

Pacific Northwest National Laboratory

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TANK VAPOR CHARACTERIZATION PROJECT

Tank 241-C-107 Temporal Study Headspace Gas and Vapor Characterization Results from Samples Collected on September 5, 1996

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August 1997

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Summary

This report presents the results from analyses of samples taken from the headspace of waste storage tank 241-C-107 (Tank C-107) at the Hanford Site in Washington State. Tank headspace samples collected by Westinghouse Hanford Company (WHC) were analyzed by Pacific Northwest National Laboratory (PNNL) to determine headspace concentrations of selected non-radioactive analytes. Analyses were performed by the Vapor Analytical Laboratory (VAL) at PNNL. Vapor concentrations from sorbent trap samples are based on measured sample volumes provided by WHC.

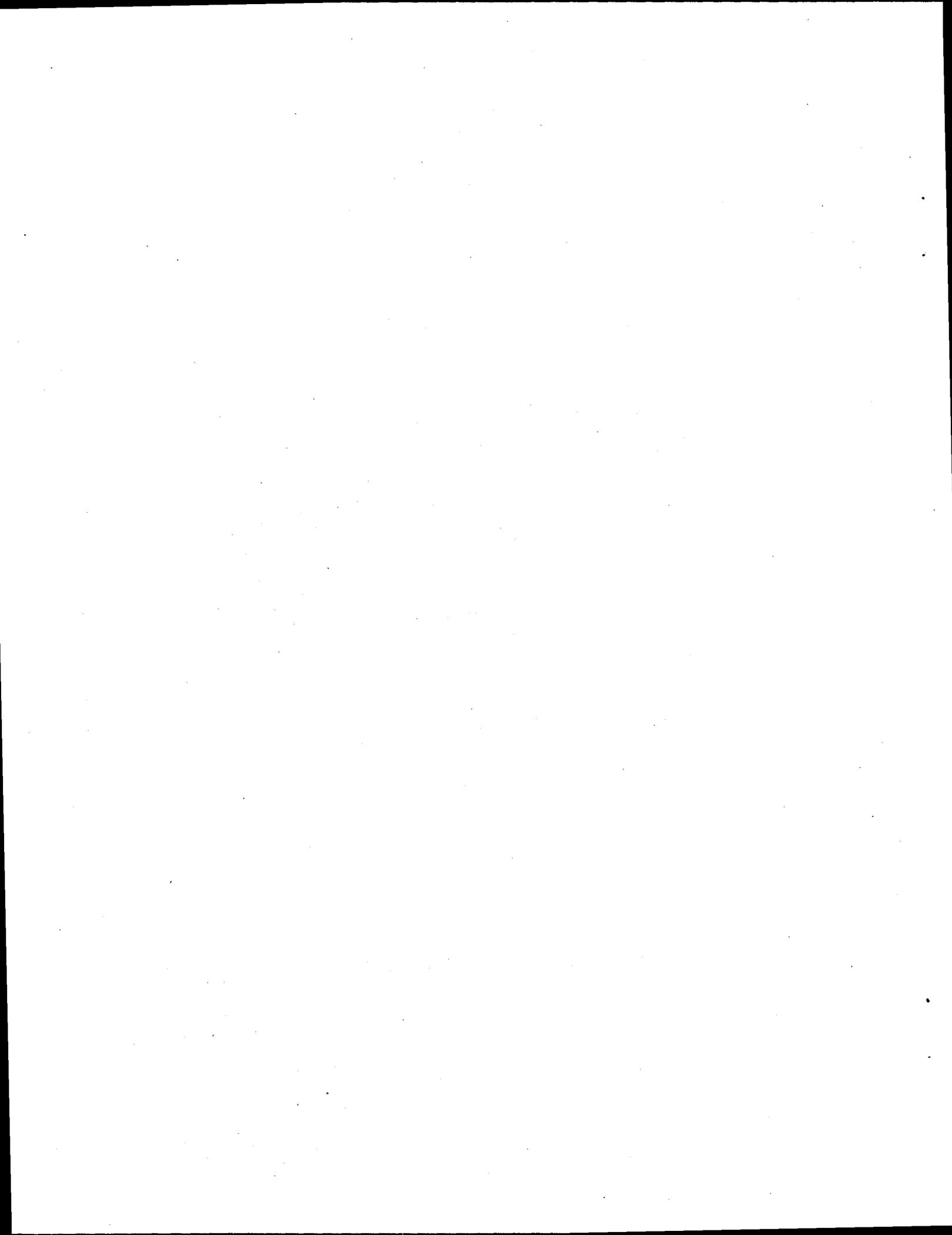
No analytes were determined to be above the immediate notification limits specified by the sampling and analysis plan (SAP) (Buckley 1996). Hydrogen was the principal flammable constituent of the Tank C-107 headspace, determined to be present at approximately 1.405% of its lower flammability limit (LFL). Total headspace flammability was estimated to be <1.519% of the LFL.

Average measured concentrations of targeted gases, inorganic vapors, and selected organic vapors are provided in Table S.1. A summary of experimental methods, including sampling methodology, analytical procedures, and quality assurance and control methods are presented in Section 2.0. Detailed descriptions of the analytical results are provided in Section 3.0.

Table S.1. Average Measured Concentrations of Gases and Inorganic and Organic Vapors in Tank C-107 Sampled on 9/5/96

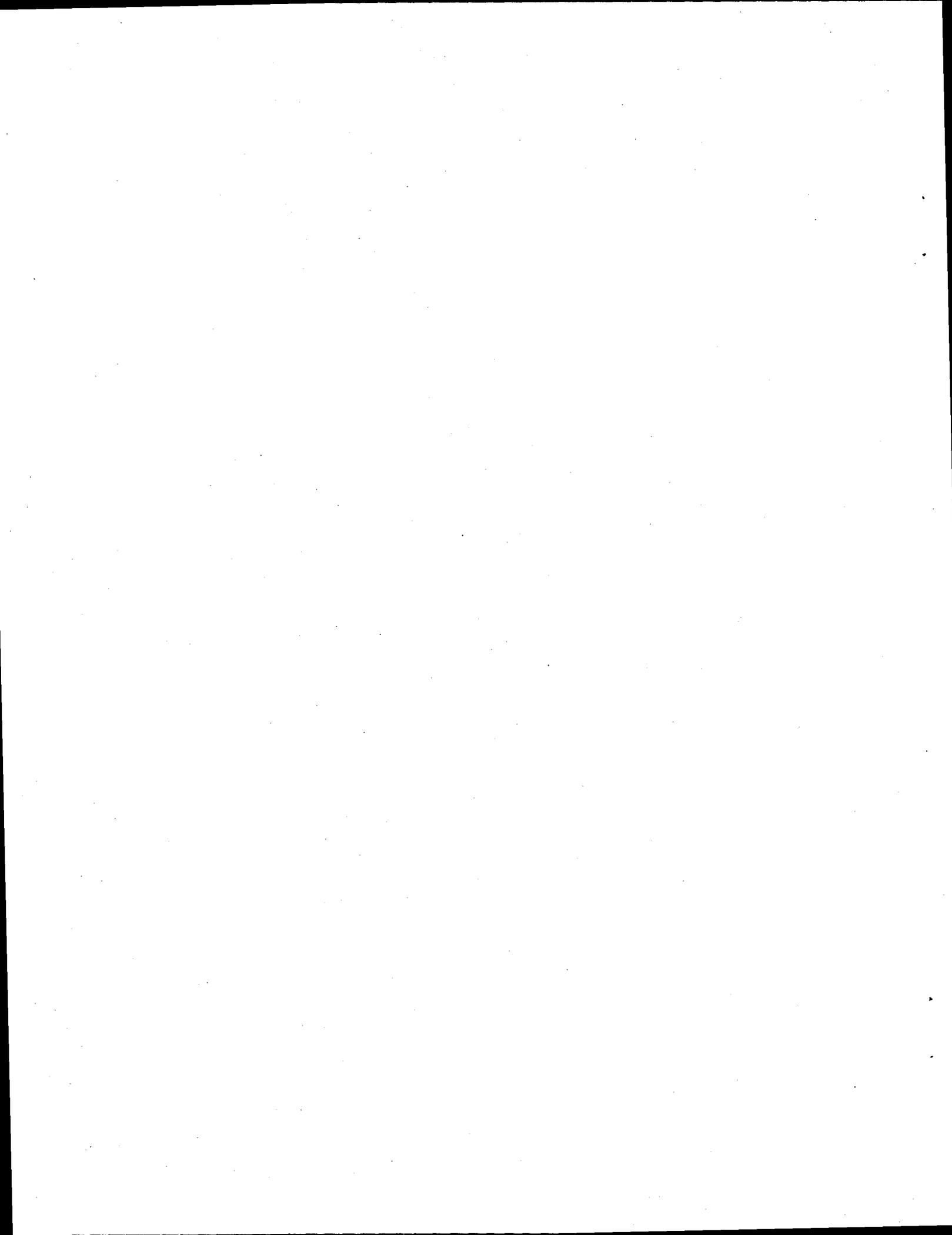
<u>Category</u>	<u>Sample Medium</u>	<u>Analyte</u>	<u>Concentration^(a)</u>	<u>Units</u>
Inorganic Vapors	Sorbent Traps	Ammonia	63	ppmv
		Nitrogen Dioxide (NO ₂)	<0.16	ppmv
		Nitric Oxide (NO)	0.37	ppmv
		Water	74.0	mg/L
Permanent Gases	SUMMA™ Canisters	Hydrogen	562	ppmv
		Methane	<25	ppmv
		Carbon Dioxide	812	ppmv
		Carbon Monoxide	<17	ppmv
		Nitrous Oxide (N ₂ O)	120	ppmv
Total Non-Methane Organic Compounds	SUMMA™ Canisters	Non-Methane Organic Compounds	3.44	mg/m ³
Volatile Organic Vapors	SUMMA™ Canisters	Acetone	1.387	mg/m ³
		Acetonitrile	1.188	mg/m ³
		Methanol	0.956	mg/m ³
Volatile Organic Vapors	Sorbent Traps	Methanol	1.624	mg/m ³
		Acetone	1.027	mg/m ³
		Acetonitrile	1.004	mg/m ³
Flammables	SUMMA™ Canisters and Sorbent Traps	Flammables	<1.519	% LFL

(a) Concentrations were determined using sample-volume data provided by Westinghouse Hanford Company and are based on averaged data from three samples. Mass concentrations are at reference temperature and pressure of 0°C and 1.013 bar.



