MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES

3 4456 0315970 2

ornl

ORNL/ER-13

OAK RIDGE
NATIONAL
LABORATORY

MARTIN MARIETTA

Sampling and Analysis of the Inactive Waste Storage Tank Contents at ORNL

J. W. Autrey
D. A. Costanzo
W. H. Griest
L. L. Kaiser
J. M. Keller
C. E. Nix
B. A. Tomkins

OAK RIDGE NATIONAL LABORATORY
CENTRAL RESEARCH LIBRARY
CIRCULATION SECTION
4500N ROOM 175
LIBRARY LOAN COPY
DO NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to see this report, send in name with report and the library will arrange a loan.

OPERATED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

ORNL/ER-13

Environmental Restoration Division
ORNL Environmental Restoration Program

Sampling and Analysis of the Inactive Waste Storage Tank Contents at ORNL

J. W. Autrey
D. A. Costanzo
W. H. Griest
L. L. Kaiser
J. M. Keller
C. E. Nix
B. A. Tomkins

Date Issued—September 1990

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
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES

3 4456 0315970 2

Author Affiliations

J. W. Autrey, L. L. Kaiser, and C. E. Nix are members of the Office of Environmental, Safety, and Health Compliance, Oak Ridge National Laboratory. D. A. Costanzo, W. H. Griest, J. M. Keller, and B. A. Tomkins are members of the Analytical Chemistry Division, Oak Ridge National Laboratory.

TABLE OF CONTENTS

LIST OF FIGURES vii

LIST OF TABLES ix

ACKNOWLEDGMENTS xi

ABSTRACT xiii

1. SAMPLE COLLECTION FROM THE INACTIVE TANKS 1-1
   1.1 INTRODUCTION 1-1
   1.2 BACKGROUND 1-1
      1.2.1 Need for Sampling and Analysis 1-1
   1.3 SAMPLING ACTIVITIES 1-5
      1.3.1 Sample-Collection Strategy 1-5
      1.3.2 Collection of Liquids 1-5
      1.3.3 Collection of Sludges 1-5
   1.4 QUALITY ASSURANCE 1-10
      1.4.1 Sample Collection 1-10
      1.4.2 Sample Analyses 1-10
   1.5 REFERENCES 1-11

2. ORGANIC ANALYTICAL METHODS 2-1
   2.1 INTRODUCTION 2-1
   2.2 VOLATILE ORGANIC COMPOUNDS ANALYSIS 2-1
      2.2.1 Method Description 2-1
         2.2.1.1 Radioactive aqueous liquids 2-1
         2.2.1.2 Aqueous liquids with very little radioactivity 2-4
         2.2.1.3 Organic liquids 2-4
      2.2.2 Method Evaluation 2-5
   2.3 MAJOR VOLATILE ORGANIC COMPOUNDS 2-8
      2.3.1 Method Description 2-8
   2.4 SEMIVOLATILE ORGANIC COMPOUND ANALYSIS 2-9
      2.4.1 Method Description 2-9
         2.4.1.1 Extraction of aqueous liquids 2-9
         2.4.1.2 Extraction of Sludges 2-11
         2.4.1.3 Preparation of organic liquids 2-15
         2.4.1.4 GC screen 2-15
         2.4.1.5 Analysis 2-16
      2.4.2 Method Evaluation 2-16
   2.5 PCB ANALYSIS 2-18
      2.5.1 Method Description 2-18
   2.6 REFERENCES 2-19

3. ORGANIC ANALYTICAL CHARACTERIZATION 3-1
   3.1 INTRODUCTION 3-1
   3.2 DATA-REPORTING CONVENTIONS 3-1
   3.3 RESULTS FOR WASTE TANK SAMPLES 3-2
      3.3.1 Tank T-3 3-2
         3.3.1.1 L-042 (aqueous liquid) 3-2

3.3.1.2 S-043 (soft sludge) 3-7
3.3.2 Tank T-4 3-7
3.3.2.1 L-044, L-045, and L-111 (aqueous liquids) 3-7
3.3.2.2 S-046 (soft sludge) 3-9
3.3.3 Tank T-1 3-9
3.3.3.1 L-035, and L-036 (aqueous liquids) 3-9
3.3.3.2 S-037 (soft sludge) 3-9
3.3.4 Tank T-2 3-9
3.3.4.1 L-038, L-039, and L-112 (aqueous liquids) 3-9
3.3.4.2 S-040 (soft sludge) 3-11
3.3.5 Tank T-9 3-11
3.3.5.1 L-047 (aqueous liquid) 3-11
3.3.5.2 S-048 (soft sludge) 3-11
3.3.6 Tank TH-1 3-11
3.3.6.1 L-049 and L-050 (aqueous liquids) 3-11
3.3.7 Tank TH-3 3-11
3.3.7.1 L-052, L-053 (aqueous liquids), and L-113 (blind water blank) . . . 3-11
3.3.8 Tank W-11 3-12
3.3.8.1 L-003, L-114, and L-115 (aqueous liquids) 3-12
3.3.8.2 H-005 (hard sludge) 3-12
3.3.9 Tank TH-4 3-12
3.3.9.1 L-055, L-056, L-057, L-116, L-117 (all aqueous liquids) 3-12
3.3.9.2 S-058 and S-059 (soft sludges) 3-16
3.3.10 Tank W-5 3-16
3.3.10.1 L-073 (aqueous liquid) 3-16
3.3.10.2 S-075 (soft sludge) 3-16
3.3.11 Tank W-6 3-16
3.3.11.1 L-077, L-078, L-079 (aqueous liquids) 3-16
3.3.11.2 S-080 (soft sludge) 3-18
3.3.12 Tank W-7 3-18
3.3.12.1 L-082 (aqueous liquid) 3-18
3.3.12.2 S-084 (soft sludge) and H-085 (hard sludge) 3-18
3.3.13 Tank W-8: S-088 (Soft Sludge), L-086 and L-087 (Aqueous Liquids) 3-18
3.3.14 Tank W-9: L-090 (Aqueous Liquid) and S-092 (Soft Sludge) 3-20
3.3.15 Tank W-1: L-007 and L-008 (Aqueous Liquids) 3-21
3.3.16 Tank W-2: L-011 and L-118 (Aqueous Liquids) 3-21
3.3.17 Tank W-4 3-22
3.3.17.1 L-022, L-023, L-119 (aqueous liquids) 3-22
3.3.17.2 S-024 (soft sludge) and H-026 (hard sludge) 3-22
3.3.18 Tank W-10 3-22
3.3.18.1 L-093, L-094, L-095 (aqueous liquids) 3-22
3.3.18.2 S-096 (soft sludge) and H-120 (hard sludge) 3-25
3.3.19 Tank 7860A 3-25
3.3.19.1 L-063, L-064, L-065, L-066, and L-067org (all organic liquids) . . 3-25
3.3.19.2 L-067, L-068, and L-069 (all aqueous liquids) 3-28
3.3.20 Tank W-3 3-29
3.3.20.1 L-016, L-017, and L-018 (all aqueous liquids) 3-29
3.3.20.2 S-020 (soft sludge) 3-29
3.3.21 Tank WC-17 3-29
3.3.21.1 O-101, O-102, L-103, L-104, and L-105 (all aqueous liquids) 3-29

3.3.21.2 O-101, O-102, L-103 (all organic layers floating on aqueous layers of the same sample number) 3-31
3.3.22 Tank W-13: L-027 and L-028 (Both Aqueous Liquids) 3-31
3.3.23 Tank W-14: L-030 and L-031 (Both Aqueous Liquids) 3-34
3.3.24 Tank W-15: L-032 and L-033 (Both Aqueous Liquids) 3-34
3.3.25 Tank W-1A: L-001 (Aqueous Liquid) 3-34
3.3.26 Tank T-30: L-121 (Aqueous Liquid) 3-34
3.3.27 Tank 7562: L-097 (Aqueous Liquid) 3-34
3.4 ANALYSIS OF BLANKS 3-35
3.5 RECOVERIES OF SURROGATE STANDARDS AND MATRIX SPIKES 3-35
3.6 ACCOUNTING OF ORGANIC CARBON 3-43
3.7 SUMMARY OF ORGANIC ANALYTICAL RESULTS 3-54
3.8 REFERENCES 3-55

4. RADIOCHEMICAL AND INORGANIC ANALYTICAL SUPPORT FOR THE INACTIVE TANK CHARACTERIZATION 4-1
4.1 INTRODUCTION 4-1
4.2 SAMPLE PREPARATION 4-7
4.2.1 Radiochemical Sample Preparation 4-7
4.2.2 Spectrochemical Sample Preparation (ICP and AA) 4-7
4.2.3 Sludge-Sampling Procedure 4-9
4.2.4 Sample Preparation for Mercury Analysis 4-9
4.2.5 Extraction Procedure Toxicity Test (EP-TOX): METHOD 1310 4-10
4.3 ANALYTICAL METHODS 4-10
4.3.1 RCRA Metal Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) 4-11
4.3.2 RCRA Metal Analysis by GFAA 4-19
4.3.3 Mercury Analysis by CV Technique 4-21
4.3.4 Cyanide and Sulfide Analysis 4-21
4.4 ANALYTICAL RESULTS 4-22
4.5 EVALUATION OF DATA 4-29
4.5.1 Overall Trends 4-29
4.5.2 Physical Measurements 4-32
4.5.3 RCRA Metals 4-33
4.5.4 Treatability Metals 4-33
4.5.5 Cyanide and Sulfide 4-33
4.5.6 Common Anions 4-34
4.5.7 Alpha Emitters (TRU Waste) 4-35
4.5.8 Beta-Gamma Emitters 4-35
4.6 RCRA EVALUATION FOR INORGANIC SPECIES 4-35
4.7 RECOMMENDATION FOR ADDITIONAL ACTIVITIES 4-39
4.8 ADDENDUM: PRELIMINARY EP-TOX RESULTS 4-40
4.9 REFERENCES 4-41

5. RCRA STATUS OF INACTIVE LIQUID LOW-LEVEL RADIOACTIVE WASTE TANKS 5-1
5.1 INTRODUCTION 5-1
5.2 RCRA HAZARDOUS CHARACTERISTICS 5-1
5.3 RCRA ORGANIC CONSTITUENTS 5-1
5.4 REFERENCES 5-7
APPENDIX: LISTING OF ORGANIC ANALYTICAL DATA HITS FOR INACTIVE TANKS AT ORNL A-1

LIST OF FIGURES

Fig. 1.1. Waste storage tanks within the main plant area 1-2
Fig. 1.2. Waste storage tanks outside the main plant area 1-3
Fig. 1.3. Typical tank configuration with proposed sample location 1-6
Fig. 1.4. Liquid sample collection system 1-7
Fig. 1.5. Soft sludge sampler 1-8
Fig. 1.6. Hard sludge sampler 1-9

Fig. 2.1. Purging apparatus being assembled in a glove box for the collection of volatile organic compounds from radioactive liquid samples 2-2
Fig. 2.2. Ultrasonic solvent extraction of semivolatile organic compounds from radioactive sludge, using a remote manipulator in a hot cell 2-12

Fig. 3.1. Comparison of the capillary column gas chromatograms of the SVOA extract from sample T-2/L-112 (A) underivatized and (B) trimethylsilyl-derivatized forms. (Preliminary peak identifications and concentration estimates are listed in Table 3.24.) 3-52

Fig. 4.1. Liquid samples from tank WC17 (Codes WC17/O101-O103 and WC17/L104-L105) 4-2
Fig. 4.2. Homogenous sludge sample from tank WC17 (Code WC17/S106) 4-3
Fig. 4.3. Nonhomogenous sludge sample from tank TH4 (Code TH4/S059) 4-4
Fig. 4.4. Perkin-Elmer Model 6500 ICP modified for radioactive work 4-16
Fig. 4.5. Close-up of torch box for ICP system 4-17
Fig. 4.6. Perkin-Elmer Model 5500 AA modified for radioactive work 4-19

LIST OF TABLES

Table 1.1. Physical data of inactive waste storage tanks 1-4

Table 2.1. MAS compounds in purge-and-trap GC-MS 2-3
Table 2.2. Comparison of supplementary volatile organic compound standard analyses with purge-and-trap in the glove box and the GC-MS lab 2-6
Table 2.3. MAS and MSD compounds used in direct aqueous GC analysis of major volatile organic compounds (about 20 mg/L each) 2-10
Table 2.4. Semivolatile surrogate standard compounds added to aqueous liquids 2-10
Table 2.5. Semivolatile MAS compounds added to aqueous samples 2-11
Table 2.6. Semivolatile organic surrogate standards added to sludge samples 2-13
Table 2.7. Semivolatile organic MAS compounds added to sludge sample W-3/S-020 2-14
Table 2.8. Results of GC-MS analysis of supplementary semivolatile appendix VIII and other organic compounds as TICs 2-17

Table 3.1. Reporting limits for volatile organic compounds in purge-and-trap GC-MS analysis (μg/L) 3-3
Table 3.2. Reporting limits for semivolatile organic compounds by extraction and GC-MS 3-4
Table 3.3. Masses of sludge samples for semivolatile organic compound extraction 3-6
Table 3.4. Listing of organic analytical data hits for tank T-4 3-8
Table 3.5. Listing of organic analytical data hits for tank T-2 3-10
Table 3.6. Listing of organic analytical data hits for tank W-11 3-13
Table 3.7. Listing of organic analytical data hits for tank TH-4 3-14
Table 3.8. Listing of organic analytical data hits for tank W-6 3-17
Table 3.9. Listing of organic analytical data hits for tank W-7 3-19
Table 3.10. Listing of organic analytical data hits for tank W-8 3-20
Table 3.11. Listing of organic analytical data hits for tank W-9 3-21
Table 3.12. Listing of organic analytical data hits for tank W-4 3-23
Table 3.13. Listing of organic analytical data hits for tank W-10 3-24
Table 3.14. Listing of organic analytical data hits for tank 7860A 3-26
Table 3.15. Listing of organic analytical data hits for tank W-3 3-30
Table 3.16. Listing of organic analytical data hits for tank WC-17 3-32
Table 3.17. Results for VOA and SVOA of blanks for aqueous liquids 3-36
Table 3.18. Summary of matrix spike recoveries for DAI-GC 3-38
Table 3.19. Summary of VOA surrogate standard and matrix spike recoveries for aqueous liquids and blanks 3-39
Table 3.20. Summary of SVOA surrogate standard and matrix spike recoveries for aqueous liquids and blanks 3-40
Table 3.21. Summary of SVOA surrogate standard recoveries from sludges 3-43
Table 3.22. Total organic carbon (TOC) accounting in aqueous liquids 3-44
Table 3.23. Total organic carbon (TOC)[a] accounting in sludges 3-48
Table 3.24. Compounds tentatively identified by cochromatography in the TMS-derivatized SVOA extract of T-2/L-112 3-53

Table 4.1. Typical spike recoveries with TOPO extraction 4-8
Table 4.2. RCRA metal regulatory limits for solid waste 4-11
Table 4.3. Analytical methods 4-12
Table 4.4. Physical and metal data for inactive tank samples 4-23
Table 4.5. Anion data for inactive tank samples 4-25
Table 4.6. Radiochemical data for inactive tanks 4-27

Table 4.7. Solubility products for RCRA metals and TRU metals 4-30
Table 4.8. Characteristics of inactive tanks for forming insoluble salts (sludges) 4-31
Table 4.9. TRU-waste classification for sludge samples 4-36
Table 4.10. Summary and status of tank analysis for rcra metals 4-38
Table 4.11. Measurement gradients observed in tank TH4 4-39
Table 4.12. EP-TOX results for RCRA metals 4-41

Table 5.1. RCRA characteristics and constituents of inactive waste storage tanks 5-2

ACKNOWLEDGMENTS

The authors would like to acknowledge the analytical chemistry support and technical assistance provided by members of the Analytical Chemistry Division, including M. G. McClung for sample preparations and distribution; C. J. Baggett for RCRA metal analyses; J. L. Botts for radiological measurements; D. O. Vick for multielement screening by spark-source mass spectrometry; and W. F. Rogers for anion analyses.

Many staff of the Analytical Chemistry Division made important contributions to this work. M. E. Garcia (now of the Environmental Compliance Division) prepared most of the samples for analysis. R. L. Schenley is working on the identification of the nonregulatory organic matter, and he also assisted in the data review and report draft editing. S. L. Harmon, M. D. Edwards, G. S. Fleming, and C. Treese (now with Hewlett Packard) performed the GC-MS analyses; L. J. Wachter was responsible for data reporting and also did GC-MS; and R. M. Edwards conducted the PCB measurements. R. Merriweather assisted with the direct aqueous GC analysis. J. E. Caton is acknowledged for his leadership of the GC-MS laboratory and his helpful advice throughout the project, and R. L. Williams and L. G. Treadwell for their tireless secretarial help.

The authors are also indebted to the sampling services performed by C. B. Scott and J. D. Brewer of the Hazardous/Radioactive Waste Operations group within the Environmental and Health Protection Division.

EXECUTIVE SUMMARY

Thirty three inactive waste storage tanks, currently managed by the ORNL Remedial Action Program (RAP), are to be characterized in preparation for final closure/decommissioning to meet environmental, safety, and long-term surveillance objectives. To date, 30 of these tanks have been characterized. The tanks have been inactive for periods of time ranging from about 1 to 30 years, and minimal information was available on the composition of materials within the tanks.

Aqueous liquid and sludge samples from the waste storage tanks were subjected to modified U.S. Environmental Protection Agency (EPA) SW-846 and Contract Laboratory Program methodology to determine Target Compound List and other Appendix VIII organic compounds. Preparation of decontaminated organic extracts in radioactive-zoned facilities permitted final organic analysis in a conventional GC-MS laboratory. Aqueous liquids were analyzed for major volatile organic compounds by direct aqueous-injection gas chromatography in a radioactive-zoned laboratory; trace volatile organics, by remote purge-and-trap followed by thermal desorption and a second purge-and-trap with GC-MS; and semivolatile organics, by solvent extraction and GC-MS. Organic liquids were analyzed for volatile and semivolatile organics and PCBs, while sludges were analyzed only for semivolatile organics.

With few exceptions, low microgram-per-liter (liquids) and milligram-per-kilogram (sludges) concentrations of regulatory organic compounds were found, and the major species were alcohols (e.g., methyl and ethyl alcohols), ketones (e.g., acetone), chlorinated hydrocarbons [e.g., bis(2-ethylhexyl)phthalate] and tributylphosphate. The modified EPA procedures performed reasonably well, although improvements in sample preparation methodology are needed in some areas (particularly for sludges). However, the regulatory analyses accounted for very little of the organic matter suggested by total organic carbon (TOC) measurements. Additional polar species including dibutylphosphate, di(2-ethylhexyl)phosphate, and ethylenediaminetetraacetic acid were tentatively identified after trimethylsilylation of a sample, but the bulk of the TOC remains unidentified. It is speculated that chromatographic "intractables" such as polar, hydrophilic macromolecular matter may be present.

The goal of the inorganic analytical support for the inactive tank-sampling and characterization task is to provide the data necessary to meet both regulatory and engineering treatability requirements. The tanks are located in several areas throughout ORNL and collected radioactive waste from a large variety of programs and facilities over many years. Because of a lack of standard regulatory methods for the analysis of highly radioactive wastes and mixed wastes, the analytical support for this project was complicated by attempts to mimic existing regulatory procedures. With the complexity of the materials analyzed, the radioactivity, and the inappropriate regulatory procedures, this has been and continues to be a difficult and challenging undertaking. To date, the inorganic tank content measurements have included metals (U, Ag, As, Ba, Cd, Cr, Hg, Ni, Pb, Se, and Tl); radionuclides (^{137}Cs, ^{60}Co, ^{90}Sr, ^{3}H, ^{233}U, ^{238}U, ^{252}Cf, ^{238}Pu, ^{239}Pu,

^{244}Cm, ^{241}Am, ^{134}Cs, ^{228}Th, ^{232}Th, ^{232}U, ^{152}Eu, ^{154}Eu, ^{155}Eu, ^{14}C); anions (Cl^-, F^-, NO_3^-, PO_4^{-2}, SO_4^{-2}, CN^-, OH^-, H^+, CO_3^{-2}, and HCO_3^-); and several physical measurements.

Determination of the RCRA status of the tank contents depends upon the hazardous characteristics of the waste and upon a comparison of the waste constituents to the list of RCRA hazardous wastes contained in the Code of Federal Regulations (40 CFR Pts. 261.31, 261.32, and 261.33). Tanks that contained no RCRA characteristically hazardous (or potential RCRA characteristic as determined by the EP Toxicity equivalent of total RCRA metal concentration) or RCRA listed organic constituents were W1, W2, and T30. At this stage of analysis, the remaining tanks should be considered as containing RCRA waste.

1. SAMPLE COLLECTION FROM THE INACTIVE TANKS

1.1 INTRODUCTION

This report presents the results of a 2-year effort to sample and analyze the contents of the 33 inactive radioactive waste storage tanks located at the Oak Ridge National Laboratory (ORNL). To date, 30 of these tanks have been sampled and analyzed; sampling activities are in progress for the remaining three tanks. All these tanks no longer comply with U.S. Environmental Protection Agency (EPA) regulations for storage or treatment of hazardous wastes and, therefore, will require final closure. The sampling and analytical activities described in this report are the first steps necessary for final closure.

This section describes the sample collection activities associated with the 30 tanks. Sections 2 and 3, respectively, describe the methods used to characterize the organic contents in the tanks and the organic analytical results. Section 4 describes the radiochemical and inorganic characterization of the tank contents; Sect. 5 presents a regulatory analysis of the analytical results.

1.2 BACKGROUND

ORNL is located 40 km (25 miles) west of Knoxville, Tennessee, and began operation in 1943. Production of radioactive and/or hazardous chemical wastes has continued since its beginning as a part of normal facility operations. Underground storage tanks have been used to collect, neutralize, store, and transfer the liquid portion of these wastes. Of these tanks, 33 have been placed out of service because of operational difficulties or system improvements to the waste-handling operations. The 33 waste-storage tanks are located throughout ORNL, but most are located within the main plant area (Figs. 1.1 and 1.2).

In general, the wastes from these tanks were pumped out as they were removed from service. But because these tanks were managed by various divisions within ORNL, little information exists on the residual liquid and sludge that remain. These tanks were constructed of steel or concrete, and they vary in size and age (Table 1.1). Of the 30 tanks sampled to date, 27 were found to contain residual liquid, and 17 to contain sludge.

1.2.1 Need for Sampling and Analysis

The primary purpose for sampling the inactive waste tanks is to determine whether these tanks contain hazardous wastes as defined by the EPA Resource Conservation and Recovery Act (RCRA) regulations (40 CFR Pt. 261, Subparts C and D). Closure deadlines would then be in force for any tanks containing such RCRA wastes. Of equal importance, the tank contents need to be characterized sufficiently to select

ORNL DWG 89-13300

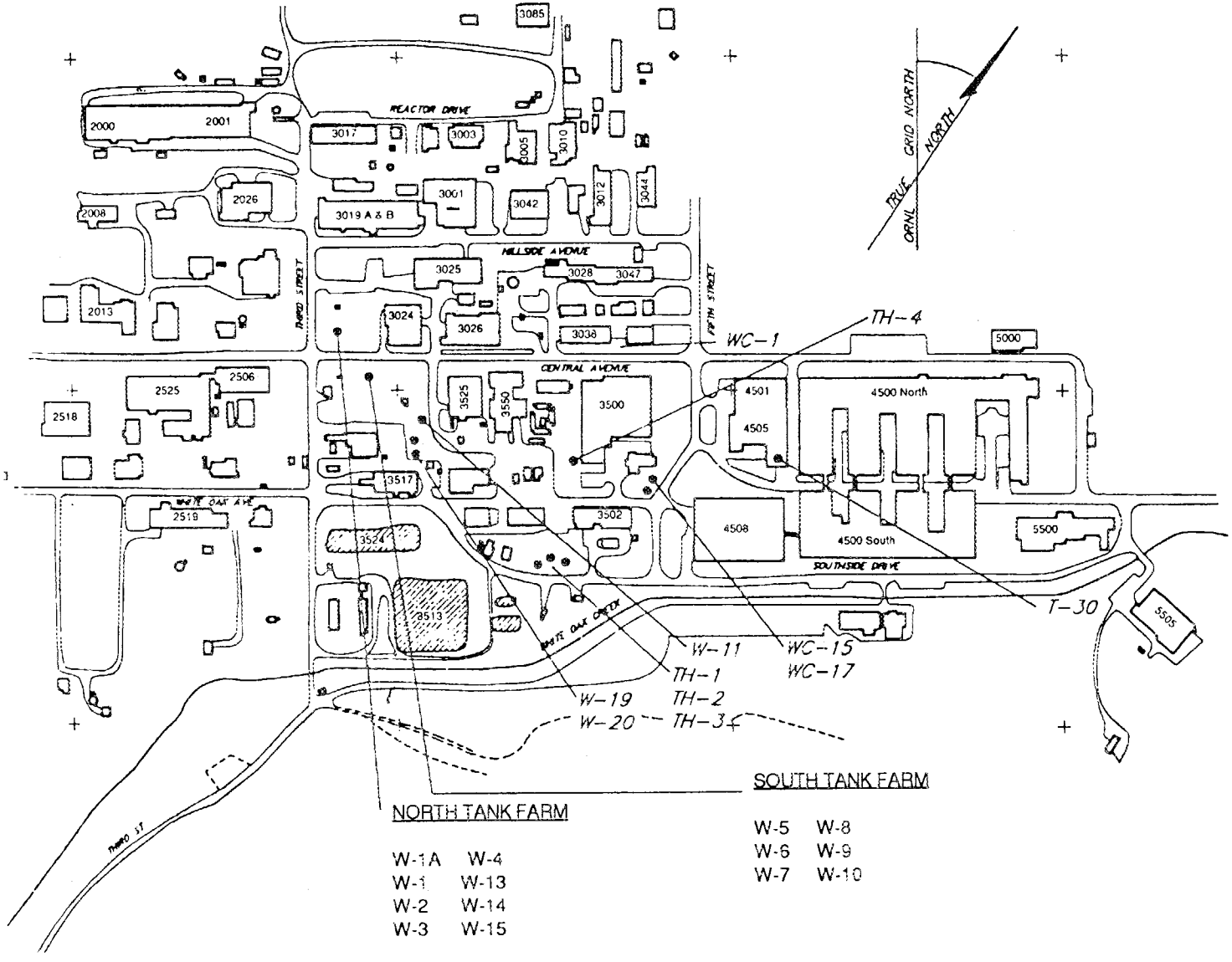


Fig. 1.1. Waste storage tanks within the main plant area.

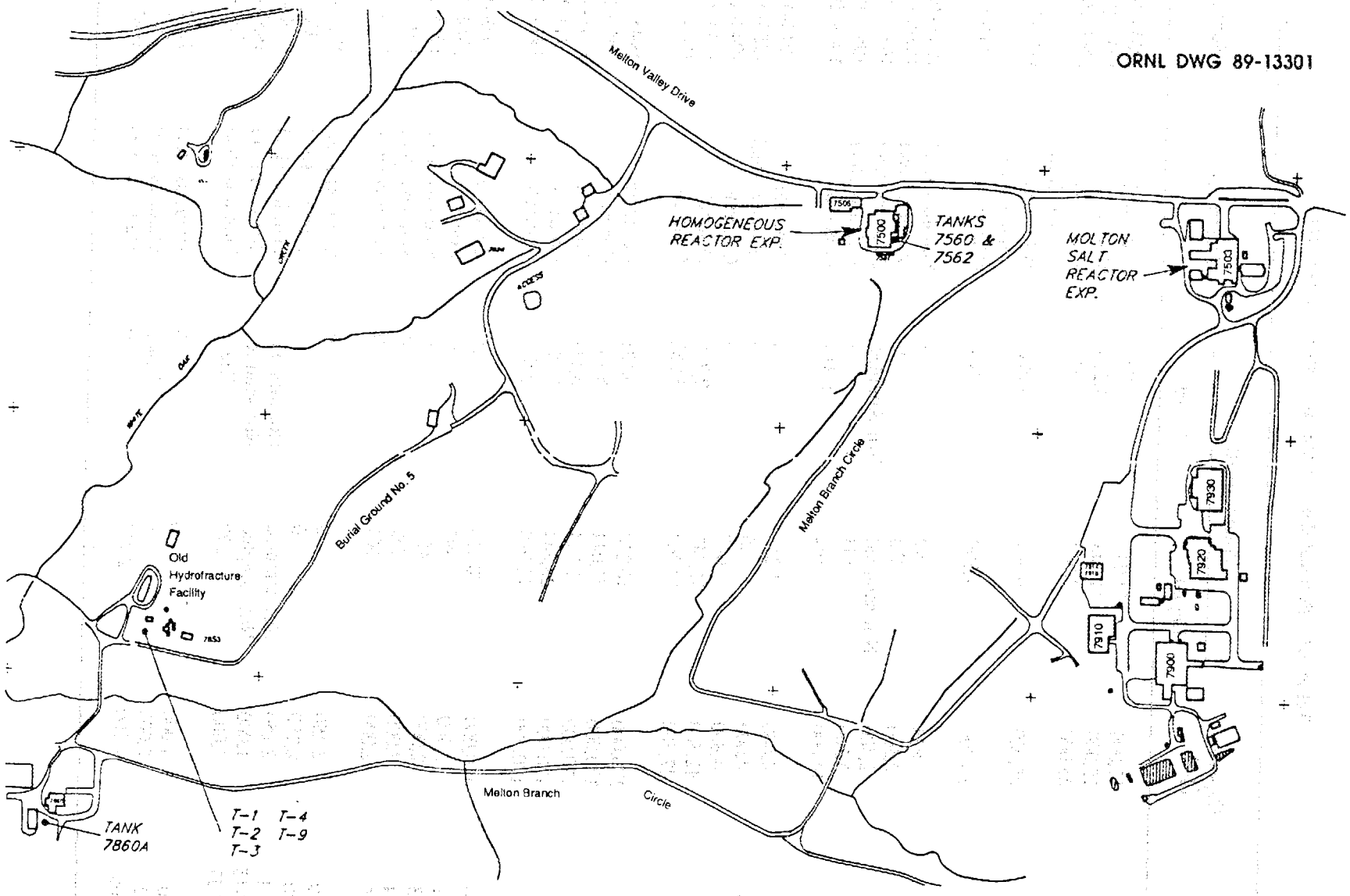


Fig. 1.2. Waste storage tanks outside the main plant area.

Table 1.1. Physical data of inactive waste storage tanks

Tank	Capacity (gal)	Volume (gal)		Construction	Service
		Liquid	Sludge		
T-1	15,000	11,047	791	Mild steel	1963–1980
T-2	15,000	11,048	1,205	Mild steel	1963–1980
T-3	25,000	2,063	2,029	Mild steel/ rubber lined	1963–1980
T-4	25,000	9,341	1,328	Mild steel/ rubber lined	1963–1980
T-9	13,000	1,290	481	Mild steel	1963–1980
T-30	824	40	0	Stainless steel	1945–1986
TH-1	2,500	278	0	Stainless steel	1948–1970
TH-2	2,400	Not sampled	Not Sampled	Stainless steel	1952–1970
TH-3	3,300	145	0	Stainless steel	1952–1970
TH-4	14,000	16,982	6,315	Concrete	1943–1970
W-1	4,800	1,213	0	Concrete	1943–1960
W-1a	4,000	25	0	Stainless steel	1955–1986
W-2	4,800	647	0	Concrete	1943–1960
W-3	42,500	31,847	2,276	Concrete	1943–1960s
W-4	42,500	17,062	3,982	Concrete	1943–1960s
W-5	170,000	10,278	5,131	Concrete	1943–1978
W-6	170,000	77,044	5,131	Concrete	1943–1978
W-7	170,000	7,044	5,131	Concrete	1943–1978
W-8	170,000	28,244	2,573	Concrete	1943–1978
W-9	170,000	12,990	2,573	Concrete	1943–1978
W-10	170,000	70,618	6,425	Concrete	1943–1986
W-11	1,500	897	62	Concrete	1944–1948
W-13	2,000	457	0	Stainless steel	1945–1958
W-14	2,000	259	0	Stainless steel	1945–1958
W-15	2,000	664	0	Stainless steel	1945–1958
W-19	2,250	0	0	Stainless steel	1956–1960
W-20	2,250	0	0	Stainless steel	1956–1960
WC-1	2,150	Not sampled	Not sampled	Stainless steel	1950–1968
WC-15	1,000	Not sampled	Not sampled	Stainless steel	1953–1960
WC-17	1,000	370	Trace	Stainless steel	1953–1960
7560	1,000	0	0	Stainless steel	1957–1961
7562	12,000	378	Trace	Stainless steel	1957–1986
7860a	4,500	2,774	0	Stainless steel	1981–1985

viable treatment strategies and meet final waste-form criteria. Among the possible treatment strategies considered are in situ grouting, in situ vitrification, incineration, chemical desorption, and transfer of the liquid wastes into the active waste treatment facility at ORNL.

1.3 SAMPLING ACTIVITIES

1.3.1 Sample-Collection Strategy

From the outset of this project, it was realized that sample analyses would provide only a relative quantification of the tank liquid and sludge contents and were not meant to be statistically defensible according to EPA SW-846 protocol. Because of the physical design of most of the tanks, sample collection could take place only from within a very limited area inside the tank (Fig. 1.3). Sample quantities were also limited to minimize radiation exposure to the field personnel collecting the samples. However, given the length of time the tank contents have had to settle, very little nonhomogeneity was expected to be found from different areas within the tank liquids.

1.3.2 Collection of Liquids

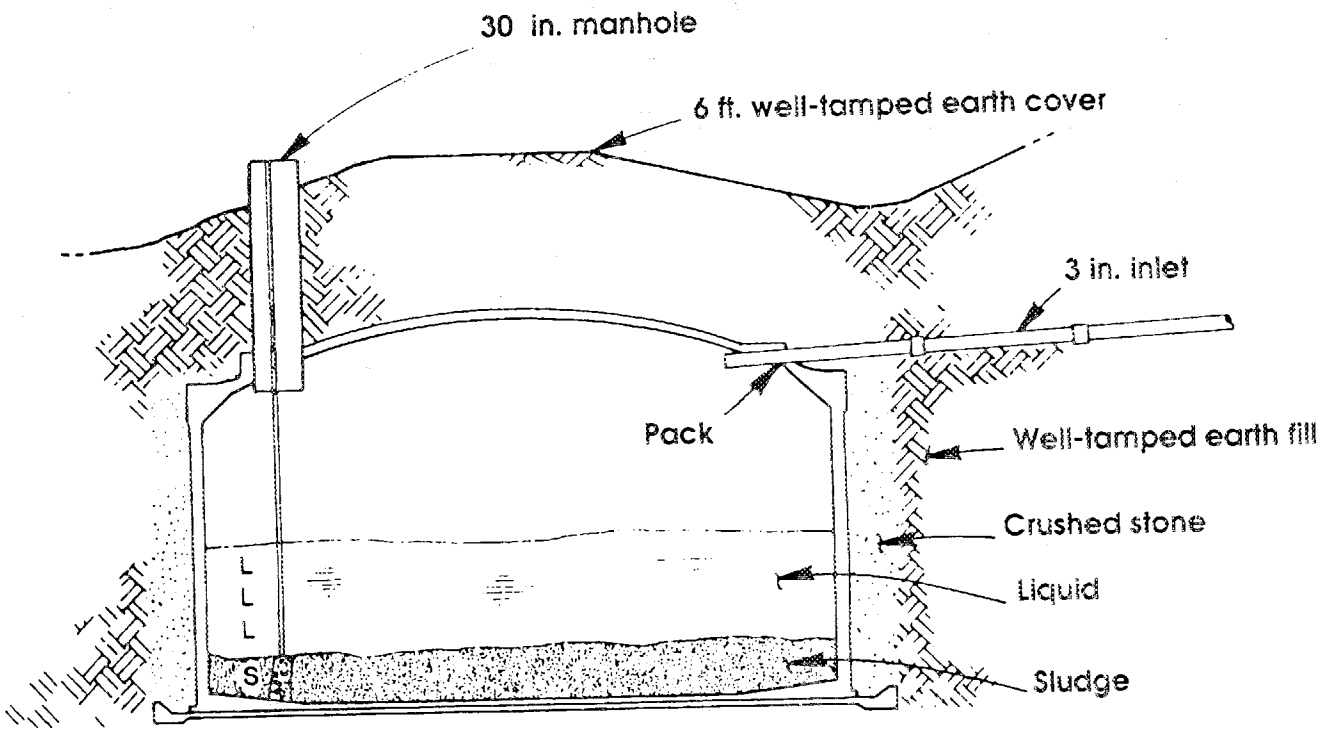
Most liquid samples were collected with a small vacuum pump, as shown schematically in Fig. 1.4. While it was realized that this procedure could volatilize the lighter organics in the liquid, this approach minimized radiation exposure to the personnel and was quite simple to operate. Liquid samples were collected into 250-mL glass sample jars with Teflon -lined caps and given unique identification numbers. The samples were surveyed by the Health Physics (HP) technician, sealed, placed in a lead pig, and immediately delivered to the High Radiation Level Analytical Laboratory (HRLAL). Usually, a second sample was collected and archived for future studies.

Vertical stratification within the liquid was expected in several tanks. Therefore, for tanks with liquid levels greater than 5 ft. deep, liquid samples were usually collected near the top at the midpoint, and at the bottom of the tank. Otherwise, samples were collected from the top and bottom of the tank. For very shallow liquid levels, only one sample was collected.

1.3.3 Collection of Sludges

As stated earlier, 17 of the tanks sampled were found to contain sludge. Liquid/sludge interfaces in the tanks were found using the Markland Model 10 Sludge Gun. This tool measures the amount of light transmitted across a fixed gap in the probe to detect changes in percent solids of the liquid. As the probe enters the sludge, an alarm sounds and the operator logs the depth. From earlier reports, "soft" and "hard" sludges were expected to be found in the tanks. Two different sludge collectors were prepared, as shown in Figs. 1.5 and 1.6. Attempts to collect sludge were made first with the soft-sludge collector. This collector

ORNL DWG 89-13302



L = liquid sample

S = sludge sample

Typical tank dimensions = 25' wide by 15'-2" high

Fig. 1.3. Typical tank configuration with proposed sample location.

ORNL DWG 89-13303

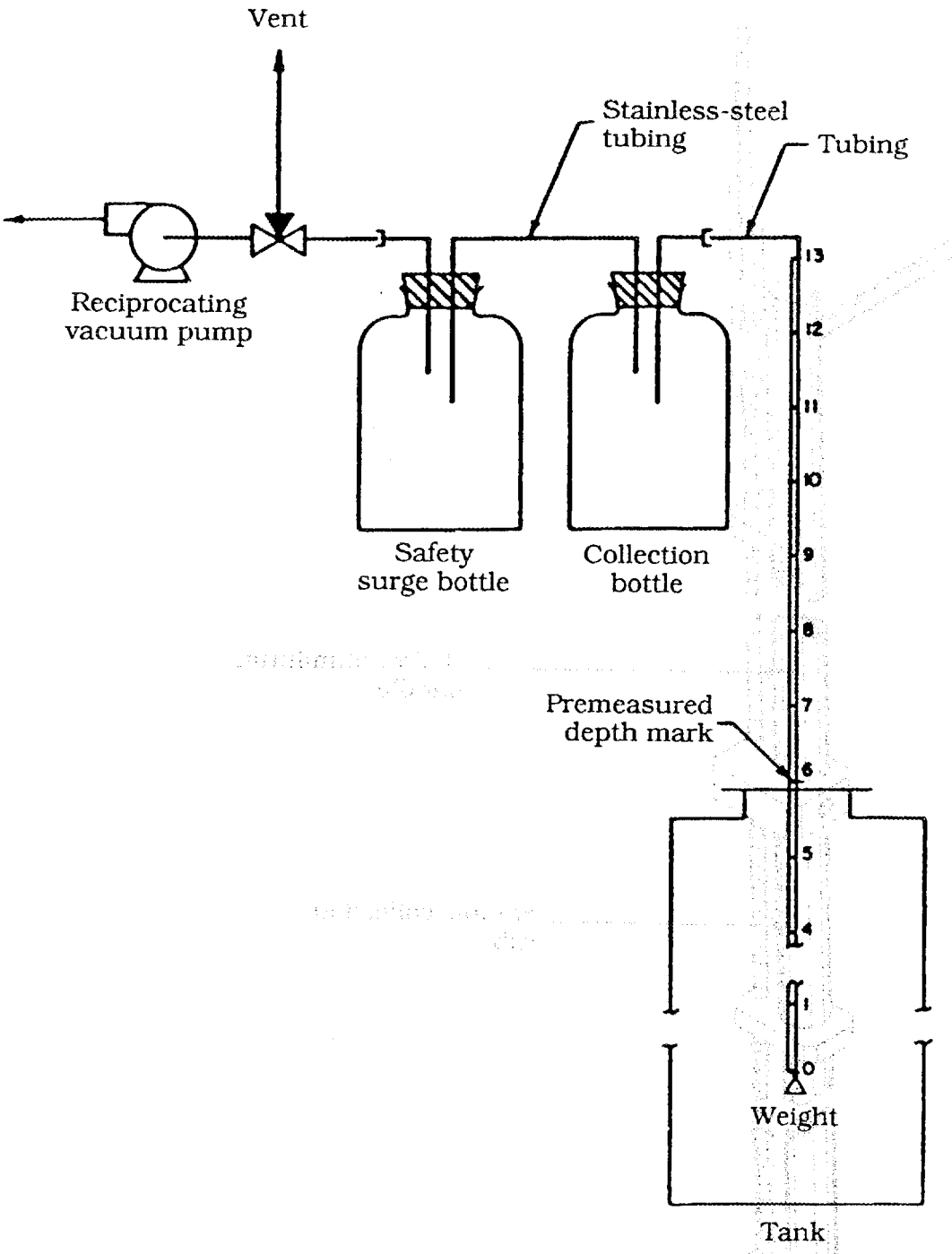


Fig. 1.4. Liquid sample collection system.

ORNL DWG 89-13304

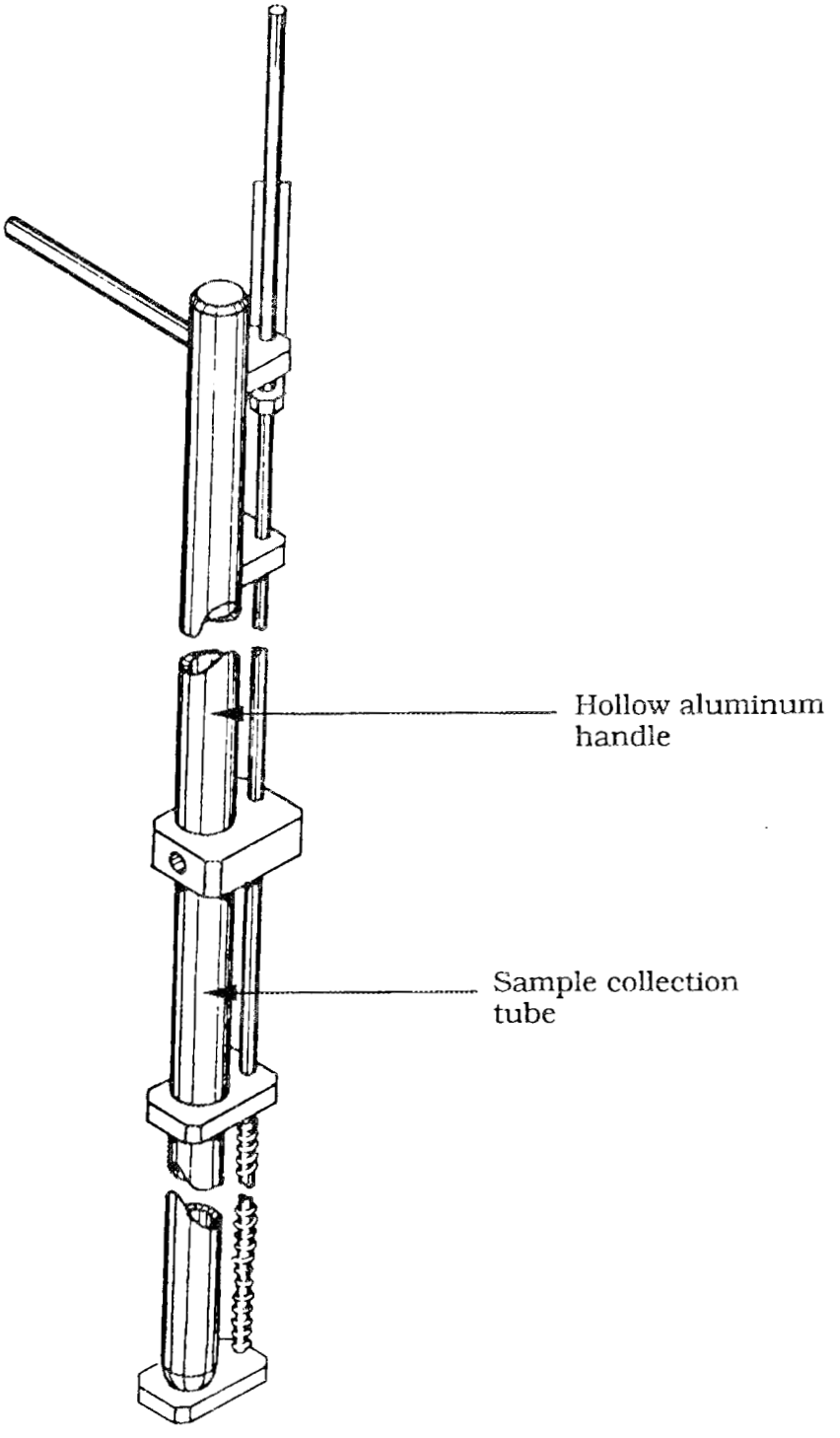


Fig. 1.5. Soft sludge sampler.

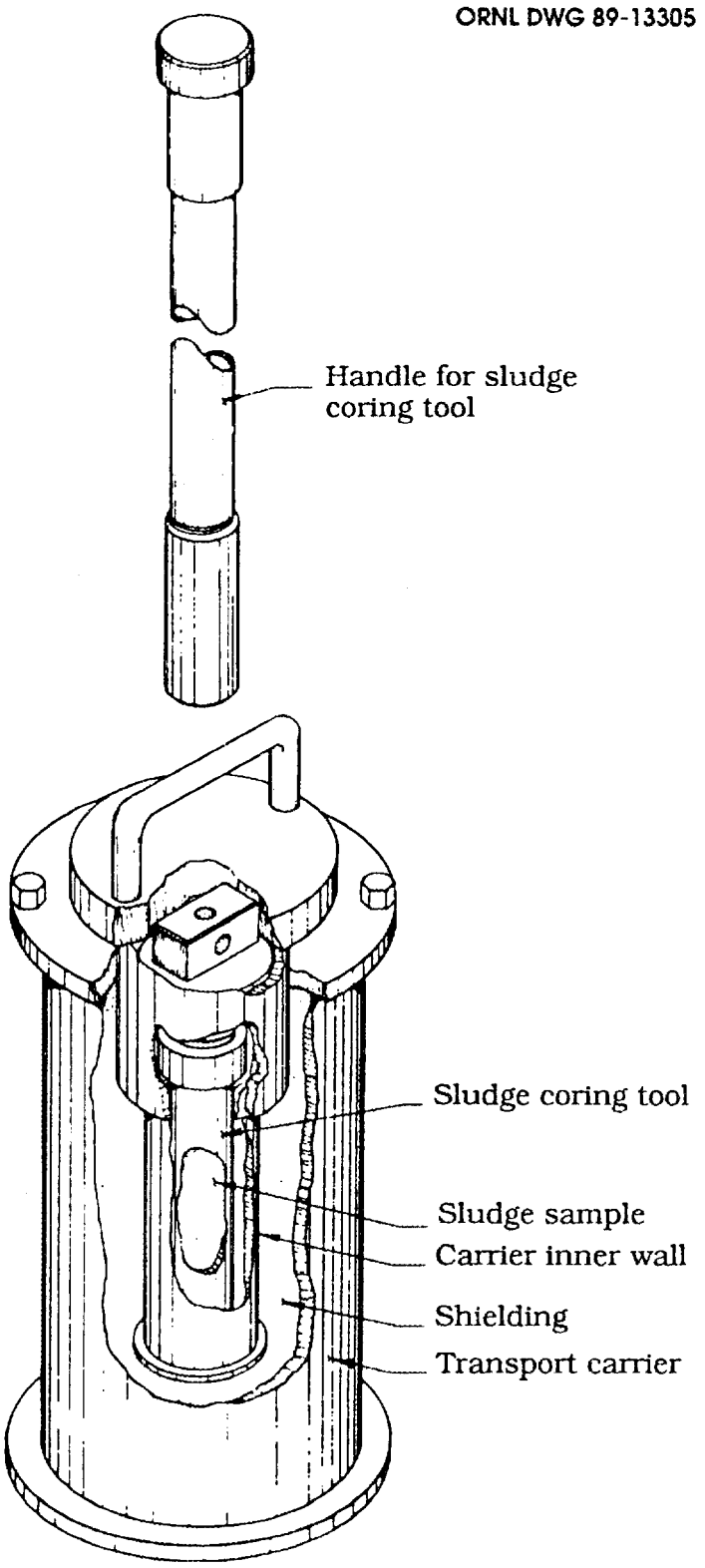


Fig. 1.6. Hard sludge sampler.

has an open-ended sample-collection tube at the bottom. After the sludge enters the collection tube, a flat plate coated with neoprene is rotated over the opening.

The hard-sludge collector uses a beveled cutting edge to core samples as it is driven into the sludge. Therefore, this collector was not used on the steel tanks for fear of puncturing the tank shell. Hard sludges were recovered from only 4 of the 12 concrete tanks sampled.

The sludge samples were surveyed by the HP technician, placed in protective containers, and immediately delivered to the HRLAL. No sludge samples were archived because of the high activity levels expected to be found.

1.4 QUALITY ASSURANCE

1.4.1 Sample Collection

Quality assurance during tank sampling activities was maintained primarily through the use of tank-specific procedures for the sampling crew to follow. These procedures provided step-by-step instructions for the collection, labeling, and shipping of each sample. If any procedural changes were required, the Project Manager was notified, and approval was required from the appropriate levels of management before operations could begin. An independent surveillance by the Quality Assurance Specialist verified that sampling procedures, chain-of-custody criteria, radiation protection, and operator training were in compliance with all written procedures.

All field data, such as liquid and sludge depths, sample identification numbers, location of the sampling port on the tank, 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 individual samples from their collection point to the individual laboratories conducting the analyses.

1.4.2 Sample Analyses

Quality assurance during sample analyses is more fully described in Sects. 2 through 4 of this report. Although there are no standard regulatory methods for the analysis of radioactive wastes or mixed wastes (i.e., radioactive wastes mixed with hazardous constituents), the HRLAL attempted to follow as closely as possible the regulatory procedures for the analysis of hazardous wastes (EPA SW-846,[1] EPA CLP,[2] and US EPA-600/4-79-020[3]). No attempts were made to spike samples during collection because spiking would increase exposure to the sampling crew. In addition, to minimize exposure to the crew, regulatory procedures for filtration and acid preservation during collection were not followed.

1.5 REFERENCES

1. 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.

2. Oak Ridge National Laboratory, *U.S. EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis*, SOW No. 787, Oak Ridge, Tenn., 1987.

3. U.S. Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, Envirnomental Monitoring and Support Laboratory, Cincinnati, Ohio, Rev. March 1983.

2. ORGANIC ANALYTICAL METHODS

2.1 INTRODUCTION

No standard U. S. Environmental Protection Agency (EPA) methods exist for the preparation and analysis of mixed wastes, and very few laboratories have experience in this area. Therefore, the organic chemical characterization in this project required that much of the methodology be developed as it was needed rather than during a preliminary developmental phase.

