{g}/\text{kg}$), two tetrachlorobiphenyls (total 15,600J $\mu\text{g}/\text{kg}$), and one pentachlorobiphenyl (8,000J $\mu\text{g}/\text{kg}$). The chlorinated biphenyl TICs support the PCB analysis, which found 119,000 $\mu\text{g}/\text{kg}$ of Aroclor-1248. The result for the matrix spike sample WC14-S-MS was 128,000 $\mu\text{g}/\text{kg}$ of Aroclor-1248. The average of the two analyses is 124,000 $\mu\text{g}/\text{kg}$. (The matrix spike sample was not spiked with PCBs, but only with certain pesticides.) The sum of the chlorinated biphenyl TICs in the SVOA was 49,100J $\mu\text{g}/\text{kg}$, which is good agreement, considering that the response factor used for the TICs was the nearest eluting SVOA internal standard, and not that of the authentic PCB. In addition, in the EPA protocol only the 20 most concentrated TICs are determined and analyzed. Other chlorinated biphenyls that may have been present would not be analyzed. Heptachlor also was reported at 7370 $\mu\text{g}/\text{kg}$ in the sample, but none was found in the matrix spike sample (<414 $\mu\text{g}/\text{kg}$), so this is not considered to be a confident hit.

The blank accompanying this sample was clean, and there were no hits or TICs. The SVOA matrix spike recoveries were good for the base/neutral spikes, but low for acidic phenols (Table E.16). The latter is probably a matrix effect from the alkaline sludge. The pesticide surrogate standard recoveries were good (Tables E.13 and E.16). Because of the dilution of the SVOA extract, the SVOA surrogate standards could not be measured accurately.

The PCB analysis (Table E.10) of the second set of sludges collected from tank WC-14 in August 1993 confirmed the results for the first set. Samples WC-14S-1 and WC-14S-2 were found to contain Aroclor-1248 at concentrations of 105,000 and 241,000 $\mu\text{g}/\text{kg}$, respectively. The blank was low (59 $\mu\text{g}/\text{kg}$), and matrix spike duplicate and surrogate standard recoveries were good (Table E.14). Results for the matrix spike and matrix spike duplicate of the WC-14S-1 sample, corrected for the spike were 126,000 and 163,000 $\mu\text{g}/\text{kg}$, respectively. The result for the three aliquots of WC-14S-1 was $131,000 \pm 29,400 \mu\text{g}/\text{kg}$ (22% relative standard deviation). The matrix spike recoveries could not be accurately measured because the Aroclor-1248 was spiked at 20,000 $\mu\text{g}/\text{kg}$, which was "overshadowed" by the native PCB and aliquot-to-aliquot inhomogeneity. The second set of sludge samples were only stirred with a spatula (per recommended SW-846 practice) before aliquots were taken. Ultrasonication before aliquotting probably would have produced more homogenous aliquots.

D.3.5 Tentative Identification of Oil Layer Type

The retention time range of the hump in the GC-FID profile of the oil layers in tanks WC-11 and WC-13 matched that of vacuum-pump oil and suggests this identification. The profiles did not match the other oils and fuels tested, which included gasoline, diesel fuel, motor oil, differential fluid, and transmission fluid. Although this tentative identification of the oils as vacuum-pump oil is consistent with laboratory operations (which could have included the use of vacuum-pumps), this analysis does not provide unambiguous identification. An oil of the same distillation range as vacuum-pump oil could produce the same profile.

