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**ENVIRONMENTAL
RESTORATION
PROGRAM**

**Sampling and Analysis of the Inactive
Waste Tanks TH-2, WC-1, and WC-15**

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W. H. Griest
J. L. Botts
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MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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Environmental Restoration Division
ORNL Environmental Restoration Program

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Date Issued—February 1992

Prepared by
Office of Environmental, Safety, and Health Compliance
Oak Ridge National Laboratory

Prepared for
U.S. Department of Energy
Office of Environmental Restoration and Waste Management
under budget and reporting code GF 72

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6285
managed by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
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under contract DE-AC05-84OR21400

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ACKNOWLEDGMENTS

The authors acknowledge the many divisions that supported the sampling and analytical efforts during this project. Members of the Analytical Chemistry Division including J. E. Caton, M. P. Maskarinec, G. S. Fleming, S. H. Harmon, S. M. Smith, R. M. Edwards, J. C. Montgomery, and S. D. Glover made important contributions to the organic analytical efforts. Other staff from the Analytical Chemistry Division who contributed to the inorganic and radiochemical analytical support include C. J. Baggett, D. A. Caquelin, L. R. Hall, H. J. Hall, K. O. Jeters, R. A. Jones, B. K. Larkins, M. G. McClung, R. T. Pack, R. F. Peacher, J. M. Peele, C. W. Scott, J. E. Sutherland, and L. C. Tabor.

The authors are also indebted to the sampling services performed by Bechtel National, Inc., and C. B. Scott and J. D. Brewer of the Waste Management Operations Group within the Office of Environmental, Safety, and Health Compliance. K. W. Cook, S. B. Garland II, D. D. Huff, R. W. Sharpe, and J. R. Trabalka assisted in the technical and editorial review of this report, for which the authors are most grateful.

EXECUTIVE SUMMARY

Thirty-eight inactive liquid low-level radioactive waste tanks are currently managed by the Environmental Restoration Program of Oak Ridge National Laboratory. The contents of these tanks are to be characterized in preparation for future corrective actions and remediation activities as part of compliance with the pending Federal Facility Agreement for the Oak Ridge Reservation. Twenty-nine of these tanks were sampled and analyzed in 1989. Three of the tanks (TH-2, WC-1, and WC-15) were not accessible from the surface and thus were not sampled until 1990. This report presents the sampling and analytical results of that campaign.

All three tanks in this report had negligible regulatory organic compounds in the samples that were collected. There were no U.S. Environmental Protection Agency (EPA) Target Compound List (TCL) constituents for volatile organics detected in any of the aqueous samples. The only semivolatile organics detected were 2-chlorophenol (52 $\mu\text{g/L}$) in tank TH-2 and dichloroethene (14–15 $\mu\text{g/L}$) and diethyl ether (15–17 $\mu\text{g/L}$) in tank WC-15. A thin oil layer was discovered floating on top of the aqueous contents in tank WC-15. The analysis of the oil layer detected no volatile organics and showed only one EPA TCL constituent, di-*n*-butylphthalate, at 1900 $\mu\text{g/L}$.

Low levels of Resource Conservation and Recovery Act (RCRA) metals were observed in the samples from tank TH-2, but only the mercury level exceeded the RCRA limit. Samples from tank WC-1 had elevated levels of the RCRA metals barium, chromium, and lead. There were also finely suspended particles in one of the samples from tank WC-1, which was filtered and analyzed separately. This solid fines have levels of transuranium elements ^{238}Pu and ^{241}Am high enough to be classified as transuranic waste.

1. SITE DESCRIPTION AND SAMPLE COLLECTION

1.1 INTRODUCTION

This report describes the sampling and analyses of the three previously inaccessible inactive liquid low-level radioactive waste (LLLW) tanks at Oak Ridge National Laboratory (ORNL) during 1990. These three tanks, TH-2, WC-1, and WC-15, are located in ORNL's main plant area (Fig. 1.1). The purpose of the sampling and analyses is to characterize the tank contents for future corrective actions and remediation activities as part of compliance with the pending Federal Facility Agreement for the Oak Ridge Reservation. The sampling methods for these tanks are the same as those used during the sampling campaign in 1989.¹ These tanks were not sampled during the 1989 campaign because of accessibility problems. This report completes the analyses of the 33 inactive LLLW tanks identified during initial investigations.¹ Additional inactive LLLW tanks have been identified, and they will be sampled in later fiscal years.

This section describes the tank sites and the sample collection activities associated with each of the tanks. Section 2 describes the organic analytical characterization. The third section describes the radiochemical and inorganic characterization of the tank contents. Section 4 summarizes the findings from all three tanks.

1.2 TANK TH-2

Tank TH-2 is located just south of Building 3503 and north of White Oak Avenue in a tank farm with six other tanks, four of which are still in use. This tank was placed in service in 1952 and received waste from the Thorium Pilot-Plant Project, located in Building 3503. In 1970 it was taken out of service and reportedly emptied. TH-2 is a small, vertical stainless steel tank with a diameter of 7 ft, a height of 10 ft, and a capacity of approximately 2400 gal. A riser pipe installed from the top of the tank to the surface provided access for sampling (Fig. 1.2). This tank was not sampled during the 1989 campaign because of earlier reports of radiation levels at 6 rem/h at the tank access without the lead shielding over the manhole. However, a survey conducted in February 1989 found the radiation level to be 90 mrem/h 1 in. below the top of the lead shielding at the surface.

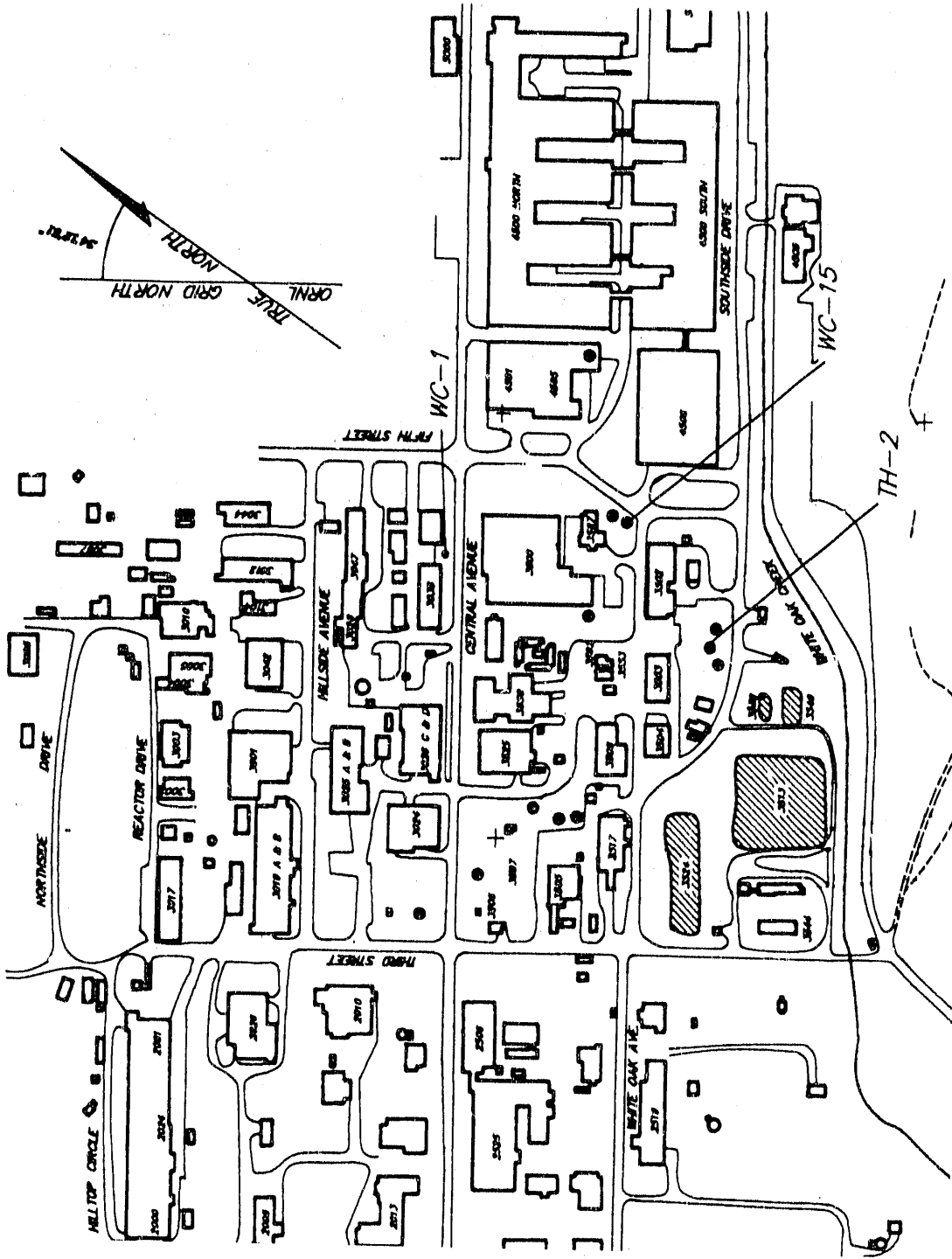


Fig. 1.1. Oak Ridge National Laboratory main plant area.