The approach taken for the determination of regulated organic compounds in the samples collected from the inactive tanks was to prepare decontaminated extracts in (radioactive) contamination-zoned facilities and to perform the analytical measurements in conventional analytical laboratories. The preparation methodologies followed EPA SW-846[1] or Contract Laboratory Program (CLP) methodology[2] as closely as possible. Method deviations required by the radioactive nature or other characteristics of the samples were documented. The quantitative measurements were conducted by EPA CLP methods. The staff of the High Radiation Level Analytical Laboratory received and logged in the samples and transferred aliquots to the Separations and Synthesis Group for organic analytical preparation. Except for the direct aqueous-injection—gas-chromatographic determination of major volatile organic compounds, all the quantitative measurements were performed by the Organic Analysis Group. All sample transfers were accompanied by chain-of-custody documentation.

The CLP analyses were specifically calibrated for the EPA CLP Target Compound List (TCL). Because EPA Region IV staff suggested analyzing for as much of the Appendix VIII list as possible within the constraints of worker protection, the detection of such compounds as Tentatively Identified Compounds (TICs) also was investigated. The following subsections describe the methods used in this project.

2.2 VOLATILE ORGANIC COMPOUNDS ANALYSIS

2.2.1 Method Description

2.2.1.1 Radioactive aqueous liquids

Volatile organic compounds were determined using modifications of SW-846 method 5030 and the CLP method for volatiles in water by purge-and-trap gas chromatography-mass spectrometry (GC-MS), which is very similar to SW-846 method 8240.

The main deviation from EPA methodology was that the purge-and-trap step was conducted in a glove box that is remote from the GC-MS laboratory. Figure 2.1 shows the purging head being assembled inside a glove box. The heavy gloves considerably limit freedom of motion and manual dexterity. The unopened bottles of aqueous liquid samples were bagged into the glove box after initial gross alpha and beta/gamma

Fig. 2.1. Purging apparatus being assembled in a glove box for the collection of volatile organic compounds from radioactive liquid samples.

contact readings. This health physics screen was conducted to determine how many samples could be taken safely into the glove box at one time. Because the volatile organic compounds can escape from an aqueous sample when the bottle is opened, the aliquot for purge and trap was taken at this first opening of the sample bottle. Five mL of liquid were pipetted into a 40-mL volatile organics analysis (VOA) vial, 10 μL of the CLP purgeables surrogate standard mix were added, and the vial was immediately sealed into the sampling head for purging. The volatile organic compounds internal standard solution was not added at this step because of the extra purge-and-trap step (see below) conducted later in the GC-MS laboratory. The sampling head was a machined block of Teflon™ with female threads fitting a 40-mL VOA vial. It also had Teflon lines for bubbling nitrogen through the sample; with these lines, the purged gas stream was carried through a bulkhead union in the wall of the glove box to an EPA method 624 triple adsorbent trap located exterior to the glove box. The sample was purged for 11 min at a flow rate of 30 mL/min. The nitrogen flow was maintained with a flow controller upstream of the sampling head and was monitored using a rotameter located downstream of the adsorbent trap. The system was checked for leaks before each purging by screwing a new VOA vial into the sampling head and monitoring the flow rate. Each sample was prepared in duplicate, but only one replicate was routinely analyzed.

The CLP purgeables surrogate standard mix was added to each sample. The following compounds were spiked at a concentration of 50 μg/L in the sample: toluene-d_8, 1,2-dichloroethane-d_4, and 4-bromofluorobenzene. A blank, a matrix spike (MAS), and matrix spike duplicate (MSD) were prepared with each sample group. The MASs and MSDs were prepared from one of the samples by adding 10 μL of an MS solution before purging. The MAS compounds and their concentrations in the MAS/MSD are listed in Table 2.1.

Table 2.1. MAS compounds in purge-and-trap GC-MS analysis of volatile organic compounds

Compound	Concentration, μg/L
1,1-Dichloroethene	51
Trichloroethene	72
Benzene	43
Toluene	43
Chlorobenzene	55

After the purging step, the adsorbent traps were screened for gross alpha and beta/gamma activity on contact and smear to detect radioactive contamination; then they were transferred with a chain-of-custody form to the Organic Analysis Group Sample Receiving Laboratory and logged in for analysis by GC-MS. The analysis was begun by thermally desorbing the adsorbent trap in a tube furnace held at 182°C while

helium was flowed though the trap at a rate of 35 mL/min for 11 min. The helium stream was bubbled through 5 mL of laboratory-distilled water held in a conventional purge-and-trap vessel in a Tekmar LSC-2 purge-and-trap apparatus. The water contained the CLP internal standards. This bubbling was part of the CLP method conventional purge-and-trap cycle that transferred the sample to the internal trap in the LSC-2 and then to the GC-MS for identification and quantitation by the CLP internal standard procedure.[2] In SW-846 method 8240, the internal and surrogate standards are added together to an aqueous sample just before purging. In this work, the surrogate standard was added before the first purge-and-trap step in the glove box, and the internal standard was not added to the sample until the second purge-and-trap step conducted in the analytical laboratory. This deviation allowed the overall recoveries from both purge-and-trap steps to be evaluated with the surrogate standard; it also allowed problems in the first and second purge-and-trap steps to be differentiated. The reporting limits for this method are essentially the same as those for the SW-846 or CLP methods, 5 to 10 μg/L.

2.2.1.2 Aqueous liquids with very little radioactivity

The aqueous liquids in tanks WC-17 and 7860A contained little radioactivity and could be analyzed in a conventional laboratory. However, several of these samples were in contact with organic layers in the tank, and the levels of organic matter dissolved in the aqueous layers were expected to be high. Therefore, small amounts were placed in 1.5-mL septum-capped vials without leaving any headspace, and these vials were screened by the Health Physics Inspector before they were transferred to the Organic Analysis Group sample receiving laboratory for analysis by the CLP purge-and-trap GC-MS method (similar to SW-846 method 8240). A 50-μL (tank 7860A) or 0.5-mL (tank WC-17) aliquot of the sample was added to 5 mL of laboratory-distilled water and was analyzed by CLP protocol. The reporting limits for these modifications, about 500 to 1000 μg/L and 50 to 100 μg/L respectively, were not as sensitive as those of the normal procedure because of the smaller sample aliquot.

2.2.1.3 Organic liquids

The organic liquids in tanks WC-17 and 7860A did not contain appreciable radioactivity and could be analyzed in a conventional laboratory. A 200-μL aliquot of each sample was diluted to 9.1 mL with Burdick and Jackson purge-and-trap grade methanol, and was sealed in a septum-capped vial. The vial was screened by the Health Physics Inspector before being transferred with a chain-of-custody form to the Organic Analysis Group sample receiving laboratory for log in and analysis by purge-and-trap GC-MS using the CLP method (similar to SW-846 method 8240). A 5-μL (tank 7860A) or 0.5 mL (tank WC-17) aliquot of the diluted sample was added to 5 mL of laboratory distilled water and analyzed by the CLP method. This procedure was much less sensitive (reporting limits of 230,000 to 460,000 μg/L and 2300 to 4600 μg/L,

respectively) than the normal purge-and-trap procedure, but this was necessary to prevent serious overloading of the GC-MS.

The data reported from the volatile organic compound analyses included the CLP TCL, the 10 to 20 most prominent TICs, and surrogate and MAS/MSD recoveries.

2.2.2 Method Evaluation

As a test of the overall procedure, Burdick and Jackson™ distilled-in-glass grade water was spiked with the CLP Purgeable Mixture A, Purgeable Mixture B, Hazardous Substance List Volatiles standards, and other selected volatile compounds from Appendix VIII at a concentration of 50 μg/L and selected alcohols and ketones at about 20 mg/L. These standards were purged onto traps in the glove box. Stock solutions of the latter two standards also were provided to the GC-MS laboratory for spiking into laboratory-distilled water and direct purge-and-trap GC-MS analysis to compare with the remote purge-and-trap procedure. A supplementary standard of additional volatile organic compounds chosen from Appendix VIII also was prepared and spiked into water at 50 to 60 μg/L for purging in the glove box as well as in the conventional laboratory for GC-MS analysis. The results of these experiments are summarized in Table 2.2.

The mixed standard was prepared from the CLP Purgeable Mixture A and B, and the Hazardous Substance List Volatiles standard was analyzed in triplicate by spiking into 5 mL of water and remote purge-and-trap in the glove box and GC-MS in a conventional laboratory; it was analyzed once by spiking into 5 mL of water at the conventional purge-and-trap GC-MS. The data in Table 2.2 demonstrate reasonable recovery of the compounds by the remote purge-and-trap, except for the ketones, vinyl acetate, bromoform, and 1,1,2,2-tetrachloroethane (low recoveries) and carbon disulfide and 1,1-dichloroethene (recoveries greater than 100%). The causes of this behavior are not known, but it is suspected that the low recoveries of the ketones may result from their high water solubilities and a consequently greater resistance to purging. The conventional purge-and-trap achieved higher recoveries, suggesting that the purging with the apparatus in the glove box was not sufficiently vigorous. However, this must be balanced against the need to prevent sample foaming and radioactive contamination of the glove box apparatus, connecting lines, and traps.

The limited experiments with 20-mg/L concentrations of alcohols and ketones (which modeled the volatile organics composition of some of the waste-tank liquids) show that the ketones are not completely recovered and quantitated at these high levels and that the alcohols (except for ethyl alcohol) are not determined at all. This behavior is not surprising considering that the purge-and-trap method is geared for μg/L concentration levels and most of the alcohols are not on the Appendix VIII list. For this reason, the purge-and-trap GC-MS was supplemented by the direct aqueous injection GC method described below.

Table 2.2. Comparison of supplementary volatile organic compound standard analyses with purge-and-trap in the glove box and the GC-MS lab

Compound	Concentration,[a] (μg/L)		
	Actual	Glovebox	GC-MS Lab
CLP Purgeable Mixture A, B, and Hazardous Substances List[b]			
Methylene chloride	50	37 ± 15 B	53 B
Acetone	50	56 ± 15 B	37 B
Carbon disulfide	50	96 ± 3.6	63
1,1-Dichloroethene	50	70 ± 1.7	52
1,1-Dichloroethane	50	57 ± 1.5	50
1,2-Dichloroethene (total)	50	54 ± 2.5	50
Chloroform	50	56 ± 2.5	48
1,2-Dichloroethane	50	43 ± 2.5	50
2-Butanone[c]	50	12 ± 1.7	43
1,1,1-Trichloroethane	50	49 ± 1.5	48
Carbon tetrachloride	50	53 ± 1.5	50
Vinyl acetate	50	20 ± 1.6	18
Bromodichloromethane	50	43 ± 2.1	49
1,2-Dichloropropane	50	50 ± 1.5	54
Cis-1,3-dichloropropene	50	36 ± 4.0	50
Trichloroethene	50	53 ± 1.5	56
Dibromochloromethane	50	38 ± 4.5	54
1,1,2-Trichloroethane	50	37 ± 3.1	54
Benzene	50	51 ± 1.5	50
Trans-1,3-dichloropropene	50	41 ± 3.1	49
Bromoform	50	23 ± 4.6	5
4-Methyl-2-pentanone[d]	50	13 ± 2.1	52
2-Hexanone	50	9 ± 1.0	40
Tetrachloroethene	50	44 ± 3.5	56
1,1,2,2-Tetrachloroethane	50	19 ± 5.1	56
Toluene	50	49 ± 1.0	50
Chlorobenzene	50	43 ± 3.5	51
Ethylbenzene	50	37 ± 4.6	48
Styrene	50	33 ± 5.7	42
Xylenes (total)	50	35 ± 2.5	47

Table 2.2 (continued)

Compound	Concentration,[a] (μg/L)		
	Actual	Glovebox	GC-MS Lab
	Appendix VIII Supplementary Standards:[b]		
1,2,3-Trichloropropane	55	11[e]	21
Dibromomethane	50	ND	ND
1,2-Dibromoethane	50	12 ± 0.6	14
Trans-1,4-Dichloro-2-butene	51	6[e]	10
Florotrichloromethane	60	43 ± 3.6	40
Methacrylonitrile	50	ND	ND
Metylmethacrylate	50	ND	8
Acrylonitrile	50	ND	ND
Iodomethane	50	33 ± 4.7	28
2-Picoline	50	ND	ND
Pyridine	51	ND	ND
	Alcohol/Ketone Standards[f]		
Methyl alcohol	20,000	ND	ND
Ethyl alcohol	22,600	5,600	ND
Acetone	21,900	9,100	4,700
i-Propyl alcohol	21,700	ND	ND
Allyl alcohol	19,900	ND	ND
2-Butanone[c]	20,500	1,000	6,200
i-Butyl alcohol	20,500	ND	ND
n-Butyl alcohol	20,800	ND	ND
4-Methyl-2-pentanone[d]	21,800	1600	3700

[a]ND = not detected; B = also detected in blank.
[b]Average ± SD for n = 3 in glovebox preparation; single preparation in GC-MS laboratory.
[c]Also named methylethylketone.
[d]Also named methylisobutylketone.
[e]Detected in 2 samples, average listed.
[f]Average of duplicate preparation in glovebox; single preparation in GC-MS laboratory.

The final evaluation consisted of the analysis of a supplementary set of volatile organic compounds selected from Appendix VIII and spiked into water (as above) both in the glove box and in the conventional GC-MS laboratory. The chlorinated compounds were the only ones detected reasonably well, and the data are estimations as TICs. The other compounds (e.g., acrylates and nitrogen heterocyclics) were not recovered. This was expected because it is recognized that they are not determined very accurately using

conventional purge-and-trap methodology and that they require special purging procedures or different analytical methods. Their inclusion here was to gain only an indication as to whether they could be detected if they were present in the samples, since it was not practical to set up specialized analytical methods for every conceivable Appendix VIII compound.

The main conclusion from this limited evaluation of the remote purge-and-trap method was that it would perform reasonably well for the bulk of the EPA TCL chemicals, with the exception of alcohols and ketones (which are determined at milligram-per-liter concentrations using direct aqueous injection GC). Additional Appendix VIII compounds can be detected, but those most successfully determined are chlorinated hydrocarbons.

2.3 MAJOR VOLATILE ORGANIC COMPOUNDS

2.3.1 Method Description

At the same time that aliquots were taken for purge-and-trap in the glove box, 1.5-mL aliquots were taken in small vials for analysis of major volatile organic compounds. This analysis was conducted to "protect" the GC-MS used for volatiles analysis by identifying those samples that were heavily contaminated with higher boiling compounds (boiling point $\geq$ about 100 to 140°C) that might be collected in the adsorbent trap and subsequently would contaminate the GC column or overload the detector in the GC-MS. It also was used to supplement the purge-and-trap analysis by providing data on highly polar, water-soluble organic compounds that are not determined very well by purge-and-trap (e.g., methyl and ethyl alcohols) but were suspected of being present from knowledge of processes used at ORNL. This procedure was originally intended to provide data of potential importance to selecting waste solidification strategies, but the finding of regulated constituents (e.g., some ketones) increased the necessity of including CLP-like quality control (QC).

The method used was direct aqueous injection GC, designed after SW-846 method 8015. The 1.5-mL vials of sample were taken into a radioactive contamination zone laboratory, and 3 μL were injected using the solvent flush technique into a gas chromatograph equipped as noted below. Aqueous samples from tank 7860A were first diluted (because of their high concentrations of organic compounds) by adding 10 μL of sample to 1 mL of laboratory-distilled water before analysis. The GC was equipped with a 3.2-mm-OD stainless steel column packed with 1% SP-1000 coated on 60/80 mesh Carbopack B (the same packing as used in the volatile organic compounds GC-MS), a flame ionization detector, and a reporting integrator. The helium carrier gas-flow rate was 30 mL/min, and the column temperature program was 70 (2-min isothermal hold) to 220°C at 16°C/min (final isothermal hold of 16 min). The inlet and detector were held at 150 and 250°C, respectively. The sample was analyzed with the method of external standards using peak areas and four concentrations of standards ranging from about 3 to 40 mg/L. Blanks and MASs were

analyzed with each group of samples. The latter were prepared by adding 10 or 20 μL of an approximately 2000 mg/L standard to 1 mL of sample. The MAS/MSD compounds that were spiked at nominal concentrations of 20 mg/L are listed in Table 2.3.

Blanks consisted either of laboratory-distilled water taken from the tap in the contamination zone or Burdick and Jackson distilled-in-glass grade water taken into the glove box and removed in the same type of vial as the tank water samples.

The GC conditions were slightly modified from those of method 8015. The other main deviation from method 8015 was that the instrument was fully calibrated each day of use with four concentration levels of standards, instead of a once weekly five-level calibration and daily single-level calibration checks. The detection limit was about 1 to 2 mg/L. An additional, qualitative confirmatory analysis on a GC column with a different stationary phase also was run at the end of the project to improve the confidence in identifications based solely on GC retention time. The second, qualitative GC analysis was performed on samples from those tanks which were found to contain any of the previous compounds in the original analysis. A 2.2-m- by 3.2-mm-OD glass column packed with a mixture of 60/80 and 35/60 mesh Tenax was used, with the same instrumental conditions as above.

The data reported included the concentrations of the compounds tentatively identified and the MAS/MSD recoveries.

2.4 SEMIVOLATILE ORGANIC COMPOUND ANALYSIS

2.4.1 Method Description

The extractions of liquid and sludge samples for semivolatile organic compounds were performed after the gross alpha and beta/gamma activity measurements were completed. This information was needed to determine how much sample could be taken for extraction in a hood or if the extraction had to be conducted in a hot cell.

2.4.1.1 Extraction of aqueous liquids

A 20-mL aliquot of aqueous sample was taken for semivolatile organic compound extraction in a contamination zone hood. The only exceptions were for the aqueous liquids from tanks 7860A and WC-17, for which 2 mL were used because of the high levels of organic matter expected from the overlying organic liquids. To each sample was added 1 mL each of the CLP base/neutral and acids surrogate standard solutions before extraction. The compounds added and their concentrations in the extract (derived from extraction of a 20-mL sample) are listed in Table 2.4.

Table 2.3. MAS and MSD compounds used in direct aqueous GC analysis of major volatile organic compounds (about 20 mg/L each)

Methyl alcohol
Ethyl alcohol
Acetone
i-Propyl alcohol
Allyl alcohol
2-Butanone
i-Butyl alcohol
n-Butyl alcohol
4-Methyl-2-pentanone

Table 2.4. Semivolatile surrogate standard compounds added to aqueous liquids

Compound	Concentration,[a] mg/L
Nitrobenzene-d_5	1.0
2-Fluorobiphenyl	1.0
4-Terphenyl-d_14	1.0
2-Fluorophenol	2.0
Phenol-d_6	2.0
2,4,6-Tribromophenol	2.0

[a]Concentrations are for 20-mL sample aliquot. The concentration is 10-fold higher for a 2-mL sample aliquot.

The sample was extracted by a modification of SW-846 method 3510. A 20-mL aliquot (or 2-mL aliquot of aqueous layers from tanks 7860A and WC-17) was taken in a 40-mL VOA vial, and the pH was checked using pH indicator paper. The pH of the initial extraction depended upon the natural pH of the sample. The pH was adjusted (if necessary) to >10 for the initial extraction of neutral and alkaline samples or was made <2 for the initial extraction of acidic samples, using 1 N sodium hydroxide or 1 N hydrochloric acid, respectively. Occasionally, stronger base or acid (ion) was required for samples exhibiting considerable buffering capacity. The sample was extracted three times using 5 mL (each time) of methylene chloride by capping the vial and tumbling about 30 to 40 times. More vigorous extraction caused emulsion formation. If particularly radioactive samples were to be extracted, vortexing followed by centrifugation was used to reduce worker exposure. The methylene chloride was pipetted out of the vial, and the occluded water was separated by passing the extract through a disposable 0.45-um Teflon filter cartridge of 25-mm diam

(Acrodisc-CR™) attached to the tip of a disposable 10-mL polypropylene syringe barrel. The pH was then adjusted to the other extreme, the sample was reextracted, and the methylene chloride was passed through the same filter and combined with the first set of extracts. The volume was reduced to 1 mL by nitrogen blowdown, and the concentrated extract was transferred to an autosampler vial. A 10-μL aliquot of the CLP semivolatile organic compound internal standard solution was added with a microsyringe, and the vial was sealed with a crimp-top cap and a Teflon-coated septum. The vial was stored at -20°C until GC screening. The reporting limits for 20- and 2-mL aqueous sample aliquots are about 500 to 2500 μg/L and 5000 to 25000 μg/L, respectively.

Extraction blanks and MAS/MSD samples were prepared with each set of samples. Blanks were prepared by extracting 20 mL of Burdick and Jackson distilled-in-glass grade water. MAS/MSDs also were prepared by spiking samples with 1 mL of the base/neutral and acid compound matrix spiking solution. The spikes and their concentrations in the sample are listed in Table 2.5.

Table 2.5. Semivolatile MAS compounds added to aqueous samples

Compound	Concentration,[a] (mg/L)
Phenol	9.7
2-Chlorophenol	10.4
1,4-Dichlorobenzene	5.3
N-nitroso-dipropylamine	7.7
1,2,4-Trichlorobenzene	6.95
4-Chloro-3-methylphenol	9.7
Acenaphthene	5.5
4-Nitrophenol	10.3
2,4-Dinitrotoluene	5.1
Pentachlorophenol	10.3
Pyrene	5.55

[a]Concentration is for 20-mL sample aliquot. Concentration is 10-fold higher for 2-mL sample aliquot.

2.4.1.2 Extraction of Sludges

Sludge samples were extracted for semivolatile organic compound analysis using a modification of SW-846 method 3550. Most of the extractions were performed in a hot cell (Fig. 2.2) because of the high levels of radioactivity in the sludges, although a few sludges were low enough in radioactivity to allow extraction in a contamination zone hood. The masses extracted varied from about 2 to 20 g because of the limited amounts of sludges collected. The amount of gas chromatographable organic matter extractable

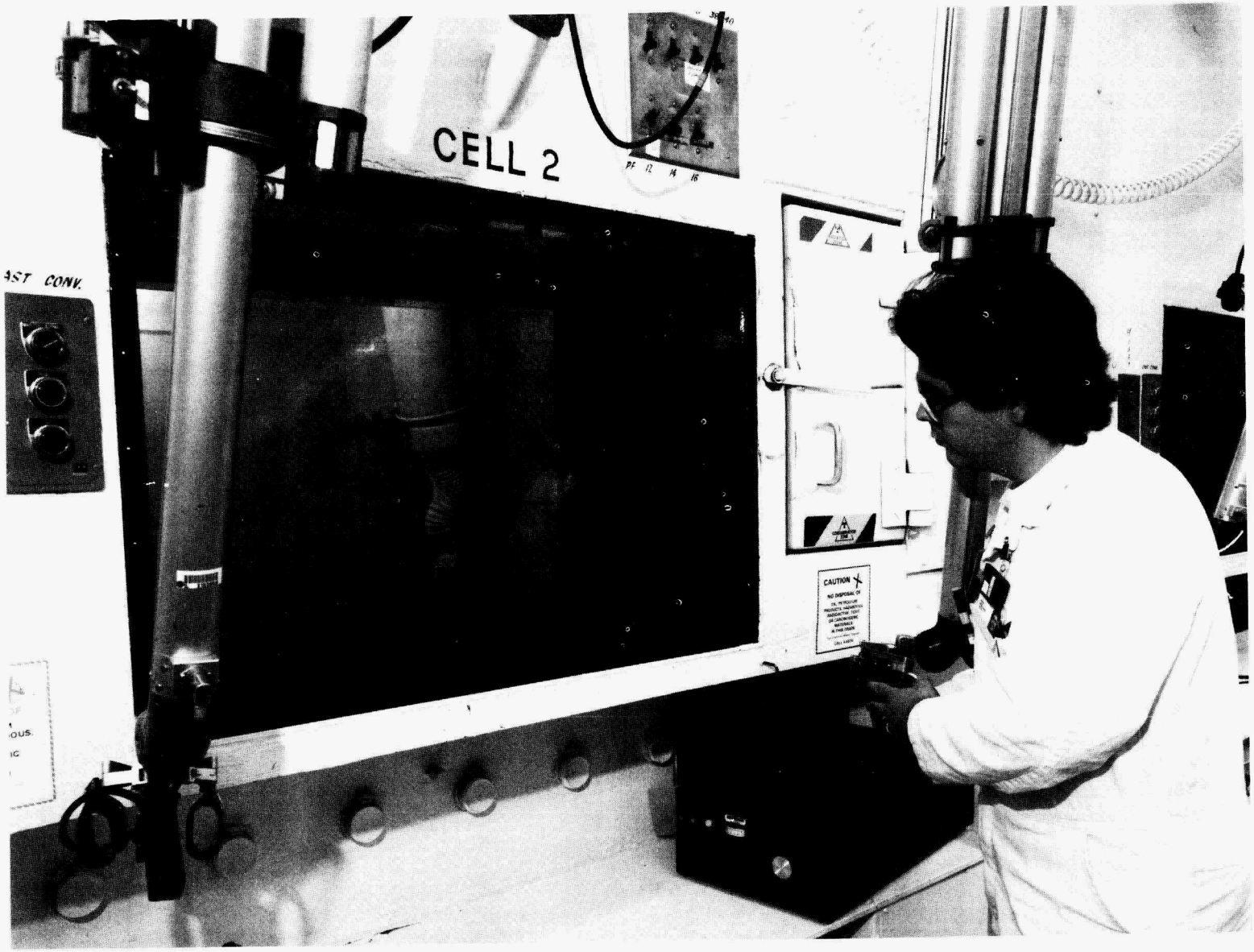


Fig. 2.2. Ultrasonic solvent extraction of semivolatile organic compounds from radioactive sludge, using a remote manipulator in a hot cell.

from the sludges required dilution of the extracts or extraction of smaller masses (e.g., 2–5 g). The reporting limits varied from about 500–2500 to 5000–25,000 μg/kg, depending upon the mass extracted and the dilution of the extract.

In the hot cell, the sludge was homogenized before aliquotting for the various analyses. The excess liquid was pipetted from the top of the sludge sample, and the remainder was then stirred with a stainless steel spatula and homogenized with a 450-W sonifier set for 50% duty cycle and the maximum power level without splattering. Early in the project, sludges (sample Nos. T-4/S-046, T-3/S-043, T-9/S-048, T-2/S-040, T-1/S-037, W-5/S-075, W-6/S-080, and W-7/S-084) were homogenized only by stirring. For semivolatile organic compound extraction, a 20-g aliquot of the homogenized sludge slurry was weighed out into a beaker. In cases where the amount of sludge collected was limited, a smaller mass (e.g., 10 g or less) was weighed out. The sludge was then spiked with the surrogate compounds by mixing with premeasured (pipetted into a vial) 1-mL volumes of the CLP surrogate standard solutions, and the slurry was stirred with a stainless steel spatula. A 20- to 40-g mass of anhydrous sodium sulfate was then slowly added with stirring. The mixture was then ultrasonically extracted in the beaker at a 50% duty cycle for 3 to 5 min with 60 to 70 mL of methylene chloride/acetone (1/1, vol/vol), and the supernatant liquid was decanted into a 150-mL, medium porosity sintered glass funnel. The solvent was recovered by filtration. The extraction was repeated twice more in the beaker. In early work with sludges through W-7/S-084, the extraction was conducted directly in the sintered glass funnel, but the sludge tended to plug the filter, especially sample W-7/S-074. The composited extracts were screened for alpha and beta/gamma activity. They were taken to a radioactive contamination zone hood for volume reduction via nitrogen gas blowdown under reduced temperature and pressure. The extracts were adjusted to a 1-mL volume, transferred to an autosampler vial, and 10 μL of internal standard were added. The vials were capped using a crimp-top with a Teflon-lined septum. The surrogate standards added to the sludges are listed in Table 2.6.

Table 2.6. Semivolatile organic surrogate standards added to sludge samples

Compound	Concentration,[a] (mg/kg)
Nitrobenzene-d_5	1.0
2-Fluorobiphenyl	1.0
4-Terphenyl-d_14	1.0
2-Fluorophenol	2.0
Phenol-d_6	2.0
2,4,6-Tribromophenol	2.0

[a]Concentrations are for 20 g sample aliquot. The concentration is 10-fold higher for a 2-g sample aliquot.

From 10 to 20 g of sludge were routinely extracted in the hot cell until it was established that this mass provided too much organic matter for the GC-MS and that a 10-fold dilution of the extract was needed before GC-MS analysis. The mass extracted was then reduced to about 2 to 5 g.

For the sludge extractions conducted in a hood, about 3 g of sludge W-3/S-020 (or about 9 g of sludges W-4/H-026 and W-4/S-024, or about 20 g of sludges W-11/H-005, TH-4/S-059, and TH-4/S-058) were mixed with an approximately twofold mass of sodium sulfate (W-3/S-020 required a fivefold mass) and 1 mL each of the CLP base/neutral and acid surrogate standard, and placed in a 60-mL medium porosity sintered glass funnel. The surrogate standards were about sevenfold more concentrated than in the 20 g-sample. The sludge was ultrasonically extracted three times (four times for W-3/S-020) with 40 mL (each time) of methylene chloride/acetone (1/1, vol/vol), using a 350-W ultrasonicator fitted with a 1.5-cm OD horn. The extracts were filtered, and the combined filtrates were reduced to a volume of 1 mL by nitrogen blowdown and transferred to an autosampler vial. A 10-μL aliquot of the internal standard solution was added, and the vial was sealed with a crimp-top septum-cap.

Blanks and MS/MSD samples were prepared but not with each extraction because the sludges generally were received one at a time (in contrast to the liquid samples) and the addition of a blank and two spikes with each extraction would have quadrupled the effort. Blanks were prepared by extracting reagent grade sea sand. Two aliquots of one sludge sample (about 3-g mass) were spiked with the base/neutral and acid compound matrix spiking standards listed in Table 2.7.

Table 2.7. Semivolatile organic MAS compounds added to sludge sample W-3/S-020

Compound	Concentration,[a] (mg/kg)
Phenol	65
2-Chlorophenol	69
1,4-Dichlorobenzene	35
N-nitroso-dipropylamine	51
1,2,4-Trichlorobenzene	46
4-Chloro-3-methylphenol	65
Acenaphthene	37
4-Nitrophenol	69
2,4-Dinitrotoluene	34
Pentachlorophenol	69
Pyrene	37

The GC screen determined that most of the extracts prepared from 20-g samples were too concentrated in organic matter and would overload the GC-MS. Therefore, a 10-fold dilution of the extracts from sludges T-1/S-037, T-2/S-040, T-3/S-043, T-4/S-046, TH-4/S-058, TH-4/S-059, T-9/S-048, and W-11/H-

005 was made by adding 10 μL of the extract to 90 μL of methylene chloride in a 100 μL crimp-top, septum-capped autosampler vial. A second group, consisting of sludges W-5/S-075, W-6/S-080, W-7/S-084, W-7/H-085, W-8/S-088, W-9/S-092, and W-10/H-120, was diluted 10-fold by adding 100 μL of extract to 900 μL of methylene chloride and an additional 9 μL of internal standard solution. Either dilution method may be used, but each affects the computerized GC-MS data report in a different way. Manual correction of the calculated results are necessary for either method with the software currently available for CLP GC-MS.

2.4.1.3 Preparation of organic liquids

Two tanks contained organic layers. These samples required only dilution and addition of internal standard, per SW-846 method 3580. After testing various dilution ratios with the GC screening method, it was determined that a 100-fold dilution would keep most of the gas chromatographable major constituents within the calibration range of the GC-MS. Ten μL of sample were added to 1 mL of methylene chloride in an autosampler vial, 10 μL of the semivolatile organic compound internal standard solution were added, and the vial was sealed with a crimped-top septum-cap. Because there was no extraction step, MAS samples were not prepared. However, for tank 7860A, 10 μL of the concentrated semivolatile organic compound surrogate standard stock solution were added to the samples to verify recoveries. The reporting limits for this dilution were high, about 1100-5500 mg/kg.

2.4.1.4 GC screen

All of the extracts were screened using a Megabore capillary column GC with flame-ionization detection. This screening was conducted to identify those samples requiring dilution to "protect" the GC-MS and to weed out those samples which contained little organic matter and which would not require GC-MS. Samples TH-3/L-052, TH-3/L-113 (a blind water blank), W-11/L-003, W-11/L-114, W-11/L-115, TH-1/L-049, TH-1/L-050, and W-14/L-031 were not submitted for GC-MS because the chromatograms were not different from the extraction blank. Almost all of the sludge sample extracts prepared from 10 to 20 g of sample required a 10-fold dilution to prevent contamination and overloading of the GC-MS.

A 0.53 mm-ID by 30-m fused silica column with 1.5-um DB-5 bonded phase was used with a helium flow rate of 7 mL/min and a column temperature program of 35 (4-min isothermal hold) to 270°C at a rate of 10°C/min with a final isothermal hold of 30 min. The inlet temperature was maintained at 270°C and the flame ionization detector at 290°C. A 1.6-μL injection was made using an autosampler. The instrument was not quantitatively calibrated, but multicomponent standards of 20- and 4-mg/L concentration were run with each set to verify sensitivity. The 4-mg/L standard (6.4-ng/compound injected) yielded peak heights on the printer/plotter of at least 1 cm at an attenuation of 2^4. The standards included the CLP TCL

base/neutral and acid standards, CLP TCL pesticide standards, and additional compounds selected from Appendix VIII. At least one standard prepared at 4 mg/L was run with each set of samples.

2.4.1.5 Analysis

After GC screening, the extracts were measured for total alpha and beta/gamma activity and then the vials were examined for contact and smear alpha and beta/gamma by the Health Physics Inspector. They were then sent to the sample receiving laboratory for GC-MS analysis. The extracts were analyzed by the CLP GC-MS procedure,[2] which utilizes the method of internal standards.

Data reported included the semivolatile organic compound TCL and the 10 to 20 most prominent TICs, the surrogate and MAS/MSD recoveries.

2.4.2 Method Evaluation

A limited evaluation was made of the ability of the GC-MS to identify and estimate other Appendix VIII compounds that might be present in the semivolatile organic extracts. In addition, the evaluation was extended to certain nonregulatory compounds such as extractants, chelators, and complexing agents unique to the nuclear industry that are highly likely to be present in the waste tank extracts. These compounds would be reported in the GC-MS analysis of the semivolatiles as TICs and the concentration data would be estimates only. For this purpose, four sets of supplementary standards were prepared at a concentration of 20 mg/L each in methylene chloride, and after addition of the semivolatiles internal standard, the standards were analyzed by the CLP GC-MS method. The results are listed in Table 2.8. The identifications and estimations were quite successful, considering that the identifications were made by machine (except for tributyl phosphate, which apparently was not included in the spectral library) with operator approval, and the concentrations were estimated based upon the response factor of the nearest eluting internal standard, as is normal procedure for TICs. A wide range of compounds, including nitrosamines, aromatic amines, chlorinated aromatics, nitrogen heterocyclics, and pesticides, were correctly identified and estimated. Exceptions apparently arose from very early-eluting species which were missed (e.g., *N*-nitrosodimethylamine and dioxane) to some compounds which may not have eluted from the GC column (e.g., some aromatic amines and hydrazines) under the conditions used. This test was conducted under normal GC-MS operation during a large sample campaign, and it is possible that sample residues in the GC inlet and column may have prevented the elution of some of the more polar species.

The general conclusion confirms a good probability that additional Appendix VIII compounds would be identified and estimated if they were present in the extracts at concentrations near 20 mg/L.

Table 2.8. Results of GC-MS analysis of supplementary semivolatile appendix VIII and other organic compounds as TICs

(All prepared at 20 mg/L solution concentration)

Compound	Estimated concentration as TIC (mg/L)
Semivolatiles supplementary standard 1	
N-nitrosodimethylamine	ND[a]
N-nitrosodiethylamine	11
Benzenethiol	13
Acetophenone	19
2,6-Dichlorophenol	12
1,4-Naphthoquinone	8
2-Naphthylamine	6
4-Aminobiphenyl	8
Benz(c)acridine	12
Dibenz(a,j)acridine	9
Semivolatiles supplementary standard 2	
p-Dioxane	ND[a]
Pentachloroethane	11
N-dimethyl-1-phenethylamine	ND
Hexachloropropene	7
1,4-Diphenylenediamine	ND
1,2,4,5-Tetrachlorobenzene	11
2,4-Diaminotoluene	ND
Pentachlorobenzene	12
1,2-Diphenylhydrazine	ND
Pentachloronitrobenzene	6[b]
Benzidine	ND
Hexachlorophene	ND
Pesticides standard	
alpha-BHC	14
beta-BHC	12
gamma-BHC	13
delta-BHC	11
Heptachlor	14
Aldrin	15
Heptachlorepoxide	12

Table 2.8 (continued)

Compound	Estimated concentration as TIC (mg/L)
Pesticides standard (continued)	
Endosulfan I	22
DDE	27
Dieldrin	22
Endrin	19
Endosulfan II	9
DDD	20
Endrin aldehyde	15
Endosulfan sulfate	18
Methoxychlor	15
Dibutylchlorendate	ND
Mirex	ND
DDT	ND
Extractants standard	
Diethylbenzene	20
2-Thenoyltrifluoroacetone	6
Tributylphosphate[c]	26
Di-s-butylphenylphosphonate	ND
2,5-Di-t-butylhydroquinone	10

[a]MS may have begun collecting data too far into GC run and may have missed these very early-eluting compounds.

[b]Pentachlorophenol was detected, suggesting decomposition in the GC or MS.

[c]Not identified by machine search; spectrum manually identified.

2.5 PCB ANALYSIS

2.5.1 Method Description

Two tanks contained layers of organic liquids or oils. These layers were analyzed for PCB content in addition to volatiles and semivolatiles. The preparation method was a modified version of method 3580. For tank WC-17, 1 g of organic liquid was weighed out, 1 mL of a dibutylchlorendate surrogate standard (0.99 mg/kg in the sample) was added, and the mixture was diluted to 10 mL using hexane/acetone (1/1, vol/vol). For tank 7860A, the same preparation procedure was used, but interferences observed in the GC analysis required a repreparation at a greater dilution. In the second preparation for tank 7860A, about 50 mg were weighed out, 50 μL of dibutylchlorendate surrogate standard were added (0.99 mg/kg in the

sample), and the mixture was diluted to 5 mL with hexane. At the GC laboratory, two additional aliquots of one sample were matrix spiked with 100 μL of a 100 ug/mL solution of Arochlor-1248 (200 mg/kg in the sample), and a drop of elemental mercury was added to each sample. A solvent blank accompanied the samples.

PCBs were measured by a capillary-column GC method, which is similar to SW-846 method 8080, but only the capillary column was used. The samples were injected into a GC equipped with a 10 m by 0.53 mm ID by 5 um film of bonded SE-30, fused silica capillary column and an electron-capture detector. The helium carrier gas flow rate was 6 mL/min, and the total flow rate to the detector was adjusted to 45 mL/min. with argon/methane (95/5, vol/vol). The column oven was programmed from 140 to 250°C at 8°C/min, with a 16 min final isothermal hold, and the injector and detector temperatures were maintained at 250 and 300°C, respectively. Quantitation was by the method of internal standards, using manually measured peak heights for peaks characteristic of each Arochlor mixture. The reporting limit was about 10 to 20 mg/kg.

Data reported were the concentration of each Arochlor and the MS/MSD recoveries.

2.6 REFERENCES

1. U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste*, SW-846, 3d ed., U.S. EPA Office of Solid Waste and Emergency Response, Washington, DC, (November 1986).

2. Oak Ridge National Laboratory, *US EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multimedia, Multiconcentration, 10/86*, rev. SOW No. 787, IFB WA-87K236, IFB WA-87K237, and IFB WA-87K238, Oak Ridge, Tennessee, 1987.

3. ORGANIC ANALYTICAL CHARACTERIZATION

3.1 INTRODUCTION

This section presents the results of the organic chemical analyses of the inactive waste tanks and attempts to provide some chemical interpretation of the findings. The impact of the findings upon the regulatory classification of the tanks is discussed in Sect. 5. This discussion is arranged by tank in the order in which the samples were prepared for analysis. Data tables are included in this chapter only for tanks where the analyses indicated levels of organic compounds appreciably above the associated blanks. The full set of data for each tank can be found in the Appendix of this report.

3.2 DATA-REPORTING CONVENTIONS

Some explanation of the reporting conventions is necessary to clarify how the data are presented in this chapter. The data are listed in the tables with letters which indicate important qualifications. Absence of a data qualifier(s) indicates that a compound was (a) identified on the basis of retention time and mass spectrum, (b) measured within the calibration range of the instrument, and (c) not found in the associated blank sample. The qualifiers present additional information, as follows:

J: The compound was identified by the appropriate retention time and mass spectrum but was observed at a concentration lower than the reporting limit, which is the lowest concentration of that compound that can be measured accurately in that sample. The quantitation is therefore an estimate. Only one significant digit is used in the discussion of the results for tentatively identified compounds (see below).

E: The concentration of the compound exceeded the calibration range of the instrument; therefore, the result must be considered an estimate.

B: The compound was detected either in the instrumental or sample preparation blank. The blanks usually contained low μg/L levels of several species, as indicated in one of the later subsections of this section.

BE: The analyte was detected in the sample at concentrations exceeding the usual calibration range, but low levels were also detected in the blank. In almost all cases, the levels observed in the sample considerably exceeded that of the blank.

In the discussion that follows the notation TIC (tentatively identified compound) means that the compound has been identified solely by its mass spectrum in a machine search of spectra contained in a computer's library. The quantitation is based upon the response factor of the nearest internal standard present in the gas chromatogram. Because of the nature of the quantitation procedure, all values reported are, by definition, estimates and bear the J qualifier.

The tables for the tank samples present data only for compounds detected above the reporting limits. Tables 3.1 and 3.2 list the target analytes, matrix, and reporting limits used in this work for different sample amounts. Exceptions to the usual sample amounts are described in Sect. 2. Table 3.3 shows the masses of the sludge samples. This is included because the masses available for the semivolatile organic compound analysis (SVOA) and the extract dilutions varied considerably, and their associated reporting limits varied accordingly.

Some reference is made to the total organic carbon (TOC) data, where such data aid in the interpretation of the organic compound analyses. The TOC results are discussed fully in Sect. 3.5. The surrogate standard and matrix spike (MAS) recoveries are not discussed in detail in this report because there are no established quality control (QC) limits for analysis of these sample matrices by modified U.S. Environmental Protection Agency (EPA) methods. Sect. 3.5 of this report does summarize the surrogate standard and MAS recoveries, and recovery problems encountered with specific samples are noted in Sect. 3.3.

3.3 RESULTS FOR WASTE TANK SAMPLES

The aqueous liquid samples for tanks T-3 and T-4 were prepared together in groups for the direct aqueous injection gas chromatographic analysis (DAI-GC), the SVOA, and the volatile organics analysis (VOA). The sludge samples for all tanks were prepared for the SVOA separately from the liquid samples.

3.3.1 Tank T-3

3.3.1.1 L-042 (aqueous liquid)

Very little organic matter was detected in the samples from this tank. The major organic compounds were determined by the DAI-GC method, and consisted of acetone (4 mg/L), methanol

Table 3.1. Reporting limits for volatile organic compounds in purge-and-trap GC-MS analysis (μg/L)

Compound	Aqueous samples 5 mL	Aqueous samples 5 μL	Organic liquids (diluted)[a]
Chloromethane	10	10,000	460,000
Bromomethane	10	10,000	460,000
Vinyl chloride	10	10,000	460,000
Chloroethane	10	10,000	460,000
Methylene chloride	5	5,000	230,000
Acetone	10	10,000	460,000
Carbon disulfide	5	5,000	230,000
1,1-Dichloroethene	5	5,000	230,000
1,1-Dichloroethane	5	5,000	230,000
1,2-Dichloroethene (total)	5	5,000	230,000
Chloroform	5	5,000	230,000
1,2-Dichloroethane	5	5,000	230,000
2-Butanone[b]	10	10,000	460,000
1,1,1-Trichloroethane	5	5,000	230,000
Carbon tetrachloride	5	5,000	230,000
Vinyl acetate	10	10,000	460,000
Bromodichloromethane	5	5,000	230,000
1,2-Dichloropropane	5	5,000	230,000
cis-1,3-Dichloropropene	5	5,000	230,000
Trichloroethene	5	5,000	230,000
Dibromochloromethane	5	5,000	230,000
1,1,2-Trichloroethane	5	5,000	230,000
Benzene	5	5,000	230,000
trans-1,3-Dichloropropene	5	5,000	230,000
Bromoform	5	5,000	230,000
4-Methyl-2-pentanone[c]	10	10,000	460,000
2-Hexanone	10	10,000	460,000
Tetrachloroethene	5	5,000	230,000
1,1,2,2-Tetrachloroethane	5	5,000	230,000
Toluene	5	5,000	230,000
Chlorobenzene	5	5,000	230,000
Ethylbenzene	5	5,000	230,000
Styrene	5	5,000	230,000
Xylenes (total)	5	5,000	230,000

[a]For organic liquids 0.2 mL of liquid was diluted to 9.1 mL, and 5 μL was taken for purge and trap in 5 mL of water. Reporting limits are 100-fold smaller where 0.5 mL was taken for purge and trap.
[b]Also named methylethylketone.
[c]Also named methylisobutylketone.

Table 3.2. Reporting limits for semivolatile organic compounds by extraction and GC-MS

Compound	Aqueous liquids (μg/L) 20 mL	Aqueous liquids (μg/L) 2 mL	Organic liquids (direct dilution, mg/kg)	Sludge[b] (mg/kg) 20 g	Sludge[b] (mg/kg) 5 g
Phenol	500	5,000	1,100	0.5	5
Bis(2-chloroethyl)ether	500	5,000	1,100	0.5	5
2-Chlorophenol	500	5,000	1,100	0.5	5
1,3-Dichlorobenzene	500	5,000	1,100	0.5	5
1,4-Dichlorobenzene	500	5,000	1,100	0.5	5
Benzyl alcohol	500	5,000	1,100	0.5	5
1,2-Dichlorobenzene	500	5,000	1,100	0.5	5
2-Methylphenol	500	5,000	1,100	0.5	5
N-nitroso-di-*n*-propylamine	500	5,000	1,100	0.5	5
Hexachloroethane	500	5,000	1,100	0.5	5
Nitrobenzene	500	5,000	1,100	0.5	5
Isophorone	500	5,000	1,100	0.5	5
2-Nitrophenol	500	5,000	1,100	0.5	5
2,4-Dimethylphenol	500	5,000	1,100	0.5	5
Benzoic acid	2,500	25,000	5,500	2.5	25
Bis(2-chloroethoxy)methane	500	5,000	1,100	0.5	5
2,4-Dichlorophenol	500	5,000	1,100	0.5	5
1,2,4-Trichlorobenzene	500	5,000	1,100	0.5	5
Naphthalene	500	5,000	1,100	0.5	5
4-Chloroaniline	500	5,000	1,100	0.5	5
Hexachlorobutadiene	500	5,000	1,100	0.5	5
4-Chloro-3-methylphenol	500	5,000	1,100	0.5	5
2-Methylnaphthalene	500	5,000	1,100	0.5	5
Hexachlorocyclopentadiene	500	5,000	1,100	0.5	5
2,4,6-Trichlorophenol	500	5,000	1,100	0.5	5
2,4,5-Trichlorophenol	2,500	25,000	500	2.5	25
2-Chloronaphthalene	500	5,000	1,100	0.5	5
2-Nitroaniline	2,500	25,000	5,500	2.5	25
Dimethylphthalate	500	5,000	1,100	0.5	5
Acenaphthylene	500	5,000	1,100	0.5	5
2,6-Dinitrotoluene	500	5,000	1,100	0.5	5
3-Nitroaniline	2,500	25,000	5,500	2.5	25
Acenaphthene	500	5,000	1,100	0.5	5
2,4-Dinitrophenol	2,500	25,000	5,500	2.5	25
4-Nitrophenol	2,500	25,000	5,500	2.5	25
Dibenzofuran	500	5,000	1,100	0.5	5
2,4-Dinitrotoluene	500	5,000	1,100	0.5	5
Diethylphthalate	500	5,000	1,100	0.5	5
4-Chlorophenyl-phenylether	500	5,000	1,100	0.5	5
Fluorene	500	5,000	1,100	0.5	5
4-Nitroaniline	2,500	25,000	5,500	2.5	25
4,6-Dinitro-2-methylphenol	2,500	25,000	5,500	2.5	25

Table 3.2 (continued)

Compound	Aqueous liquids (μg/L)		Organic liquids (direct dilution, mg/kg)	Sludge[b] (mg/kg)	
	20 mL	2 mL		20 g	5 g
N-nitrosodiphenylamine[c]	500	5,000	1,100	0.5	5
4-Bromophenyl-phenylether	500	5,000	1,100	0.5	5
Hexachlorobenzene	500	5,000	1,100	0.5	5
Pentachlorophenol	2,500	25,000	5,500	2.5	5
Phenanthrene	500	5,000	1,100	0.5	5
Anthracene	500	5,000	1,100	0.5	5
Di-*n*-butylphthalate	500	5,000	1,100	0.5	5
Fluoranthene	500	5,000	1,100	0.5	5
Pyrene	500	5,000	1,100	0.5	5
Butylbenzylphthalate	500	5,000	1,100	0.5	5
3,3′-Dichlorobenzidine	1,000	10,000	2,200	1	10
Benzo(a)anthracene	500	5,000	1,100	0.5	5
Chrysene	500	5,000	1,100	0.5	5
Bis(2-ethylhexyl)phthalate	500	5,000	1,100	0.5	5
Di-*n*-octylphthalate	500	5,000	1,100	0.5	5
Benzo(b)fluoranthene	500	5,000	1,100	0.5	5
Benzo(k)fluoranthene	500	5,000	1,100	0.5	5
Benzo(a)pyrene	500	5,000	1,100	0.5	5
Indeno(1,2,3-cd)pyrene	500	5,000	1,100	0.5	5
Dibenz(a,h)anthracene[d]	500	5,000	1,100	0.5	5
Benzo(g,h,i)perylene	500	5,000	1,100	0.5	5

[a]For example, 9.1 mg (10 μL) of organic liquid in 1.0 mL of methylene chloride.

[b]Calculated without sample dilution. For ten-fold dilution, reporting limit is ten-fold greater.

[c]Cannot be separated from diphenylamine.

[d]And/or dibenz(a,c)anthracene.

Table 3.3. Masses of sludge samples for semivolatile organic compound extraction

Sludge sample No.	Mass[a] (g)	Sludge sample No.	Mass[a] (g)
T-4/S-046	19.4	W-7/S-084	19.5
T-3/S-043	20.1	W-7/H-085	4.6
T-9/S-048	20.9	W-8/S-088	5.0
T-2/S-040	10.2	W-9/S-092	6.9
T-1/S-037	19.1	W-4/S-024	9.5[b]
W-11/H-005	21.0	W-4/H-026	9.1[b]
TH-4/S-058	19.5	W-10/S-096	6.4
TH-4/S-059	18.7	W-10/H-120	7.5
W-5/S-075	19.2	W-3/S-020	2.7[b]
W-6/S-086	22.6		

[a]Except for those noted, all SVOA extracts required ten-fold dilution before GC-MS. Reporting limits are increased ten-fold by dilution.

[b]These SVOA extracts did not require dilution.

(28 mg/L), and *n*-butyl alcohol (80 mg/L). As noted in the methods section (Sect. 2), these identifications cannot be considered absolute because the method does not include mass spectrometer (MS) confirmation. However, where the constituent also was identified in the VOA, there is more confidence in the DAI-GC result. Of the CLP Target Compound List (TCL) constituents, only acetone was detected at 180 μg/L by the purge-and-trap GC-MS VOA. The lower value for the VOA than for the DAI-GC is probably due to a low purging efficiency at high concentration levels.

Volatile TICs were insignificant. Only benzyl alcohol was estimated in the SVOA of the aqueous liquid at 100 μg/L. Among the TICs were dibromonitrophenol at 2000 μg/L and tributylphosphate (TBP) at 2000 μg/L. Both of these TICs were found in other samples as well. The presence of a nitrated phenol suggests nitration reactions in the tank. The brominated product was unexpected, but TBP has been used in large amounts at the laboratory, and its presence was expected.

The SVO phenol and 2-fluorophenol surrogate standards were not recovered. This may reflect the highly alkaline nature of the samples (the pH of T-3/L-042 was 12.7).

3.3.1.2 S-043 (soft sludge)

Two phthalates, diethyl- and di-*n*-butylphthalate, were determined at 2 and 3 mg/kg, respectively. It is not clear why neither dibromonitrophenol nor TBP were detected in the sludge SVOA TICs, unless the analysis was not sufficiently sensitive.