D.4 CONCLUSIONS

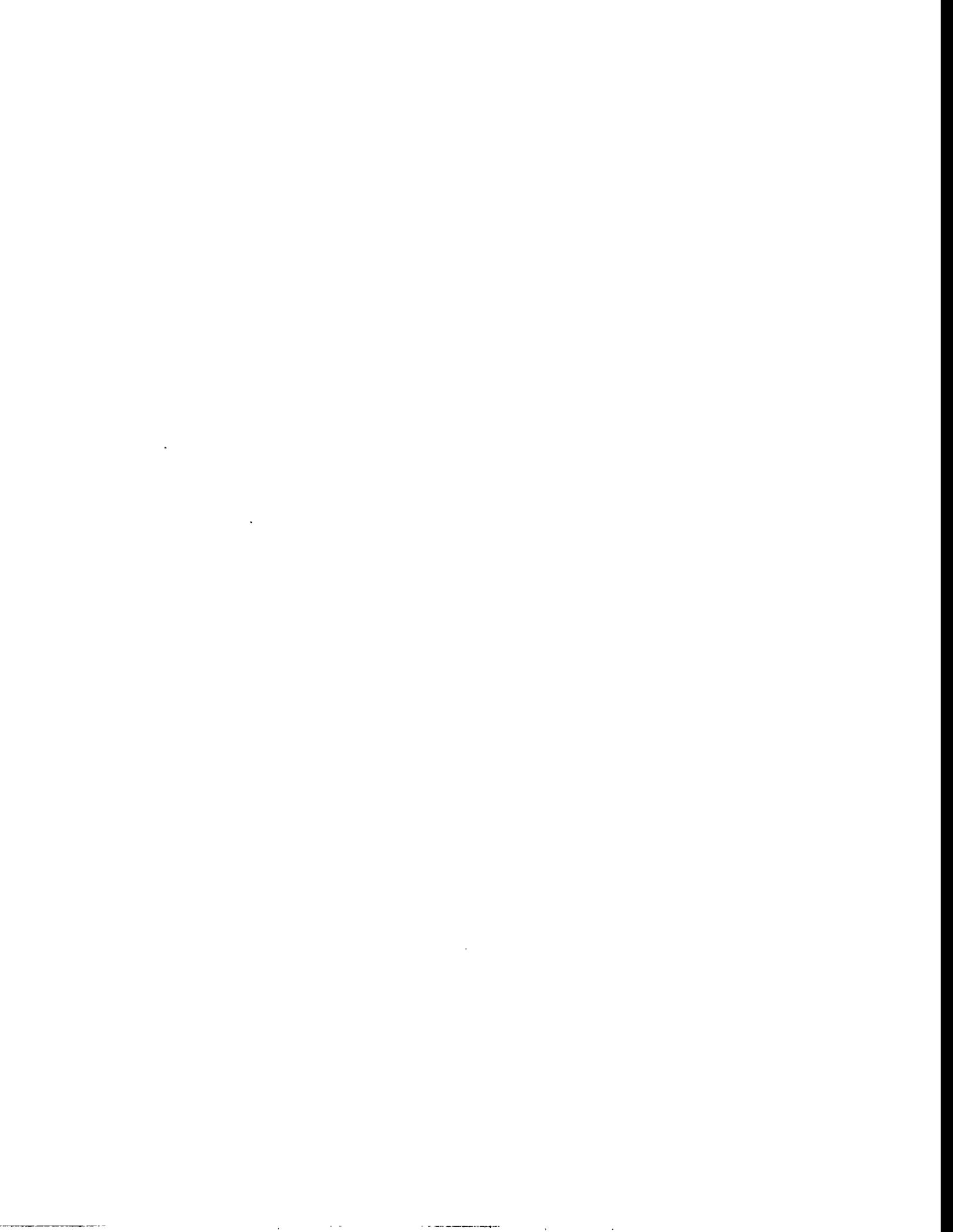
The results of these analyses show that, in general, the aqueous liquids present in these tanks at the times of their sampling did not contain organic compounds of regulatory interest at significant concentrations. The low microgram-per-liter concentrations of solvents in WC-13 and WC-14 may be the exceptions. Except for the oils, there was very little detectable organic material present. The oils appeared to be vacuum-pump oil (or an oil with the same distillation range) and were found to contain low concentrations of Aroclor-1254. In contrast, the sludges contained considerable organic material, including what appeared to be vacuum-pump oil (tank WC-13) and PCBs (especially tank WC-14). The reactivity of nuclear wastes was illustrated by the identification of a nitrated product of an SVOA surrogate standard in one sample (WC12-L3-C filtrate).

D.5 REFERENCES

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Appendix E

LISTING OF ORGANIC ANALYTICAL DATA



The analytical data for the organic compound analyses are presented in this appendix. For an introduction to the tables and a discussion of the results, see Appendix D, Sect. D.3.

Some initialisms and data reporting conventions used in the tables are as follows:

Reporting limits	The reporting limits are the concentrations above which the response of the instrument for a calibrated range of concentrations is linear.
B	Data qualifier meaning that the compound also was found in the accompanying laboratory blank sample.
J	Data qualifier meaning that the compound was estimated at a concentration below the reporting limit. Also used to indicate that the concentrations for TICs are estimates.
MS	Matrix spike
MSD	Matrix spike duplicate
PCB	Polychlorinated biphenyl
QC	Quality control
RPD	Relative percent difference
SVOA	Semivolatile organic compound analysis
TIC	Tentatively identified compound. The identification is based upon the mass spectrum only and the quantitation upon the response factor of the nearest eluting internal standard. All TIC values are estimates and bear the "J" qualifier.
VOA	Volatile organic compound analysis