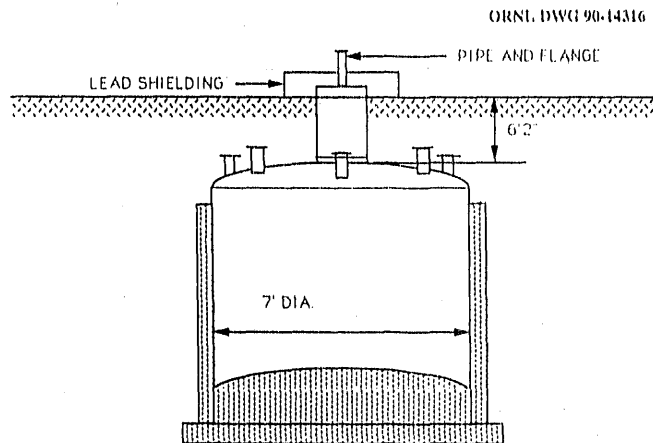


Fig. 1.2. Tank TH-2.

Tank TH-2 was sampled in February 1990. Only 3 in. (72 gal) of liquid were found in the bottom of the tank; no sludge was detected. The background readings were 1000 mrem/h at ground surface and 200-300 mrem/h at the top of the tank located 6 ft below ground. The liquid samples collected from the tank read 1 mrem/h on contact. The low activity of the tank liquids confirms the reports that the tank was flushed and emptied prior to removal from service. Surprisingly, bromide was detected in the samples; this suggests that a hot-cell window may have been emptied into the tank. However, zinc was not detected in the samples.

1.3 TANK WC-1

Tank WC-1 is a small, vertical, stainless steel tank located between Buildings 3038 and 3037. This 2500-gal tank has an outside diameter of 8.75 ft and a height of approximately 6 ft. Tank WC-1 was placed in service in 1950 and was used for collecting and monitoring process liquid waste from isotope production and development laboratories in Buildings 3038, 3028, 3029, 3030, 3031, 3032, 3033, 3047, Building 3110 filter house, stack 3039, and scrubber 3092. The tank was abandoned in 1968 because of a leaking discharge line and was reportedly emptied upon abandonment. Tank WC-1 was not sampled during the 1989 campaign because of tank inaccessibility and safety concerns resulting from potential soil contamination. In October 1989, soil samples were collected at the site. Samples from the first 6 ft were clean (no detectable alpha or beta activity). Beta-gamma readings ranged from 0.2 to 110 mrem/h to a depth of 10 ft and showed no detectable alpha (Table 1.1).

Prior to sampling, no above-ground access was available to this tank. Excavation activities² to access tank WC-1 began April 1990 and were completed 4 weeks later. Tank excavation and shoring were impeded when piping was encountered (Fig. 1.3). After the tank flange was uncovered, groundwater infiltrated the excavation. The groundwater was pumped to the process waste system. A riser was installed from flange No. 4 to the surface (Fig. 1.4), and the site was backfilled. Tank sampling indicated that 375 gal of liquid remains

Table 1.1. Soil radiological screening results from WC-1 riser installation

Depth (ft)	Date	Gross alpha (Bq/g)	Gross beta (Bq/g)	¹³⁷ Cs (Bq/g)	⁴⁰ K (Bq/g)	⁶⁰ Co (Bq/g)	²⁴¹ Am (Bq/g)	²² Na (Bq/g)
0.0	4-25-90	<0.48	2.331	0.152	0.999	<i>a</i>	<i>a</i>	<i>a</i>
1.0	4-25-90	<0.481	1.295	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>
2.0	4-26-90	<0.444	5.920	1.998	1.036	<i>a</i>	<i>a</i>	<i>a</i>
3.0	4-26-90	<0.437	1.998	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>
4.0	4-27-90	<0.429	2.960	0.081	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>
5.0	4-30-90	<0.477	1.036	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>
6.0	5-01-90	<0.414	96.200	0.264	0.966	3.093	<i>a</i>	<i>a</i>
6.5	5-01-90	<0.814	166.500	155.770	<i>a</i>	3.245	0.699	0.082
7.0	5-01-90	0.444	77.700	38.850	0.999	2.745	<i>a</i>	<i>a</i>
8.0	5-02-90	0.426	9.620	2.109	0.607	0.459	<i>a</i>	<i>a</i>
9.0	5-03-90	0.411	5.550	0.148	0.929	<i>a</i>	<i>a</i>	<i>a</i>
10.0	5-03-90	0.429	3.219	0.496	0.466	<i>a</i>	<i>a</i>	<i>a</i>
10.3	5-03-90	0.477	7.460	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>	<i>a</i>

^aNot detected.

in the tank. Two samples were collected from the top and bottom of the liquid layer. The bottom liquid sample was colored dark because of suspended fines, and the sample was separated at the laboratory into a liquid and solid sample for separate analyses.

1.4 TANK WC-15

Tank WC-15 is located south of Building 3587 and west of Building 4508, near the intersection of Fifth Street and White Oak Avenue, on a concrete pad that is common to six other tanks, one (WC-17) of which is inactive. Tank WC-15 is a small, vertical, stainless steel tank with an outside diameter of 5 ft, a height of 6.8 ft, and a capacity of 1000 gal. This tank was placed in service in 1951 and received waste from research laboratories in Building 4500. The tank was removed from service in 1960 because of leaks and is currently known to be in-leaking at the seams and the dish head. The tank was reportedly emptied at the time it was removed from service, and the piping was isolated to preclude further use. Because of this, the tank was not accessible from the ground surface and was not sampled during the 1989 campaign. Soil cores taken over WC-15 in October 1989 found no contamination all the way down to the top of the tank. However, probe readings were 32 mrem/h on contact with the tank.

