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Waste Tank Vapor Program

**Vapor Space Characterization of
Waste Tank 241-TX-118 (*In Situ*):
Results from Samples Collected
on 9/7/94**

B. L. Thomas
T. W. Clauss
M. W. Ligothke
K. H. Pool

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October 1995

Prepared for Westinghouse Hanford Company
under a Related Services Agreement
with the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy



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WASTE TANK VAPOR PROGRAM

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Richland, Washington 99352

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Summary

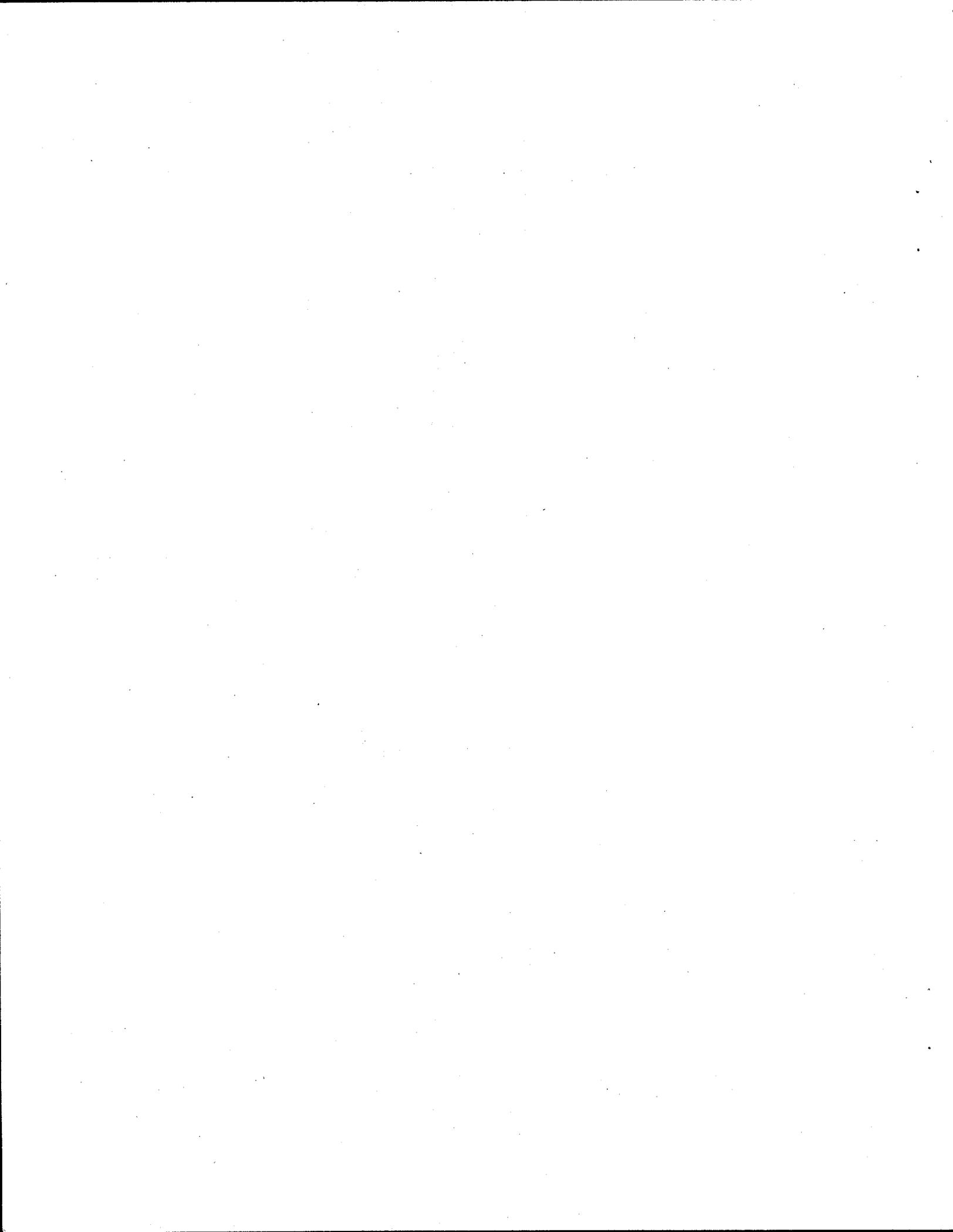
This report describes inorganic and organic analyses results from *in situ* samples obtained from the headspace of the Hanford waste storage Tank 241-TX-118 (referred to as Tank TX-118). The results described here were obtained to support safety and toxicological evaluations. A summary of the results for inorganic and organic analytes is listed in Table 1. Detailed descriptions of the results appear in the text.

Quantitative results were obtained for the inorganic compounds ammonia (NH₃), nitrogen dioxide (NO₂), nitric oxide (NO), hydrogen cyanide (HCN), and water (H₂O). Sampling for sulfur oxides (SO_x) was not requested. In addition, quantitative results were obtained for the 39 TO-14 compounds plus an additional 13 analytes. Hexane, normally included in the additional analytes, was removed because a calibration standard was not available during analysis of Tank TX-118 SUMMA™ canisters. Of these, 12 were observed above the 5-ppbv reporting cutoff. Fourteen tentatively identified compounds (TICs) were observed above the reporting cutoff of (ca.) 10 ppbv and are reported with concentrations that are semiquantitative estimates based on internal-standard response factors. The 10 organic analytes with the highest estimated concentrations are listed in Table 1 and account for approximately 86% of the total organic components in Tank TX-118. Permanent gas analysis was not conducted on the tank-headspace samples.

Table 1. Summary Results of Inorganic and Organic Samples Collected from the Headspace of Tank TX-118 on 9/7/94

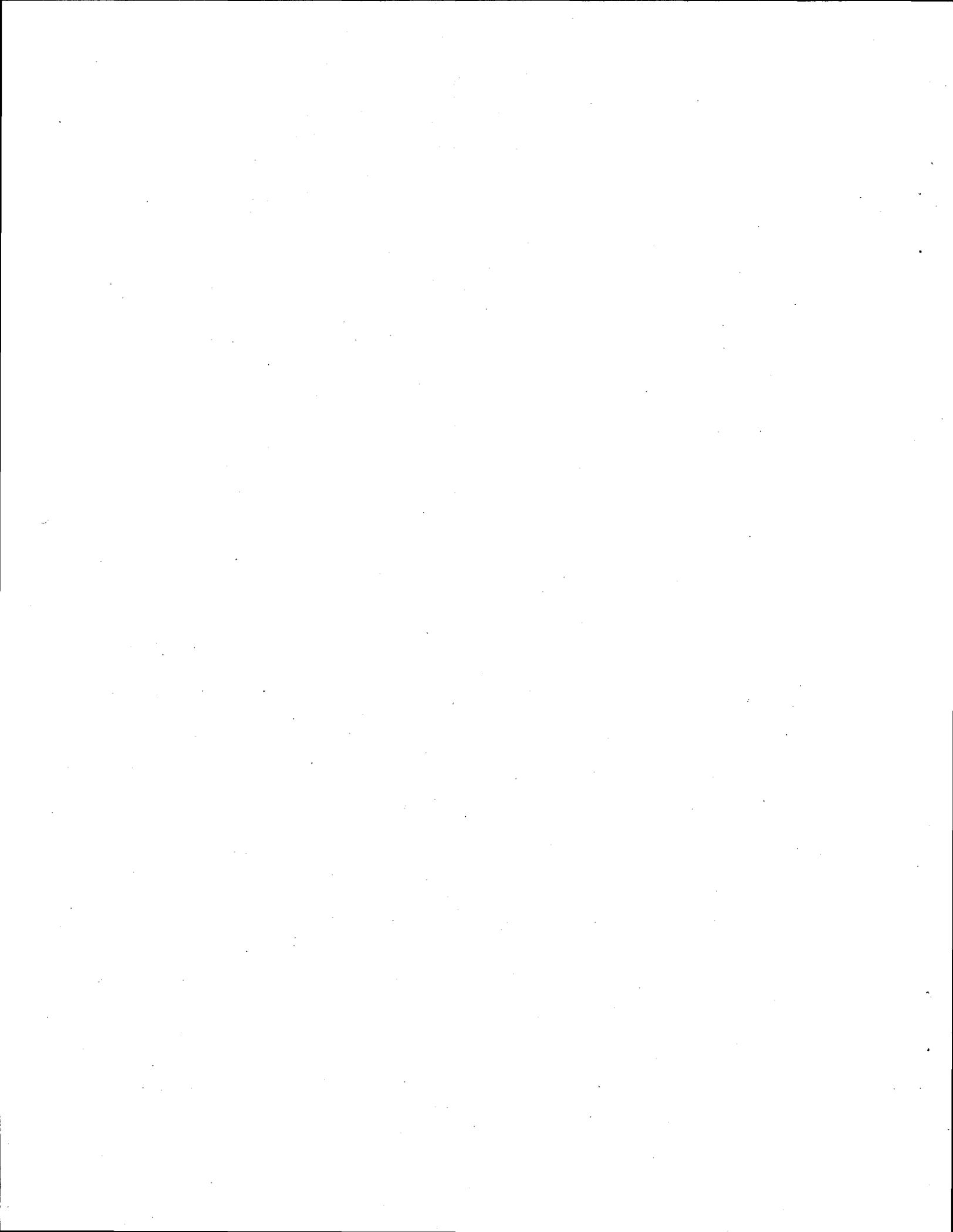
<u>Category</u>	<u>Analyte</u>	<u>Vapor^(a) Concentration</u>	<u>Units</u>
Inorganic	NH ₃	38 ± 1	ppmv
	NO ₂	≤ 0.02	ppmv
	NO	0.62 ± 0.01	ppmv
	HCN	≤ 0.02	ppmv
	H ₂ O	16 ± 2	mg/L
Organic	Acetone	3.39	mg/m ³
	Trichlorofluoromethane	2.44	mg/m ³
	1-Butanol	1.09	mg/m ³
	Methyl Alcohol	1.06	mg/m ³
	Ethanol	0.41	mg/m ³
	Propane	0.38	mg/m ³
	Unknown	0.36	mg/m ³
	3-Heptanone	0.33	mg/m ³
	Butanal	0.29	mg/m ³
	Hexane, 3-methyl	0.28	mg/m ³

(a) Vapor concentrations were determined using sample-volume data provided by Westinghouse Hanford Company and are based on averaged data.