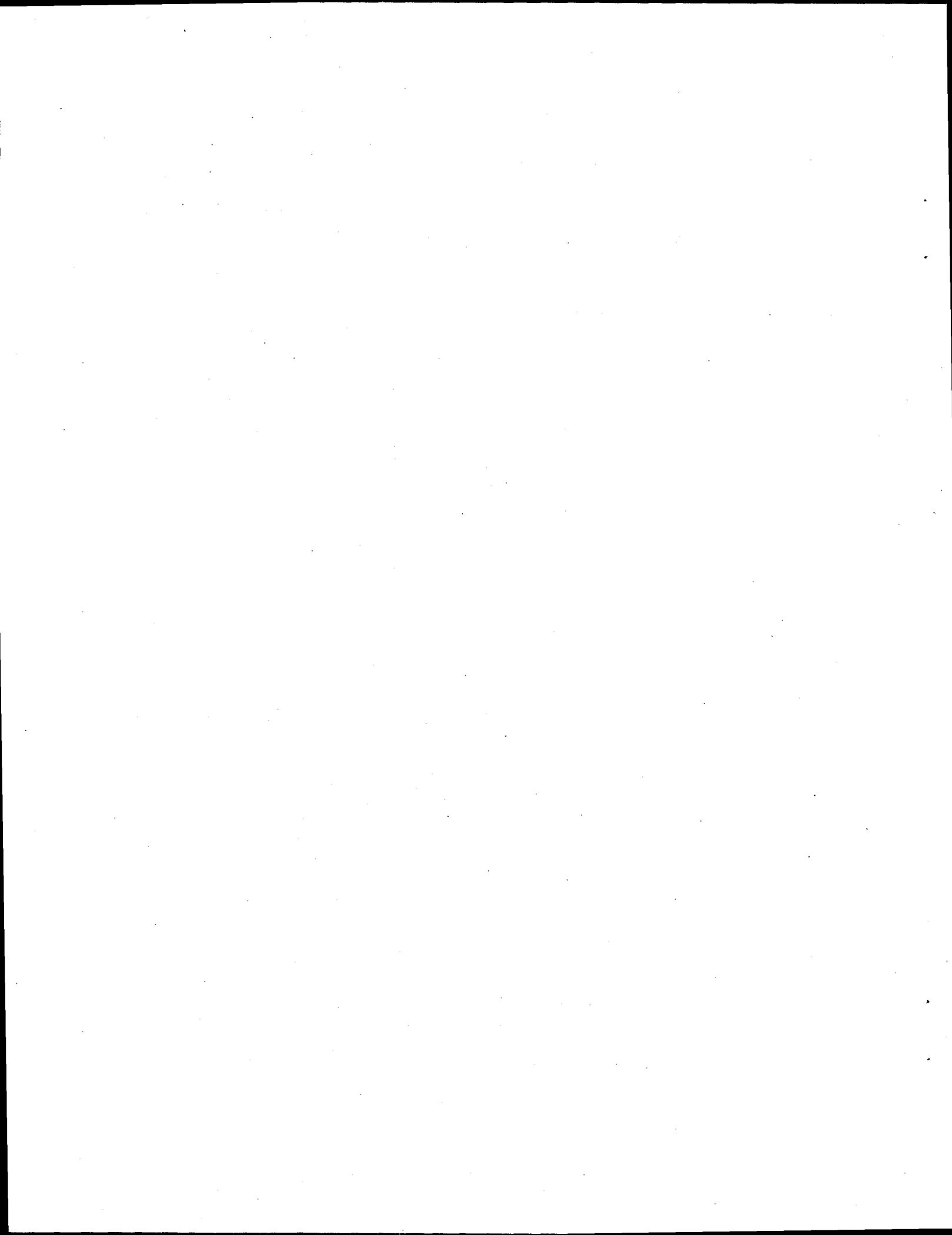
Acknowledgments

The authors gratefully acknowledge the support of other project staff at PNNL who contributed to the successful completion of this sampling and analysis activity. S. O. Slate, K. P. Schielke, L. M. P. Thomas, and G. W. Dennis supported inorganic laboratory work.



Terms and Abbreviations

% D	% Difference
CAS	Chemical Abstracts Service
CCB	continuing calibration blank
CCV	continuing calibration verification
COC	chain-of-custody
DIW	deionized water
EPA	U.S. Environmental Protection Agency
EQL	estimated quantitation limit
GC/FID	gas chromatography/flame ionization detector
GC/MS	gas chromatograph/mass spectrometer
GC/TCD	gas chromatography/thermal conductivity detection
IC	ion chromatography
ICB	initial calibration blank
ICV	initial calibration verification
IDL	instrument detection limit
IS	internal standard
ISE	ion selective electrode
ISVS	In Situ Vapor Sampling System
LFL	lower flammability limit
MW	molecular weight
NIST	National Institute for Standards and Technology
OSHA	Occupational Safety and Health Administration
PNL	Pacific Northwest Laboratory (previous name for the laboratory)
PNNL	Pacific Northwest National Laboratory
ppbv	part per billion by volume
ppm	parts per million
ppmv	part per million by volume
QA	quality assurance
RPD	relative percent difference
RSD	relative standard deviation
SAP	sample and analysis plan
SCIC	suppressed-conductivity ion chromatography
SRM	standard reference material
STP	standard temperature and pressure
SUMMA™	process for passivating stainless steel
TBP	tributyl phosphate
TEA	triethanolamine
TIC	tentatively identified compound
TNMOC	total non-methane organic compound
TST	triple sorbent trap
UHP	ultra high purity
UQL	upper quantitation limit
VAL	Vapor Analytical Laboratory
VSS	vapor sampling system
WHC	Westinghouse Hanford Company



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1.0 Introduction

This report presents results of chemical analyses of vapor samples collected by WHC on September 5, 1996 from the headspace of waste storage tank 241-C-107 (Tank C-107) at the Hanford Site in Washington State. Pacific Northwest National Laboratory^(a) provided SUMMA™ canisters and sorbent traps for sample collection, and analyzed the samples according to instructions in the SAP (Buckley 1996). Analytical work was performed by the PNNL VAL in the 300 Area of the Hanford Site under the PNNL Tank Vapor Characterization Project.

Pacific Northwest National Laboratory provided six sets of sorbent traps for selected inorganic analytes (four samples and two field blanks), five SUMMA™ canisters for permanent gases and organic analytes (three headspace samples and two ambient air samples), and eight triple sorbent traps (TSTs) for organic analytes (four samples, two field blanks, and two trip blanks). Sample devices and controls were provided to WHC on September 3, 1996 and were returned to PNNL on September 16, 1996. Westinghouse Hanford Company measured and reported to PNNL the sample volumes needed to determine headspace concentrations from sorbent trap samples.

Specific analytical methods for sample analysis are described in Section 2.0. Results and known sampling and analytical variances from established quality assurance (QA) requirements, where significant, are documented in Section 3.0. Chain-of-custody forms used to document possession and transfer of samples and controls are provided in Appendix A. Appendix B contains a complete listing of target analyte results for the organic analyses.

⁰ Pacific Northwest National Laboratory is operated for the U. S. Department of Energy by Battelle under Contract DE-AC06-76RLO 1830. The previous name of the laboratory was Pacific Northwest Laboratory. The former name is used when previously published documents are referenced.

2.0 Analytical Methods

Table 2.1 summarizes the analytes, sampling media, analytical methods, and laboratory procedures. Table 2.2 summarizes information regarding sample media, handling, and storage procedures.