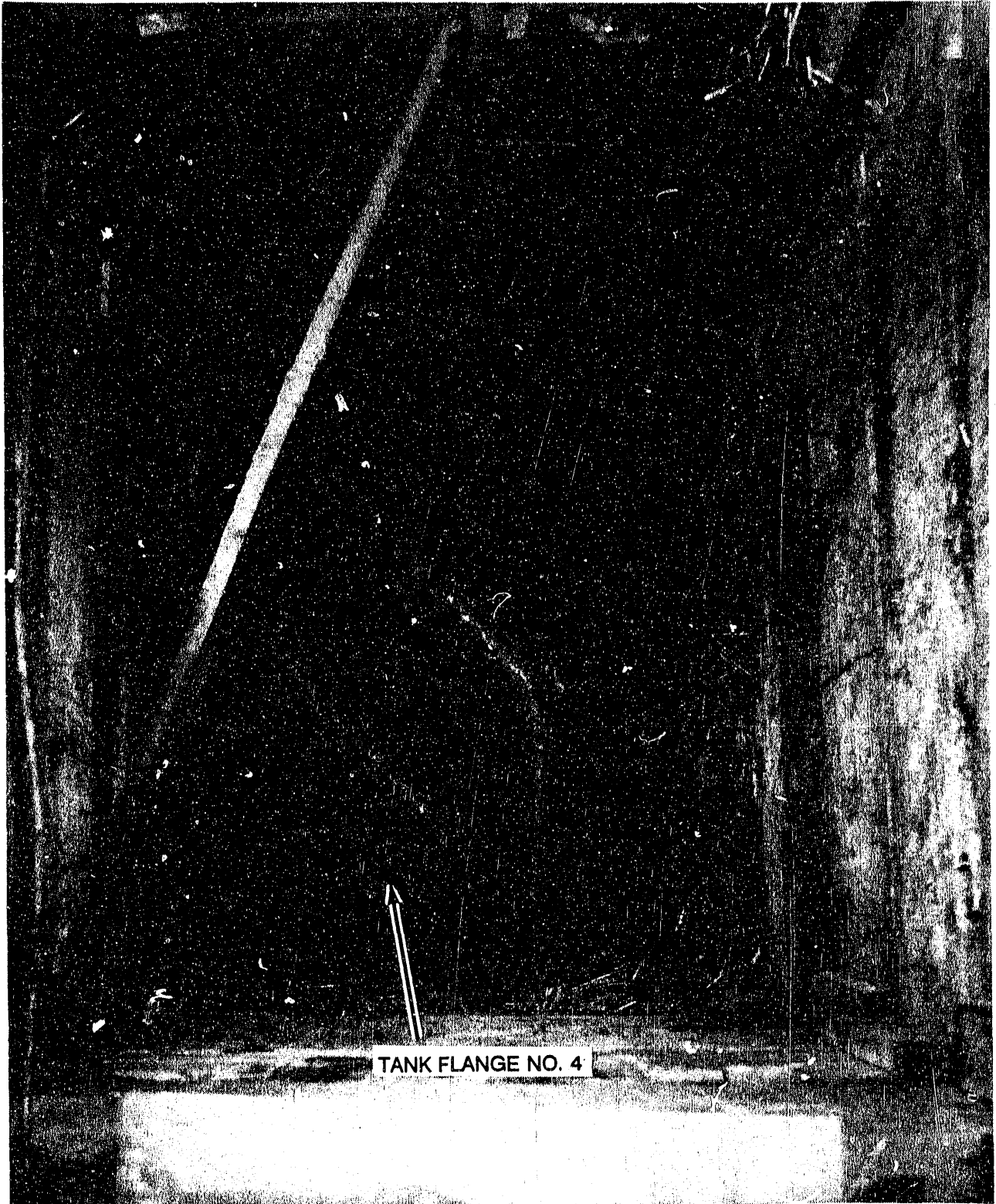


Fig. 1.3. Excavation to tank WC-1.

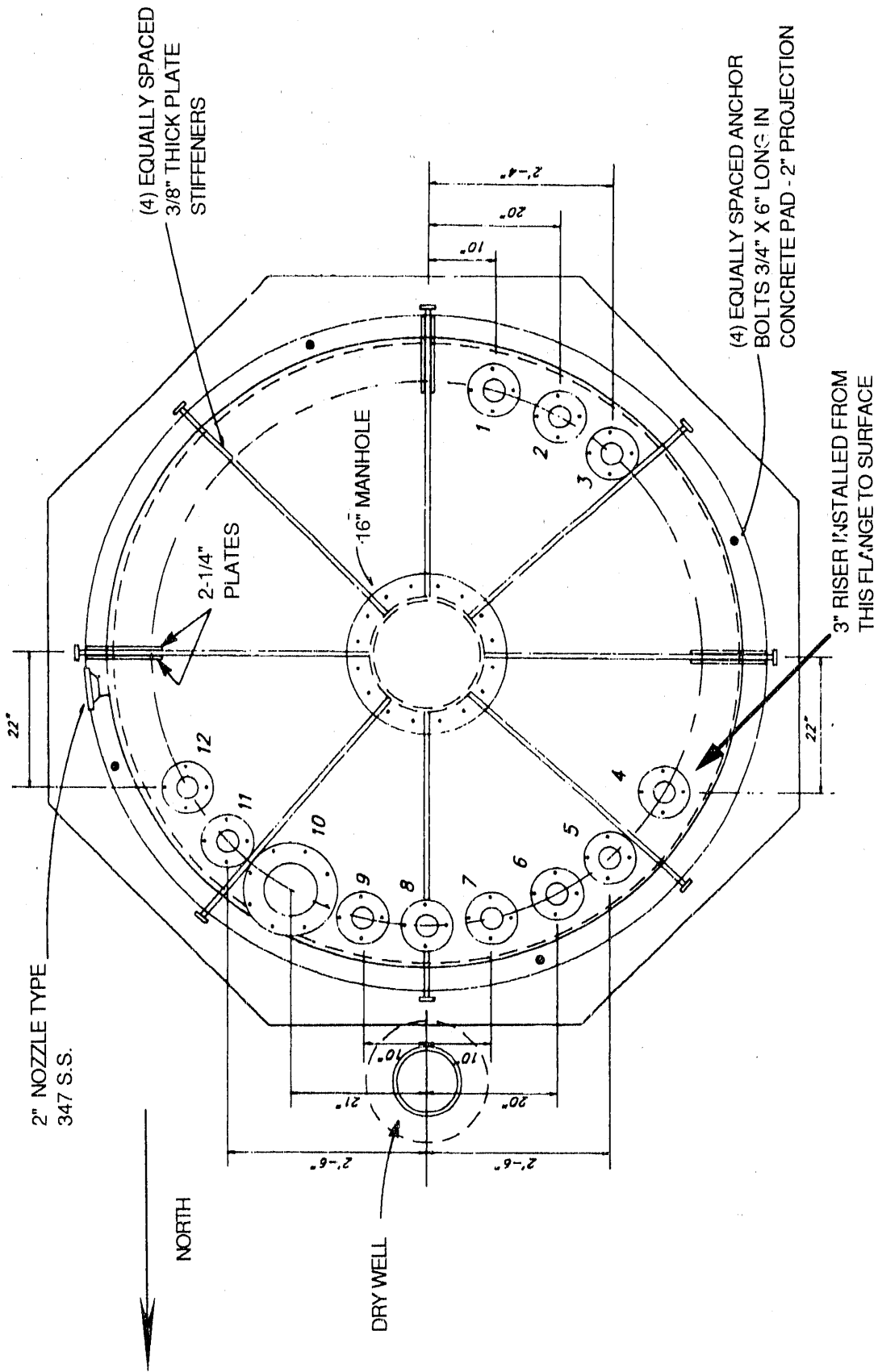


Fig. 1.4. Top view of tank WC-1.

Because of the active tanks within the area of tank WC-15, it was decided to auger rather than to excavate to WC-15 in order to install a riser to the surface.² During the site preparation, the coordinates for the centerline of tank WC-15 (from ORNL drawing D-8670, rev. 2) were discovered to be incorrect. The area was resurveyed, and the coordinates were found to be N-21730.0 and E-31771.0. Again, no contamination was found during the augering process (Table 1.2). Augering was initiated in June 1990, and the riser was installed 7 d later. Upon sampling, the tank was discovered to be completely full of liquid (1000 gal), and no sludge was detected. Four liquid samples were taken from different levels in the tank. The sample from the top of the tank had an organic layer that appeared to be ninety-weight oil. Each of the four samples had a contact reading of only 1 mrem/h.

Table 1.2. Soil radiological screening results from WC-15 riser installation

Depth (ft)	Date	Gross alpha (Bq/g)	Gross beta (Bq/g)	¹³⁷ Cs (Bq/g)	⁴⁰ K (Bq/g)
0.0	5-29-90	<0.463	<0.736	0.029	0.529
1.0-2.0	6-04-90	<0.426	1.147	<i>a</i>	<i>a</i>
2.0-3.17	6-05-90	<0.315	1.110	<i>a</i>	<i>a</i>
3.17-4.0	6-05-90	<0.477	1.480	<i>a</i>	<i>a</i>
4.0-5.0	6-05-90	<0.488	1.073	<i>a</i>	<i>a</i>
5.0-6.25	6-05-90	<0.429	1.628	<i>a</i>	<i>a</i>
6.25-7.0	6-06-90	0.518	1.073	<i>a</i>	<i>a</i>
7.0-8.0	6-06-90	<0.301	1.480	<i>a</i>	<i>a</i>
8.0-8.83	6-06-90	0.629	0.814	<i>a</i>	<i>a</i>
8.83-10.5	6-07-90	0.592	1.554	<i>a</i>	<i>a</i>
10.5	6-08-90	0.259	1.628	<i>a</i>	<i>a</i>

^aNot detected.

