Vapor Space Characterization of Waste Tank 241-C-106: Results from Samples Collected on 2/15/94

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June 1995

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**Pacific Northwest Laboratory
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Pacific Northwest Laboratory
Richland, Washington 99352

Summary

This report describes inorganic and organic analyses results from samples obtained from the headspace of the Hanford waste storage Tank 241-C-106 (referred to as Tank C-106). The results described here were obtained to support safety and toxicological evaluations. A summary of the results for inorganic and organic analytes is listed in Summary Table 1. Detailed descriptions of the results appear in the text.

Quantitative results were obtained for the inorganic compounds ammonia (NH_3), nitrogen dioxide (NO_2), nitric oxide (NO), and water (H_2O). No chain of custody was utilized for the inorganic samples, in part, because procedures were still being developed at the time Tank C-106 was sampled. The tank headspace concentrations of inorganic compounds were reported as less than or equal to probable maximum values; this was done because potential sampling errors were identified that may have effected some of the samples. While more definitive results could be obtained by re-sampling the headspace, the relatively low concentrations of analytes found in the present job would tend to support a lower prioritization for a second sampling job.

Two organic tentatively identified compounds (TICs) were observed above the detection limit of (ca.) 10 ppbv, but appropriate gas standards were not available at the time of analysis, and the reported concentrations are semiquantitative estimates. In addition, we looked for the 40 standard TO-14 analytes. None were observed above the 2-ppbv detection limit. The two TICs are listed in Table 1, and account for 100% of the total organic components from the samples obtained from the headspace of Tank C-106. Because the SUMMA™ canisters were not heated, n-alkanes greater than C_{11} and polar compounds which may have adhered to the inside surfaces of the canister may also be under represented in these results.

Table 1. Results of Inorganic and Organic Samples Collected from the Headspace of Tank C-106 on 2/15/94

<u>Category</u>	<u>Analyte</u>	<u>Vapor^(a) Concentration</u>	<u>Units</u>
Inorganic	NH_3	≤ 9	ppmv
	NO_2	≤ 0.1	ppmv
	NO	≤ 0.1	ppmv
	H_2O	20 ± 3	mg/L
Organic	Tridecane	0.13	mg/m^3
	1-Butanol	0.06	mg/m^3

(a) Vapor concentrations were determined using sample-volume data provided by Westinghouse Hanford Company and are based on averaged data. The inorganic samples were obtained and analyzed using procedure that were subsequently improved.

Acknowledgments

The authors gratefully acknowledge the support of other project staff at Pacific Northwest Laboratory who contributed to the successful completion of this sampling and analysis activity. Jeff Edwards served as the PNL single-point-of-contact and coordinated sample handling and communications with Westinghouse Hanford Company. K. B. Olsen provided data analysis. Bruce Lerner performed method development and validation work that supported the inorganic vapor sampling. Sally Slate and May-Lin Thomas analyzed inorganic samples, and Annalisa Krupsha and Gary Dennis prepared the solid-sorbent sample trains. Rose Nipper and Brenda Thornton provided word processing support.

Abbreviations

CAS	Chemical Abstracts Service
COC	chain of custody
Cv	concentration by volume
DIW	deionized water
emf	electromotive force
EPA	U.S. Environmental Protection Agency
GC/MS	gas chromatography/mass spectrometry
HP	Hewlett Packard
IC	ion chromatography
IL	impact level
IS	internal standard
MDL	minimum detection limit
NIST	National Institute for Standards and Technology
NPH	normal paraffin hydrocarbon
OSHA	Occupational Safety and Health Administration
PNL	Pacific Northwest Laboratory
ppbv	part per billion by volume
ppmv	part per million by volume
QA	quality assurance
REL	recommended exposure limit
SCIC	suppressed-conductivity ion chromatography
SIE	selective ion electrode
SRM	standard reference material
STP	standard temperature and pressure
TEA	triethanolamine
TIC	tentatively identified compound
VSS	vapor sampling system
WHC	Westinghouse Hanford Company

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1.0 Introduction

This report describes results of the analyses of tank-headspace samples taken from the Hanford waste Tank 241-C-106 (referred to as Tank C-106). Pacific Northwest Laboratory (PNL)^(a) contracted with Westinghouse Hanford Company (WHC) to provide sampling devices and to analyze inorganic and organic analytes collected from the tank headspace. Jerry Osborne sent a letter of instruction to S. C. Goheen on February 15, 1994, requesting that SUMMA™ canisters and sorbent traps be analyzed by PNL quality assurance (QA) impact level (IL) III. The sample job was designated S4006, and samples were collected by WHC on February 15, 1994, using the vapor sampling system (VSS). The results of the analyses are expected to be used to estimate the potential toxicity of tank-headspace gas as described in *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issue Resolution*, WHC-SD-WM-DQO-002, Rev. 0.

No chain of custody (COC) forms are available for inorganic samples, so for the purpose of this report, the inorganic samples are identified by the numbers affixed to the samples by WHC before transfer to PNL. Thirteen SUMMA™ canisters were supplied to WHC staff on February 14, six SUMMA™ canisters were listed on COC 006115 for Tank C-106 (Figure 1.1a), and seven SUMMA™ canisters were listed on COC 006108 for Tank C-104. Three SUMMA™ canisters were returned to PNL on February 18, 1994, on COC 006115 (Figure 1.1c), which contains two different SUMMA™ canisters than the original COC 006115 (Figure 1.1a). Canister PNL #061 (sample S4006-04.B15) was sent out on COC 006115 (Figure 1.1a) as a surrogate sample, but was returned on COC 006115 (Figure 1.1c) as a sample for analysis. Two of the returned canisters (one also intended as a surrogate sample) were originally sent out on COC 00610 (Figure 1.1b) for sampling Tank C-104.

The samples were inspected upon delivery to the 326/23B laboratory and logged into PNL record book 55408 before analyses. Custody of the sorbent traps was transferred to PNL personnel performing the inorganic analysis and stored at refrigerated ($\leq 10^{\circ}\text{C}$) temperature until the time of analysis. The canisters were stored in the 326/23B laboratory at ambient (25°C) temperature until the time of the analysis. Access to the 326/23B laboratory is limited to PNL personnel working on the waste-tank safety program. Analyses described in this report were performed at PNL in the 300 area of the Hanford Reservation. Analytical methods that were used are described in the text. Organic analyses were performed using cryogenic preconcentration followed by gas chromatography/mass spectrometry (GC/MS).