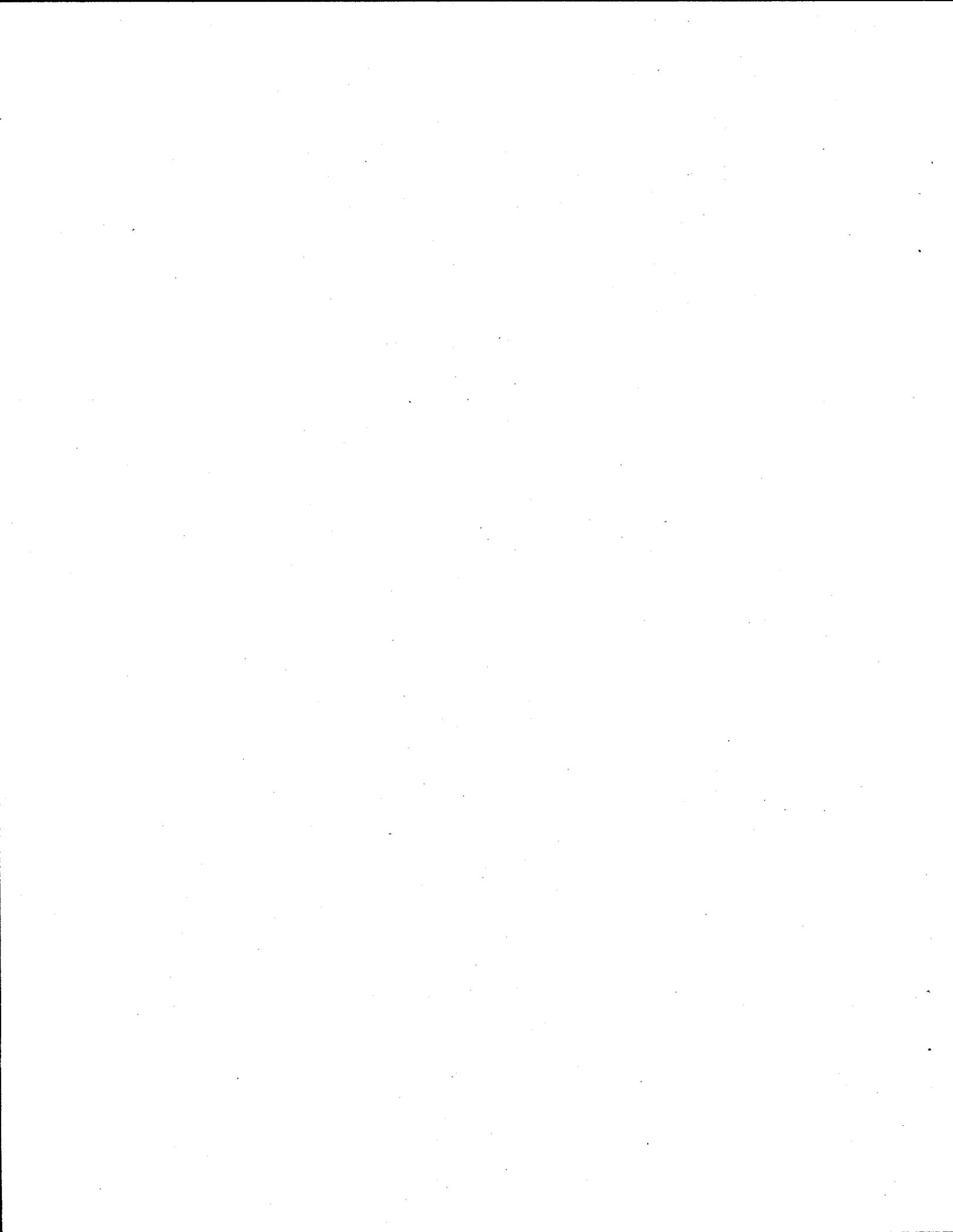
Acknowledgments

The authors gratefully acknowledge the support of other project staff at Pacific Northwest Laboratory (PNL) 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. Sally Slate, May-Lin Thomas, and Karen Schielke analyzed inorganic samples, and Gary Dennis prepared the solid-sorbent sample trains. Brenda M. Thornton provided word processing support.



Abbreviations

CAS	Chemical Abstracts Service
COC	chain of custody
C _v	concentration by volume
DIW	deionized water
emf	electromotive force
EPA	U.S. Environmental Protection Agency
GC/MS	gas chromatography/mass spectrometry
GC/TCD	gas chromatography/thermal conductivity detection
HP	Hewlett Packard
IC	ion chromatography
IL	impact level
IS	internal standard
ISS	<i>in situ</i> sampling system
MDL	minimum detection limit
NIST	National Institute for Standards and Technology
OSHA	Occupational Safety and Health Administration
PFA	perfluoroalkoxy
PNL	Pacific Northwest Laboratory
ppbv	part per billion by volume
ppmv	part per million by volume
QA	quality assurance
REL	recommended exposure limit
RPD	relative percent difference
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 *in situ* tank-headspace samples taken from the Hanford waste Tank 241-TX-118 (referred to as Tank TX-118). 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 and ambient air near the tank. The organic analytes for TO-14 compounds were extended to include 14 analytes identified by the Toxicological Review Panel for Tank C-103 and reported by Mahlum et al. (1994). Program management included these analytes for future tank analyses as identified in the fiscal year work plan. This plan is attached to a letter dated 9/30/94 and addressed to Mr. T. J. Kelly of WHC. The sample job was designated S4066, and samples were collected by WHC on September 7, 1994, using the *in situ* sampling system (ISS).

Sampling devices, including six sorbent trains (for inorganic analyses), and four SUMMA™ canisters (for organic analyses) were supplied to the WHC sampling staff on August 15. Samples were taken (by WHC) from the tank headspace on September 7 and were returned to PNL from the field on September 8. Inorganic (sorbent trap) samples actually used to sample TX-118 (see above note) were delivered to PNL on chain-of-custody (COC) 007506 (see Figure 1.1a). The SUMMA™ canisters were delivered on COC 007505 (see Figure 1.1b).

The samples were inspected upon delivery to the 326/23B laboratory and logged into PNL record book 55408 before implementation of PNL Technical Procedure PNL-TVP-07^(b). 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 canister was stored in the 326/23B laboratory at ambient (25°C) temperature until the time of 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. In summary, sorbent traps for inorganic analyses containing sample materials were either weighed (for water analysis) or weighed and desorbed with the appropriate aqueous solutions (for hydrogen cyanide, ammonia, nitrogen dioxide, and nitric oxide analyses). The aqueous extracts were analyzed by either selective electrode or ion chromatography (IC). 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.

(b) PNL-TVP-07, Rev. 0, October 1994, *Sample Shipping and Receiving Procedure for PNL Waste Tank Samples*, PNL-Technical Procedure, Tank Vapor Project, Richland, Washington.

Westinghouse Hanford Company	CHAIN OF CUSTODY	WHC 007506
---	-------------------------	-------------------

Custody Form Initiator	J. A. Edwards	Telephone	(509) 373-0141
		Pager	85-3009
Company Contact	L. A. Pingel	Telephone	(509) 373-4051
Project Designation/Sampling Locations	200 East Tank Farm	Collection Date	5-4-94 / 08-07-94
241-TX-118 Tank	Vapor Sample SAF S4066	Preparation Date	08-15-94
Ice Chest No.	(IN-SITU)	Field Logbook No.	WHC-N-____-
Bill of Lading/Airbill No.	N/A	Offsite Property No.	N/A
Method of Shipment	Government Truck		
Shipped to	WHC		

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

- | | |
|-----------------------------|--|
| S4066 - SOL .61W | NH ₃ /NO _x /H ₂ O (Sample #1) |
| S4066 - SOL .62W | NH ₃ /NO _x /H ₂ O (Sample #2) |
| S4066 - SOL .63W | NH ₃ /NO _x /H ₂ O (Sample #3) |
| S4066 - SOL .64W | NH ₃ /NO _x /H ₂ O (Spare) |
| S4066 - SOL .65W | HCN/H ₂ O (Sample #1) |
| S4066 - SOL .66W | HCN/H ₂ O (Sample #2) |
| S4066 - SOL .67W | HCN/H ₂ O (Sample #3) |
| S4066 - SOL .68W | HCN/H ₂ O (Spare) |
| S4066 - SOL .69W | OVS/Drier (Radiological survey) |
- SMB 9/10/94

[X] Field Transfer of Custody			[] Chain of Possession			(Sign and Print Names)		
Relinquished By	Date	Time	Received By	Date	Time	Relinquished By	Date	Time
J. A. Edwards	08-15-94	0830	Andy Johnson	08-15-94	0850	J. A. Edwards		
LAP	9-2-94	0730	LAP	9-2-94	1300	LAP		
LAP	9-7-94	1310	SM Brown	9-7-94	1300	LAP		
SM Brown	9-8-94	1100	Hmit Sharma	9-8-94	1330	SM Brown		

(version 02-28-94)

Final Sample Disposition

Disposal Method:
 Disposed by:
 Date/Time:
 Comments

A-6000-407 (12/92) WEF061

Figure 1.1a Chain-of-Custody for Inorganic Samples for Tank TX-118

Westinghouse Hanford Company	CHAIN OF CUSTODY	WHC 007505
---	-------------------------	-------------------

Custody Form Initiator	J. A. Edwards	Telephone	(509) 373-0141
		Pager	85-3009
Company Contact	L. A. Pingel	Telephone	(509) 373-4051
Project Designation/Sampling Locations	200 East Tank Farm	Collection Date	08-07-94
241-TX-118 Tank	Vapor Sample SAF S4066	Preparation Date	08-17-94
Ice Chest No.	(IN-SITU)	Field Logbook No.	WHC-N-____
Bill of Lading/Airbill No.	N/A	Offsite Property No.	N/A
Method of Shipment	Government Truck		
Shipped to	WHC		
Possible Sample Hazards/Remarks	Unknown at time of sampling		

Sample Identification

S4066 - S44 .060	(PNL) SUMMA Ambient Air
S4066 - S44 .086	(PNL) SUMMA Sample #1
S4066 - S44 .180	(PNL) SUMMA Sample #2
S4066 - S44 .184	(PNL) SUMMA Sample #3

[X] Field Transfer of Custody		[] Chain of Possession				(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time		
J. A. Edwards <i>J. Edwards</i>	08-17-94	08:58	Tim Utecht <i>T-B Utecht</i>	08-17-94	08:58		
<i>L.A.P.</i>	9-2-94	07:30	<i>L.A.P.</i>	9-2-94	13:00		
<i>L.A.P.</i>	9-7-94	13:10	<i>SM (Brown)</i>	9-7-94	13:10		
<i>SM Brown</i>	9-8-94	11:00	<i>Amit Sharma</i>	9-8-94	12:30		

(version 02-28-94)

Final Sample Disposition

Disposal Method:

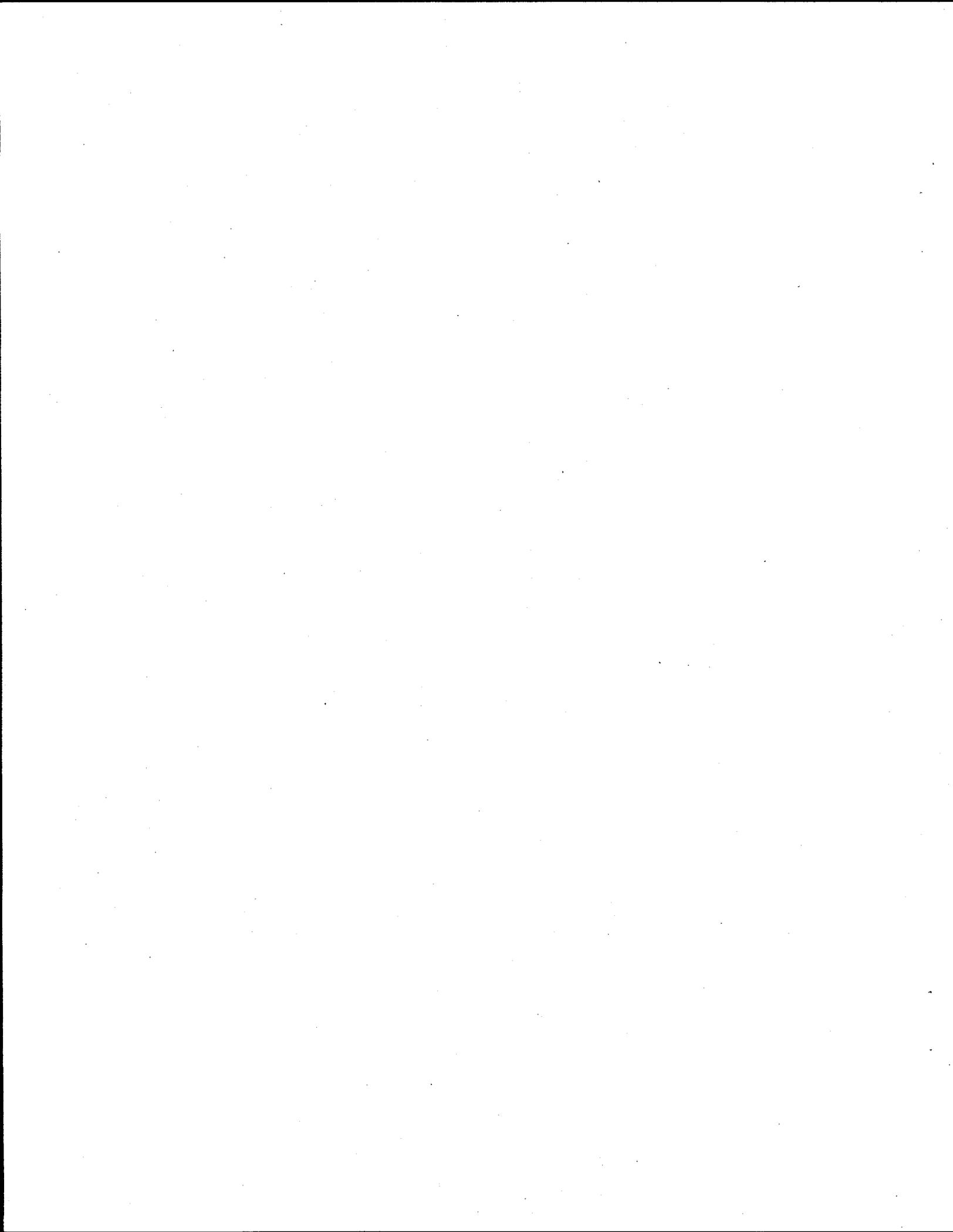
Disposed by:

Date/Time:

Comments

A-6000-407 (12/92) WEF061

Figure 1.1b Chain-of-Custody for Organic Samples for Tank TX-118



2.0 Inorganic

Solid sorbent traps, prepared in sampling trains, were supplied to WHC for sampling the tank headspace using the ISS. Controls and exposed samples were returned to PNL for analysis. Analyses were performed to provide information on the tank-headspace concentration of the following analytes: ammonia (NH₃), nitrogen dioxide (NO₂), nitric oxide (NO), hydrogen cyanide (HCN), and water (H₂O). Procedures were similar to those developed previously during sample jobs performed with the vapor sampling system (VSS) connected to the tank headspace of Tank C-103 (Ligotke et al. 1994). Analytical accuracy was estimated based on procedures used. Sample preparation and analyses were performed following PNL quality assurance (QA) impact level (IL) III requirements.

2.1 Standard Sampling Methodology

Standard glass traps containing sorbent materials to trap vapors of selected analytes of NH₃, NO₂, NO, HCN, and H₂O (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. Sorbent traps were connected end-to-end to prepare multi-trap sorbent trains for sampling.

The type and nominal quantity of sorbent material varied by application. Sorbent traps were selected for the tank sample job and included the following products. The NH₃ 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 NH₃ was chemisorbed as ammonium sulfate {(NH₄)₂SO₄}. The NO₂ traps contained a zeolite impregnated with triethanolamine (TEA), with 400 mg in the primary and 200 mg in the breakthrough sections. The NO₂ was absorbed and disproportionated to equi-molar quantities of nitrite ions (NO₂⁻) and nitrate ions (NO₃⁻). Glass tubes containing 800 mg of an oxidant such as chromate were used to convert NO to NO₂. The converted NO was then collected as nitrite and nitrate in an NO₂ trap. The HCN traps contained soda lime, with 600 mg in the primary and 200 mg in the breakthrough sections. The water traps contained 300 mg of silica gel in the primary and 150 mg in the breakthrough sections.

Samples provided by PNL to trap inorganic compounds include all or some of the following: samples, spiked samples, spares, blanks, and spiked blanks. The samples of each were prepared from same-lot batches, with the oxidizer sections of the NO_x sorbent trains having been stored previously in a freezer. After sample preparation, all samples, spiked samples, blanks, and spiked blanks were stored at ≤ 10°C, primarily because of handling recommendations for the oxidizer tubes attached to some samples. 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

(a) Pacific Northwest Laboratory. 10/94. *Sorbent Trap Preparation for sampling and Analysis: Waste Tank Inorganic Vapor Samples*, PNL-TVP-09 (Rev.0), PNL Technical Procedure, Richland, Washington.

temperature, and selected oxidizer sections were returned to a freezer until completion of analyses.

The sorbent traps were prepared in multi-trap sorbent trains configured so sample flow passed in order through the traps, targeting specific analytes, and then through a desiccant trap. The specific order of traps within the various sorbent trains is described in Section 2.4. The ends of the glass-tube traps were broken, and the traps were weighed and then connected to each other using uniform lengths of 3/8-in. perfluoroalkoxy (PFA)-grade Teflon® tubing. The tubing was heated in hot air and forced over the open ends of the traps to form a tight seal. Both the inlet and outlet ends of the sorbent trains (the downstream ends of the traps always contained silica gel) were each sealed with red-plastic end caps provided by the manufacturer. The leading and trailing ends of the sorbent trains remained sealed other than during the actual sampling periods. C-Flex® tubing was provided by WHC to connect the downstream ends of the sorbent trains to the sampling exhaust manifold connections.

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 compound 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 the conditions used by WHC; 21.1°C and 760 torr), in L, by 24.1 L/mol. For example, the concentration (C_v) of a 3.00-L sample containing 75.0 μg of NH_3 equals

$$C_v = \frac{75.0 \mu\text{g}}{17 \text{ g/mol}} \left(\frac{3.00 \text{ L}}{24.1 \text{ L/mol}} \right)^{-1} = 35.4 \text{ ppmv} \quad (2.1)$$

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 flowmeter. 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 in the text. All are 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^(a). Briefly, this method includes 1) preparing a 1000- $\mu\text{g}/\text{mL}$ (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_3 concentration data obtained for the set of working standards; 4) performing a calibration-verification check, using one of the midrange standards, after analyzing every four or five 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^(b) 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.

-
- (a) Procedure entitled "Ammonia (Nitrogen) in Aqueous Samples," PNL-ALO-226, in the *Analytical Chemistry Laboratory (ACL) Procedure Compendium*, Vol. 3: Inorganic Instrumental Methods. Pacific Northwest Laboratory, Richland, Washington.
- (b) Procedure entitled "Determination of Inorganic Anions by Ion Chromatography," PNL-ALO-212, in the *Analytical Chemistry Laboratory (ACL) Procedure Compendium*, Vol. 3: Inorganic Instrumental Methods. Pacific Northwest Laboratory, Richland, Washington.

2.2.3. Cyanide Analysis. The HCN samples were desorbed in 3.0 mL of 0.02 N sodium hydroxide and analyzed by amperometric detection ion chromatography according to PNL-ALO-271^(a). Calibration standards, typically 0, 20, 50, and 100 ppb CN⁻, were prepared from a stock 1000 ppm CN⁻ standard on the day of sample analysis in 0.02 N NaOH matrix. The same analysis sequence described above in Section 2.2.2 was used. Instrument responses (peak height) observed for samples were compared to those for standards to determine the CN⁻ concentration of the samples.

2.2.4 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, presumed to be dominated by water vapor, was determined by dividing the combined change in mass from all traps in a sorbent train by the actual volume of gas sampled. Controls were used 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 impact level (IL) III. The PNL documents include some or all of the following: 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

Table 2.1 Analysis Procedures and Typical Detection Limits of Target Inorganic Analytes.

Analyte	Formula	Procedure	REL ^(a) (ppmv)	0.1 x REL ^(a) (ppmv)	MDL ^(b) (ppmv)
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
Hydrogen Cyanide	HCN	PNL-ALO-271	4.8	0.48	0.01
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 gravimetrically.

(a) Procedure entitled "Procedure for Analysis of Free Cyanide in Water and Solid Sample Leachates" PNL-ALO-271, in the *Analytical Chemistry Laboratory (ACL) Procedure Compendium*, Vol. 3: Inorganic Instrumental Methods. Pacific Northwest Laboratory, Richland, Washington.

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 NH₃).

The accuracy of concentration measurements depends on errors associated with both sampling and analysis (see Section 2.4). Sampling information, including sample volumes, was provided by WHC; sample volume uncertainty was not provided. The accuracy of analytical results depends on the method used. For NH₃ 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. Similarly, no known NIST SRM is available for nitrite analysis (for NO₂ and NO). Based on experience in comparing nitrite working standards prepared from several different sources and factors mentioned for NH₃ above, the estimated maximum bias for samples derived from sampling for NO₂ is $\pm 10\%$, and for samples derived from 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. The analytical accuracy of measurements of the change in mass of sorbent trains is typically about ± 2 mg per 5-trap sorbent train.