2. ORGANIC ANALYTICAL METHODS AND CHARACTERIZATION

2.1 SAMPLE PREPARATION AND ANALYSIS METHODS

The aqueous samples collected from tanks TH-2, WC-1, and WC-15 were subjected to organic compound analyses by modified EPA SW-846 methods.³ Modifications of SW-846 methods 5030 and 3510 were used for the preparation of aqueous liquids for subsequent determination of volatile organic compounds and semivolatile organic compounds respectively. The final analyses used U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) gas chromatography—mass spectrometry (GC-MS) methods equivalent to SW-846 methods 8240 and 8270 respectively. Major volatile organic compounds not amenable to purge-and-trap GC-MS were analyzed in a contamination-zoned laboratory using a direct aqueous injection GC method similar to SW-846 method 8015. This method determines highly water soluble alcohols and ketones that are not detected or that are difficult to accurately measure by the purge-and-trap GC-MS method. These compounds include methanol, ethanol, *i*-propanol, allyl alcohol and/or *n*-propyl alcohol, *i*-butanol, *n*-butanol, acetone, methylethyl ketone, and methylisobutyl ketone. Organic layers were analyzed for volatile organic compounds, semivolatile organic compounds, and polychlorinated biphenyls (PCBs) by a modification of SW-846 waste dilution method 3580 followed by CLP equivalents to SW-846 methods 8240, 8270, and 8080 respectively.

Modifications to the EPA SW-846 methods were necessary to minimize worker radiation exposure and radioactive contamination of equipment and instruments. The sample preparation was conducted in radiation contamination-zoned facilities, and the final analyses for volatile and semivolatile organic compounds were carried out in conventional laboratories using unmodified CLP determinative methods. The protocols included the use of surrogate standards in each sample, of matrix spiked samples, and of blanks. More detailed descriptions of the analytical methods have been published elsewhere.⁴⁻⁶

The reporting limits of the specific EPA Target Compound List (TCL) volatile and semivolatile organic constituents are listed in Tables 2.1 and 2.2 respectively. The reporting limits for the volatile organic compounds analyses were 5–10 $\mu\text{g/L}$ for aqueous liquids and 10,000–20,000 $\mu\text{g/kg}$ for the organic layer. For the semivolatile organic compounds analyses, the corresponding reporting limits are 250–1300 $\mu\text{g/L}$ and 20,000–100,000 $\mu\text{g/kg}$ respectively. The detection limits for the alcohols and ketones determined by the direct aqueous injection GC method are 1000 $\mu\text{g/L}$ for each compound except for allyl alcohol/*n*-propyl alcohol, for which the detection limit is 2000 $\mu\text{g/L}$. The reporting limit for the PCB analysis of the organic layer is 2 $\mu\text{g/g}$.

Table 2.1. Reporting limits for volatile organic compounds in purge and trap GC-MS analysis

Compound	Reporting limit	
	Aqueous samples ($\mu\text{g/L}$)	Organic liquids ^a ($\mu\text{g/kg}$)
Chloromethane	10	20,000
Bromomethane	10	20,000
Vinyl Chloride	10	20,000
Chloroethane	10	20,000
Methylene Chloride	5	10,000
Acetone	10	20,000
Carbon Disulfide	5	10,000
1,1-Dichloroethene	5	10,000
1,1-Dichloroethane	5	10,000
1,2-Dichloroethene (total)	5	10,000
Chloroform	5	10,000
1,2-Dichloroethane	5	10,000
2-Butanone ^b	10	20,000
1,1,1-Trichloroethane	5	10,000
Carbon Tetrachloride	5	10,000
Vinyl Acetate	10	10,000
Bromodichloromethane	5	10,000
1,2-Dichloropropane	5	10,000
Cis-1,3-dichloropropene	5	10,000
Trichloroethene	5	10,000
Dibromochloromethane	5	10,000
1,1,2-Trichloroethane	5	10,000
Benzene	5	10,000
Trans-1,3-dichloropropene	5	10,000
Bromoform	5	10,000
4-Methyl-2-pentanone ^c	10	20,000
2-Hexanone	10	20,000
Tetrachloroethene	5	10,000
1,1,2,2-Tetrachloroethane	5	10,000
Toluene	5	10,000
Chlorobenzene	5	10,000
Ethylbenzene	5	10,000
Styrene	5	10,000
Xylenes (total)	5	10,000

^a1 g of organic liquid diluted to 2 mL in methanol, and 10 μL of this mixture taken for purge and trap in 5 mL of water.

^bAlso named methylethyl ketone.

^cAlso named methylisobutyl ketone.

Table 2.2. Reporting limits for semivolatile organic compounds by extraction or waste dilution and GC-MS

Compound	Reporting limit	
	Aqueous samples ($\mu\text{g/L}$)	Organic liquids ^a ($\mu\text{g/kg}$)
Phenol	250	20,000
Bis(2-chloroethyl)ether	250	20,000
2-Chlorophenol	250	20,000
1,3-Dichlorobenzene	250	20,000
1,4-Dichlorobenzene	250	20,000
Benzyl alcohol	250	20,000
1,2-Dichlorobenzene	250	20,000
2-Methylphenol	250	20,000
N-Nitroso-di-n-propylamine	250	20,000
Hexachloroethane	250	20,000
Nitrobenzene	250	20,000
Isophorone	250	20,000
2-Nitrophenol	250	20,000
2,4-Dimethylphenol	250	20,000
Benzoic acid	1,300	100,000
Bis(2-chloroethoxy)methane	250	20,000
2,4-Dichlorophenol	250	20,000
1,2,4-Trichlorobenzene	250	20,000
Naphthalene	250	20,000
4-Chloroaniline	250	20,000
Hexachlorobutadiene	250	20,000
4-Chloro-3-methylphenol	250	20,000
2-Methylnaphthalene	250	20,000
Hexachlorocyclopentadiene	250	20,000
2,4,6-Trichlorophenol	250	20,000
2,4,5-Trichlorophenol	1300	100,000
2-Chloronaphthalene	250	20,000
2-Nitroaniline	1,300	100,000
Dimethylphthalate	250	20,000
Acenaphthylene	250	20,000
2,6-Dinitrotoluene	250	20,000
3-Nitroaniline	1,300	100,000
Acenaphthene	250	20,000
2,4-Dinitrophenol	1,300	100,000
4-Nitrophenol	1,300	100,000
Dibenzofuran	250	20,000
2,4-Dinitrotoluene	250	20,000
Diethylphthalate	250	20,000
4-Chlorophenyl-phenylether	250	20,000
Fluorene	250	20,000

Table 2.2 (continued)

Compound	Reporting limit	
	Aqueous samples ($\mu\text{g/L}$)	Organic liquids ^a ($\mu\text{g/kg}$)
4-Nitroaniline	1,300	100,000
4,6-Dinitro-2-methylphenol	1,300	100,000
<i>n</i> -nitrosodiphenylamine ^b	250	20,000
4-Bromophenyl-phenylether	250	20,000
Hexachlorobenzene	250	20,000
Pentachlorophenol	1,300	100,000
Phenanthrene	250	20,000
Anthracene	250	20,000
Di- <i>n</i> -butylphthalate	250	20,000
Fluoranthene	250	20,000
Pyrene	250	20,000
Butylbenzylphthalate	250	20,000
3,3'-Dichlorobenzidine	500	40,000
Benzo(a)anthracene	250	20,000
Chrysene	250	20,000
Bis(2-ethylhexyl)phthalate	250	20,000
Di- <i>n</i> -octylphthalate	250	20,000
Benzo(b)fluoranthene	250	20,000
Benzo(k)fluoranthene	250	20,000
Benzo(a)pyrene	250	20,000
Indeno(1,2,3- <i>cd</i>)pyrene	250	20,000
Dibenz(<i>a,h</i>)anthracene ^c	250	20,000
Benzo(<i>g,h,i</i>)perylene	250	20,000

^a1 g of organic liquid in 2 mL of methylene chloride.