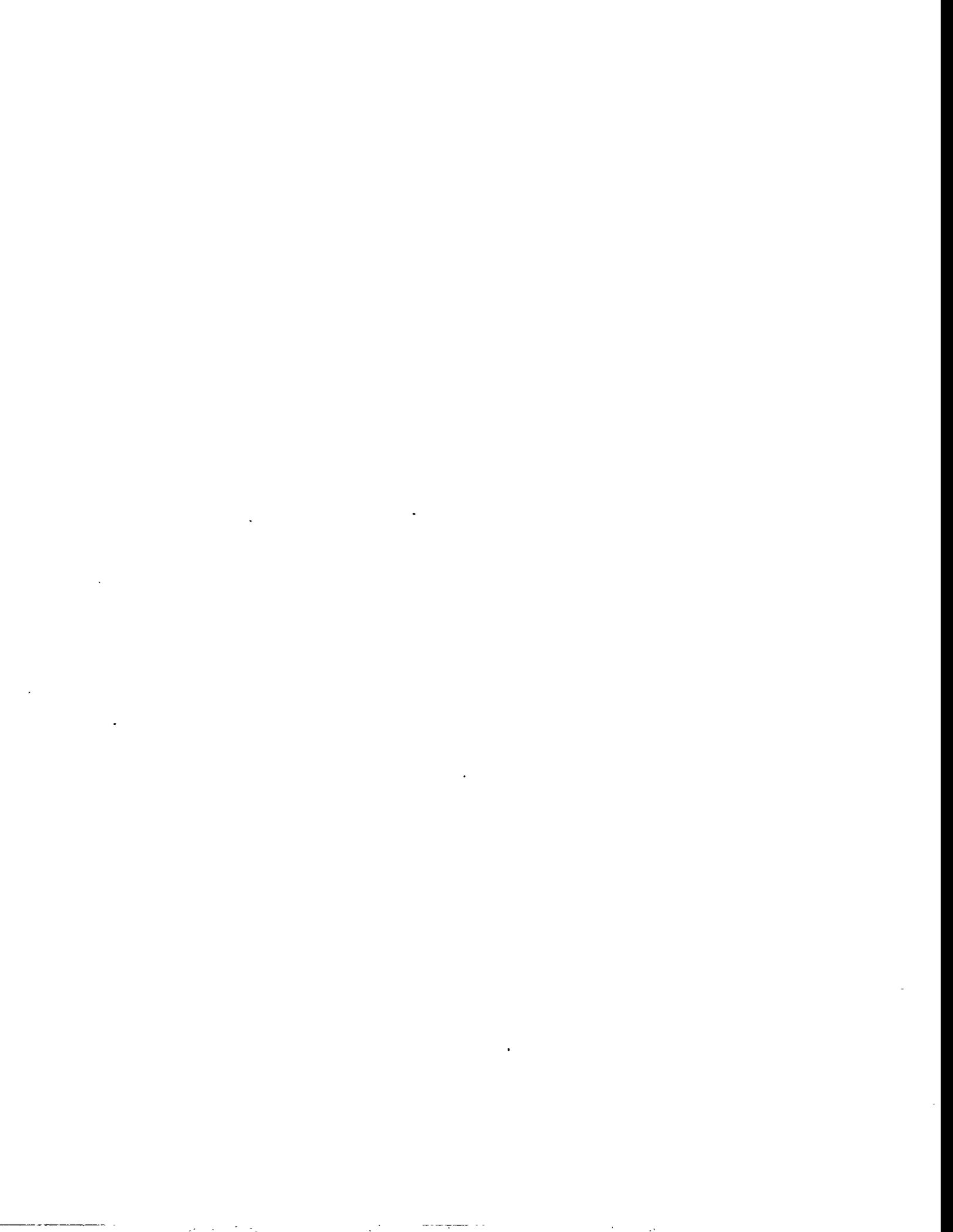


Table E.1. Waste tank sample aliquot volumes/masses and conversion factors for calculating reporting limits

Sample	Sample bottle	pH	Volatile organic compound analyses		Semivolatile organic compound and pesticide/PCB analyses			
			Aliquot volume (mL)	Conversion factor	Aliquot		Conversion factor	
					Volume (mL)	Mass (g)		SVOA
W17-L1	A	11.6	5.0	1	190		5 (a)	5
W17-L1-MS (b)	B	11.6			215		5 (a)	5
W17-L1-MSD (c)	C	11.6			195		5 (a)	5
W18-L1	A	10.0	5.0	1	190		5 (a)	5
W18-L1-MS	B	10.0			210		5 (a)	5
W18-L1-MSD	C	10.0			200		5 (a)	5
Water blank		6.3	5.0	1	1,000		1 (a)	1
WC5-L1	E, F, G	12.9	0.5	10	605		1.6 (a)	1.6
WC6-L1	E, F, G	12.8	1.0	5	612		1.6 (a)	1.6
WC8-L1	E, F, G	13.0	0.5	10	562		1.7 (a)	1.7
Water blank		5.9	0.5	10	1,000		1 (a)	1
WC11-L1	E	9.5	(d)		200		2.5	10
WC11-L1-MS	F	9.5			200		2.5	10
WC11-L1-MSD	G	9.5			200		2.5	10
WC11-Surface (oil layer)	3		(e)	10,000	(e)		5,500	1,050
Solvent blank				10,000			5,500	1,050
WC12-L1	E	10.0	(d)		200		2.5	10
Water blank		6.5			200		2.5	5
WC13-L1	E	>11	(d)		200		25 (f)	50
WC13-Surface (oil layer)	2		(e)	10,000	(e)		5,500	2,100
WC14-L1	E	6.0	(d)		200		25 (f)	50

Table E.1. Waste tank sample aliquot volumes/masses and conversion factors for calculating reporting limits (continued)