2.4 Inorganic Sample Results

Sorbent trap trains and controls were prepared on 8/13/94, submitted to WHC on 8/15/94, and used by WHC to sample the tank headspace of Tank TX-118 on 9/7/94 using the ISS. The sample job designation number was S4066. The exposed samples were returned to PNL on 9/8/94 and subsequently analyzed on 9/12/94 (H₂O), 9/16/94 (NH₃), 9/15 and 9/16/94 (NO_x), and 9/15/94 (HCN) to provide information on the vapor-space concentrations of selected inorganic compounds. Sampling and analysis for sulfur oxides was not requested. The sample-volume information was received from WHC on 11/15/94.

A list of samples, sampling information, sample volumes, and gravimetric results is shown in Table 2.2. The types of sample trains used and the order of sorbent traps within each train are also shown in the table. For example, the sorbent train NH₃/NO_x/H₂O contained an NH₃ trap at the inlet end, an NO_x series in the middle (Section 2.4.2), and a desiccant trap at the outlet end. 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 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 average of the blanks less one standard deviation from the average of the samples plus one standard deviation. Results of control samples, such as spiked blanks, are discussed in this section. Spiked blanks, when used, were transported to the field but not opened. Spiked samples, when used, were opened in the field and used to collect tank vapors. Sample results were not corrected for the percentage recoveries of spiked blanks.

Table 2.2 List of PNL Inorganic Samples, Blanks, and Gravimetric Results Obtained From *In Situ* Sampling of the Headspace of Tank TX-118 on 9/7/94

<u>Sample Number</u>	<u>Sorbent Type</u>	<u>Sample Port and Volume Information^(a)</u>			
		<u>Flow Rate (mL/min)</u>	<u>Duration (min)</u>	<u>Volume (L)</u>	<u>Mass Gain (g)</u>
<u>Samples:</u>					
S4066-066-61W	NH ₃ /NO _x /H ₂ O Sample	270	15	4.05	0.0625
S4066-066-62W	NH ₃ /NO _x /H ₂ O Sample	264	15	3.96	0.3654 ^(b)
S4066-066-63W	NH ₃ /NO _x /H ₂ O Sample	269	15	4.04	0.0703
S4066-066-64W	NH ₃ /NO _x /H ₂ O Blank/Spare	n/a ^(c)	n/a	n/a	0.0017
S4066-066-65W	HCN/H ₂ O Sample	240	15.0	3.60	0.0570
S4066-066-66W	HCN/H ₂ O Sample	206	15.0	3.08	0.0508
S4066-066-68W	HCN/H ₂ O Sample	212	15.0	3.17	0.0605
S4066-066-67W	HCN/H ₂ O Blank/Spare	n/a	n/a	n/a	-0.0008

- (a) Sampling information and dry-gas sample volumes, corrected to 21°C and 760 torr, were provided by WHC. Uncertainty values were not provided with sample-volume results.
- (b) Reason for unusual mass change not determined. Sample result neglected from analysis.
- (c) n/a = not applicable.

2.4.1 Ammonia Results. The concentration of NH₃ was 38 ± 1 ppmv, based on all three samples. The NH₃ quantities in the sorbent traps ranged from 6.2 to 6.4 μmol. Blank corrections, ≤ 0.06 μmol in front and ≤ 0.03 μmol in back sorbent sections, were not significant (≤ 1%) and were neglected. Although spiked blanks were not tested, the percentage recoveries of three sets of blanks spiked with 12.2, 22.3, and 46.4 μmol of NH₃ were 101 ± 4%, 109 ± 2%, and 104 ± 1%, respectively, during related sample jobs (Clauss et al. 1994; Ligothke et al. 1994). The analysis of one sample was duplicated and yielded a repeatability of ± 1%. Two sample leachates were spiked after initial analysis with roughly the quantity of NH₃ in the sample and yielded percentage recoveries of 102 and 115%. An unknown error occurred during the spike, yielding the 115% recovery; consequently, a second spike was performed, yielding 102% recovery. A 5-point calibration was performed over an NH₃ range of 0.1 to 1000 μg/mL.

2.4.2 Nitrogen Oxides Results. Measurements of NO₂ and NO were made using three 5-segment NH₃/NO_x/H₂O sorbent-trap trains (the NO_x trains consisted of NO₂ trap, oxidizer, and NO₂ trap). Related sample jobs, performed using the vapor sampling system (VSS) in Tanks BY-104, -105, and -106 both with and without NO_x trains protected by a leading NH₃ trap (e.g., Clauss et al. 1994), indicated that the presence of the upstream NH₃ traps resulted in NO concentrations that were about 1.3- to 1.6-fold less than those from unprotected NO₂ traps. The NO₂ concentrations were also potentially less following an NH₃ trap.

The concentrations of NO₂ and NO were ≤ 0.02 and 0.62 ± 0.01 ppmv, respectively. Blank-corrected NO₂ quantities in the sorbent traps averaged ≤ 0.002 μmol (NO₂ samples) and 0.052 μmol (NO samples). Nitrite blank levels used to correct data were 0.0140 ± 0.0006 μmol in front and were not analyzed in back sorbent sections. The nitrite blank levels were based on the

analytical results from the spare/blank sorbent train. Although spiked blanks were not tested, blanks spiked with 0.0064, 0.047, 0.11, and 0.74 μmol of 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. 1994). 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. A 4-point calibration was performed over a concentration range of 0 to 0.5 $\mu\text{g NO}_2^-$ per mL in the desorbing matrix.

2.4.3 Hydrogen Cyanide Results. The concentration of HCN was ≤ 0.02 ppmv. The samples contained $0.0157 \pm 0.0013 \mu\text{mol}$ in front and $0.0020 \pm 0.0001 \mu\text{mol CN}^-$ in back sections. The blank contained $0.0148 \mu\text{mol}$ in the front and $0.0021 \mu\text{mol CN}^-$ in the back section. Blanks spiked with $0.0099 \pm 0.0001 \mu\text{mol CN}^-$ were found previously to yield an average blank-corrected percentage recovery of $69 \pm 10\%$ after a hold time of 11 weeks (ISS sample job in BY-111). It is possible that the relatively poor spike recovery was related to the relatively long hold time. No samples were reanalyzed to check repeatability. No sample leachates were spiked after initial analysis with quantities of CN^- to test analytical percentage recoveries.

2.4.4 Gravimetric Results. The mass concentration of material collected in the sorbent-trap trains, believed to be primarily water vapor, was $16 \pm 2 \text{ mg/L}$. The result was based on an average blank-corrected mass gain of 58 mg from 5 of 6 sets of $\text{NH}_3/\text{NO}_x/\text{H}_2\text{O}$ and $\text{HCN}/\text{H}_2\text{O}$ sample trains. The actual mass gains were corrected by subtracting a blank mass gain of 2 mg. The blank correction was determined as the average of blank sorbent trap trains from six related ISS sample jobs, a group from which the individual results ranged between - 1 and + 4 mg (NH_3 trains) and - 1 and + 6 mg (HCN trains). The overall measurement uncertainty was estimated based on the variability of the samples and the range of blank data. Although no spiked blanks were tested, the percentage recovery of mass from three blank H_2O traps spiked with 51 mg water was $103 \pm 2\%$ during a related sample job (Clauss et al. 1994).

Table 2.3 Inorganic Vapor Sample Results Obtained From *In Situ* Samples Inserted into the Headspace of Tank TX-118 on 9/7/94

Sample	Analytical Results (μmol)			Sample Volume (L)	Vapor ^(a) Concentration (ppmv)
	Front Section	Back Section	Total ^(b) Blank-Corrected		
<u>NH₃ Samples:</u>			<u>6.3^(c)</u>	<u>4.02^(c)</u>	<u>38 ± 1^(c)</u>
S4066-066-61W	6.2	≤0.03	6.2	4.05	37
S4066-066-62W	6.4	NA ^(d)	6.4	3.96	39
S4066-066-63W	6.4	NA	6.4	4.04	38
<u>NO₂ Samples:</u>			<u>≤0.002</u>	<u>4.02</u>	<u>≤ 0.02</u>
S4066-066-61W	0.0136	0.0071	n/a ^(d)	4.05	n/a
S4066-066-62W	0.0131	NA	n/a	3.96	n/a
S4066-066-63W	0.0134	0.0075	n/a	4.04	n/a
<u>NO Samples:</u>			<u>0.052</u>	<u>4.02</u>	<u>0.62 ± 0.01</u>
S4066-066-61W	0.0652	0.0079	0.051	4.05	0.61
S4066-066-62W	0.0652	NA	0.051	3.96	0.62
S4066-066-63W	0.0667	0.0087	0.053	4.04	0.63
<u>HCN Samples:</u>			<u>≤ 0.002</u>	<u>3.28</u>	<u>≤ 0.02</u>
S4066-066-65W	0.0153	0.0021	n/a	3.60	n/a
S4066-066-66W	0.0172	0.0019	n/a	3.08	n/a
S4066-066-68W	0.0146	0.0020	n/a	3.17	n/a
<u>Gravimetric Samples (mg.mg/L):</u>			<u>58 mg</u>	<u>3.65</u>	<u>16 ± 2 mg/L</u>
S4066-066-61W	n/a	n/a	60	4.05	14.8
S4066-066-62W	n/a	n/a	NA	3.69	NA
S4066-066-63W	n/a	n/a	68	4.04	16.8
S4066-066-65W	n/a	n/a	55	3.60	15.3
S4066-066-66W	n/a	n/a	49	3.08	15.9
S4066-066-68W	n/a	n/a	58	3.17	18.3

- (a) Blank-corrected vapor concentrations were calculated using WHC-reported dry-air sample volumes (corrected to 21°C and 760 torr). In the calculation for concentration, the nitrite values (listed) were doubled to account for unanalyzed nitrate. Sample results were not corrected for percentage recovery of spiked samples or spiked blanks.
- (b) 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 level of analytes found in blanks is described in the subsections of Section 2.4.
- (c) Underlined values represent the average of the set samples. Concentration uncertainty equals ± 1 standard deviation (absolute) for each set of samples other than mass concentration. The uncertainty in mass concentration was determined based on the added uncertainty caused by the range of results of six related blanks. The use of "≤" is defined in Section 2.0.
- (d) NA = not analyzed. Only selected back sorbent sections were analyzed. n/a = not applicable.

3.0 Organic

3.1 SUMMA™ Canister Preparation

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant free according to PNL Technical Procedure PNL-TVP-02^(a). The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis by PNL Technical Procedure PNL-TVP-01^(b), which is a modification of U.S. Environmental Protection Agency (EPA) compendium Method TO-14. If the canister is verified as clean, free of TO-14 and unknown contaminants to a level of 5 parts per billion by volume (ppbv), the canister is evacuated to 5 mtorr, tagged, and stored for use in the field. Before sending the canisters out to the field for sampling, the canister vacuum is measured to determine if any leakage has occurred. If the vacuum has remained constant during storage, the canisters are prehumidified with 100 μ L of distilled water and labeled with a field-sampling identification. Canisters stored more than 30 but less than 60 days are re-evacuated and rehumidified before use. If stored more than 60 days, the canisters are recleaned and validated before use.