Table 2.1. Sampling and Analysis Methods Summary

<u>Analyte</u>	<u>Sampling Media</u>	<u>Extraction Method</u>	<u>Analysis Method</u>	<u>Procedure</u>
Ammonia	Acidified carbon bead sorbent trap	Aqueous extraction	ISE analysis	PNL-ALO-226
Nitric Oxide and Nitrogen Dioxide	Triethanolamine impregnated sorbent traps	Aqueous extraction	IC	PNL-ALO-212
Water Vapor	Other inorganic sorbent traps + silica gel sorbent trap	None	Gravimetric analysis (sample weight gain)	PNL-TVP-09
Carbon Monoxide, Carbon Dioxide, Hydrogen, Methane, and Nitrous Oxide	SUMMA™ canisters	Analysis of SUMMA™ canister subsample	GC/TCD	PNL-TVP-05
Organic Vapors	Triple sorbent traps	Thermal desorption	GC/MS	PNL-TVP-10
Organic Vapors	SUMMA™ canisters	Cryo-focusing of SUMMA™ canister subsample	GC/MS	PNL-TVP-03
Total Non-Methane Organic Compounds	SUMMA™ canisters	Cryo-focusing of SUMMA™ canister subsample	GC/FID	PNL-TVP-08

Table 2.2. Sample Media Preparation, Handling, and Storage

<u>Sampling Media</u>	<u>Supplier and Catalog Number</u>	<u>Preparation Procedure</u>	<u>Handling and Storage Procedure</u>
Inorganic vapor sorbent traps	SKC No. 226-29 SKC No. 226-40-02 SKC No. 226-10-04	PNL-TVP-09	PNL-TVP-07
SUMMA™ canisters	Scientific Instrumentation Specialist	PNL-TVP-02	PNL-TVP-07
Triple sorbent traps	Supelco CarboTrap 300	PNL-TVP-10	PNL-TVP-07

2.1 Inorganic Vapors

Solid sorbent traps, prepared in multi-trap sampling trains, were supplied to WHC for sampling the tank headspace. Blanks and exposed samples were returned to PNNL for analyses. Analyses were performed to provide information on the tank-headspace concentration of the following analytes: ammonia, nitrogen dioxide (NO₂), nitric oxide (NO), and water. Samples were prepared, handled, and disassembled as described in Technical Procedure PNL-TVP-09^(a).

2.1.1 Sampling Methodology

Standard glass tubes containing sorbent materials to trap ammonia, NO, NO₂, and water vapors were obtained, prepared, and submitted for vapor sampling. 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. Each sorbent trap contained two sorbent sections separated by a glass wool plug. Sorbent media in the two sections were segregated and analyzed separately (except for analysis for water). Analyses of the second sorbent (breakthrough) sections were performed to demonstrate complete collection of the target analyte by the first sorbent section.

The ammonia 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 ammonia 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 were used to convert NO to NO₂. The converted NO was then collected as nitrite and nitrate in an NO₂ trap. The water traps contained a total of 450 mg of silica gel. All sorbent traps for a given analyte were from a single manufacturer's batch.

After sample preparation, sorbent trains were stored at ≤ 10°C because of handling recommendations for the oxidizer tubes attached to some samples. After receipt of exposed and

^(a) Pacific Northwest Laboratory. 12/95. *Sorbent Trap Preparation for Sampling and Analysis: Waste Tank Inorganic Vapor Samples*, PNL-TVP-09 (Rev. 2), PNL Technical Procedure, Richland, Washington.

radiologically cleared samples from WHC and disassembly of the sorbent trains, samples were provided to the analytical laboratory at ambient temperature.

The sorbent traps were prepared in multi-trap trains configured so sample air flow passed in order through the ammonia, nitrogen dioxide, oxidizer, nitric oxide, and desiccant traps. Traps were weighed and then connected to each other using uniform lengths of 3/8-in. perfluoroalkoxy-grade (PFA) Teflon® tubing. The perfluoroalkoxy-grade tubing was heated in hot air and forced over the open ends of the traps to form a tight seal. The inlets of the sorbent trains each consist of a short section of tubing having a 3/8-in. stainless steel Swagelok® nut, and sealed using a Swagelok® cap. The trailing ends of the sorbent trains were each sealed with red plastic caps provided by the manufacturer.

2.1.1.1 Concentration Calculations. Concentration, in parts per million by volume (ppmv), was determined by dividing the amount of analyte, in μmol , by the moles of the dried tank air sampled. For example, the concentration of a 3.00-L sample containing 75.0 μg of ammonia equals

$$\frac{(75.0 \mu\text{g})(22.4 \text{ L/mol})}{(17.0 \mu\text{g}/\mu\text{mol})(3.00 \text{ L})} = 32.9 \text{ ppmv} \quad (2.1)$$

Measured sample volumes were specified by WHC at standard temperature and pressure (STP; 0°C, 1.013 bar). Because water vapor is removed as an analyte before the sample air stream passes through the mass flow meters, sample volumes exclude water vapor.

2.1.2 Analytical Procedures

2.1.2.1 Ammonia Analysis. The sorbent material from the ammonia traps was placed into labeled 20-mL glass scintillation vials. Vials containing front-section sorbent material were treated with 10.0 mL of deionized water (DIW), and vials containing breakthrough-section sorbent material were treated with 5.0 mL of DIW. Ammonia present was measured using the ion selective electrode (ISE) procedure PNL-ALO-226^(a). Briefly, the method includes 1) preparing a 1000- $\mu\text{g}/\text{mL}$ ammonia stock standard solution from dried reagent-grade NH_4Cl and DIW, 2) preparing 0.1-, 0.5-, 1.0-, 10-, and 100- $\mu\text{g}/\text{mL}$ ammonia working calibration standards by serial dilution of the freshly made stock standard, 3) generating an initial calibration curve from the measured electromotive force signal versus ammonia concentration data obtained for the set of working standards, 4) performing a calibration-verification check, using a mid-range dilution of a certified National Institute for Standards and Technology (NIST)-traceable 0.1 M NH_4Cl standard from an independent source, at a minimum of once per batch, 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). Electromotive force signal measurements obtained for samples are compared to those for standards to determine ammonia concentration in the samples.

2.1.2.2 Nitrite Analysis. The sorbent material for NO_2 and NO traps were desorbed in an aqueous TEA and n-butanol solution and analyzed by suppressed-conductivity ion chromatography

^(a) Procedure entitled "Ammonia (Nitrogen) in Aqueous Samples," PNL-ALO-226 Rev. 0, in the *Analytical Chemistry Laboratory (ACL) Procedure Compendium*, Vol. 3: Inorganic Instrumental Methods. Pacific Northwest National Laboratory, Richland, Washington.

(SCIC) for nitrite according to PNL-ALO-212, Rev. 1^(a) modified to obviate interferences by concentrations of non-target analytes. Specifically, the modifications used were 1) eluent 1.44 mM Na₂CO₃ + 1.8 mM NaHCO₃ 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 sample loop through 0.45- μ m syringe filters.

Primary and breakthrough section materials were analyzed separately using identical procedures. Each analytical session was conducted as follows. Working nitrite standards were prepared by diluting a stock nitrite standard with desorbing solution. An initial calibration curve was prepared from the instrument response versus nitrite standard concentration data for the set of working standards. A calibration verification check using one of the midrange standards was performed at a minimum of once per batch. If the instrument response indicated that sample nitrite concentration was outside the calibration range, 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. Instrument responses observed for samples were compared to those for standards to determine the nitrite concentration of the samples. Because the analytes were collected on the sorbent as equal quantities of nitrite and nitrate, and the analysis was specific for nitrite, the moles of NO₂ and NO were determined by doubling the analytically determined moles of nitrite.

2.1.2.3 Water Analysis. All sorbent traps used to make each multi-trap 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. Mass gain was assumed to be entirely due to collection of water vapor. Field blanks were used to correct results.

2.1.3 Quality Assurance/Quality Control

Analytical work was performed according to quality levels identified in PNL-ALO-212, PNL-ALO-226, and Quality Assurance Plan ETD-002. Estimated quantitation limits (EQLs) for the inorganic vapors are given in Table 2.3.

The accuracy of concentration measurements depends on errors associated with both sampling and analysis. Ammonia results were estimated to be within 5% of their true values. The uncertainty includes preparation of standards, purity of the ammonium salt used to prepare standards, potential operator bias, ambient temperature variations, etc. Working standards are traceable to NIST standard reference material (SRM) by using an independent calibration verification standard certified to be NIST-traceable. Nitrite analyses (for NO₂ and NO) are performed using certified but not NIST-traceable SRM; this is because NIST does not make a nitrite SRM. Based on experience in comparing nitrite working standards prepared from several different sources and factors mentioned for ammonia above, the estimated maximum bias for NO₂ results is $\pm 10\%$, and for NO results it is $\pm 5\%$.

^(a) 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 National Laboratory, Richland, Washington.

Table 2.3 Quantitation Limits for Selected Inorganic Analytes

<u>Analyte</u>	<u>Formula</u>	EQL ^(a) (ppmv)
Ammonia	NH ₃	0.71
Nitrogen Dioxide	NO ₂	0.16
Nitric oxide	NO	0.16
Mass (water) ^(b)	n/a	0.3 mg/L

(a) The lowest calibration standard is defined as the EQL.

(b) The vapor-mass concentration, thought to be largely water vapor, is determined gravimetrically.

n/a = not applicable.

The accuracy of measurements of sample mass is typically ± 0.1 mg, or less than 1% of the mass changes of most samples. The analytical accuracy of measurements of the change in mass of sorbent trains, based on the variability in mass change of field blank multi-trap trains, is determined for each sample job and is typically about ± 1 mg per five-trap sorbent train.

2.2 Permanent Gases

SUMMA™ canister samples were analyzed for carbon dioxide, carbon monoxide, hydrogen, methane, and nitrous oxide (N₂O).

2.2.1 SUMMA™ Canister Preparation

All SUMMA™ canisters are cleaned and verified contaminant-free according to PNNL Technical Procedure PNL-TVP-02^(a) before use. The cleaning procedure uses an EnTech 3000 cleaning system that alternately fills the canisters with purified humid air and evacuates them for several cycles while the canister is heated. If the canister is verified as clean, the canister is evacuated to 5 mtorr and tagged. Before sending the canisters 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. 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. All canisters are stored at room temperature.