^bCannot be separated from diphenylamine.

^cAnd/or dibenz(*a,c*)anthracene.

2.2 TANK TH-2

The samples from tank TH-2 consisted of two aqueous liquids, L-131 and L-132. No sludge or organic layers were found. The TCL constituents found and major Tentatively Identified Compounds (TICs) are included in Table 2.3 for the volatile organic compounds analyses and in Table 2.4 for the semivolatile organic compounds analyses.

Table 2.3. Results for volatile organic compound analysis of waste samples^a

	Tank identification and component concentration ($\mu\text{g/L}$)								Water blank ^b	
	TH-2		WC-1		WC-15					
	L-131	L-132	L-124	L-134	O-130	L-126	L-127			L-128
Target compound list										
Methylene chloride			240	160		10	8			240
Carbon disulfide				3					1	
1,2-Dichloroethene						15	15		14	
Chloroform				1						
1,1,1-Trichloroethane						16				
Toluene	6	6								5
Tentatively identified compounds ^c										
Unknown				44 (2)						
Unknown hydrocarbon									9 (2)	
Ethyl ether						16	17		15	
Iodomethane	40	230								44
Trichlorofluoromethane	25	190								29

^aOnly compounds whose possible presence was indicated by the analysis are listed.

^bRepresentative blank.

^cNumber of tentatively identified compounds in parentheses.

Table 2.4. Results for semivolatile organic compound analysis of waste samples^a

	Tank identification and component concentration ($\mu\text{g/L}$)								Water blank ^b	
	TH-2		WC-1		WC-15					
	L-131	L-132	L-124	L-134	O-130	L-126	L-127			L-128
Target compound										
2-Chlorophenol		52								
Di-n-butylphthalate					1,900					
Tentatively identified compounds ^c										
Unknown				13,110 (13)	39,100 (3)					
Unknown hydrocarbons					189,100 (15)					
Brominated Benzene Deriv.					13,700 (2)					

^aOnly compounds whose possible presence was indicated by the analysis are listed.

^bWater blank.

^cNumber of tentatively identified compounds in parentheses.

2.2.1 Volatile Organic Compounds Analysis

There were no TCL constituents measured above the reporting limits (5–10 $\mu\text{g/L}$) except for toluene (6 $\mu\text{g/L}$ in both samples). The toluene was also found in the blank (5 $\mu\text{g/L}$); thus, it cannot be attributed to the tank contents. The main TICs were iodomethane and trichlorofluoromethane, but they also were found in the blank. Thus, they cannot be positively ascribed to the tank contents. Only in L-132 were the TICs more concentrated (230 and 190 $\mu\text{g/L}$ respectively) than in the blank (44 and 29 $\mu\text{g/L}$ respectively). The very low results for the volatile organic compounds analyses are consistent with the very low total organic carbon content of 13.6 mg/L for L-131 and 7.8 mg/L for L-132 (see Sect. 3.3).

Surrogate standard and matrix spike recoveries were generally good. One sample apparently had been double-spiked with the surrogate standard. After correction for this mistake, the recoveries were very good. The matrix spike was omitted from the matrix spike duplicate sample, but the recoveries of the first matrix spike were very good. The blank was clean.

2.2.2 Major Volatile Organic Compounds Analysis

No compounds were detected in this analysis. Matrix spike recoveries were very good, and the blank was clean.

2.2.3 Semivolatile Organic Compounds Analysis

There were no TCL constituents measured above the reporting limits (250–1300 $\mu\text{g/L}$) in either sample, although 52 $\mu\text{g/L}$ of 2-chlorophenol was estimated in sample L-132. There were no TICs for either sample. The surrogate standard and matrix spike recoveries all were very good, and the blank was clean.

2.3 TANK WC-1

Two aqueous liquids, L-124 and L-134, were collected from this tank. The results of the volatile organic compounds analysis and semivolatile organic compounds analysis of these samples are also included in Tables 2.3 and 2.4.

2.3.1 Volatile Organic Compounds Analysis

Two TCL constituents, methylene chloride and carbon disulfide, were determined in L-134 at levels below the reporting limits. The former compound also was detected in the blank at a comparable level. The carbon disulfide was very low (estimated at 3 $\mu\text{g/L}$). Two unknown TICs were detected at a total of 44 $\mu\text{g/L}$ in L-134. These results are consistent with the low total organic carbon (TOC) found in these samples (35 mg/L for L-124 and 38 mg/L for L-134) (see Sect. ?).

The recoveries of both the surrogate standards and the matrix spikes were very low (<14%) for these samples. The most likely cause is leakage in the new purge-and-trap apparatus used in the glove box. These samples will be rechecked after the apparatus is leak-checked and its performance is verified with control samples.

2.3.2 Major Volatile Organic Compounds Analysis

No volatile organic compounds were detected in these samples. Matrix spikes were good, and the blanks were clean. These results support the findings of the volatile organic compounds analyses.

2.3.3 Semivolatile Organic Compounds Analysis

There were no TCL constituents found for either sample. Thirteen TICs totaled 13,000 $\mu\text{g/L}$ in sample L-134. Surrogate standard recoveries were good (but tribromophenol was above quality control limits for water), and matrix spike recoveries were good except for phenol, 2-chlorophenol, 1,4-dichlorobenzene, and 1,2,4-trichlorobenzene in the matrix spike duplicate. The blank was clean.

2.4 TANK WC-15

The samples from this tank consisted of three aqueous liquids (L-126, L-127, L-128) and a thin oil layer (O-130) that appeared much like 90-weight oil. These samples had very low levels of radioactivity. The results of the volatile organic compounds analyses and semivolatile organic compounds analyses for these samples are included in Tables 2.3 and 2.4.

2.4.1 Volatile Organic Compounds Analysis

Because of the low radioactivity of these samples, the aqueous liquids were not prepared off-line by remote purge-and-trap in a glove box, but rather they were prepared by direct purge-and-trap GC-MS. TCL constituents measured included traces of methylene chloride and 1,1,1-trichloroethane (these also were found in the blank) and traces of 1,2-dichloroethene (14–15 $\mu\text{g/L}$). Carbon disulfide in L-128 (estimated at 1 $\mu\text{g/L}$) was essentially insignificant. All three samples contained the TIC ethyl ether (approximately 15–17 $\mu\text{g/L}$), and L-128 also exhibited two unknown TICs totalling 9 $\mu\text{g/L}$. These very low concentrations of volatile organic compounds are consistent with the low total organic carbon determinations of 21, 19, and 24 mg/L for L-126, L-127, and L-128 respectively (see Sect. 3.3). Surrogate standard recoveries were all within quality control limits, but the holding time for groundwater samples exceeded by 7 d the time allowed.

The oil sample did not contain TCL compounds or TICs detectable by the volatile organic compounds analyses; this indicates that it is a relatively heavy oil devoid of volatile organic matter. It apparently does not have an appreciable solubility in the aqueous layer.

2.4.2 Major Volatile Organic Compounds Analysis

No compounds were detected in the aqueous samples. This method is not appropriate for the oil layer, and that sample was not analyzed. Matrix spike recoveries were good, and the blank was clean.

2.4.3 Semivolatile Organic Compounds Analysis

The semivolatile organic compounds analysis did not have any TCL constituents or TICs for the aqueous samples. The oil layer did have one TCL, di-*n*-butylphthalate, at 1900 $\mu\text{g/kg}$. The essentially hydrocarbon nature of the oil is suggested by the 15 unknown hydrocarbon TICs totalling about 189,000 $\mu\text{g/kg}$, the 3 unknown TICs totally about 39,000 $\mu\text{g/kg}$, and the 2 TICs apparently consisting of brominated benzene derivatives (possibly bromodiethyl benzenes), all of which added up to 14,000 $\mu\text{g/kg}$. The sum of the TCL constituents and the TICs is approximately 243,800 $\mu\text{g/kg}$ (about 0.02 wt %). The bulk of the GC-MS profile was a large unresolved hump (typical of a heavy oil).