(a) Pacific Northwest Laboratory is operated for the U. S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

Westinghouse Hanford Company	CHAIN OF CUSTODY	PNL 006115
---------------------------------	------------------	------------

Custody Form Initiator C. M. Jones/ J. E. Darling

Company Contact J. L. Huckaby

Telephone (509) 373-3443

Project Designation/Sampling Locations 200 East Tank Farm: Tank

Collection Date 02-15-94

241-C-106 vapor sample SAF S4006, 241-C-106

Ice Chest No.

Field Logbook No. WHC-N-647-4

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment

Shipped to PNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S4006- 04 .B15	SAP Ref	9A	PNL summa	PNL# 065
S4006- 05 .B15	SAP Ref	9B	PNL summa	PNL# 064
S4006- 06 .B15	SAP Ref	19	PNL summa	PNL# 058
S4006- 07 .B15	SAP Ref	15A	PNL surrogate	PNL# 061
S4006- 08 .B15	SAP Ref	---	PNL surrogate	PNL# 071
S4006- 09 .B15	SAP Ref	---	PNL surrogate	PNL# 059

Field Transfer of Custody			Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time	
Thore Clauer	2/14/94	14:29				

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:
------------------	--------------	------------

Comments:

WHC purchased canisters are returned from PNL to WHC.
 PNL # above can be removed. PNL will not receive
 canister samples for this tank sample job.
 Thore Clauer
 2/14/94

Figure 1.1a Chain of Custody for Tank C-106 (Organic Samples)

Custody Form Initiator C. M. Jones/ J. E. Darling

Company Contact J. L. Huckaby

Telephone (509) 373-3443

Project Designation/Sampling Locations 200 East Tank Farm: Tank
241-C-104 vapor sample SAF S4004, 241-C-104

Collection Date 02- -94

Ice Chest No.

Field Logbook No. WHC-N-647-4

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment

Shipped to PNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S4004- 04 .B17	SAP Ref	11A	PNL summa	PNL# 074
S4004- 05 .B17	SAP Ref	11B	PNL summa	PNL# 056 -
S4004- 06 .B17	SAP Ref	22	PNL summa	PNL# 060
S4004- 07 .B17	SAP Ref	---	PNL surrogate	PNL# 072
S4004- 08 .B17	SAP Ref	---	PNL surrogate	PNL# 070
S4004- 09 .B17	SAP Ref	---	PNL surrogate	PNL# 067
S4004- 49 .B17	SAP Ref	18A	PNL surrogate	PNL# 057

[] Field Transfer of Custody

Chain of Possession

(Sign and Print Names)

Relinquished By	Date	Time	Received By	Date	Time
<i>Shane Clavin</i>	2/14/94	16:30			

Final Sample Disposition

Disposal Method:

Disposed by:

Date/Time:

Comments: WHC purchased canisters are returned from PNL to WHC.

PNL# above can be removed. PNL will not receive
canister samples for this ^{200E tank} ~~tank~~ sample job.
Shane Clavin
2/14/94

Figure 1.1b Chain of Custody for Tank C-106 (Organic Samples)

Westinghouse Hanford Company	CHAIN OF CUSTODY	PNL 006115
---------------------------------	------------------	------------

Custody Form Initiator C. M. Jones/ J. E. Darling

Company Contact J. L. Huckaby Telephone (509) 373-3443

Project Designation/Sampling Locations 200 East Tank Farm: Tank 241-C-106 vapor sample SAF S4006, 241-C-106 Collection Date 02-15-94

Ice Chest No. Field Logbook No. WHC-N-647-4

Bill of Lading/Airbill No. N/A Offsite Property No. N/A

Method of Shipment

shipped to PNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification					
S4006- 04 .B15	SAP Ref	9A	PNL summa	PNL# Can 061	
S4006- 05 .B15	SAP Ref	9B	PNL summa	PNL# Can 072	
S4006- 06 .B15	SAP Ref	19	PNL summa	PNL# Can 056	
S4006- 07 .B15	SAP Ref	15A	PNL surrogate	PNL# 2/15/94	
S4006- 08 .B15	SAP Ref	---	PNL surrogate	PNL# 2/15/94	
S4006- 09 .B15	SAP Ref	---	PNL surrogate	PNL# 2/15/94	

Field Transfer of Custody			Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time	
<i>[Signature]</i>	2-15-94	1645	<i>[Signature]</i> J.E. Darling	2-15-94	1645	
<i>[Signature]</i> J.E. Darling	2-18-94	0930	<i>[Signature]</i> B. Jean	2-18-94	1310	

Final Sample Disposition		
Disposal Method:	Disposed by:	Date/Time:
Comments:		

Figure 1.1c Chain of Custody for Tank C-106 (Organic Samples)

2.0 Inorganic Task

Solid sorbent traps were supplied to WHC for sampling the headspace of Tank C-106 using the VSS. Samples were returned to PNL for analysis. Analyses were performed to provide information on the tank-headspace concentration of the following analytes: ammonia (NH_3), nitrogen dioxide (NO_2), nitric oxide (NO), and water (H_2O). It is important to note that both sampling and analytical procedures were developmental as Tank C-106 was one of the first four trial sample jobs. Differences in sample preparation, sampling, and sample handling, compared with subsequent jobs, varied from those used after April 1994 (Ligotke et al. 1994). Analytical accuracy was estimated based on procedures used. Sample preparation and analyses were performed following PNL QA IL III requirements.

2.1 Standard Sampling Methodology

Standard glass tubes containing sorbent materials to trap vapors of selected analytes of NH_3 , NO_x , and H_2O (SKC Inc., Eighty Four, Pennsylvania) were obtained, prepared, and submitted for use by WHC. The sorbent traps were selected based on their use by the Occupational Safety and Health Administration (OSHA) to perform workplace monitoring, and because of available procedures and verification results associated with that particular application. The typical sorbent traps used consisted of a glass tube containing a sorbent material specific to the compound of interest. In general, the tubes contained two sorbent layers, or sections: the first layer was the primary trap, and the second layer provided an indication of breakthrough. In the tubes, sorbent layers are generally held in packed layers separated by glass wool. The sorbent tubes, having glass-sealed ends, were received from the vendor.