3.2 Sample Analysis Methods

The SUMMA™ canister sample was analyzed according to PNL Technical Procedure PNL-TVP-03^(c), which is a modified version of EPA compendium Method TO-14. The method uses an EnTech cryoconcentration system interfaced with either a 5971 or a 5972 Hewlett Packard (HP) benchtop 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. Twenty-four hours before the analysis, the SUMMA™ canister samples were pressurized with purified air (Aadco Instruments, Inc., 1920 Sherwood St., Clearwater, Florida 34625). The starting pressure was first measured using a calibrated diaphragm gauge (Cole Parmer), then pressurized to a level exactly twice the original pressure. For example, if the canister had a starting pressure of 740 torr, it was pressurized to 1480 torr. This dilution was an effort to improve the precision of the analysis. The sample dilution was taken into account when calculating the analysis results.

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- (a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, Richland, Washington.
 - (b) Pacific Northwest Laboratory. 8/94. *Determination of TO-14 Volatile Organic Compounds in Ambient Air Using SUMMA™ Passivated Canister Sampling and Gas Chromatographic-Mass Spectrometric Analysis*, PNL-TVP-01 (Rev. 0), PNL Technical Procedure, Richland, Washington.
 - (c) Pacific Northwest Laboratory. 8/94. *Determination of TO-14 Volatile Organic Compounds in Hanford Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Gas Chromatographic-Mass Spectrometric Analysis*, PNL-TVP-03 (Rev. 0), PNL Technical Procedure, Richland, Washington.

The instrument calibration mixture for the TO-14 analysis consists of the standard 39 organic analytes with an additional 13 tank-related compounds. Hexane normally included in the additional analytes, was removed because a calibration standard was not available during analysis of TX-118 SUMMA™ canisters. Together, these 52 compounds that are directly quantified in this analysis make up the target analyte list (these 52 compounds will be referred to as target analytes). The calibration mixture was prepared by blending a commercially prepared 39-compound TO-14 calibration mixture with a 13-compound mixture created using a Kin-Tek® permeation-tube standard generation system. The operation of the permeation tube system follows the method detailed in PNL Technical Procedure PNL-TVP-06^(a). The standard calibration mix was analyzed using four aliquot sizes ranging from 30 mL to 200 mL, and a response factor for each compound was calculated. The GC/MS response for these compounds has been previously determined to be linearly related to concentration. Currently, 1-butanol is not being measured in the samples as a calibrated analyte. It is being quantified as a tentatively identified compound (TIC). Once the appropriate permeation tube has been obtained, 1-butanol will be measured as a calibrated compound. Performance-based detection limits for the target analytes will be developed as a pool of calibration data becomes available. Currently, the nominal detection limit of 5 ppbv is met.

3.3 Quality Assurance/Quality Control

Before the tank sample was analyzed, a diagnostic check was performed on the GC/MS instrument by running an instrument "high-sensitivity 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 using a standard gas mixture containing 39 volatile organic compounds listed in EPA compendium Method TO-14 and an additional 13 tank-related compounds. A gas mixture containing bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d₅ was used as an internal standard (IS) for all blank, calibration standard, and sample analyses. Analyte responses from sample components, ISs, and standards were obtained from the extracted ion plot from their selected mass ion. The calibration 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. Once it is determined that the relative response is linear with increasing concentration, an average response factor is calculated for each target analyte and used to determine the concentration of target compounds in each sample. Method blanks are analyzed prior to and after analysis of calibration standards and tank-headspace samples.

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- (a) Pacific Northwest Laboratory. 8/94. *Preparation of TO-14 Volatile Organic Compounds Gas Standards*, PNL-TVP-06 (Rev. 0). PNL Technical Procedure, Richland, Washington.
- (b) Pacific Northwest Laboratory. 8/94. *Analysis Method for the Determination of Permanent Gases in Hanford Waste Tank Vapor Samples Collected in SUMMA™ Passivated Stainless Steel Canisters*, PNL-TVP-05 (Rev. 0). PNL Technical Procedure, Richland, Washington.

3.3.1 Quantitation Results of Target Analytes. The quantitative-analysis results for the target analytes 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:

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

3.3.2 Identification and Quantitation of Tentatively Identified Compounds. The 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 5971/5972 instrument operating system. Chromatographic peaks with an area count greater than, or equal to, one tenth of the total area count of the nearest eluting IS are tentatively identified and quantitatively estimated. 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 the total peak area for the nearest eluting IS. The IS peak area was used to calculate a response factor using the IS concentration in mg/m^3 :

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

The calculated response factor was then multiplied by the TIC peak area to give an estimated concentration for that compound.

The ppbv concentrations are calculated from mg/m^3 and the molecular weight of the analyte.

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

The IS level added to all blank, standard, and sample injections was 104 ppbv for bromochloromethane, 101 ppbv for 1,4-difluorobenzene, and 98.5 ppbv for chlorobenzene- d_5 . The IS concentrations were converted from ppbv to mg/m^3 at STP using a molecular weight of 129.39 (g/mol) for bromochloromethane, 114.09 for 1,4-difluorobenzene, and 117.6 for chlorobenzene- d_5 . All sample concentrations were multiplied by a factor of two to account for the dilution step described in Section 3.2.

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. The results of replicate analyses on a single SUMMA™ canister are presented in Tables 3.3 and 3.4. A representative total ion chromatogram showing the identity of major constituents is given in Figure 3.1.

Table 3.1 lists the quantitative results for target analyte compounds. Twelve target analytes above the 5-ppbv reporting cutoff were detected in the tank-headspace samples. Acetone

(3.39 mg/m³), trichlorofluoromethane (2.44 mg/m³), and carbon tetrachloride (0.25 mg/m³) accounted for 88% of the target analyte concentration and 52% of the total concentration identified by both the target and TIC analyses. Acetone accounted for 49% of the target analyte concentration and 29% of the total concentration identified by both analyses. The total concentration of the target analytes was measured to be 6.93 mg/m³ or 59% of the total concentration identified by both the target and TIC analyses.

Table 3.2 lists the semi-quantitative results for the TICs. A total of 14 TICs were identified. Twelve TICs were identified in two or more of the tank-headspace samples. The predominant species observed in these samples were 1-butanol (1.09 mg/m³), methyl alcohol (1.06 mg/m³), and ethanol (0.41 mg/m³). The highest concentration TIC, 1-butanol, accounted for 23% of the TIC concentration and 9% of the total concentration identified by both analyses. The total concentration of the TICs was measured to be 4.75 mg/m³ or 41% of the total concentration identified by both the target and TIC analyses.

SUMMA™ canister PNL 184 was analyzed in replicate for target analytes and TICs to determine analytical precision. The relative percent difference (RPD) results are presented in Tables 3.3 and 3.4. The RPD was calculated for analytes detected above the detection limit and found in both replicates. Six of the 12 target analytes and 7 of 14 TICs had RPDs of less than 10%.

Neither target analytes nor TICs were identified in the ambient-air sample collected upwind of Tank TX-118.

Permanent gas analysis was not conducted in the tank headspace or ambient-air samples.

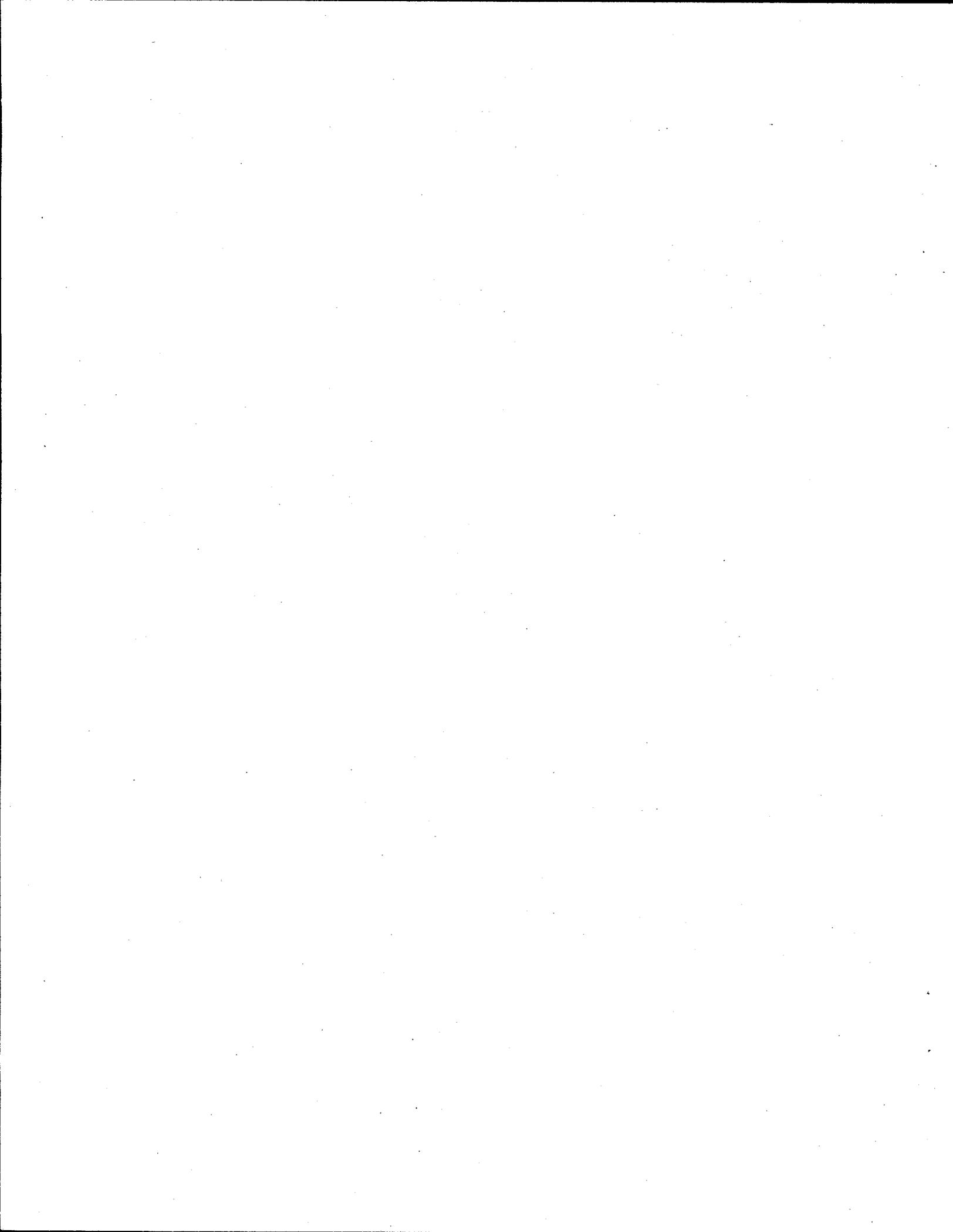
4.0 Conclusions

The concentrations of selected inorganic and organic compounds were determined from *in situ* samples of the headspace of Tank TX-118 on 9/7/94. Sampling and analysis methods followed those described by Ligothke et al. (1994) for VSS samples obtained from Tank C-103, containing a relatively complex headspace composition. Method-validation measurements during that study did appear to validate the trapping and analysis of NH₃, but did not eliminate the possibility of interferences that could affect NO_x results. It is recommended that additional control samples be obtained if a tank is discovered in the future to contain significant quantities of NO_x. In the current sample job, NO_x samples were obtained after first passing the sample flow through an NH₃ trap. The average and the standard deviation of the concentration results from inorganic sorbent trains were 38 ± 1 ppmv (NH₃), ≤ 0.02 ppmv (NO₂), 0.62 ± 0.01 ppmv (NO), ≤ 0.02 ppmv (HCN), and 16 ± 2 mg/L (vapor-mass concentration). The vapor-mass concentration is expected to consist largely of water vapor. Uncertainties were based on one standard deviation of analytical results; information on sample-volume uncertainty was not provided. It is recommended that sample-volume uncertainties be evaluated and reported along with analytical uncertainties in subsequent sample jobs.