The surrogate standard and matrix spike recoveries were all good except for the slightly low 2-fluorobiphenyl in L-127 and the lack of recovery of 4-nitrophenol in both the matrix spike and matrix spike duplicate. The blank was clean.

2.4.4 PCB Analysis

Negative-ion, chemical-ionization GC-MS was used to examine the oil sample O-130 for PCBs and polybrominated biphenyls. Neither was detected. However, chlordane was positively identified by GC-MS and measured by SW 846 method 8080 at 60,000 $\mu\text{g}/\text{kg}$. This would explain the response observed in the PCB analysis. In addition, pentachloronaphthalene was qualitatively identified by GC-MS.

2.5 CONCLUSIONS

The aqueous samples for tanks TH-2, WC-1, and WC-15 do not contain appreciable concentrations of EPA TCL compounds. These conclusions are supported by low total organic carbon concentrations, the generally good surrogate standard and matrix spike recoveries, and the clean blanks. The oil layer in tank WC-15 appears to be essentially a heavy hydrocarbon mixture with low concentrations of chlorinated aromatic compounds. The oil does not appear to have contaminated the aqueous layer.

3. RADIOCHEMICAL AND INORGANIC ANALYTICAL SUPPORT

3.1 INTRODUCTION

Radiochemical and inorganic characterization of radioactive liquid waste from numerous inactive waste tanks is currently managed by the Environmental Restoration Program. Because information obtained from these studies will be used in support of various activities, standardized analytical procedures were used as much as possible for all measurements.^{3,7,8} Frequently, modifications to the standardized procedures were necessary because of the radiation levels and the generally complex chemical nature of the samples. Deviations from standard procedures were documented in the raw data files for these measurements.

The analytical methodology required for the characterization of the radioactive waste has gone through numerous changes since the initial sampling campaign¹ for the inactive LLLW tanks. There was limited experience with LLLW tank analyses during the first tank-sampling campaign, and there was an attempt to follow regulatory procedures for many of the measurements. However, it has been found that many of the regulatory procedures are inadequate or simply do not give valid data for LLLW tank samples. The reason for failure of the regulatory procedures is related to both the restrictions involved with handling radioactive materials and the complex chemical composition of most LLLW samples. As experience was developed with LLLW tank measurements, a number of instrumentation needs were identified, procured, and placed into service to support the various programs involved with LLLW tank characterization. This expansion of analytical capabilities, along with the experience gained, has resulted in a significant improvement in the analytical data for LLLW tank samples and is discussed in this section. The analytical results are presented in Tables 3.1 through 3.3.

3.2 SAMPLE PREPARATION

Liquid samples were filtered and digested with nitric acid prior to analysis for metals. A 0.5-mL aliquot of a 1-mg/mL scandium standard was added prior to dilution to serve as an internal standard for inductively coupled plasma (ICP) analyses. The liquids were either analyzed directly or after a dilution with 2 M nitric acid for the radiochemical measurements.

Table 3.1. Analytical data for tank TH-2 liquid samples

Sample ^a	TH2-L131	TH2-L132
Physical properties and miscellaneous data		
TDS, mg/mL	4.9	5.0
TS, mg/mL	5.1	5.3
Density, g/mL	0.9989	1.0002
IC, mg/L	<0.1	0.2
TC, mg/L	13.6	8.0
TOC, mg/L	13.6	7.8
RCRA metals (mg/L)		
Ag	<0.2	<0.2
As	<2	<2
Ba	<0.1	<0.1
Cd	0.083	<0.08
Cr	0.80	0.68
Hg	2.8	2.7
Ni	0.58	0.43
Pb	1.9	2.1
Se	<1	<1
Tl	<0.5	<0.5
Process metals (mg/L)		
B	<0.7	<0.7
Si	<1	<1
Th	1.68	1.71
U	<8	<8
Anions (mg/L)		
Bromide	1510	1510
Chloride	18	18
Cyanide	0.28	0.22
Fluoride	17	17
Nitrate	<10	<10
Phosphate	<10	<10
Sulfate	26	26
Sulfide	<0.1	<0.1
Alkalinity		
pH	2.44 (2.12) ^b	2.46 (2.14)
H ⁺ , M	0.056	0.056
OH ⁻ , M	NA ^c	NA
CO ₃ ²⁻ , M	NA	NA
HCO ₃ ⁻ , M	NA	NA
Beta/gamma emitters (Bq/mL)		
Gross alpha	87.6 (58)	84.5 (52)
Gross beta	9280 (8690)	9450 (8730)
¹⁴ C	2350	<1
⁶⁰ Co	4 (3.6)	<3 (2.2)
¹³⁷ Cs	2710 (2730)	2740 (2640)
³ H	2.34	2.39
⁹⁰ Sr	2820 (2780)	2920 (2720)
Alpha emitters (Bq/mL)		
²³² U	ND ^d	5
²³³ U	15	17
²³⁹ Pu/ ²⁴⁰ Pu	ND	ND
²³⁸ Pu	ND	ND
²⁴¹ Am	ND	ND
²⁴⁴ Cm	ND	ND

^aACD sample request numbers IPA6285, TRU9069, CPA10586.

^bData enclosed in parentheses were duplicate measurements by TAL group.

^cNA = analysis not applicable for sample.

^dND = analyte not detected for sample.

Table 3.2. Analytical data for tank WC-1 liquid and sludge samples

Sample ^a	WC1-L124 (liquid)	WC1-L134 ^b (liquid)	WC1-L134S (sludge)
Physical properties and miscellaneous data			
TDS, mg/mL or mg/g ^c	<1	<1 ^d	NA ^e
TS, mg/mL or mg/g	<1	<1	NA
Density, g/mL or g/g	0.9750	0.9760	NA
IC, mg/L or mg/kg	32	23	560
TC, mg/L or mg/kg	67	61	9930
TOC, mg/L or mg/kg	35	38	9370
RCRA metals (mg/L or mg/kg)			
Ag	<0.04	0.33	<40
As	1.3	2.0	<300
Ba	<0.02	93	927
Cd	<0.02	0.10	4.0
Cr	<0.02	2.9	112
Hg	<2	<2	193
Ni	<0.1	<0.1	10
Pb	<0.2	61	771
Se	2.4	<0.02	<20
Tl	<0.1	<0.1	<90
Process metals (mg/L or mg/kg)			
B	<0.04	<0.04	<40
Si	10	90	NA
Th	<0.3	<0.3	<200
U	<2	<2	<100
Anions (mg/L or mg/kg)			
Bromide	<10	<10	<495 ^f
Chloride	113	114	220
Cyanide	<0.05	<0.05	0.33
Fluoride	0.60	0.70	75
Nitrate	<10	<10	3680
Phosphate	<10	<10	<99
Sulfate	196	204	449
Sulfide	<0.1	1.4	0.99
Alkalinity			
pH	8.25 (6.50) ^g	7.46 (6.39)	NA
Beta/gamma emitters (Bq/mL or Bq/g)			
Gross alpha	5 (<50)	286 (1560)	8.07e+04 (8.62e+04)
Gross beta	64,200 (68,600)	73,000 (110,000)	1.09e+06 (1.36e+05)
²⁴¹ Am	<300	131 (<300)	1.57e+04 (1.73e+04)
¹⁴ C	276	375	1.88e+04
⁶⁰ Co	767 (852)	432 (594)	1.30e+05 (1.29e+05)
¹³⁷ Cs	59,200 (58,000)	61,700 (56,500)	2.69e+05 (2.73e+05)
¹⁵² Eu	<30	<30	<2.68e+02 (<1.86e+03)
¹⁵⁴ Eu	<30	<30	4.12e+03 (4.66e+03)
¹⁵⁵ Eu	<150	<150	3.16e+03 (4.12e+03)
³ H	1,600	1,590	4.30e+02
⁹⁰ Sr	1,290 (1,270)	1,420 (3,310)	3.03e+05 (4.03e+05)
Alpha emitters (Bq/mL or Bq/g)			
²³² U	ND ^h	ND	ND
²³³ U	ND	ND	ND
²³⁹ Pu/ ²⁴⁰ Pu	ND	ND	ND
²³⁸ Pu	ND	1,110	4.06e+04
²⁴¹ Am	ND	200	3.48e+04
²⁴⁴ Cm	ND	251	1.08e+04

^aACD sample request numbers IPA6324, TRU9239.