The type and nominal quantity of sorbent material varied by application. Sorbent traps selected for the tank sample job included the following products. The NH_3 sorbent traps contained carbon beads impregnated with sulfuric acid; nominally, 500 mg were contained in the primary and 250 mg in the breakthrough sections. The NH_3 was chemisorbed as ammonium sulfate $\{(\text{NH}_4)_2\text{SO}_4\}$. The NO_2 traps contained a zeolite impregnated with triethanolamine (TEA), with 200 mg in the primary and 100 mg in the breakthrough sections. The NO_2 was absorbed and disproportionated to equi-molar quantities of nitrite ions (NO_2^-) and nitrate ions (NO_3^-). The water trap contained 300 mg of silica gel in the primary and 150 mg in the breakthrough sorbent sections.

Samples provided by PNL to trap inorganic compounds included samples and single-trap blanks. The samples of each type were prepared from same-lot batches, with the NO_x sorbent traps having been stored previously in a freezer. After receipt of exposed and radiologically cleared samples from WHC and disassembly of the sorbent trains, samples were provided to the analytical laboratory at ambient temperature.

2.1.1 Concentration Calculations. The concentrations of target compounds in the tank headspace were determined from sample results, assuming effective sample transport to the sorbent traps. Concentration, in parts per million by volume (ppmv), was determined by dividing the mass of the compound, in μmol , by the volume of the dried tank air sampled in mol. The micromolar sample mass was determined by dividing the compound mass, in μg , by the molecular weight of the compound, in g/mol. The molar sample volume was determined, excluding water vapor, by dividing

the standard sample volume (at 0°C and 760 torr), in L, by 22.4 L/mol. For example, the concentration (C_v) of a 3.00-L sample containing 75.0 μg of ammonia equals

$$C_v = \frac{75.0 \mu\text{g}}{17 \text{ g/mol}} \left(\frac{3.00 \text{ L}}{22.4 \text{ mol}} \right)^{-1} = 32.9 \text{ ppmv} \quad (2.1)$$

This calculational method produces concentration results that are slightly conservative (greater than actual) because the volume of water vapor in the sample stream is neglected. The volume of water vapor is not included in the measured sampled volume because of its removal in desiccant traps upstream of the mass flowmeters. However, the bias is generally expected to be small. For a tank-headspace temperature of 35°C, the magnitude of the bias would be about 1 to 6%, assuming tank-headspace relative humidities of 20 to 100%, respectively. The concentration of mass (determined gravimetrically) was also per dry-gas volume at standard conditions.

2.2 Analytical Procedures

The compounds of interest were trapped using solid sorbents and chemisorption (adsorption of water vapor). Analytical results were based on extraction and analysis of selected ions. Analytical procedures used are specified herein and compiled in PNL-MA-599.

2.2.1 Ammonia Analysis. The sorbent material from the NH_3 -selective sorbent traps was placed into labeled 20-mL glass scintillation vials. Vials containing front-, or primary-, section sorbent material were treated with 10.0 mL of deionized water (DIW), and vials containing back-up-section sorbent material were treated with 5.0 mL of DIW. After extraction, the NH_3 sorbent traps were analyzed using the selective ion electrode (SIE) procedure PNL-ALO-226 {Ammonia (Nitrogen) in Aqueous Samples}. Briefly, this method includes 1) preparing a 1000- $\mu\text{g/mL}$ (ppm) NH_3 stock standard solution from dried reagent-grade NH_4Cl and DIW on the day analyses are performed; 2) preparing 0.1-, 0.5-, 1.0-, 10-, and 100-ppm NH_3 working calibration standards by serial dilution of the freshly made stock standard; 3) generating an initial calibration curve from the measured electromotive force (emf) signal versus NH_2 concentration data obtained for the set of working standards; 4) performing a calibration-verification check, using one of the midrange standards, after analyzing every 4 or 5 samples; 5) continuing this sequence until all samples of the batch have been measured, including duplicates and spiked samples; and 6) remeasuring the complete set of calibration standards (at the end of the session). Emf signal measurements obtained for samples are compared to those for standards, either graphically or algebraically (using linear regression), to determine NH_3 concentration in the samples.

2.2.2 Nitrite Analysis. The sorbent traps for NO_2 and NO were desorbed in an aqueous TEA and n-butanol solution and analyzed by suppressed-conductivity ion chromatography (SCIC) for nitrite according to PNL-ALO-212, Rev. 1 (*Determination of Inorganic Anions by Ion Chromatography*) modified to obviate interferences by concentrations of non-target analytes. Specifically, the modifications used were 1) eluent 1.44 mM Na_2CO_3 + 1.8 mM NaHCO_3 at 2.0 mL/min, 2) one guard column (AG4A) and two separator columns (AS4A) in series instead of just one separator column, and 3) all standards, samples, and blanks injected into the IC sample loop through 0.45- μm syringe filters.

For the analysis, the sorbent materials were placed into labeled 20-mL glass scintillation vials. To each vial, 3.0 mL of desorbing solution (15 g TEA + 1 mL n-butanol in 1.0 L DIW) was added.

Primary sorbent-tube sample materials and back-up (breakthrough) sorbent-trap materials were analyzed separately using identical procedures. Each analytical session was conducted as follows. Working nitrite standards (0, 0.1, 0.25, and 0.5 ppm) were prepared by diluting a stock nitrite standard with desorbing solution. An initial calibration curve was prepared from the instrument response (chromatographic peak height) versus nitrite standard concentration data for the set of working standards. A calibration verification check using one of the midrange standards was performed after the analysis of every six samples. If the instrument response indicated that sample nitrite concentration was outside the calibration range (> 0.5 ppm nitrite), the sample was diluted with desorbing solution and reanalyzed. After all samples of a batch were analyzed, the complete set of calibration standards was remeasured to verify consistent instrument response, and the analytical session was terminated.