Organic analysis of the tank-headspace samples from Tank TX-118 identified 12 target analytes above the 5-ppbv reporting cutoff and 14 TICs above the 10-ppbv reporting cutoff. Twelve TICs were observed in two or more of the SUMMA™ canisters. Acetone, the highest concentration target analyte, accounted for 49% of the total concentration of target analytes and 29% of the total concentration identified by both analyses. The total concentration of the target analytes accounted for 59% of the total concentration identified by both the target and TIC analyses. Tentatively identified compounds accounted for 41% of the total compounds identified by both the target and TIC analyses. The highest concentration TIC, 1-butanol, accounted for 23% of the TIC concentration and 9% of the total concentration identified by both analyses. Results of replicate analysis on a single SUMMA™ canister observed 6 of 12 target analytes and 7 of 14 TICs having RPDs of less than 10%. No target analytes or TICs above their respective MDLs were identified in the ambient air collected upwind of Tank TX-118.

5.0 References

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- Ligothke, 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 - 5/25/94)*. PNL-10172, Pacific Northwest Laboratory, Richland, Washington.
- Mahlum, D. 1994. *Toxicological Evaluation of Analytes from Tank 241-C-103*. PNL-10189, 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 Positively Identified and Quantitated Target Analytes^(a) of Samples Collected from the Headspace of Tank TX-118 in SUMMA™ Canisters on 9/7/94

Analyte	CAS No.	Mol Wt	S4066-SUM.086 ^(b)		S4066-SUM.180 ^(b)		S4066-SUM.184 ^(b)		Means and Standard Deviations	
			PNL 86 ^(c) (mg/m ³)	(ppbv)	PNL 180 ^(c) (mg/m ³)	(ppbv)	PNL 184 ^{(c)(d)} (mg/m ³)	(ppbv)	(mg/m ³)	St Dev (ppbv)
Dichlorodifluoromethane	75-71-8	120.9	<0.03	<5	<0.03	<5	<0.03	<5	(e)	(e)
Chloromethane	74-87-3	50.5	0.03	11	0.02	10	0.02	10	0.01	10
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	170.9	<0.04	<5	<0.04	<5	<0.04	<5	(e)	(e)
Vinyl Chloride	75-01-4	62.5	<0.01	<5	<0.01	<5	<0.01	<5	(e)	(e)
Bromomethane	74-83-9	94.9	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
Chloroethane	75-00-3	64.5	<0.01	<5	<0.01	<5	<0.01	<5	(e)	(e)
Acetonitrile	75-05-8	41.1	0.14	79	0.14	74	0.13	68	0.01	74
Acetone	67-64-1	58.1	3.30	1273	3.60	1388	3.26	1256	0.19	1306
Trichlorofluoromethane	75-69-4	137.4	2.51	409	2.49	407	2.32	378	0.10	398
1,1-Dichloroethene	75-35-4	96.9	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
Methylene Chloride	75-09-2	84.9	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	187.4	<0.04	<5	<0.04	<5	<0.04	<5	(e)	(e)
Propanenitrile	107-12-0	55.1	0.04	15	0.04	18	0.04	18	0.00	17
Propanol	71-23-8	60.1	0.20	74	0.14	54	0.16	60	0.03	62
1,1-Dichloroethane	75-34-3	99.0	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
2-Butanone	78-93-3	72.1	0.18	55	0.27	84	0.26	79	0.05	73
cis-1,2-Dichloroethene	156-59-2	96.9	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
Chloroform	67-66-3	119.4	<0.03	<5	<0.03	<5	<0.03	<5	(e)	(e)
Tetrahydrofuran	109-99-9	72.1	0.05	15	0.06	18	0.06	18	0.01	17
1,2-Dichloroethane	107-06-2	99.0	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
1,1,1-Trichloroethane	71-55-6	133.4	<0.03	<5	<0.03	<5	<0.03	<5	(e)	(e)
Butanenitrile	109-74-0	69.1	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
Benzene	71-43-2	78.1	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
Carbon Tetrachloride	56-23-5	153.8	0.25	36	0.26	38	0.25	36	0.01	37
Cyclohexane	110-82-7	84.2	0.08	21	0.07	20	0.07	19	0.01	20
1,2-Dichloropropane	78-87-5	113.0	<0.03	<5	<0.03	<5	<0.03	<5	(e)	(e)
Trichloroethene	79-01-6	131.4	<0.03	<5	<0.03	<5	<0.03	<5	(e)	(e)
Heptane	142-82-5	100.2	0.08	19	0.08	17	0.07	16	0.01	17
cis-1,3-Dichloropropene	10061-01-5	111.0	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
4-Methyl-2-Pentanone	108-10-1	100.2	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
Pyridine	110-86-1	79.1	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
trans-1,3-Dichloropropene	10061-02-6	111.0	<0.02	<5	<0.02	<5	<0.02	<5	(e)	(e)
1,1,2-Trichloroethane	79-00-5	133.4	<0.03	<5	<0.03	<5	<0.03	<5	(e)	(e)

TX-118 Table 3.1 (Contd)

Analyte	CAS No.	Mol Wt	S4066-SUM.086 ^(b)		S4066-SUM.180 ^(b)		S4066-SUM.184 ^(b)		Means and Standard Deviations			
			PNL 86 ^(c) (mg/m ³) (ppbv)	12	PNL 180 ^(c) (mg/m ³) (ppbv)	0.04	11	PNL 184 ^(c) (mg/m ³) (ppbv)	0.04	10	(mg/m ³)	St Dev (ppbv)
Toluene	108-88-3	92.1	0.05	12	0.04	11	0.04	10	0.04	0.01	11	0.8
1,2-Dibromochthane	106-93-4	187.9	<0.04	<5	<0.04	<5	<0.04	<5	(c)	(c)	(c)	(c)
Tetrachloroethylene	127-18-4	165.8	<0.04	<5	<0.04	<5	<0.04	<5	(c)	(c)	(c)	(c)
Chlorobenzene	108-90-7	112.6	<0.03	<5	<0.03	<5	<0.03	<5	(c)	(c)	(c)	(c)
Ethylbenzene	100-41-4	106.2	<0.02	<5	<0.02	<5	<0.02	<5	(c)	(c)	(c)	(c)
p/m-Xylene ^(d)	106-42-3	106.2	<0.02	<5	<0.02	<5	<0.02	<5	(c)	(c)	(c)	(c)
Cyclohexanone	108-94-1	98.1	<0.02	<5	<0.02	<5	<0.02	<5	(c)	(c)	(c)	(c)
Styrene	100-42-5	104.2	<0.02	<5	<0.02	<5	<0.02	<5	(c)	(c)	(c)	(c)
1,1,2,2-Tetrachloroethane	79-34-5	167.9	<0.04	<5	<0.04	<5	<0.04	<5	(c)	(c)	(c)	(c)
o-Xylene	95-47-6	106.2	<0.02	<5	<0.02	<5	<0.02	<5	(c)	(c)	(c)	(c)
1,3,5-Trimethylbenzene	108-67-8	120.2	<0.03	<5	<0.03	<5	<0.03	<5	(c)	(c)	(c)	(c)
1,2,4-Trimethylbenzene	95-63-6	120.2	<0.03	<5	<0.03	<5	<0.03	<5	(c)	(c)	(c)	(c)
Decane	124-18-5	142.3	<0.03	<5	<0.03	<5	<0.03	<5	(c)	(c)	(c)	(c)
1,3-Dichlorobenzene	541-73-1	147.0	<0.03	<5	<0.03	<5	<0.03	<5	(c)	(c)	(c)	(c)
1,4-Dichlorobenzene	106-46-7	147.0	<0.03	<5	<0.03	<5	<0.03	<5	(c)	(c)	(c)	(c)
1,2-Dichlorobenzene	95-50-1	147.0	<0.03	<5	<0.03	<5	<0.03	<5	(c)	(c)	(c)	(c)
1,2,4-Trichlorobenzene	120-82-1	181.5	<0.04	<5	<0.04	<5	<0.04	<5	(c)	(c)	(c)	(c)
Hexachloro-1,3-butadiene	87-68-3	260.8	<0.06	<5	<0.06	<5	<0.06	<5	(c)	(c)	(c)	(c)

(a) TO-14 plus 14 additional target analytes.

(b) WHC sample identification number.

(c) PNL canister number.

(d) Replicates of this sample are found in Table 3.3

(e) Average and/or standard deviation are not meaningful for this analyte.