2.2.2 Analytical Procedure

The SUMMA™ canister samples were analyzed for five permanent gases by gas chromatography/thermal conductivity detection (GC/TCD). Aliquots of sampled air (undiluted) are drawn directly from each SUMMA™ canister into a 5-mL gas-tight syringe and injected into a

^(a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, Pacific Northwest Laboratory, Richland, Washington.

Hewlett-Packard 5890 GC/TCD fitted with a loop injector valve and a column switching valve. An aliquot of 5 mL is used so that the 1.0-mL injection loop is completely purged with sample air, ensuring that no dilution of the sample takes place within the injection loop. One set of GC conditions is used to analyze for carbon monoxide, carbon dioxide, N₂O, and methane using helium as the carrier gas. A second GC/TCD analysis is performed for hydrogen using nitrogen as the carrier gas to enhance the signal sensitivity and lower the detection limit for this analyte. The permanent gases and the derived EQLs are listed in Table 2.4.

Table 2.4 Quantitation Limits for Permanent Gases

<u>Analyte</u>	<u>Formula</u>	<u>EQL (ppmv)</u>
Carbon Dioxide	CO ₂	17
Carbon Monoxide	CO	17
Methane	CH ₄	25
Hydrogen	H ₂	17
Nitrous Oxide	N ₂ O	17

2.2.3 Quality Assurance/Quality Control

Standards for the permanent gas analyses were blended from commercially prepared and certified standards. The instrument was calibrated at five different concentrations for methane over a range of 25 to 2100 ppmv, calibrated for carbon monoxide, carbon dioxide, and N₂O over a range of 17 to 2100 ppmv, and calibrated for hydrogen over a range of 17 to 2120 ppmv. An average response factor from the calibration was used for quantification.

Each analyte was quantitated by comparison of sample analyte peak area to the calibration plot generated for the compound. The EQL for the method has been established as the low level calibration standard. Before and after each sample analysis set, a gas standard was run to evaluate system performance and to measure system accuracy. The calculated concentration of the individual gases in the standards fell within ± 25% of the expected concentrations.

2.3 Total Non-Methane Organic Compounds

2.3.1 Analytical Procedure

SUMMA™ canister samples were analyzed for total non-methane organic compounds (TNMOCs) according to PNNL Technical Procedure PNL-TVP-08^(a), which is similar to U.S. Environmental Protection Agency (EPA) Task Order 12 (TO-12). Twenty-four hours before analysis, SUMMA™ canister samples are pressurized with purified air (Aadco Instruments, Inc., 1920 Sherwood St., Clearwater, Florida 34625). The original pressure is first measured using a calibrated diaphragm gauge (Cole Parmer), then pressurized to a level exactly twice the original pressure.

^(a) Pacific Northwest Laboratory. 12/95. *Determination of TO-12 Total Nonmethane Organic Compounds in Hanford Waste Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Flame Ionization Detection*, PNL-TVP-08 (Rev. 1), PNL Technical Procedure, Richland, Washington.

The method uses an EnTech 7000 cryoconcentration system interfaced with a Hewlett-Packard 5890 gas chromatograph/flame ionization detector (GC/FID). The EnTech concentrator is used to pull a metered volume of 50 to 100 mL of sample air from the SUMMA™ canister mounted on an EnTech 7016CA 16-canister autosampler. The sample is cryogenically concentrated, and constituents are trapped in a stainless steel tube containing glass beads and Tenax. The glass bead/Tenax trap is heated to 180°C and purged with ultra high purity (UHP) helium into the GC/FID. The GC oven is programmed to run at a 150°C isothermal temperature. Chromatographic separation is not needed in this method since quantitation is from the entire FID response over the run time.

Concentration in mg/m³ was derived from the 10-point multilevel calibration curve from the propane standard using the following equation:

$$\text{mg/m}^3 = \frac{(\text{ng TNMOC}) \times (\text{dilution factor})}{\text{mL sampled volume}} \quad (2.2)$$

2.3.2 Quality Assurance/Quality Control

The TNMOC is calibrated by using propane as the calibration standard. The instrument calibration mixture for the TNMOC analysis consists of NIST 99.999% propane analyzed using a 10-point, multilevel, linear regression curve.

Immediately before running the analysis sequence, a leak-check procedure, which includes evacuating the transfer lines and monitoring the pressure, must be performed on the sample manifold tower. The control limits on this test require that the change in pressure is <1.5 psi, and the absolute pressure after evacuation is <3 psi for each manifold position specified in the sequence table. If either criterion is not met, it must be corrected before the samples are analyzed.

Before the tank samples were analyzed, a diagnostic check was performed on the GC/FID instrument by running a system cleanliness procedure and an instrument continuing calibration as described in PNL-TVP-08. First, two blank volumes of Aadco purified air were analyzed to check the cleanliness of the system. This demonstrates through the analysis of a zero-air blank that the level of interference is acceptable in the analytical system. The analysis of purified air must be below 0.1 mg/m³. Second, an instrument continuing calibration is run using 100-mL UHP propane followed by one blank volume of Aadco air.

2.4 SUMMA™ Canister Sample Analyses

2.4.1 Analytical Procedure

SUMMA™ canister samples were analyzed according to PNNL Technical Procedure PNL-TVP-03^(a), which is a modified version of EPA Task Order 14 (TO-14). The method uses an EnTech 7000 cryoconcentration system interfaced with a 5972 Hewlett-Packard benchtop gas chromatograph/mass spectrometer (GC/MS). The EnTech concentrator is used to pull a metered volume of sample air from the SUMMA™ canister, cryogenically concentrate the condensable organic

^(a) Pacific Northwest National Laboratory. 8/96. *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. 2), PNNL Technical Procedure, Richland, Washington.

vapors, then transfer the vapors 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 a J&W Scientific DB-1 phase, 60-m by 0.32-mm internal diameter with 3- μ m film thickness column. The GC oven is programmed to run a temperature gradient beginning at 40°C, hold for 5 min, and ramp at 4°C per min to a final temperature of 260°C, with a 5-min hold.

The instrument calibration mixture for the PNL-TVP-03 analysis consists of organic analytes listed in Appendix B, Table B.1. These target analytes are quantitated in this analysis. The calibration mixture was prepared by blending a commercially prepared TO-14 calibration mixture with a mixture created using a Kin-Tek® permeation-tube standard generation system. The operation of the permeation-tube system follows the method detailed in PNNL Technical Procedure PNL-TVP-06^(a). The standard calibration mix was analyzed using six different concentrations, and a response factor for each compound was calculated. The GC/MS response for these compounds has been determined previously to be linearly related to concentration. Instrument detection limits and EQLs have been determined.

Quantitative concentrations for the target analytes were calculated using the average response factors generated using the internal standard (IS) method described in procedure PNL-TVP-03. The conversion from ppbv to mg/m³ assumes STP conditions (0°C and 1.013 bar) and was calculated using the gross molecular weight (MW) of the compound from the following equation:

$$\text{concentration in mg/m}^3 = \frac{(\text{concentration in ppbv})(\text{MW})}{(1000)(22.4 \text{ L/mol})} \quad (2.3)$$

The tentatively identified compounds (TICs) are determined by mass-spectral interpretation and comparison of the spectra with the EPA/NIST and WILEY electronic mass spectra libraries. 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 their concentrations are estimated. The quality of the mass spectra match 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/m³:

$$\text{Response Factor} = \frac{\text{IS conc. (mg/m}^3\text{)}}{\text{IS peak area}} \quad (2.4)$$

The calculated response factor was then multiplied by the TIC peak area to give an estimated concentration for that compound. All calculated sample concentrations were multiplied by a factor of 2 to account for the dilution step described in Section 2.3.1.

^(a) Pacific Northwest Laboratory. 11/94. *Preparation of TO-14 Volatile Organic Compounds Gas Standards*, PNL-TVP-06 (Rev. 0). PNL Technical Procedure, Richland, Washington.

2.4.2 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 the organic compounds listed in Table B.1. A gas mixture containing bromochloromethane, 1,4-difluorobenzene, chlorobenzene-d₅, and bromofluorobenzene was used as an 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 was determined that the relative response was linear with increasing concentration, an average response factor was calculated for each target analyte and used to determine the concentration of target compounds in each sample. Method blanks were analyzed before and after calibration verification standards.

2.5 Triple Sorbent Trap Sample Analyses

2.5.1 Sampling Methodology

Samples are collected on Supelco 300 graphite-based TSTs. Before field deployment, each trap is heated to 380°C under inert gas flow for a minimum of 60 min. Traps are prepared in batches with each tank sampling job constituting one batch. One trap is selected from each batch and run immediately to verify cleanliness. All remaining traps in the batch receive equal amounts of three surrogate compounds (hexafluorobenzene, toluene-d₈, and bromobenzene-d₅). One trap per batch is run immediately to verify successful addition of surrogate spikes to that batch. Traps are then placed in individually labeled plastic shipping tubes (Supelco TD³), which are sealed with gasketed end caps. This provides a rugged, headspace-free shipping and storage method. As a precautionary measure, sample tubes are kept in refrigerated storage before and after sampling.