Instrument responses (peak height) observed for samples were compared to those for standards to determine the nitrite concentration of the samples. Because NO_2 and NO converted to NO_2 were collected on the sorbent as equal quantities of nitrite and nitrate, and the analysis was specific for nitrite, the molar masses of NO_2 and NO were determined by doubling the analytically determined molar mass of nitrite.

2.2.3 Mass (Water) Analysis. Sorbent traps used to make each sample train were weighed using a semi-micro mass balance after labeling and breaking the glass tube ends, without plastic end caps. After receipt of exposed samples, the sorbent traps were again weighed to determine the change in mass. Records of the measurements were documented on sample-preparation data sheets. The mass concentration, generally roughly equal to the concentration of water, was determined by dividing the combined change in mass from all traps in a sorbent train by the actual volume of gas sampled. Blanks and spiked blanks were included to provide information on uncertainty.

2.3 Quality Assurance/Quality Control

Analytical work was performed according to quality levels identified in the project QA plan and several PNL documents. The samples were analyzed following PNL QA IL III. The PNL documents include PNL-MA-70 (Part 2), PNL-MA-599, PNL-ALO-212, PNL-ALO-226, PNL-ALO-271, and MCS-033. A summary of the analysis procedures and limits for the target inorganic compounds is provided in Table 2.1. From the table, it can be seen that the minimum detection limit (MDL) required to resolve the analyte at one-tenth of the recommended exposure limit (REL) for each of the target analytes is achieved using current procedures and with a vapor-sample volume of 3 L and a desorption-solution volume of 3 mL (10 mL for ammonia).

The accuracy of concentration measurements depends on errors associated with both sampling and analysis. Sampling information was provided by WHC. The accuracy of analytical results depends on the method used. For NH_3 analyses, the accuracy of laboratory measurements by SIE was estimated to be $\pm 5\%$ relative, independent of concentration at $1 \mu\text{g/mL}$ or greater levels. The uncertainty includes preparation of standards, purity of the ammonium salt used to prepare standards, potential operator bias, ambient temperature variations, etc. Unfortunately, no known National Institute for Standards and Technology (NIST)-traceable standard reference material (SRM) is available against which to compare working standards. As for NH_3 , no known NIST SRM is available for nitrite analysis (for NO_2 and NO). Based on experience in comparing nitrite working standards prepared from several different sources and factors mentioned for NH_3 above, the estimated maximum bias for samples derived from sampling for NO_2 is $\pm 10\%$, and for samples derived from

Table 2.1 Analysis Procedures and Detection Limits of Target Inorganic Analytes

<u>Analyte</u>	<u>Formula</u>	<u>Procedure</u>	<u>REL^(a)</u> <u>(ppmv)</u>	<u>0.1 x REL^(a)</u> <u>(ppmv)</u>	<u>MDL^(b)</u> <u>(ppmv)</u>
Ammonia	NH ₃	PNL-ALO-226	25	2.5	0.5
Nitrogen Dioxide	NO ₂	PNL-ALO-212	1	0.1	0.02
Nitric oxide	NO	PNL-ALO-212	25	2.5	0.02
Mass (water) ^(c)	n/a	n/a	n/a	n/a	n/a

(a) Target analytical limits are equal to one tenth of the REL.

(b) MDL is defined as the vapor concentration that can be detected with an uncertainty equal to about the magnitude of the measurement. The uncertainty is expected to reduce to about one quarter of the magnitude of the measurement at a concentration of four times the MDL. The MDLs were based on the assumption that 3 L of vapor are sampled; if greater volumes of vapor are sampled, correspondingly smaller MDLs can be achieved. The MDLs were also based on desorbing-solution volumes of 10 mL for NH₃ and 3 mL for the other analytes.

(c) The vapor-mass concentration, thought to be largely water vapor, is determined for estimates of humidity.

sampling for NO, it is $\pm 5\%$ relative. The accuracy of measurements of sample mass is ± 0.05 mg, or much less than 1% of the mass changes of most samples, and roughly 5% or less of the mass change of most blanks.

2.4 Inorganic Sample Results

Samples were obtained by WHC from the headspace of Tank C-106 on 2/15/94 using VSS. The sample job designation number was S4006. Samples were prepared, submitted to WHC, and then analyzed to provide information on the concentrations of NH₃, NO₂, NO, and H₂O. Sampling and analysis for SO_x and HCN were not requested. The inorganic samples were received from WHC on 3/4/94; the sample-volume information was received on 3/25/95.

A list of samples, sampling information, and sample volumes is shown in Table 2.2. The types of solid sorbent traps used are also shown in the table. Analytical mass and concentration results are shown in Table 2.3. Sample volumes were provided by WHC; sample-volume uncertainty was not provided. Tank-headspace concentration results (Table 2.3) are based on this information, and the listed uncertainties equal plus-or-minus one standard deviation of the individual results from each set of samples. Where analytical results from each set of samples were nearly indistinguishable from those of blanks, indicating very low vapor concentrations of the analyte, the concentration results (Table 2.3) are listed as "less than or equal to" a probable maximum value determined by subtracting the blank result from the sample result. Sample results were not corrected for the percentage recoveries of spiked samples.

2.4.1 Ammonia Results. The concentration of NH_3 was ≤ 9 ppmv. The NH_3 quantities found in the exposed sorbent traps were 0.1 to 1.2 μmol in the front sorbent sections and ≤ 0.06 μmol in the back sections (indicating no breakthrough). Blank corrections, generally ≤ 0.06 μmol per section, were used to correct the results. The results indicate that sample results are dependent on sample port number and indicate that Port 2 was not connected effectively to the headspace. Although spiked blanks were not tested, the percentage recoveries of three sets of blanks spiked with 12.2, 22.3, and 46.4 μmol NH_3 were $101 \pm 4\%$, $109 \pm 2\%$, and $104 \pm 1\%$, respectively, during related sample jobs (Clauss et al. 1994; Ligotke et al. 1994). Two sample analyses were duplicated after initial analysis and yielded repeatabilities of 109 and 106%; the relatively poor repeatabilities were attributed to the very small quantities of NH_3 found in the sorbent traps. No sample leachates were spiked after initial analysis to check percentage recovery.