Sample	Sample bottle	pH	Volatile organic compound analyses		Semivolatile organic compound and pesticide/PCB analyses				
			Aliquot volume (mL)	Conversion factor	Volume (mL)	Mass (g)	SVOA	Pest./PCBs	
WC12-L3-C					146			3.8	68
Water blank					150			3.7	67
WC13-S (sludge)	Composite sample (g)					10.8		279	28
WC13-S-MS	Composite					10.5		(h)	29
WC13-S-MSD	Composite					11.2		(h)	27
Sand blank						10.0		30	30
WC14-S (sludge)	Composite sample (t)					6.6		45	46
WC14-S-MS	Composite					5.8		51	52
Sand blank						10.1		30	30

(a) The conversion factor for the acid/neutral compounds in the semivolatile organic compound analysis is 10% higher than listed because of the removal of a 10% volume of the acid/neutral fraction for pesticide/PCB analysis (see Sect. D.2.3.2). The alkaline compounds, pesticides, and PCBs are not affected by the split, and the conversion factor is correct as shown.

(b) MS = matrix spike sample.

(c) MSD = matrix spike duplicate sample.

(d) The volatile organic analysis was not run because the sample was too radioactive for a nonzoned laboratory (see Sects. D.1 and D.2.1.1).

(e) Oil layer; 0.2 mL diluted to 2.0 mL and surrogate standards added.

(f) The extract for the semivolatile organic compound analysis required a 10-fold dilution over normal protocol before GC-MS. The extract for the pesticide/PCB analysis was also diluted 10-fold.

(g) Sample WC13-S was a composite of samples WC13-S1 and WC13-S2, collected March 2, 1993.

(h) The matrix spike and matrix spike duplicate samples were not quantitated because of the dilution required to protect the GS-MS from overloading (see Sect. D.2.5.2).

(i) Sample WC14-S was a composite of samples WC14-S1 and WC14-S2, collected March 24, 1993.

Table E.2. Waste tank sample aliquot volumes/masses and conversion factors for PCB analyses of second and third sets of samples from tank WC-14

Sample	Aliquot		Conversion factor
	Volume (mL)	Mass (g)	
WC14-L1 (a)	196		5.1
WC14-Surface (a)	200		5.0
Water blank	200		5.0
WC14-L2-B (b)	180		0.52
WC14-L2-C (b)	195		0.52
Water blank	200		0.50
WC-14S-1 (a)		10.4	2.9
WC-14S-1-MS (a)		10.9	2.8
WC-14S-1-MSD (a)		10.2	2.9
WC-14S-2 (a)		10.4	2.9
Sand blank		10.0	3.0

(a) Second set of samples collected in August 1993.

(b) Third set of samples collected in December 1994.

Table E.3. Compound list and reporting limits for conventional analyses of nonradioactive samples: Volatile organic compound analyses

Compound	Reporting limits	
	Liquid sample, 5-mL aliquot ($\mu\text{g/L}$)	Solid sample, 5-g aliquot ($\mu\text{g/kg}$)
Chloromethane	10	10
Bromomethane	10	10
Vinyl chloride	10	10
Chloroethane	10	10
Methylene chloride	5	5
Acetone	10	10
Carbon disulfide	5	5
1,1-Dichloroethene	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethene (total)	5	5
Chloroform	5	5
1,2-Dichloroethane	5	5
2-Butanone	10	10
1,1,1-Trichloroethane	5	5
Carbon tetrachloride	5	5
Vinyl acetate	10	10
Bromodichloromethane	5	5
1,2-Dichloropropane	5	5
cis-1,3-Dichloropropene	5	5
Trichloroethene	5	5
Dibromochloromethane	5	5
1,1,2-Trichloroethane	5	5
Benzene	5	5
trans-1,3-Dichloropropene	5	5
Bromoform	5	5
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
Tetrachloroethene	5	5
1,1,2,2-Tetrachloroethane	5	5
Toluene	5	5
Chlorobenzene	5	5
Ethylbenzene	5	5
Styrene	5	5
Xylenes (total)	5	5

Table E.4. Compound list and reporting limits for conventional analyses of nonradioactive samples: Semivolatile organic compound analyses

Compound	Reporting limits	
	Liquid sample, 1000-mL aliquot ($\mu\text{g/L}$)	Solid sample, 30-g aliquot ($\mu\text{g/kg}$)
Phenol	10	330
bis(2-Chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
Benzyl alcohol	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
bis(2-Chloroisopropyl)ether	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Benzoic acid	50	1650
bis(2-Chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	10	330
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1650
Dimethylphthalate	10	330
Acenaphthylene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	50	1650
Acenaphthene	10	330

Table E.4. Compound list and reporting limits for conventional analyses of nonradioactive samples: Semivolatile organic compound analyses (continued)

Compound	Reporting limits	
	Liquid sample, 1000-mL aliquot ($\mu\text{g/L}$)	Solid sample, 30-g aliquot ($\mu\text{g/kg}$)
2,4-Dinitrophenol	50	1650
4-Nitrophenol	50	1650
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenylether	10	330
Fluorene	10	330
4-Nitroaniline	50	1650
4,6-Dinitro-2-methylphenol	50	1650
N-Nitrosodiphenylamine	10	330
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	50	1650
Phenanthrene	10	330
Anthracene	10	330
Di-n-butylphthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	20	660
Benzo(a)anthracene	10	330
Chrysene	10	330
bis(2-Ethylhexyl)phthalate	10	330
Di-n-octylphthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenz(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