(f) 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) of Samples from the Headspace of Tank IX-118 in SUMMA™ Canisters Collected on 9/7/94

Tentatively Identified Compound ^(a)	CAS No. ^(e)	Mol Wt	Ret Time	S4066-SUM.08 ^(b)		S4066-SUM.180 ^(b)		S4066-SUM.184 ^(b)		Means and Standard Deviations			
				PNL 86 ^(b) (mg/m ³)	ppbv	PNL 180 ^(b) (mg/m ³)	ppbv	PNL 184 ^(b) (mg/m ³)	ppbv	(mg/m ³)	St Dev	(ppbv)	St Dev
Propane	74-98-6	44	5.5	0.41	210	0.38	191	0.36	185	0.38	0.03	195	13.0
Methyl Alcohol	67-56-1	32	6.5	1.06	739	1.10	771	1.01	708	1.06	0.05	739	31.5
Butane	106-97-8	58	7.1	0.09	34	0.08	32	0.08	29	0.08	0.01	32	2.6
Ethanol	64-17-5	46	8.2	0.45	217	0.41	200	0.38	185	0.41	0.03	200	16.3
Unknown			11.1	0.38	(g)	0.38	(g)	0.32	(g)	0.36	0.04	(f)	(f)
Unknown			13.5	(g)	(g)	(g)	(g)	0.06	(g)	(f)	(f)	(f)	(f)
Butanal	123-72-8	72	13.8	0.23	70	0.29	91	0.34	106	0.29	0.06	88.9	18.0
Unknown			16.1	(g)	(g)	0.06	(g)	0.02	(g)	0.04	(f)	(f)	(f)
1-Butanol	71-36-3	74	17.9	0.98	295	0.82	249	1.48	449	1.09	0.35	331	104.8
Hexane, 2-methyl-	591-76-4	100	19.0	0.18	41	0.17	37	0.16	35	0.17	0.01	37.5	2.9
Pentane, 2,3-dimethyl-	565-59-3	100	19.2	0.15	34	0.13	30	0.12	26	0.13	0.02	30.1	4.3
Hexane, 3-methyl-	589-34-4	100	19.5	0.32	71	0.27	60	0.25	56	0.28	0.03	62.3	7.6
Unknown Alkane			20.1	(g)	(g)	0.05	(g)	(g)	(g)	(f)	(f)	(f)	(f)
Unknown Alkane			22.4	0.21	(g)	(g)	(g)	0.17	(g)	(f)	(f)	(f)	(f)
Nitric acid, butyl ester	928-45-0	119	26.3	0.07	12	0.07	13	0.07	12	0.07	0.00	12.4	0.2
3-Heptanone	106-35-4	114	29.8	0.33	64	0.34	66	0.33	65	0.33	0.01	64.8	1.0
Unknown			37.7	0.06	(g)	0.06	(g)	0.06	(g)	0.06	0.00	(f)	(f)

(a) Semi-quantitative estimate calculated using concentration of closest eluting IS.

(b) WHC sample identification number.

(c) Replicates of this sample are found in Table 3.4.

(d) PNL SUMMA™ canister number.

(e) Obtained by mass spectral interpretation and comparison with the EPANIST/WILEY Library.

(f) Mean and/or standard deviation are not meaningful for this analyte.

(g) Molecular weight not available for this analyte.

Table 3.3 Positively Identified and Quantitated Target Analytes^(a) of Replicate Analyses of a Single SUMMA™ Canister Collected from the Headspace of Tank TX-118 on 9/7/94

Analyte	CAS No.	Mol Wt	S4066-SUM.184 ^(b)		S4066-SUM.184 ^(c)		Relative Percent Difference
			PNL 184 ^(c) (mg/m ³)	(ppbv)	PNL 184 ^(c) (mg/m ³)	(ppbv)	
Dichlorodifluoromethane	75-71-8	120.9	<0.03	<5	<0.03	<5	
Chloromethane	74-87-3	50.5	0.02	10	0.02	11	0.0
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	170.9	<0.04	<5	<0.04	<5	
Vinyl Chloride	75-01-4	62.5	<0.01	<5	<0.01	<5	
Bromomethane	74-83-9	94.9	<0.02	<5	<0.02	<5	
Chloroethane	75-00-3	64.5	<0.01	<5	<0.01	<5	
Acetonitrile	75-05-8	41.1	0.13	68	0.14	75	7.4
Acetone	67-64-1	58.1	3.26	1256	3.64	1404	11.0
Trichlorofluoromethane	75-69-4	137.4	2.32	378	2.73	446	16.2
1,1-Dichloroethene	75-35-4	96.9	<0.02	<5	<0.02	<5	
Methylene Chloride	75-09-2	84.9	<0.02	<5	<0.02	<5	
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	187.4	<0.04	<5	<0.04	<5	
Propanenitrile	107-12-0	55.1	0.04	18	0.06	25	40.0
Propanol	71-23-8	60.1	0.16	60	0.29	108	57.8
1,1-Dichloroethane	75-34-3	99.0	<0.02	<5	<0.02	<5	
2-Butanone	78-93-3	72.1	0.26	79	0.28	88	7.4
cis-1,2-Dichloroethene	156-59-2	96.9	<0.02	<5	<0.02	<5	
Chloroform	67-66-3	119.4	<0.03	<5	<0.03	<5	
Tetrahydrofuran	109-99-9	72.1	0.06	18	0.06	19	0.0
1,2-Dichloroethane	107-06-2	99.0	<0.02	<5	<0.02	<5	
1,1,1-Trichloroethane	71-55-6	133.4	<0.03	<5	<0.03	<5	
Butanenitrile	109-74-0	69.1	<0.02	<5	<0.02	<5	
Benzene	71-43-2	78.1	<0.02	<5	<0.02	<5	
Carbon Tetrachloride	56-23-5	153.8	0.25	36	0.27	39	7.7
Cyclohexane	110-82-7	84.2	0.07	19	0.08	20	13.3
1,2-Dichloropropane	78-87-5	113.0	<0.03	<5	<0.03	<5	
Trichloroethene	79-01-6	131.4	<0.03	<5	<0.03	<5	
Heptane	142-82-5	100.2	0.07	16	0.08	17	13.3
cis-1,3-Dichloropropene	10061-01-5	111.0	<0.02	<5	<0.02	<5	
4-Methyl-2-Pentanone	108-10-1	100.2	<0.02	<5	<0.02	<5	
Pyridine	110-86-1	79.1	<0.02	<5	<0.02	<5	
trans-1,3-Dichloropropene	10061-02-6	111.0	<0.02	<5	<0.02	<5	

TX-118 Table 3.3 (Contd)

Analyte	CAS No.	Mol Wt	S4066-SUM.184 ^(b)		S4066-SUM.184 ^(b)		Relative Percent Difference %
			PNL 184 ^(c) (mg/m ³)	(ppbv)	PNL 184 ^(c) (mg/m ³)	(ppbv)	
1,1,2-Trichloroethane	79-00-5	133.4	<0.03	<5	<0.03	<5	
Toluene	108-88-3	92.1	0.04	10	0.04	11	0.0
1,2-Dibromoethane	106-93-4	187.9	<0.04	<5	<0.04	<5	
Tetrachloroethylene	127-18-4	165.8	<0.04	<5	<0.04	<5	
Chlorobenzene	108-90-7	112.6	<0.03	<5	<0.03	<5	
Ethylbenzene	100-41-4	106.2	<0.02	<5	<0.02	<5	
p/m-Xylene ^(d)	106-42-3	106.2	<0.02	<5	<0.02	<5	
Cyclohexanone	108-94-1	98.1	<0.02	<5	<0.02	<5	
Styrene	100-42-5	104.2	<0.02	<5	<0.02	<5	
1,1,2,2-Tetrachloroethane	79-34-5	167.9	<0.04	<5	<0.04	<5	
o-Xylene	95-47-6	106.2	<0.02	<5	<0.02	<5	
1,3,5-Trimethylbenzene	108-67-8	120.2	<0.03	<5	<0.03	<5	
1,2,4-Trimethylbenzene	95-63-6	120.2	<0.03	<5	<0.03	<5	
Decane	124-18-5	142.3	<0.03	<5	<0.03	<5	
1,3-Dichlorobenzene	541-73-1	147.0	<0.03	<5	<0.03	<5	
1,4-Dichlorobenzene	106-46-7	147.0	<0.03	<5	<0.03	<5	
1,2-Dichlorobenzene	95-50-1	147.0	<0.03	<5	<0.03	<5	
1,2,4-Trichlorobenzene	120-82-1	181.5	<0.04	<5	<0.04	<5	
Hexachloro-1,3-butadiene	87-68-3	260.8	<0.06	<5	<0.06	<5	

(a) TO-14 plus 14 additional target analytes.

(b) WHC sample identification number.

(c) PNL canister number.

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

Table 3.4 Tentatively Identified Compounds and Estimated Concentrations^(a) of Replicate Analyses of a Single SUMMA™ Canister Collected from the Headspace of Tank TX-118 on 9/7/94

Tentatively Identified Compound ^(b)	CAS No. ^(c)	Mol Wt	Ret Time	S4066-SUM.184 ^(b)		S4066-SUM.184 ^(b)		Relative Percent Difference %
				(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	
Propane	74-98-6	44	5.5	0.36	185	0.42	214	14.8
Methyl Alcohol	67-56-1	32	6.5	1.01	708	1.11	778	9.4
Butane	106-97-8	58	7.1	0.08	29	0.10	39	30.5
Ethanol	64-17-5	46	8.2	0.38	185	0.48	235	23.9
Unknown			11.1	0.32	(e)	0.40	(e)	22.5
Unknown			13.5	0.06	(e)	0.06	(e)	8.5
Butanal	123-72-8	72	13.8	0.34	106	0.38	117	10.1
Unknown			16.1	0.02	(e)	(e)	(e)	
1-Butanol	71-36-3	74	17.9	1.48	449	1.74	528	16.2
Hexane, 2-methyl-	591-76-4	100	19.0	0.16	35	0.16	36	3.8
Pentane, 2,3-dimethyl-	565-59-3	100	19.2	0.12	26	0.13	28	8.3
Hexane, 3-methyl-	589-34-4	100	19.5	0.25	56	0.27	60	6.9
Unknown Alkane			20.1	(e)	(e)	0.05	(e)	
Unknown Alkane			22.4	0.17	(e)	(e)	(e)	
Nitric acid, butyl ester	928-45-0	119	26.3	0.07	12	0.08	14	12.8
3-Heptanone	106-35-4	114	29.8	0.33	65	0.35	68	5.0
Unknown			37.7	0.06	(e)	0.07	(e)	9.1

(a) Semi-quantitative estimate calculated using concentration of closest eluting IS.

(b) WHC sample identification number.

(c) PNL SUMMA™ canister number.

(d) Obtained by mass spectral interpretation and comparison with the EPA/NIST/WILEY Library.

(e) No molecular weight available for calculation.