Table 2.2 List of PNL Inorganic Samples, Controls, and Gravimetric Results Obtained From a Heated Tube Inserted into the Headspace of Tank C-106 on 2/15/94

Sample Port and Volume Information ^(a)					
<u>Sample Number</u>	<u>Sorbent Type</u>	<u>Sample Port</u>	<u>Flow Rate (mL/min)</u>	<u>Duration (min)</u>	<u>Volume (L)</u>
<u>Samples:</u>					
S4006- 23-B15	NH ₃ Trap	1	200	15.0	3.00
S4006- 24-B15	NH ₃ Trap	10	200	15.0	3.00
S4006- 37-B15	NH ₃ Trap	2	200	15.0	3.00
S4006- 38-B15	NH ₃ Trap	10	200	15.0	3.00
S4006- 46-B15	NH ₃ Trap	2	200	15.0	3.00
S4006- 47-B15	NH ₃ Trap	10	200	15.0	3.00
S4006- 19-B15	NO _x Trap	1	200	15.0	3.00
S4006- 20-B15	NO _x Trap	10	200	15.0	3.00
S4006- 35-B15	NO _x Trap	1	200	15.0	3.00
S4006- 36-B15	NO _x Trap	10	200	15.0	3.00
S4006- 44-B15	NO _x Trap	1	200	15.0	3.00
S4006- 45-B15	NO _x Trap	10	200	15.0	3.00
S4006- 21-B15	H ₂ O Trap	2	200	15.0	3.10
S4006- 25-B15	H ₂ O Trap	2	200	15.0	3.00
S4006- 26-B15	H ₂ O Trap	10	200	15.0	3.00
S4006- 39-B15	H ₂ O Trap	1	200	15.0	3.00
S4006- 40-B15	H ₂ O Trap	10	200	15.0	3.00
<u>Controls:</u>					
S4006- 50-B15	NH ₃ Spare	n/a ^(b)	n/a	n/a	n/a
S4006- 51-B15	NO ₂ Blank	n/a	n/a	n/a	n/a
S4006- 53-B15	H ₂ O Blank	n/a	n/a	n/a	n/a

(a) Sampling information and dry-gas sample volumes, corrected to 0°C and 760 torr, were provided by WHC.

Uncertainty values were not provided with sample-volume results.

(b) n/a = not applicable.

Table 2.3 Inorganic Vapor Sample Results Obtained From a Heated Tube Inserted into the Headspace of Tank C-106 on 2/15/94

Sample	Analytical Results (μmol)			Sample Volume (L)	Vapor Concentration (ppmv)
	Front Section	Back Section	Total ^(a) Blank-Corrected		
<u>NH₃ Samples:</u>			<u>$\leq 1.2^{(c)}$</u>	<u>3.00^(c)</u>	<u>$\leq 9^{(c)}$</u>
S4006- 23-B15	1.24	0.06	1.18	3.00	8.8
S4006- 24-B15	0.31	0.06	0.25	3.00	1.9
S4006- 37-B15	0.13	0.06	0.07 x ^(d)	3.00	0.5 x
S4006- 38-B15	0.29	0.06	0.23	3.00	1.7
S4006- 46-B15	0.09	0.06	0.03 x	3.00	0.2 x
S4006- 47-B15	0.33	0.06	0.27	3.00	2.0
<u>NO₂ Samples:</u>			<u>≤ 0.01</u>	<u>3.00</u>	<u>≤ 0.1</u>
S4006- 19-B15	0.0155	NA ^(d)	n/a ^(d)	3.00	n/a
S4006- 20-B15	0.0217	NA	n/a	3.00	n/a
S4006- 35-B15	0.0186	NA	n/a	3.00	n/a
S4006- 36-B15	0.0180	NA	n/a	3.00	n/a
S4006- 44-B15	0.0168	NA	n/a	3.00	n/a
S4006- 45-B15	0.0149	NA	n/a	3.00	n/a
<u>NO Samples:</u>			<u>≤ 0.009</u>	<u>3.00</u>	<u>≤ 0.1</u>
S4006- 19-B15	0.0212	NA	n/a	3.00	0.01
S4006- 20-B15	0.0236	NA	n/a	3.00	0.07
S4006- 35-B15	0.0194	NA	n/a	3.00	0.33
S4006- 36-B15	0.0220	NA	n/a	3.00	0.37
S4006- 44-B15	0.0220	NA	n/a	3.00	0.44
S4006- 45-B15	0.0193	NA	n/a	3.00	0.35
<u>Gravimetric Samples (mg.mg/L):</u>			<u>60 mg</u>	<u>3.00</u>	<u>20 $\pm$ 3 mg/L</u>
S4006- 21-B15	n/a	n/a	11.5 x	3.00	3.8 x
S4006- 25-B15	n/a	n/a	2.7 x	3.00	0.9 x
S4006- 26-B15	n/a	n/a	58.8	3.00	19.6
S4006- 39-B15	n/a	n/a	69.8	3.00	23.3
S4006- 40-B15	n/a	n/a	51.8	3.00	17.3

- (a) Total blank-corrected analyte masses (nitrite for NO₂ and NO) were determined, when significant, by subtracting the quantity of analyte found in blanks from that found in samples. The levels of analytes found in blanks are described in the subsections of Section 2.4.
- (b) Blank-corrected vapor concentrations as per Section 2.1.1. In the calculation for concentration, the nitrite values (listed) were doubled to account for unanalyzed nitrate.
- (c) Underlined values represent the average of the set of samples. Concentrations uncertainty equals ± 1 standard deviation (absolute) for each set of samples. The use of " $\leq$ " is defined in Section 2.4.
- (d) NA = not analyzed; n/a = not applicable; x = not included in determination of average concentration.