3.3.2 Tank T-4

3.3.2.1 L-044, L-045, and L-111 (aqueous liquids)

The three aqueous liquid samples for tank T-4 yielded very similar results, as shown in Table 3.4. The DAI-GC found concentrations exceeding 1000 μg/L each of acetone, ethyl alcohol, isopropyl alcohol, methanol, and *n*-butyl alcohol. The VOA confirmed the presence of acetone and also indicated chloroform. Other TCL volatile organics were present at very low levels.

Volatile TICs were low in concentration; the major one was heptanone (80 μg/L, found in L-044). No semivolatile TCL organic compounds were detected in the liquids. Major SVOA TICs were methylhexanone (800 μg/L in L-045) and TBP (3000 μg/L in L-111).

Table 3.4. Listing of organic analytical data hits for tank T-4

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	T-4/L-044	Acetone		7	mg/L
	T-4/L-044	Ethyl alcohol		37	mg/L
	T-4/L-044	I-propyl alcohol		3	mg/L
	T-4/L-044	Methyl alcohol		46	mg/L
	T-4/L-044	*n*-butyl alcohol		3	mg/L
	T-4/L-045	Acetone		8	mg/L
	T-4/L-045	Ethyl alcohol		37	mg/L
	T-4/L-045	I-propyl alcohol		3	mg/L
	T-4/L-045	Methyl alcohol		27	mg/L
	T-4/L-045	*n*-butyl alcohol		6	mg/L
	T-4/L-111	Acetone		7	mg/L
	T-4/L-111	Ethyl alcohol		37	mg/L
	T-4/L-111	I-propyl alcohol		3	mg/L
	T-4/L-111	Methyl alcohol		42	mg/L
	T-4/L-111	*n*-butyl alcohol		3	mg/L
VOA	T-4/L-044A	Acetone	BE	400	μg/L
	T-4/L-044A	Benzene		7	μg/L
	T-4/L-044A	Bromodichloromethane	B	7	μg/L
	T-4/L-044A	Chloroform	B	170	μg/L
	T-4/L-044A	Ethylbenzene		20	μg/L
	T-4/L-044A	Methylene chloride	B	12	μg/L
	T-4/L-044A	Toluene	B	78	μg/L
	T-4/L-044A	4-Methyl-2-pentanone		11	μg/L
	T-4/L-045A	Acetone	BE	220	μg/L
	T-4/L-045A	Benzene		10	μg/L
	T-4/L-045A	Chlorobenzene	J	4	μg/L
	T-4/L-045A	Chloroform	B	160	μg/L
	T-4/L-045A	Ethylbenzene		20	μg/L
	T-4/L-045A	Methylene chloride	B	14	μg/L
	T-4/L-045A	Toluene	B	98	μg/L
	T-4/L-045A	Trichloroethene	J	3	μg/L
	T-4/L-045A	Xylene (total)		170	μg/L
	T-4/L-045A	2-Hexanone		20	μg/L
	T-4/L-045A	4-Methyl-2-pentanone		10	μg/L
	T-4/L-111A	Acetone	B	72	μg/L
	T-4/L-111A	Benzene		7	μg/L
	T-4/L-111A	Chloroform	B	120	μg/L
	T-4/L-111A	Ethylbenzene		5	μg/L
	T-4/L-111A	Methylene chloride	B	12	μg/L
	T-4/L-111A	Toluene	B	60	μg/L
	T-4/L-111A	Xylene (total)		36	μg/L
	T-4/S-046	Di-*n*-butylphthalate	J	3400	μg/kg
	T-4/S-046	Diethylphthalate	J	2400	μg/kg

3.3.2.2 S-046 (soft sludge)

The sludge contained only the ubiquitous phthalates.

Two of the neutral and two of the acid surrogate standard recoveries were low for the sludge. Low recoveries of acidic surrogate standards from the alkaline sludges was often encountered, suggesting a need for pH adjustment currently unspecified in EPA method 3550. Studies are under way to improve the surrogate standard recoveries in such sample matrices.

The samples for tanks T-1, T-2, and T-9 were each prepared as a group. These are discussed below.

3.3.3 Tank T-1

3.3.3.1 L-035, and L-036 (aqueous liquids)

Very little organic matter was detected in the samples from this tank. The VOA revealed apparently high levels of methylene chloride in both the sample and the blank. These high levels can be attributed to contamination of the solid sorbent traps during storage. When the quantitation procedure was modified, permitting traps to be analyzed within 48 h of receipt, the level of methylene chloride in the blank was reduced to acceptable levels. In the SVOA extraction, the methylene chloride and aqueous layers were difficult to separate for samples L-035 and L-036. Sample L-036 required centrifugation to achieve separation. However, surrogate standard and MAS recoveries were good. There were no SVOA TCL hits in the aqueous liquids.

SVOA TICs included diphenyloxazole (300–400 μg/L), hexadecanoic acid (300 μg/L in one sample), and TBP (30–40 mg/L). TBP was often the major organic compound detected.

3.3.3.2 S-037 (soft sludge)

Of the SVOA TCL compounds in the sludge, only phthalates were detected. SVOA TICs in the sludge included TBP at 2 mg/kg.

3.3.4 Tank T-2

3.3.4.1 L-038, L-039, and L-112 (aqueous liquids)

Very little volatile organic matter was detected in the aqueous liquids. Methylene chloride probably was a contaminant, as for tank T-1. As shown in Table 3.5, however, the SVOA showed the presence of 2-nitrophenol, 2,4-dichlorophenol, and 2,4,5-trichlorophenol in the liquids. These compounds could represent chemical reactions occurring in the tanks.

Table 3.5. Listing of organic analytical data hits for tank T-2

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	T-2/L-038A	Acetone	B	20	μg/L
	T-2/L-038A	Benzene	J	3	μg/L
	T-2/L-038A	Chloroform	BE	340	μg/L
	T-2/L-038A	Methylene chloride	BE	1,000	μg/L
	T-2/L-038A	Toluene	B	12	μg/L
	T-2/L-039A	Acetone	B	22	μg/L
	T-2/L-039A	Chloroform	BE	370	μg/L
	T-2/L-039A	Methylene chloride	BE	820	μg/L
	T-2/L-039A	Toluene	B	7	μg/L
	T-2/L-112A	Acetone	B	17	μg/L
	T-2/L-112A	Chloroform	BE	310	μg/L
	T-2/L-112A	Methylene chloride	BE	860	μg/L
	T-2/L-112A	Toluene	B	8	μg/L
SVO	T-2/L-038	Bis(2-ethylhexyl)phthalate	J	200	μg/L
	T-2/L-038	2-Nitrophenol	J	170	μg/L
	T-2/L-038	2,4-Dichlorophenol	J	100	μg/L
	T-2/L-038	2,4,5-Trichlorophenol	J	99	μg/L
	T-2/L-039	Bis(2-ethylhexyl)phthalate	J	300	μg/L
	T-2/L-039	2-Nitrophenol	J	200	μg/L
	T-2/L-039	2,4-Dichlorophenol	J	140	μg/L
	T-2/L-039	2,4,5-Trichlorophenol	J	110	μg/L
	T-2/L-112	Bis(2-ethylhexyl)phthalate	J	280	μg/L
	T-2/L-112	Di-*n*-butylphthalate	J	24	μg/L
	T-2/L-112	2-Nitrophenol	J	180	μg/L
	T-2/L-112	2,4,5-Trichlorophenol	J	120	μg/L
	T-2/S-040	Bis(2-ethylhexyl)phthalate		24,000	μg/kg
	T-2/S-040	Di-*n*-butylphthalate	J	4600	μg/kg
	T-2/S-040	Diethylphthalate	J	2,800	μg/kg
	T-2/S-040	Naphthalene	J	2,300	μg/kg

SVOA TICs in the three liquids included TBP at 20 mg/L and tetramethylbutylphenoxyethoxyethanol at 800–1000 μg/L. Some foaming was observed in samples L-039 and L-112 upon acidification.

Samples L-038 and L-112, which were collected sequentially, are duplicates within the limits of field sampling. The agreement between the results for the two samples is quite good, especially considering the low levels determined.

3.3.4.2 S-040 (soft sludge)

The sludge contained the usual phthalates and also naphthalene. The latter could be a component of liquid scintillator solutions. TBP also was detected in the sludge, but the amount could not be estimated because the mass spectrum of the chromatographic peak indicated coeluting interferences.

3.3.5 Tank T-9

3.3.5.1 L-047 (aqueous liquid)

Tank T-9 also did not contain appreciable concentrations of either volatile or semivolatile organic compounds, except for 17 mg/L of methanol determined by DAI-GC. Sample L-047 contained 400 μg/L of benzoic acid and 200 μg/L of benzyl alcohol. TBP at 30 mg/L was the main TIC.

3.3.5.2 S-048 (soft sludge)

Only phthalates were determined as TCL constituents in the sludge; TBP was determined as a TIC at 2 mg/kg.

Samples from tanks TH-1, TH-3, and W-11 were each processed together. These are discussed below.

3.3.6 Tank TH-1

3.3.6.1 L-049 and L-050 (aqueous liquids)

The samples from this tank were not significantly different from the blank. The TOC was among the lowest determined. None of the SVOA extracts was submitted for GC-MS because the GC screening did not reveal any constituents different from the blank. A precipitate was formed when sodium hydroxide was added to sample L-050, and it was difficult to increase the pH above 10 even when concentrated sodium hydroxide solution was employed.

3.3.7 Tank TH-3

3.3.7.1 L-052, L-053 (aqueous liquids), and L-113 (blind water blank)

These samples were very similar to those drawn from tank TH-1. The lack of significant levels of organic compounds is reflected by the low TOC values, viz., 6.1, 5.1, and 0.8 mg/L for L-052, L-053, and L-113, respectively. A precipitate formed during the basic pH adjustment of L-053, and achieving pH >10 for the base/neutral extraction was difficult.

The absence of detectable compounds in L-113, a blank submitted blind as a regular sample, is a good check of the entire handling, preparation, and analytical procedures for contamination. The toluene VOA matrix spike was not recovered, but the perdeuterated toluene surrogate standard was recovered well.

3.3.8 Tank W-11

3.3.8.1 L-003, L-114, and L-115 (aqueous liquids)

As shown in Table 3.6, the volatile organic compounds in tank W-11 samples also were very low. The SVOA extracts of all but the sludge were not submitted for GC-MS analysis because the GC screen did not detect sufficient concentrations of chromatographable organic compounds. The screening data are consistent with the sample TOCs, being the lowest of all the tanks in this study (2.0, <0.1, and <0.1 mg/L for L-003, L-114, and L-115, respectively). Samples L-003 and L-115 were sequentially collected and are useful as duplicates, within the usual errors of sampling. The VOA results are in reasonable agreement, considering the very low concentrations (2–30 μg/L) determined.

3.3.8.2 H-005 (hard sludge)

The sludge contained several polycyclic aromatic hydrocarbons (PAHs) at concentrations of several tens to hundreds of mg/kg, as shown in Table 3.6. The presence of these species was quite unexpected, since PAH-bearing wastes, such as tars, were not known to be disposed of in the tank. The PAHs were not detected in the liquid samples, as expected from their low aqueous solubilities (low μg/L for the five-ring PAH), and they selectively partitioned into the sludge. This trend was observed for other sludge samples. Phthalates were also present.

3.3.9 Tank TH-4

3.3.9.1 L-055, L-056, L-057, L-116, L-117 (all aqueous liquids)

The aqueous samples from tank TH-4 were processed together. There was an insufficient amount of L-117 for the SVOA extraction, so this sample received only the DAI-GC and VOA characterization. Results for the tank are listed in Table 3.7.

The DAI-GC analysis indicated that the aqueous liquids contained methanol, acetone, 2-butanone, and 4-methyl-2-pentanone at high mg/L concentrations. The VOA confirmed the presence of acetone, 4-methyl-2-butanone and benzene; did not confirm the presence of 4-methyl-2-pentanone, and could not

Table 3.6. Listing of organic analytical data hits for tank W-11

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	W-11/L-003	Acetone	B	27	μg/L
	W-11/L-003	Chlorobenzene	J	5	μg/L
	W-11/L-003	Methylene chloride	B	7	μg/L
	W-11/L-003	Toluene	B	23	μg/L
	W-11/L-003	Trichloroethene	J	4	μg/L
	W-11/L-114	Acetone	B	21	μg/L
	W-11/L-114	Chlorobenzene	J	3	μg/L
	W-11/L-114	Methylene chloride	B	8	μg/L
	W-11/L-114	Toluene	B	24	μg/L
	W-11/L-115	Acetone	B	15	μg/L
	W-11/L-115	Chlorobenzene	J	2	μg/L
	W-11/L-115	Methylene chloride	B	13	μg/L
	W-11/L-115	Toluene	B	13	μg/L
SVO	W-11/H-005	Benzo(a)anthracene		44,000	μg/kg
	W-11/H-005	Benzo(a)pyrene		64,000	μg/kg
	W-11/H-005	Benzo(b)fluoranthene	E	160,000	μg/kg
	W-11/H-005	Benzo(g,h,i)perylene		22,000	μg/kg
	W-11/H-005	Bis(2-ethylhexyl)phthalate		38,000	μg/kg
	W-11/H-005	Chrysene		130,000	μg/kg
	W-11/H-005	Di-*n*-butylphthalate		17,400	μg/kg
	W-11/H-005	Fluoranthene	E	240,000	μg/kg
	W-11/H-005	Indeno(1,2,3-cd)pyrene		42,000	μg/kg
	W-11/H-005	Phenanthrene		24,000	μg/kg
	W-11/H-005	Pyrene		90,000	μg/kg

Table 3.7. Listing of organic analytical data hits for tank TH-4

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	TH-4/L-055	Methyl alcohol		13	mg/L
	TH-4/L-056	Acetone		22	mg/L
	TH-4/L-056	Methyl alcohol		27	mg/L
	TH-4/L-056	2-Butanone		2	mg/L
	TH-4/L-056	4-Methyl-2-pentanone		15	mg/L
	TH-4/L-056	*n*-Butyl alcohol		20	mg/L
	TH-4/L-057	Methyl alcohol		12	mg/L
	TH-4/L-117	Methyl alcohol		20	mg/L
VOA	TH-4/L-055A	Acetone	B	22	μg/L
	TH-4/L-055A	Benzene		3	μg/L
	TH-4/L-055A	Chloroform		6	μg/L
	TH-4/L-055A	Methylene chloride	B	8	μg/L
	TH-4/L-055A	Toluene	B	4	μg/L
	TH-4/L-056A	Acetone	BE	630	μg/L
	TH-4/L-056A	Benzene		149	μg/L
	TH-4/L-056A	Carbon tetrachloride		11	μg/L
	TH-4/L-056A	Chloroform		11	μg/L
	TH-4/L-056A	Methylene chloride	B	8	μg/L
	TH-4/L-056A	Toluene	B	18	μg/L
	TH-4/L-056A	2-Butanone		85	μg/L
	TH-4/L-056A	4-Methyl-2-pentanone	BE	2682	μg/L
	TH-4/L-057A	Acetone	B	71	μg/L
	TH-4/L-057A	Benzene		115	μg/L
	TH-4/L-057A	Chloroform		7	μg/L
	TH-4/L-057A	Methylene chloride	B	7	μg/L
	TH-4/L-057A	Toluene	B	21	μg/L
	TH-4/L-057A	Xylene (total)		10	μg/L
	TH-4/L-057A	4-Methyl-2-pentanone	BE	397	μg/L
	TH-4/L-057B	Acetone	B	54	μg/L
	TH-4/L-057B	Benzene		118	μg/L
	TH-4/L-057B	Chloroform		5	μg/L
	TH-4/L-057B	Methylene chloride	B	6	μg/L
	TH-4/L-057B	Toluene	B	23	μg/L
	TH-4/L-057B	Xylene (total)		22	μg/L
	TH-4/L-057B	4-Methyl-2-pentanone	B	179	μg/L
	TH-4/L-116A	Acetone	B	150	μg/L
	TH-4/L-116A	Benzene		10	μg/L
	TH-4/L-116A	Chlorobenzene		8	μg/L
	TH-4/L-116A	Methylene chloride	B	6	μg/L
	TH-4/L-116A	Toluene	B	13	μg/L
	TH-4/L-116A	Trichloroethene		5	μg/L
	TH-4/L-116A	4-Methyl-2-pentanone	B	298	μg/L
	TH-4/L-117A	Acetone	B	16	μg/L

Table 3.7 (continued)

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	TH-4/L-117A	Benzene		21	μg/LT
	TH-4/L-117A	Chloroform		4	μg/L
	TH-4/L-117A	Methylene chloride	B	210	μg/L
	TH-4/L-117A	Toluene	B	79	μg/L
	TH-4/L-117A	4-Methyl-2-pentanone	B	9	μg/L
SVO	TH-4/L-056	Acenaphthene	J	42	μg/L
	TH-4/L-056	Naphthalene	J	110	μg/L
	TH-4/L-056	Phenanthrene	J	56	μg/L
	TH-4/S-058	Acenaphthene	J	4600	μg/kg
	TH-4/S-058	Anthracene		26000	μg/kg
	TH-4/S-058	Benzo(a)anthracene		6300	μg/kg
	TH-4/S-058	Benzo(a)pyrene	J	4300	μg/kg
	TH-4/S-058	Benzo(b)fluoranthene		5600	μg/kg
	TH-4/S-058	Benzo(g,h,i)perylene	J	3400	μg/kg
	TH-4/S-058	Bis(2-ethylhexyl)phthalate		20000	μg/kg
	TH-4/S-058	Chrysene		6800	μg/kg
	TH-4/S-058	Di-*n*-butylphthalate		17000	μg/kg
	TH-4/S-058	Fluoranthene		23000	μg/kg
	TH-4/S-058	Fluorene	J	2800	μg/kg
	TH-4/S-058	Indeno(1,2,3-cdpyrene	J	3600	μg/kg
	TH-4/S-058	Naphthalene		8400	μg/kg
	TH-4/S-058	Phenanthrene		26000	μg/kg
	TH-4/S-058	Pyrene		18000	μg/kg
	TH-4/S-059	Acenaphthene		6700	μg/kg
	TH-4/S-059	Benzo(a)anthracene		5800	μg/kg
	TH-4/S-059	Benzo(b)fluoranthene		8100	μg/kg
	TH-4/S-059	Bis(2-ethylhexyl)phthalate		26000	μg/kg
	TH-4/S-059	Chrysene		6200	μg/kg
	TH-4/S-059	Di-*n*-butylphthalate		13000	μg/kg
	TH-4/S-059	Dibenzofuran	J	4100	μg/kg
	TH-4/S-059	Fluoranthene		20000	μg/kg
	TH-4/S-059	Fluorene	J	3700	μg/kg
	TH-4/S-059	Naphthalene		17000	μg/kg
	TH-4/S-059	Phenante		3000	μg/kg
	TH-4/S-059	Pyrene		21000	μg/kg

detect methanol. The lower quantitative results for acetone and 4-methyl-2-pentanone probably result from the reduced purging efficiency of these species when present at mg/L levels.

Among the volatile TICs was di-isopropylether at 20, 1000, 1000, and 50 μg/L for L-055, L-056, L-057, and L-117, respectively.

Except for three two-ring and three-ring PAHs in L-056, no semivolatile TCL organics were determined in the aqueous liquid samples. The major TIC was TBP, at 20 and 30 mg/L in L-056 and L-057, respectively.

3.3.9.2 S-058 and S-059 (soft sludges)

PAHs were found at milligram-per-kilogram concentrations in the sludges. TBP also was found as a TIC at 2 mg/kg in both sludges.

The sludges were hard to dry, even with an approximately fourfold-mass excess of sodium sulfate. However, the surrogate standard recoveries were good, exception for nitrobenzene, which was usually recovered poorly, possibly because of evaporative loses during solvent concentration by nitrogen blow-down.

Except for the sludges, the samples for tanks W-5, W-6, and W-7 were each processed as a group. These are discussed below.

3.3.10 Tank W-5

3.3.10.1 L-073 (aqueous liquid)

The major constituents observed in the VOA were tri- and tetrachloroethenes at 138 and 267 μg/L concentrations, respectively. Other target analytes were observed, but at much lower levels. The SVOA did not detect any significant material in the liquids. The aqueous liquid sample foamed upon acidification during the preparation for the SVOA.

3.3.10.2 S-075 (soft sludge)

TBP was found as a TIC in the sludge at 8 mg/kg. The SVOA suggested the presence of phthalates.

3.3.11 Tank W-6

3.3.11.1 L-077, L-078, L-079 (aqueous liquids)

The results for this tank are shown in Table 3.8. The principal volatile organics were tri- and tetrachloroethenes. The SVOA of the aqueous liquids showed only benzoic acid at 300 ug/L in L-079. Sample L-078 turned green upon the addition of the SVOA MAS, but the surrogate recoveries were acceptable.

Table 3.8. Listing of organic analytical data hits for tank W-6

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-6/L-079	*I*-propyl alcohol		3	mg/L
VOA	W-6/L-077A	Acetone	B	67	μg/L
	W-6/L-077A	Carbon tetrachloride	JB	4	μg/L
	W-6/L-077A	Chloroform	J	5	μg/L
	W-6/L-077A	Methylene chloride	B	9	μg/L
	W-6/L-077A	Tetrachloroethene	B	47	μg/L
	W-6/L-077A	Toluene	B	6	μg/L
	W-6/L-077A	Trichloroethene		13	μg/L
	W-6/L-078A	Acetone	B	44	μg/L
	W-6/L-078A	Benzene	J	2	μg/L
	W-6/L-078A	Chloroform		9	μg/L
	W-6/L-078A	Methylene chloride	B	8	μg/L
	W-6/L-078A	Tetrachloroethene	B	158	μg/L
	W-6/L-078A	Toluene	B	10	μg/L
	W-6/L-078A	Trichloroethene		24	μg/L
	W-6/L-078A	4-Methyl-2-pentanone	J	9	μg/L
	W-6/L-079A	Acetone	B	111	μg/L
	W-6/L-079A	Benzene		10	μg/L
	W-6/L-079A	Chloroform	J	3	μg/L
	W-6/L-079A	Methylene chloride	B	7	μg/L
	W-6/L-079A	Tetrachloroethene	BE	507	μg/L
	W-6/L-079A	Toluene	B	10	μg/L
	W-6/L-079A	Trichloroethene		82	μg/L
	W-6/L-079A	2-Butanone		75	μg/L
	W-6/L-079A	2-Hexanone		9	μg/L
	W-6/L-079A	4-Methyl-2-pentanone		91	μg/L
SVO	W-6/L-079	Benzoic acid	J	290	μg/L
	W-6/S-080	Bis(2-ethylhexyl)phthalate		31,000	μg/kg
	W-6/S-080	Di-*n*-butylphthalate	J	210	μg/kg
	W-6/S-080	Diethylphthalate	J	150	μg/kg
	W-6/S-080	Fluoranthene	J	340	μg/kg
	W-6/S-080	Phenanthrene	J	680	μg/kg
	W-6/S-080	Pyrene	J	350	μg/kg

3.3.11.2 S-080 (soft sludge)

A few PAHs and phthalates were determined in the sludge.

3.3.12 Tank W-7

3.3.12.1 L-082 (aqueous liquid)

The measurements for tank W-7 are presented in Table 3.9. The major organic constituents were 14 mg/L of methanol and ~ 1000 μg/L of carbon tetrachloride in L-082. The data for the sample identification numbers with A and B suffixes are for duplicate determinations. These data show very good agreement, considering the low concentrations and two purging steps in the analysis. Benzoic acid (2000 μg/L) was the only TCL compound detected. Dibromonitrophenol was estimated at 700 μg/L as a TIC.

3.3.12.2 S-084 (soft sludge) and H-085 (hard sludge)

Fluoranthene and several phthalates were determined in the sludges. Four of the five SVOA surrogate standard recoveries (particularly for the acid surrogate standards) from the soft sludge, S-084, were very low, suggesting problems with the extraction step. Surrogate standard recoveries were somewhat better for the hard sludge, H-085. This difference in recovery for the surrogates between soft and hard sludges was observed with other samples.

The samples for tanks W-8 and W-9, except for the sludges, were each prepared together. These are discussed below.

3.3.13 Tank W-8: S-088 (Soft Sludge); L-086 and L-087 (Aqueous Liquids)

Table 3.10 lists the findings for this tank. The concentrations of both volatile and semivolatile organic compounds were very low.

The main SVOA TICs were dibromocyclohexane (300 μg/L in L-086 alone), dibromonitrophenol (500 and 700 μg/L in L-086 and L-087, respectively), and TBP (20 mg/L in each liquid). The results suggest bromination reactions may have occurred.

Only the sludge was found to contain significant detectable organic compounds, which were mostly PAHs. The TICs were mostly unidentified hydrocarbons and TBP (700 mg/kg).

Table 3.9. Listing of organic analytical data hits for tank W-7

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-7/L-082	Methyl alcohol		14	mg/L
VOA	W-7/L-082A	Acetone	B	26	μg/L
	W-7/L-082A	Carbon tetrachloride	BE	1,088	μg/L
	W-7/L-082A	Chloroform	J	3	μg/L
	W-7/L-082A	Methylene chloride	B	7	μg/L
	W-7/L-082A	Tetrachloroethene	B	24	μg/L
	W-7/L-082A	Toluene	B	14	μg/L
	W-7/L-082A	4-Methyl-2-pentanone		7	μg/L
	W-7/L-082B	Acetone	B	31	μg/L
	W-7/L-082B	Carbon tetrachloride	BE	1,021	μg/L
	W-7/L-082B	Chloroform	J	3	μg/L
	W-7/L-082B	Methylene chloride	B	4	μg/L
	W-7/L-082B	Tetrachloroethene	B	39	μg/L
	W-7/L-082B	Toluene	B	9	μg/L
	W-7/L-082B	4-Methyl-2-pentanone		7	μg/L
SVO	W-7/H-085	Di-*n*-Butylphthalate	J	2,200	μg/kg
	W-7/H-085	Diethylphthalate	J	510	μg/kg
	W-7/L-082	Benzoic acid	J	1,900	μg/L
	W-7/S-084	Bis(2-ethylhexyl)phthalate		57,000	μg/kg
	W-7/S-084	Di-*n*-Butylphthalate	J	300	μg/kg
	W-7/S-084	Diethylphthalate	J	200	μg/kg
	W-7/S-084	Fluoranthene	J	260	μg/kg

Table 3.10. Listing of organic analytical data hits for tank W-8

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-8/L-086	Acetone		1	mg/L
	W-8/L-087	Acetone		3	mg/L
	W-8/L-087	Methyl alcohol		1	mg/L
	W-8/L-087	2-Butanone		1	mg/L
	W-8/L-087	*n*-Butyl alcohol		2	mg/L
VOA	W-8/L-086A	Acetone	B	64	μg/L
	W-8/L-086A	Benzene	J	3	μg/L
	W-8/L-086A	Methylene chloride	B	12	μg/L
	W-8/L-086A	Toluene	BJ	3	μg/L
	W-8/L-086A	2-Hexanone		41	μg/L
	W-8/L-086A	4-Methyl-2-pentanone		15	μg/L
	W-8/L-087A	Acetone	B	45	μg/L
	W-8/L-087A	Benzene		6	μg/L
	W-8/L-087A	Methylene chloride	B	7	μg/L
	W-8/L-087A	Toluene	BJ	4	μg/L
SVO	W-8/L-087	Di-*n*-butylphthalate	J	17	μg/L
	W-8/L-087	Naphthalene	J	28	μg/L
	W-8/S-088	Di-*n*-butylphthalate	J	630	μg/kg
	W-8/S-088	Diethylphthalate	J	430	μg/kg
	W-8/S-088	Fluoranthene	J	740	μg/kg
	W-8/S-088	Naphthalene	J	2,600	μg/kg
	W-8/S-088	Phenanthrene	J	1,300	μg/kg
	W-8/S-088	Pyrene	J	850	μg/kg
	W-8/S-088	2-Methylnaphthalene	J	1,300	μg/kg

The acidic surrogate standard compounds were very poorly recovered from the aqueous liquids, suggesting that acidic compounds, if present in the samples, may not have been extracted or detected. The two chlorinated phenolic MAS compounds also were poorly recovered.

3.3.14 Tank W-9: L-090 (Aqueous Liquid) and S-092 (Soft Sludge)

The results for this tank were similar to those for W-8. The VOA and SVOA did not reveal any significant levels of TCL constituents, except for the low mg/kg levels of PAHs and phthalates in the sludge, as shown in Table 3.11. The SVOA TIC, TBP, was estimated at 10 mg/L in L-090. The recoveries of the surrogate standard compounds in L-090 all were zero, suggesting either that an error had been made in surrogate standard spiking or that the matrix seriously interfered with the extraction.

Table 3.11. Listing of organic analytical data hits for tank W-9

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	W-9/L-090A	Acetone	B	70	μg/L
	W-9/L-090A	Benzene	J	2	μg/L
	W-9/L-090A	Chlorobenzene		5	μg/L
	W-9/L-090A	Chloroform		44	μg/L
	W-9/L-090A	Methylene chloride	B	13	μg/L
	W-9/L-090A	Tetrachloroethene	J	4	μg/L
	W-9/L-090A	Toluene	B	9	μg/L
	W-9/L-090A	Trichloroethene		5	μg/L
	W-9/L-090A	4-Methyl-2-pentanone		11	μg/L
SVO	W-9/L-090	Di-*n*-butylphthalate	J	20	μg/L
	W-9/S-092	Bis(2-ethylhexyl)phthalate		160,000	μg/kg
	W-9/S-092	Di-*n*-butylphthalate	J	760	μg/kg
	W-9/S-092	Diethylphthalate	J	450	μg/kg
	W-9/S-092	Fluoranthene	J	1,100	μg/kg
	W-9/S-092	Naphthalene	J	370	μg/kg
	W-9/S-092	Phenanthrene	J	1,300	μg/kg
	W-9/S-092	Pyrene	J	850	μg/kg

Except for the sludges, the samples for tanks W-1, W-2, and W-4 were prepared together. These are discussed below.

3.3.15 Tank W-1: L-007 and L-008 (Aqueous Liquids)

No TCL compounds were detected at significant concentrations in the samples from this tank. This is consistent with the very low TOC, 6.4 and 10.1 mg/L for L-007 and L-008, respectively. Two aliquots from L-007 were analyzed for VOA. The VOA surrogate standard recoveries were very poor for the A aliquot (10–19%) but were good for the B aliquot. The latter should be considered the more accurate analysis.

3.3.16 Tank W-2: L-011 and L-118 (Aqueous Liquids)

Neither sample contained significant levels of TCL constituents. This again is consistent with the low TOC, 18.9 and 22.6 mg/L, for L-011 and L-118. These two samples were collected in series and are useful as field duplicates, within normal sampling errors.

3.3.17 Tank W-4

3.3.17.1 L-022, L-023, L-119 (aqueous liquids)

As shown in Table 3.12, some of the samples from this tank contained elevated levels of organic compounds. Sample L-023 contained mg/L quantities of 4-methyl-2-pentanone (7 mg/L by DAI-GC, 1.1 mg/L by VOA). The VOA was not effective for determining mg/L concentrations of volatile organics, and the DAI-GC result probably is more accurate. Sample L-023 also contained 200 μg/L of naphthalene. These data are consistent with the elevated TOC in L-023 (558.5 mg/L) compared to the other liquids (49.5 and 59.9 mg/L for L-022 and L-119, respectively).

3.3.17.2 S-024 (soft sludge) and H-026 (hard sludge)

The two sludges contained PAHs and phthalates at levels of hundreds of mg/kg. Their SVOA extracts had to be filtered twice because of crystal precipitation. The surrogate standard recoveries of nitrobenzene, 2-fluorophenol, and fluorobiphenyl were low for the soft sludge, but better for the hard sludge. The SVOA extraction for S-024 was repeated, using a pH adjustment to obtain base/neutral and acid fractions. Two organic layers separated when the extracts were concentrated to 1 mL. Also, a lump of dark tar was found in the S-024 sample and could be a source of the PAHs which were detected previously.

3.3.18 Tank W-10

3.3.18.1 L-093, L-094, L-095 (aqueous liquids)

All the aqueous samples for tank W-10 were processed as a group. The analytical measurements for the tank are listed in Table 3.13. The DAI-GC indicated the presence of 30 to 40 mg/L of methanol. The acetone determined by DAI-GC was qualitatively confirmed by the VOA, but the latter indicated much lower levels to be present. The presence of 2-butanone was not verified by the VOA. Except for the

Table 3.12. Listing of organic analytical data hits for tank W-4

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-4/L-023	4-Methyl-2-pentanone		7	mg/L
VOA	W-4/L-022A	Methylene chloride	B	10	μg/L
	W-4/L-022A	Toluene	B	13	μg/L
	W-4/L-023A	Acetone		55	μg/L
	W-4/L-023A	Benzene	J	3	μg/L
	W-4/L-023A	Methylene chloride	B	10	μg/L
	W-4/L-023A	Toluene	B	14	μg/L
	W-4/L-023A	Trichloroethene		49	μg/L
	W-4/L-023A	4-Methyl-2-pentanone		1,102	μg/L
	W-4/L-119A	Benzene	J	2	μg/L
	W-4/L-119A	Methylene chloride	B	10	μg/L
	W-4/L-119A	Toluene	B	46	μg/L
	W-4/L-119A	Trichloroethene		14	μg/L
	W-4/L-119A	4-Methyl-2-pentanone	J	3	μg/L
SVO	W-4/H-026	Bis(2-ethylhexyl)phthalate		5,700	μg/kg
	W-4/H-026	Di-*n*-butylphthalate		1,400	μg/kg
	W-4/H-026	Diethylphthalate	J	49	μg/kg
	W-4/H-026	Fluoranthene	J	160	μg/kg
	W-4/H-026	Phenanthrene	J	110	μg/kg
	W-4/H-026	Pyrene	J	130	μg/kg
	W-4/L-022	Di-*n*-butylphthalate	J	11	μg/L
	W-4/L-023	Naphthalene	J	160	μg/L
	W-4/L-119	Naphthalene	J	35	μg/L
	W-4/S-024	Bis(2-ethylhexyl)phthalate		11,000	μg/kg
	W-4/S-024	Chrysene	J	69	μg/kg
	W-4/S-024	Di-*n*-butylphthalate		2,100	μg/kg
	W-4/S-024	Di-*n*-octylphthalate	J	51	μg/kg
	W-4/S-024	Diethylphthalate	J	33	μg/kg
	W-4/S-024	Fluoranthene	J	220	μg/kg
	W-4/S-024	Phenanthrene	J	170	μg/kg
	W-4/S-024	Pyrene	J	170	μg/kg

Table 3.13. Listing of organic analytical data hits for tank W-10

Analysis	Sample ID	Compound	Qualifier	Results	Units
DAI-GC	W-10/L-093	Methyl alcohol		27	mg/L
	W-10/L-094	Methyl alcohol		37	mg/L
	W-10/L-095	Acetone		3	mg/L
	W-10/L-095	Ethyl alcohol		1	mg/L
	W-10/L-095	Methyl alcohol		41	mg/L
	W-10/L-095	2-Butanone		1	mg/L
	W-10/L-095	*n*-Butyl alcohol		1	mg/L
VOA	W-10/L-094A	Benzene	B	6	μg/L
	W-10/L-094A	Methylene chloride	B	13	μg/L
	W-10/L-094A	Tetrachloroethene		21	μg/L
	W-10/L-094A	Toluene		12	μg/L
	W-10/L-094A	Trichloroethene		27	μg/L
	W-10/L-094A	1,2-Dichloroethene (total)		11	μg/L
	W-10/L-094B	Benzene	BM	38	μg/L
	W-10/L-094B	Methylene chloride	B	19	μg/L
	W-10/L-095A	Acetone	B	97	μg/L
	W-10/L-095A	Benzene	B	22	μg/L
	W-10/L-095A	Chlorobenzene	B	5	μg/L
	W-10/L-095A	Chloroform		16	μg/L
	W-10/L-095A	Methylene chloride	B	12	μg/L
	W-10/L-095A	Tetrachloroethene		48	μg/L
	W-10/L-095A	Toluene	B	26	μg/L
	W-10/L-095A	Trichloroethene		81	μg/L
	W-10/L-095A	1,2-Dichloroethene (total)		31	μg/L
	W-10/L-095A	4-Methyl-2-pentanone	B	23	μg/L
SVO	W-10/H-120	Benzo(*a*)pyrene	J	1,900	μg/kg
	W-10/H-120	Benzo(*b*)fluoranthene	J	2,200	μg/kg
	W-10/H-120	Benzo(*g,h,i*)perylene	J	870	μg/kg
	W-10/H-120	Di-*n*-butylphthalate	J	770	μg/kg
	W-10/H-120	Fluoranthene	J	8,000	μg/kg
	W-10/H-120	Naphthalene	J	270	μg/kg
	W-10/H-120	Phenanthrene	J	5,100	μg/kg
	W-10/H-120	Pyrene	J	8,100	μg/kg
	W-10/L-094	Benzoic acid	J	400	μg/L
	W-10/L-095	Benzoic acid		2,900	μg/L
	W-10/L-095	Naphthalene	J	20	μg/L
	W-10/S-096	Benzo(*a*)anthracene	J	660	μg/kg
	W-10/S-096	Bis(2-ethylhexyl)phthalate		130,000	μg/kg
	W-10/S-096	Chrysene	J	760	μg/kg
	W-10/S-096	Di-*n*-Butylphthalate	J	2,600	μg/kg
	W-10/S-096	Fluoranthene	J	1,800	μg/kg
	W-10/S-096	Phenanthrene	J	1700	μg/kg
	W-10/S-096	Pyrene	J	1400	μg/kg

methanol, there were only very low levels of volatile organic compounds in the aqueous liquids, and in L-093, none were detected at all. This is consistent with the much lower TOC in L-093 (8.7 mg/L) versus that in L-094 and L-095 (86.4 and 54.6 mg/L, respectively).

The only TCL semivolatile organic compounds determined in the liquids were benzoic acid in L-094 and L-095 (400 and 2900 μg/L, respectively). The naphthalene determined in L-095 was very low (20 μg/L). TBP was estimated as a TIC only in L-094 at 2 mg/L.

3.3.18.2 S-096 (soft sludge) and H-120 (hard sludge)

As with the other tanks, these sludges contained PAHs and phthalates, with H-120 having higher concentrations of PAHs than S-096, which is in line with their TOCs (14,600 and 8180 mg/kg, respectively). The SVOA TIC, ethylphenylethanone, was estimated at 200 and 2300 μg/L in L-094 and L-095, respectively, and 9 and 40 mg/kg in S-096 and H-120, respectively. Tributylphospahte was detected as a TIC in the sludges at 60 and 300 mg/kg in S-096 and H-120, respectively. The hard sludges appear to contain generally higher concentrations of organic compounds than do the soft sludges.

The acidic MAS recoveries for L-093 were very poor; however, the acidic surrogate standard recoveries were good. The reason for this discrepancy is not clear. Two of the acidic compound and two of the neutral compound surrogate standard recoveries were low for S-096, but were better for the hard sludge, H-120. The hard sludges exhibited better surrogate standard and MAS recoveries than did the soft sludges. The reasons for this behavior are not clear, but may be related to the greater aqueous character of the soft sludges.

3.3.19 Tank 7860A

3.3.19.1 L-063, L-064, L-065, L-066, and L-067org (all organic liquids)

The samples from tank 7860A, which were prepared as a group, consisted of four organic liquids, an organic liquid overlaying an aqueous liquid, and two aqueous liquids. The organic liquids were not subjected to the DAI-GC. The radioactivity of the organic and aqueous layers also was low enough that these liquids could be diluted in methanol (organic layers) or water (aqueous layers) for purge-and-trap GC-MS, and in methylene chloride for the SVOA GC-MS. Samples of the organic layers diluted in hexane also were subjected to GC-ECD analysis for PCBs. The results of the analyses are presented in Table 3.14.

The organic layer appears to be a hydrocarbon liquid. PCBs were not detected with a reporting limit of 10 mg/kg, and in the SVOA, naphthalene and 2-methylnaphthalene averaged 88 and 77 mg/kg. Phthalates also were determined. The SVOA TICs consisted mostly of unidentifiable hydrocarbons which totaled 15

Table 3.14. Listing of organic analytical data hits for tank 7860A

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	7860A/L-067AQ	Acetone		1,500	mg/L
	7860A/L-067AQ	Ethyl alcohol		2,600	mg/L
	7860A/L-067AQ	*I*-propyl alcohol		770	mg/L
	7860A/L-067AQ	Methyl alcohol		1,400	mg/L
	7860A/L-067AQ	2-Butanone		1,400	mg/L
	7860A/L-067AQ	*n*-Butyl alcohol		1,000	mg/L
	7860A/L-068	Acetone		2,300	mg/L
	7860A/L-068	Ethyl alcohol		4,300	mg/L
	7860A/L-068	*I*-propyl alcohol		940	mg/L
	7860A/L-068	Methyl alcohol		1,700	mg/L
	7860A/L-068	2-Butanone		2,000	mg/L
	7860A/L-068	*n*-Butyl alcohol		1,200	mg/L
	7860A/L-069	Acetone		1,900	mg/L
	7860A/L-069	Ethyl alcohol		3,400	mg/L
	7860A/L-069	*I*-propyl alcohol		860	mg/L
	7860A/L-069	Methyl alcohol		1,600	mg/L
	7860A/L-069	2-Butanone		1,600	mg/L
	7860A/L-069	*n*-Butyl alcohol		1,000	mg/L
VOA	7860A/L-063	Acetone	B	2,200,000	μg/L
	7860A/L-063	Ethylbenzene		440,000	μg/L
	7860A/L-063	Methylene chloride	JB	61,000	μg/L
	7860A/L-063	Tetrachloroethene	E	39,000,000	μg/L
	7860A/L-063	Toluene	J	85,000	μg/L
	7860A/L-063	Trichloroethene	E	39,000,000	μg/L
	7860A/L-063	Xylene (total)	E	9,800,000	μg/L
	7860A/L-063	2-Butanone		4,100,000	μg/L
	7860A/L-064	Acetone	B	1,100,000	μg/L
	7860A/L-064	Ethylbenzene		420,000	μg/L
	7860A/L-064	Methylene chloride	JB	97,000	μg/L
	7860A/L-064	Tetrachloroethene	E	35,000,000	μg/L
	7860A/L-064	Toluene	J	81,000	μg/L
	7860A/L-064	Trichloroethene	E	36,000,000	μg/L
	7860A/L-064	Xylene (total)		8,900,000	μg/L
	7860A/L-064	2-Butanone		1,400,000	μg/L
	7860A/L-065	Acetone	B	1,000,000	μg/L
	7860A/L-065	Methylene chloride	JB	64,000	μg/L
	7860A/L-065	Tetrachloroethene	E	36,000,000	μg/L
	7860A/L-065	Toluene	J	90,000	μg/L
	7860A/L-065	Trichloroethene	E	38,000,000	μg/L
	7860A/L-065	Xylene (total)	E	9,300,000	μg/L
	7860A/L-065	2-Butanone		1,300,000	μg/L
	7860A/L-066	Acetone	B	1,000,000	μg/L
	7860A/L-066	Ethylbenzene		450,000	μg/L
	7860A/L-066	Methylene chloride	JB	170,000	μg/L

Table 3.14 (continued)

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	7860A/L-066	Tetrachloroethene	E	40,000,000	µg/L
	7860A/L-066	Trichloroethene	E	39,000,000	µg/L
	7860A/L-066	Xylene (total)	E	11,000,000	µg/L
	7860A/L-066	2-Butanone		1,300,000	µg/L
	7860A/L-067AQ	Acetone	BE	1,300,000	µg/L
	7860A/L-067AQ	Methylene chloride	JB	1,600	µg/L
	7860A/L-067AQ	Tetrachloroethene		19,000	µg/L
	7860A/L-067AQ	Trichloroethene		65,000	µg/L
	7860A/L-067AQ	Vinyl acetate	J	2,300	µg/L
	7860A/L-067AQ	Xylene (total)		14,000	µg/L
	7860A/L-067AQ	2-Butanone	E	1,800,000	µg/L
	7860A/L-067AQ	4-Methyl-2-pentanone	J	3,100	µg/L
	7860A/L-067OR	Acetone	B	1,000,000	µg/L
	7860A/L-067OR	Ethylbenzene		430,000	µg/L
	7860A/L-067OR	Methylene chloride	JB	81,000	µg/L
	7860A/L-067OR	Tetrachloroethene	E	35,000,000	µg/L
	7860A/L-067OR	Toluene	J	94,000	µg/L
	7860A/L-067OR	Trichloroethene	E	36,000,000	µg/L
	7860A/L-067OR	Xylene (total)	E	9,500,000	µg/L
	7860A/L-067OR	2-Butanone		1,700,000	µg/L
	7860A/L-068	Acetone	BE	1,300,000	µg/L
	7860A/L-068	Methylene chloride	JB	1,600	µg/L
	7860A/L-068	Tetrachloroethene	J	3,900	µg/L
	7860A/L-068	Trichloroethene		15,000	µg/L
	7860A/L-068	Vinyl acetate	J	1,600	µg/L
	7860A/L-068	Xylene (total)		8,800	µg/L
	7860A/L-068	2-Butanone	E	1,700,000	µg/L
	7860A/L-068	4-Methyl-2-pentanone	J	5,000	µg/L
	7860A/L-069	Acetone	BE	1,400,000	µg/L
	7860A/L-069	Methylene chloride	JB	1,200	µg/L
	7860A/L-069	Tetrachloroethene		8,500	µg/L
	7860A/L-069	Trichloroethene		41,000	µg/L
	7860A/L-069	Vinyl acetate	J	2,100	µg/L
	7860A/L-069	Xylene (total)		11,000	µg/L
	7860A/L-069	2-Butanone	E	2,000,000	µg/L
	7860A/L-069	4-Methyl-2-pentanone	J	5,300	µg/L
SVO	7860A/L-063	Bis(2-ethylhexyl)phthalate	J	600,000	µg/kg
	7860A/L-063	Isophorone	J	41,000	µg/kg
	7860A/L-063	Naphthalene	J	82,000	µg/kg
	7860A/L-063	2-Methylnaphthalene	J	71,000	µg/kg
	7860A/L-064	Bis(2-ethylhexyl)phthalate	J	630,000	µg/kg
	7860A/L-065	Bis(2-ethylhexyl)phthalate	J	530,000	µg/kg
	7860A/L-065	Di-*n*-butylphthalate	J	62,000	µg/kg
	7860A/L-065	Naphthalene	J	92,000	µg/kg

Table 3.14 (continued)

Analysis	Sample ID	Compound	Qualifier	Result	Units
SVO	7860A/L-065	2-Methylnaphthalene	J	86,000	μg/kg
	7860A/L-066	Bis(2-ethylhexyl)phthalate	J	560,000	μg/kg
	7860A/L-066	Di-*n*-butylphthalate	J	59,000	μg/kg
	7860A/L-066	Naphthalene	J	89,000	μg/kg
	7860A/L-066	2-Methylnaphthalene	J	74,000	μg/kg
	7860A/L-067AQ	Bis(2-ethylhexyl)phthalate		20,000	μg/L
	7860A/L-067AQ	Di-*n*-butylphthalate	J	1,760	μg/L
	7860A/L-067AQ	Naphthalene	J	3,200	μg/L
	7860A/L-067AQ	2-Methylnaphthalene	J	3,200	μg/L
	7860A/L-067OR	Bis(2-ethylhexyl)phthalate	J	490,000	μg/kg
	7860A/L-067OR	Di-*n*-butylphthalate	J	60,000	μg/kg
	7860A/L-067OR	Naphthalene	J	91,000	μg/kg
	7860A/L-067OR	2-Methylnaphthalene	J	79,000	μg/kg
	7860A/L-069AQ	Naphthalene	J	240	μg/L

to 20 wt %. TBP was estimated at 40, 80, 30, 70, and 80 mg/g in L-063, L-064, L-065, L-066, and L-067org, respectively. A more detailed analysis could have been achieved in the SVOA by using a lesser dilution, but the danger of contaminating the GC-MS with large amounts of organic matter would have been greater. Percentage concentrations of several volatile solvents also were determined by the VOA. Tetra- and trichloroethenes averaged ~4 wt. % each. Substantial concentrations (thousands of mg/L) of acetone, xylene, and 2-butanone, and lesser levels (averages of 88 to 440 mg/L) of ethylbenzene, methylene chloride, and toluene also were found.

3.3.19.2 L-067, L-068, and L-069 (all aqueous liquids)

The aqueous layer had substantial concentrations of organic compounds, as would be expected from its contact with the organic layer. In contrast to the organic layer, the major identified compounds in the aqueous layer were those which are more water-soluble. Hydrophilic compounds such as the ketones were approximately equally distributed between the aqueous layer and the organic layer, while hydrophobic compounds such as the aromatic and chlorinated hydrocarbons were more concentrated in the organic layer. The major compounds determined in the VOA of the aqueous layer were acetone (average of 1300 mg/L), 2-butanone (average of 1800 mg/L), and trichloroethene (240 mg/L). Lesser concentrations of xylene, tetrachloroethane, methylene chloride, 4-methyl-2-pentanone, and vinyl acetate also were determined. Among the VOA TICS in the aqueous layer were ethyl, isopropyl, and *n*-butyl alcohols. They probably were more accurately measured by the DAI-GC than by the SVOA because of their high concentrations. The 4-methyl-2-pentanone and vinyl acetate were not detected in the organic layer, probably because of their low

concentrations and high water solubilities. For the SVOA in the aqueous layers, only naphthalene, 2-methylnaphthalene, and two phthalates were detected in L-067aq (and also naphthalene in L-069). TBP was the major TIC, estimated at 2, 300, and 300 mg/L in L-067aq, L-068, and L-069, respectively.

The agreement between the DAI-GC and the VOA for acetone and 2-butanone is generally good, considering that all of the GC-MS measurements for these compounds were above the calibration range (as flagged with the "E" data qualifier). The data for the DAI-GC probably are more accurate because the measurements were within the calibration range.

3.3.20 Tank W-3

3.3.20.1 L-016, L-017, and L-018 (all aqueous liquids)

The analytical results for these samples, which were prepared as a group, are listed in Table 3.15. The major organic compound was methanol. The DAI-GC also determined mg/L levels of acetone and 2-butanone that were not confirmed by the VOA. Very low concentrations of other volatiles were detected in the VOA. The SVOA determined very low levels of phthalates that were not significantly different from those in the blank. PAH also were determined in L-018, which did have a TOC approximately double that of the other two liquids. TBP was estimated as a TIC at 30 mg/L in L-016, L-017, and L-018.

3.3.20.2 S-020 (soft sludge)

As observed in other samples, somewhat higher levels of PAHs and phthalates were measured in the sludge. The latter were not significantly different in concentration from the blank. TBP was estimated at 4 mg/kg as a TIC.