2.5.2 Analytical Procedure

The Supelco 300 tubes were analyzed according to PNNL Technical Procedure PNL-TVP-10^(a), with the exceptions noted in Section 3.4.1. The method employs Supelco Carbotrap™ 300 traps for sample collection and preconcentration. The traps are ground-glass tubes (11.5 cm long X 6 mm OD, 4 mm ID) containing a series of sorbents arranged in order of increasing retentivity. Each trap contains 300 mg of Carbotrap™ C, 200 mg of Carbotrap™ B, and 125 mg of Carbosieve™ S-III. The first two sorbents are deactivated graphite with limited sorption power for less volatile compounds. The final trapping stage, the Carbosieve™ S-III, is a graphitized molecular sieve used to retain the most volatile components, including some permanent gases such as Freon-12. Following sample collection and addition of IS, the traps are transferred to a Dynatherm ACEM 900 thermal desorber unit for analysis. The trap on the ACEM 900 is then desorbed by ballistic heating to 350°C with the sample then transferred to a smaller focusing trap. A 10:1 split is used during the transfer with 10% of the sample analyzed and the rest retained for reanalysis. The split sample

(a) Pacific Northwest National Laboratory. 2/96. *Determination of Volatile Organic Compounds in Hanford Waste Tank Headspace Samples Using Triple Sorbent Trap Sampling and Gas Chromatograph-Mass Spectrometer Analysis*, PNL-TVP-10 (Rev. 2), PNNL Technical Procedure, Richland, Washington.

collected on a second identical Carbotrap™ 300 trap is used for repeat analysis on at least one sample per batch. Since the IS also follows the same path, quantitation may be performed directly on the repeat run without changing the calibration. Following desorption from the Carbotrap™ 300 trap, the analyte is transferred to a long, thin focusing trap filled with the same type of trapping materials as the Carbotrap™ 300 traps and in approximately the same ratios. The purpose of the focusing trap is to provide an interface to a capillary GC column, which may be thermally desorbed using helium at a flow rate compatible with the column and MS interface (1.2 mL/min). The focusing trap is ballistically heated to thermally desorb components onto a capillary GC column. The column is subsequently temperature programmed to separate the method analytes, which are then detected by the MS.

The instrument calibration mixture for the TST analysis consists of the compounds listed in Appendix B, Table B.2. These compounds that are directly quantified in this analysis make up the target analyte list (these compounds will be referred to as target analytes). The calibration mixture is prepared in common with the mixture used for the SUMMA™ analysis (see Section 2.4.2). The standard calibration mix was analyzed using six different concentrations, and a response factor for each compound was calculated. Volumes of standard added to the traps are measured by pressure difference on a SUMMA™ canister of known volume. The GC/MS response for these compounds has been determined previously to be linearly related to concentration.

Quantitative concentrations for the target analytes were calculated directly from the calibration curve generated using the IS method described in procedure PNL-TVP-10. The conversion from ppbv to mg/m³ assumes STP conditions (0°C and 1.013 bar) and was calculated directly from the following equation:

$$\text{mg/m}^3 = \frac{(\text{ppbv}/1000) \times \text{g mol wt of compound}}{22.4 \text{ L/mol}} \quad (2.5)$$

NOTE: Because of laboratory error, STP conditions were not used. All TST tank sample results are reported as mg/m³ are at 21°C and 760 mm Hg pressure. All TST tank sample results reported as ppbv must be multiplied by 1.077. Blank sample results need no further corrections.

The TICs are determined by mass-spectral interpretation and comparison of the spectra with the EPA/NIST and WILEY Libraries, which are a part of the Hewlett-Packard 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/m³:

$$\text{Response Factor} = \frac{\text{IS conc. (mg/m}^3\text{)}}{\text{IS peak area}} \quad (2.6)$$

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

2.5.3 Quality Assurance/Quality Control

Before tank samples were analyzed, a diagnostic check was performed on the GC/MS instrument by running a full auto tune, as described in PNL-TVP-10. Upon satisfactory completion of the instrument diagnostic check, a blank trap was analyzed to check the cleanliness of the system. The instrument was then calibrated using six different concentrations of standard gas mixture containing the compounds listed in Table B.2. A gas mixture containing difluorobenzene, chlorobenzene-d₅, and 1,4 bromofluorobenzene was used as an IS for all 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. A continuing 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 was determined that the relative response was linear with increasing concentration, an average response factor was calculated for each target analyte and used to determine the concentration of target compounds in each sample.

3.0 Analysis Results

Results from the sampling of the headspace of Tank C-107 on September 5, 1996 (Sample Job S6091) are provided below.

3.1 Inorganic Vapors

Measured vapor concentrations of ammonia, nitric oxide (NO), nitrogen dioxide (NO₂), and water are given in Table 3.1. The vapor concentration results were based on four samples for each compound, with the exception of only three samples for NO₂. The four inorganic vapors were collected at the same time using sorbent traps connected in series. Sample air was drawn first through an ammonia trap, then through a three-tube system that collected NO₂ and NO (described below), and then through a desiccant trap to remove any remaining water vapor.

Two field blank multi-trap trains, identical to sample multi-trap trains discussed in Section 2.1, were included in the tube bundle lowered into the headspace of Tank C-107 during sample job S6091. No air was pulled through these field blank multi-trap trains. Any analyte found in the field blank multi-trap trains over and above levels in unexposed tubes was attributed to passive sampling. Data in Table 3.1 have been corrected for these minor effects of passive sampling.

Results provided in Table 3.1 are estimated to be accurate to within $\pm 10\%$ and within the $\pm 30\%$ specified by the SAP. Percent relative standard deviations of the measured concentrations were $<6\%$, which is within the 25% specified by the SAP.

3.1.1 Ammonia

Ammonia analyses were performed on September 24, 1996, 19 days after sample collection and within established holding times (Ligotke et al. 1995). All samples (100%) were successfully analyzed, and no deviations from the procedure were noted.

The blank-corrected ammonia quantities in the sorbent traps ranged from 5.06 to 5.41 μmol in front sections; blank corrected back sorbent section ammonia concentrations were $<0.01 \mu\text{mol}$. Blank corrections of 0.08 μmol in front and 0.05 μmol in the back sections, were about 1.5% of collected quantities. The analysis of one sample was a duplicate and indicated a reproducibility of $\pm 2.3\%$. One blank sorbent trap was spiked with 17 ppm of ammonia and yielded a percentage recovery of 104%. One sample leachate was spiked after initial analysis with approximately the quantity of ammonia in the sample and yielded a percentage recovery of 99%. The initial and continuing calibration verification (ICV, CCV) standards, using NIST-traceable material, yielded percentage recoveries of 100% (ICV) and 100% (CCV) during the analytical session. A five-point calibration was performed over an ammonia range of 0.1 to 100 $\mu\text{g/ml}$.

3.1.2 Nitric Oxide and Nitrogen Dioxide

Nitric oxide and nitrogen oxide analyses were performed on September 23 and 24, 1996, 18 and 19 days after sample collection, and within established holding times (Ligotke et al. 1995). All samples (100%) were successfully analyzed. No deviations from the procedure were noted.

Blank-corrected NO_2^- quantities in the NO_2 sorbent traps were all $<0.013 \mu\text{mol}$. Blank corrected NO_2^- quantities in the NO sorbent traps ranged from 0.007 to 0.008 μmol . Nitrite blank levels used to correct data were 0.0055 μmol in front (four of four blanks analyzed) and 0.0036 μmol in back (two of four blanks analyzed) sorbent sections. The analyses of two samples were duplicated and yielded repeatabilities of $\pm 0\%$. Two sample leachates were spiked with 0.125 and 0.50 ppm NO_2^- and yielded percentage recoveries of 96% and 95%, respectively. A four-point calibration was performed over a concentration range of 0 to 0.5 $\mu\text{g/mL NO}_2^-$ in the desorbing matrix.

3.1.3 Water

Analyses for water vapor were performed on September 19, 1996, within established holding times (Ligotke et al. 1995). All samples (100%) were successfully analyzed.

All multi-trap sample mass gain is assumed to be due to adsorption of water. This is justified because the total mass concentration of other vapors in the headspace of Hanford waste tanks are typically two to three orders of magnitude less than the mass concentration of the water vapor found in even relatively dry tanks. Water vapor concentrations are given in Table 3.1 for both moist air at tank conditions and for dry air at STP (0°C and 1.013 bar). Because the sample volumes were measured after all water vapor was removed by the sorbent traps, the measured sample volumes are for dry air. The average water vapor concentration was 74.0 mg of water per L of dry air at STP. The result was determined from an average mass gain of 137.9 mg from all multi-trap trains. The blank correction applied to the results was - 0.3 mg per multi-trap train. A control mass was measured and indicated a measurement accuracy of ± 0.1 mg. The average water vapor concentration corresponds to a tank headspace dew point at 42.4°C and relative humidity at 92% at the time of sampling.

3.2 Permanent Gases

Hydrogen analyses were performed on September 20, 1996, and analyses for other permanent gases were performed on September 18, 1996. All analyses were conducted within the 60-day administrative holding time as specified in the WHC Tank Vapor Characterization QA Plan (WHC 1994). All samples (100%) were successfully analyzed and used in the averages. No deviations from standard procedures were noted.

Measured concentrations of carbon monoxide, carbon dioxide, hydrogen, methane, and nitrous oxide are provided in Table 3.2. Results were based on three samples for each compound. Hydrogen (562 ppmv), carbon dioxide (812 ppmv), and nitrous oxide (120 ppmv) were detected in Tank C-107 at concentrations above those in ambient air. The relative percent difference (RPD) between duplicate analyses of a single SUMMA™ canister was $<1\%$ for all three compounds detected.

Results provided in Table 3.2 are estimated to be accurate to within $\pm 30\%$ as specified by the SAP.