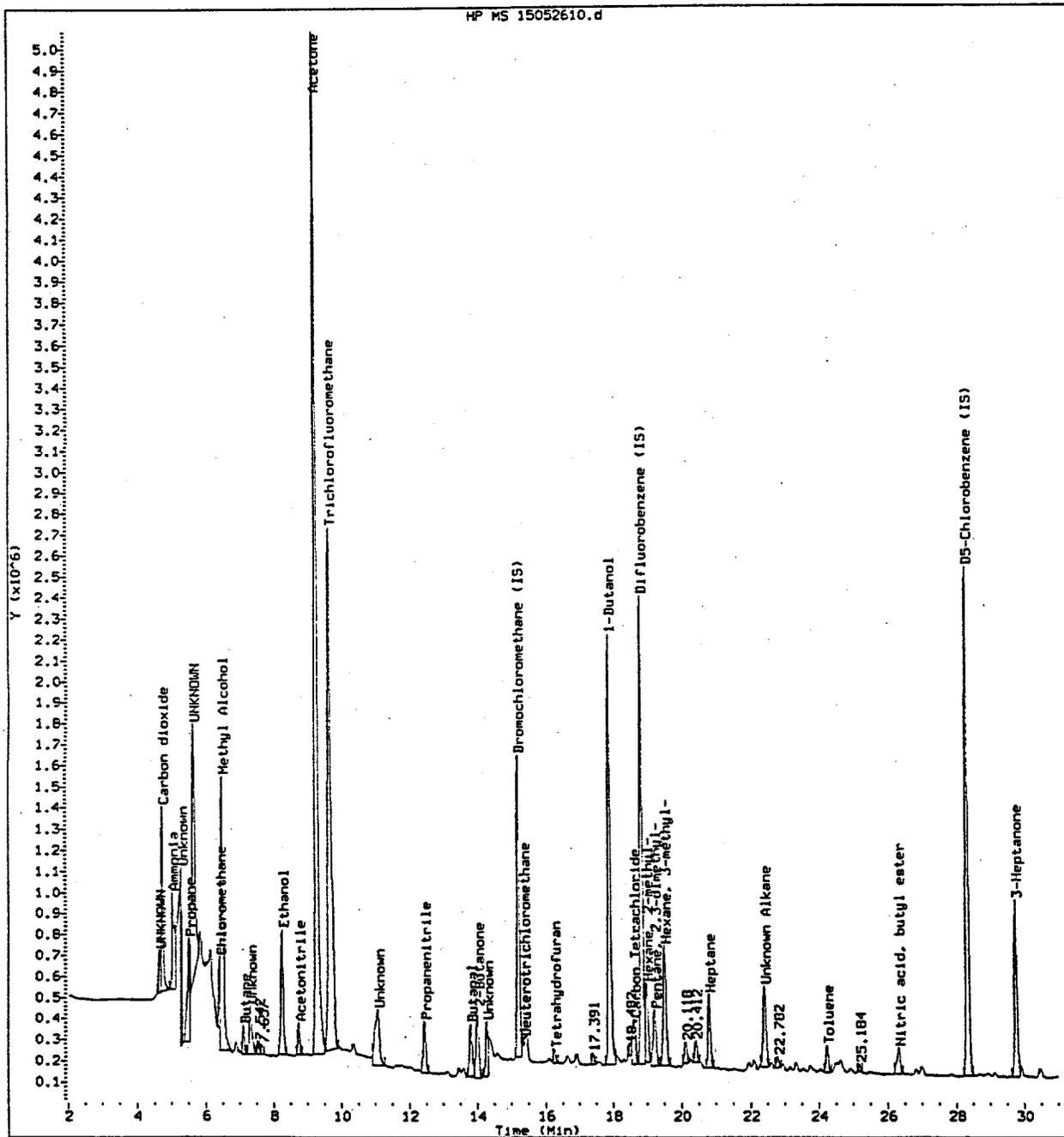


Figure 3.1a Total Ion Chromatogram (2 - 30 min) for Hanford Waste Tank TX-118
SUMMA™ Canister Sample S4066-SUM-086 Collected on 9/7/94

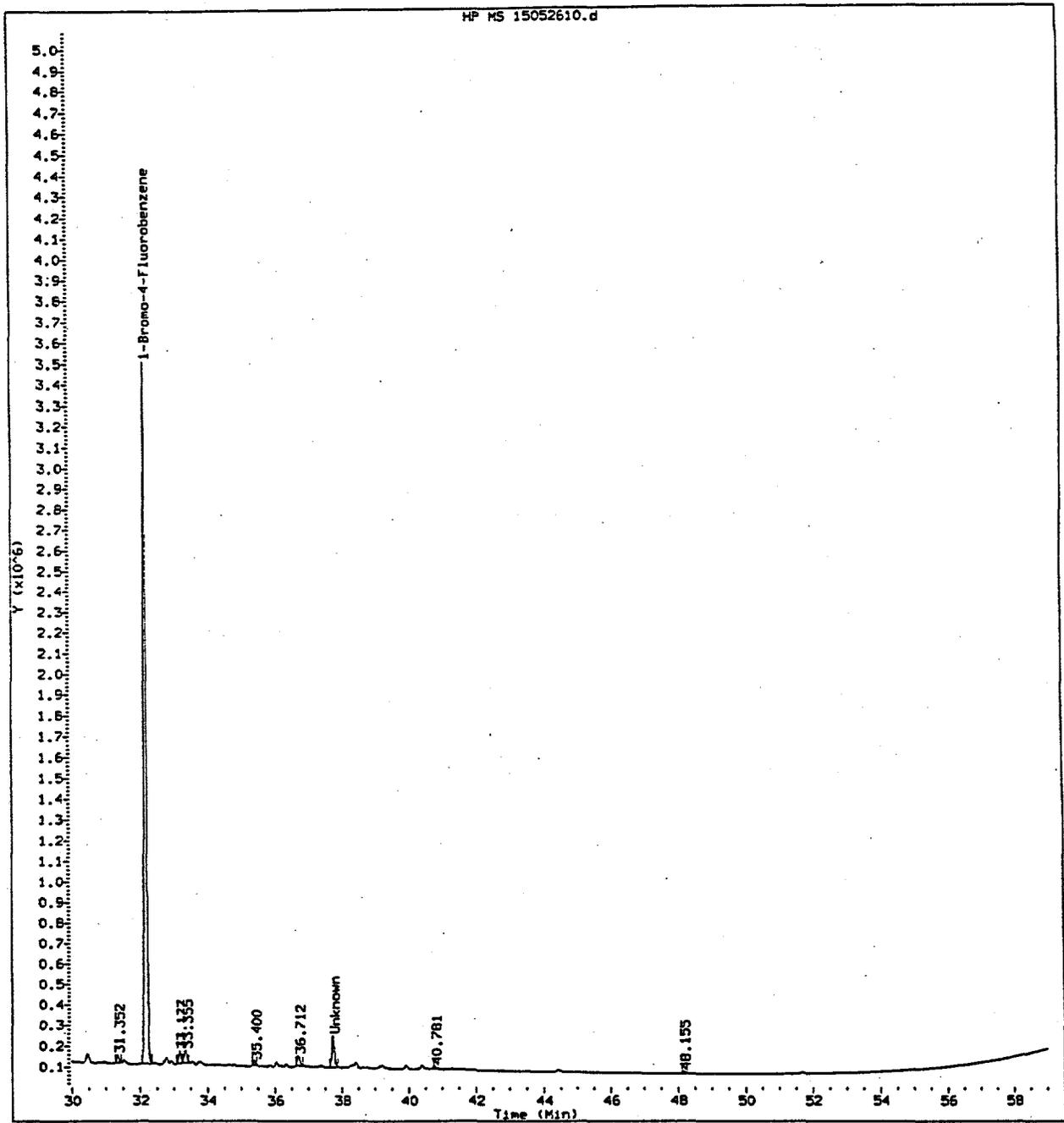
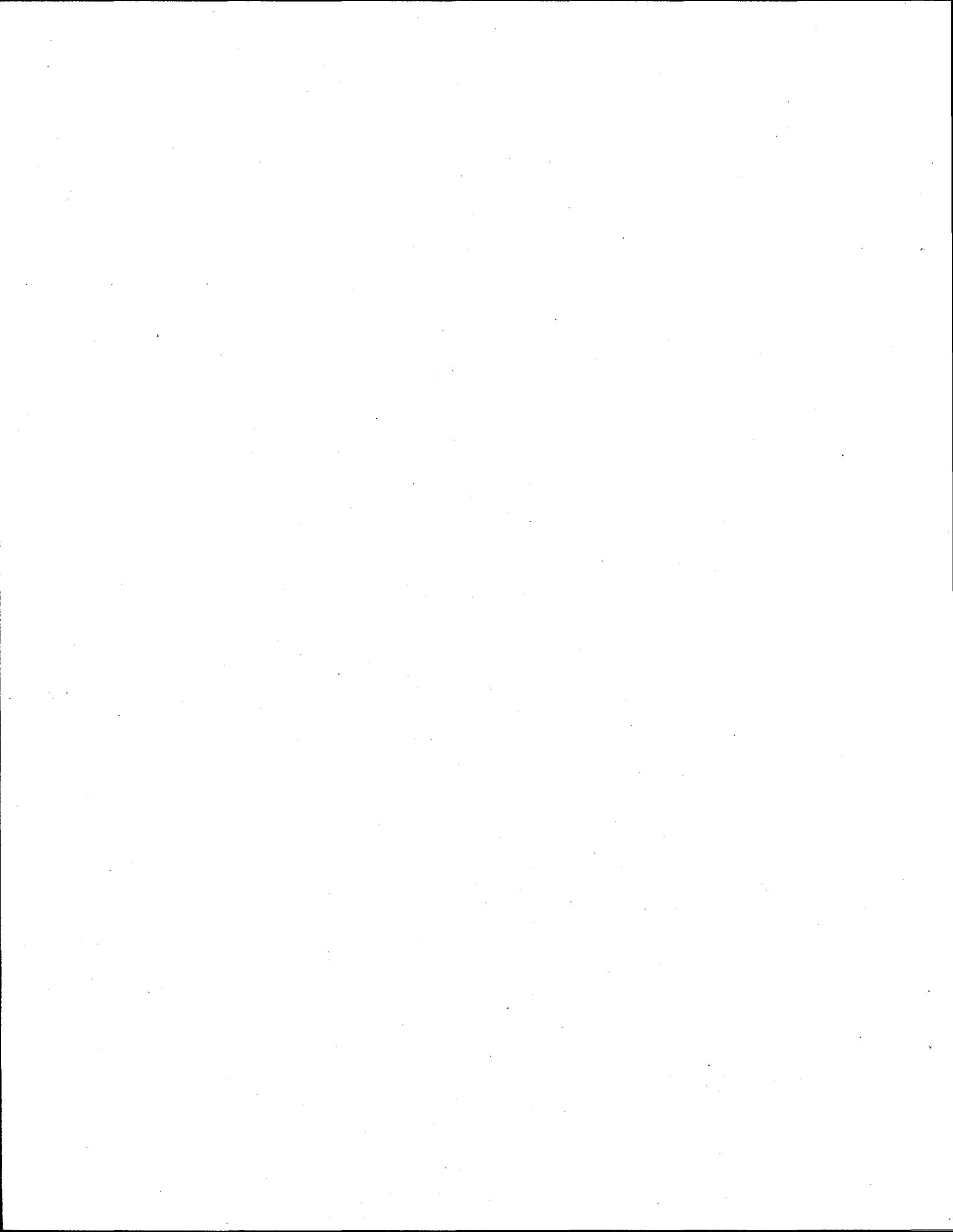


Figure 3.1b Total Ion Chromatogram (30 - 58 min) for Hanford Waste Tank TX-118 SUMMA™ Canister Sample S4066-SUM-086 Collected on 9/7/94



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