3.3.21 Tank WC-17

3.3.21.1 O-101, O-102, L-103, L-104, and L-105 (all aqueous liquids)

This complex sample set, prepared as a group, consisted of three samples having an organic layer floating on an aqueous liquid, as well as two additional aqueous liquids. The activity of these samples was low enough that the aqueous and organic layers both could be subjected to purge-and-trap GC-MS in the

Table 3.15. Listing of organic analytical data hits for tank W-3

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-3/L-016	Methyl alcohol		38	mg/L
	W-3/L-017	Methyl alcohol		40	mg/L
	W-3/L-018	Acetone		7	mg/L
	W-3/L-018	Methyl alcohol		20	mg/L
	W-3/L-018	2-Butanone		5	mg/L
	W-3/L-018	*n*-Butyl alcohol		7	mg/L
VOA	W-3/L-016A	Acetone	JB	2	μg/L
	W-3/L-016A	Methylene chloride	JB	4	μg/L
	W-3/L-016A	Tetrachloroethene	B	6	μg/L
	W-3/L-016A	Toluene	JB	5	μg/L
	W-3/L-016A	Trichloroethene	B	11	μg/L
	W-3/L-017A	Acetone	B	26	μg/L
	W-3/L-017A	Chloroform	J	1	μg/L
	W-3/L-017A	Methylene chloride	JB	4	μg/L
	W-3/L-017A	Tetrachloroethene	B	8	μg/L
	W-3/L-017A	Toluene	B	14	μg/L
	W-3/L-017A	Trichloroethene	B	14	μg/L
	W-3/L-018A	Acetone	JB	5	μg/L
	W-3/L-018A	Benzene	J	4	μg/L
	W-3/L-018A	Chlorobenzene		8	μg/L
	W-3/L-018A	Methylene chloride	JB	3	μg/L
	W-3/L-018A	Toluene	B	37	μg/L
	W-3/L-018A	Trichloroethene	B	11	μg/L
	W-3/L-018A	4-Methyl-2-pentanone		18	μg/L
SVO	W-3/L-016	Di-*n*-butylphthalate	J	60	μg/L
	W-3/L-016	Di-*n*-octylphthalate	J	49	μg/L
	W-3/L-017	Di-*n*-butylphthalate	J	48	μg/L
	W-3/L-017	Di-*n*-octylphthalate	J	80	μg/L
	W-3/L-018	Di-*n*-butylphthalate	J	230	μg/L
	W-3/L-018	Fluoranthene	J	56	μg/L
	W-3/L-018	Phenanthrene	J	33	μg/L
	W-3/L-018	Pyrene	J	60	μg/L
	W-3/L-018	2-Nitrophenol	J	53	μg/L
	W-3/S020	Phenanthrene	J	12	μg/kg
	W-3/S020	Di-*n*-butylphthalate	JB	47	μg/kg
	W-3/S020	Chrysene	J	4	μg/kg
	W-3/S020	Bis(2-ethylhexyl)phthalate	JB	23	μg/kg

GC-MS laboratory. Small volumes were diluted in water at the time of purge and trap because of the expected high concentrations of volatile organic compounds. There was insufficient sludge S-106 for SVOA. The data for these samples are presented in Table 3.16.

The main compounds identified in the VOA were acetone, benzene, methylene chloride, and xylene. However, considering the high blank levels (see Table 3.17), only xylene appears to be present in the sample at 50-80 μg/L. The reason for the high blank level is not known. The lack of detectable organic compounds in the DAI-GC supports the contention that most of the results for the VOA were blank-related.

In the SVOA, only phthalates were detected at levels similar to those in the blank.

3.3.21.2 O-101, O-102, L-103 (all organic layers floating on aqueous layers of the same sample number)

Considering the elevated levels of compounds detected in the VOA blank, only styrene (at 5000 to 8000 μg/L) can be confidently identified in the organic layer. Trichloroethylene and vinyl acetate also were found, but in only one of the samples. The SVOA detected only phthalates, which appear to be related to the blank. The TICs were mostly unknown hydrocarbons, except for 1,1-biphenyl-2-ol (200 mg/kg in all three organic layers). A low level of PCBs was determined in the organic layers. This appeared to be a mixture of Aroclors 1254 and 1260, with a total concentration of 29-33 mg/kg.

The samples from tanks W-13, W-14, W-15, and W-1A were each processed together.

3.3.22 Tank W-13: L-027 and L-028 (Both Aqueous Liquids)

Very little organic matter was determined in these samples. The main compound measured by DAI-GC was methanol. Toluene was the main TCL species found by the VOA. The VOA surrogate standard recoveries were low for L-028, which probably is the reason that the VOA for L-027 yielded more hits. The SVOA did not detect any TCL constituents, and the TICs were not significant, except for TBP (90 μg/L in L-027). The SVOA extracts for both samples were not different from the blank, and L-028 was not submitted for GC-MS. The TOCs for these samples were low, 41.4 and 39 mg/L for L-027 and L-028, respectively.

Table 3.16. Listing of organic analytical data hits for tank WC-17

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	WC-17/L-103AQ	Acetone	B	170	μg/L
	WC-17/L-103AQ	Benzene	JB	10	μg/L
	WC-17/L-103AQ	Methylene chloride	JB	38	μg/L
	WC-17/L-103AQ	Xylene (total)	J	50	μg/L
	WC-17/L-103OR	Acetone	B	6,400	μg/L
	WC-17/L-103OR	Benzene	JB	420	μg/L
	WC-17/L-103OR	Methylene chloride	B	5,300	μg/L
	WC-17/L-103OR	Styrene		7,500	μg/L
	WC-17/L-103OR	Xylene (total)	B	2,500	μg/L
	WC-17/L-104AQ	Acetone	B	150	μg/L
	WC-17/L-104AQ	Benzene	JB	10	μg/L
	WC-17/L-104AQ	Methylene chloride	JB	44	μg/L
	WC-17/L-104AQ	Xylene (total)		59	μg/L
	WC-17/L-105AQ	Acetone	B	130	μg/L
	WC-17/L-105AQ	Benzene	JB	10	μg/L
	WC-17/L-105AQ	Methylene chloride	JB	40	μg/L
	WC-17/L-105AQ	Xylene (total)	J	50	μg/L
	WC-17/O-101AQ	Acetone	B	150	μg/L
	WC-17/O-101AQ	Benzene	JB	7	μg/L
	WC-17/O-101AQ	Methylene chloride	JB	39	μg/L
	WC-17/O-101AQ	Xylene (total)	J	46	μg/L
	WC-17/O-101OR	Acetone	B	6,700	μg/L
	WC-17/O-101OR	Acetone	B	6,700	μg/L
	WC-17/O-101OR	Benzene	JB	450	μg/L
	WC-17/O-101OR	Benzene	JB	410	μg/L
	WC-17/O-101OR	Methylene chloride	B	6,500	μg/L
	WC-17/O-101OR	Methylene chloride	B	4,000	μg/L
	WC-17/O-101OR	Styrene		7,100	μg/L
	WC-17/O-101OR	Styrene		8,200	μg/L
	WC-17/O-101OR	Trichloroethene	J	920	μg/L
	WC-17/O-101OR	Vinyl acetate	J	1,200	μg/L
	WC-17/O-101OR	Xylene (total)	JB	2,200	μg/L
	WC-17/O-101OR	Xylene (total)	JB	2,300	μg/L
	WC-17/O-102AQ	Acetone	B	140	μg/L
	WC-17/O-102AQ	Benzene	JB	9	μg/L
	WC-17/O-102AQ	Methylene chloride	JB	37	μg/L
	WC-17/O-102AQ	Xylene (total)		76	μg/L
	WC-17/O-102OR	Acetone	B	7,400	μg/L
	WC-17/O-102OR	Benzene	JB	400	μg/L
	WC-17/O-102OR	Methylene chloride	B	6,600	μg/L
	WC-17/O-102OR	Styrene		4,700	μg/L
	WC-17/O-102OR	Xylene (total)	B	2,700	μg/L
	WC-17/L-103AQ	Bis(2-ethylhexyl)phthalate	J	38	μg/L
	WC-17/L-103AQ	Di-*n*-butylphthalate	JB	56	μg/L
	WC-17/L-103AQ	Di-*n*-octylphthalate	JB	93	μg/L
	WC-17/L-103OR	Di-*n*-butylphthalate	JB	68,000	μg/kg

Table 3.16 (continued)

Analysis	Sample ID	Compound	Qualifier	Result	Units
	WC-17/L-104AQ	Di-*n*-octylphthalate	JB	33	μg/L
	WC-17/O-101AQ	Di-*n*-octylphthalate	JB	40	μg/L
	WC-17/O-101OR	Di-*n*-butylphthalate	JB	74,000	μg/kg
	WC-17/O-101OR	Di-*n*-butylphthalate	JB	57,000	μg/kg
	WC-17/O-102AQ	Di-*n*-octylphthalate	JB	24	μg/L
	WC-17/O-102OR	Di-*n*-butylphthalate	JB	44,000	μg/kg
PCB	WC-17/L-103OR	AROCLOR-1254		20	mg/kg
	WC-17/L-103OR	AROCLOR-1260		13	mg/kg
	WC-17/O-101OR	AROCLOR-1254		18	mg/kg
	WC-17/O-101OR	AROCLOR-1260		11	mg/kg
	WC-17/O-102OR	AROCLOR-1254		20	mg/kg
	WC-17/O-102OR	AROCLOR-1260		13	mg/kg

3.3.23 Tank W-14: L-030 and L-031 (Both Aqueous Liquids)

The results for this tank were essentially the same as for W-13, i.e., methanol was the major compound detected by DAI-GC, very little was measured in the VOA, and only a phthalate was found in the SVOA. As for the previous tank, the SVOA extract of only one sample (L-030) was submitted for GC-MS. TBP was estimated at 200 ug/L in L-030. The TOCs were higher for this tank (121 and 132 mg/L, respectively).

3.3.24 Tank W-15: L-032 and L-033 (Both Aqueous Liquids)

The results for this tank were very similar to those for the previous two tanks. The DAI-GC indicated 7 mg/L of methanol for both samples, and the VOA determined very little material. No TCL compounds were detected by the SVOA. TBP was estimated at 400 μg/L in both L-032 and L-033. This is consistent with the low TOCs, 53.7 mg/L for L-032 and 42.7 mg/L for L-033. The deuterated perylene internal standard was missing from the L-032 SVO extract. The cause is not clear. A matrix effect is possible because the other internal standards were found in the extract and all were added together before the GC screen. It may be significant that the pH for both of these samples was 0.2, which was the lowest of all the inactive tank samples. However, all the internal standards were found in the L-033 sample, and the inorganic characterization did not suggest any unusual properties.

3.3.25 Tank W-1A: L-001 (Aqueous Liquid)

The results for this tank were very similar to those for the previous three tanks. The DAI-GC determined 7 mg/L of methanol, and only very low levels of volatiles were detected in the VOA. The SVOA determined only a phthalate. TICs included C_4-benzenes (probably diethylbenzenes used in radiochemical extractions) at 5 mg/L and ethylphenylethanone at 3 mg/L.

The samples for tanks T-30 and 7562 were each processed together.

3.3.26 Tank T-30: L-121 (Aqueous Liquid)

As for the previous group of tanks, 11 mg/L of methanol were determined by the DAI-GC, and very little in the way of volatile organics was found by the VOA. The SVOA identified only a phthalate. These findings are in line with the low TOC, 13.7 mg/L.

3.3.27 Tank 7562: L-097 (Aqueous Liquid)

Again, very little organic matter was detected in this sample: methanol at 14 mg/L by DAI-GC, very low VOA results, and no SVOA TCL hits. The TOC was 29.6 mg/L. There was insufficient sludge in S-098 for the SVOA.

3.4 ANALYSIS OF BLANKS

Method blanks were prepared with almost every group of samples. Burdick and Jackson "distilled-in-glass" grade water or sea sand were prepared and analyzed identically to and simultaneously with the samples. The results for the VOA and SVOA of the aqueous sample blanks are summarized in Table 3.17. Only very low levels of a few TCL compounds were determined by the VOA. The two exceptions, methylene chloride in the T-1/T-2/T-9 sample group-apparently resulted from contamination of the solid sorbent traps during storage. When this contamination was detected, the traps were analyzed soon after preparation. It is not clear why the methanol used for the dilution of one of the organic liquids apparently contained such high levels of TCL compounds.

Only two phthalates were detected in two groups of SVOA blanks. Di-*n*-butylphthalate (21 μg/kg) and bis(2-ethylhexyl)phthalate (8 μg/kg) were the only TCL compounds detected in the SVOA blanks for the sludges. The blanks for the DAI-GC did not contain detectable concentrations (<1–2 mg/L) of the alcohols and ketones.

3.5 RECOVERIES OF SURROGATE STANDARDS AND MATRIX SPIKES

The recoveries of standard compounds added to aliquots of the waste samples before preparation and analysis are good indicators of the accuracy and reproducibility of the determinations. Surrogate standards were added to all samples in the VOA and SVOA. Matrix spiked samples were included in the VOA, SVOA, DAI-GC, and PCB analyses.

Table 3.17. Results for VOA and SVOA of blanks for aqueous liquids

(Concentration in blank for sample group,[a] μg/L)

Compound	T-3 T-4	T-1 T-2 T-9	TH-1 TH-3 W-11	TH-4	W-5 W-6 W-7	W-8, W-9	W-1 W-2 W-4	W-10	W-3	WC-17	W-13 W-14 W-15 W-1A	WC17ORG[b]
VOA:												
Acetone	28	23	23	16	36	27	—[c]	—	2	150	28	6,700
Bromodichloromethane	10	—	—	—	—	—	—	—	—	—	—	—
Chloroform	45	9	—	—	—	—	28	—	—	13	—	—
Methylene chloride	9	880	18	17	13	14	7	13	8	18	7	4,100
Toluene	8	10	16	18	18	8	16	—	5	—	43	—
Benzene	—	5	—	—	—	—	—	—	—	—	2	370
4-Methyl-2-pentanone	—	—	—	11	—	—	—	—	—	—	—	—
Tetrachloroethene	—	—	—	—	22	—	—	—	10	—	—	—
Trichloroethene	—	—	—	—	—	—	—	—	16	—	4	—
Xylenes	—	—	—	—	—	—	—	—	—	—	—	1,800
Carbon tetrachloride	—	—	—	—	52	—	—	—	—	—	—	—
SVOA:												
Di-*n*-butylphthalate	—	—	—	—	—	—	—	—	13	110	—	—
Di-*n*-octylphthalate	—	—	—	—	—	—	—	—	69	170	—	—

[a]Results listed for aqueous blanks only, unless otherwise indicated.
[b]Results for methanol used to dilute organic layers.
[c]Data not available.

The recoveries of MASs for the DAI-GC of aqueous liquids is shown in Table 3.18. The data are for eight pairs of MAS/MSD. One data point for methyl alcohol was dropped because of an apparent misintegration in its measurement. Except for i-propyl (89.9% mean recovery) and allyl/*n*-propyl alcohols (85.5% mean recovery), the mean spike recoveries were greater than 93%. The allyl/*n*-propyl alcohol was not detected in any sample. The relative standard deviation of the recoveries ranged from 9.7 to 21.6%. These data suggest good accuracy and precision for the measurements. There are no SW-846 surrogate standards or QC Acceptance Limits for this method.

The three VOA surrogate standards were recovered in reasonable yield from both the aqueous radioactive waste tank samples and from water blanks prepared inside the glove box. Using a data base of 65 waste tank samples, the mean recoveries and standard deviations (both expressed in % recovery) of d_8-toluene, bromofluorobenzene, and 1,2-dichloroethane-d_4 were 89 $\pm$ 17%, 59 $\pm$ 15%, and 81 $\pm$ 11%, respectively (Table 3.19). Both the mean recovery and precision are acceptable at the 50 μg/L level in this hostile matrix. The mean values are similar to the QC Acceptance Limits specified by EPA Method 8240 for ground water analyses,[1] viz., 88–110% for d_8-toluene, 86–115% for bromofluorobenzene, and 76–114% for 1,2-dichloroethane-d_4. The recoveries of the surrogate standards from seven water blanks were 91 $\pm$ 11%, 61 $\pm$ 12%, and 86 $\pm$ 13%, respectively. Data from four blanks were discarded because of apparent laboratory error. The applicability of QC Acceptance Limits developed for groundwater analysis to our waste tank liquids analysis is unknown, but those limits are useful guidelines for evaluating these data. The reasons for the lower recoveries of bromofluorobenzene from both the blanks and the samples are not clear. We suggest that this surrogate has the highest boiling point (and is therefore the least volatile) of the three surrogates, and the sparging conditions employed may not have been vigorous enough to transfer it quantitatively to the sorbent trap.

The recoveries of the five VOA MAS compounds were evaluated using 10 pairs of MAS/MSD. The mean recoveries and relative standard deviations of 1,1-dichloroethene, trichloroethene, benzene, toluene, and chlorobenzene ranged from 105 to 67%. Some of the raw-data values had to be deleted from the evaluation because they either exceeded 150% recovery (one for benzene, two for toluene, and one for chlorobenzene), or they were inexplicably zero when all other MAS compounds were recovered reasonably (two for toluene, composing one pair). Again, the recoveries of the five MAS compounds generally fell within the QC Acceptance Limits specified by the EPA CLP,[1] which are typically between ~ 70 to 130%. SW-846 does not list QC Acceptance Limits for MASs. The agreement between results for MAS and MSDs was typically within 10%.

The recoveries of the six SVOA surrogate standard compounds from 67 waste tank aqueous liquids and eight blanks are shown in Table 3.20. The recovery data for the acid surrogates taken from four samples were rejected because of apparent laboratory error and are not included in the tabulation. The recoveries

Table 3.18. Summary of matrix spike recoveries for DAI-GC
(Percentages unless otherwise indicated)

Compound	Mean	Std. Dev.	RSD	No. of samples
Methyl alcohol	97.8	16.6	16.9	15
Ethyl alcohol	94.1	10.7	11.3	16
Acetone	94.8	10.6	11.1	16
i-Propyl alcohol	89.9	11.4	12.7	16
Allyl/*n*-propyl alcohol	85.5	18.3	21.4	16
2-Butanone	93.6	9.1	9.7	16
i-Butyl alcohol	97.3	10.6	10.9	16
n-Butyl alcohol	102.4	13.3	13.0	16
4-Methyl-2-pentanone	94.2	20.4	21.6	16

Table 3.19. Summary of VOA surrogate standard and matrix spike recoveries for aqueous liguids and blanks

(Percentages unless otherwise indicated)

Compound and QC acceptance range	Waste samples				Blanks			
	No. of samples	Mean	Std. dev.	RSD	No. of blanks	Mean	Std. dev.	RSD
Surrogate standards								
d_8-Toluene (88-110)	65	89	17	19	7	91	11	12
Bromofluorobenzene (86-115)	65	59	15	25	7	61	12	20
1,2-Dichloroethane-d_4 (76-114)	65	81	11	14	7	86	13	15
Matrix spikes								
1,1-Dichloroethene (61-145)	20	105	18	17				
Trichloroethene (71-120)	20	87	14	16				
Benzene (76-127)	19	89	15	17				
Toluene (76-125)		15	81	20	25			
Chlorobenzene (75-130)	19	67	13	19				

Table 3.20. Summary of SVOA surrogate standard and matrix spike recoveries for aqueous liquids and blanks

(Percentages unless otherwise indicated)

Compound and QC acceptance range	Waste samples				Blanks			
	No. of samples	Mean	Std. dev.	RSD	No. of blanks	Mean	Std. dev.	RSD
Surrogate standards								
Nitrobenzene d_5 (35-114)	67	70	15	21	8	65	18	28
2-Fluorobiphenyl (43-116)	67	64	14	22	8	58	13	23
Terphenyl-d_{14} (33-141)	67	88	17	19	8	85	18	21
Phenol-d_5 (10-94)	63	53	12	23	8	49	12	24
2-Fluorophenol (21-100)	63	48	11	24	8	42	8	19
2,4,6-Tribromophenol (10-123)	63	71	17	24	8	69	14	20
Acid matrix spikes								
Phenol (12-89)		10	40	9	22			
2-Chlorophenol (27-127)	10	43	7	17				
4-Chloro-3-methylphenol (23-97)	10	55	19	34				
4-Nitrophenol (10-80)	10	69	31	46				
Pentachlorophenol (9-103)	8	74	32	44				

Table 3.20. (continued)

Compound and QC acceptance range	Waste samples				Blanks			
	No. of samples	Mean	Std. dev.	RSD	No. of blanks	Mean	Std. dev.	RSD
Base/neutral matrix spikes								
1,4-Dichlorobenzene (36-97)	14	50	10	20				
N-Nitroso-di-*n*-propylamine (41-116)	14	66	10	15				
1,2,4-Trichlorobenzene (39-98)	14	53	12	22				
Acenaphthene (46-118)	14	64	13	21				
2,4-Dinitrotoluene (24-96)	14	71	18	25				
Pyrene (26-127)	14	78	12	15				

of the acid surrogates were zero in those samples, while the recoveries of the base/neutral surrogates were good, suggesting that an error was made either in the addition of the surrogate standards or in the pH adjustment of the extraction. Overall, the mean recoveries were well within the SW-846 QC Acceptance Limits,[1] which are quite wide. The precision of the measurements is approximately 20%. These observations also hold for the surrogate standard recoveries in the eight blank samples.

Seven pairs of SVOA MAS/MSD recoveries also were evaluated, and are included in Table 3.20. While all of the available data for base/neutral MASs were used, two pairs of acid MAS data were rejected. The recoveries of the acid MAS compounds were inexplicably zero, while the acid surrogate standard compounds were recovered well, suggesting a laboratory error. The mean recovery values for both the acid and base/neutral MASs fell within the EPA CLP QC Acceptance Limits for soil.[2] The standard deviations and relative standard deviations of the base/neutral MASs were almost identical to those observed for the base/neutral surrogate standards. These two parameters tended to be somewhat greater for the acid MASs. Possible reasons for the poorer precision for the latter may be that (*a*) the pH of the sample was insufficiently low to allow extraction of the more acidic phenols and that (*b*) reliable quantitation of at least one species, pentachlorophenol, by GC-MS is historically difficult. The agreement between the MASs and MSDs was typically between 10 and 20%.

The sludge samples also were routinely spiked with surrogate standards, and the recovery data for 19 samples and one blank are presented in Table 3.21. Data for two samples and one blank were discarded because of uniformly very low recoveries. The means and relative standard deviations of the surrogate standard recoveries from the sludge were poorer than those for the aqueous liquid analyses. In contrast, the recoveries from the blank (sea sand) were good, suggesting that the problems were mainly in extracting the sludge matrix. Recoveries were particularly poor for nitrobenzene, and the recoveries of fluorobiphenyl and the fluoro- and tribromophenols were not much better. It is possible that the nitrobenzene and fluorobiphenyl losses arose from the rotary evaporation or nitrogen blow-down steps used for volume reduction of the extracts, while the more acidic phenols may not have been efficiently extracted from the alkaline sludges. SW-846 method 3550 does not expressly incorporate a pH adjustment. Methodology for achieving base/neutral and acid fraction extractions of sludges is under development.

Only one pair of MAS/MSDs was prepared from one sludge sample at the end of the study, and the recoveries were generally poor. However, because the surrogate standard recoveries for that sample and for the MAS/MSDs all were good, we concluded that either the matrix spiking solution had degraded or its addition was faulty, and that the MAS recovery data therefore were not valid.

Table 3.21. Summary of SVOA surrogate standard recoveries from sludges

(Percentage)

Surrogate std. and QC limits[a]	Sludge samples[b] Mean	Sludge samples[b] Std. dev.	Sludge samples[b] RSD	Blank[c] Mean
Nitrobenzene-d5 (23-120)	18	17	99	33
2-Fluorobiphenyl (30-115)	37	19	52	35
Terphenyl-d_{14} (18-137)	66	20	30	99
Phenol-d5 (24-113)	47	20	43	46
2-Fluorophenol ((25-121)	35	19	56	39
2,4,6-Tribromophenol (19-122)	37	18	45	92

[a]From SW-846; see ref. 1, Sect. 3.7.
[b]N = 19.
[c]N = 1.

Two dilutions of an organic oil sample were spiked with PCB-1248 to evaluate potential interferences. The recoveries of 120 and 108% suggest that no interferences were encountered in the PCB determinations and that the measurements were accurate.

3.6 ACCOUNTING OF ORGANIC CARBON

Comparison of the results of the TOC analysis with the organic carbon calculated from the GC and GC-MS measurements of individual compounds allows an assessment of the completeness of the accounting of organic matter present in the waste samples. Such a comparison is presented in Tables 3.22 and 3.23 for the aqueous liquids and sludges, respectively. The results of the DAI-GC determination of major volatile organic compounds, the TCL and TIC compounds measured by the VOA (compounds common to the DAI-GC analysis have been omitted from the VOA data so that they will not be counted twice), and

Table 3.22. Total organic carbon (TOC) accounting in aqueous liquids[a]

Tank	Sample	TOC,	DAI-GC		VOA-TCL[b]		SVOA-TIC		VOA-TCL		SVOA-TIC		TOC Accounted for (%)
T-30	L-121	13.7	4.1	ALC	–		–		–		0.2	NID	31
TH-4	L-055	19.4	4.9	ALC	–		–		–		0.6	NID	28
	L-056	352	23.7	ALC	0.1	OTR	0.20		0.9	ETR	3.1	TBP	19
			25.7	KET					0.5	OTR	11.0	NID	
	L-057	2,170	4.5	ALC	0.4	KET	<0.1		–		4.9	TBP	0.8
	L-116	14.6	–		0.1	OTR	<0.1		0.1O TR		0.8	NID	7
											7.5	NID	
	L-117	31.6	7.5	ALC	<0.1		NR		<0.1		NR		24
W-10	L-094	86.4	13.9	ALC	<0.1		0.3	BA	<0.1		0.3	TBP	18
											0.8	NID	
	L-095	54.6	16.5	ALC	0.1	OTR	2.0	BA	<0.1		1.8	EPE	45
			2.6	KET							1.7	NID	
W-6	L-077	2	–		<0.1		<0.1		<0.1		1.1	NID	55
	L-078	2	–		<0.1		<0.1		<0.1		2.0	NID	100
	L-079	13.2	1.9	ALC	0.3		0.2	BA	0.1		11.2	NID	104
W-7	L-082	50	5.3	ALC	0.08	CH	1.3	BA	<0.1		20.3	NID	54
7562	L-097	29.6	5.3	ALC	<0.1		<0.1		<0.1		0.3	NID	19
W-10	L-093	8.7	10.1	ALC	–		–		–		0.1	NID	117
	L-094	86.4	13.8	ALC	<0.1		–		<0.1		0.3	TBP	18
											0.2	EPE	
											1.4	NID	
	L-095	54.6	16.6	ALC	<0.1		–		<0.1		3.7	NID	42
			2.6	KET									
T-1	L-035	836	–		0.1	BA	0.35	PHTH	<0.1		6.7	TBP	1.6
					0.1	OTR					6.0	NID	
	L-036	790	–		<0.1		0.15	PHTH	–		5.4	TBP	1.9
											9.4	NID	

Table 3.22 (continued)

Tank	Sample	TOC,	DAI-GC	VOA-TCL[b]	SVOA-TIC	VOA-TCL	SVOA-TIC	TOC Accounted for (%)
T-2	L-038	1120	–	0.03 CH	0.1 PHTH 0.17 PNL	– –	3.8 TBP 6.1 NID	0.9
	L-039	1310	–	0.04 CH	0.2 PHTH	–	3.4 TBP 8.9 NID	1.0
	L-112	1100	–	0.03 CH	0.2 PHTH 0.13 PNL	–	3.2 TBP 7.1 NID	1.0
T-3	L-042	12,600	65.7 ALC	<0.1	0.09 ALC	<0.1	0.4 TBP 11.6 NID	0.6
T-4	L-044	460	40.5 ALC 4.3 KET	0.06 CH 0.04 AH	<0.1	0.1 OTR	5.4 NID	11.0
	L-045	473	40.3 ALC 5.0 KET	0.24 AH 0.06 OTR	<0.1	0.1 OTR	5.7 NID	9.8
	L-111	460	39.0 ALC 4.3 KET	0.08 CH 0.05 AH 0.04 OTR	<0.1	0.1 OTR	0.5 TBP 8.0 NID	11
T-9	L-047	850	6.4 ALC	<0.1	0.2 BA 0.1 ALC	–	4.5 TBP 10.7 NID	2.6
TH-3	L-053	5.1	–	<0.1	<0.1	<0.1	3.4 NID	67
W-1	L-007	6.4	–	<0.1	<0.1	–	0.4 NID	6.3
	L-008	10.1	–	<0.1	<0.1	<0.1	0.6 NID	5.9
W-13	L-027	41.4	4.1 ALC	<0.1	–	–	0.02 TBP 0.3 NID	11
	L-028	39	1.9 ALC	<0.1	–	–	–	4.9
W-14	L-030	121	9.2 ALC	<0.1	<0.1	–	0.03 TBP 0.4 NID	8.0

Table 3.22 (continued)

Tank	Sample	TOC,	DAI-GC	VOA-TCL[b]	SVOA-TIC	VOA-TCL	SVOA-TIC	TOC Accounted for (%)
W-15	L-032	53.7	2.6 ALC	<0.1	–	<0.1	0.07 TBP 2.6 NID	9.8
	L-033	42.7	2.6 ALC	<0.1	–	–	0.07 TBP 0.8 NID	8.1
W-2	L-011	18.9	–	<0.1	<0.1	<0.1	0.5 NID	3
	L-118	22.6	–	<0.1	–	<0.1	0.6 NID	3
W-3	L-016	772	14.3 ALC	–	0.1 OTR	–	5.2 TBP 1.6 NID	2.7
	L-017	790	15.0 ALC	–	0.1 OTR	–	6.1 TBP 4.0 NID	3.2
	L-018	1,500	12.3 ALC	<0.1	0.16 PHTH 0.14 PAH 0.03 PNL	<0.1	4.7 TBP 3.3 NID	1.9
W-4	L-022	49.5	–	–	–	–	0.4 NID	0.8
	L-023	558.5	5.0 KET	<0.1	–	<0.1	16.5 NID	3.8
	L-119	59.9	–	<0.1	–	<0.1	0.7 NID	0.1
W-5	L-073	700	–	0.07 CH	<0.1	–	0.5 TBP 12.7 NID	1.9
W-8	L-086	720	0.6 KET	<0.1	–	<0.1	3.8 TBP 13.0 NID	2.4
	L-087	1,460	1.8 ALC 2.6 KET	<0.1	<0.1	<0.1	4.5 TBP 9.2 NID	1.2
W-9	L-090	290	–	<0.1	<0.1	–	2.0 TBP 11.3 NID	4.6

Table 3.22 (continued)

Tank	Sample		TOC,	DAI-GC		VOA-TCL[b]		SVOA-TIC		VOA-TCL	SVOA-TIC		TOC Accounted for (%)
WC-17	L-101	AQ	191	–		<0.1		<0.1		–	6,680	NID	>100
	L-102	AQ	143	–		<0.1		<0.1		–	1.2	NID	0.8
	L-103	AQ	309	–		<0.1		<0.1		–	10	NID	3.2
	L-104	AQ	153	–		<0.1		<0.1		–	0.9	NID	0.6
	L-105	AQ	127	–		<0.1		–		–	0.9	NID	0.7
7562	L-097		29.6	5.3	ALC	<0.1		–		<0.1	0.3	NID	19
7860A	L-067	AQ	43.8	3,042	ALC	12.7	AH	16.0	PHTH	–[c]	0.3	TBP	>100
				1,868	KET	14.6	CH	6.0	AH		581	NID	
						2.0	OTR						
	L-068		43.8	4,289.	ALC	8.0	AH	–		–[c]	50.4	TBP	>100
				2,250.	KET	3.4	CH				68.6	NID	
						0.9	OTR						
	L-069		45.6	3,593	ALC	10.0	AH	0.2	AH	–[c]	46.8	TBP	>100
				2,250	KET	8.8	CH				82.3	NID	

[a]Organic carbon calculated for different chemical classes using data for specific compounds determined with direct aqueous injection gas chromatography (DAI-GC), the volatile organics analysis (VOA), and the semivoaltile organics analysis (SVOA). The target compound list (TCL) and tentatively identified compounds (TICs) are listed separately. A factor of 0.85 was used to convert TICs from compound concentration to organic carbon concentration. Abbreviations of chemicals: ALC=alcohols, NID=not identified (TICs), KET=ketones, TBP=tributylphosphate, BA=benzoic acid, EPE=1-(4-ethylphenyl)-ethanone, CH=chlorinated hydrocarbon, AH=aromatic hydrocarbon, PHTH=phthalate, PNL=phenol, PAH=polycyclic aromatic hydrocarbon, OTR=other compound, NR=analysis not run.

[b]Does not include compounds also measured by DAI-GC, to eliminate duplication of results.

Table 3.23. Total organic carbon (TOC)[a] accounting in sludges

(mg/kg)

Tank	Sludge	TOC	Organic carbon from SVOA				TOC Accounted for %
			TCL		TIC		
T-1	S-037	18,600	2.4	PHTH	1.2	TBP	0.1
					15.9	NID	
T-2	S-040	28,000	22.8	PHTH	17.6	NID	0.2
			2.2	PAH			
T-3	S-043	9,140	3.3	PHTH	56.1	NID	0.7
T-4	S-046	4,620	3.9	PHTH	51.3	NID	1.2
T-9	S-048	7,620	4.6	PHTH	1.2	TBP	0.2
					13.3	NID	
TH-4	S-058	6,940	131.1	PAH	1.2	TBP	2.3
			26.5	PHTH	3.7	NID	
	S-059	7,530	115.6	PAH	1.2	TBP	1.9
			28.2	PHTH	0.8	NID	
W-10	S-096	8,180	5.9	PAH	30.9	TBP	4.5
			98.0	PHTH	230	NID	
	H-120	14,600	25.1	PAH	162.0	TBP	4.3
			0.5	PHTH	436	NID	
W-3	S-020	3,410	0.014	PAH	1.8	TBP	0.03
			0.05	PHTH	0.5	NID	
W-4	S-024	9,190	0.67	PAH	53.3	NID	0.7
			9.56	PHTH			
	H-026	9,020	0.4	PAH	43.9	NID	0.5
			5.23	PHTH			
W-5	S-075	4,020	0.8	PAH	4.2	TBP	2.3
			30.0	PHTH	56.3	NID	

Table 3.23 (continued)

Tank	Sludge	TOC	Organic carbon from SVOA TCL		Organic carbon from SVOA TIC		TOC Accounted for %
W-6	S-080	9,110	1.2	PAH	78.5	NID	1.3
			23.1	PHTH			
W-7	S-084	9,030	0.2	PAH	5.4	TBP	0.8
			42.5	PHTH	23.0	NID	
	H-085	4,010	1.8	PHTH	64.8	NID	1.7
W-8	S-088	11,300	6.3	PAH	357	TBP	8.4
			0.7	PHTH	587	NID	
W-9	S-092	13,900	3.4	PAH	298	NID	3.0
			119	PHTH			

[a]For abbreviations, see Table 3.22.

the TCL and TIC compounds from the SVOA are summarized by chemical class (or specific compound if they were major species) and are quantified as mg of organic carbon per L or kg of sample. For TICs that could not be identified, a factor of 0.85 was used to convert the estimated compound weight concentration to an estimated organic carbon weight concentration. This factor is similar to those for C_{10}-C_{20} *n*-hydrocarbons. Samples which did not receive a TOC or SVOA analysis are not included in the tables unless the DAI-GC analysis accounted for a significant portion of the TOC. A dash in Tables 3.22 and 3.23 indicates that the analysis did not detect any compounds, while a "less than sign" shows that some compounds were detected but their contribution to the TOC was insignificant.

The TOC accounting for aqueous liquids (Table 3.22) was generally quite low, and only for three samples (W-6/L-078, W-6/L-079, and W-10/L-093) was the accounting near quantitative. For two of these three samples, however, unidentified TICs constituted most of the organic carbon, and thus the identities of the organic compounds remained unknown. For approximately 50% of the samples listed, the TOC accounting was less than 5%, and for 78%, the accounting was less than 20%. The accounting for samples from tank 7860A was much greater than 100%. This probably resulted from a low TOC measurement. Volatile compounds can be lost from the TOC analytical procedure when inorganic carbon is purged. The samples from tank 7860A contained percentage concentrations of several volatile organic compounds that would be easily lost during even gentle purging. The TOC accounting also was very high for WC-17/L-101AQ, probably from a contamination of the aqueous sample taken for the SVOA extraction with the overlying organic layer.

Most of the TOC accounted for in the aqueous liquids was measured by the DAI-GC. This analysis found common laboratory solvents (mainly methyl, ethyl, and *n*-butyl alcohols; acetone; 2-butanone; and 4-methyl-2-pentanone). The SVOA TICs also were major identified contributors to the TOC, and TBP was the major TIC. This finding is not surprising because of the large volume of TBP used as an extractant. The TCL compounds determined by the VOA and SVOA generally accounted for relatively little of the organic carbon. For a few tanks, however, aromatic hydrocarbons (xylenes and toluene) and chlorinated hydrocarbons (tri- and tetraethenes, methylene chloride, and chloroform) were major VOA TCL compounds, and phthalates [especially bis-(2-ethylhexyl)phthalate and dibutylphthalate], phenols (nitro-, dichloro-, and trichlorophenols), and benzoic acid were major SVOA TCL compounds.

The TOC accounting for the sludges (Table 3.23) was much poorer than for the aqueous liquids, and for 95% of the listed samples the accounting was less than 5%. Phthalates [especially bis-(2-ethylhexyl)phthalate and di-*n*-butylphthalate], polycyclic aromatic hydrocarbons (unsubstituted 2–5 ring aromatic hydrocarbons), TBP, and unidentified TICs were the major contributors to the calculated organic carbon.

For both aqueous liquids and sludges, the bulk of the measured TOC remains unaccounted for at the present. It was initially hypothesized that this unaccounted organic matter consists of highly polar, water-soluble compounds derived from chelators, extractants, decontamination agents, and their degradation products. Such compounds would not be efficiently recovered by the SVOA extraction procedures because of their hydrophilicity and would not pass through the GC and be detected by the MS because of their thermal lability and nonvolatility. Indeed, studies at the Pacific Northwest Laboratory have demonstrated

the presence of such compounds and have found that the TOC content of some nuclear wastes can be accounted by them.[3,4] Among the compounds specifically identified were ethylenediaminetetraacetic acid, citric acid, N-(2-hydroxyethyl)ethylenediaminetriacetic acid, N-(ethylene)ethylenediaminetriacetic acid, ethylenediaminetriacetic acid, N-(2-hydroxyethyl)-N′-(methyl)ethylenediamine-N,N′-diacetic acid, iminodiacetic acid, methane tricarboxylic acid, C_2-C_9 dicarboxylic acids, and C_3-C_{18} monocarboxylic acids.

A study has been initiated to identify and account for the TOC in these waste samples, but only preliminary results are available at this time. It has been found that the SVOA extracts contain appreciable amounts of organic matter not detectable by SW-846 procedures. Some of this organic matter can be analyzed by GC and GC-MS if it is first chemically derivatized to block polar functional groups, improve thermal stability, and increase volatility. A good example is shown in Fig. 3.1. The gas chromatogram in Figure 3.1(A) is of the underivatized SVOA extract remaining after the GC-MS analysis was performed on sample T-2/L-112. The chromatogram in (B) is of the same extract after trimethylsilyl (TMS) derivatization. A larger number of compounds were detected after derivatization. At least 20 major new chromatographic peaks and many more minor peaks were visible. The total chromatographic peak area increased about twofold, suggesting that twice the amount of organic carbon was visualized.

GC-MS has not yet been performed upon the TMS-derivatized extract, but some preliminary identifications were made on the basis of gas chromatographic retention time matching and co-chromatography. Major peaks tentatively identified on this basis are listed in Table 3.24. These compounds were TBP (identified as a TIC in the SVOA), dibutylphosphate, di(2-ethylhexyl)phosphate, 2-ethyl-1-hexanol, ethylenediaminetetraacetic acid, hexadecanoic acid, and octadecenoic acid. All but the last two compounds have been used in considerable volumes at the Laboratory or are their expected degradation products (i.e., dibutylphosphate). The last two compounds also have been identified in studies of nuclear wastes at the Pacific Northwest Laboratory.[3,4] Minor species also tentatively identified in our work included oxalic acid, 2-nitrophenol (which was identified in the SVOA as a TCL compound), *p*-nitrobenzoic acid, tartaric acid, and several monocarboxylic acids ranging from C_{17} to C_{25}. The total of these TICs (except for TBP and 2-nitrophenol, which already were counted in the SVOA) accounted for ~14.8 mg/L of TOC, and the remaining unidentified new compounds added ~2.7 mg/L TOC. This doubled the TOC accounted for, and 3.2% of the TOC was accounted for or at least detected.

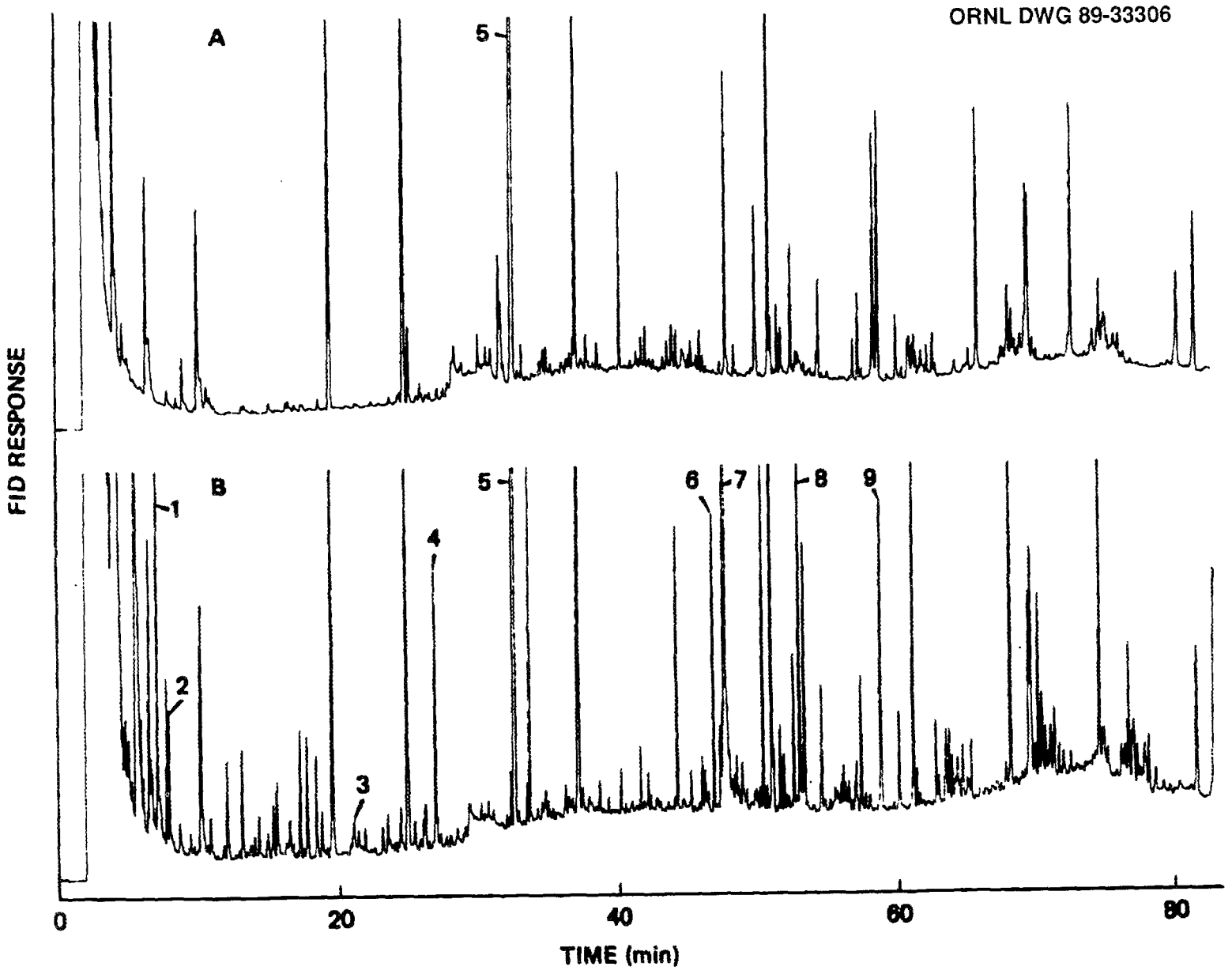


Fig. 3.1. Comparison of the capillary column gas chromatograms of the SVOA extract from sample T-2/L-112 (A) underivatized and (B) trimethylsilyl-derivatized forms. (Preliminary peak identifications and concentration estimates are listed in Table 3.24.)

Table 3.24. Compounds tentatively identified by cochromatography in the TMS-derivatized SVOA extract of T-2/L-112

Peak No.[a]	Tentative Identification[b]	Estimated Concentration[c], ug/L
1	2-Ethyl-1-Hexanol	1.3
2	Oxalic Acid	0.4
3	2-Nitrophenol	0.2[d]
4	Dibutylphosphate	1[d]
5	Tributylphosphate	18[d]
6	Hexadecanoic Acid	1.0
7	Di(2-Ethylhexyl)phosphate	18
8	Octadecanoic Acid	1.6
9	Ethylenediaminetetraacetic Acid	1.5

[a]As labeled in Fig. 3.1.
[b]Based only upon retention time matching and cochromatography; subject to verification by GC-MS.
[c]Estimate only. Recovery in SVOA extract is unknown.
[d]Concentration from SVOA.

It must be emphasized that these identifications are tentative until they are confirmed or disproved by other evidence, such as mass spectra. Although this does show that appreciable amounts of polar compounds were extracted into the SVOA extract, their recoveries are not known. Thus, their calculated concentrations in the aqueous sample also must be considered only as estimates.

Because polar hydrophilic compounds are not expected to extract efficiently into organic solvents even with extreme pH adjustments of the aqueous phase, we have studied means of directly derivatizing such compounds without extractions. Aliquots of several aqueous waste samples have been evaporated with and without acidification to convert acids to their free forms, and the dried residues have been trimethylsilylated. Additional aliquots were spiked with standards to gauge derivatization efficiencies, and others were made more alkaline for comparison. Although recoveries of spikes were variable, the additional gas chromatographable matter was minimal and clearly did not account for the TOC. Additional experiments are under way to substantiate our hypothesis that the missing TOC does not consist of the initially suspected chelators, extractants, and degradation products. These findings suggest that the bulk of the TOC may consist of chromatographic "intractable matter" — for example, polar, water-soluble, perhaps macromolecular species—which require an entirely different analytical approach. Mass spectral and liquid chromatographic methods are being tested.

3.7 SUMMARY OF ORGANIC ANALYTICAL RESULTS

Generally, only low microgram-per-liter concentrations of a few EPA Target Compound List (TCL) compounds were determined in the aqueous liquids by the volatile organics analysis (VOA) and the semivolatile organics analysis (SVOA). The main TCL compounds were ketones (e.g., acetone), chlorinated hydrocarbons (eg, methylene chloride), and aromatic hydrocarbons (eg, xylenes) for the VOA, and phthalates [e.g. bis-(2-ethylhexyl)phthalate], and polycyclic aromatic hydrocarbons (PAHs, e.g., naphthalene and pyrene) for the SVOA. Tentatively identified compounds (TICs) were detected mainly in the SVOA. TBP was the major TIC.

The direct aqueous injection gas chromatographic (DAI-GC) analysis indicated that many tanks contained mg/L concentrations of common alcohols (e.g., methyl and ethyl alcohols) and ketones (e.g., 4-methyl-2-pentanone), which were not readily detectable by the VOA or were above the calibration range of the VOA.

A few tanks (especially one with an organic upper layer) contained mg/L to percentage levels of chlorinated hydrocarbons (e.g., tri- and tetrachloroethenes), aromatic hydrocarbons (e.g., xylenes), and ketones (e.g., 2-butanone) in the aqueous phase and percentage levels in the organic phase.

Hydrophobic organic compounds preferentially accumulated in the sludges. Phthalates, PAHs, and TBP were measured at mg/kg concentrations. Sludge/aqueous-layer distribution coefficients for PAHs ranged from ~100 to 500, depending on PAH ring system size. The coefficient for TBP was <1, probably because of its greater polarity.

Chemical and/or radiochemical reactions are suggested by the presence of nitro-, dibromonitro-, and chlorophenols, benzoic acid, and benzyl alcohol.

The total organic carbon (TOC) was generally only poorly accounted for by the specific compounds measured by the regulatory analyses. Trimethylsilylation and capillary-column GC of a SVOA extract suggested the presence (cochromatography only) of dibutylphosphate, di(2-ethylhexyl)phosphate, 2-ethylhexanol, oxalic acid, and monocarboxylic acids. However, the TOC still was not accounted for, suggesting that the bulk of the TOC might consist of chromatographic "intractables" such as polar, hydrophilic macromolecular matter.

Characterization of the unaccounted organic matter will require the development of analytical methods capable of identifying and measuring polar, hydrophilic organics in high-ionic-strength, radioactive aqueous liquids.

3.8 REFERENCES

1. *Test Methods for Evaluating Solid Waste,* 3rd ed., U.S. EPA Office of Solid Waste and Emergency Response, Washington, D.C., November 1986.

2. *US EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multimedia, Multiconcentration, 10/86,* Rev. SOW no. 787, IFB WA-87K236, IFB WA-87K237, and IFB WA-87K238, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1987 .

3. A. P. Toste, T. J. Lechner-Fish, D. J. Hendren, R. D. Scheele, and W. G. Richmond, "Analysis of Organics in Highly Radioactive Nuclear Wastes," *J. of Radiochemical and Nuclear Chemistry, Articles,* **123**, 149–66 (1988).

4. A. P. Toste, T. R. Pahl, R. B. Lucke, and R. B. Myers, "Analysis of Complex Organic Mixtures in Nuclear Wastes," pp. 133–50 in *24th Hanford Life Sciences Symposium: Health and Environmental Research on Complex Organic Mixtures,* ed. R. H. Gray, E. K. Chess, P. J. Mellinger, R. G. Riley, and D. L. Springer, Pacific Northwest Laboratory, Richland, Wash., 1987.

4. RADIOCHEMICAL AND INORGANIC ANALYTICAL SUPPORT FOR THE INACTIVE TANK CHARACTERIZATION

4.1 INTRODUCTION

Twenty-seven inactive waste storage tanks, currently managed by the Oak Ridge National Laboratory (ORNL) Remedial Action Program (RAP), were characterized in preparation for final closure/decommissioning to meet environmental, safety, and long-term surveillance objectives. The sampling of the tanks resulted in 86 samples that consisted of 65 liquid samples and 21 sludge samples. The analytical work for this task has been in progress for nearly a year. The analyses for this project have not been completed at this time, and this report includes only the data available through June 1989. This project has involved the cooperation and participation of groups from all sections of the Analytical Chemistry Division at ORNL. The organization, sample preparation, sample distribution, and the bulk of the analytical work were performed by the High Radiation Level Analytical Laboratory (HRLAL).

The goal of the inorganic analytical support for the inactive tank-sampling and characterization task is to provide the data necessary to meet both regulatory and engineering treatability requirements. The tanks are located in several areas throughout ORNL and contain radioactive waste collected from a large variety of programs and facilities over many years. The tanks have been inactive for periods of time ranging from about 1 to 30 years, and minimal information was available on the composition of materials within the tanks. Because of a lack of standard regulatory methods for the analysis of highly radioactive wastes and mixed wastes, the analytical support for this project was complicated by attempts to mimic existing regulatory procedures (SW-846,[1] U. S. EPA CLP,[2] and EPA-600[3]). With the complexity of the materials analyzed, the radioactivity, and the inappropriate regulatory procedures, this has been a difficult and challenging project.

Because of the nature of the material and the design of the containment vessels, it is not probable that representative samples were collected from the inactive storage tanks; consequently, the samples can be considered only as indicators of what might be found elsewhere in the tanks. Radiation fields up to 6.5 rad/h were measured at the access to the tanks. This procedure limited the number and type of samples (sludge samples have higher activities) collected. To meet shielding requirements, the tanks have been located underground with most transfer operations designed for remote handling. The tanks have very limited access, and additional sampling would require excavation in contaminated soil.