2.4.2 Nitrogen Oxides Results. Measurements of NO₂ and NO were made using six NO_x sorbent-trap trains (the NO_x trains consisted of NO₂ trap, oxidizer, and NO₂ trap). The concentrations of NO₂ and NO were each ≤ 0.1 ppmv. Blank-corrected NO₂ quantities in the sorbent traps averaged ≤ 0.01 μmol (NO₂ samples) and ≤ 0.009 μmol (NO samples). One nitrite blank was analyzed and used to correct data for each analyte: 0.0118 μmol for NO₂ and 0.0150 μmol for NO. Although spiked blanks were not tested, blanks

spiked with 0.0064, 0.047, 0.11, and 0.74 $\mu\text{mol NO}_2^-$ during related sample jobs yielded percentage recoveries of $153 \pm 14\%$, $103 \pm 4\%$, $106 \pm 8\%$, and $111 \pm 7\%$, respectively (Clauss et al. 1994; Ligothke et al.). No samples were reanalyzed to check repeatability. No sample leachates were spiked after initial analysis with quantities of NO_2^- to test analytical percentage recoveries.

2.4.3 Gravimetric Results. The mass concentration of material collected in the sorbent-trap trains, believed to be primarily water vapor, was $20 \pm 3 \text{ mg/L}$. The result was based on an average mass gain of 60 mg from three of five silica-gel sorbent traps. As with the NH_3 results, samples connected to Port 2 yielded low results. Only samples from Ports 1 and 10 were included in the determination of average vapor mass concentration.

3.0 Organic Task

3.1 SUMMA™ Canister Preparation

SUMMA™ canisters sent out to the field for sampling were new and clean, prehumidified with 100 μ L of distilled water, and labeled with a field-sampling identification number.

3.2 Sample Analysis Method

The SUMMA™ canister samples were analyzed according to PNL Technical Procedure PNL-TVP-03, *Determination of TO-14 Volatile Organic Compounds in Hanford Waste Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Gas Chromatographic-Mass Spectrometry Analysis*, which is a modified version of U. S. Environmental Protection Agency (EPA) compendium Method TO-14. The method uses an EnTech cryoconcentration system interfaced with a Hewlett Packard (HP) 5971 GC/MS. The EnTech concentrator is used to pull a metered volume of sample air from the SUMMA™ canister, cryogenically concentrate the air volume, then transfer the volume to the GC/MS for analysis. A 100-mL volume of sample is measured and analyzed from the tank headspace. The organic components in the sampled air are separated on an analytical column, J&W Scientific DB-1 phase, 60-m by 0.32-mm internal diameter with 3- μ m film thickness. The GC oven is programmed to run a temperature gradient beginning at 40°C, holding for 5 min, and ramping at 4°C per min to a final temperature of 260°C, with a 5-min hold.

3.3 Quality Assurance/Quality Control

Before the SUMMA™ tank samples were analyzed, a diagnostic check was performed on the GC/MS instrument by running an instrument "quick tune," as described in PNL-TVP-03. Upon satisfactory completion of the instrument diagnostic check, a blank volume of purified nitrogen was analyzed to check the cleanliness of the system. The instrument was then calibrated over six data points ranging from 2 ppbv to 100 ppbv, using a standard gas mixture containing 40 volatile organic compounds listed in EPA compendium Method TO-14. A gas mixture containing bromochloromethane, 1,4-difluorobenzene, and chlorobenzene- d_5 was used as an internal standard (IS) for all blank, calibration standard, and sample analyses. Analyte response from sample components, ISs, and standards were obtained from the extracted ion plot from their selected mass ion. The calibration curve was generated by calculating the relative response ratios of the IS to calibration standard responses and plotting the ratios against the ratio of the calibration-standard concentration (in ppbv) to the IS concentration. A least-squares linear-regression routine was applied to the data set to generate the best-fit line for each compound. The equation for that line was then used to quantify the TO-14 compounds found in the tank samples.

3.3.1 Quantitation of TO-14 Results. The quantitative-analysis results for the TO-14 volatile organic compounds were calculated directly from the calibration curve generated using the IS method described above and in PNL-TVP-03. The conversion from ppbv to mg/m^3 assumes standard temperature and pressure (STP) conditions of 760 torr and 273K and was calculated directly from the following equation:

$$\text{mg/m}^3 = \frac{(\text{ppbv}/1000) \times \text{g mol wt of compound}^{(a)}}{22.4 \text{ L/mole}} \quad (3.1)$$

3.3.2 Identification and Quantitation of Tentatively Identified Compounds. The tentatively identified compounds (TICs) are determined by mass-spectral interpretation and comparison of the spectra with the EPA/NIST/WILEY Library, which is a part of the HP 5971 instrument operating system. Chromatographic peaks with an area count greater than, or equal to, one half of the total area count of the chlorobenzene-d₅ IS peak at the 20-ppbv calibration level are tentatively identified and quantitatively estimated. This standard was chosen to determine the integration cutoff as it is in the middle of the chromatographic range and not in a region typically affected by coelution of other compounds. The quality of the mass-spectral searches was then reviewed by the principal investigators before the identification was assigned to each chromatographic peak.

The concentration of each TIC was estimated using a relative response factor calculated using a corrected total peak area for the IS chlorobenzene-d₅. Specifically, the total integrated area for the chlorobenzene-d₅ peak had to be corrected for possible coeluting compounds before calculating the response factor. The corrected total peak area for the IS was calculated by multiplying the IS quantitation ion by a correction factor based on the ratio of the total integrated peak area to the quantitation ion as measured in blank runs. The corrected peak area was then used to calculate a response factor using the IS concentration in mg/m³:

$$\text{Response Factor} = \frac{\text{IS conc. (mg/m}^3\text{)}}{\text{IS peak area}} \quad (3.2)$$

The calculated response factor was then multiplied by the TIC peak area to give an estimated concentration for that compound. For tridecane, the total peak area was multiplied by the response factor for chlorobenzene-d₅ to give an estimated concentration of 0.13 mg/m³. The ISs bromochloromethane and difluorobenzene were not used to quantitate the TICs because coeluting compounds appeared to have greatly altered the signal of the quantitation ions for those two ISs.