3.3 Total Non-Methane Organic Compounds

Analyses for TNMOCs were performed on October 25, 1996, which is within the 60-day administrative holding time as specified in the WHC Tank Vapor Characterization QA Plan (WHC 1994). All three tank samples and the two ambient samples (100%) were successfully analyzed and used in the averages. No deviations from standard procedures were noted.

Table 3.3 lists results of the EPA TO-12 analysis of SUMMA™ canister samples for TNMOCs. Results in Table 3.3 are reported in two different units; in the upper row the mass concentration (mg/m^3) of non-methane organic compounds is given at STP (0°C and 1.013 bar), and in the lower row, by EPA TO-12 convention, as ppmv of carbon based on propane as the standard. The average concentration in the three tank headspace samples was $3.44 \text{ mg}/\text{m}^3$ or 1.44 ppmv of carbon. The $3.44 \text{ mg}/\text{m}^3$ value compares to $4.52 \text{ mg}/\text{m}^3$ for the sum of all target compounds identified in the analysis of the SUMMA™ canisters. The $4.52 \text{ mg}/\text{m}^3$ value should be considered a minimum value since TICs were not reported in the temporal study. Results provided in Table 3.3 are estimated to be accurate to within $\pm 30\%$ as specified by the SAP. The RPD for duplicate analysis of a single SUMMA™ canister was 4%.

3.3.1 Procedural Deviations, Observations, and Anomalies.

The calibration method described in Section 2.3.2 reflects a deviation from procedure PNL-TVP-08. Refer to Deviation Report JAE082996 for further details.

3.4 Organic Compound Characterization

Organic vapors in the Tank C-107 headspace were sampled with SUMMA™ canisters and TSTs. These sampling methods are fundamentally different, but provide comparable results for many of the target analytes.

3.4.1 SUMMA™ Canister Results

Analyses of SUMMA™ canisters for organic vapors were performed on September 28, 1996. All analyses were conducted within the 60-day administrative holding time as specified in the WHC Tank Vapor Characterization QA Plan (WHC 1994). All samples (100%) were successfully analyzed. Procedural deviations, observations, and anomalies are presented in Section 3.4.1.1.

Measured concentrations of target analytes are presented in Table 3.4; because this is part of a temporal study, TICs are not reported. Twenty-three target analytes above the instrument detection limit (IDL) were detected in the tank headspace samples. Of these, 21 target analytes were identified in two or more tank headspace samples. Acetone at $1.387 \text{ mg}/\text{m}^3$, acetonitrile at $1.188 \text{ mg}/\text{m}^3$, and methanol at $0.956 \text{ mg}/\text{m}^3$ were the three most abundant compounds identified in the tank headspace samples. The total average concentration was $4.52 \text{ mg}/\text{m}^3$ for the target analytes. This compares to a total concentration of $3.44 \text{ mg}/\text{m}^3$ identified in the TO-12 analysis of the three tank headspace samples.

SUMMA™ canister S6091-A05.285 was analyzed in replicate for target analytes to determine analytical precision. The RPD results are presented in Table 3.5. The RPDs were calculated for

analytes detected above the IDL and found in both replicates. Fifteen of 20 target analytes had RPDs of less than 10%.

Results of analyses of target analytes in ambient air and ambient air through the VSS samples are provided in Table 3.6. Fourteen target compounds were observed in one or both of the ambient air samples. All target compounds were observed below the EQL with the exception of 2-butanone which was observed at 0.029 mg/m³.

A representative total ion chromatogram showing the major constituents identified in the SUMMA™ analysis is provided in Figure 3.1.

Appendix B, Table B.1 contains complete listings of target analyte results from the SUMMA™ analyses. These data originate directly from the evaluated instrument data and have not been rounded. The intent of the data is to list the analytes that were analyzed and to present the detection limit values associated with analytes that were not detected.

3.4.1.1 Procedural Deviations, Observations, and Anomalies. The SUMMA™ canister samples were analyzed in one batch. The sample analytical sequence run (batch) was as follows:

Batch #1 (File Identifier # 16092801.b) - S6091-A01.275, S6091-A02.283, S6091-A04.284, S6091-A05.285, S6091-A05.285 REP, and S6091-A06.287.

One target compound (tetradecane - 51.16%) surpassed the 30% relative standard deviation (%RSD) acceptance criteria for the initial calibration. Tetradecane was not found in any samples above its IDL, except for tank sample S6091-A04.284, where it was observed at a concentration above the IDL but below the EQL.

The %D for the continuing calibration was less than 25% for all target compounds except chloromethane (34.5%), 1,2-dichloro-1,1,2,2-tetrafluoroethane (33.5%), cyclohexanone (28.9%), and decane (31.6%). The response factor for tetradecane was less than 0.05 (0.022). However, the CCV passed the procedural criterion requiring ±25%D passage for 85% of all target compounds. Cyclohexanone and 1,2-dichloro-1,1,2,2-tetrafluoroethane were not found in tank samples at concentrations above their IDLs, except for ambient air sample S6091-A01.275, where cyclohexanone was found at concentrations between its IDL and EQL. Chloromethane and decane were found in all tank samples at concentrations between their IDL and EQL, but were not found above their IDL in the two ambient air samples.

Target compound 1,2,4-trichlorobenzene was found in the CCB above its EQL, but its concentration was less than 1.5 ppbv. Two target compounds (1,2,4-trichlorobenzene and 1,2-dichlorobenzene) were found in the ICB above their EQLs, but their concentrations were less than 2.1 ppbv.

Instrument detection limits, precision, and accuracy have not been experimentally evaluated for methanol, ethanol, or 1,3-butadiene. Sample results are flagged with a "<" when the absolute number of nanograms calculated in the sample is less than the lowest concentration standard used in the initial calibration. Methanol, ethanol, and 1,3-butadiene results falling within their calibration range are quantitative results as evidenced by a valid calibration for these compounds.

3.4.2 Triple Sorbent Trap Sample Results

Analyses of TSTs were performed on October 2 and 3, 1996. All analyses were conducted within the 60-day administrative holding time as specified in the WHC Tank Vapor Characterization QA Plan (WHC 1994). Three samples were successfully analyzed. Sample S6091-A11.1201 was damaged during sampling and could not be analyzed (see Appendix A page A.3). Procedural deviations, observations, and anomalies are presented in Section 3.4.2.1.

NOTE: All TST tank sample results reported in mg/m³ are at 21°C and 760 mm Hg pressure. To correct to 0°C and 760 mm Hg pressure multiply by 1.077. All TST tank sample results reported in ppb must be corrected by multiplying by 1.077. TST blank sample results need no further correction.

Measured concentrations of target analytes are presented in Table 3.7; because this is part of a temporal study, TICs are not reported. Results are based on three samples for each compound. Twenty-three target analytes above the IDL were detected in the tank headspace samples. Of these, all were identified in two or more tank headspace samples. Methanol at 1.624 mg/m³, acetone at 1.027 mg/m³, and acetonitrile at 1.004 mg/m³ were the three most abundant compounds identified in the tank headspace sample. The total concentration of the target analytes was 4.95 mg/m³.

Triple sorbent trap sample S6091-A13.1203 was analyzed in replicate for target analytes to determine analytical precision. The RPD results are presented in Table 3.8. The RPDs were calculated for analytes detected above the detection limit and found in both replicates. Eleven of 20 target analytes had RPDs of less than 10%.

Results of analyses of target analytes in the field and trip blanks are provided in Table 3.9. Three target analytes were identified in one or more of the blank samples. None of the target compounds observed were above the EQL in any sample.

A representative total ion chromatogram showing the major constituents identified in the triple sorbent trap analysis is provided in Figure 3.2.

Appendix B, Tables B.2 and B.3 contain complete listings of target analyte results from the TST analyses. These data originate directly from the evaluated instrument data and have not been rounded. The intent of the data is to list the analytes that were analyzed and to present the detection limit values associated with analytes that were not detected.

3.4.2.1 Procedural Deviations, Observations, and Anomalies. Samples were run under the protocols of TVP-10, Rev. 2 with the initial calibration performed on September 9, 1996 and quantitated against a CCV run at the beginning of the batch.

The TST samples were analyzed in two batches. The sample analytical sequence runs were as follows:

Batch #1 (file identifier #46100201.b) - S6091-A17.1205, S6091-A18.1206, S6091-A19.1207, S6091-A20.1208.

Batch #2 (file identifier #46100301.b) - S6091-A12.1202, S6091-A13.1203, S6091-A13.1203 REP, S6091-A14.1204.

Batch #1:

Samples included in this batch consisted of the two field blanks and two trip blanks.

The CCV showed acceptable performance as specified in the procedure for all target compounds except tetradecane (49%). Tetradecane results have a higher uncertainty due to the CCV performance. The CCB was clean of all target and TIC compounds except for methylene chloride which was below the EQL. The CCB concentration of methylene chloride was comparable to that in the samples and therefore were flagged with a B.

Batch #2:

Samples included in this batch consisted of the three tank samples and one repeat analysis of a tank sample. These samples were collected using the VSS truck on 9/5/96.

The CCB was clean of all target and TIC compounds except for methylene chloride which was below the EQL.

Information common to both batches:

These samples were collected by the VSS truck method and did not show the environmental contamination problems apparently associated with field bundle preparation used in the ISVS cart method of sampling.