The composition and condition of several waste tank samples are illustrated in Figs. 4.1-4.3. A problem associated with obtaining a representative sample can be observed in Fig. 4.1 for tank WC17. This example illustrates the difficulty in taking samples at various depths. The three samples from the left were taken at the surface and at 1 and 6 in. below the surface, respectively. The organic material, which was a

ORNL PHOTO 07174-88

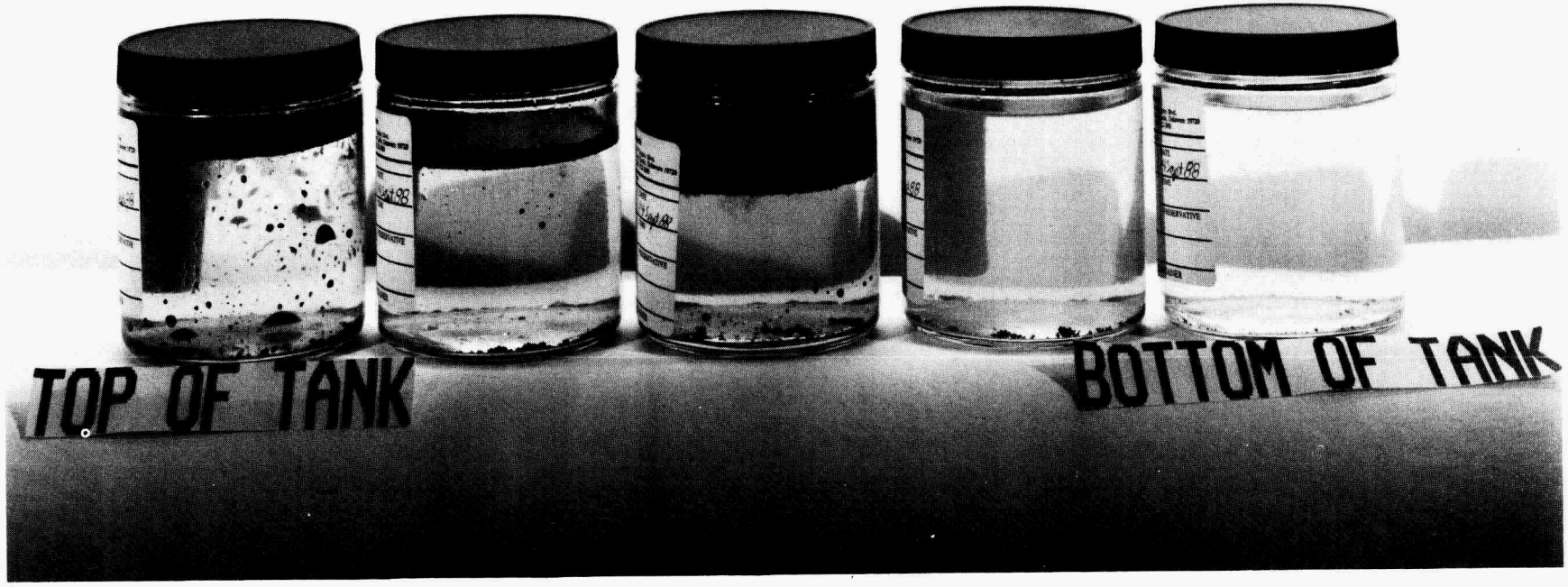


Fig. 4.1. Liquid samples from tank WC17 (Codes WC17/O101-O103 and WC17/L104-L105).

ORNL PHOTO 7173-88

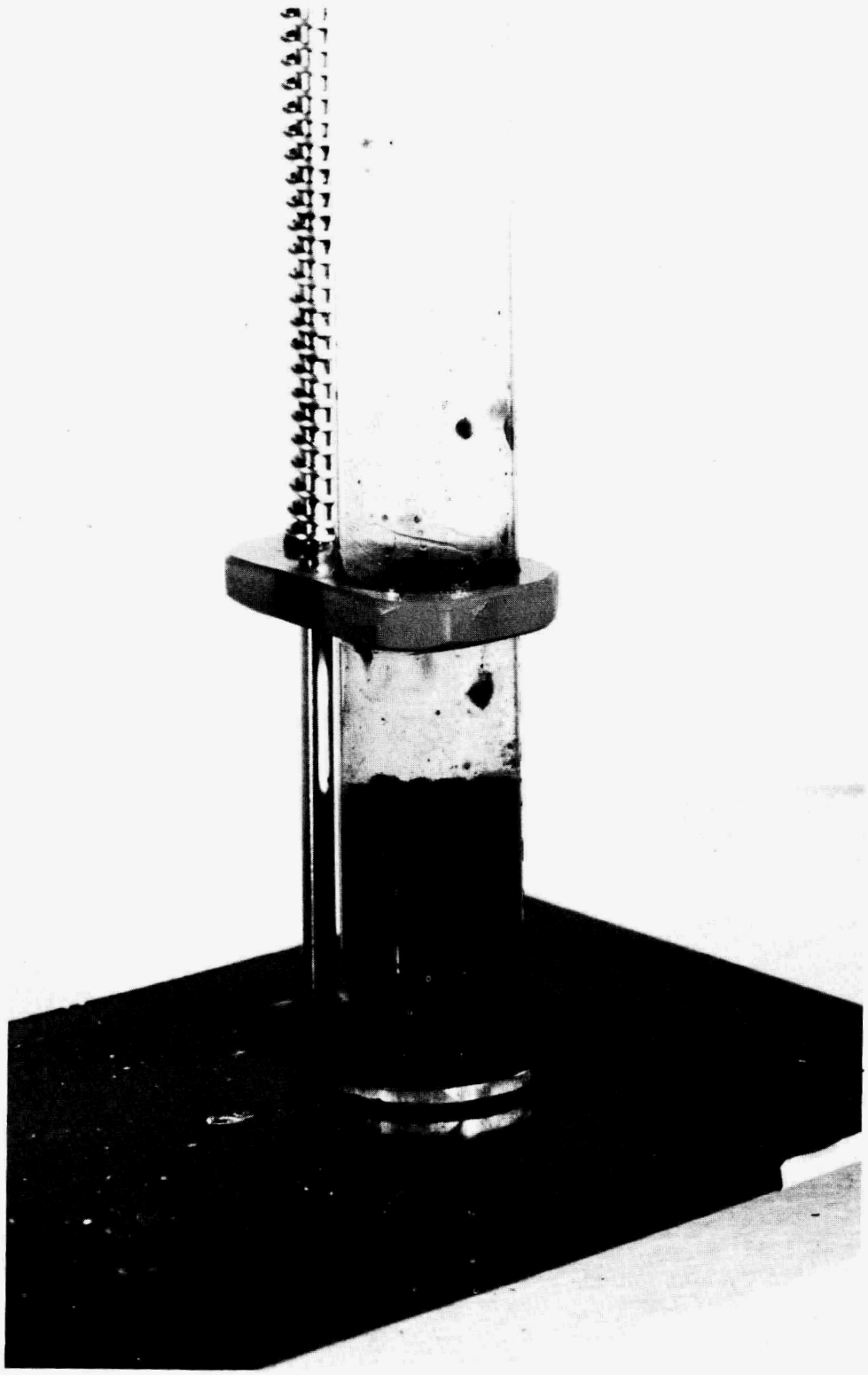


Fig. 4.2. Homogenous sludge sample from tank WC17 (Code WC17/S106).

ORNL PHOTO 6384-88

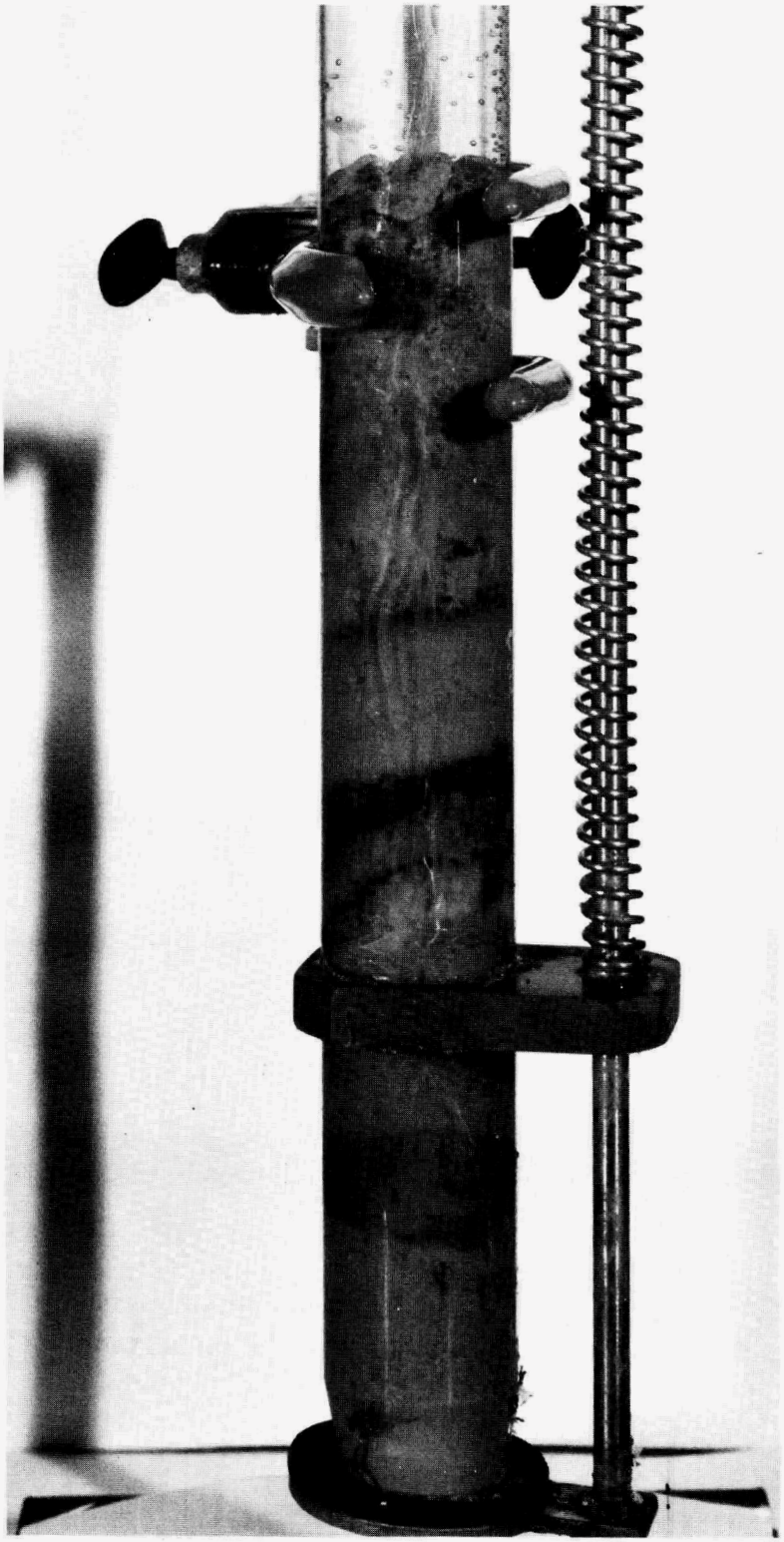


Fig. 4.3. Nonhomogenous sludge sample from tank TH4 (Code TH4/S059).

thin layer on the surface of the liquid, was present to a greater degree in the sample obtained at 6 in. than the sample taken at the surface. The last two samples in Fig. 4.1 were taken at 14 and 26 in. and did not contain the entrained organic material. The sludge layer from tank WC17, which is shown in Fig. 4.2, was fairly homogeneous.

An example of a nonhomogenous sludge, which is shown in Fig. 4.3, was taken from tank TH4. Not only are various bands, or striations, observed in this core sample, but also the solids appear to be more finely divided near the bottom of the sample. Another point of interest in this sample was the off-gassing, which can be observed at the solid-liquid interface. Although it has not been determined at this time, we can speculate that the gases may be the releases of radiolytic products or simply the releases of dissolved gases which resulted from the change in temperature between the waste tank and the laboratory.

Several miscellaneous deviations, not discussed under procedures, involve the initial handling of samples. Frequently the analytical methodology, as written for regulatory measurements, was not appropriate for characterizing the contents of radioactive waste tanks. It was then necessary to modify the regulatory procedures to (1) reduce radiation exposure to personnel to meet the policy of maintaining radiation exposure *As Low As Reasonably Achievable* (ALARA), (2) accommodate the limitations of the instrumentation and facilities available, and (3) handle the complex nature of the sample composition (e.g. high levels of salt, dissolved solids, uranium, and thorium) to obtain valid data. To reduce radiation exposure, sample-size requirements were reduced significantly for numerous measurements. The reduced sample sizes and instrumental limitations resulted in reduced sensitivities and other conflicts with some of the regulatory quality assurance/quality control (QA/QC) procedures that were too stringent to accommodate these problems. The deviations from regulatory QA/QC practices will be discussed for individual procedures under the topic of Analytical Methods.

First, the U. S. Environmental Protection Agency (EPA) sample-preservation procedures that involve maintenance of samples at 4°C were avoided because of the lack of refrigeration facilities for radioactive samples and to prevent shifts in the equilibrium states of solutions with elevated levels of both pH and dissolved solids. In addition, the on-site filtration upon collection and the on-site acid preservation of metals in aqueous samples were not performed because of the danger of handling radioactive materials. The EPA requirement that states that all samples must initially be run undiluted was not met for samples with high radiation levels or for techniques in which high-salt content interfered with the measurement. Finally, it was not possible to meet the holding-time requirements for mercury (26 d) and cyanide (14 d) because of the additional time necessary to analyze radioactive samples.

To meet the analytical requirements of this project, a new facility for atomic emission and atomic absorption spectroscopy was established. This facility included specially adapted instrumentation for Inductively Coupled Argon Plasma Atomic Emission Spectroscopy (ICP) and an Atomic Absorption (AA)

Spectrometer for graphite furnace (GFAA) work and mercury analysis by the cold-vapor (CV) technique. Each of these systems was set up and modified for operation in stainless steel enclosures. Both the ICP and the AA spectrometer were equipped with auxiliary exhaust systems to handle the bulk of radioactive aerosols generated during sample analysis. A Preoperational Readiness Review was conducted by the Radioactive Operations Committee on each of these systems prior to operation of these systems with radioactive samples.

The inactive waste-tank samples were inspected by a health physics representative, and a field survey was performed on each sample before it was packaged and delivered to HRLAL. Based upon the field survey, all liquid samples were handled in radiochemical laboratories (classified as radiation contamination zones). Most of the sludge samples required remote handling for dissolution and dilution before their removal from the hot cell and distribution to various laboratories. Sample volumes or weights were adjusted to comply with Appendix 7 of the ORNL Health Physics Manual. The limiting factors for laboratory operations were based upon the ^{90}Sr (Class 1; very high radiotoxicity) and ^{137}Cs (Class 2; high radiotoxicity) content. Most of the laboratory procedures were simple wet operations; this permitted the application of a 10-fold modifying factor to the activity levels allowed. Dose rates did not exceed 200-400 mrad/h at 1.5 in. for laboratory operations; the ^{90}Sr activity levels were maintained below 0.1-0.2 mCi, and the ^{137}Cs below 1-2 mCi. The only problem area involved the determination of total and dissolved solids. This required the weighing of the residue from dried samples. Special care and handling were exercised with the dry samples to prevent dispersing, or dusting, of the material.

The complex chemical composition of the waste-tank solutions was compounded by the effects of radiolysis.[4] To generate detectable levels of radiolytic products within a solution, large doses of radiation are required. If one considers the length of time the contents of the waste tanks have been exposed to radiation (1-30 years) and the intimate contact of the solutions with radioactive species, the cumulative dose the samples have received is sufficient to generate 0.01-0.1 M levels of radiolytic products. A basic observation in the irradiation of aqueous solutions with gamma-ray or beta-particle radiations is that the major chemical changes occur in the dissolved material. The changes result from free radical reactions with the solute species. Alpha radiation, on the other hand, leads to extensive decomposition of the water into hydrogen gas, hydrogen peroxide, and oxygen, all of which can go on to subsequent redox reactions with solute species. In addition, the alpha radiation can interact directly with the solute molecules and form numerous reactive ionic species. In general, we expected an oxidizing environment to exist in most waste-tank samples. However, depending upon the solution composition, reducing environments may also be possible.

Many of the samples obtained from the inactive waste tanks have high dissolved solids; this phenomenon translates into a high ionic strength. The effects of high ionic strength can have a significant effect on the solution chemistry and various chemical measurements. For example, a potentiometric pH measurement is actually based upon the hydronium ion activity and not the hydronium activity. The

hydronium activity will differ considerably at high ionic strengths. Therefore, the pH measurements on many of the tank samples can be considered only as apparent pH values that are not directly related to hydronium ion concentration. The ionic strength will also affect the kinetics of ionic reactions and result in deviations from solubility product behavior. In general, the solution chemistry is far from ideal, and the expected behavior for many species may not be observed.

4.2 SAMPLE PREPARATION

4.2.1 Radiochemical Sample Preparation

The sample preparation for radiochemical analysis requires a complete dissolution of the sample to ensure that all activity is measured. Approximately 20 mL of 8 M nitric acid was added to 1-2 grams of sample in a 100-mL beaker covered with a watch glass. The sample was then heated to boiling, and the heating was continued under mild reflux for 30 min. After cooling, 10 mL of 30% hydrogen peroxide was carefully added in 1-mL increments, followed by an additional 30 min of mild reflux. The sample was then cooled and filtered through No. 42 Whatman™ paper. Any residue present on the filter paper was checked for activity. If no activity was observed in the residue, the filtrate was diluted to 100 mL with distilled/deionized water and submitted for radiochemical analysis. If activity was observed on the filter paper, the residue and filter paper were carefully dried, ignited, and ashed in a platinum crucible. The ashed residue was then fused with 1-2 g of lithium metaborate, and the resulting melt was dissolved in 4 M nitric acid. The dissolved residue solution was then quantitatively transferred to the original filtrate, and the resulting solution was mixed well and built to volume with distilled/deionized water and submitted for radiochemical analysis.

4.2.2 Spectrochemical Sample Preparation (ICP and AA)

The acid-digestion procedures recommended by EPA for GFAA and ICP analytical techniques were not appropriate for the Inactive Tank liquid samples. The instrumentation for both GFAA and ICP are housed in stainless steel enclosures for containment of radioactivity and must be maintained in a relatively clean condition. Therefore, procedures which employ hydrochloric acid (used in EPA procedure for ICP analysis) or high levels of chloride are avoided to minimize a corrosive environment that would degrade the stainless steel enclosure. Initially a decision was made to use the acid-digestion procedure recommended for furnace AA analysis for both the ICP and GFAA work. The AA-digestion procedure employs only nitric acid and hydrogen peroxide for sample digestion. However, the oxidation step with hydrogen peroxide for many samples resulted in severe precipitation problems caused by high levels of thorium. The sample-preparation procedure finally chosen involved a simple dilution of liquid samples with 5% nitric acid.

The sample-preparation procedure recommended by EPA for the sludge samples did not display any obvious problems and was used as written. About 10 mL of 1:1 nitric acid was added to 1-2 g of sludge sample, which was then heated for 15 min at 95°C in a 50-mL beaker covered with a watch glass. The sample was allowed to cool; following a 5-mL addition of concentrated nitric acid, the sample was refluxed for 30 min. Then 1-mL increments of 30% hydrogen peroxide were added and warmed until effervescence ceased or a maximum of 10 mL was added. The sample was then cooled and filtered through No. 42 Whatman™ paper and diluted to 100 mL.

Additional preparation was required for many of the samples because of severe spectral/matrix interferences from high levels of uranium and thorium. The uranium and thorium levels were lowered to below 100 mg/L with a tri-*n*-octylphosphine oxide (TOPO) extraction.[5] The extraction procedure involved the addition of 5 mL of 4 M nitric acid to 5 mL of sample (either liquid sample or dissolved sludge) and then the acidified solution was extracted for 5 min with 10 mL of 0.1 M TOPO/cyclohexane. Then 5 mL of the aqueous phase was removed and diluted to 25 mL with 5% nitric acid (scandium was also added as an internal standard for ICP analysis). This procedure results in an additional 10-fold dilution that is free of uranium and thorium interference.

The following observations on the effects of a TOPO extraction on the Resource Conservation and Recovery Act (RCRA) metals were taken from the literature: Ag(I), As(V), Cr(III), Ba, and Ni are not extracted; Pb as the nitrate is not extracted from a nitric acid matrix; As(III), Cd, Hg(II) are slightly extracted from hydrochloric acid media but not from nitric acid; Cr(VI) is readily extracted from hydrochloric acid matrix but to a lesser extent from nitric acid.

As can be seen in Table 4.1, our experimental results on spike recoveries are in agreement with the above reported observations.

Table 4.1. Typical spike recoveries with TOPO extraction

Element	Spike recovery (%)	Relative standard deviation (%)
As	100	11
Ba	100	2
Cd	96	5
Cr	100	7
Hg	72	24
Ni	98	3
Pb	96	11
Se	100	9
Tl	86	25

4.2.3 Sludge-Sampling Procedure

Prior to taking a sample aliquot, it was necessary to homogenize the sludges to ensure a representative sample (although we have reservations concerning how well the sample represents the tank contents). All initial handling of the sludges was performed remotely in the hot cells. When a sludge sample was received, the sample was removed from a carrier and set up in a vertical position on a ring stand to permit settling of suspended particles and measurement of the settling time. After the sludge had settled, generally within 16-24 h, the supernatant was removed by aspiration from above the sludge layer, and then the retainer cap on the bottom of the sampler was removed to permit the sludge to drain out of the sampler. The soft sludges were first mixed with a stainless steel stirring paddle and then homogenized. A Branson sonicator was installed in a hot cell, and the sludges homogenized with the sonicator adjusted to a power level to provide vigorous agitation. In the case of very hard sludges, a weighed amount of sludge was diluted with a weighted amount of water and then sonicated to produce a soft, mixed slurry for measurement.

4.2.4 Sample Preparation for Mercury Analysis

In addition to inorganic forms of mercury, the EPA procedures suggest that organo-mercury compounds may be present which will not respond to the CV technique unless they are first broken down to the mercuric ions. Because of biological activity, this expectation is reasonable for environmental or sewage-type samples but is questionable for the type of waste found in the inactive tank samples. Based upon the operations and processes that employed the waste storage system, only elemental or inorganic mercury is likely to be present. As a result of the harsh environment (high or low pH, high salt, and radioactivity), it is unlikely that sufficient biological activity is present in the waste-storage tanks to convert inorganic mercury to organo-mercury compounds. Additional evidence to support the lack of biological activity is the absence of the organic acids associated with the metabolic pathways (such as the Kreb cycle) in the waste tank samples. Therefore, to reduce sample handling and radiation exposure to personnel the sample digestion steps to oxidize organo-mercury compounds to mercuric ions were not used.

In general, the presence of hydrogen peroxide, which results from radiolytic decomposition of water[4] in aqueous solutions, provides an oxidizing environment which should tend to stabilize the mercury in radioactive solutions. Hydrogen peroxide is a stronger oxidizing agent in alkaline solutions than in acidic solutions and may not have sufficient oxidizing potential to stabilize the mercury in the acidic waste tanks. Potassium permanganate was used to evaluate the reducing power of the waste-tank samples before mercury measurements were taken. The samples were tested by observing the decolorization following the addition of several drops of 5% potassium permanganate. Most of the samples retained the purple color; however, several of the liquid samples consumed the permanganate. This reaction indicated a possible reducing environment. The reducing species oxidized by the permanganate have not been identified at this time. The

sludge samples, which were prepared for spectrochemical analysis by the EPA GFAA dissolution procedure, were used for the mercury measurements. Therefore, the sludge samples were in an oxidizing environment, which resulted from the nitric acid and hydrogen peroxide used in the sample preparation. Consequently, none of the sludge samples decolorized the permanganate. In contrast, the liquid samples were analyzed directly, without prior treatment, which provided an indication of the actual environment present in the waste tanks.

4.2.5 Extraction Procedure Toxicity Test (EP-TOX): Method 1310

A representative sample of waste containing greater that 0.5% solids must be extracted with deionized water, which is maintained within a pH range of 4.8 to 5.2 with acetic acid. This requirement applies to all sludge samples and may apply to some of the liquid samples, especially the organic layers found in tanks WC17 and 7860A. A minimum of 100 g defines a representative sample for this procedure; however, because of the high radiotoxicity associated with many of the sludge samples and the lack of sufficient sample available for testing, it was necessary to scale down the procedure by a factor of 10. Therefore, a 10-g sample was extracted with 200 mL (Method 1310 states 100 g/2000 mL). With the exception of the sample-size deviation, the EP-TOX procedure was followed as written.

During the initial planning of the Inactive Tank Characterization project, it was decided to demonstrate that the EP-TOX procedure was not required if the total metal measurement did not exceed the EP-TOX equivalent limits. Because of a 20-fold dilution factor during the extraction procedure, the EP-TOX equivalent limit is defined as 20 times the EPA regulatory limits (Table 4.2). However, the total metal measurements for the sludge samples yielded results that exceeded the EP-TOX equivalent limits for chromium, lead, and mercury in most of the sludge samples. Therefore, the EP-TOX procedure was required for all sludge samples, but with remeasurement of only the RCRA metals that exceeded the EP-TOX equivalent limit.

4.3 ANALYTICAL METHODS

The analytical methods employed for the characterization of the inactive tank samples are summarized in Table 4.3. An expanded discussion of procedures required for regulatory measurements is provided to inform the reader of deviations from EPA methods and QA/QC requirements. In addition, any unusual occurrences observed during sample analysis or measurements requiring explanations necessary for interpretation of the data are discussed.

Table 4.2. RCRA metal regulatory limits for solid waste

Element	(Liquid) Regulatory limit (mg/L)	(Sludge) EP-TOX equivalent (mg/kg)
As	5.0	100
Ba	100	2000
Cd	1.0	20
Cr	5.0	100
Pb	5.0	100
Hg	0.2	4
Se	1.0	20
Ag	5.0	100
Ni	50	1000
Tl	0.9	18
pH	<2 or >12	

4.3.1 RCRA Metal Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES)

A Perkin-Elmer XR 6500 ICP was modified for use with radioactive samples by isolating the ICP torch box in a stainless steel enclosure for containment of radioactivity (Figs. 4.4 and 4.5). The enclosure is provided with sliding glass doors and ports, which may be gloved if higher levels of radioactivity must be handled. The performance of the system with the separation of the torch box and monochromator was evaluated and found to be comparable with normal operation.

The Plant and Equipment (P&E) ICP, used for the analysis of the tank samples, has a poor dynamic range relative to most other ICP systems. Therefore, the narrow calibration range resulted in numerous measurements exceeding the high standard of the calibration curve. When a measurement exceeded the calibration range, several dilutions were usually required to obtain a valid result. With the eight RCRA metals plus nickel and thallium, the additional dilutions and sample handling resulted in a drastic increase in the analysis time and radiation exposure to the analyst.

A Perkin-Elmer Cross-Flow, a Hildebrand Grid, and a Meinhard Type C nebulizer were evaluated for operation and effectiveness with samples containing high dissolved solids content while maintaining acceptable standard deviations. The Meinhard Type C nebulizer, which is similar to a standard glass concentric type but has the inner capillary recessed from the tip to improve performance with samples containing high solids, was chosen for measurements on the inactive tank samples. In addition, a mass-flow controller was added to the nebulizer gas flow to improve precision of the ICP measurements.

The initial liquid tank samples were analyzed by the method of standard addition (MSA), which proved to be highly wasteful of the limited sample volumes available. In addition, the MSA generated larger

Table 4.3. Analytical methods

Parameter	Reference	Method summary	Approximate detection limit
		Radiochemical procedures	
Gross alpha	ACD 9 002301, EPA-600-900.0	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	None	Sample is dissolved, is 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	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 re-analyzed.	Varies with energy
^{241}Am ^{244}Cm	ACD 2 31621	Sample is dissolved, if necessary. After a extraction procedure with thenoyltrifluoroactone (TTA) -xylene, the sample is dried on a stainless steel planchet and analyzed by alpha spectroscopy (ACD 1 003115).	3 pCi/L
^{232}U, ^{233}U, ^{235}U, ^{238}U	ACD 2 31926	Sample is dissolved, if necessary. Uranium isotopes are extracted with methyl isobutyl ketone (hexone) using aluminium nitrate as a salting agent; then the sample is dried on a stainless steel planchet and analyzed by alpha spectroscopy (ACD 1 003115).	0.04 pCi/L
^{238}Pu, ^{239}Pu, ^{240}Pu	ACD 2 31621	Sample is dissolved, if necessary. After a chemical valence adjustment, the sample is extracted with TTA-xylene, dried on a stainless steel planchet, and analyzed by alpha spectroscopy (ACD 1 003115).	0.04 pCi/L
^{90}Sr	EPA-600-905.0	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
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
^{14}C	ACD 2 0952	Aqueous solutions are acidified with sulfuric acid, and the CO_2 is distilled into a hyamine solution, a portion of which is counted on a scintillation counter.	5 pCi/mL
^{99}Tc	ACD 5 118/3	Technetium is first separated from other fission products by an oxidation step followed by extraction of the pertechnetate anion. The ^{99}Tc is then stripped into water and loaded onto an anion exchange resin. The ^{99}Tc content is determined by neutron activation analysis of the resin column.	
^{129}I	ACD 2 21393	Iodine activity is separated from other fission products by oxidation to periodate with sodium hypochlorite in basic solution, followed by reduction to iodine with hydroxylamine hydrochloride and extraction of iodine into carbon tetrachloride. The iodine is stripped into an aqueous solution of potassium metabisulfate, which reduces the iodine to iodide. The iodide is loaded onto an anion exchange resin which is dried and analyzed by neutron activation. The induced ^{130}I activity is measured along with added ^{131}I activity for yield corrections.	

Table 4.3. continued

Parameter	Reference	Method summary			Approximate detection limit
		Inorganic analytical procedures			
Metals by ICP	EPA 200.7/ SW-846-6010	Liquid samples or dissolved sludges are nebulized, and the aerosol that is produced is transported to a argon plasma torch where excitation occurs. Characteristic atomic-line spectra are produced by an (ICP); the intensity of the emission lines from the excited elements is measured with a computer controlled scanning monochromator (Perkin-Elmer 6500 ICP). The measured intensity is compared to stored calibration curves for quantitation.			
		Wavelength (nm)			
Ag		328.068			69 ng/mL
As		197.197			265 ng/mL
Ba		233.527			13 ng/mL
Cd		226.502			19 ng/mL
Cr		205.552			35 ng/mL
Ni		231.604			56 ng/mL
Pb		220.353			144 ng/mL
Se		196.026			312 ng/mL
Tl		190.864			177 ng/mL
Al		308.215			45 ng/mL
B		249.773			5 ng/mL
Ca		317.933			10 ng/mL
Fe		259.940			7 ng/mL
K		766.491			
Mg		279.079			30 ng/mL
Na		588.995			29 ng/mL
Th					
Zn		213.856			2 ng/mL
Metals by GFAA	SW-846-7000	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 the 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.			
		Wavelength(nm)	Pyrolysis (°C)	Atomization (°C)	
Ag	EPA 272.2	328.1	650	1600	0.16 ng/mL
As	EPA 206.2	193.7	1300	2300	11.4 ng/mL
Ba	EPA 208.2	553.6	1200	2550	15.6 ng/mL
Cd	EPA 213.2	228.8	700	1600	0.13 ng/mL
Cr	EPA 218.2	357.9	1650	2500	1.32 ng/mL
Ni	EPA 249.2	232.0	1400	2500	3.55 ng/mL
Pb	EPA 239.2	283.3	850	1800	1.24 ng/mL
Se	EPA 270.2	196.0	900	2100	8.80 ng/mL
Tl	EPA 279.2	276.8	600	1300	9.40 ng/mL
Cs	none	852.1	900	1900	

Table 4.3. continued

Parameter	Reference	Method summary	Approximate detection limit
Metals by spark source mass spectrometry	ACD 4 0200	Approximately 50 elements are determined simultaneously by sparking a sample which was dried on a graphite electrode in the source of a mass spectrometer. This technique was not used to report data, but to provide screening or ICP and GFAA analysis.	Depends on the metal, but is generally in the ppm range; analysis. precision is about a factor of 10.
Mercury by CV technique	CLP-M 245.1	The flameless 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 area) is measured as a function of concentration.	1-2 ng/mL for a 1-mL sample size
Soluble silica	EPA-600/370.1	The silica in a filtered sample is converted to molybdosilicic acid which is reduced by 1-amino-2-naphthol-4-sulfonic acid to hetropoly blue. The color is measured at 815 nm or 650 nm, and the signal is compared to a calibration curve for quantitation.	1 mg/L for a 1-mL sample size
Total uranium	ACD 1 219240	Uranium is extracted from 3-M HNO_3 solutions with trioctylphosphine oxide (TOPO); an aliquot of the organic extractant is pipetted onto pellets of sodium fluoride which is dried and sintered. The fluorescence due to the measured uranium is measured on a fluorophotometer.	
Anions by ion chromatography		Samples, diluted if necessary, are injected directly into an ion chromatograph, and the resultant chromatogram is analyzed to identify anions and determine concentration.	
F^-	EPA300.0		1 mg/L
Cl^-	EPA300.0		1 mg/L
PO_4^{-2}	EPA300.0		5 mg/L
NO_3^-	EPA300.0		5 mg/L
SO_4^{-2}	EPA300.0		5 mg/L
pH	SW-846-9040	The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference indicating electrode. 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^-)	ACD 1 003105	The OH^-, CO_3^{-2}, HCO_3^-, and H^+ were all determined by potentiometric titration with either 0.1 N HCl or 0.1 N NaOH. The change in potential as a function of reagent consumption was recorded with an automatic potentiograph.	0.01 N
Cyanide (CN^-)	SW-846-9010	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 manually determined colorimetrically.	0.5 mg/L for a 10 ml sample size
Sulfide (S^{-2})	SW-846-9030	An aliquot of the absorbing solution from the cyanide analysis is treated with iodine to oxidize the sulfide to sulfur under acidic conditions. The excess iodine is back-titrated with phenylarsine oxide.	2 mg/L for a 10 ml sample size
Total Organic Carbon (TOC)	SW-846-9060	The organic carbon in a sample is converted to carbon dioxide (CO_2) by a catalytic combustion. The CO_2 is then measured directly by an infrared detector.	1 mg/L

Table 4.3. continued

Parameter	Reference	Method summary	Approximate detection limit
		Physical measurement procedures	
Total solids	EPA-600/160.3	A well-mixed aliquot of the sample is quantitatively transferred to a preweighted evaporating dish and evaporate to dryness at 103-105°C for 16 h.	10 mg/L
Dissolved solids	EPA-600/160.2	A well-mixed sample is filtered and the filtrate quantitatively transferred to a pre-weighed evaporating dish and evaporate to dryness at 103-105°C for 16 h.	4 mg/L
Suspended solids	EPA-600/160.2	The suspended solids were calculated by subtracting the dissolved solids from the total solids.	4 mg/L
Density	ACD 1 1011	A measured volume of sample is weighed at ambient temperature.	

ORNL PHOTO 6720-87

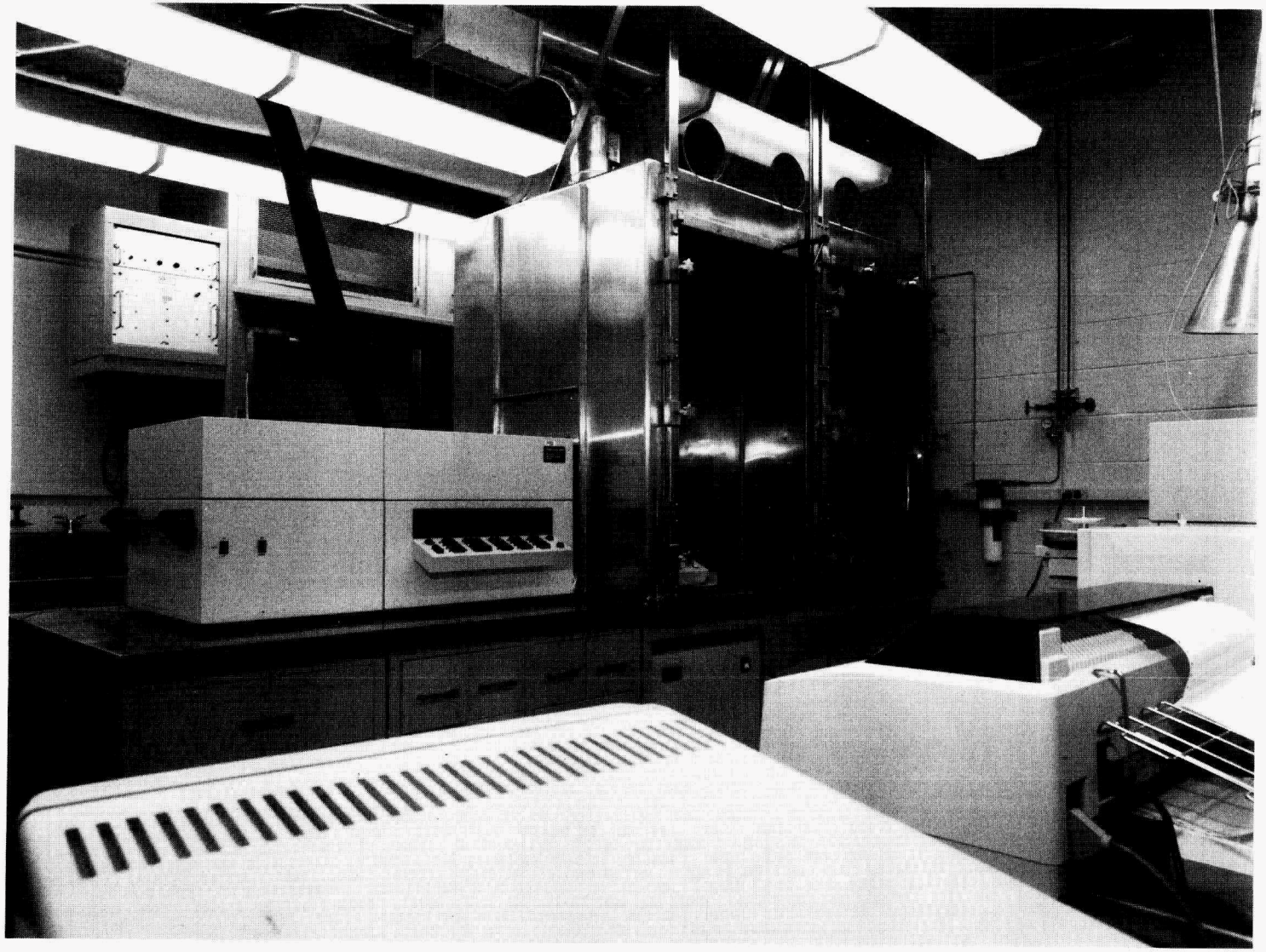


Fig. 4.4. Perkin-Elmer Model 6500 ICP modified for radioactive work.

ORNL PHOTO 6722-87

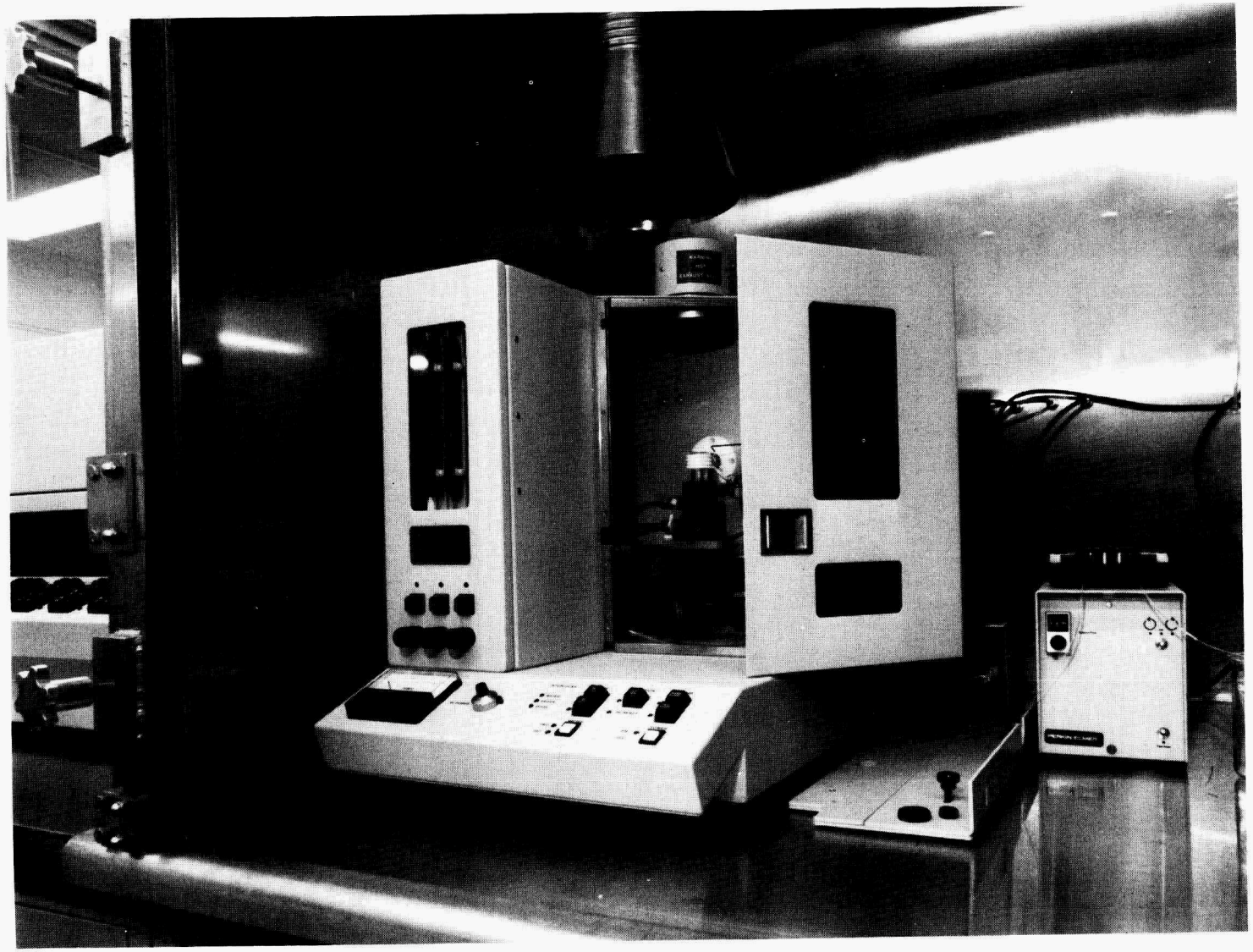


Fig. 4.5. Close-up of torch box for ICP system.

sample volumes; this in turn resulted in unnecessary radiation exposure to the analyst. After testing several batches of samples, it was found that the data obtained with a standard calibration curve were comparable to the MSA technique. Therefore, the remaining tank samples were analyzed with a calibration curve and appropriate QA checks of data reliability.

QC was applied to batches of tank samples, where a batch was defined by grouping samples with similar matrices based upon the screening results obtained for each sample with a spark source mass spectrometer (SSMS). The initial segregation of the samples was based upon the criteria of (1) samples with high uranium and thorium and (2) samples with low uranium and thorium (<100 mg/L). Because of the variability observed in the sample matrix composition, each sample was measured along with a spiked sample (provided enough sample was available) to monitor matrix effects and interferences. All samples were also analyzed with an internal standard (scandium) to compensate for matrix differences on the nebulization of the sample.

Batch acceptance criteria for the ICP measurements were based upon several factors. The percent relative difference (PRD) for the calibration check standard (CCS) was required to be within the range of ±20% of the known standard value. The PRD for duplicates and serial dilutions were required to be within the range of ±20% for analytes whose concentration exceeded ten times the instrument detection limit (IDL). Spike recoveries were required to be within the range of 75 to 125%. If an analyte spike recovery was outside this range, the analytical line used was inspected graphically, and a hard copy saved with the original sample data. The PRD for an interference check standard was required to be within the range of ±25% of the known standard concentration. The relative errors for analytes at or near the IDL were not used for batch acceptance. The calibration blank and reagent blanks were monitored to check for contamination of the reagent and/or the sample introduction system. It was up to the discretion of the analyst to determine if there was a problem with the blank measurements and take the necessary action to correct.

4.3.2 RCRA Metal Analysis by GFAA

The instrumentation used for the GFAA measurements was a Perkin-Elmer 5100 Atomic Absorption System with the HGA-600 furnace option with Zeeman-effect background correction. The spectrometer was placed in a stainless steel enclosure (Fig. 4.6), similar to the ICP system, to permit measurements on radioactive samples. The technique used for all GFAA samples was an approach referred to as the stabilized temperature platform furnace (STPF)[6] concept, which was developed by Perkin-Elmer Corporation. The STPF approach is based on the use of the L'vov platform, fast electronics, quantitation by peak area, matrix modifiers, pyrolytically coated graphite tubes, fast heating of furnace, no flow of support gas during the atomization step, and Zeeman-effect background correction. Based on recent literature, an additional step

ORNL PHOTO 7175-88

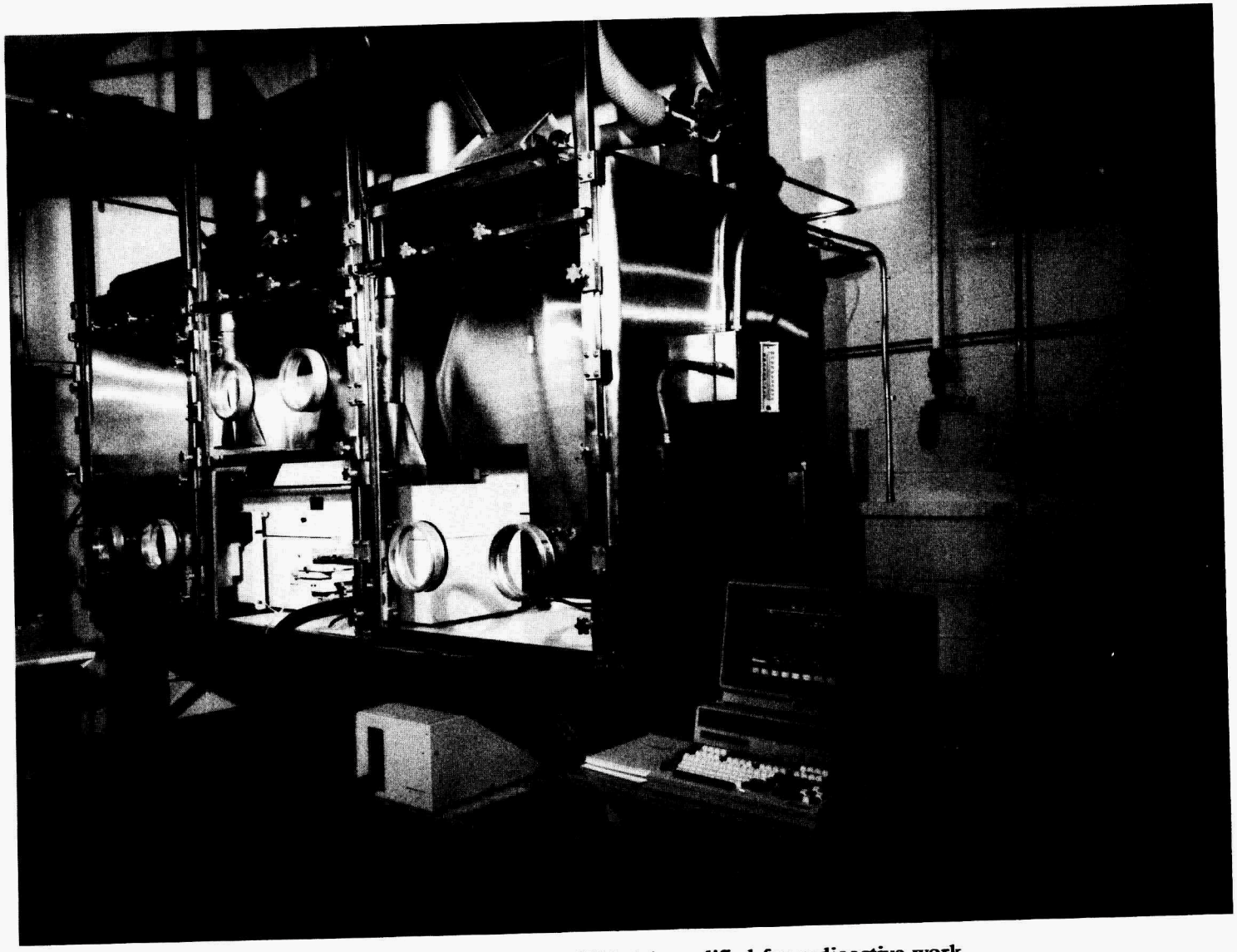


Fig. 4.6. Perkin-Elmer Model 5500 AA modified for radioactive work.

was added to the typical furnace temperature program that consists of a cool-down step.[7] It was reported that for some samples, better results were obtained if the furnace was allowed to cool down to ambient temperature before the atomization step. Also reported was that for some real samples the use of a cool-down step reduced or completely eliminated interferences that could not be controlled otherwise. Therefore, the furnace temperature program for all GFAA measurements included a cool-down step before atomization.

The analytical approach developed for GFAA measurements provided adequate QA/QC and followed the ALARA principle by minimizing the radiation exposure of the analyst. Instrument calibration consisted of constructing calibration curves from the average absorbance values, obtained with double injections of a blank and 3-4 standards vs the known concentrations. The concentration of the standards was chosen to be within the optimum range, as listed by the EPA GFAA procedures.[2] Calibration verification, which followed the generation of the calibration curve, consisted of measurements for a calibration blank (CB) and a CCS. The tolerance limits for CB and CCS have not been set at this time because of a lack of enough data to statistically define the limits. Additional QC data were obtained for CB and CCS at a frequency such that no more than five samples were analyzed between calibration verification samples. For each batch of samples (4-5 samples), an additional divisional QC sample was measured for the elements of interest.

The analysis of samples, which followed the initial calibration verification, included sample and spike measurements, each with double injections from which the average concentrations were reported or employed for spike recovery calculations. With the double injection for samples and spikes, the analysis of 5 samples implied 20 furnace injections between QC samples. For concentrations greater than 5 times the IDL, the duplicate sample measurement should have agreed within a 30% relative standard deviation (RSD); otherwise, the sample was rerun once. If the RSD still exceeded 30%, the sample was flagged. If the spike recoveries were within the range of 75 to 125% the data were quantitated directly from the calibration curve and reported to the IDL. If spike recoveries were outside the range, the sample was flagged for a more detailed examination at a later date. Up to this point in the analysis scheme, details are similar to EPA CLP[2] procedures with the exception of the broader spike recovery range and duplicate RSD. The EPA CLP[2] procedure specifies analysis by MSA when spike recoveries are poor. However, for this work the MSA technique was not used because of increased sample handling, which would increase the radiation exposure to the analyst. Because this project has not been completed at this time, it may be necessary to use the MSA technique to verify and document tanks that are identified as non-RCRA during the current phase of the project.

Quite frequently an obvious reason exists for the poor spike recoveries. To illustrate, let us consider the precipitation of barium and lead in samples with elevated levels of carbonate, phosphate, or sulfate. Similarly, the loss of silver in samples with high chloride or phosphate are other good examples. The presence of high levels of uranium also resulted in poor spike recoveries for many of the elements of

interest, but the mechanism of this problem seems to be an interference with the atomization process rather than a chemical interference. The samples with uranium levels in excess of 100 mg/L were TOPO extracted to reduce this interference; however, the extraction process introduced some additional error to the measurements.

4.3.3 Mercury Analysis by CV Technique

The instrumentation for the mercury CV technique was a MHS-20 (Mercury-Hydride System) attachment for the Perkin-Elmer 5100 AA system. The MHS-20 includes a heated 10-cm quartz cell, which is placed in the flame AA light path of the 5100 system; a sample-handling system; and a system controller for programing the addition of reductant and reaction times. The signal from the spectrometer is then processed by the 5100 system computer, and peak areas are used for quantitation.

4.3.4 Cyanide and Sulfide Analysis

The regulatory limits for cyanide and sulfide in solid waste are defined under the characteristic of reactivity and are based upon the release of toxic gases when exposed to pH conditions between 2 and 12.5. The current EPA action level for cyanide is 250 mg/kg; for sulfides, 500 mg/kg. The procedure recommended by EPA (SW-846) for the determination of total available cyanide and sulfide involves a nitrogen purge of a 10-g sample from an acidic solution to evolve the hydrogen cyanide and sulfide, which are collected in a basic scrubber solution. The amount of cyanide is then determined by Method 9010, and the amount of sulfide, by Method 9030.

A procedure for the measurement of total cyanide and sulfide, which exceeds the regulatory requirements and was already in place for the total measurement, was used for this project. The total cyanide and sulfide determination uses a distillation process (in contrast to purge technique recommended in SW-846) in which the detection limit is dependent upon the weight of sample taken for analysis. To reduce personnel radiation exposure, the sample volume recommended by EPA (100 mL) for liquid samples was reduced by a factor of 10. The reduced sample volume resulted in a decrease of the overall sensitivity; however, the decreased sensitivity still exceeded the regulatory limits for solid waste. The elevated levels of nitrate required all samples to be pretreated with sulfamic acid[1,2] to ensure elimination of a possible positive interference, which results from the decomposition of oximes to generate hydrogen cyanide. A Wheaton cyanide still was used to drive the cyanide and sulfide from an acidic solution into a basic scrubber solution. Because sulfide adversely affects the colorimetric procedure for cyanide, an aliquot of the distillate was first analyzed for sulfide. The determination of sulfide consisted of the addition of a standard iodine solution to an aliquot of the distillate, which was back-titrated with phenylarsine oxide to determine the excess iodine. If sulfide was present, an aliquot taken for cyanide analysis was first treated with cadmium to precipitate the sulfide. The cyanide concentration was determined by a colorimetric procedure that closely followed the EPA Method 9010.