The ppbv concentrations are calculated from mg/m³ and the molecular weight of the analyte.

$$\text{TIC in ppmv} = \frac{\text{TIC (mg/m}^3\text{)} \times 22.4 \text{ L/mole} \times 1000}{\text{TIC g mol wt}^{(a)}} \quad (3.3)$$

The IS level added to all blank, standard, and sample injections was 18.3 ppbv for bromochloromethane, 20.3 ppbv for 1,4-difluorobenzene, and 18.2 ppbv for chlorobenzene-d₅. The IS concentrations were converted from ppbv to mg/m³ at STP using a molecular weight of 129.39 (g/mol) for bromochloromethane, 114.09 for 1,4-difluorobenzene, and 117.6 for chlorobenzene-d₅.

(a) Nominal molecular weight.

3.4 Analysis Results

The results from the GC/MS analysis of the tank-headspace samples are presented in Tables 3.1 and 3.2. A representative total ion chromatogram showing the identity of major constituents is given in Figure 3.1. Close inspection of the total ion chromatogram will indicate that two IS peaks, chlorobenzene-d5 and bromofluorobenzene, elute in two different places each in the data. The analysis was conducted overnight. The liquid nitrogen ran out during the stage in the Entech samplers' sampling sequence, at a point after it had already loaded ISs onto the trap. The liquid nitrogen stopped the sequence and restarted it. The trap was warmed and retained only the two less volatile ISs (bromochloromethane and difluorobenzene were lost), and those were valved onto the analytical column. Since they elute in different places than the ISs added after the sequence was restarted, and since sample was added only once, data quality has not been impacted. The IS areas meet all QC criteria.

Table 3.1 lists the quantitative results for compounds listed in Method TO-14. The levels of TO-14 analytes observed in the sample collected from Tank C-106 were below the quantitation limit (2 ppb). No average concentrations were calculated because only one canister contained analytes above the instrumental detection limit.

Table 3.2 lists the semiquantitative results for the TICs observed in the samples. Two compounds were observed above the instrument detection limit, a total average concentration 0.19 mg/m³. The normal paraffin hydrocarbons (NPH), defined as n-alkanes from C₁₁ to C₁₅, present included only tridecane, which accounted for 68% of the TIC compounds. It should be noted that because the SUMMA™ canisters were not heated at the time of analysis, the NPH concentrations listed after the retention time of decane may not be a true accounting of all the NPH in the sample. Similarly, polar compounds, which may adhere to the inside surface of the canister, may also be under represented in this analysis. In addition, 1-butanol was detected.

4.0 Conclusions

The concentrations of selected inorganic and organic compounds were determined from samples of the headspace of Tank C-106. It is important to note that the inorganic samples were obtained and analyzed using procedures that were being developed and that final procedures were not completed until later (Ligotke et al. 1994). Consequently, it is not known with certainty whether significant sampling or analytical errors occurred, and it is recommended that the headspace of Tank C-106 be sampled again using the improved methods should less qualified results be needed. Based on the NH_3 results, it is possible that sample port 2 was not effectively connected to the tank headspace, and results from samples connected to that port were neglected. The qualified results of inorganic samples were NH_3 (≤ 9 ppmv), NO_2 (≤ 0.1 ppmv), NO (≤ 0.1 ppmv), and mass concentration (20 ± 3 mg/L). The mass concentration was expected to consist largely of water vapor. Listed uncertainties reflect repeatability; actual uncertainties were not determined.

Two of the SUMMA™ canisters analyzed were originally identified as surrogate samples (which means they contained standards and were not considered clean canisters) on COCs 006115 and 006108 (Figures 1.1a and b). However, the composition and consistency of the TIC results suggest that the canisters did contain actual headspace samples. A small amount of the TO-14 compound methyl benzene (toluene) was above the instrumental detection limit in the one canister sample (S4006-06.B15) that was not sent as a surrogate sample. Small amounts of tridecane and 1-butanol appeared in the TIC analyses; the tridecane was about twice the level found in the blank. The 1-butanol is known to be a degradation product of TBP. Tank C-106 is vented, which may account for low concentrations of only a few organic compounds found in the headspace.

5.0 References

- Clauss, T. W., M. W. Ligotke, B. D. McVeety, K. H. Pool, R. B. Lucke, J. S. Fruchter, and S. C. Goheen. 1994. *Vapor Space Characterization of Waste Tank 241-by-104: Results from Samples Collected on 6/24/94*. PNL-10208. Pacific Northwest Laboratory, Richland, Washington.
- Ligotke, M. W., K. H. Pool, and B. D. Lerner. 1994. *Vapor Space Characterization of Waste Tank 241-C-103: Inorganic Results from Sample Job 7B (5/12/94)*. PNL-10172, Pacific Northwest Laboratory, Richland, Washington.

6.0 Further Reading

- Pacific Northwest Laboratory. Analytical Laboratory Procedure Compendium. Procedures PNL-ALO-212, -226, -271. PNL-MA-599, Richland, Washington.
- Pacific Northwest Laboratory. Quality Assurance Manual, Part 2: Good Practices Standard. PNL-MA-70, Part 2, Richland, Washington.
- Pacific Northwest Laboratory. Quality Assurance Plan for Activities Conducted by the Analytical Chemistry Laboratory (ACL). MCS-033, Analytical Chemistry Laboratory, Richland, Washington.

Pacific Northwest Laboratory. 1994. *Determination of TO-14 Volatile Organic Compounds in Hanford Waste Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Gas Chromatographic-Mass Spectrometry Analysis*, PNL-TVP-03 (Rev. 0), PNL Technical Procedure, Richland, Washington.

Pacific Northwest Laboratory. 1994. *Sample Shipping and Receiving Procedure - DRAFT for PNL Waste Tank Samples*. PNL-TVP-07 (Rev. 0), PNL Technical Procedure, Richland, Washington.