Table E.5. Compound list and reporting limits for conventional analyses of nonradioactive samples: Pesticide/PCB analyses

Compound	Reporting limits	
	Liquid sample, 1000-mL aliquot ($\mu\text{g/L}$)	Solid sample, 30-g aliquot ($\mu\text{g/kg}$)
Alpha-BHC	0.05	8.0
Beta-BHC	0.05	8.0
Delta-BHC	0.05	8.0
Gamma-BHC (Lindane)	0.05	8.0
Heptachlor	0.05	8.0
Aldrin	0.05	8.0
Heptachlor epoxide	0.05	8.0
Endosulfan I	0.05	8.0
Dieldrin	0.10	16
4,4'-DDE	0.10	16
Endrin	0.10	16
Endosulfan II	0.10	16
4,4'-DDD	0.10	16
Endosulfan sulfate	0.10	16
4,4'-DDT	0.10	16
Methoxychlor	0.50	80
Endrin ketone	0.10	16
Alpha chlordane	0.50	80
Gamma chlordane	0.50	80
Toxaphene	1.0	160
Aroclor-1016	0.50	80
Aroclor-1221	0.50	80
Aroclor-1232	0.50	80
Aroclor-1242	0.50	80
Aroclor-1248	0.50	80
Aroclor-1254	1.0	160
Aroclor-1260	1.0	160

**Table E.6. Compound list and reporting limits for
conventional analyses of nonradioactive samples:
Water-soluble volatile organic
compound analyses**

Compound	Reporting limits for liquid sample, 3- μ L, aliquot (μ g/L)
Methyl alcohol	1000
Ethyl alcohol	1000
Acetone	1000
i-Propyl alcohol	1000
Allyl/n-propyl alcohol	1000
Methylethyl ketone	1000
i-Butyl alcohol	1000
n-Butyl alcohol	1000
Methyl-i-butyl ketone	1000

Table E.7. Summary of results for organic characterization of liquid samples from tanks W-17, W-18, WC-5, WC-6, and WC-8

Analysis class and compound hits, µg/L (a)	W17-L1	W18-L1	Water blank	WC5-L1	WC6-L1	WC8-L1	Water blank
Volatiles organics							
Acetone	39B	37B	17				
Water-soluble organics							
Semivolatiles organics							
Butylbenzylphthalate				2J	2J		
Pesticides/PCBs							
Endosulfan II		0.046J					
α-Chlordane		0.12J					
Aroclor-1254		4J					
Aroclor-1260		1.9J					
Semivolatiles organic TICs (b)							
Unknown	1938J (3)	918J (3)		711J (3)	184J (3)	713J (3)	
Unknown hydrocarbons					13J (1)	11J (1)	

(a) Only compounds whose possible presence was indicated by the analysis are listed. See Tables E.3-E.6 for the target compound lists and Table E.1 for the conversion factors to calculate the reporting limits.

(b) Result is the sum of the tentatively identified compounds (TICs). The number of TICs are shown in parenthesis.

Table E-8. Summary of results for organic characterization of liquid samples from tanks WC-11 through WC-14

Analysis class and compound hits, µg/L (a)	WC11-L1 (b)	WC12-L1 (b)	Water blank	WC13-L1 (b)	WC14-L1 (b)	WC11-Surface (oil layer) (c)	WC13-Surface (oil layer) (c)	Solvent blank (c)
Volatile organics								
Water-soluble organics								
Methyl alcohol				9,600	14,700	(d)	(d)	(d)
n-Butyl alcohol				8,900				
Semivolatile organics								
Diethylphthalate	7J							
Di-n-octylphthalate	17J							
Di-n-butylphthalate			4J				1.7 × 10 ⁷ J	
Benzoic acid					100J			
Pesticides/PCBs								
Aroclor-1248					278 (e)	1,700 (e)		
Aroclor-1254							5,060 (e)	

Table E.8. Summary of results for organic characterization of liquid samples:
from tanks WC-11 through WC-14 (continued)

Analysis class and compound hits, µg/L (a)	WC11-L1	WC12-L1	Water blank	WC13-L1	WC14-L1	WC11-Surface (oil layer)	WC13-Surface (oil layer)	Solvent blank
<i>Semivolatile organic TICs (f)</i>								
Unknown	1,276J (8)	943J (10)		2,480J (3)	2,870J (10)			
Unknown hydrocarbons	124J (7)	74J (5)	33J (3)		1,700J (2)			
Tributylphosphate				470J	9,700J			
Diethylbenzene					280J			

(a) Only compounds whose possible presence was indicated by the analysis are listed. See Tables E.3-E.6 for the target compound lists and Table E.1 for the conversion factors to calculate the reporting limits.

(b) Analysis was not run because the sample was too radioactive for a nonzoned laboratory.