One trip blank and two samples contained minor amounts of 1-chloro-1,1-difluoroethane. This compound has appeared persistently in most samples sent to the field in the past including blanks. It is believed to be a fugitive refrigerant. This material is never present in tubes archived for a similar amount of time in the 326 Vapor Lab or 329 Building temporary storage. The origin of the material is unclear but since it has shown up in trip blanks as well as field blanks, the most likely candidate is one of the refrigerators used for interim storage.

Very narrow air spikes (mass 32) occasionally are present in chromatograms from this period. This problem has been traced to high-frequency air bursts from the surface of MS vacuum system o-rings. Attempts at permanently eliminating this problem have been unsuccessful to date and the matter has been referred to HP for further investigation. There is no known impact on data quality but the spikes do appear as features on the total ion chromatogram.

Methanol, ethanol, and 1,3-butadiene are not currently included in the method performance section of the procedure; however, these analytes were analyzed by this method. The low level standard is used as the EQL for these compounds. Sample results are flagged with a less-than symbol (<) when less than the EQL value.

Tributyl phosphate (TBP) is included in the target list based on a calibration performed on January 5 and 9, 1996. The TBP was introduced onto a series of double sorbent traps as a methanolic solution standard rather than a vapor standard. This served to determine the retention time and verify the mass spectral characteristics of the compound. However, verification of the calibration acceptability was not performed because the compound is not present in the CCV. At present, it is not possible to prepare a gas standard from this material. The calibration information on TBP demonstrated that detectability at 0.08 ppbv (based on a 200-mL sample) was possible. Tributyl phosphate was not detected in the samples.

3.5 Flammability

The analytical results presented above can be used to estimate the tank C-107 headspace flammability at the time of sampling. Flammability is calculated using the ammonia concentration from the inorganic analysis, carbon monoxide, hydrogen, and methane concentrations measured from the permanent gas analysis, and the total nonmethane organic concentration determined from the TO-12 analysis. Table 3.10 summarizes the calculated flammability data. Hydrogen was the principal flammable constituent of the Tank C-107 headspace, determined to be present at approximately 1.405% of its LFL. Total headspace flammability was estimated to be <1.519% of the LFL.

Table 3.1 Inorganic Vapor Concentrations from Tank C-107 Sampled on 9/5/96

Analyte	CAS Number	S6091-A07-94R	S6091-A08-95R	S6091-A09-96R	S6091-A10-97R	Average	Standard Deviation
Ammonia (ppmv)	7664-41-7	61	66	63	63	63	2
Nitric Oxide (ppmv)		0.33	0.39	0.37	0.39	0.37	0.02
Nitrogen Dioxide (ppmv)		<0.16	<0.16	NA	<0.16	<0.16	
Water ^(a) (mg/L)		72.5	75.3	74.1	74.1	74.0	1.2
Water ^(b) (mg/L)		56.3	58.3	57.4	57.4	57.3	0.9

(a) Dry air at 0°C and 1.013 bar.

(b) Moist air at tank temperature and pressure.

NA Interference during analysis.

Table 3.2 Permanent Gas Analysis Results from Tank C-107 Sampled on 9/5/96

Analyte	Permanent Gas	Ambient Air		Tank Samples						Mean	St.Dev.	
		Upwind	Through VSS	S6091-A01.275 ^(a)	S6091-A02.283 ^(a)	S6091-A04.284 ^(a)	S6091-A06.287 ^(a)	S6091-A05.285 ^(a)	S6091-A05.285 ^{(a)(b)}			RPD ^(c)
		(ppmv)	(ppmv)	(ppmv)	(ppmv)	(ppmv)	(ppmv)	(ppmv)	(ppmv)	(%)	(ppmv)	
Hydrogen		<17	<17	562	560	563	562	562	562	0.2	562	2
Methane		<25	<25	<25	<25	<25	<25	<25	<25		<25	
Carbon Dioxide		373	373	812	806	818	818	818	818	0.0	812	6
Carbon Monoxide		<17	<17	<17	<17	<17	<17	<17	<17		<17	
Nitrous Oxide		<17	<17	118	118	123	122	122	120	0.8	120	3

(a) WHC sample identification number.

(b) Replicate analysis for S6091-A06.285; results are not included in the calculation of average concentrations.

(c) Relative percent difference (RPD) based on replicate analysis.

Table 3.3 Total Non-Methane Organic Compound Analysis Results from Tank C-107 Sampled on 9/5/96

Ambient Air		Tank Samples			
Upwind	Ambient Air Through Bundle	S6091-A04.284 ^(a)	S6091-A06.287 ^(a)	S6091-A05.285 ^(a)	S6091-A05.285 ^{(a)(b)}
Concentration	Concentration	Concentration	Concentration	Concentration	RPD ^(c)
< 0.59	< 0.59	3.43	3.46	3.44	4
				3.31	
					Mean
					3.44
					St.Dev.
					0.02
					1.44
TO-12 (mg/m ³)					
Total Carbon (ppmv)					

(a) WHC sample identification number.

(b) Replicate analysis for S6091-A05.285; results are not included in the calculation of average concentrations.

(c) Relative percent difference (RPD) based on replicate analysis.

Table 3.4 Organic Compound Concentrations in SUMMA™ Canisters from Tank C-107 Sampled on 9/5/96

Target Analytes ^(a)	C.A.S.	MW	Ret Time	S6091-A04.284 ^(b) VSS		S6091-A05.285 ^{(b)(c)} VSS		S6091-A06.287 ^(b) VSS		Mean and Standard Deviation			
				(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	St. Dev. (ppbv)	St. Dev. (mg/m ³)	
Dichlorodifluoromethane	75-71-8	121	4.6	0.003	0.59	0.003	0.58	0.003	0.61	0.003	0.000	0.59	0.015
Chloromethane	74-87-3	50	4.9	0.009	3.8	0.008	3.6	0.008	3.4	0.008	0.000	3.6	0.19
Methanol	67-56-1	32	5.2	0.872	610	1.008	705	0.989	691	0.956	0.073	669	51
Butane	106-97-8	58	5.9	0.008	2.9	0.007	2.8	0.007	2.7	0.007	0.000	2.8	0.12
Bromomethane	74-83-9	95	6.3	0.022	5.1	0.021	4.9	0.019	4.6	0.021	0.001	4.8	0.25
Ethanol	64-17-5	46	6.8	0.121	59	0.129	63	0.125	61	0.125	0.004	61	2.1
Acetonitrile	75-05-8	41	7.3	1.152	628	1.199	654	1.212	661	1.188	0.032	648	17
Acetone	67-64-1	58	7.8	1.369	528	1.392	537	1.402	541	1.387	0.017	535	6.6
Trichlorofluoromethane	75-69-4	137	8.3	0.161	26	0.164	27	0.164	27	0.163	0.002	27	0.31
Methylene Chloride	75-09-2	85	9.7	0.015	4.1	0.015	3.9	0.015	4.0	0.015	0.000	4.0	0.067
Propanol	71-23-8	60	10.9	0.031	11	0.030	11	0.029	11	0.030	0.001	11	0.27
Propanenitrile	107-12-0	55	10.9	0.248	101	0.258	105	0.257	104	0.254	0.006	103	2.3
2-Butanone	78-93-3	72	12.5	0.095	30	0.090	28	0.079	24	0.088	0.008	27	2.6
Butanenitrile	109-74-0	69	15.8	0.129	42	0.137	44	0.130	42	0.132	0.005	43	1.5
1-Butanol	71-36-3	74	16.3	0.015	4.6	0.016	4.8	0.013	3.9	0.015	0.002	4.4	0.50
Heptane	142-82-5	100	19.2	0.002	0.40	0.002	0.45	0.002	0.40	d	d	d	d
Pentanitrile	110-59-8	83	21.6	0.015	3.9	0.015	4.0	0.013	3.6	0.014	0.001	3.8	0.19
Hexanenitrile	628-73-9	97	27.1	0.010	2.4	0.011	2.5	0.010	2.3	0.010	0.000	2.4	0.095
Decane	124-18-5	142	34.4	0.004	0.69	0.005	0.72	0.003	0.51	0.004	0.001	0.64	0.11
Undecane	1120-21-4	156	38.8	0.012	1.7	0.010	1.5	0.008	1.1	0.010	0.002	1.4	0.29
Dodecane	112-40-3	170	42.8	0.033	4.4	0.028	3.6	0.015	2.0	0.025	0.009	3.3	1.2
Tridecane	629-50-5	184	46.5	0.105	13	0.071	8.6	0.025	3.1	0.067	0.040	8.2	4.9
Tetradecane	629-59-4	198	50.0	0.024	2.7	0.004	0.47	0.004	0.47	d	d	d	d
Total Concentration of Target Analytes										4.522			

Data Quality Flags

- J Target compound detected above the IDL but below the EQL.
- U Target compound not detected at or above the IDL.
- Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.
- (a) Detected target analytes.
- (b) WHC sample identification number.
- (c) Replicates of this sample are found in Table 3.5.
- (d) Mean and/or standard deviation are not meaningful for this analyte.