^bLarge amount of suspended particles present; difficult to obtain representative sample. For the physical and inorganic data, the particulates were allowed to settle prior to sampling for analysis; the duplicate radiochemical data and the alpha emitter data were obtained from a well-mixed sample.

^cThe sludge sample data are reported on a weight basis.

^dTS/TDS data obtained after suspended solids had settled out.

^eNA = analysis not applicable for sample.

^fSludge anions obtained from microwave-heated water-leach of sample.

^gData enclosed in parentheses were duplicate measurements by TAL group.

^hND = analyte not detected for sample.

Table 3.3. Analytical data for tank WC-15 liquid samples

Sample ^a	WC15-L126	WC15-L127	WC15-L128	WC15-O130
Physical properties and miscellaneous data				
TDS, mg/mL	<1	<1	<1	<1
TS, mg/mL	<1	<1	<1	<1
Density, g/mL	0.9560	0.9610	0.9620	0.9570
IC, mg/L	98	99	102	100
TC, mg/L	119	118	126	435
TOC, mg/L	21	19	24	335
RCRA metals (mg/L)				
Ag	<1	0.22	0.16	<1.0
As	<8	<8	<8	<8
Ba	0.10	0.03	0.08	0.05
Cd	<0.5	<0.5	<0.5	<0.5
Cr	<0.5	<0.5	0.3	<0.5
Hg	<2	<2	<2	<2
Ni	0.11	<2	0.11	0.16
Pb	<5	<5	<5	<5
Se	0.58	0.05	0.3	<0.5
Tl	<2	<2	<2	<2
Process metals (mg/L)				
B	<1	<1	<1	<1
Si	5.2	<2	<2	<2
Th	<7	<7	<7	<7
U	1.4	3.7	<50	2.4
Anions (mg/L)				
Bromide	<10	<10	<10	<10
Chloride	89	87	89	85
Cyanide	<0.05	<0.05	<0.05	<0.05
Fluoride	1.2	1.2	1.2	1.2
Nitrate	<10	<10	<10	<10
Phosphate	<10	<10	<10	<10
Sulfate	50	50	38	41
Sulfide	<0.01	<0.01	0.2	1.3
Alkalinity				
pH	8.8 (8.52) ^b	8.7 (8.50)	7.7 (8.53)	6.5 (8.39)
H ⁺ , M	NA ^c	NA	NA	NA
OH ⁻ , M	NA	NA	NA	NA
CO ₃ ²⁻ , M	NA	NA	NA	NA
HCO ₃ ⁻ , M	NA	NA	NA	NA
Beta/gamma emitters (Bq/mL)				
Gross alpha	0.89 (<1)	0.13 (<1)	0.43 (<1)	0.56 (<1)
Gross beta	11.7 (<10)	10.3 (<10)	14.6 (<10)	15.4 (<10)
¹⁴ C	<1	<1	33	95
⁶⁰ Co	<0.2	<0.2	<0.2	<0.3
¹³⁷ Cs	3.0 (<5)	3.1 (<5)	4.0 (<5)	3.5 (<5)
³ H	18.4	18.6	18.9	18.5
⁹⁰ Sr	1.40 (<10)	1.71 (<10)	1.93 (<10)	3.36 (<10)
Alpha emitters (Bq/mL)				
²³² U	<i>d</i>			
²³³ U				
²³⁹ Pu/ ²⁴⁰ Pu				
²³⁸ Pu				
²⁴¹ Am				
²⁴⁴ Cm				

^aACD sample request numbers IPA6332, TRU9238.

^bData enclosed in parentheses were duplicate measurements by TAL group.

^cNA = analysis not applicable for sample.

^dGross alpha too low for alpha spectroscopy measurements.

Sludge samples from tank WC-1 were prepared by microwave digestion with a CEM MDS-81D microwave instrument. The digestion procedure is similar to the proposed SW-846 method 3051, *Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils*. Approximately 1 g of sludge sample was placed in a Teflon® vessel (120-mL size) with 10 mL of nitric acid. After heating for 2.5 min at 100% power and 10 min at 80% power, the sample was allowed to cool to room temperature. After cooling, 2–5 mL of 30% hydrogen peroxide was added a drop at a time to the solution. On the completion of effervescence, the samples were filtered through a No. 541 Whatman filter and diluted to 100 mL with American Society for Testing Materials Type II water. A 1.0-mL aliquot of a 1.0-mg/mL scandium standard was added prior to the dilution. The scandium serves as an internal standard to account for matrix interferences with ICP analyses.

3.3 CARBON ANALYZER

Liquids were assayed for total carbon (TC) and inorganic carbon (IC) on the Dohrmann carbon analyzer. The TC was measured by direct sample injection into a combustion tube packed with cobalt oxide and maintained at 900 °C. The furnace was continually sparged with oxygen; this converted all carbon to carbon dioxide and transported the off-gases through a detector chamber. The carbon dioxide was detected by a nondispersive infrared (IR) detector. Liquids were assayed for IC by sample injection into a phosphoric acid bath that converted carbonates to carbon dioxide. The phosphoric acid bath was continually sparged with oxygen; this transported the evolved carbon dioxide to the IR detector.

An optional solids adapter for the Dohrmann carbon analyzer was used to measure TC and TOC in the sludge samples. Sludges were assayed for TC by placing the sample in a platinum boat, then heating the sludge to 900°C to oxidize organic species and any elemental carbon and to decompose carbonates. The furnace was continually sparged with oxygen; this passed through a combustion bed packed with cobalt oxide for conversion of carbon-bearing species to carbon dioxide. The evolved carbon dioxide was transported to the IR detector. Sludges were assayed for organic carbon after pretreatment with concentrated nitric acid. The sample was weighed, acidified, and dried before placement in the same furnace used for TC determination.

3.4 ION CHROMATOGRAPHY

A Dionex 4506i ion chromatograph having a pulsed electrochemical detector was installed in a radiochemical hood to improve analytical capabilities for (1) anions (fluoride, chloride, bromide, phosphate, nitrate and sulfate) and (2) alkali metals (sodium, potassium, and cesium) and for cyanide/sulfide measurements. Another improvement over earlier LLLW tank studies¹ was the inclusion of an HPIC-AS2 (Dionex column) anion separator column; this column has a high affinity for nitrate and permits earlier eluting anions to be determined without significant interference from nitrate.

The determination of the common anions listed above was primarily for treatability studies, but the anion data were also useful for understanding the distribution of various metals between the liquid and solid phases. The measurement of these anions on a system that was configured for radioactive samples was a much needed improvement over the last sampling campaign.¹ For this project, only the liquid samples were analyzed for the anions.

The determination of anions within the sludge phase of the LLLW tanks is a problem area. Ion chromatography may not be appropriate for anion determination in sludge samples because the solid phase must first be placed into solution with a strong acid. The use of a strong acid to dissolve the sludge samples places severe limitations on the anions that can be determined by ion chromatography. Considerable development work may be required to obtain useful anion data for the sludge phase of the waste tanks. An alternative to direct measurement of anions in a sludge sample is to develop a protocol that involves several leaching steps to characterize the soluble anion content in a sludge material. A procedure where the sludge sample was leached with successively increasing concentrations of acid would account for greater than 90% of the sample anion content (greater than 50% of most sludges observed to date will dissolve in distilled water).

A basic limitation of ion chromatography is that the relative concentration of all anions must be within a factor of about 100 or the predominate species behaves as an eluent for the other anionic species resulting in all anions eluting in a single band. High levels of certain anions can be tolerated by the use of special precolumns to reduce the level of a predominate species; however, these precolumns were not available for this project. The new ion chromatograph will permit more methods development work, and improved anion/cation data should be obtainable for future LLLW waste-tank samples.