(c) Traces of methylene chloride and hexane were detected. Their presence in the blank suggests cross contamination among the diluted oil samples during transport.

(d) Analysis not appropriate for organic samples.

(e) Some pesticide peaks also were detected, but these are believed to be the result of co-eluting components from the Aroclor mixture, and not pesticides.

(f) Result is the sum of the tentatively identified compounds (TICs). The number of TICs are shown in parenthesis.

Table E.9. Summary of results for organic characterization of sludge and filtrate samples from tanks WC-12 through WC-14

Analysis class and compound hits (a)	WC12-L3-C Filtrate	Water blank	WC13-S Sludge	Sand blank	WC14-S Sludge	Sand blank
<i>Volatiles organics</i>	(b)		(b)		(b)	
<i>Water-soluble organics (µg/L)</i>						
Methyl alcohol	(c)		5,000 (d)		(e)	
n-Butyl alcohol			5,200 (d)			
<i>Semivolatiles organics (µg/L or µg/kg)</i>						
Diethylphthalate	89B	12J				
Di-n-butylphthalate	390		31,000B	4,100J	14,000J	
Bis(2-ethylhexyl)phthalate	25J					
Di-n-octylphthalate	12J					
Naphthalene					3,500J	
<i>Pesticides/PCBs (µg/L or µg/kg)</i>						
Heptachlor					7,370	
Atroclor-1248					119,000	
Atroclor-1254			1,630J (f)			

Table E.9. Summary of results for organic characterization of sludge and filtrate samples from tanks WC-12 through WC-14 (continued)

Analysis class and compound hits (a)	WC12-L3-C Filtrate	Water blank	WC13-S Sludge	Sand blank	WC14-S Sludge	Sand blank
<i>Semivolatile organic TICs (µg/L or µg/kg)</i>						
Unknown (g)	7,540J (18)		377,000J (6)		734,000J (7)	
Unknown hydrocarbons (g)					674,200J (7)	
Tributylphosphate	630J		47,000J		270,000J	
C4-Benzene (g)					95,200J (2)	
Propanol derivative	64J					
2,6-Dibromo-4-nitrophenol	54J					
Dibutylphthalate	66J					
Trichlorobiphenyl (g)					25,500J (2)	
Tetrachlorobiphenyl (g)					15,600J (2)	
Pentachlorobiphenyl					8,000J	
<i>Oil analysis (wt %)</i>						
Vacuum-pump oil range						

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(a) Only compounds whose possible presence was indicated by the analysis are listed. See Tables E.3-E.6 for the target compound lists and Table E.1 for the conversion factors to calculate the reporting limits.

(b) Analysis was not run because the sample was too radioactive for a nonzoned laboratory.

(c) Analysis was not run because of instrument breakdown.

(d) Analysis run on interstitial liquid in sludge; units are µg/L of liquid.

(e) Analysis was not run because of insufficient sample to collect interstitial liquid.

(f) Some pesticide peaks also were observed, but these are believed to be the result of co-eluting components from the Aroclor mixture, and not pesticides.

(g) Result is the sum of the tentatively identified compounds (TICs). The number of TICs are shown in parenthesis.

Table E.10. PCB analyses of second and third sets of samples from tank WC-14

Phase	Sample	Aroclor-1248
Liquid	WC14-L1 (a)	4.7 µg/L
	WC14-Surface (a)	6.9 µg/L
	Water blank	<2.5 µg/L
	WC14-L2-B (b)	13.2 µg/L
	WC14-L2-C (b)	10.9 µg/L
	Water blank	<0.25 µg/L
	Sludge	WC-14S-1 (a)
	WC-14S-1-MS (a)	126,000 µg/kg (c)
	WC-14S-1-MSD (a)	163,000 µg/kg (c)
	WC-14S-2 (a)	241,000 µg/kg
	Sand blank	59 µg/kg

(a) Second set of samples collected in August 1993.

(b) Third set of samples collected in December 1994.

(c) "Native" Aroclor-1248 in the aliquot after correction for that added by the matrix spike.

Table E.11. Surrogate standard percent (%) recoveries for liquid samples from tanks W-17, W-18, WC-5, WC-6, and WC-8

Class	Surrogate standard	QC limits (a)	W17-L1	W18-L1	Water blank	WC5-L1	WC6-L1	WC8-L1	Water blank
VOA	Toluene-d ₈	88-110	101	101	103	(b)	(b)	(b)	(b)
	Bromofluorobenzene	86-115	103	102	105				
	1,2-Dichloroethane-d ₄	76-114	96	94	97				
SVOA	Nitrobenzene-d ₅	35-114	61	58	73	48	56	50	49
	2-Fluorobiphenyl	43-116	59	53	62	47	52	46	47
	Terphenyl-d ₁₄	33-141	87	83	87	70	73	67	75
	Phenol-d ₅	10-94	22	21	30	20	21	20	22
	2-Fluorophenol	21-100	39	34	43	34	38	32	30
	2,4,6-Tribromophenol	10-123	71	73	71	71	70	74	57
Pesticides and PCBs	Decachlorobiphenyl (DCBP)		85 (c)	77	85 (c)	53	60	61	63

(a) The QC limits for surrogate standard recoveries are from the U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste, SW-846*, 3rd ed., Update I, July 1992.