Although the determination of sulfide approximated the regulatory procedures for this project, there appeared to be some interferences from oxidizing agents (radiolysis can result in free chlorine or oxides of nitrogen which will oxidize the iodide to iodine and give a low result) that distilled with the hydrogen sulfide. Additional development is needed to improve the sulfide measurement for complex radioactive waste samples.

It should be noted that the EPA preservation requirements, which include pH adjustment to a value greater than or equal to 12 and refrigeration of samples to stabilize the sample, were not satisfied. Also, there was no attempt to meet the holding-time (14-d) requirement for the cyanide analysis.

4.4 ANALYTICAL RESULTS

The physical, inorganic, and radiochemical analyses were conducted by three facilities: the HRLAL (Bldg. 2026) and the Transuranium Laboratory (TRU, Bldg. 7920), which are in the Radioactive Materials Analysis Section, and the Chemical and Physical Analysis Laboratory (CPA, Bldg. 4500S), which is in the Inorganic Chemistry Section. The sample receiving, sample distribution, sample preparation, physical measurements, metal analyses, cyanide and sulfide analyses, total organic carbon (TOC), ^{14}C and ^{3}H analyses, and some of the other radiochemical measurement were performed by HRLAL. The general anion (F^-, Cl^-, PO_4^{-2}, NO_3^-, and SO_4^{-2}) measurements by ion chromatography were performed by the CPA group. The bulk of the radiochemical data, which include the gross alpha, gross beta, gamma emitters, ^{90}Sr, and alpha emitters, was provided by the TRU laboratory. In addition, SSMS data were provided on each sample by a group from the Analytical Spectroscopy Section located at HRLAL. Because of the complexity of the chemical composition for the inactive tank samples, the SSMS technique was employed as a screening tool to provide qualitative information, which was used to alert the analyst to possible elemental and spectral interferences in the variety of different analytical procedures employed.

The inactive tank sample data are presented in three tables: Table 4.4 physical and metal data, Table 4.5 anion data, and Table 4.6 radiochemical data. Each table includes the tank sample identification number and the depth (inches) at which the sample was collected. The sample identification numbers include the tank identification (i.e., T1, T2, WC17, etc.) followed by a slash, a phase designation (L–liquid, S–soft sludge, H–hard sludge, and O–organic), and an additional number to uniquely distinguish the sample. The pH is included in each table for convenience and interpretation of trends that follow the hydrogen (H^+) or hydroxide (OH^-) ion concentration. Each set of tank samples is grouped by increasing depth with the sludge sample(s) at the end of a group. Included within these data tables are three blind duplicates (T2/L38 and T2/L112, W11/L3 and W11/L115, W2/L11 and W2/L118) and a blind sample blank (TH3/L113) that was distilled deionized water. An attempt was made to organize the data in a manner such that interrelationships between the different parameters may be observed and trends identified.

Table 4.4. Physical and metal data for inactive tank samples

Sample ID	Depth (in.)	Density (g/mL)	Suspended solids (mg/mL)	Dissolved solids (mg/mL)	Tot. Sol. L:(mg/mL) S:(mg/g)	TOC L:(mg/L) S:(mg/kg)	Soluble Si (mg/L)	Metals (Liquid: mg/L; sludge: mg/kg)											
								U	Ag	As	Ba	Cd	Cr	Hg	Ni	Pb	Se	Tl	pH
T4/L111	0	1.016	0.3	23.6	23.9	460	1.96	23.3	0.018	<0.4	<0.02	<0.01	13*	7.9	<0.1	<0.5	<0.23	<0.23	11.7
T4/L44	4	1.020	2.7	23.6	26.3	460	1.96	25.7	0.017	<0.8	<0.04	<0.02	9.4	1.1	<0.2	<1	<0.09	<0.09	11.7
T4/L45	34	1.016	<0.1	23.7	23.4	473	1.45	27.8	0.02	<0.8	<0.04	<0.02	14	2.7	<0.2	<1	<0.09	<0.09	11.7
T4/S46	NA	1.235	NA	NA	253	4620	NA	1850	(1.7)	<4	<50	10	(102)	585	160	(510)	(1.5)	0.73	NA
T3/L42	5	1.035	1.5	51.9	53.4	12600	77.1	0.2	<0.01	0.4	<0.02	<0.01	14	5.7	<0.1	<0.5	<0.5	<0.5	12.7
T3/S43	NA	1.930	NA	NA	364	9140	NA	3060	0.15	<3	76	8.5	(69)	40	57	(300)	(0.74)	<0.6	NA
T9/L47	6.5	1.034	0.7	41.5	42.2	850	9.76	852	0.01	<0.8	0.12	<0.02	0.4	3.4	<0.2	<1	<0.09	<0.09	9.1
T9/S48	NA	NA	NA	NA	343	7620	NA	2930	0.21	<2	115	7.8	(<10)	39	380	(540)	(<2)	<2	NA
T2/L38	4	1.013	1.1	11.4	12.5	1120	5.07	166	<0.002	<0.8	<0.04	<0.02	0.44	0.1	<0.2	<1	<0.09	<0.09	9.9
T2/L112	4	1.008	0.6	11.5	12.1	1100	6.81	161	<0.002	<0.8	0.06	<0.02	<0.1	0.1	<0.2	<1	<0.09	<0.09	9.9
T2/L39	48	1.013	0.9	10.7	11.6	1310	6.97	158	0.002	<0.8	<0.04	<0.02	<0.1	0.15	<0.2	<1	<0.09	<0.09	9.9
T2/S40	NA	NA	NA	NA	324	28000	NA	1000	2.9	<1	33	6.6	(180)	70	72	(350)	(<1)	<1	NA
T1/L35	4	1.005	0.8	6.4	7.2	836.0	9.34	172	0.005	<0.8	<0.04	<0.02	0.29	0.06	<0.2	<1	<0.2	<0.2	9.7
T1/L36	48	1.007	<0.1	6.9	6.9	790.0	6.81	175	0.005	<0.8	0.05	<0.02	0.18	0.07	<0.2	<1	<0.2	<0.2	9.7
T1/S37	NA	NA	NA	NA	921	18600	NA	2800	2.1	<2	88	12.9	(130)	74	190	(860)	(<2)	1.7	NA
TH1/L49	4	1.034	<0.1	44.4	41.3	26.0	35.3	0.5	0.003	<4	<0.2	<0.2	1.2	16	1.5	<2	<0.09	<0.09	1.8
TH1/L50	10	1.034	<0.1	43.6	35.5	22.0	38.3	0.4	0.005	<4	0.23	<0.2	1.4	16	1.7	2.2	<0.09	<0.09	1.8
TH3/L52	2.5	1.015	<0.1	18.0	15.1	6.1	15.7	<0.1	<0.002	<2.1	0.89	<0.08	2.2	11	2.6	1.9	<0.09	<0.09	1.8
TH3/L53	5	1.015	<0.1	18.6	15.6	5.1	18.2	<0.1	<0.01	<2.1	0.86	<0.08	2.2	9	2.5	2.2	<0.5	<0.5	1.8
TH3/L113	NA	1.000	0.1	<0.1	0.1	0.8	3.23	<0.1	<0.002	<2	<0.1	<0.08	<0.1	<0.01	<0.2	<1.4	<0.09	<0.09	6.6
W11/L3	4	0.999	0.2	0.1	0.3	2.0	6.91	12.1	<0.002	<0.8	<0.04	<0.02	<0.1	<0.04	<0.2	<1	<0.09	(<0.09)	8.0
W11/L115	4	0.998	0.2	0.1	0.3	<0.1	7.03	8.88	(<0.002)	<0.8	<0.04	<0.02	<0.1	<0.01	<0.2	<1	<0.09	(<0.09)	7.8
W11/L114	24	1.000	0.2	0.1	0.3	<0.1	7.29	8.82	<0.002	<0.8	0.05	<0.02	<0.1	<0.01	<0.2	<1	<0.09	(<0.09)	7.7
W11/H5	NA	2.080	NA	NA	1200		NA		2.4	(1.8)	64	(1.2)	670	17	10	(14000)	<0.3	<0.3	NA
TH4/L116	0	1.000	<0.1	<0.1	<0.1	14.6	0.57	0.84	<0.002	<2	<0.02	<0.06	<0.1	<0.01	<0.1	<2	<0.1	<0.09	7.1
TH4/L55	4	1.003	0.1	1.4	1.5	19.4	0.95	15.3	<0.002	<2	<0.02	<0.06	<0.1	<0.01	<0.1	<2	<0.1	<0.09	6.9
TH4/L117	6+	1.007	0.1	0.5	0.6	31.6	1.52	24		<5	<0.3	<0.2	<0.4	<0.05	<0.5	<4	<3	<4	6.9
TH4/L56	39	1.056	<0.1	70.6	70.6	352	3.3	7230	<0.002	<4	<0.2	<0.2	1.6	0.25	<1	<2	<0.1	<0.09	7.1
TH4/L57	72	1.083	0.3	103.8	104.1	2170	2.66	16700	<0.005	<10	<0.6	<0.5	5.6	0.04	<3	<5	<0.2	<0.2	7.8
TH4/S58	NA	NA	NA	NA	256	6940	NA	4530	0.068	(4.6)	5.6	<0.3	(72)	3.6	16	38	(<0.4)	<0.4	NA
TH4/S59	NA	NA	NA	NA	405	7530	NA	2590	0.15	(2.1)	(12)	0.37	(290)	4.9	20	99	(<0.2)	<0.2	NA
W5/L73	4	1.044	0.7	47.5	48.2	700	12.9	296	<0.002	<4	<0.2	<0.08	2.7	0.41	<1	<2	<0.09	<0.09	10.6
W5/S75	NA	NA	NA	NA	348	4020	NA	1420	0.26	<1	140	10	(580)	18	52	388	(0.81)	(<0.5)	NA
W6/L77	4	0.999	<0.1	2.5	2.5	2.0	3.99	16.2	<0.002	<4	<0.2	<0.08	<0.3	<0.01	<1	<2	<0.09	<0.09	8.7
W6/L78	30	1.009	<0.1	14.8	14.7	2.0	2.98	52.3	<0.002	<4	<0.2	<0.08	2.6	<0.01	<1	<2	<0.09	<0.09	10.4
W6/L79	54	1.059	0.1	74.0	74.1	180	1.65	69.8	<0.002	<4	<0.2	<0.08	32	0.07	1.5	<2	<0.09	<0.09	11.8
W6/S80	NA	NA	NA	NA	406	9110	NA	6340	0.006	(<2)	(350)	<1	(2400)	36	85	1100	(<2)	<2	NA
W7/L82	3	1.130	11.3	158.7	170	50.0	7.35	8530	-	<4	<0.2	<0.08	145	(11)	<1	<2	<2	<2	10.7
W7/S84	NA	NA	NA	NA	367	9030	NA	45000	0.36	<6	(1300)	2.0	(660)	141	84	(300)	(<1)	<1	NA
W7/H85	NA	NA	NA	NA	360	4010	NA	86000	0.12	<6	(16)	2.2	(130)	52	22	(77)	(<1)	<1	NA
W8/L86	4	1.020	<0.1	29.4	29.4	720	1.39	817	0.007	<4	<0.2	<0.2	12	0.09	<1	<2	<0.09	<0.09	9.6
W8/L87	16	1.027	<0.1	37.8	37.6	1460	1.14	1760	<0.002	<4	<0.2	<0.2	18	0.17	<1	<2	<0.09	<0.09	9.7
W8/S88	NA	NA	NA	NA	558	11300	NA	8560	1.3	<4	(38)	4.1	(410)	50	160	(1800)	(<0.7)	<0.7	NA

Table 4.4. Continued

Sample ID	Depth (in.)	Density (g/mL)	Suspended solids (mg/mL)	Dissolved solids (mg/mL)	Tot. Sol. L:(mg/mL) S:(mg/g)	TOC L:(mg/L) S:(mg/kg)	Soluble Si (mg/L)	Metals (Liquid: mg/L; sludge: mg/kg) U	Ag	As	Ba	Cd	Cr	Hg	Ni	Pb	Se	Tl	pH
W9/L90	5	1.017	<0.1	21.2	21.2	290	2.53	2390	<0.002	<4	<0.2	<0.2	**7.3**	0.06	<1	<2	<0.09	<0.09	10.2
W9/S92	NA	NA	NA	NA	944	13900	NA	25800	0.77	<5	(200)	4.9	**160**	**40**	110	**620**	<0.7	<0.7	NA
W1/L7	4	1.002	<0.1	0.2	0.2	6.4	8.38	<0.1	<0.002	<2	0.13	<0.04	<0.1	<0.01	<0.6	<1	<0.09	(<0.09)	7.9
W1/L8	9	1.002	<0.1	0.2	0.2	10.1	8.69	<0.1	<0.002	<2	0.13	<0.04	<0.1	<0.01	<0.6	<1	<0.09	(<0.09)	8.0
W2/L11	4	1.002	<0.1	0.4	0.3	18.9	4.94	0.18	<0.002	<2	0.19	<0.04	<0.1	<0.01	<0.5	1.1	<0.09	(<0.09)	8.5
W2/L118	4	1.003	0.2	0.3	0.5	22.6	4.5	0.17	<0.002	<2	0.14	<0.04	<0.1	<0.01	<0.6	<1	<0.09	(<0.09)	8.5
W4/L22	4	1.013	0.2	6.1	6.3	49.5	2.55	910	0.003	<4	<0.2	<0.08	2.8	<0.01	<1	<2	<0.09	<0.09	9.1
W4/L119	30	1.014	0.2	10.1	10.3	59.9	2.07	1780	<0.002	<4	<0.2	0.09	**5**	<0.01	<1	<2	<0.09	<0.09	10.0
W4/L23	54	1.027	0.5	27.4	27.9	558.5	1.75	3680	<0.002	<4	<0.2	0.09	**13.5**	<0.01	<1	<2	<0.09	<0.09	10.9
W4/S24	NA	NA	NA	NA	307	9190	NA	33.0%	0.08	<4	(11)	22	(**729**)	2.4	15	(**150**)	(<0.7)	<0.7	NA
W4/H26	NA	NA	NA	NA	349	9020	NA	29.6%	0.06	<2	(5.8)	2.1	(**200**)	0.82	14	62	(0.49)	<0.4	NA
W10/L93	4	1.004	<0.1	5.7	5.5	8.7	1.71	34.7	<0.002	<4	<0.2	<0.2	0.55	<0.01	<1	2.2	<0.09	<0.09	9.2
W10/L94	28	1.009	<0.1	13.7	13.6	86.4	1.9	64.5	<0.002	<4	<0.2	<0.2	4.4	0.05	<1	<2	<0.09	<0.09	10.5
W10/L95	49	1.027	<0.1	37.1	36.7	54.6	2.34	460	<0.002	<4	<0.2	<0.2	**19**	**0.37**	<1	<2	<0.09	<0.09	10.9
W10/H120	NA	NA	NA	NA	845	14600	NA	82300	0.89	<7	(24)	6.1	(97)	**11**	31	**190**	(<1)	<1	NA
W10/S96	NA	NA	NA	NA	449	8180	NA	10800	0.79	5.7	(94)	4.7	(**140**)	**48**	83	**480**	(<0.9)	(<0.9)	NA
7860A/L63	8	0.909	NA	NA	NA	NA	NA	212						0.10					NA
7860A/L64	16	0.914	NA	NA	NA	NA	NA	252						0.06					NA
7860A/L65	24	0.910	NA	NA	NA	NA	NA	188						0.12					NA
7860A/L66	32	0.909	NA	NA	NA	NA	NA	192						0.06					NA
7860A/L67	40	1.004	<0.1	3.1	2.7	43.8	1.4	10	<0.002					0.01			<0.09	<0.09	4.0
7860A/L68	48	1.002	<0.1	3.5	2.3	43.8	1.7	9.8	<0.002					0.03			<0.09	<0.09	3.8
7860A/L69	54	1.003	<0.1	3.8	3.5	45.6	1.5	40	<0.002					**0.85**			<0.09	<0.09	4.0
W3/L16	4	1.004	<0.1	2.9	2.7	772	6.5	88	0.002	<4	<0.2	<0.2	3.3	<0.01	<1	<2	<0.09	<0.09	9.0
W3/L17	60	1.006	<0.1	4.6	4.4	1130	5.3	163	0.002	<4	<0.2	<0.2	**6.8**	0.01	<1	<2	<0.09	<0.09	10.1
W3/L18	106	1.009	<0.1	8.6	8.4	1500	7.2	285	0.002	<4	<0.2	<0.2	**20**	0.02	<1	<2	<0.09	<0.09	11.1
W3/S19	NA	NA	NA	NA	360	3410	NA	61300	0.05	(<2)	(6.4)	0.52	**1300**	0.83	<7	52	(<0.3)	<0.3	NA
WC17/O101	0	0.996	0.3	0.3	0.6	193	4.7	0.02	<0.001	<2	0.19	<0.09	<0.1	**0.73**	<0.6	<1	<0.05	<0.05	7.6
WC17/O102	1	0.999	0.3	0.4	0.7	143	4.67	0.01	<0.001	<2	0.15	<0.09	<0.1	0.08	<0.6	<1	<0.05	<0.05	7.6
WC17/L103	6	0.989	0.3	0.4	0.7	309	4.51	0.03	(<0.001)	<2	0.17	<0.09	<0.1	**49**	<0.6	<1	<0.05	<0.05	7.6
WC17/L104	14	1.005	0.1	0.4	0.5	153	4.66	0.01	<0.001	<2	0.18	<0.09	<0.1	0.04	<0.6	<1	<0.05	<0.05	7.6
WC17/L105	26	1.004	<0.1	0.4	0.3	127	7.64	0.01	<0.001	<2	0.14	<0.09	<0.1	<0.005	<0.6	<1	<0.05	<0.05	7.9
WC17/S106	NA	NA	NA	NA	122		NA	60.4	4.5	(0.76)	1400	**79**	(**2000**)	**380**	270	**390**	<0.5	(<0.5)	NA
W13/L27	4	1.033	0.6	34.2	34.8	41.4	14.9	18.8	0.003	<4	0.69	0.22	2.5	**31**	8.4	**8.8**	<0.09	<0.09	3.0
W13/L28	18	1.033	<0.1	33.9	33.6	39.0	14.5	17.4	0.004	<4	0.7	0.3	2.7	**35**	8.7	**8.9**	<0.09	<0.09	3.0
W14/L30	4	1.049	<0.1	29.8	27.5	121	16.5	0.56	<0.002	<4	0.45	<0.2	**69**	0.08	50	**7.5**	<0.09	<0.09	**0.6**
W14/L31	14	1.052	1.4	27.7	29.1	132	16.2	0.56	0.008	<4	0.63	<0.2	**64**	0.18	52	**7.5**	<0.09	<0.09	**0.5**
W15/L32	4	1.080	2.0	38.1	40.1	53.7	22.3	387	<0.002	<4	3.2	<0.2	**53**	**0.40**	48	**7.5**	<0.09	<0.09	**0.2**
W15/L33	21	1.081	<0.1	41.1	38.4	42.7	21.9	383	0.002	<4	3.4	<0.2	**55**	**0.39**	48	**7.5**	<0.09	<0.09	**0.2**
W1A/L1	2	1.002	0.3	0.4	0.7		4.47	477	0.008	<4	<0.2	**7.3**	0.33	**0.96**	<1	<2	<0.09	<0.09	8.8
T30/L121	2	1.001	<0.1	<0.1	<0.1	13.7	0.62	<1.0	0.002	<2	<0.1	<0.09	<0.1	0.01	<0.6	<1	<0.05	<0.05	8.7
7562/L97	4	1.001	0.1	0.2	0.3	29.6	2.36	6.77	<0.001	<2	<0.1	<0.09	<0.1	<0.01	<0.6	<1	<0.05	<0.05	7.4
7562/S98	NA	NA	NA	NA	75.9		NA	1990	0.0020	(<2)	140	18	(**2300**)	1.8	320	**900**	<0.006	(<0.006)	NA

* Values in bold exceed regulatory limits, () represents suspect data.
+ Top 6 in. were sampled.

Table 4.5. Anion data for inactive tank samples

Sample ID	Depth (in.)	pH	Cl^- (U[a])	F^- (U)	NO_3^- (U)	PO_4^{-2} (U)	SO_4^{-2} (U)	CN^- (U)	OH^- (N)	H^+ (N)	CO_3^{-2} (M)	HCO_3^- (M)
4/L111	0	11.7	3.4	1.1	26	50	6.3	*	0.01	*	0.05	*
T4/L44	4	11.7	3.1	1.1	26	51	6.3	0.54	0.01	*	0.05	*
T4/L45	34	11.7	3.4	1.3	25	50	5.9	0.59	0.01	*	0.05	*
T4/S46	NA	NA	<0.83%	<0.83%	*	<42%	<42%	*	*	*	*	*
T3/L42	5	12.7	7.0	1.8	53	160	15	1.10	0.03	*	0.08	*
T3/S43	NA	NA	29%	<12.7%	*	<62.5%	<62.5%	0.20	*	*	*	*
T9/L47	6.5	8.8	290	1.0	110	<5	37	*	*	*	<0.01	0.03
T9/S48	NA	NA	3%	<0.7%	*	*	6.8%	*	*	*	*	*
T2/L38	4	9.4	12	1.0	8.4	<5	22	0.14	*	*	0.04	0.08
T2/L112	4	9.4	12	1.0	8.0	<5	21	0.12	*	*	0.04	0.09
T2/L39	48	9.4	12	1.0	8.2	<5	22	0.10	*	*	0.04	0.09
T2/S40	NA	NA	4.9%	<0.8%	*	*	14%	*	*	*	*	*
T1/L35	4	9.3	7.8	1.0	16	<5	7.5	*	*	*	0.01	0.04
T1/L36	48	9.3	8.0	1.0	17	<5	8.1	0.22	*	*	0.01	0.04
T1/S37	NA	NA	*	*	*	*	*	*	*	*	*	*
TH1/L49	4	1.8	<1	<1	1400	<250	<250	<0.04	*	0.41	*	*
TH1/L50	10	1.8	<1	<1	1300	<250	<250	<0.10	*	0.40	*	*
TH3/L52	2.5	1.8	<1	<1	510	<50	<50	<0.04	*	0.17	*	*
TH3/L53	5	1.8	<1	<1	500	<50	<50	<0.04	*	0.18	*	*
TH3/L113	NA	6.6	<1	<1	<5	<5	<5	<0.05	*	*	*	*
W11/L3	4	8.0	<1	<1	<5	<5	<5	<0.04	*	*	*	*
W11/L115	4	7.8	<1	<1	<5	<5	<5	<0.04	*	*	*	*
W11/L114	24	7.7	<1	<1	<5	<5	<5	<0.04	*	*	*	*
W11/H5	NA	NA	*	*	*	*	*	*	*	*	*	*
TH4/L116	0	7.1	1.7	1.3	49	6.6	17	<0.05	<0.02	*	<0.01	*
TH4/L55	4	6.9	8.2	2.0	1,300	<50	140	<0.07	<0.01	*	<0.01	*
TH4/L117		6	6.9	5.7	1.0	550	<50	91	*	<0.02	*	<0.01 *
TH4/L56	39	7.1	140	86	25,000	<2500	83,000	*	<0.02	*	<0.01	*
TH4/L57	72	7.6	160	80	31,000	<2500	14,000	*	0.02	*	0.02	*
TH4/S58	NA	NA	<0.4%	<0.4%	*	*	4.8%	*	*	*	*	*
TH4/S59	NA	NA	370	960	*	*	0.47%	0.16	*	*	*	*
W5/L73	4	10.1	8.6	56	21	420	26	0.24	*	*	0.16	0.26
W5/S75	NA	NA	<84	1,300	*	<840	<840	*	*	*	*	*
W6/L77	4	8.7	1.3	5.1	73	<5	4.8	<0.07	*	*	<0.01	0.02
W6/L78	30	10.4	4.5	27	250	31	34	0.05	*	*	0.02	0.11
W6/L79	54	11.4	12.0	49	710	210	330	0.41	*	*	0.18	0.10
W6/S80	NA	NA	<800	4,000	*	<2,500	<2,500	*	*	*	*	*
W7/L82	3	10.3	29	11	440	29	55	<0.06	*	*	0.21	0.33
W7/S84	NA	NA	<1.2%	1,000	*	*	6.7%	*	*	*	*	*
W7/H85	NA	NA	*	*	*	*	*	*	*	*	*	*
W8/L86	4	9.2	290	<50	2,700	<250	1,400	0.33	*	*	0.01	0.04
W8/L87	16	9.3	840	<125	7,300	<625	4,900	0.58	*	*	0.02	0.07
W8/S88	NA	NA	<100	<100	*	<500	<500	*	*	*	*	*

Table 4.5. Continued

Sample ID	Depth (in.)	pH	Cl^- (U[a])	F^- (U)	NO_3^- (U)	PO_4^{-2} (U)	SO_4^{-2} (U)	CN^- (U)	OH^- (N)	H^+ (N)	CO_3^{-2} (M)	HCO_3^- (M)
W9/L90	5	9.8	340	<50	1,650	2,600	900	0.14	*[b]	*	0.03	0.09
W9/S92	NA[c]	NA	<100	<100	*	<500	<500	*	*	*	*	*
W1/L7	4	7.9	13	6.1	32	<5	47	<0.06	*	*	<0.01	<0.01
W1/L8	9	8.0	13	6.2	32	<5	47	<0.05	*	*	<0.01	<0.01
W2/L11	4	8.5	7.2	9.2	<5	<5	15	<0.05	*	*	<0.01	<0.01
W2/L118	4	8.5	7.6	9.9	<5	<5	15	<0.05	*	*	<0.01	<0.01
W4/L22	4	9.1	<20	<20	1,700	<100	570	<0.04	*	*	<0.01	0.02
W4/L119	30	10.0	<20	<20	1,600	<100	930	<0.04	*	*	0.01	0.04
W4/L23	54	10.9	<20	<20	2,200	250	2600	<0.07	*	*	0.08	0.19
W4/S24	NA	NA	<10	<10	*	<50	<50	*	*	*	*	*
W4/H26	NA	NA	<10	<10	*	<50	<50	*	*	*	*	*
W10/L93	4	9.2	170	74	2,900	<200	280	<0.04	*	*	*	0.02
W10/L94	28	10.5	410	190	4,400	<200	690	<0.04	*	*	0.02	<0.01
W10/L95	49	10.9	990	430	8,500	<200	2,100	0.54	*	*	0.09	0.02
W10/H120	NA	NA	<60	<60	*	<300	<300	*	*	*	*	*
W10/S96	NA	NA	<120	<120	*	<600	<600	*	*	*	*	*
7860A/L63	8	*	*	*	*	*	*	*	*	*	*	*
7860A/L64	16	*	*	*	*	*	*	*	*	*	*	*
7860A/L65	24	*	*	*	*	*	*	*	*	*	*	*
7860A/L66	32	*	*	*	*	*	*	*	*	*	*	*
7860A/L67	40	4.0	280	<20	3,800	<100	<100	*	*	0.03	*	*
7860A/L68	48	3.8	270	<20	3,800	<100	<100	*	*	0.03	*	*
7860A/L69	54	4.0	280	<20	3,800	<100	<100	*	*	0.03	*	*
W3/L16	4	9.0	7.8	<10	<50	490	280	0.08	*	*	<0.01	0.03
W3/L17	60	10.1	7.9	<10	<50	600	430	<0.07	*	*	0.02	*
W3/L18	106	11.1	10.0	<10	<50	800	620	0.10	*	*	0.05	*
W3/S19	NA	NA	<100	<100	*	<500	<500	*	*	*	*	*
WC17/O101	0	7.6	35	<1	8.2	<5	99	<0.06	*	*	*	<0.01
WC17/O102	1	7.6	32	<1	13	<5	98	0.06	*	*	*	<0.01
WC17/L103	6	7.6	39	1.0	<5	<5	89	<0.06	*	*	*	<0.01
WC17/L104	14	7.6	30	1.0	15	<5	82	0.06	*	*	*	<0.01
WC17/L105	26	7.9	31	<1	16	<5	91	<0.06	*	*	*	<0.01
WC17/S106	NA	NA	<50	<50	*	<300	<300	*	*	*	*	*
W13/L27	4	3.0	<3	<3	43	<13	<13	<0.05	*	0.26	*	*
W13/L28	18	3.0	<3	<3	43	<13	<13	<0.05	*	0.27	*	*
W14/L30	4	0.6	<3	<3	70	<13	<13	<0.05	*	0.19	*	*
W14/L31	14	0.5	<3	<3	70	<13	<13	<0.05	*	0.21	*	*
W15/L32	4	0.2	<4	<4	120	<20	<20	<0.06	*	0.70	*	*
W15/L33	21	0.2	<4	<4	120	<20	<20	<0.07	*	0.73	*	*
W1A/L1	2	8.8	33	1.0	<5	<5	58.0	<0.06	*	*	*	<0.01
T30/L121	2	8.7	<1	<1	<5	<5	<5	<0.06	*	*	*	<0.01
7562/L97	4	7.4	13	<10	160	<50	120	<0.05	*	*	*	<0.01
7562/S98	NA	NA	<90	<90	*	<450	<450	*	*	*	*	*

[a]For liquids U = mg/L; for sludges U = mg/kg.
[b]Asterisk indicates that data are not available.
[c]NA = not available.

Table 4.6. Radiochemical data for inactive tanks
(Liquids: Bq/mL; Sludges: Bq/g)

Sample ID	Depth (in.)	pH	Gross alpha	Gross beta	^{137}Cs	^{60}Co	^{90}Sr	H-3	^{233}U	^{238}U	^{252}Cf	^{238}Pu	^{239}Pu	^{244}Cm	^{241}Am	^{134}Cs	^{228}Th ^{232}Th	^{232}U	^{152}Eu	^{154}Eu	^{155}Eu	^{14}C
T4/L111	0	11.7	3.8E+01	3.1E+05	3.0E+05	5.2E+01	1.4E+03	1.1E+02	2.3E+01	•[a]	•	•	•	•	•	•	•	•	•	•	•	•
T4/L44	4	11.7	3.6E+01	2.8E+05	3.0E+05	6.4E+01	1.2E+03	1.1E+02	2.2E+01	•	•	•	•	•	•	•	•	•	•	•	•	•
T4/L45	34	11.7	4.9E+01	3.1E+05	3.0E+05	5.2E+01	1.4E+03	1.1E+02	2.9E+01	•	•	•	•	•	•	•	•	•	•	•	•	•
T4/S46	NA[b]	NA	3.7E+05	3.6E+07	4.5E+05	6.0E+04	2.2E+07	2.8E+01	7.1E+03	•	•	2.2E+04	4.6E+03	2.1E+05	8.2E+03	•	5.8E+02	•	5.2E+04	4.4E+04	7.0E+03	5.1E+02
T3/L42	5	12.7	2.0E+00	2.8E+05	2.7E+05	3.6E+02	3.0E+02	1.7E+02	2.0E+00	•	•	•	•	•	•	•	•	•	•	•	•	•
T3/S43	NA	NA	2.0E+05	2.5E+07	1.3E+06	1.6E+05	8.1E+06	7.7E+01	8.3E+03	•	<200	1.4E+04	5.3E+03	1.8E+05	•	•	•	•	5.1E+04	5.3E+04	•	7.6E+02
T9/L47	6.5	8.8	7.0E+02	3.4E+05	2.9E+05	6.0E-03	3.6E+04	1.6E+02	6.6E+02	•	•	•	•	•	•	•	•	•	•	•	•	•
T9/S48	NA	NA	1.5E+05	1.6E+07	4.0E+05	4.3E+04	1.4E+07	3.4E+01	4.4E+03	•	<2	1.0E+04	4.3E+03	9.7E+04	•	•	•	•	3.5E+04	8.9E+03	•	2.2E+03
T2/L38	4	9.4	2.0E+02	1.4E+05	1.4E+05	<75	2.5E+03	2.1E+02	1.9E+02	•	•	•	•	•	•	•	•	•	•	•	•	4.8E+02
T2/L112	4	9.4	2.1E+02	1.4E+05	1.4E+05	<75	2.8E+03	2.1E+02	1.8E+02	•	•	•	•	•	•	•	•	•	•	•	•	3.6E+02
T2/L39	48	9.4	2.0E+02	1.4E+05	1.4E+05	<75	2.7E+03	2.1E+02	1.8E+02	•	•	•	•	•	•	•	•	•	•	•	•	2.3E+02
T2/S40	NA	NA	2.5E+05	2.0E+07	2.5E+05	6.4E+04	1.2E+07	9.5E+01	8.3E+03	•	1.8E+05	3.1E+03	5.1E+03	<200	•	•	•	•	3.8E+04	2.6E+04	3.8E+03	1.7E+01
T1/L35	4	9.3	1.9E+02	8.1E+04	7.4E+04	<50	3.3E+03	7.1E+01	1.8E+02	•	•	•	•	•	•	•	•	•	•	•	•	•
T1/L36	48	9.3	2.1E+02	7.8E+04	7.5E+0	<50	3.4E+03	7.1E+01	2.0E+02	•	•	•	•	•	•	•	•	•	•	•	•	•
T1/S37	NA	NA	6.5E+05	5.9E+07	3.9E+05	2.6E+05	3.2E+07	2.6E+01	•	•	•	3.4E+04	6.5E+03	•	•	•	•	•	1.4E+05	1.2E+05	2.3E+04	4.8E+01
TH1/L49	4	1.8	3.9E+02	3.2E+04	9.0E+03	<20	1.0E+04	5.0E+00	7.5E+01	•	<5	<6	<3	<2	•	•	4.0E+01	•	•	•	•	5.0E-01
TH1/L50	10	1.8	4.2E+02	3.9E+04	9.1E+03	<20	1.0E+04	4.9E+00	8.8E+01	•	<4	<5	<3	<2	•	•	5.1E+01	•	•	•	•	5.0E-01
TH3/L52	2.5	1.8	1.5E+02	3.2E+04	7.7E+03	<20	8.8E+03	2.6E+00	2.6E+01	•	<2	<2	<1	<1	•	•	1.7E+01	•	•	•	•	•
TH3/L53	5	1.8	1.4E+02	3.0E+04	8.4E+03	<20	8.7E+03	9.4E+00	7.1E+01	•	<2	<1	<1	<1	•	•	1.5E+01	•	•	•	•	•
TH3/L113	NA	6.6	<1	<20	<27	<20	7.2E-01	1.7E-01	<1	•	•	<1	<1	<1	•	•	<1	•	•	•	•	2.0E-01
W11/L3	4	8.0	<1	<20	<20	<20	2.0E+01	6.0E-01	•	•	•	•	•	•	•	•	•	•	•	•	•	2.0E-01
W11/L115	4	7.8	<1	<20	<20	<20	4.2E+01	5.0E-01	•	•	•	•	•	•	•	•	•	•	•	•	•	1.0E-02
W11/L114	24	7.7	<1	<20	<20	<20	2.1E+01	5.0E-01	•	•	•	•	•	•	•	•	•	•	•	•	•	1.0E-01
W11/H5	NA	NA	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
TH4/L116	0	7.1	<20	<20	5.4E-01	<20	3.0E-02	3.0E-03	<0.2	•	<0.2	<0.2	<0.2	<0.2	•	•	1.3E-01	•	•	•	•	•
TH4/L55	4	6.9	<20	<10	7.4E+00	<20	1.4E-01	3.0E-02	<0.2	•	<0.2	<0.2	<0.2	<0.2	•	•	•	•	•	•	•	5.0E-03
TH4/L117	[6]	6.9	<20	<20	3.6E+00	<20	4.6E-01	•	<0.2	•	<0.2	<0.2	<0.2	<0.2	•	•	1.3E-01	•	•	•	•	•
TH4/L56	39	7.1	1.1E+02	3.7E+02	1.8E+02	2.2E-01	2.5E+01	5.0E-02	<0.3	•	<0.3	<0.3	<0.3	<0.3	•	•	•	•	•	•	•	•
TH4/L57	72	7.6	1.6E+02	7.0E+02	2.4E+02	3.7E-01	2.0E+01	•	<0.4	•	<0.4	<0.4	<0.4	<0.4	•	•	•	•	•	•	•	•
TH4/S58	NA	NA	1.7E+03	7.0E+03	3.9E+02	3.0E+00	1.9E+03	1.3E+01	<25	•	<239	<180	4.2E+01	<55	•	•	1.9E+02	•	•	•	•	•
TH4/S59	NA	NA	2.9E+03	9.9E+03	4.2E+02	4.3E+00	2.2E+03	2.2E+01	1.2E+03	•	<310	<2	1.3E+02	<124	•	•	4.4E+02	•	•	•	•	•
W5/L73	4	10.1	6.0E+00	4.2E+03	5.5E+03	1.6E+02	9.0E+00	1.9E+00	•	•	<0.2	<2	<4	<0.4	•	•	•	•	•	•	•	•
W5/S75	NA	NA	1.3E+03	1.8E+05	3.0E+04	2.6E+02	2.9E+04	1.1E+01	4.6E+01	•	<4	5.4E+01	6.2E+02	3.5E+02	•	•	•	•	•	•	•	•
W6/L77	4	8.7	1.0E+00	2.8E+03	1.6E+03	1.3E+01	3.1E+02	6.3E-01	•	•	<0.2	<2	<4	<0.4	•	•	•	•	•	•	•	•
W6/L78	30	10.4	3.0E+00	6.0E+03	5.6E+03	2.8E+01	7.8E+01	1.4E+00	•	•	<0.2	<2	<4	<0.4	•	•	•	•	•	•	•	•
W6/L79	54	11.4	3.0E+00	1.9E+04	2.0E+04	9.0E+01	1.9E+02	3.2E+00	•	•	<0.2	<2	<4	<0.4	•	•	•	•	•	•	•	•
W6/S80	NA	NA	8.0E+03	1.0E+06	8.2E+04	9.1E+02	3.6E+05	9.2E+00	2.7E+02	•	<8	2.1E+02	1.8E+03	4.2E+03	•	•	•	•	•	•	•	•
W7/L82	3	10.3	2.0E+02	5.6E+05	6.4E+05	2.9E+02	6.0E+02	1.9E+01	•	•	<0.2	<2	<4	<0.4	•	•	8.5E+01	•	•	•	•	•
W7/S84	NA	NA	1.4E+04	3.1E+06	1.2E+06	5.0E+03	8.1E+05	1.2E+02	5.4E+02	•	<14	1.1E+03	2.4E+03	4.2E+03	5.7E+02	1.0E+02	•	•	1.6E+03	1.3E+03	•	•
W7/H85	NA	NA	8.0E+03	1.7E+06	1.0E+06	2.0E+03	3.1E+05	8.3E+01	1.7E+03	1.6E+03	<4	6.4E+02	4.8E+02	2.5E+03	•	•	•	•	•	4.5E+02	•	•
W8/L86	4	9.2	1.5E+02	2.4E+05	3.4E+05	3.0E+02	6.6E+02	8.0E+00	<2	•	<2	<2	<2	<2	•	•	•	•	•	•	•	•
W8/L87	16	9.3	2.0E+02	2.8E+05	4.0E+05	5.9E+02	4.6E+02	8.8E+00	<2	•	<2	<2	<2	<2	•	•	•	•	•	•	•	•
W8/S88	NA	NA	2.9E+04	6.7E+06	4.7E+04	4.0E+02	2.8E+06	0.0E+00	1.6E+03	•	<4	2.9E+03	3.2E+03	1.5E+04	2.0E+02	•	•	•	9.0E+01	1.4E+02	•	•

Table 4.6. Continued

Sample ID	Depth (in.)	pH	Gross alpha	Gross beta	^{137}Cs	^{60}Co	^{90}Sr	H-3	^{233}U	^{238}U	^{252}Cf	^{238}Pu	^{239}Pu	^{244}Cm	^{241}Am	^{134}Cs	^{228}Th ^{232}Th	^{232}U	^{152}Eu	^{154}Eu	^{155}Eu	^{14}C
W9/L90	5	9.8	6.0E+01	7.1E+04	6.3E+04	1.1E+02	3.7E+02	1.0E+01	<3	*	<3	<3	<3	<3	*	8.5E+01	*	*	*	*	*	*
W9/S92	NA	NA	1.1E+05	5.0E+06	2.2E+04	7.8E+02	2.2E+06	0.0E+00	3.1E+03	*	*	2.2E+04	9.2E+03	5.4E+04	7.2E+03	2.0E+01	*	*	2.3E+02	4.4E+02	5.2E+01	*
W1/L7	4	7.9	<3	8.7E+02	1.8E+01	<10	*	3.9E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	*
W1/L8	9	8.0	<3	8.7E+02	1.8E+01	<10	*	2.5E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	*
W2/L11	4	8.5	<3	7.1E+02	2.2E+01	<10	*	1.5E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	*
W2/L118	4	8.5	<3	7.4E+02	2.0E+01	<10	*	1.7E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	*
W4/L22	4	9.1	<3	1.6E+03	8.9E+02	<10	*	1.2E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	*
W4/L119	30	10.0	<3	2.6E+03	1.4E+03	<10	*	2.0E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	*
W4/L23	54	10.9	<3	2.2E+03	2.1E+03	<13	2.9E+02	2.1E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	*
W4/S24	NA	NA	5.9E+03	3.3E+05	3.1E+04	1.2E+01	8.9E+04	0.0E+00	2.0E+03	2.3E+03	<6	<6	4.4E+02	<7	*	*	*	*	*	*	*	*
W4/H26	NA	NA	6.6E+03	8.7E+04	1.3E+04	*	2.1E+04	3.3E+00	2.7E+03	3.0E+03	<7	<7	2.1E+02	<6	*	*	*	*	*	*	*	*
W10/L93	4	9.2	5.0E+00	3.1E+04	3.1E+04	2.8E+01	1.2E+03	3.2E+01	<2	<2	<2	<2	<2	<3	*	*	*	*	*	*	*	*
W10/L94	28	10.5	1.0E+01	7.8E+04	8.3E+04	3.4E+01	7.6E+02	6.8E+01	<2	<2	<2	<2	<2	<3	*	*	*	*	*	*	*	*
W10/L95	49	10.9	2.8E+01	1.9E+05	2.4E+05	1.4E+02	2.1E+02	1.4E+02	7.0E+00	3.0E+00	<2	2.0E+00	2.0E+00	<3	*	*	*	*	*	*	*	*
W10/H120	NA	NA	4.1E+04	1.2E+07	1.1E+07	1.4E+03	1.2E+06	9.0E-01	2.1E+03	1.3E+03	<25	6.7E+03	1.1E+04	1.4E+04	*	1.4E+03	*	*	*	*	*	*
W10/S96	NA	NA	8.4E+04	5.3E+06	8.6E+05	1.3E+04	8.6E+06	1.4E+02	<50	*	<50	2.9E+04	8.2E+03	4.7E+04	4.5E+03	*	*	*	4.6E+03	8.0E+03	*	*
7860A/L63	8	*	3.0E+00	<10	4.0E-02	<10	<10	6.1E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	1.8E+00
7860A/L64	16	*	2.0E+00	<10	8.7E-01	<10	<10	5.1E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	1.9E+00
7860A/L65	24	*	3.0E+00	<10	7.9E-01	<10	<10	5.4E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	1.7E+00
7860A/L66	32	*	2.0E+00	<10	9.8E-02	<10	<10	5.7E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	1.4E+00
7860A/L67	40	4.0	<1	7.7E+01	1.0E+01	7.3E+00	2.2E+01	3.3E+01	*	*	*	*	*	*	*	*	*	*	*	*	*	*
7860A/L68	48	3.8	2.0E+00	7.0E+01	9.5E+00	6.3E+00	2.1E+01	3.4E+01	*	*	*	*	*	*	*	*	3.4E-01	*	*	*	*	*
7860A/L69	54	4.0	6.0E+00	9.2E+01	1.0E+01	6.7E+00	2.1E+01	3.3E+01	*	*	*	*	*	*	*	*	8.0E-01	*	*	*	*	*
W3/L16	4	9.0	6.0E+00	4.9E+02	5.8E+02	<10	4.0E+01	6.9E-01	<5	*	<5	<5	<5	<5	*	*	*	*	*	*	*	*
W3/L17	60	10.1	1.2E+01	6.7E+02	8.4E+02	1.0E-01	6.4E+01	9.4E-01	<5	*	<5	<5	<5	<5	*	*	*	*	*	*	*	*
W3/L18	106	11.1	1.7E+01	1.2E+03	1.3E+03	2.6E-01	9.0E+01	1.1E+00	<5	*	<5	<5	<5	<5	*	*	*	*	*	*	*	*
W3/S19	NA	NA	8.4E+03	1.5E+06	4.7E+04	2.4E+01	1.5E+05	2.2E+00	1.1E+03	4.3E+02	<10	1.7E+02	3.9E+03	<10	2.6E+02	*	*	*	*	7.6E+01	*	*
WC17/O101	0	7.6	<10	<20	1.2E+00	*	<1	9.4E-01	*	*	*	*	*	*	*	*	*	*	*	*	*	*
WC17/O102	1	7.6	<10	<20	1.3E-01	*	<1	4.0E-01	*	*	*	*	*	*	*	*	*	*	*	*	*	*
WC17/L103	6	7.6	<10	<20	1.3E+00	*	<1	8.2E-01	*	*	*	*	*	*	*	*	*	*	*	*	*	*
WC17/L104	14	7.6	<10	<20	4.8E-01	*	<1	1.6E-01	*	*	*	*	*	*	*	*	*	*	*	*	*	*
WC17/L105	26	7.9	<10	<20	9.7E-01	*	<1	2.7E-01	*	*	*	*	*	*	*	*	1.6E-02	*	*	*	*	*
WC17/S106	NA	NA	2.7E+03	3.5E+03	1.5E+01	8.2E-01	2.9E+02	9.1E+01	*	*	<3	4.3E+02	6.2E+01	1.6E+03	2.6E+02	*	*	*	*	3.3E+00	5.6E-01	*
W13/L27	4	3.0	7.4E+02	1.4E+06	6.1E+05	1.5E+02	5.5E+05	2.8E+03	*	*	<4	5.5E+02	5.8E+01	<4	*	*	1.2E+02	*	*	1.6E+03	*	*
W13/L28	18	3.0	7.6E+02	1.3E+06	5.8E+05	1.9E+02	5.9E+05	2.8E+03	*	*	<4	5.5E+02	6.9E+01	<4	*	*	1.3E+02	*	*	1.5E+03	*	*
W14/L30	4	0.6	8.3E+03	9.7E+05	4.1E+05	1.1E+03	4.5E+05	2.4E+00	*	*	<50	<150	<20	<100	*	*	8.0E+03	*	*	*	*	*
W14/L31	14	0.5	8.4E+03	1.0E+06	4.1E+05	1.1E+03	4.3E+05	3.0E+00	*	*	<50	<150	<50	<150	*	*	8.2E+02	*	*	*	*	*
W15/L32	4	0.2	2.6E+03	3.6E+06	1.6E+06	1.4E+03	1.7E+06	3.0E+01	1.1E+02	*	<10	2.5E+02	6.3E+02	<25	*	*	1.5E+03	*	*	*	*	*
W15/L33	21	0.2	2.3E+03	3.6E+06	1.5E+06	1.2E+03	1.5E+06	3.2E+01	1.4E+02	*	<10	2.4E+02	5.8E+02	<15	*	*	1.3E+03	*	*	*	*	*
W1A/L1	2	8.8	1.1E+03	1.3E+03	1.4E+02	4.4E+00	3.2E+02	2.6E+00	7.3E+02	5.0E+00	<2	1.9E+01	6.7E+01	9.0E+00	5.4E+01	4.6E+00	*	2.7E+02	*	*	*	*
T30/L121	2	8.7	9.4E+01	2.7E+02	4.0E+00	*	1.5E+01	1.6E+00	<2	4.0E+00	<2	<2	3.5E+00	3.1E+01	<10	*	*	*	*	*	*	*
7562/L97	4	7.4	<1	1.2E+03	1.7E+00	*	1.7E+03	1.6E+00	*	*	*	*	*	*	*	*	*	*	*	*	*	*
7562/S98	NA	NA	<1	8.8E+05	3.8E+03	*	2.2E+05	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*

[a]Asterisk indicates that data are not available.
[b]NA = not applicable.

Table 4.4 summarizes the physical measurements, total organic carbon (TOC), eight RCRA metals plus two proposed RCRA metals (Ni and Tl), and completed treatability metal data for all the inactive tank samples collected. Most of the RCRA metals in the liquid sample were determined by ICP except for silver, selenium, and thallium. The sensitivity of the ICP method for selenium and thallium was insufficient; thus, these analytes were determined by GFAA. The silver was also determined by GFAA rather than by ICP. Because of the limitations of the sequential ICP used, the analytical wavelength for silver was much higher than the other metals, and the time required to scan to the silver wavelength consumed too much of the sample. The RCRA metals for the sludge samples were all (except for mercury) determined by GFAA because of the large dilution that resulted from the dissolution of the samples (dilution factors range from 50 to 200 for the sludge samples). The mercury for all tank samples were determined by the CV technique.

The anion data are summarized in Table 4.5, which includes the common anions determined by ion chromatography along with the cyanide, alkalinity, acidity, carbonate, and bicarbonate. The sulfide data were not reported because no reliable data have been obtained at this time. However, a qualitative test based upon the precipitation of cadmium sulfide indicated that the concentration of sulfide in the samples analyzed for cyanide had not exceeded the regulatory limit. Also, the nitrate was not reported for the sludge samples because the sludges were dissolved in nitric acid.

The radiochemical data are summarized in Table 4.6, which includes the identified nuclides along with the gross alpha and gross beta measurements. The gross beta values are not considered reliable, but they are included for comparison to identified beta emitters.