Table 3.1 TO-14 Analysis for Samples from the Headspace of Tank C-106 from SUMMA™ Canister Samples Collected on 2/15/94

TO-14 Analyte	CAS No.	Mol Wt	S4006-04.B15 ^(a)		S4006-05.B15 ^(a)		S4006-06.B15 ^(a)		Means and Standard Deviations	
			PNL 061 ^(b)		PNL 072 ^(b)		PNL 056 ^(b)			
			Concentration		Concentration		Concentration			
			(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	St Dev
Dichlorodifluoromethane (FREON-12)	75-71-8	120	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Methyl Chloride (Chloromethane)	74-87-3	50	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,2-Dichloro-1,1,2,2,-Tetrafluoroethane (FREON-114)	76-14-2	170	< 0.02	< 2	< 0.02	< 2	< 0.02	< 2	(c)	(c)
Chloroethene (Vinyl Chloride)	75-01-4	64	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Methyl Bromide (Bromomethane)	74-83-9	94	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Ethyl Chloride	75-00-3	62	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Trichlorofluoromethane (FREON-11)	75-69-4	136	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,1-Dichloroethene (1,1-Dichloroethylene)	75-35-4	96	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Dichloromethane (Methylene Chloride)	75-09-2	84	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,1,2-Trichloro-1,2,2-Trifluoroethane (FREON-113)	76-13-1	186	< 0.02	< 2	< 0.02	< 2	< 0.02	< 2	(c)	(c)
1,1-Dichloroethane	75-34-3	98	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
cis-1,2-Dichloroethene (cis-1,2-Dichloroethylene)	156-59-2	96	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Trichloromethane (Chloroform)	67-66-3	118	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
cis 1,2-Dichloroethane	107-06-2	98	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,1,1-Trichloroethane	71-55-6	132	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Benzene	71-43-2	78	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Carbon Tetrachloride	56-23-5	152	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,2-Dichloropropane	78-87-5	112	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Trichloroethylene	79-01-6	130	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
cis 1,3-Dichloropropene	61-01-5	110	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
trans 1,3-Dichloropropene	61-02-6	110	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,1,2-Trichloroethane	79-00-5	132	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Methyl Benzene (Toluene)	108-88-3	92	< 0.01	< 2	< 0.01	< 2	0.01	3.45	(c)	(c)
1,2-Dibromoethane	106-93-4	186	< 0.02	< 2	< 0.02	< 2	< 0.02	< 2	(c)	(c)
Tetrachloroethene (Perchloroethylene)	127-18-4	164	< 0.02	< 2	< 0.02	< 2	< 0.02	< 2	(c)	(c)
Chlorobenzene	108-90-7	112	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Ethylbenzene	100-41-4	106	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
m-Xylene (1,3-Dimethylbenzene) ^(d)	108-38-3	106	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
p-Xylene (1,4-Dimethylbenzene) ^(d)	106-42-3	106								
Styrene	100-42-5	104	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)

Table 3.1 (Contd)

TO-14 Analyte	CAS No.	Mol Wt	S4006-04.B15 ^(a)		S4006-05.B15 ^(a)		S4006-06.B15 ^(a)		Means and Standard Deviations	
			PNL 061 ^(b)		PNL 072 ^(b)		PNL 056 ^(b)			
			Concentration	Concentration	Concentration	Concentration	Concentration	Concentration		
			(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	St Dev
1,1,2,2-Tetrachloroethane	79-34-5	166	< 0.02	< 2	< 0.02	< 2	< 0.02	< 2	(c)	(c)
o-Xylene (1,2-Dimethylbenzene)	95-47-6	106	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,3,5-Trimethylbenzene	108-67-8	120	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,2,4-Trimethylbenzene	95-63-6	120	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
Chloromethylbenzene, alpha (Benzyl Chloride)	100-44-7	126	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
m-Dichlorobenzene (1,3-Dichlorobenzene)	541-73-1	146	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
p-Dichlorobenzene (1,4-Dichlorobenzene)	106-46-7	146	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
o-Dichlorobenzene (1,2-Dichlorobenzene)	95-50-1	146	< 0.01	< 2	< 0.01	< 2	< 0.01	< 2	(c)	(c)
1,2,4-Trichlorobenzene	120-82-1	180	< 0.02	< 2	< 0.02	< 2	< 0.02	< 2	(c)	(c)
Hexachloro-1,3-Butadiene	87-68-3	258	< 0.02	< 2	< 0.02	< 2	< 0.02	< 2	(c)	(c)

(a) WHC sample identification number.

(b) PNL canister number.

(c) Average and standard deviation are not meaningful for this analyte.

(d) m-Xylene and p-Xylene coelute; the reported concentration is the sum of these two compounds.

Table 3.2 Tentatively Identified Compounds and Estimated Concentrations^(a) in Headspace of Tank C-106 from SUMMATM Canister Samples Collected on 2/15/94

Tentatively Identified Compound ^(d)	CAS No. ^(d)	Mol. Wt.	Ret Time	S4006-04.B15 ^(b)		S4006-05.B15 ^(b)		S4006-06.B15 ^(b)		Means and Standard Deviations			
				PNL 061 ^(c)		PNL 072 ^(c)		PNL 056 ^(c)		(mg/m ³)	St Dev	(ppbv)	St Dev
				(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(e)	(e)	(e)	(e)
Acetone	67-64-1	58	8.433	< 0.03	< 10.00	0.06	22.29	< 0.03	< 10.00				
1-Butanol	71-36-3	74	17.52	0.06	19.02	0.06	18.75	0.06	18.04	0.06	0.00	18.4	0.5
Tridecane	629-50-5	184	47.98	0.10	12.13	0.13	16.13	0.15	17.67	0.13	0.03	16.9	1.09

- (a) Semi-quantitative estimate calculated using concentration of closest eluting internal standard.
 (b) WHC sample identification number.
 (c) PNL SUMMATM canister number.
 (d) Obtained by mass spectral interpretation and comparison with the EPA/NIST/WILEY Library.
 (e) Average and standard deviation are not meaningful for this analyte.

Figure 3.1a Total Ion Chromatogram (2 through 30 min) for Hanford Waste Tank C-106 SUMMA™ Canister Sample S4006-04.B15 Collected on 2/15/94

Figure 3.1b Total Ion Chromatogram (30 through 58 min) for Hanford Waste Tank C-106 SUMMA™ Canister Sample S4006-04.B15 Collected on 2/15/94

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		4	<u>Westinghouse Hanford Company</u> H. Babad, R2-78 D. R. Bratzel, S7-21 (2) J. E. Meacham, S7-15
		27	<u>Pacific Northwest Laboratory</u> S. C. Goheen, P8-08 (20) J. L. Huckaby, K6-55 M. W. Ligothe, P7-59 Technical Report Files (5)