(b) A gas chromatography screen for volatile organic compounds was conducted. The internal standard recoveries were acceptable.

(c) Extracts of sample W17-L1 and the blank were inadvertently mixed and analyzed together.

Table E.12. Surrogate standard percent (%) recoveries for liquid samples from tanks WC-11 through WC-14

Class	Surrogate standard	QC limits (a)	WC11-L1	WC12-L1	Water blank	WC13-L1	WC14-L1	WC11-Surface, oil layer	WC13-Surface, oil layer	Hexane blank
VOA	Toluene-d ₈	88-110	(b)	(b)		(b)	(b)	(c)	(c)	(c)
	Bromofluorobenzene	86-115								
	1,2-Dichloroethane-d ₄	76-114								
SVOA	Nitrobenzene-d ₅	35-114	72	71	61	(d)	(d)	11	11	111
	2-Fluorobiphenyl	43-116	70	67	60			21	21	207
	Terphenyl-d ₁₄	33-141	95	85	77			18	19	159
	Phenol-d ₅	10-94	26	24	34			14	16	166
	2-Fluorophenol	21-100	36	36	44			11	14	141
	2,4,6-Tribromophenol	10-123	92	84	73			2	131	24
Pesticides and PCBs	Decachlorobiphenyl (DCBP)		55	81	66	100	77	141	175	141

(a) The QC limits for surrogate standard recoveries are from the U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste, SW-846*, 3rd ed., Update I, July 1992.

(b) The VOA was not run because the sample was too radioactive for a nonzoned laboratory.

(c) A gas chromatography screen for volatile organic compounds was conducted. The internal standard recoveries were acceptable.

(d) The SVOA surrogate standard recoveries could not be measured because of the sample extract dilution required for GC-MS. This procedure resulted in the dilution of the internal standards below the calibration range.

Table E.13. Surrogate standard percent (%) recoveries for sludge and filtrate samples

Class	Surrogate standard	QC limits (a)		WC12-L3-C Filtrate	Water blank	WC13-S Sludge	Sand blank	WC14-S Sludge	Sand blank
		Water	Soil						
VOA	Toluene-d ₈	88-110	81-117	(b)		(c)		(c)	
	Bromofluorobenzene	86-115	74-121						
	1,2-Dichloroethane-d ₄	76-114	70-121						
SVOA	Nitrobenzene-d ₅	35-114	23-120	56	53	(d)		(d)	
	2-Fluorobiphenyl	43-116	30-115	52	44				
	Terphenyl-d ₁₄	33-141	18-137	78	48				
	Phenol-d ₅	10-94	24-113	0	34				
	2-Fluorophenol	21-100	25-121	1	47				
	2,4,6-Tribromophenol	10-123	19-122	31	62				
Pesticides and PCBs	Decachlorobiphenyl (DCBP)			86	77	126	93	100	112

(a) The QC limits for surrogate standard recoveries are from the U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed., Update I, July 1992.

(b) The VOA was not run because the sample was too radioactive for a nonzoned laboratory.

(c) The VOA was not run because of insufficient sample quantity and high radioactivity.

(d) The SVOA surrogate standard recoveries could not be measured because of the sample extract dilution required to protect the gas chromatograph—mass spectrometer from the complex mixture of organics. This procedure resulted in the dilution of the internal standards below the calibration range.

Table E.14. Surrogate standard percent (%) recoveries
in PCB analyses of second and third sets of
samples from tank WC-14

Phase	Sample	Decachlorobiphenyl recovery
Liquid	WC14-L1 (a)	57
	WC14-Surface (a)	47
	Water blank	79
	WC14-L2-B (b)	73
	WC14-L2-C (b)	76
	Water blank	52
	Sludge	WC-14S-1 (a)
WC-14S-1-MS (a)		150
WC-14S-1-MSD (a)		105
WC-14S-2 (a)		107
Sand blank		104

(a) Second set of samples collected in August 1993.

(b) Third set of samples collected in December 1994.

Table E.15. Matrix spike (MS) and matrix spike duplicate (MSD) percent (%) recoveries and relative percent differences (RPDs) for duplicates:
Liquid samples