4.5 EVALUATION OF DATA

4.5.1 Overall Trends

Of the 27 tanks characterized, 17 tanks contained insoluble solids or sludges. The presence of the sludges is dependent on pH and on cation and anion content. No sludge was observed in tanks with a pH less than 5 (6 tanks), but all tanks with a pH greater than 9 (12 tanks) contained a sludge. With the exception of alkali metals (Group Ia), most other metals form insoluble hydroxides. Basic solutions tend to dissolve atmospheric carbon dioxide and accumulate increasing amounts of carbonate with time, which results in the precipitation of many carbonate salts. Based upon on the solubility products (Table 4.7) for the various cation-anion precipitates and the anion distribution (Table 4.8) in the waste tanks, the elevated levels of RCRA metals found in sludges from tanks with high pH liquids can largely be accounted for by the insolubility of the hydroxide and carbonate salts. Most alpha emitters also form insoluble hydroxides and carbonates. This formation accounts for the low level of transuranium nuclides in the liquid phase of most waste tanks. The ^{90}Sr was observed to be 3 to 4 orders of magnitude greater in the sludge relative to the

Table 4.7. Solubility products for RCRA metals and TRU metals

Compound	Formula	pK_{sp}[a]
RCRA metals		
Barium carbonate	$BaCO_3$	8.29
Barium chromate	$BaCrO_4$	9.93
Barium fluoride	BaF_2	5.98
Barium hydroxide	$Ba(OH)_2$	2.30
Barium hydrogen phosphate	$BaHPO_4$	6.50
Barium phosphate	$Ba_3(PO_4)_2$	22.47
Barium nitrate	$Ba(NO_3)_2$	2.35
Barium sulfate	$BaSO_4$	9.96
Cadmium carbonate	$CdCO_3$	11.28
Cadmium fluoride	CdF_2	2.19
Cadmium hydroxide	$Cd(OH)_2$	13.60
Cadmium phosphate	$Cd_3(PO_4)_2$	32.60
Cadmium sulfide	CdS	26.10
Chromium(III) fluoride	CrF_3	10.18
Chromium(II) hydroxide	$Cr(OH)_2$	15.70
Chromium(III) hydroxide	$Cr(OH)_3$	30.20
Chromium(III) phosphate (green)	$CrPO_4 \cdot 4H_2O$	22.62
Chromium(III) phosphate (violet)	$CrPO_4 \cdot 4H_2O$	17.00
Lead carbonate	$PbCO_3$	13.13
Lead chromate	$PbCrO_4$	12.55
Lead chloride	$PbCl_2$	4.79
Lead fluoride	PbF_2	7.57
Lead chloride fluoride	$PbClF$	8.62
Lead hydroxide	$Pb(OH)_2$	14.93
Lead(IV) hydroxide	$Pb(OH)_4$	65.50
Lead hydroxide chloride	$PbOHCl$	13.70
Lead hydroxide nitrate	$PbOHNO_3$	3.55
Lead hydrogen phosphate	$PbHPO_4$	6.24
Lead phosphate	$Pb_3(PO_4)_2$	42.10
Lead sulfate	$PbSO_4$	7.79
Lead sulfide	PbS	27.90
Mercury(I) carbonate	Hg_2CO_3	16.05
Mercury(I) chloride	Hg_2Cl_2	17.88
Mercury(I) chromate	Hg_2CrO_4	8.70
Mercury(I) hydroxide	$Hg_2(OH)_2$	23.70
Mercury(II) hydroxide	$Hg(OH)_2$	25.52
Mercury(I) hydrogen phosphate	Hg_2HPO_4	12.40
Mercury(I) sulfide	Hg_2S	47.00
Mercury(II) sulfide (black)	HgS	51.80
Mercury(II) sulfide (red)	HgS	52.40
Nickel(II) carbonate	$NiCO_3$	8.18
Nickel(II) hydroxide	$Ni(OH)_2$	14.70
Nickel(II) phosphate	$Ni_3(PO_4)_2$	30.30
Nickel(II) sulfide (alpha)	NiS	18.50
Nickel(II) sulfide (beta)	NiS	24.00
Nickel(II) sulfide (gamma)	NiS	25.70
Silver carbonate	Ag_2CO_3	11.09
Silver chloride	$AgCl$	9.75
Silver chromate	Ag_2CrO_4	11.95
Silver hydroxide	$AgOH$	7.71
Silver phosphate	Ag_3PO_4	15.84
Silver sulfate	Ag_2SO_4	4.84
Silver sulfide (alpha)	Ag_2S	49.18
Silver sulfide (beta)	Ag_2S	49.00
Thallium(I) chloride	$TlCl$	3.76
Thallium(I) chromate	Tl_2CrO_4	12.00
Thallium(I) sulfide	Tl_2S	20.30
Thallium(III) hydroxide	$Tl(OH)_3$	45.20
TRU and other elements of interest		
Americium(III) hydroxide	$Am(OH)_3$	19.57
Americium(IV) hydroxide	$Am(OH)_4$	56.00
Europium hydroxide	$Eu(OH)_3$	23.05
Neptunyl hydroxide	$NpO_2(OH)_2$	21.60
Plutonium(III) hydroxide	$Pu(OH)_3$	19.70
Plutonium(IV) hydroxide	$Pu(OH)_4$	55.00
plutonyl(IV) hydroxide	$PuO_2(OH)_2$	24.70
Plutonium hydrogen phosphate	$Pu(HPO_4)_2$	27.70
Scandium fluoride	ScF_3	17.37
Scandium hydroxide	$Sc(OH)_3$	30.10
Strontium carbonate	$SrCO_3$	9.96
Strontium chromate	$SrCrO_4$	4.65
Strontium fluoride	SrF_2	8.61
Strontium phosphate	$Sr_3(PO_4)_2$	27.39
Strontium sulfate	$SrSO_4$	6.49
Thorium hydroxide	$Th(OH)_4$	44.40
Thorium phosphate	$Th_3(PO_4)_4$	78.60
Thorium hydrogen phosphate	$Th(HPO_4)_2$	20.00
Uranyl carbonate	UO_2CO_3	11.73
Uranyl hydroxide	$UO_2(OH)_2$	21.95
Uranyl phosphate	$(UO_2)_3(PO_4)_2$	46.70
Uranyl hydrogen phosphate	UO_2HPO_4	10.67
Yttrium fluoride	YF_3	12.14
Yttrium hydroxide	$Y(OH)_3$	22.10

Table 4.8. Characteristics of inactive tanks for forming insoluble salts (sludges)

Tank	Sludge present	Average pH	Presence of anions efficient at forming insoluble salts					
			CO_3^{-2}	SO_4^{-2}	PO_4^{-2}	CrO_4^{-2}	Cl^-	F^-
pH > 9								
T1	Yes	9.3	+[a]	+	-[b]	+	+	-
T2	Yes	9.4	+	+	-	+	+	-
T3	Yes	12.7	+	+	+	+	+	-
T4	Yes	11.7	+	+	+	+	+	-
W3	Yes	10.1	+	++[c]	++	+	+	-
W4	Yes	10.0	+	++	-	+	-	-
W5	Yes	10.1	+	+	++	+	+	+
W6	Yes	10.2	+	++	++	+	+	+
W7	Yes	10.3	+	++	+	+	+	+
W8	Yes	9.3	+	++	-	+	++	-
W9	Yes	9.8	+	++	++	+	++	-
W10	Yes	10.2	+	++	-	+	++	++
pH < 5								
TH1	no	1.8	-	-	-	-	-	-
TH3	no	1.8	-	-	-	-	-	-
W13	no	3.0	-	-	-	-	-	-
W14	no	0.6	-	-	-	+	-	-
W15	no	0.2	-	-	-	+	-	-
7860A	no	3.9	-	-	-	?[d]	+	-
5 < pH < 9								
W1	no	8.0	-	+	-	-	+	+
W2	no	8.5	-	+	-	-	+	+
W1A	no	8.8	-	+	-	-	+	-
T30	no	8.7	-	-	-	-	-	-
T9	Yes	8.8	+	+	-	-	++	-
W11	Yes	7.9	-	-	-	+	-	-
TH4	Yes	7.1	+	+++[e]	+++	+	++	++
WC17	Yes	7.6	-	+	-	+	+	-
7562	Yes	7.4	-	+	-	+	+	-

[a] + = low concentration.
[b] - = none observed.
[c] ++ = high concentration.
[d] ? = no data
[e] +++ = very high concentration.

liquid phase of basic tanks. This distribution of ^{90}Sr in the sludge is most likely the result the precipitation of strontium carbonate.

In general, for waste tanks with a pH greater than 5, most species remain in solution, whereas in tanks with a pH greater than 9, an insoluble sludge consisting primarily of hydroxide and carbonate salts is found. However, for waste tanks with an intermediate pH ($5 < pH < 9$), the formation of sludge is difficult to predict. As shown in Table 4.8, five out of nine tanks with an intermediate pH had a sludge. Four of the five tanks (no anion data are available for waste tank W11 because it contained a very hard, thin sludge layer which was difficult to sample) containing sludge within this group contained moderate to high levels of sulfate, which tends to form insoluble salts. Three of the remaining four tanks with an intermediate pH also had moderate levels of sulfate present, but no sludge was formed.

4.5.2 Physical Measurements

Dissolved solids observed in the inactive tank samples ranged from 0.1 to 159 mg/mL, and suspended solids ranged from <0.1 to 11.3 mg/mL. Low dissolved solids were observed in most of the tanks with a pH between 5 and 9. However, TH4 with an average pH equal to 7.1, had the second highest dissolved solids observed. Vertical concentration gradients were observed for dissolved solids (and most other species measured) in a number of the tanks with a pH greater than 5. In acidic ($pH < 5$) tanks, the dissolved solids content was invariant, and the liquid appeared to be homogeneous based on samples taken at various levels. Also, little or no suspended solids were observed in the acidic tanks.

The density measurements ranged from 0.9982 to 1.1303 g/mL for the aqueous samples. For the only tank (7860A) with a significant organic layer, the density of the organic samples ranged from 0.9088 to 0.9135 g/mL. As expected, the density measurements followed the dissolved solid measurements, and a number of tanks with pH greater than 5 exhibited vertical gradients in density.

The highest measurements for suspended solids, dissolved solids, and density were observed in the W7 tank. These high measurements probably result from the high level of uranium present in the liquid phase of the W7 waste tank (W7 had the highest level of uranium found in a liquid phase).

4.5.3 RCRA Metals

For the most part, the distribution of the RCRA metals within liquid and sludge phases in the inactive waste tank was dependent first upon the pH and second upon the anion content of the liquid phase. If one refers to Table 4.7, on solubility products, most of the RCRA metals form hydroxides with low solubilities. Also, many of the common anions found in the waste tanks form highly insoluble salts with the RCRA metals. The distribution of anions within the waste tanks is illustrated by Table 4.8.

The high levels of chromium, mercury, and lead observed in many of the inactive waste tanks was not surprising because of the various laboratory activities that were associated with the waste-tank system. For many years a common cleaner for glassware was a mixture of sulfuric acid and potassium dichromate, which was routinely flushed down the hot drains. Another common practice was the cleaning and decontamination of lead bricks and other forms of lead shielding. The use of mercury in electrochemical techniques and for other processes or activities would account for the levels of mercury observed. Cadmium was present in most of the sludge samples, however, usually below the regulatory limits. Little or no cadmium was observed in most of the liquid phases analyzed, but the cadmium observed in the liquid phase of one tank (W1A) was above the regulatory limit.

For basic waste tanks (pH > 9), much higher levels of RCRA metals were found in the sludge phase. Excluding the acidic tanks, the only EPA hazardous metals observed at significant levels in the liquid phases were chromium and mercury. Some of the acidic tanks also had elevated levels of lead and nickel in the liquid phase.

4.5.4 Treatability Metals

The metals determined for waste-treatability studies include species that must be known for various waste-solidification and other waste-handling processes (Al, B, Ca, Fe, K, Mg, Na, Si, and Sr), and uranium and thorium for criticality calculations and accountability requirements. The TOC and total solids are important measurements to the waste-processing groups. Currently, only the uranium and soluble silica measurements have been completed, and the data are reported in Table 4.4 with other data on metals.

Silica measurements were not performed on the sludge samples, and only the soluble silica was measured in the liquid samples. The soluble silica ranged from less than 1 to about 80 mg/L, but most of the liquid samples were below 20 mg/L. The soluble silica observed in the acidic liquids was consistently higher than average; however, the highest level observed was in the most basic tank (tank T3, pH 12.7).

Uranium was found in all sludge samples and ranged from 60 mg/kg to 33% by weight in the sludges. The uranium in the liquid phase ranged from <0.1 to in excess of 8500 mg/L, and uranium concentration gradients were observed in a number of the tanks. Although the uranyl carbonate and hydroxide are fairly insoluble, the uranyl cation (UO_2^{2+}) can form unusually stable anionic species such as uranyl tricarbonate , $UO_2(CO_3)_3^{4-}$, which is water soluble.

4.5.5 Cyanide and Sulfide

The cyanide detection limit for the procedure and sample volume (10 mL) used during this project was 0.04 mg/L, which is significantly below the regulatory limits for labile cyanide. The total cyanide found in the liquid waste tank samples ranged from less than 0.04 mg/L to a maximum of 1.1 mg/L, which is 2 orders of magnitude below the regulatory limits. However, poor spike recoveries were observed with many of the waste tank samples, and a significant cyanide demand was observed with some samples. The chemical

complexity and the variable composition observed with the waste tank samples make it very difficult to identify all the possible interferences, such as the formation of metal complexes with cyanide, such as $Hg(CN)_2$ and $Cu(CN)_2$, which are difficult to dissociate and can prevent distillation or cause poor distillation. The poor distillation would result in poor spike recoveries and low results. Also, the presence of free chlorine in basic solutions can consume cyanide by alkaline chlorination to gaseous cyanogen chloride. Although several possible interferences to the cyanide measurement are discussed, numerous mechanisms could result in poor spike recoveries. Therefore, if more nearly accurate cyanide measurements are required, additional method development is necessary to identify the cause of poor spike recoveries and cyanide consumption observed with the waste-tank samples.

The method used for the determination of sulfide is based upon the separation of the sulfide from the complex sample matrix by distillation of hydrogen sulfide from strong acid solution and trapping of the hydrogen sulfide in a basic scrubber solution. The sulfide in the basic trapping solution is reacted with excess iodine and the remaining iodine is determined by titration. Therefore, the sulfide determination was actually a measurement of the sample's reducing power relative to iodine and cannot be exclusively attributed to the sulfide concentration. The sulfide measurement depends upon the quantitative reduction of iodine to iodide which requires a relatively simple system to be valid. The waste tank samples are complex and may contain volatile species that can reduce the iodine, thus contributing to positive bias. Also, any volatile species that can oxidize the iodide to iodine would result in a negative bias. Here again, more method development is required for accurate sulfide measurements.

4.5.6 Common Anions

The determination of common anions was primarily for treatability studies, but the studies were also useful for understanding the distribution of various metals in the liquid and sludge phases of the waste tanks. The liquid samples were diluted and then measured directly on an ion chromatograph. The sludge samples were first dissolved with nitric acid (measurements for nitrate in sludge samples were not reported) and then filtered prior to measurement by ion chromatography. Because of a limitation of ion chromatography, the high nitrate concentration in the dissolved sludge samples prevented measurement of other anionic species much below percent levels. The ion-exchange separation of anionic species requires that the relative concentration of all anions be within a factor of about 100, or the predominate species behaves as an eluent for the other species and all anions elute in a single band. Also, because the ion chromatography was not done in a radiochemical laboratory, the detection limits for some of the more radioactive samples were limited.

4.5.7 Alpha Emitters (TRU Waste)

In general, most of the alpha activity was found in the sludge of basic waste tanks, with little to no alpha activity observed in the basic liquid phase. Low levels of alpha activity were observed in the acidic waste tanks, and one acidic tank (W13) approached the limits for classification as TRU-waste. The presence of transuranic alpha emitters with half-lives greater than 20 years and activities in excess of 3.7×10^3 Bq/g (100 nCi/g) is the basic criterion for classification as "TRU-waste." In addition to the transuranics, the isotope ^{233}U is included for classification as TRU-waste. The isotope ^{244}Cm has a half-life of 18 years and is excluded from the TRU-waste definition. However, due to the high specific activity, ^{244}Cm is included in these discussions independent from the TRU discussion. The only TRU-waste identified was found in the waste tanks with a sludge. Eleven of the 17 tanks with sludge are classified as TRU-waste (Table 4.9). Based on the total gross alpha, the average TRU content was about 41 ± 23% and ranged from 2% to 95%. The balance of the gross alpha observed, for some samples, resulted from other alpha emitters such as thorium and curium. However, for many samples the alpha balance could not be accounted for. Also included in Table 4.9 is a check of the gross alpha balance obtained by a ratio of the sum of all the alpha emitters identified by alpha spectroscopy to the gross alpha. This gross alpha balance averaged about 73 ± 28% and ranged from 2.5% to 109%.

The curium isotope, ^{244}Cm, which has a reputation of being difficult to handle because of its high specific activity, was found only in the sludge of basic tanks. The ^{244}Cm activity ranged from 1 to 2 to in excess of 10^5 Bq/g in the sludge samples.

4.5.8 Beta-Gamma Emitters

The most abundant radionuclides observed were ^{137}Cs and ^{90}Sr with their associated short-lived daughters ^{137}Ba and ^{90}Y, respectively. Most of the beta activity observed in the liquid phase of the inactive waste tanks resulted from ^{137}Cs, which was 1-2 decades greater than the water-soluble ^{90}Sr. The liquid-phase gamma activity was also caused by the ^{137}Cs, which was usually several orders of magnitude greater than other water-soluble gamma emitters. With the exception of several of the more acidic liquid wastes, the gamma active europium isotopes were not observed in the liquid phase of the inactive tanks. When present, the europium isotopes were observed only in the sludge of basic tanks. This observation is not surprising considering the low solubility of europium hydroxide (see Table 4.6).

4.6 RCRA EVALUATION FOR INORGANIC SPECIES

The inactive waste tanks are categorized as solid waste and are classified as hazardous if the contents of a tank exhibits any of the following specific characteristics: (1) ignitability, (2) reactivity, (3) corrosivity, or (4) toxicity according to an extraction procedure or EP-TOX test.

Table 4.9. TRU-waste classification for sludge samples

Sludge sample	Gross alpha	TRU status	TRU (Bq/g)	TRU:alpha ratio	All:alpha ratio
T4/S46	374,000	TRU	42,030	0.112	0.685
T3/S43	202,000	TRU	27,030	0.134	1.000
T9/S48	147,000	TRU	19,110	0.130	0.790
T2/S40	202,000	TRU	191,430	0.948	0.948
T1/S37	647,000	TRU	40,830	0.063	0.063
TH4/S58	1,730	Non-TRU	42	0.024	0.024
TH4/S59	2,890	Non-TRU	1,336	0.462	0.462
W5/S75	1,330	Non-TRU	721	0.542	0.802
W6/S80	8,040	Non-TRU	2,276	0.283	0.804
W7/S84	13,600	TRU	3,974	0.292	0.600
W7/H85	7,990	Non-TRU	2,824	0.353	0.670
W8/S88	29,000	TRU	11,560	0.399	0.899
W9/S92	105,000	TRU	41,140	0.392	0.908
W4/S24	5,940	TRU	4,690	0.790	0.790
W4/H26	6,600	TRU	5,901	0.894	0.894
W10/H120	41,100	TRU	21,450	0.522	0.858
W10/S96	83,500	TRU	44,040	0.527	1.088
W3/S19	8,430	TRU	5,610	0.665	0.665
WC17/S106	2,720	Non-TRU	753	0.277	0.880
7562/S98	<1	Non-TRU	0		

A liquid waste is classified as ignitable if the liquid has a flash point that is 60°C or lower. A majority of the inactive waste tanks contained an aqueous-liquid phase and were not tested for ignitability. Two of the waste tanks had an organic layer that would char only when exposed to an open flame. Therefore, none of the waste tanks were classified as ignitable waste.

The inorganic regulatory characterization for the inactive waste tanks is separated into classification of the liquid and solid phase for each tank. The two-category classification provides additional information that may affect decisions concerning waste handling or disposal. For example, a non-RCRA liquid might be removed for a waste tank with a RCRA sludge, and the sludge then processed separately. The following discussion concerning the sludge samples is preliminary because the measurements are based upon the total metal content obtained with a nitric acid leach and not the EP-TOX results. The current available data show only that if a sludge completely dissolves in the EP-TOX acetate buffer, the data are applicable for the determination of RCRA classification because the measurements would be equivalent to the extraction procedure. Therefore, to arrive at the following classifications, the total metal content has been compared to an EP-TOX equivalent (Table 4.2), which accounts for the 20-fold dilution obtained with the extraction procedure. Also note that this evaluation is based on only the inorganic composition and does not include the RCRA status based upon the organic content.

The classification of the inactive waste tank is summarized in Table 4.10 (note that the RCRA status on the sludge samples is preliminary until the EP-TOX data are completed). The current data indicate that 12 of the 27 tanks are RCRA in both the liquid and sludge phases, 4 tanks have a RCRA sludge with an apparent non-RCRA liquid phase, 6 tanks without any sludge have an apparent RCRA liquid, and 3 tanks (W1, W2, and T30) without any sludge appear to be non-RCRA. Two tanks (7860A and WC17), which contained an organic layer, have insufficient data for classification of the liquid phase. Four acidic tanks (TH1, TH3, W14, and W15) and one basic tank (T3) are classified as corrosive waste due to measured pH values that exceed the regulatory limits.

All the sludge samples exceeded the EP-TOX equivalent limits for two or more of the EPA toxic metals (17 tanks). Most of the sludge samples contain chromium, lead, and mercury in amounts that are near or exceed the regulatory limits (16 RCRA in Cr, 15 RCRA in Pb, 14 RCRA in Hg,) and the sludge from 12 tanks are RCRA in all 3. Cadmium was also found in most of the sludge samples, but exceeded the limits in only two of the sludge samples.

The same EPA toxic metals were observed in the liquid samples. However, no lead was present in the basic liquids. Lead was observed in all of the acidic liquid samples and exceeded regulatory limits in several cases. In addition, nickel was observed at or near the regulatory limit in two acidic tanks (W14 and W15). Measured levels of silver and barium, below the regulatory limits, were observed in most of the sludge samples. Low levels of silver and barium were found in most of the acidic liquids and a few of the neutral to slightly basic liquids.

Arsenic, selenium, and thallium were not found at quantitative levels in any of the waste tank samples. Although several of the sludge samples measured positive for arsenic, selenium, and thallium, the signal was most likely the result of some type of interference.

Four tanks (TH1, TH3, W14, and W15) that are classified as hazardous waste based on corrosivity (pH < 2) have no sludge associated with them, but they do contain quantities of chromium, lead, and mercury near or above the regulatory limit. If the pH of these tanks is carelessly raised to a noncorrosive level, the EPA toxic metals present may precipitate and produce a hazardous sludge. One tank (T3), which has a very basic liquid and is classified as corrosive (pH > 12.5), could be made noncorrosive by lowering the pH below (the small change in pH required would not likely effect the solution chemistry).

If the concentration of cyanide and sulfide present in a waste tank is sufficient to generate toxic gases when exposed to pH conditions between 2 and 12.5, the waste has the characteristic of reactivity. The interim guidance for reactive cyanide and sulfide are 250 and 500 mg/kg, respectively. Although, there were some problems with the cyanide and sulfide measurements, none of the liquid samples approached the regulatory limits. At this time only two of the sludge samples have been analyzed for cyanide and sulfide, and both samples were well below the regulatory limits. The remaining sludge samples need to be analyzed, especially for the sulfide which forms insoluble salts with numerous common metals.

Table 4.10. Summary and status of tank analysis for RCRA metals

Tank	Liquid	Sludge	Comments	
1 T4	RCRA	RCRA	Liquid: Cr, Hg	Sludge: (Cd), Cr, Pb, Hg
2 T3	RCRA	RCRA	Liquid: Cr, Hg?, pH	Sludge: (Cd), (Cr), Pb, Hg
3 T9	RCRA	RCRA	Liquid: Hg	Sludge: (Cd), Pb, Hg
4 T2	NR[a]	RCRA	Liquid: (Hg)	Sludge: (Cd), Cr, Pb, Hg
5 T1	NR	RCRA	Liquid: (Hg)	Sludge: (Cd), Cr, Pb, Hg
6 TH1	RCRA	NA	Liquid: (Cr), (Pb), Hg, pH	
7 TH3	RCRA	NA	Liquid: (Cr), (Pb), Hg, pH	
8 W11	NR	RCRA	Liquid:	Sludge: Cr, Pb, Hg
9 TH4	RCRA	RCRA	Liquid: (Cr)*	Sludge: Cr, Hg, (Pb)*
10 W5	RCRA	RCRA	Liquid: (Cr), Hg	Sludge: (Cd), Cr, Pb, Hg
11 W6	RCRA	RCRA	Liquid: Cr	Sludge: Cr, Pb, Hg
12 W7	RCRA	RCRA	Liquid: Cr, Hg?	Sludge: (Cd), Cr, Pb, Hg
13 W8	RCRA	RCRA	Liquid: Cr	Sludge: (Cd), Cr, Pb, Hg
14 W9	RCRA	RCRA	Liquid: Cr	Sludge: (Cd), Cr, Pb, Hg
15 W1	NR	NA		
16 W2	NR	NA		
17 W4	RCRA	RCRA	Liquid: Cr	Sludge: Cd, Cr, Pb, (Hg)
18 W10	RCRA	RCRA	Liquid: Cr, Hg	Sludge: (Cd), Cr, Pb, Hg
19 7860A	RCRA	NA	Liquid: Hg	
20 W3	RCRA	RCRA	Liquid: Cr	Sludge: Cr, (Hg)
21 WC17	RCRA	RCRA	Liquid: Hg	Sludge: Cd, Cr, Pb, Hg
22 W13	RCRA	NA	Liquid: (Cr), Pb, Hg	
23 W14	RCRA	NA	Liquid: Cr, Pb, (Hg), Ni, pH	
24 W15	RCRA	NA	Liquid: Cr, Pb, Hg, (Ni)*, pH	
25 W1A	RCRA	NA	Liquid: Cd, Hg	
26 T30	NR	NA		
27 7562	NR	RCRA	Liquid:	Sludge: (Cd), Cr, Pb, (Hg)

[a]NR–not RCRA; NA–not applicable; ( )–element near RCRA limit; ( )*–element very near RCRA limit; Hg?–no data.

The analysis of liquid samples obtained at different levels within waste tanks indicate that a number of the tanks were not homogeneous but that they actually have concentration gradients which increase from top to bottom of the tanks. This behavior is illustrated in tank TH4 (Table 4.11). Physical parameters related to water-soluble species (density and dissolved solids) or the concentration of several water-soluble species (pH, chromium, and ^{137}Cs) were a good measure of the gradients. The physical measurements, density and dissolved solids, were simple and reliable indicators for the presence of a gradient, with the dissolved solids measurement more sensitive to small changes. The measurement of ^{137}Cs was comparable to the dissolved solids for detecting gradients in the waste tanks. The classification of some of the tanks (such as TH4) that exhibit concentration gradients in the liquid and sludge phase is difficult because of the unknown

bulk RCRA content which would result from mixing of the liquid or solid phase of these tanks. The tanks that exhibit a liquid gradient also have a sludge, and the higher concentrations are observed near the tank sludge. If the liquid phase of these tanks were separated from the sludge and the liquid mixed thoroughly, the redistribution of the metals might result in concentrations below the regulatory limits.

Table 4.11. Measurement gradients observed in tank TH4

Depth (in.)	Density (g/mL)	Dissolved solid (mg/mL)	pH	Cr (mg/L)	^{137}Cs (Bq/mL)
0	1.0002	<0.1	5.8	<0.13	0.54
4	1.0034	1.4	6.9	<0.13	7.4
0-6 in. col.	1.0065	0.5	7.6	<0.36	3.5
39	1.0557	70.6	7.1	1.6	180
72	1.0832	103.8	7.8	5.6	240

4.7 RECOMMENDATION FOR ADDITIONAL ACTIVITIES

The analytical support for the radioactive waste tanks was a learning experience for this laboratory, and to our knowledge, a project of this type and requirements has not been attempted elsewhere. The identification of problem areas has been a positive outcome of this work. The problem areas include inappropriate regulatory procedures for highly radioactive samples, complexity of the samples (each tank and sometimes each sample required additional method development), unrealistic expectations for QA/QC measurements for solid waste, and the expense in both time and money.

Additional studies are needed to improve some of the procedures and reduce radiation exposure, understand the complex chemistry, and identify interferences. Improved methods need to be developed for the measurement of cyanide and sulfide. The long-term results of radiation exposure of the sample need to be studied, and the effects the radiolytic products may have on the analysis of waste tank sample need to be identified. Both reducing and oxidizing environments were observed during the analysis of several waste tank samples. The characteristics of these chemically active samples need to be better understood to ensure valid results for the measurement of species such as mercury and sulfide.

An inductively coupled plasma-mass spectrometer (ICP-MS) is designed for the rapid multielement determination of elemental species at low concentration. ICP-MS is complementary to both ICP-emission spectroscopy and GFAA and in many ways combines the beneficial characteristics of both techniques. Although the initial expense for an ICP-MS is high (>$250K), the advantages realized would include a significant reduction in labor costs (it has taken nearly a year for the 10 EPA metals in 87 samples), faster sample turnaround (typically, 20 elements can be determined in 1 to 3 min depending upon the precision

required), and much more information from a single analysis (up to 75 elements for a single analysis). With the excellent detection limits possible, ICP-MS could provide all ten regulatory metals and the treatability metals in a single analysis, which currently requires three separate analytical techniques to complete (ICP, GFAA, and mercury CV). Also, the single analysis would drastically reduce the sample handling and radiation exposure to the analyst. Therefore, if the analytical support for the characterization of radioactive waste is to continue, it is recommended that our analytical capabilities be upgraded with an ICP-MS.

The determination of the anionic species in the waste tanks is important to both the treatability studies and the understanding of the distribution of regulatory metals found in the waste tanks. The measurement of the anions for this project was done in a facility not equipped to handle the radioactivity present and required very high dilution before analysis by ion chromatography. In many cases this large dilution prevented the measurement of important species because of insufficient sensitivity. In addition to the measurement of common anions, the ion chromatograph could be used for the measurement of cyanide and sulfide with good sensitivity and better selectivity than could be achieved for this current project. Therefore, it is recommended that an ion chromatograph be obtained for one of the facilities capable of handling the higher levels of radioactivity.

4.8 ADDENDUM: PRELIMINARY EP-TOX RESULTS

Just prior to the release of this report some additional data was obtained which was needed to establish the RCRA status of several of the inactive waste tanks. Sufficient time was not available to integrate this information into the body of the report. However, the data had a significant impact on the RCRA status of several tanks and was included in this report so that regulatory classification of the waste tanks discussed could be concluded.

In an effort to identify the gross RCRA status for the inactive waste tanks it was necessary to complete the EP-TOX measurements on the sludge from several of the tanks which contained a non-RCRA liquid phase and an apparent RCRA sludge (the apparent RCRA classification is based upon the total metal content). Sufficient sample was not available for the waste tanks in question, which included T1, T2, and 7562, and these tanks were re-sampled for the EP-TOX measurements. The total metal content and the percent extracted, in Table 4.12, are based upon the original sludge samples. Only the metals which had a total metal content that exceeded the EP-TOX equivalent limit (for most sludge samples Cr, Pb, and Hg exceeded the limits) were measured in the EP-TOX leachate.

The results of the extraction procedure, which are summarized in Table 4.12, show that the toxic metals of interest (Cr, Pb, and Hg) are weakly extracted into the 0.05 M acetate buffer. Although approximately 50% of the solid material from the sludges below dissolved in the buffer solution, the toxic metals are present as insoluble species.

Table 4.12. EP-TOX results for RCRA metals

Sample ID	Weight (g)	Final volume (mL)	0.05 M HOAc[a] (mL)	Initial pH	Cr Total (μg/g)	Cr EP-TOX (μg/mL)	Cr Ext. (%)	Pb Total (μg/g)	Pb EP-TOX (μg/mL)	Pb Ext. (%)	Hg Total (μg/g)	Hg EP-TOX (μg/mL)	Hg Ext. (%)
T1/S145[c]	10.0574	202	38.4	9.7	134	0.03	0.45	863	<0.21	<0.5	74.	0.04	1.1
T2/S147[c]	10.0680	202	30.8	9.5	184	0.06	0.65	354	<0.21	<1.2	70.	0.01	0.3
T4/S046	10.2694	202	39.9	9.8	102	<0.02	<0.39	512	<0.21	<0.8	585.	6.4	21.5
TH4/S058	10.2020	204	25.0	9.5	72	<0.02	<0.56	38	0.39	20.5	3.6	0.01	4.4
TH4/S059	10.8130	216	28.0	9.8	290	<0.02	<0.14	99	<0.21	<4.2	4.9	0.01	3.3
7562/L143[c]	9.9141	200	0.5	6.7	2300	0.02	0.02	900	<0.21	<0.5	1.8	<0.01	<9.0

[a]0.05 M acetic acid.
[b]Extracted column summarizes the % extracted.
[c]Tanks T1, T2, and 7562 were resampled for the EP-TOX measurements. For the sample 7562/L143, the original sample was labeled as a sludge, however, the sample actually consisted primarily as a liquid with a fine suspended precipitate.

Therefore, supported by the EP-TOX results for the inorganic metals, the waste tanks T1, T2, and 7562, which had non-RCRA liquids with an apparent RCRA sludge (based on total metal content), are not RCRA according to the inorganic contents. Several other sludges, listed in Table 4.12, demonstrated a similar behavior for the extraction of the toxic metals.

4.9 REFERENCES

1. USEPA, *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, D.C., November 1986.

2. USEPA Contract Laboratory Program, *Statement of Work for Inorganic Analysis*, SOW No. 787.

3. USEPA, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, rev. March 1983.

4. A. O. Allen, *The Radiation Chemistry of Water and Aqueous Solutions*, D. Van Nostrand Company, Inc., Princeton, N.J., 1961.

5. J. C. White and W. J. Ross, *Separations by Solvent Extraction with Tri-n-octylphosphine Oxide*, NAS-NS 3102, Oak Ridge National Laboratory, Oak Ridge, Tenn., February 1961.

6. W. Slavin, D. C. Manning, and G. R. Carnrick, "The Stabilized Temperature Platform Furnace," *Atomic Spectroscopy*, 2(5), 137-145, 1981.

7. G. R. Carnrick and W. Slavin, "Modern Graphite Furnace AA: Part 2," *American Laboratory*, pp. 90-95, February 1989.

5. RCRA STATUS OF INACTIVE LIQUID LOW-LEVEL RADIOACTIVE WASTE TANKS

5.1 INTRODUCTION

A summary of the Resource Conservation and Recovery Act (RCRA) hazardous characteristics and listed organic constituents is presented in Table 5.1. A more complete discussion of the inorganic and organic species detected, including differences found between the liquid and sludge phases, can be found in Sects. 2, 3, and 4 of this report. Table 5.1 is meant to provide only a quick overview of the RCRA status of the contents of those tanks sampled in this study.

The first two columns summarize the RCRA hazardous characteristics of the liquid and sludge phases, if applicable. It should be noted that the measurements listed for the sludge samples are total metal content obtained with a nitric acid leach and not the Extraction Procedure (EP) Toxicity[1] results. Classification as EP toxic is based upon an EP Toxicity equivalent; in other words, it is a value which accounts for the 20-fold dilution of the EP Toxicity procedure and represents the concentration that would result in classification of the sample as RCRA hazardous if the sludge completely dissolves in the acetate buffer(s) used in the EP Toxicity or Toxicity Characteristic Leaching Procedure (TCLP).

5.2 RCRA HAZARDOUS CHARACTERISTICS

None of the waste tanks contained a RCRA ignitable waste; only 5 (T3, TH1, TH3, W14, and W15) were classified as RCRA corrosive. Of the 27 tanks sampled, the liquid contents of 7 (T1, T2, W1, W2, W11, T30, and 7862) did not exceed the EP Toxicity regulatory level for at least one of the 8 RCRA regulated metals. In those tanks containing a sludge that could be sampled, all exceeded the EP Toxicity equivalent. Until the EP Toxicity (or TCLP) data are completed, the RCRA status of the sludge samples based upon content of regulated metals must be considered preliminary. Three tanks (W1, W2, and T30) do not contain any RCRA characteristically hazardous material. It can also be seen from Table 5.1 that these tanks do not contain any RCRA listed organic constituents. Although methylene chloride and toluene were detected at concentrations ranging from 10-15 ppb, they were also detected in the blanks samples at similar concentrations.

5.3 RCRA ORGANIC CONSTITUENTS

Determination of the RCRA status of the tank contents based upon the types of organic constituents present involves a comparison with the list of RCRA hazardous wastes contained in the Code of Federal Regulations (CFR). These are listed at 40 CFR Pts. 261.31, 261.32, and 261.33. Whether wastewater containing the listed organic constituents is considered a RCRA hazardous waste depends upon the manner in which the waste was generated. For instance, a listed solvent or a discarded commercial chemical product

Table 5.1. RCRA characteristics and constituents of inactive waste storage tanks

Tank No.	Hazardous characteristic		TCLP organic constituents				Other organic constituents detected			
	Liquid	Sludge	Volatile	ug/L	Semivolatile	(ug/L)	Volatiles (DAI-GC)[a]	ug/L	Semivolatiles[b]	ug/L
T1	None	EP-Toxicity (Cr, Pb, Hg)	Carbon disulfide	91	None		None		Di-n-butyl phthalate	3,500
T2	None	EP-Toxicity (Cr, Pb, Hg)	None		2,4,5-Trichlorophenol	110	None		Ethyl hexyl phthalate	24,000
									Dibutyl phthalate	4,600 (J)
									Diethylephythalate	2,800 (J)
									Naphthalene	2,300 (J)
T3	EP-Tox (Cr) Corrosive (pH=12.7)	EP-Toxicity (Pb, Hg)	None		None		Acetone	4,000	Di-n-butyl phthalate	3,100 (J)
							Methanol	28,000	Diethyl phthalate	2,400 (J)
							N-Butyl alcohol	80,000		
T4	EP-Toxicity (Cr, Hg)	EP-Toxicity (Cr, Pb, Hg)	Toluene	98 (B)[b]	None		Acetone	8,000	Dibutyl phthalate	3,400 (J)
			Trichloroethylene	3 (J)			Methanol	46,000	Diethylphathalate	2,400 (J)
			Chlorobenzene	4 (J)			N-Butyl alcohol	6,000		
			Methylene chloride	14 (B)			Xylene	170		
T9	EP-Toxicity (Hg)	EP-Toxicity (Pb, Hg)	Methylene chloride	600 (BE)	None		Methanol	17,000	Dibutyl phthalate	3,700 (J)
			Carbon Disulfide	4 (J)					Diethyl phthalate	3,000 (J)
			Chlorobenzene	6						
			Toluene	19 (B)						
			Trichloroethylene	5 (J)						
TH1	EP-Tox (Hg) Corrosive (pH=1.8)	NA	Methylene chloride	6 (B)	None		None		None	
			Toluene	45 (B)						
TH3	EP-Tox (Hg) Corrosive (pH=1.8)	NA	Chlorobenzene	2 (J)	None		None		None	
			Methylene chloride	8 (B)						
			Toluene	65 (B)						
TH4	EP-Toxicity (Cr)	EP-Toxicity (Cr, Hg, Pb)	Toluene	79 (B)	None		Acetone	22,000	Various phthalates and polycylic	
			Carbon tetrachloride	11			Methanol	27,000	aromatic hydrocarbons (PAH) were	
			Chlorobenzene	8			Methyl isobutyl keton	15,000	detected in the sludge ranging	
			Trichloroethylene	5			n-Butyl alcohol	20,000	from 4 to 30 ppm.	
			Methylene chloride	210 (B)						
			Methyl ethyl ketone	2,000						
W1	None	NA	Methylene chloride	11 (B)	None		None		None	
			Toluene	11 (B)						

Table 5.1 (continued)

Tank No.	Hazardous characteristic		TCLP organic constituents				Other organic constituents detected			
	Liquid	Sludge	Volatile	ug/L	Semivolatile	(ug/L)	Volatiles (DAI-GC)[a]	ug/L	Semivolatiles[b]	ug/L
W1A	EP-Toxicity (Cd, Hg)	NA	Toluene	63	None		Methanol	7,000	None	
			Trichloroethylene	20						
			Methylene chloride	4 (JB)						
W2	None	NA	Methylene chloride	10 (B)	None		None		None	
			Toluene	15 (B)						
W3	EP-Toxicity (Cr)	EP-Toxicity (Cr)	Toluene	37 (B)	None		Acetone	7,000	None	
			Trichloroethylene	14 (B)			Methanol	40,000		
			Chlorobenzene	8			n-Butyl alcohol	7,000		
			Methylene chloride	4 (JB)						
			Tetrachloroethylene	8 (B)						
			Methyl ethyl ketone	5,000						
W4	EP-Toxicity (Cr)	EP-Toxicity (Cd, Cr, Pb)	Trichloroethylene	14	None		Methyl isobutyl ketone	7,000	Ethyl hexyl phthalate	11,000
			Methylene chloride	10 (B)					Dibutyl phthalate	2,100
			Toluene	46 (B)						
W5	EP-Toxicity (Hg)	EP-Toxicity (Cr, Pb, Hg)	Carbon tetrachloride	68 (B)	None		None		Ethyl hexyl phthalate	28,000
			Trichloroethylene	138						
			Methylene chloride	6 (B)						
			Tetrachloroethylene	267 (B)						
W6	EP-Toxicity (Cr)	EP-Toxicity (Cr, Pb, Hg)	Toluene	10 (B)	None		None		Ethyl hexyl phthalate	31,000
			Carbon tetrachloride	4 (JB)						
			Trichloroethylene	82						
			Methylene chloride	9 (B)						
			Methyl ethyl ketone	75						
			Tetrachloroethylene	507 (BE)						
W7	EP-Toxicity (Cr)	EP-Toxicity (Cr, Pb, Hg)	Carbon tetrachloride	1088 (BE)	None		Methanol	14,000	Ethyl hexyl phthalate	57,000
			Toluene	14 (B)					Dibutylphthalate	2,200
			Methylene chloride	7 (B)						
			Tetrachloroethylene	39 (B)						
W8	EP-Toxicity (Cr)	EP-Toxicity (Cr, Pb, Hg)	Methyl ethyl keton	1,000	None		Acetone	3,000	Naphthalene	2,600
			Methylene chloride	12 (B)			Methanol	1,000		
			Toluene	4 (BJ)			n-Butyl alcohol	2,000		

Table 5.1 (continued)

Tank No.	Hazardous characteristic		TCLP organic constituents				Other organic constituents detected			
	Liquid	Sludge	Volatile	ug/L	Semivolatile	(ug/L)	Volatiles (DAI-GC)[a]	ug/L	Semivolatiles[b]	ug/L
W9	EP-Toxicity (Cr)	EP-Toxicity (Cr, Pb, Hg)	Toluene	9 (B)	None		None		Ethyl hexyl phthalate	160,000
			Chlorobenzene	5					Fluoranthene	1,100
			Trichloroethylene	5						
			Methylene chloride	13 (B)						
			Tetrachloroethylene	4 (J)						
W10	EP-Toxicity (Cr, Hg)	EP-Toxicity (Cr, Pb, Hg)	Toluene	26 (B)	None		Acetone	3,000	Ethyl hexyl phthalate	130,000
			Chlorobenzene	5 (B)			Methanol	41,000	Dibutyl phthalate	2,600
			Trichloroethylene	81			n-Butyl alcohol	1,000	Floranthene	8,000
			Methylene chloride	19 (B)					Benyo(a) pyrene	1,900
			Tetrachloroethylene	48						
			Methyl ethyl ketone	1,000						
W11	None	EP-Toxicity (Cr, Pb, Hg)	Chlorobenzene	5 (J)	None		None		Ethyl hexyl phthalate	38,000
			Methylene chloride	13 (B)					Dibutyl phthalate	17,400
			Toluene	24 (B)					Benzo(a) anthracene	44,000
			Trichloroethylene	4 (J)					Benzo(a) pyrene	64,000
									Chrysene	130,000
									Fluoranthene	240,000
W13	EP-Toxicity (Pb, Hg)	NA	Toluene	100	None		Methanol	11,000	None	
			Chlorobenzene	4 (J)						
			Methylene chloride	24 (B)						
W14	EP-Toxicity (Cr, Pb) Corrosive (pH=0.5)	NA	Toluene	21	None		Methanol	15,000	None	
			Chlorobenzene	6						
			Methylene chloride	23 (B)						
			Tetrachloroethylene	5						
			Trichloroethylene	6						
W15	EP-Toxicity (Cr, Pb, Hg) Corrosive (pH=0.2)	NA	Trichloroethylene	4 (J)	None		Methanol	7,000	None	
			Chlorobenzene	5						
			Methylene chloride	24 (B)						
			Toluene	29						
WC17	EP-Toxicity (Hg)	EP-Toxicity (Cd, Cr, Pb, and Hg)	Trichloroethylene	920 (JB)	None		Acetone, xylene, styrene vinyl acetate in both liquid and organic phases (VOA analysis)		Dibutyl phthalate	74,000
			Methylene chloride	6,600 (JB)					PCB-Aroclor 1254	20
									PCB-Aroclor 1260	13

Table 5.1 (continued)

Tank No.	Hazardous characteristic		TCLP organic constituents				Other organic constituents detected			
	Liquid	Sludge	Volatile	ug/L	Semivolatile	(ug/L)	Volatiles (DAI-GC)[a]	ug/L	Semivolatiles[b]	ug/L
T30	None	NA	Trichloroethylene	4 (J)	None		Methanol	11,000	None	
			Methylene chloride	24 (B)						
			Tetrachloroethylene	9						
			Toluene	10						
7862	None	EP-Toxicity (Cr, Pb)	Toluene	10	None		Methanol	14,000	None	
			Methylene chloride	18 (B)						
			Tetrachloroethylene	4 (J)						
7860A	EP-Toxicity (Hg)	NA	Methylene chloride[c]	170 (JB)	None		Acetone[c]	2,300	Ethyl hexyl phthalate	630,000
			Tetrachloroethylene[c]	40,000 (E)			Methanol[c]	1,700	Dibutyl phthalate	62,000
			Toluene[c]	85 (J)			Methyl isobutyl ketone[c]	5	Naphthalene	92,000
			Trichloroethylene[c]	39,000 (E)			n-Butyl alcohol[c]	1,200		
			Methyl ethyl ketone[c]	2,000			Xylene[c]	9,500		
			Ethyl benzene[c]	450						

[a]DAI-GC, Direct Aqueous Injection-Gas Chromatography.
[b]For an explanation of data qualifiers see Chapter 3, Organic Analysis Characterization.
[c]Concentrations expressed in mg/L.

is an RCRA waste if it meets certain conditions as defined in the regulations, but its presence in a process wastewater does not mean that such wastewaters are automatically classified as RCRA listed waste. The manner in which the constituents were discarded is the sole determinant. Another method whereby a wastewater that contains listed RCRA organic constituents may be classified as RCRA waste is by applications of the TCLP. Currently, the TCLP has not been approved for any testing outside the land disposal restriction program; nevertheless, the regulatory levels of various organic constituents can be used as a guide. The concentration of F001-F005 spent solvent wastes treatment standards for wastewater may be compared to concentrations found in the contents of the inactive liquid low-level waste (LLLW) tanks.

The history of discharges of chemical constituents into the LLLW collection system is not well documented; therefore, little can be said concerning the origin of organic constituents. Based upon the organic analyses presented in this study, the types of RCRA organic waste present in the tank contents are primarily various solvents that could have been discarded as F-listed solvents (exceptions are the various phthalates and polycyclic aromatic hydrocarbons (PAHs)). In the absence of knowledge concerning the solvent content (volume percent) when discarded, the concentrations are compared to regulatory thresholds contained in the TCLP. An additional consideration pertaining to F003-listed solvents that are mixed with a solid waste is described in 40 CFR Pt. 261.3 (a)(2)iii. It states that a mixture of a solid waste and a hazardous waste that is listed solely because it exhibits one or more hazardous waste characteristics is no longer considered RCRA hazardous if the mixture does not exhibit the hazardous waste characteristic. This exclusion can be applied to the contents of those tanks that contain F003 listed constituents since they are listed because of the ignitability characteristic.

Several of the tanks contain one or more organic constituents that are listed as F003. Since these solvents are listed under F003 because of ignitability and since none of the tanks exhibited this RCRA characteristic, those tanks containing F003-listed solvents are excluded from RCRA by application of the "mixture" rule, 40 CFR Pt. 261.3 (a)(2)iii.

Application of the treatment standards or regulatory thresholds for those F001-F005 constituents listed in the TCLP and detected in tank samples reveals that most tanks contain trace amounts of RCRA organic constituents; however, the concentrations are below regulatory thresholds. Furthermore, because most are also detected in the blank samples, their presence in the analytical sample is probably an artifact.

Several tanks (TH4, W3, W8, and W10) contain methyl ethyl ketone (MEK) at concentrations ranging from 1000 to 5000 ppb (the TCLP regulatory threshold for wastewaters is 50 ppb; for wastes other than wastewater the treatment standard is 750 ppb). In several other tanks (T9, W5, W6, W7, WC17), tetrachloroethylene, trichloroethylene, methylene chloride, and/or carbon tetrachloride were detected at concentrations above TCLP standards for wastewater and/or other wastes (tetrachloroethylene = 79, 50 ppb; trichloroethylene = 62, 91 ppb; methylene chloride = 200, 960 ppb; carbon tetrachloride = 50, 960 ppb).

In tank 7860A, methylene chloride, tetrachloroethylene, toluene, trichloroethylene, ethyl benzene, and methyl ethyl ketone were detected at concentrations ranging from 85 ppm (toluene) to 40,000 ppm (tetrachloroethylene).

In summary, it can be concluded that ten of the tanks sampled (T9, TH4, W3, W5, W6, W7, W8, W10, WC17, and 7860A) contain volatile organic RCRA constituents above the regulatory thresholds listed in the TCLP. Also, several of the tanks contain milligram-per-liter quantities of F003 listed solvents (various alcohols and ketones) but the contents are no longer ignitable; thus, they are not classified as containing RCRA waste because of the presence of F003-listed solvents. Slightly greater than one-half (16) of the tanks sampled contained concentrations of RCRA listed semivolatile organic constituents at concentrations greater than 1 ppm. These consisted primarily of various phthalates and PAHs. The origin of the chemical constituents are unknown; however, it is unlikely that they were discarded as P- or U-listed RCRA waste.

Tanks that contained no RCRA characteristically hazardous (or potential RCRA characteristic as determined by the EP Toxicity equivalent of total RCRA metal concentration) or RCRA listed organic constituents included W1, W2, and T30. At this stage of analysis, the remaining tanks should be considered as containing RCRA waste.

5.4 REFERENCES

1. U. S. Environmental Protection Agency, 40 CFR, Pts. 190-299, Office of the Federal Register National Archives and Records Administration, Washington, July 1988.