3.5 CYANIDE/SULFIDE ANALYSIS

Numerous attempts have been made to use the regulatory procedures (EPA methods 9010 and 9030) for cyanide and sulfide with little or no success. The regulatory procedure uses 500 mL of sample, but this is unacceptable for radioactive samples because of their high activity. In addition, a more serious problem with the EPA procedures is a result of severe chemical interferences resulting from the complex chemical composition of radioactive waste samples. To overcome the problems with cyanide and sulfide measurements, a two-fold solution was required. First, a Lachat Micro-Dist, which is a microdistillation apparatus, was used to handle the cyanide/sulfide distillation from radioactive samples. The advantage of the Lachat system is that minimal sample handling is required by the analyst, and up to 21 samples can be distilled at the same time in a small space (available radiochemical hood space is very limited). The Lachat distillation tube is a disposable unit that separates the sample from a basic scrubber with a hydrophobic membrane. This distillation tube is simple, easy to handle, and could be adapted to a glove box operation if necessary. The second problem, chemical interferences, was resolved by measuring the cyanide and sulfide by ion chromatography with electrochemical detection.

The cyanide and sulfide were distilled from 6 mL of sample using the Lachat Micro-Dist method "Cyanide 1" procedure. The Lachat procedure was modified to include sulfamic acid, which is recommended for a high nitrate matrix, in the releasing solution. The releasing solution is simply an aliquot of acid added to the sample to evolve hydrogen cyanide and hydrogen sulfide. The cyanide and sulfide were then measured in the distillates by ion chromatography with a 0.025-mL sample loop injection and an HPIC AS-7 separator column. The cyanide and sulfide were detected by direct current amperometry. Both standard measurements and spike recoveries have been excellent with this procedure.

3.6 RADIOCHEMICAL CHARACTERIZATION

The radiochemical characterization of the LLLW tanks included gross alpha, gross beta, gamma spectrometry, ^{90}Sr , and actinides by alpha spectrometry. To ensure valid data, all of the previous radiochemical measurements except the alpha spectrometry were duplicated by two separate laboratories. A brief description of the radiochemical methods, along with typical detection limits,¹ is given elsewhere. In addition, ^{14}C and tritium were determined by liquid scintillation on all liquid samples, and ^{14}C was determined for all sludge samples. Although not measured for this project, it is recommended that both ^{99}Tc ($t_{1/2} = 2.13 \times 10^5$ years) and ^{129}I ($t_{1/2} = 1.6 \times 10^7$ years) be determined on selected samples from this project.

3.6.1 Liquid Scintillation Counting

A Packard 2500TR liquid scintillation system was used for ^{14}C and tritium measurements. The tritium was measured in liquid samples after distillation from a basic solution. A Dohrmann carbon analyzer was used for ^{14}C analyses.

The Dohrmann carbon analyzer is equipped with a combustion tube for solids analysis that was adapted to the analysis of ^{14}C . In this procedure samples were heated to 900°C to volatilize organic compounds, to decompose carbonates, and to oxidize elemental carbon. The furnace was continually sparged with oxygen, and all off-gases were passed through an oxidizing bed (cobalt oxide) to convert organic species to carbon dioxide. Samples were collected in gas-washing bottles filled with a carbon dioxide-absorbent solution. Aliquots of the carbon dioxide-absorbent solutions were pipetted into a compatible liquid scintillation cocktail and counted.

3.6.2 Sample Preparation for Alpha Counting

A rapid and reliable method was needed to prepare clean alpha plates from samples of inactive waste tanks. Excessive solid materials on these prepared alpha plates led to self absorption, flaking, and poor alpha spectrometry for isotopic determination. The method used to clean up these samples is based on a double precipitation with ferric iron. Ferric nitrate is first added to the sample solution; this is then heated to boiling and followed by a drop-by-drop addition of NH_4OH to precipitate what is best described as hydrous ferric oxide ($\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$) but is commonly called ferric hydroxide. Yields have been reported⁹ in excess of 97% for uranium and the transuranic actinides coprecipitated with the ferric hydroxide.

Because of excessive solids, the isotopic composition could not be accurately determined on the LLLW tank samples that were plated directly. There was no significant difference in the pulse-height analysis of the single and double iron precipitates; however, the second precipitation was required for valid gross alpha data. This method is reliable, rapid, and generates very little waste. It can easily be carried out on samples with low alpha activity and results in minimal dilution of the sample (dilution factor less than 5).

4. SUMMARY

4.1 TANK TH-2

Tank TH-2 contains about 70 gal of liquid and no sludge. Low levels of Resource Conservation and Recovery Act (RCRA) metals (Cr, Hg, Pb, and Ni) were observed in this tank, but only the mercury level exceeded the RCRA limit. Only long-lived fission products ^{137}Cs and ^{90}Sr (along with their short-lived daughter products ^{137}Ba and ^{90}Y) were observed at an appreciable level. The alpha activity in this tank was less than 100 Bq/mL; this is well below the transuranic (TRU) waste limit of 3700 Bq/g. An unusually high level of bromide measured in this tank suggested that the contents (zinc bromide) from a used hot-cell window were discharged into the tank. However, only trace levels of zinc were measured in the tank.

Concentrations of regulatory organic compounds were very low in the liquid. There were no EPA TCL constituents for volatile organics in either of the two liquid samples, but 230 $\mu\text{g/L}$ of iodomethane and 190 $\mu\text{g/L}$ of trichlorofluoromethane were estimated in the TICs for sample L-132. Only 2-chlorophenol (52 $\mu\text{g/L}$) was found in the semivolatile organics analysis of sample L-132.

4.2 TANK WC-1

Approximately 375 gal of liquid remain in tank WC-1. Samples were taken from the top and bottom of the liquid layer. The bottom sample contained finely suspended particles that were filtered from the liquid for separate analysis and analyzed as a sludge sample. The liquid portion associated with the suspended solid has elevated levels of RCRA metals (Ba, Cr, and Pb), and the Pb level is greater than the RCRA limit by a factor of 10. The solid portion of this sample measured high in Ba, Cd, Cr, Hg, Ni, and Pb. The Cr, Hg, and Pb all exceed the RCRA limits in the solid material. Only ^{137}Cs and ^{90}Sr were observed at an appreciable level in the liquid phase of this tank. A much higher level of long-lived fission products was observed in the suspended solids. Also, the level of transuranium elements (^{238}Pu and ^{241}Am) is high enough to classify the solid as TRU waste. The alpha activity in the liquid phase is below this limit for TRU waste classification.

Similar to tank TH-2, the two aqueous samples for WC-1 contained very little detectable organic matter. The main findings were 2 unknown TICs totaling 44 $\mu\text{g/L}$ in the volatiles and 13 unknown TICs adding up to 13,000 $\mu\text{g/L}$ in the semivolatiles. However, the volatile organic surrogate standard and matrix spike recoveries were unacceptably low.

4.3 TANK WC-15

Tank WC-15 has a capacity of 1000 gal and was discovered to be completely full during sampling. A thin, light oil layer is present above the aqueous phase. Four samples were collected from the tank, and both the aqueous and oil layers measured only trace levels of metals or radionuclides.

The aqueous samples contained very little organic matter suggesting that the oil has little solubility in the aqueous layer. There were no TCL constituents detected or TICs for the semivolatile organics analysis, and the volatile organics analysis showed only traces of the TCL compounds dichloroethene (14–15 $\mu\text{g/L}$) and ethyl ether (15–17 $\mu\text{g/L}$).

The analysis of the oil layer detected no volatile organics compounds and only one TCL constituent, di-*n*-butylphthalate at 1,900 $\mu\text{g/L}$. The hydrocarbon nature of the oil was evident by the 189,000 $\mu\text{g/kg}$ of hydrocarbon TICs and the large, unresolved "hump" in the GC-MS chromatogram. Chlordane was detected at 60,000 $\mu\text{g/kg}$, and pentachloronaphthalene was qualitatively identified.

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