Class	Matrix spike	QC limits (σ)			W17-L1			W18-L1			WC11-L1		
		Recovery	RPD	MS	MSD	RPD	MS	MSD	RPD	MS	MSD	RPD	
VOA	1,1-Dichloroethene	61-145	14	89	88	1							
	Trichloroethene	71-120	14	99	95	4							
	Benzene	76-127	11	100	97	3							
	Toluene	76-125	13	100	99	1							
	Chlorobenzene	75-130	13	100	99	1							
SVOA	Phenol	12-89	42	23	28	19	22	19	14	36	32	12	
	2-Chlorophenol	27-123	40	67	79	16	62	65	4	72	56	25	
	1,4-Dichlorobenzene	36-97	28	56	63	11	48	38	23	67	61	9	
	N-nitroso-di-n-propylamine	41-116	38	79	88	10	72	70	2	94	83	12	
	1,2,4-Trichlorobenzene	39-98	28	65	74	12	57	45	23	78	72	8	
	4-Chloro-3-methylphenol	23-97	42	79	97	20	85	94	10	67	61	9	
	Acenaphthene	46-118	31	71	71	0	70	61	13	78	72	8	
	4-Nitrophenol	10-80	50	21	28	28	24	28	15	61	52	16	
	2,4-Dinitrotoluene	24-96	38	56	67	17	61	66	7	78	72	8	
	Pentachlorophenol	9-103	50	66	79	17	70	81	14	94	94	0	
	Pyrene	26-127	31	85	86	1	82	85	3	94	94	0	

**Table E.15. Matrix spike (MS) and matrix spike duplicate (MSD) percent (%) recoveries and relative percent differences (RPDs) for duplicates:
Liquid samples (continued)**

Class	Matrix spike	QC limits (a)			W17-L1			W18-L1			WC11-L1		
		Recovery	RPD	MS	MSD	RPD	MS	MSD	RPD	MS	MSD	RPD	
Pesticides and PCBs	γ -BHC	56-123	15	69	54	24	73	68	7	0	0	0	
	Heptachlor	40-131	20	74	67	10	70	59	17	35	20	54	
	Aldrin	40-120	22	65	55	17	60	46	26	56	34	49	
	Dieldrin	52-126	18	85	75	13	78	68	14	0	0	0	
	Endrin	56-121	21	86	76	12	79	75	5	0	0	0	
	DDT	38-127	27	85	80	6	84	81	4	5	9	70	
	DCBP (surrog. std.)	(b)		92	88	4	83	69	18	94	55	52	
Water-soluble volatile organics	Methyl alcohol	(b)					86	67				25	
	Ethyl alcohol						92	93				1	
	Acetone						93	93				0	
	i-Propyl alcohol						94	95				1	
	Allyl alcohol						95	94				1	
	Methylethyl ketone						96	93				3	
	i-Butyl alcohol						96	97				1	
	n-Butyl alcohol						97	98				1	
	Methyl-i-butyl ketone						92	92				0	

(a) The QC limits for matrix spike recoveries and RPDs are from the U. S. Environmental Protection Agency Contract Laboratory Program (CLP), "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration," February 1988.

(b) There are no QC limits for these compounds in the CLP statement of work.

Table E.16. Matrix spike (MS) and matrix spike duplicate (MSD) percent (%) recoveries and relative percent differences (RPDs) for duplicates:
Sludge samples

Class	Matrix spike	QC limits (a)		WC13-S (b)		WC14-S (b), (c)		
		Recovery	RPD	MS	MSD	RPD	MS	
SVOA	Phenol	26-90	35	(d)			69	
	2-Chlorophenol	25-102	50				55	
	1,4-Dichlorobenzene	28-104	27				34	
	N-nitroso-di-n-propylamine	41-126	38				66	
	1,2,4-Trichlorobenzene	38-107	23				48	
	4-Chloro-3-methylphenol	26-103	33				12	
	Acenaphthene	31-137	19				67	
	4-Nitrophenol	11-114	50				40	
	2,4-Dinitrotoluene	28-89	47				51	
	Pentachlorophenol	17-109	47				0	
	Pyrene	35-142	36				62	
	Pesticides and PCBs	DCBP (surrog. std.)	(e)	166		127	(f)	91

Table E.16. Matrix spike (MS) and matrix spike duplicate (MSD) percent (%) recoveries and relative percent differences (RPDs) for duplicates: Sludge samples (continued)

Class	Matrix spike	QC limits (a)			WC13-S (b)		WC14-S (b), (c)	
		Recovery	RPD	MS	MSD	RPD	MS	
Water-soluble volatile organics	Methyl alcohol	(e)		106	109	3	(g)	
	Ethyl alcohol			91	93	2		
	Acetone			97	108	11		
	i-Propyl alcohol			97	115	17		
	Allyl alcohol			89	101	13		
	Methylethyl ketone			98	101	3		
	i-Butyl alcohol			99	111	11		
	n-Butyl alcohol			101	97	4		
	Methyl-i-butyl ketone			97	112	14		

(a) The QC limits for matrix spike recoveries and RPDs are from the U. S. Environmental Protection Agency Contract Laboratory Program (CLP), "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration," February, 1988.

(b) The pesticide matrix spikes could not be measured because of interferences from the Aroclor mixture.

(c) Insufficient sample for a matrix spike duplicate.

(d) The SVOA matrix spikes could not be measured because of the dilution required to protect the gas chromatograph-mass spectrometer from overloading (see Sect. D.2.5.2). This procedure resulted in the dilution of the spikes below the calibration range.

(e) There are no QC limits for these compounds in the CLP statement of work.

(f) The RPD is not calculated for surrogate standards.

(g) The water-soluble volatile organics analysis was not conducted because there was insufficient sample to collect the interstitial liquid.

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