APPENDIX

LISTING OF ORGANIC ANALYTICAL DATA HITS FOR INACTIVE TANKS AT ORNL

Table A.1. Listing of organic analytical data hits for tank T-1

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	T-1/L-035A	ACETONE	B	51	UG/L
	T-1/L-035A	BENZENE		130	UG/L
	T-1/L-035A	CARBON DISULFIDE		91	UG/L
	T-1/L-035A	CARBON TETRACHLORIDE	J	2	UG/L
	T-1/L-035A	CHLOROFORM	B	12	UG/L
	T-1/L-035A	METHYLENE CHLORIDE	BE	1300	UG/L
	T-1/L-035A	TOLUENE	B	71	UG/L
	T-1/L-035A	TRICHLOROETHENE	J	4	UG/L
	T-1/L-036A	ACETONE	B	27	UG/L
	T-1/L-036A	BENZENE	J	5	UG/L
	T-1/L-036A	CHLOROFORM	B	6	UG/L
	T-1/L-036A	METHYLENE CHLORIDE	BE	970	UG/L
	T-1/L-036A	TOLUENE	B	12	UG/L
SVO	T-1/L-035	BIS(2-ETHYLHEXYL)PHTHALATE	J	400	UG/L
	T-1/L-035	DI-N-BUTYLPHTHALATE	J	75	UG/L
	T-1/L-036	BIS(2-ETHYLHEXYL)PHTHALATE	J	190	UG/L
	T-1/L-036	DI-N-BUTYLPHTHALATE	J	76	UG/L
	T-1/S-037	DI-N-BUTYLPHTHALATE	J	3500	UG/KG

Table A.2. Listing of organic analytical data hits for tank T-2

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	T-2/L-038A	ACETONE	B	20	UG/L
	T-2/L-038A	BENZENE	J	3	UG/L
	T-2/L-038A	CHLOROFORM	BE	340	UG/L
	T-2/L-038A	METHYLENE CHLORIDE	BE	1000	UG/L
	T-2/L-038A	TOLUENE	B	12	UG/L
	T-2/L-039A	ACETONE	B	22	UG/L
	T-2/L-039A	CHLOROFORM	BE	370	UG/L
	T-2/L-039A	METHYLENE CHLORIDE	BE	820	UG/L
	T-2/L-039A	TOLUENE	B	7	UG/L
	T-2/L-112A	ACETONE	B	17	UG/L
	T-2/L-112A	CHLOROFORM	BE	310	UG/L
	T-2/L-112A	METHYLENE CHLORIDE	BE	860	UG/L
	T-2/L-112A	TOLUENE	B	8	UG/L
SVO	T-2/L-038	BIS(2-ETHYLHEXYL)PHTHALATE	J	200	UG/L
	T-2/L-038	2-NITROPHENOL	J	170	UG/L
	T-2/L-038	2,4-DICHLOROPHENOL	J	100	UG/L
	T-2/L-038	2,4,5-TRICHLOROPHENOL	J	99	UG/L
	T-2/L-039	BIS(2-ETHYLHEXYL)PHTHALATE	J	300	UG/L
	T-2/L-039	2-NITROPHENOL	J	200	UG/L
	T-2/L-039	2,4-DICHLOROPHENOL	J	140	UG/L
	T-2/L-039	2,4,5-TRICHLOROPHENOL	J	110	UG/L
	T-2/L-112	BIS(2-ETHYLHEXYL)PHTHALATE	J	280	UG/L
	T-2/L-112	DI-N-BUTYLPHTHALATE	J	24	UG/L
	T-2/L-112	2-NITROPHENOL	J	180	UG/L
	T-2/L-112	2,4,5-TRICHLOROPHENOL	J	120	UG/L
	T-2/S-040	BIS(2-ETHYLHEXYL)PHTHALATE		24000	UG/KG
	T-2/S-040	DI-N-BUTYLPHTHALATE	J	4600	UG/KG
	T-2/S-040	DIETHYLPHTHALATE	J	2800	UG/KG
	T-2/S-040	NAPHTHALENE	J	2300	UG/KG

Table A.3. Listing of organic analytical data hits for tank T-3

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	T-3/L-042	ACETONE		4	MG/L
	T-3/L-042	METHYL ALCOHOL		28	MG/L
	T-3/L-042	N-BUTYL ALCOHOL		80	MG/L
VOA	T-3/L-042A	ACETONE	B	180	UG/L
	T-3/L-042A	CHLOROFORM	JB	3	UG/L
	T-3/L-042A	METHYLENE CHLORIDE	JB	3	UG/L
	T-3/L-042A	TOLUENE	B	9	UG/L
SVO	T-3/L-042	BENZYL ALCOHOL	J	120	UG/L
	T-3/S-043	DI-N-BUTYLPHTHALATE	J	3100	UG/KG
	T-3/S-043	DIETHYLPHTHALATE	J	2000	UG/KG

Table A.4. Listing of organic analytical data hits for tank T-30

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	T-30/L-121	METHYL ALCOHOL		11	MG/L
VOA	T-30/L-121A	ACETONE	B	21	UG/L
	T-30/L-121A	BENZENE	J	3	UG/L
	T-30/L-121A	METHYLENE CHLORIDE	B	24	UG/L
	T-30/L-121A	TETRACHLOROETHENE		9	UG/L
	T-30/L-121A	TOLUENE		10	UG/L
	T-30/L-121A	TRICHLOROETHENE	J	4	UG/L
SVO	T-30/L-121	BIS(2-ETHYLHEXYL)PHTHALATE	J	37	UG/L

Table A.5. Listing of organic analytical data hits for tank T-4

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	T-4/L-044	ACETONE		7	MG/L
	T-4/L-044	ETHYL ALCOHOL		37	MG/L
	T-4/L-044	I-PROPYL ALCOHOL		3	MG/L
	T-4/L-044	METHYL ALCOHOL		46	MG/L
	T-4/L-044	N-BUTYL ALCOHOL		3	MG/L
	T-4/L-045	ACETONE		8	MG/L
	T-4/L-045	ETHYL ALCOHOL		37	MG/L
	T-4/L-045	I-PROPYL ALCOHOL		3	MG/L
	T-4/L-045	METHYL ALCOHOL		27	MG/L
	T-4/L-045	N-BUTYL ALCOHOL		6	MG/L
	T-4/L-111	ACETONE		7	MG/L
	T-4/L-111	ETHYL ALCOHOL		37	MG/L
	T-4/L-111	I-PROPYL ALCOHOL		3	MG/L
	T-4/L-111	METHYL ALCOHOL		42	MG/L
	T-4/L-111	N-BUTYL ALCOHOL		3	MG/L
VOA	T-4/L-044A	ACETONE	BE	400	UG/L
	T-4/L-044A	BENZENE		7	UG/L
	T-4/L-044A	BROMODICHLOROMETHANE	B	7	UG/L
	T-4/L-044A	CHLOROFORM	B	170	UG/L
	T-4/L-044A	ETHYLBENZENE		20	UG/L
	T-4/L-044A	METHYLENE CHLORIDE	B	12	UG/L
	T-4/L-044A	TOLUENE	B	78	UG/L
	T-4/L-044A	4-METHYL-2-PENTANONE		11	UG/L
	T-4/L-045A	ACETONE	BE	220	UG/L
	T-4/L-045A	BENZENE		10	UG/L
	T-4/L-045A	CHLOROBENZENE	J	4	UG/L
	T-4/L-045A	CHLOROFORM	B	160	UG/L
	T-4/L-045A	ETHYLBENZENE		20	UG/L
	T-4/L-045A	METHYLENE CHLORIDE	B	14	UG/L
	T-4/L-045A	TOLUENE	B	98	UG/L
	T-4/L-045A	TRICHLOROETHENE	J	3	UG/L
	T-4/L-045A	XYLENE (TOTAL)		170	UG/L
	T-4/L-045A	2-HEXANONE		20	UG/L
	T-4/L-045A	4-METHYL-2-PENTANONE		10	UG/L
	T-4/L-111A	ACETONE	B	72	UG/L
	T-4/L-111A	BENZENE		7	UG/L
	T-4/L-111A	CHLOROFORM	B	120	UG/L
	T-4/L-111A	ETHYLBENZENE		5	UG/L
	T-4/L-111A	METHYLENE CHLORIDE	B	12	UG/L
	T-4/L-111A	TOLUENE	B	60	UG/L
	T-4/L-111A	XYLENE (TOTAL)		36	UG/L
SVO	T-4/S-046	DI-N-BUTYLPHTHALATE	J	3400	UG/KG
	T-4/S-046	DIETHYLPHTHALATE	J	2400	UG/KG

Table A.6. Listing of organic analytical data hits for tank T-9

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	T-9/L-047	METHYL ALCOHOL		17	MG/L
VOA	T-9/L-047A	ACETONE	B	23	UG/L
	T-9/L-047A	BENZENE		11	UG/L
	T-9/L-047A	CARBON DISULFIDE	J	4	UG/L
	T-9/L-047A	CHLOROBENZENE		6	UG/L
	T-9/L-047A	CHLOROFORM	B	13	UG/L
	T-9/L-047A	METHYLENE CHLORIDE	BE	600	UG/L
	T-9/L-047A	TOLUENE	B	19	UG/L
	T-9/L-047A	TRICHLOROETHENE	J	5	UG/L
	T-9/L-047A	1,1-DICHLOROETHENE	J	2	UG/L
SVO	T-9/L-047	BENZOIC ACID	J	340	UG/L
	T-9/L-047	BENZYL ALCOHOL	J	160	UG/L
	T-9/S-048	DI-N-BUTYLPHTHALATE	J	3700	UG/KG
	T-9/S-048	DIETHYLPHTHALATE	J	3000	UG/KG

Table A.7. Listing of organic analytical data hits for tank TH-1

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	TH-1/L-049	ACETONE	B	18	UG/L
	TH-1/L-049	METHYLENE CHLORIDE	B	6	UG/L
	TH-1/L-049	TOLUENE	B	44	UG/L
	TH-1/L-050	ACETONE	B	15	UG/L
	TH-1/L-050	METHYLENE CHLORIDE	B	6	UG/L
	TH-1/L-050	TOLUENE	B	45	UG/L

Table A.8. Listing of organic analytical data hits for tank TH-3

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	TH-3/L-052	ACETONE	B	16	UG/L
	TH-3/L-052	METHYLENE CHLORIDE	B	8	UG/L
	TH-3/L-052	TOLUENE	B	7	UG/L
	TH-3/L-053	ACETONE	B	14	UG/L
	TH-3/L-053	METHYLENE CHLORIDE	B	6	UG/L
	TH-3/L-053	TOLUENE	B	65	UG/L
	TH-3/L-113	ACETONE	B	45	UG/L
	TH-3/L-113	CHLOROBENZENE	J	2	UG/L
	TH-3/L-113	METHYLENE CHLORIDE	JB	4	UG/L
SVO	TH-3/L-053	BIS(2-ETHYLHEXYL)PHTHALATE	J	79	UG/L

Table A.9. Listing of organic analytical data hits for tank TH-4

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	TH-4/L-055	METHYL ALCOHOL		13	MG/L
	TH-4/L-056	ACETONE		22	MG/L
	TH-4/L-056	METHYL ALCOHOL		27	MG/L
	TH-4/L-056	METHYL ETHYL KETONE		2	MG/L
	TH-4/L-056	METHYL I-BUTYL KETONE		15	MG/L
	TH-4/L-056	N-BUTYL ALCOHOL		20	MG/L
	TH-4/L-057	METHYL ALCOHOL		12	MG/L
	TH-4/L-117	METHYL ALCOHOL		20	MG/L
VOA	TH-4/L-055A	ACETONE	B	22	UG/L
	TH-4/L-055A	BENZENE		3	UG/L
	TH-4/L-055A	CHLOROFORM		6	UG/L
	TH-4/L-055A	METHYLENE CHLORIDE	B	8	UG/L
	TH-4/L-055A	TOLUENE	B	4	UG/L
	TH-4/L-056A	ACETONE	BE	630	UG/L
	TH-4/L-056A	BENZENE		149	UG/L
	TH-4/L-056A	CARBON TETRACHLORIDE		11	UG/L
	TH-4/L-056A	CHLOROFORM		11	UG/L
	TH-4/L-056A	METHYLENE CHLORIDE	B	8	UG/L
	TH-4/L-056A	TOLUENE	B	18	UG/L
	TH-4/L-056A	2-BUTANONE		85	UG/L
	TH-4/L-056A	4-METHYL-2-PENTANONE	BE	2682	UG/L
	TH-4/L-057A	ACETONE	B	71	UG/L
	TH-4/L-057A	BENZENE		115	UG/L
	TH-4/L-057A	CHLOROFORM		7	UG/L
	TH-4/L-057A	METHYLENE CHLORIDE	B	7	UG/L
	TH-4/L-057A	TOLUENE	B	21	UG/L
	TH-4/L-057A	XYLENE (TOTAL)		10	UG/L
	TH-4/L-057A	4-METHYL-2-PENTANONE	BE	397	UG/L
	TH-4/L-057B	ACETONE	B	54	UG/L
	TH-4/L-057B	BENZENE		118	UG/L
	TH-4/L-057B	CHLOROFORM		5	UG/L
	TH-4/L-057B	METHYLENE CHLORIDE	B	6	UG/L
	TH-4/L-057B	TOLUENE	B	23	UG/L
	TH-4/L-057B	XYLENE (TOTAL)		22	UG/L
	TH-4/L-057B	4-METHYL-2-PENTANONE	B	179	UG/L
	TH-4/L-116A	ACETONE	B	150	UG/L
	TH-4/L-116A	BENZENE		10	UG/L
	TH-4/L-116A	CHLOROBENZENE		8	UG/L
	TH-4/L-116A	METHYLENE CHLORIDE	B	6	UG/L
	TH-4/L-116A	TOLUENE	B	13	UG/L
	TH-4/L-116A	TRICHLOROETHENE		5	UG/L
	TH-4/L-116A	4-METHYL-2-PENTANONE	B	298	UG/L
	TH-4/L-117A	ACETONE	B	16	UG/L
	TH-4/L-117A	BENZENE		21	UG/L
	TH-4/L-117A	CHLOROFORM		4	UG/L
	TH-4/L-117A	METHYLENE CHLORIDE	B	210	UG/L

Table A.9 (continued)

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	TH-4/L-117A	TOLUENE	B	79	UG/L
	TH-4/L-117A	4-METHYL-2-PENTANONE	B	9	UG/L
SVO	TH-4/L-056	ACENAPHTHENE	J	42	UG/L
	TH-4/L-056	NAPHTHALENE	J	110	UG/L
	TH-4/L-056	PHENANTHRENE	J	56	UG/L
	TH-4/S-058	ACENAPHTHENE	J	4600	UG/KG
	TH-4/S-058	ANTHRACENE		26000	UG/KG
	TH-4/S-058	BENZO(A)ANTHRACENE		6300	UG/KG
	TH-4/S-058	BENZO(A)PYRENE	J	4300	UG/KG
	TH-4/S-058	BENZO(B)FLUORANTHENE		5600	UG/KG
	TH-4/S-058	BENZO(G,H,I)PERYLENE	J	3400	UG/KG
	TH-4/S-058	BIS(2-ETHYLHEXYL)PHTHALATE		20000	UG/KG
	TH-4/S-058	CHRYSENE		6800	UG/KG
	TH-4/S-058	DI-N-BUTYLPHTHALATE		17000	UG/KG
	TH-4/S-058	FLUORANTHENE		23000	UG/KG
	TH-4/S-058	FLUORENE	J	2800	UG/KG
	TH-4/S-058	INDENO(1,2,3-CD)PYRENE	J	3600	UG/KG
	TH-4/S-058	NAPHTHALENE		8400	UG/KG
	TH-4/S-058	PHENANTHRENE		26000	UG/KG
	TH-4/S-058	PYRENE		18000	UG/KG
	TH-4/S-059	ACENAPHTHENE		6700	UG/KG
	TH-4/S-059	BENZO(A)ANTHRACENE		5800	UG/KG
	TH-4/S-059	BENZO(B)FLUORANTHENE		8100	UG/KG
	TH-4/S-059	BIS(2-ETHYLHEXYL)PHTHALATE		26000	UG/KG
	TH-4/S-059	CHRYSENE		6200	UG/KG
	TH-4/S-059	DI-N-BUTYLPHTHALATE		13000	UG/KG
	TH-4/S-059	DIBENZOFURAN	J	4100	UG/KG
	TH-4/S-059	FLUORANTHENE		20000	UG/KG
	TH-4/S-059	FLUORENE	J	3700	UG/KG
	TH-4/S-059	NAPHTHALENE		17000	UG/KG
	TH-4/S-059	PHENANTHRENE		30000	UG/KG
	TH-4/S-059	PYRENE		21000	UG/KG

Table A.10. Listing of organic analytical data hits for tank W-1

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	W-1/L-007B	METHYLENE CHLORIDE	B	10	UG/L
	W-1/L-007B	TOLUENE	B	5	UG/L
	W-1/L-008A	METHYLENE CHLORIDE	B	11	UG/L
	W-1/L-008A	TOLUENE	B	11	UG/L
SVO	W-1/L-007	DI-N-BUTYLPHTHALATE	J	9	UG/L
	W-1/L-008	DI-N-BUTYLPHTHALATE	J	8	UG/L

Table A.11. Listing of organic analytical data hits for tank W-1A

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-1A/L-001	METHYL ALCOHOL		7	MG/L
VOA	W-1A/L-001A	ACETONE	B	17	UG/L
	W-1A/L-001A	BENZENE	JB	3	UG/L
	W-1A/L-001A	CHLOROBENZENE	J	3	UG/L
	W-1A/L-001A	ETHYLBENZENE	J	4	UG/L
	W-1A/L-001A	METHYLENE CHLORIDE	JB	4	UG/L
	W-1A/L-001A	TOLUENE	B	63	UG/L
	W-1A/L-001A	TRICHLOROETHENE	B	20	UG/L
	W-1A/L-001A	XYLENE (TOTAL)		13	UG/L
SVO	W-1A/L-001	DIETHYLPHTHALATE	J	24	UG/L

Table A.12. Listing of organic analytical data hits for tank W-10

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-10/L-093	METHYL ALCOHOL		27	MG/L
	W-10/L-094	METHYL ALCOHOL		37	MG/L
	W-10/L-095	ACETONE		3	MG/L
	W-10/L-095	ETHYL ALCOHOL		1	MG/L
	W-10/L-095	METHYL ALCOHOL		41	MG/L
	W-10/L-095	METHYL ETHYL KETONE		1	MG/L
	W-10/L-095	N-BUTYL ALCOHOL		1	MG/L
VOA	W-10/L-094A	BENZENE	B	6	UG/L
	W-10/L-094A	METHYLENE CHLORIDE	B	13	UG/L
	W-10/L-094A	TETRACHLOROETHENE		21	UG/L
	W-10/L-094A	TOLUENE		12	UG/L
	W-10/L-094A	TRICHLOROETHENE		27	UG/L
	W-10/L-094A	1,2-DICHLOROETHENE (TOTAL)		11	UG/L
	W-10/L-094B	BENZENE	BM	38	UG/L
	W-10/L-094B	METHYLENE CHLORIDE	B	19	UG/L
	W-10/L-095A	ACETONE	B	97	UG/L
	W-10/L-095A	BENZENE	B	22	UG/L
	W-10/L-095A	CHLOROBENZENE	B	5	UG/L
	W-10/L-095A	CHLOROFORM		16	UG/L
	W-10/L-095A	METHYLENE CHLORIDE	B	12	UG/L
	W-10/L-095A	TETRACHLOROETHENE		48	UG/L
	W-10/L-095A	TOLUENE	B	26	UG/L
	W-10/L-095A	TRICHLOROETHENE		81	UG/L
	W-10/L-095A	1,2-DICHLOROETHENE (TOTAL)		31	UG/L
	W-10/L-095A	4-METHYL-2-PENTANONE	B	23	UG/L
SVO	W-10/H-120	BENZO(A)PYRENE	J	1900	UG/KG
	W-10/H-120	BENZO(B)FLUORANTHENE	J	2200	UG/KG
	W-10/H-120	BENZO(G,H,I)PERYLENE	J	870	UG/KG
	W-10/H-120	DI-N-BUTYLPHTHALATE	J	770	UG/KG
	W-10/H-120	FLUORANTHENE	J	8000	UG/KG
	W-10/H-120	NAPHTHALENE	J	270	UG/KG
	W-10/H-120	PHENANTHRENE	J	5100	UG/KG
	W-10/H-120	PYRENE	J	8100	UG/KG
	W-10/L-094	BENZOIC ACID	J	400	UG/L
	W-10/L-095	BENZOIC ACID		2900	UG/L
	W-10/L-095	NAPHTHALENE	J	20	UG/L
	W-10/S-096	BENZO(A)ANTHRACENE	J	660	UG/KG
	W-10/S-096	BIS(2-ETHYLHEXYL)PHTHALATE		130000	UG/KG
	W-10/S-096	CHRYSENE	J	760	UG/KG
	W-10/S-096	DI-N-BUTYLPHTHALATE	J	2600	UG/KG
	W-10/S-096	FLUORANTHENE	J	1800	UG/KG
	W-10/S-096	PHENANTHRENE	J	1700	UG/KG
	W-10/S-096	PYRENE	J	1400	UG/KG

Table A.13. Listing of organic analytical data hits for tank W-11

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	W-11/L-003	ACETONE	B	27	UG/L
	W-11/L-003	CHLOROBENZENE	J	5	UG/L
	W-11/L-003	METHYLENE CHLORIDE	B	7	UG/L
	W-11/L-003	TOLUENE	B	23	UG/L
	W-11/L-003	TRICHLOROETHENE	J	4	UG/L
	W-11/L-114	ACETONE	B	21	UG/L
	W-11/L-114	CHLOROBENZENE	J	3	UG/L
	W-11/L-114	METHYLENE CHLORIDE	B	8	UG/L
	W-11/L-114	TOLUENE	B	24	UG/L
	W-11/L-115	ACETONE	B	15	UG/L
	W-11/L-115	CHLOROBENZENE	J	2	UG/L
	W-11/L-115	METHYLENE CHLORIDE	B	13	UG/L
	W-11/L-115	TOLUENE	B	13	UG/L
SVO	W-11/H-005	BENZO(A)ANTHRACENE		44000	UG/KG
	W-11/H-005	BENZO(A)PYRENE		64000	UG/KG
	W-11/H-005	BENZO(B)FLUORANTHENE	E	160000	UG/KG
	W-11/H-005	BENZO(G,H,I)PERYLENE		22000	UG/KG
	W-11/H-005	BIS(2-ETHYLHEXYL)PHTHALATE		38000	UG/KG
	W-11/H-005	CHRYSENE		130000	UG/KG
	W-11/H-005	DI-N-BUTYLPHTHALATE		17400	UG/KG
	W-11/H-005	FLUORANTHENE	E	240000	UG/KG
	W-11/H-005	INDENO(1,2,3-CD)PYRENE		42000	UG/KG
	W-11/H-005	PHENANTHRENE		24000	UG/KG
	W-11/H-005	PYRENE		90000	UG/KG

Table A.14. Listing of organic analytical data hits for tank W-13

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-13/L-027	METHYL ALCOHOL		11	MG/L
	W-13/L-028	METHYL ALCOHOL		5	MG/L
VOA	W-13/L-027A	ACETONE	B	15	UG/L
	W-13/L-027A	BENZENE	JB	3	UG/L
	W-13/L-027A	CHLOROBENZENE	J	4	UG/L
	W-13/L-027A	METHYLENE CHLORIDE	B	24	UG/L
	W-13/L-027A	STYRENE	J	3	UG/L
	W-13/L-027A	TOLUENE	B	100	UG/L
	W-13/L-028A	METHYLENE CHLORIDE	B	14	UG/L

Table A.15. Listing of organic analytical data hits for tank W-14

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-14/L-030	ETHYL ALCOHOL		3	MG/L
	W-14/L-030	I-PROPYL ALCOHOL		2	MG/L
	W-14/L-030	METHYL ALCOHOL		17	MG/L
	W-14/L-031	ETHYL ALCOHOL		2	MG/L
	W-14/L-031	METHYL ALCOHOL		15	MG/L
VOA	W-14/L-030A	ACETONE	B	20	UG/L
	W-14/L-030A	BENZENE	JB	3	UG/L
	W-14/L-030A	METHYLENE CHLORIDE	B	22	UG/L
	W-14/L-030A	TOLUENE	B	14	UG/L
	W-14/L-031A	ACETONE	B	25	UG/L
	W-14/L-031A	BENZENE	JB	4	UG/L
	W-14/L-031A	CHLOROBENZENE		6	UG/L
	W-14/L-031A	METHYLENE CHLORIDE	B	23	UG/L
	W-14/L-031A	TETRACHLOROETHENE		5	UG/L
	W-14/L-031A	TOLUENE	B	21	UG/L
	W-14/L-031A	TRICHLOROETHENE	B	6	UG/L
	W-14/L-031A	XYLENE (TOTAL)	J	4	UG/L
SVO	W-14/L-030	DI-N-BUTYLPHTHALATE	B	12	UG/L

Table A.16. Listing of organic analytical data hits for tank W-15

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-15/L-032	METHYL ALCOHOL		7	MG/L
	W-15/L-033	METHYL ALCOHOL		7	MG/L
VOA	W-15/L-032A	ACETONE	B	24	UG/L
	W-15/L-032A	BENZENE	JB	4	UG/L
	W-15/L-032A	CHLOROBENZENE		5	UG/L
	W-15/L-032A	METHYLENE CHLORIDE	B	21	UG/L
	W-15/L-032A	STYRENE	J	2	UG/L
	W-15/L-032A	TOLUENE	B	29	UG/L
	W-15/L-032A	TRICHLOROETHENE	JB	4	UG/L
	W-15/L-032A	XYLENE (TOTAL)	J	5	UG/L
	W-15/L-033A	ACETONE	B	26	UG/L
	W-15/L-033A	BENZENE	JB	3	UG/L
	W-15/L-033A	CHLOROBENZENE	J	2	UG/L
	W-15/L-033A	METHYLENE CHLORIDE	B	24	UG/L
	W-15/L-033A	TOLUENE	B	26	UG/L
	W-15/L-033A	TRICHLOROETHENE	JB	1	UG/L
	W-15/L-033A	XYLENE (TOTAL)	J	4	UG/L

Table A.17. Listing of organic analytical data hits for tank W-2

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	W-2/L-011A	METHYLENE CHLORIDE	B	10	UG/L
	W-2/L-118A	METHYLENE CHLORIDE	B	9	UG/L
	W-2/L-118A	TOLUENE	B	15	UG/L
SVO	W-2/L-011	DI-N-BUTYLPHTHALATE	J	10	UG/L

Table A.18. Listing of organic analytical data hits for tank W-3

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-3/L-016	METHYL ALCOHOL		38	MG/L
	W-3/L-017	METHYL ALCOHOL		40	MG/L
	W-3/L-018	ACETONE		7	MG/L
	W-3/L-018	METHYL ALCOHOL		20	MG/L
	W-3/L-018	METHYL ETHYL KETONE		5	MG/L
	W-3/L-018	N-BUTYL ALCOHOL		7	MG/L
VOA	W-3/L-016A	ACETONE	JB	2	UG/L
	W-3/L-016A	METHYLENE CHLORIDE	JB	4	UG/L
	W-3/L-016A	TETRACHLOROETHENE	B	6	UG/L
	W-3/L-016A	TOLUENE	JB	5	UG/L
	W-3/L-016A	TRICHLOROETHENE	B	11	UG/L
	W-3/L-017A	ACETONE	B	26	UG/L
	W-3/L-017A	CHLOROFORM	J	1	UG/L
	W-3/L-017A	METHYLENE CHLORIDE	JB	4	UG/L
	W-3/L-017A	TETRACHLOROETHENE	B	8	UG/L
	W-3/L-017A	TOLUENE	B	14	UG/L
	W-3/L-017A	TRICHLOROETHENE	B	14	UG/L
	W-3/L-018A	ACETONE	JB	5	UG/L
	W-3/L-018A	BENZENE	J	4	UG/L
	W-3/L-018A	CHLOROBENZENE		8	UG/L
	W-3/L-018A	METHYLENE CHLORIDE	JB	3	UG/L
	W-3/L-018A	TOLUENE	B	37	UG/L
	W-3/L-018A	TRICHLOROETHENE	B	11	UG/L
	W-3/L-018A	4-METHYL-2-PENTANONE		18	UG/L
SVO	W-3/L-016	DI-N-BUTYLPHTHALATE	J	60	UG/L
	W-3/L-016	DI-N-OCTYLPHTHALATE	J	49	UG/L
	W-3/L-017	DI-N-BUTYLPHTHALATE	J	48	UG/L
	W-3/L-017	DI-N-OCTYLPHTHALATE	J	80	UG/L
	W-3/L-018	DI-N-BUTYLPHTHALATE	J	230	UG/L
	W-3/L-018	FLUORANTHENE	J	56	UG/L
	W-3/L-018	PHENANTHRENE	J	33	UG/L
	W-3/L-018	PYRENE	J	60	UG/L
	W-3/L-018	2-NITROPHENOL	J	53	UG/L

Table A.19. Listing of organic analytical data hits for tank W-4

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-4/L-023	METHYL I-BUTYL KETONE		7	MG/L
VOA	W-4/L-022A	METHYLENE CHLORIDE	B	10	UG/L
	W-4/L-022A	TOLUENE	B	13	UG/L
	W-4/L-023A	ACETONE		55	UG/L
	W-4/L-023A	BENZENE	J	3	UG/L
	W-4/L-023A	METHYLENE CHLORIDE	B	10	UG/L
	W-4/L-023A	TOLUENE	B	14	UG/L
	W-4/L-023A	TRICHLOROETHENE		49	UG/L
	W-4/L-023A	4-METHYL-2-PENTANONE		1102	UG/L
	W-4/L-119A	BENZENE	JB	2	UG/L
	W-4/L-119A	METHYLENE CHLORIDE	B	10	UG/L
	W-4/L-119A	TOLUENE	B	46	UG/L
	W-4/L-119A	TRICHLOROETHENE		14	UG/L
	W-4/L-119A	4-METHYL-2-PENTANONE	J	3	UG/L
SVO	W-4/H-026	BIS(2-ETHYLHEXYL)PHTHALATE		5700	UG/KG
	W-4/H-026	DI-N-BUTYLPHTHALATE		1400	UG/KG
	W-4/H-026	DIETHYLPHTHALATE	J	49	UG/KG
	W-4/H-026	FLUORANTHENE	J	160	UG/KG
	W-4/H-026	PHENANTHRENE	J	110	UG/KG
	W-4/H-026	PYRENE	J	130	UG/KG
	W-4/L-022	DI-N-BUTYLPHTHALATE	J	11	UG/L
	W-4/L-023	NAPHTHALENE	J	160	UG/L
	W-4/L-119	NAPHTHALENE	J	35	UG/L
	W-4/S-024	BIS(2-ETHYLHEXYL)PHTHALATE		11000	UG/KG
	W-4/S-024	CHRYSENE	J	69	UG/KG
	W-4/S-024	DI-N-BUTYLPHTHALATE		2100	UG/KG
	W-4/S-024	DI-N-OCTYLPHTHALATE	J	51	UG/KG
	W-4/S-024	DIETHYLPHTHALATE	J	33	UG/KG
	W-4/S-024	FLUORANTHENE	J	220	UG/KG
	W-4/S-024	PHENANTHRENE	J	170	UG/KG
	W-4/S-024	PYRENE	J	170	UG/KG

Table A.20. Listing of organic analytical data hits for tank W-5

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	W-5/L-073A	ACETONE	B	19	UG/L
	W-5/L-073A	CARBON TETRACHLORIDE	B	68	UG/L
	W-5/L-073A	CHLOROFORM		6	UG/L
	W-5/L-073A	METHYLENE CHLORIDE	B	6	UG/L
	W-5/L-073A	TETRACHLOROETHENE	B	267	UG/L
	W-5/L-073A	TRICHLOROETHENE		138	UG/L
SVO	W-5/L-073	DI-N-BUTYLPHTHALATE	J	18	UG/L
	W-5/S-075	BIS(2-ETHYLHEXYL)PHTHALATE		28000	UG/KG
	W-5/S-075	DI-N-BUTYLPHTHALATE	J	350	UG/KG
	W-5/S-075	DIETHYLPHTHALATE	J	230	UG/KG
	W-5/S-075	FLUORANTHENE	J	230	UG/KG
	W-5/S-075	PHENANTHRENE	J	440	UG/KG
	W-5/S-075	PYRENE	J	230	UG/KG

Table A.21. Listing of organic analytical data hits for tank W-6

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-6/L-079	I-PROPYL ALCOHOL		3	MG/L
VOA	W-6/L-077A	ACETONE	B	67	UG/L
	W-6/L-077A	CARBON TETRACHLORIDE	JB	4	UG/L
	W-6/L-077A	CHLOROFORM	J	5	UG/L
	W-6/L-077A	METHYLENE CHLORIDE	B	9	UG/L
	W-6/L-077A	TETRACHLOROETHENE	B	47	UG/L
	W-6/L-077A	TOLUENE	B	6	UG/L
	W-6/L-077A	TRICHLOROETHENE		13	UG/L
	W-6/L-078A	ACETONE	B	44	UG/L
	W-6/L-078A	BENZENE	J	2	UG/L
	W-6/L-078A	CHLOROFORM		9	UG/L
	W-6/L-078A	METHYLENE CHLORIDE	B	8	UG/L
	W-6/L-078A	TETRACHLOROETHENE	B	158	UG/L
	W-6/L-078A	TOLUENE	B	10	UG/L
	W-6/L-078A	TRICHLOROETHENE		24	UG/L
	W-6/L-078A	4-METHYL-2-PENTANONE	J	9	UG/L
	W-6/L-079A	ACETONE	B	111	UG/L
	W-6/L-079A	BENZENE		10	UG/L
	W-6/L-079A	CHLOROFORM	J	3	UG/L
	W-6/L-079A	METHYLENE CHLORIDE	B	7	UG/L
	W-6/L-079A	TETRACHLOROETHENE	BE	507	UG/L
	W-6/L-079A	TOLUENE	B	10	UG/L
	W-6/L-079A	TRICHLOROETHENE		82	UG/L
	W-6/L-079A	2-BUTANONE		75	UG/L
	W-6/L-079A	2-HEXANONE		9	UG/L
	W-6/L-079A	4-METHYL-2-PENTANONE		91	UG/L
SVO	W-6/L-079	BENZOIC ACID	J	290	UG/L
	W-6/S-080	BIS(2-ETHYLHEXYL)PHTHALATE		31000	UG/KG
	W-6/S-080	DI-N-BUTYLPHTHALATE	J	210	UG/KG
	W-6/S-080	DIETHYLPHTHALATE	J	150	UG/KG
	W-6/S-080	FLUORANTHENE	J	340	UG/KG
	W-6/S-080	PHENANTHRENE	J	680	UG/KG
	W-6/S-080	PYRENE	J	350	UG/KG

Table A.22. Listing of organic analytical data hits for tank W-7

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-7/L-082	METHYL ALCOHOL		14	MG/L
VOA	W-7/L-082A	ACETONE	B	26	UG/L
	W-7/L-082A	CARBON TETRACHLORIDE	BE	1088	UG/L
	W-7/L-082A	CHLOROFORM	J	3	UG/L
	W-7/L-082A	METHYLENE CHLORIDE	B	7	UG/L
	W-7/L-082A	TETRACHLOROETHENE	B	24	UG/L
	W-7/L-082A	TOLUENE	B	14	UG/L
	W-7/L-082A	4-METHYL-2-PENTANONE		7	UG/L
	W-7/L-082B	ACETONE	B	31	UG/L
	W-7/L-082B	CARBON TETRACHLORIDE	BE	1021	UG/L
	W-7/L-082B	CHLOROFORM	J	3	UG/L
	W-7/L-082B	METHYLENE CHLORIDE	B	4	UG/L
	W-7/L-082B	TETRACHLOROETHENE	B	39	UG/L
	W-7/L-082B	TOLUENE	B	9	UG/L
	W-7/L-082B	4-METHYL-2-PENTANONE		7	UG/L
SVO	W-7/H-085	DI-N-BUTYLPHTHALATE	J	2200	UG/KG
	W-7/H-085	DIETHYLPHTHALATE	J	510	UG/KG
	W-7/L-082	BENZOIC ACID	J	1900	UG/L
	W-7/S-084	BIS(2-ETHYLHEXYL)PHTHALATE		57000	UG/KG
	W-7/S-084	DI-N-BUTYLPHTHALATE	J	300	UG/KG
	W-7/S-084	DIETHYLPHTHALATE	J	200	UG/KG
	W-7/S-084	FLUORANTHENE	J	260	UG/KG

Table A.23. Listing of organic analytical data hits for tank W-8

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	W-8/L-086	ACETONE		1	MG/L
	W-8/L-087	ACETONE		3	MG/L
	W-8/L-087	METHYL ALCOHOL		1	MG/L
	W-8/L-087	METHYL ETHYL KETONE		1	MG/L
	W-8/L-087	N-BUTYL ALCOHOL		2	MG/L
VOA	W-8/L-086A	ACETONE	B	64	UG/L
	W-8/L-086A	BENZENE	J	3	UG/L
	W-8/L-086A	METHYLENE CHLORIDE	B	12	UG/L
	W-8/L-086A	TOLUENE	BJ	3	UG/L
	W-8/L-086A	2-HEXANONE		41	UG/L
	W-8/L-086A	4-METHYL-2-PENTANONE		15	UG/L
	W-8/L-087A	ACETONE	B	45	UG/L
	W-8/L-087A	BENZENE		6	UG/L
	W-8/L-087A	METHYLENE CHLORIDE	B	7	UG/L
	W-8/L-087A	TOLUENE	BJ	4	UG/L
SVO	W-8/L-087	DI-N-BUTYLPHTHALATE	J	17	UG/L
	W-8/L-087	NAPHTHALENE	J	28	UG/L
	W-8/S-088	DI-N-BUTYLPHTHALATE	J	630	UG/KG
	W-8/S-088	DIETHYLPHTHALATE	J	430	UG/KG
	W-8/S-088	FLUORANTHENE	J	740	UG/KG
	W-8/S-088	NAPHTHALENE	J	2600	UG/KG
	W-8/S-088	PHENANTHRENE	J	1300	UG/KG
	W-8/S-088	PYRENE	J	850	UG/KG
	W-8/S-088	2-METHYLNAPHTHALENE	J	1300	UG/KG

Table A.24. Listing of organic analytical data hits for tank W-9

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	W-9/L-090A	ACETONE	B	70	UG/L
	W-9/L-090A	BENZENE	J	2	UG/L
	W-9/L-090A	CHLOROBENZENE		5	UG/L
	W-9/L-090A	CHLOROFORM		44	UG/L
	W-9/L-090A	METHYLENE CHLORIDE	B	13	UG/L
	W-9/L-090A	TETRACHLOROETHENE	J	4	UG/L
	W-9/L-090A	TOLUENE	B	9	UG/L
	W-9/L-090A	TRICHLOROETHENE		5	UG/L
	W-9/L-090A	4-METHYL-2-PENTANONE		11	UG/L
SVO	W-9/L-090	DI-N-BUTYLPHTHALATE	J	20	UG/L
	W-9/S-092	BIS(2-ETHYLHEXYL)PHTHALATE		160000	UG/KG
	W-9/S-092	DI-N-BUTYLPHTHALATE	J	760	UG/KG
	W-9/S-092	DIETHYLPHTHALATE	J	450	UG/KG
	W-9/S-092	FLUORANTHENE	J	1100	UG/KG
	W-9/S-092	NAPHTHALENE	J	370	UG/KG
	W-9/S-092	PHENANTHRENE	J	1300	UG/KG
	W-9/S-092	PYRENE	J	850	UG/KG

Table A.25. Listing of organic analytical data hits for tank WC-17

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	WC-17/L-103 AQ	ACETONE	B	170	UG/L
	WC-17/L-103 AQ	BENZENE	JB	10	UG/L
	WC-17/L-103 AQ	METHYLENE CHLORIDE	JB	38	UG/L
	WC-17/L-103 AQ	XYLENE (TOTAL)	J	50	UG/L
	WC-17/L-103 ORG	ACETONE	B	6400	UG/L
	WC-17/L-103 ORG	BENZENE	JB	420	UG/L
	WC-17/L-103 ORG	METHYLENE CHLORIDE	B	5300	UG/L
	WC-17/L-103 ORG	STYRENE		7500	UG/L
	WC-17/L-103 ORG	XYLENE (TOTAL)	B	2500	UG/L
	WC-17/L-104 AQ	ACETONE	B	150	UG/L
	WC-17/L-104 AQ	BENZENE	JB	10	UG/L
	WC-17/L-104 AQ	METHYLENE CHLORIDE	JB	44	UG/L
	WC-17/L-104 AQ	XYLENE (TOTAL)		59	UG/L
	WC-17/L-105 AQ	ACETONE	B	130	UG/L
	WC-17/L-105 AQ	BENZENE	JB	10	UG/L
	WC-17/L-105 AQ	METHYLENE CHLORIDE	JB	40	UG/L
	WC-17/L-105 AQ	XYLENE (TOTAL)	J	50	UG/L
	WC-17/O-101 AQ	ACETONE	B	150	UG/L
	WC-17/O-101 AQ	BENZENE	JB	7	UG/L
	WC-17/O-101 AQ	METHYLENE CHLORIDE	JB	39	UG/L
	WC-17/O-101 AQ	XYLENE (TOTAL)	J	46	UG/L
	WC-17/O-101 ORG	ACETONE	B	6700	UG/L
	WC-17/O-101 ORG	ACETONE	B	6700	UG/L
	WC-17/O-101 ORG	BENZENE	JB	450	UG/L
	WC-17/O-101 ORG	BENZENE	JB	410	UG/L
	WC-17/O-101 ORG	METHYLENE CHLORIDE	B	6500	UG/L
	WC-17/O-101 ORG	METHYLENE CHLORIDE	B	4000	UG/L
	WC-17/O-101 ORG	STYRENE		7100	UG/L
	WC-17/O-101 ORG	STYRENE		8200	UG/L
	WC-17/O-101 ORG	TRICHLOROETHENE	J	920	UG/L
	WC-17/O-101 ORG	VINYL ACETATE	J	1200	UG/L
	WC-17/O-101 ORG	XYLENE (TOTAL)	JB	2200	UG/L
	WC-17/O-101 ORG	XYLENE (TOTAL)	JB	2300	UG/L
	WC-17/O-102 AQ	ACETONE	B	140	UG/L
	WC-17/O-102 AQ	BENZENE	JB	9	UG/L
	WC-17/O-102 AQ	METHYLENE CHLORIDE	JB	37	UG/L
	WC-17/O-102 AQ	XYLENE (TOTAL)		76	UG/L
	WC-17/O-102 ORG	ACETONE	B	7400	UG/L
	WC-17/O-102 ORG	BENZENE	JB	400	UG/L
	WC-17/O-102 ORG	METHYLENE CHLORIDE	B	6600	UG/L
	WC-17/O-102 ORG	STYRENE		4700	UG/L
	WC-17/O-102 ORG	XYLENE (TOTAL)	B	2700	UG/L
SVO	WC-17/L-103 AQ	BIS(2-ETHYLHEXYL)PHTHALATE	J	38	UG/L
	WC-17/L-103 AQ	DI-N-BUTYLPHTHALATE	JB	56	UG/L
	WC-17/L-103 AQ	DI-N-OCTYLPHTHALATE	JB	93	UG/L
	WC-17/L-103 ORG	DI-N-BUTYLPHTHALATE	JB	68000	UG/KG

Table A.25 (continued)

Analysis	Sample ID	Compound	Qualifier	Result	Units
SVO	WC-17/L-104 AQ	DI-N-OCTYLPHTHALATE	JB	33	UG/L
	WC-17/O-101 AQ	DI-N-OCTYLPHTHALATE	JB	40	UG/L
	WC-17/O-101 ORG	DI-N-BUTYLPHTHALATE	JB	74000	UG/KG
	WC-17/O-101 ORG	DI-N-BUTYLPHTHALATE	JB	57000	UG/KG
	WC-17/O-102 AQ	DI-N-OCTYLPHTHALATE	JB	24	UG/L
	WC-17/O-102 ORG	DI-N-BUTYLPHTHALATE	JB	44000	UG/KG
PCB	WC-17/L-103 ORG	PCB (AROCLOR-1254)		20	MG/KG
	WC-17/L-103 ORG	PCB (AROCLOR-1260)		13	MG/KG
	WC-17/O-101 ORG	PCB (AROCLOR-1254)		18	MG/KG
	WC-17/O-101 ORG	PCB (AROCLOR-1254)		16	UG/G
	WC-17/O-101 ORG	PCB (AROCLOR-1260)		10	MG/KG
	WC-17/O-101 ORG	PCB (AROCLOR-1260)		11	MG/KG
	WC-17/O-102 ORG	PCB (AROCLOR-1254)		20	MG/KG
	WC-17/O-102 ORG	PCB (AROCLOR-1260)		13	MG/KG

Table A.26. Listing of organic analytical data hits for tank 7562

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	7562/L-097	METHYL ALCOHOL		14	MG/L
VOA	7562/L-097A	ACETONE	B	15	UG/L
	7562/L-097A	BENZENE	J	3	UG/L
	7562/L-097A	METHYLENE CHLORIDE	B	18	UG/L
	7562/L-097A	TETRACHLOROETHENE	J	4	UG/L
	7562/L-097A	TOLUENE		10	UG/L

Table A.27. Listing of organic analytical data hits for tank 7860A

Analysis	Sample ID	Compound	Qualifier	Result	Units
DAI-GC	7860A/L-067	ACETONE		1500	MG/L
	7860A/L-067	ETHYL ALCOHOL		2600	MG/L
	7860A/L-067	I-PROPYL ALCOHOL		770	MG/L
	7860A/L-067	METHYL ALCOHOL		1400	MG/L
	7860A/L-067	METHYL ETHYL KETONE		1400	MG/L
	7860A/L-067	N-BUTYL ALCOHOL		1000	MG/L
	7860A/L-068	ACETONE		2300	MG/L
	7860A/L-068	ETHYL ALCOHOL		4300	MG/L
	7860A/L-068	I-PROPYL ALCOHOL		940	MG/L
	7860A/L-068	METHYL ALCOHOL		1700	MG/L
	7860A/L-068	METHYL ETHYL KETONE		2000	MG/L
	7860A/L-068	N-BUTYL ALCOHOL		1200	MG/L
	7860A/L-069	ACETONE		1900	MG/L
	7860A/L-069	ETHYL ALCOHOL		3400	MG/L
	7860A/L-069	I-PROPYL ALCOHOL		860	MG/L
	7860A/L-069	METHYL ALCOHOL		1600	MG/L
	7860A/L-069	METHYL ETHYL KETONE		1600	MG/L
	7860A/L-069	N-BUTYL ALCOHOL		1000	MG/L
VOA	7860A/L-063	ACETONE	B	2200000	UG/L
	7860A/L-063	ETHYLBENZENE		440000	UG/L
	7860A/L-063	METHYLENE CHLORIDE	JB	61000	UG/L
	7860A/L-063	TETRACHLOROETHENE	E	39000000	UG/L
	7860A/L-063	TOLUENE	J	85000	UG/L
	7860A/L-063	TRICHLOROETHENE	E	39000000	UG/L
	7860A/L-063	XYLENE (TOTAL)	E	9800000	UG/L
	7860A/L-063	2-BUTANONE		4100000	UG/L
	7860A/L-064	ACETONE	B	1100000	UG/L
	7860A/L-064	ETHYLBENZENE		420000	UG/L
	7860A/L-064	METHYLENE CHLORIDE	JB	97000	UG/L
	7860A/L-064	TETRACHLOROETHENE	E	35000000	UG/L
	7860A/L-064	TOLUENE	J	81000	UG/L
	7860A/L-064	TRICHLOROETHENE	E	36000000	UG/L
	7860A/L-064	XYLENE (TOTAL)		8900000	UG/L
	7860A/L-064	2-BUTANONE		1400000	UG/L
	7860A/L-065	ACETONE	B	1000000	UG/L
	7860A/L-065	METHYLENE CHLORIDE	JB	64000	UG/L
	7860A/L-065	TETRACHLOROETHENE	E	36000000	UG/L
	7860A/L-065	TOLUENE	J	90000	UG/L
	7860A/L-065	TRICHLOROETHENE	E	38000000	UG/L
	7860A/L-065	XYLENE (TOTAL)	E	9300000	UG/L
	7860A/L-065	2-BUTANONE		1300000	UG/L
	7860A/L-066	ACETONE	B	1000000	UG/L
	7860A/L-066	ETHYLBENZENE		450000	UG/L
	7860A/L-066	METHYLENE CHLORIDE	JB	170000	UG/L
	7860A/L-066	TETRACHLOROETHENE	E	40000000	UG/L
	7860A/L-066	TRICHLOROETHENE	E	39000000	UG/L

Table A.27 (continued)

Analysis	Sample ID	Compound	Qualifier	Result	Units
VOA	7860A/L-066	XYLENE (TOTAL)	E	11000000	UG/L
	7860A/L-066	2-BUTANONE		1300000	UG/L
	7860A/L-067 AQ	ACETONE	BE	1300000	UG/L
	7860A/L-067 AQ	METHYLENE CHLORIDE	JB	1600	UG/L
	7860A/L-067 AQ	TETRACHLOROETHENE		19000	UG/L
	7860A/L-067 AQ	TRICHLOROETHENE		65000	UG/L
	7860A/L-067 AQ	VINYL ACETATE	J	2300	UG/L
	7860A/L-067 AQ	XYLENE (TOTAL)		14000	UG/L
	7860A/L-067 AQ	2-BUTANONE	E	1800000	UG/L
	7860A/L-067 AQ	4-METHYL-2-PENTANONE	J	3100	UG/L
	7860A/L-067 ORG	ACETONE	B	1000000	UG/L
	7860A/L-067 ORG	ETHYLBENZENE		430000	UG/L
	7860A/L-067 ORG	METHYLENE CHLORIDE	JB	81000	UG/L
	7860A/L-067 ORG	TETRACHLOROETHENE	E	35000000	UG/L
	7860A/L-067 ORG	TOLUENE	J	94000	UG/L
	7860A/L-067 ORG	TRICHLOROETHENE	E	36000000	UG/L
	7860A/L-067 ORG	XYLENE (TOTAL)	E	9500000	UG/L
	7860A/L-067 ORG	2-BUTANONE		1700000	UG/L
	7860A/L-068	ACETONE	BE	1300000	UG/L
	7860A/L-068	METHYLENE CHLORIDE	JB	1600	UG/L
	7860A/L-068	TETRACHLOROETHENE	J	3900	UG/L
	7860A/L-068	TRICHLOROETHENE		15000	UG/L
	7860A/L-068	VINYL ACETATE	J	1600	UG/L
	7860A/L-068	XYLENE (TOTAL)		8800	UG/L
	7860A/L-068	2-BUTANONE	E	1700000	UG/L
	7860A/L-068	4-METHYL-2-PENTANONE	J	5000	UG/L
	7860A/L-069	ACETONE	BE	1400000	UG/L
	7860A/L-069	METHYLENE CHLORIDE	JB	1200	UG/L
	7860A/L-069	TETRACHLOROETHENE		8500	UG/L
	7860A/L-069	TRICHLOROETHENE		41000	UG/L
	7860A/L-069	VINYL ACETATE	J	2100	UG/L
	7860A/L-069	XYLENE (TOTAL)		11000	UG/L
	7860A/L-069	2-BUTANONE	E	2000000	UG/L
	7860A/L-069	4-METHYL-2-PENTANONE	J	5300	UG/L
SVO	7860A/L-063	BIS(2-ETHYLHEXYL)PHTHALATE	J	600000	UG/KG
	7860A/L-063	ISOPHORONE	J	41000	UG/KG
	7860A/L-063	NAPHTHALENE	J	82000	UG/KG
	7860A/L-063	2-METHYLNAPHTHALENE	J	71000	UG/KG
	7860A/L-064	BIS(2-ETHYLHEXYL)PHTHALATE	J	630000	UG/KG
	7860A/L-065	BIS(2-ETHYLHEXYL)PHTHALATE	J	530000	UG/KG
	7860A/L-065	DI-N-BUTYLPHTHALATE	J	62000	UG/KG
	7860A/L-065	NAPHTHALENE	J	92000	UG/KG
	7860A/L-065	2-METHYLNAPHTHALENE	J	86000	UG/KG
	7860A/L-066	BIS(2-ETHYLHEXYL)PHTHALATE	J	560000	UG/KG
	7860A/L-066	DI-N-BUTYLPHTHALATE	J	59000	UG/KG
	7860A/L-066	NAPHTHALENE	J	89000	UG/KG

Table A.27 (continued)

Analysis	Sample ID	Compound	Qualifier	Result	Units
SVO	7860A/L-066	2-METHYLNAPHTHALENE	J	74000	UG/KG
	7860A/L-067 AQ	BIS(2-ETHYLHEXYL)PHTHALATE		20000	UG/L
	7860A/L-067 AQ	DI-N-BUTYLPHTHALATE	J	1760	UG/L
	7860A/L-067 AQ	NAPHTHALENE	J	3200	UG/L
	7860A/L-067 AQ	2-METHYLNAPHTHALENE	J	3200	UG/L
	7860A/L-067 ORG	BIS(2-ETHYLHEXYL)PHTHALATE	J	490000	UG/KG
	7860A/L-067 ORG	DI-N-BUTYLPHTHALATE	J	60000	UG/KG
	7860A/L-067 ORG	NAPHTHALENE	J	91000	UG/KG
	7860A/L-067 ORG	2-METHYLNAPHTHALENE	J	79000	UG/KG
	7860A/L-069 AQ	NAPHTHALENE	J	240	UG/L

INTERNAL DISTRIBUTION

1-3. J. W. Autrey
4. L. D. Bates
5. T. W. Burwinkle
6. J. S. Colley
7. K. W. Cook
8. D. A. Costanzo
9. H. R. Gaddis
10. W. H. Griest
11. D. D. Huff
12. J. M. Keller
13. C. E. Nix
14-17. P. T. Owen
18. R. E. Pudelek
19. J. Reafsnyder
20. R. W. Sharpe
21. V. P. Spalding
22. C. L. Stair
23. D. W. Swindle
24. B. A. Tomkins
25. J. R. Trabalka
26. L. D. Voorhees
27. L. F. Willis
28. R. K. White
29. Central Research Library
30. Document Reference Section
31-52. ER Document Management Center
53. Laboratory Records, ORNL—RC
54-55. Laboratory Records Department
56. ORNL Patent Section

EXTERNAL DISTRIBUTION

57. Department of Energy, Oak Ridge Operations Office, Office of Assistant Manager for Energy Research and Develpment, P.O. Box 2001, Oak Ridge, TN 37831-8600
58. L. L. Kaiser, Project Manager, 807 Matzatlan, Grand Junction, CO 81506
59. D. Taylor, Bechtel National, Inc., P.O. Box 350, Oak Ridge, TN 37831-0350
60. T. J. Wheeler, Department of Energy, Oak Ridge Operations Office, P.O. Box 2001, Oak Ridge, TN 37831-8600
61-62. P. H. Edmonds, Lee Wan and Associates, 120 S. Jefferson Circle, Oak Ridge, TN 37830