Table 3.5 Comparison of Organic Compound Concentrations from Replicate Analysis of a Single SUMMA™ Canister from Tank C-107 Sampled on 9/5/96

Target Analytes ^(a)	CAS	MW	Ret Time	S6091-A05.285 ^(b) VSS (mg/m ³)	S6091-A05.285 ^(b) VSS (ppbv)	S6091-A05.285 ^(b) VSS (mg/m ³)	S6091-A05.285 ^(b) VSS (ppbv)	VSS REP Flag	RPD ^(c) %
Dichlorodifluoromethan	75-71-8	121	4.6	0.003	0.58	0.003	0.53	J	7
Chloromethane	74-87-3	50	4.9	0.008	3.6	0.009	3.8	J	6
Methanol	67-56-1	32	5.2	1.008	705	1.044	730	Y	4
Butane	106-97-8	58	5.9	0.007	2.8	0.006	2.4	J	12
Bromomethane	74-83-9	95	6.3	0.021	4.9	0.021	5.0	J	2
Ethanol	64-17-5	46	6.8	0.129	63	0.124	60	Y	5
Acetonitrile	75-05-8	41	7.3	1.199	654	1.210	660		1
Acetone	67-64-1	58	7.8	1.392	537	1.396	539		0
Trichlorofluoromethane	75-69-4	137	8.3	0.164	27	0.157	26		4
Methylene Chloride	75-09-2	85	9.7	0.015	3.9	0.015	4.0	J	2
Propanol	71-23-8	60	10.9	0.030	11	0.030	11		0
Propanenitrile	107-12-0	55	10.9	0.258	105	0.261	106		1
2-Butanone	78-93-3	72	12.5	0.090	28	0.087	27		3
Butanenitrile	109-74-0	69	15.8	0.137	44	0.136	44		1
1-Butanol	71-36-3	74	16.3	0.016	4.8	0.015	4.6	J	5
Heptane	142-82-5	100	19.2	0.002	0.45	0.002	0.40	U	
Pentanenitrile	110-59-8	83	21.6	0.015	4.0	0.015	4.0	J	1
Hexanenitrile	628-73-9	97	27.1	0.011	2.5	0.009	2.0	J	22
Decane	124-18-5	142	34.4	0.005	0.72	0.004	0.56	J	24
Undecane	1120-21-4	156	38.8	0.010	1.5	0.008	1.1	J	26
Dodecane	112-40-3	170	42.8	0.028	3.6	0.015	2.0	J	57
Tridecane	629-50-5	184	46.5	0.071	8.6	0.006	0.73	U	

Data Quality Flags

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

(a) Detected target analytes.

(b) WHC sample identification number.

(c) Relative percent differences (RPDs) based on mg/m³ values.

Table 3.6 Organic Compound Concentrations in Ambient Air and Ambient Air through the VSS in SUMMA™ Canisters Associated with the Sampling of Tank C-107 on 9/5/96

Target Analytes ^(a)	CAS	MW	Ret Time	S6091-A02.283 ^(b)		S6091-A01.275 ^(b)	
				Ambient Air through VSS (mg/m ³)	Flag	Ambient Air through VSS (mg/m ³)	Flag
Dichlorodifluoromethane	75-71-8	121	4.6	0.003	J	0.004	J
Acetone	67-64-1	58	7.8	0.018	J	0.016	J
Methylene Chloride	75-09-2	85	9.7	0.014	J	0.014	J
2-Butanone	78-93-3	72	12.5	0.029	J	0.010	J
1-Butanol	71-36-3	74	16.3	0.004	J	0.010	J
Cyclohexanone	108-94-1	98	28.5	0.006	U	0.008	J
1-Ethyl-2-methylbenzene	611-14-3	120	32.9	0.002	U	0.002	J
1,3,5-Trimethylbenzene	108-67-8	120	32.7	0.002	U	0.002	J
1,2,4-Trimethylbenzene	95-63-6	120	34.2	0.003	U	0.003	J
1,3-Dichlorobenzene	541-73-1	147	34.7	0.002	U	0.004	J
1,4-Dichlorobenzene	106-46-7	147	34.9	0.002	U	0.004	J
1,2-Dichlorobenzene	95-50-1	147	36.1	0.002	U	0.008	J
1,2,4-Trichlorobenzene	120-82-1	181	42.3	0.003	U	0.022	J,B
Hexachloro-1,3-butadiene	87-68-3	261	44.1	0.005	U	0.012	J

Data Quality Flags

- B Compound found in associated laboratory blank.
 - J Target compound detected above the IDL but below the EQL.
 - U Target compound not detected at or above the IDL.
- (a) Detected target analytes.
 (b) WHC sample identification number.

Table 3.7 Organic Compound Concentrations in Triple Sorbent Traps from Tank C-107 Sampled on 9/5/96

NOTE: All data in this table is reported at 21°C and 760 mm Hg pressure. To correct to 0°C and 760 mm Hg pressure multiply by 1.077.

Target Analytes ^(a)	CAS	MW	Ret Time	S6091-A12.1202 ^(b) VSS		S6091-A13.1203 ^{(b)(c)} VSS		S6091-A14.1204 ^(b) VSS		Mean and Standard Deviation			
				(mg/m ³)	(ppbv) Flag	(mg/m ³)	(ppbv) Flag	(mg/m ³)	(ppbv) Flag	(mg/m ³) St. Dev	(ppbv) St. Dev.		
Methanol	67-56-1	32	10.0	1.577	1102 Y	1.665	1164 Y	1.630	1140 Y	1.624	0.045	1135	31
Chloroethane	75-00-3	65	12.1	0.076	26	0.085	29	0.088	31	0.083	0.006	29	2.2
Ethanol	64-17-5	46	12.5	0.300	146 Y	<0.275	<133 Y	0.305	148 Y	0.302	0.004	147	2
Acetonitrile	75-05-8	41	13.0	0.970	529	1.085	592	0.959	523	1.004	0.070	548	38
Acetone	67-64-1	58	13.6	0.973	375	1.161	448	0.948	366	1.027	0.116	396	45
Trichlorofluoromethane	75-69-4	137	14.1	0.181	30	0.188	31	0.183	30	0.184	0.003	30	0.57
Pentane	109-66-0	72	14.9	0.006	1.7 J	0.010	3.1 J	0.010	3.2 J	0.009	0.003	2.7	0.82
Methylene Chloride	75-09-2	85	15.7	0.047	12 J	0.041	11 J	0.041	11 J	0.043	0.003	11	0.85
Propanenitrile	107-12-0	55	17.0	0.246	100	0.265	108	0.248	101	0.253	0.011	103	4.4
Propanol	71-23-8	60	17.0	0.078	29	0.068	25	0.075	28	0.073	0.005	27	1.9
2-Butanone	78-93-3	72	18.6	0.070	22	0.093	29	0.075	23	0.080	0.012	25	3.8
Hexane	110-54-3	86	19.8	0.004	1.2 J	0.004	1.1 J	0.004	1.0 J	0.004	0.000	1.1	0.070
Butanenitrile	109-74-0	69	21.8	0.113	37	0.124	40	0.113	36	0.117	0.007	38	2.2
1-Butanol	71-36-3	74	22.3	0.026	7.8 J	0.019	5.6 J	0.020	5.9 J	0.021	0.004	6.4	1.2
Benzene	71-43-2	78	22.8	0.006	1.7 J	0.001	0.40 U	0.005	1.5 J	0.005	d	1.6	d
Pyridine	110-86-1	79	26.5	0.031	8.9 J	0.021	6.0 J	0.022	6.2 J	0.025	0.006	7.0	1.6
Pentanenitrile	110-59-8	83	27.4	0.014	3.8	0.013	3.5	0.014	3.7	0.014	0.001	3.7	0.17
Toluene	108-88-3	92	28.5	0.002	0.55 J	0.003	0.63 J	0.002	0.38 J	0.002	0.001	0.52	0.13
Hexanenitrile	628-73-9	97	32.8	0.011	2.6 J	0.010	2.4 J	0.011	2.6 J	0.011	0.001	2.5	0.14
Undecane	1120-21-4	156	44.4	0.009	1.3 J	0.009	1.3 J	0.010	1.4 J	0.009	0.001	1.4	0.074
Dodecane	112-40-3	170	48.4	0.015	1.9 J	0.015	2.0 J	0.017	2.3 J	0.016	0.001	2.1	0.20
Tridecane	629-50-5	184	52.2	0.021	2.6 J	0.030	3.6 J	0.033	4.0 J	0.028	0.006	3.4	0.71
Tetradecane	629-59-4	198	55.7	0.017	1.9 J	0.008	0.93 J	0.006	0.73 J	0.010	0.006	1.2	0.63
Total Concentration of Target Analytes										4.945			

Data Quality Flags

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

(a) Detected target analytes.

(b) WHC sample identification number.

(c) Replicates of this sample are found in Table 3.8.

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

Table 3.8 Comparison of Organic Compound Concentrations from Replicate Analysis of a Single Triple Sorbent Trap from Tank C-107 Sampled on 9/5/96

NOTE: All data in this table is reported at 21°C and 760 mm Hg pressure. To correct to 0°C and 760 mm Hg pressure multiply by 1.077.

Target Analytes ^(a)	CAS	MW	Ret Time	S6091-A13.1203 ^(b) VSS (mg/m ³)	S6091-A13.1203 ^(b) VSS REP (ppbv)	S6091-A13.1203 ^(b) VSS REP (mg/m ³)	S6091-A13.1203 ^(b) VSS REP (ppbv)	Flag	RPD ^(c) %
Methanol	67-56-1	32	10.0	1.665	1164	1.497	1046	Y	11
Chloroethane	75-00-3	65	12.1	0.085	29	0.042	15		67
Ethanol	64-17-5	46	12.5	<0.275	<133	<0.275	<133	Y	
Acetonitrile	75-05-8	41	13.0	1.085	592	1.073	585		1
Acetone	67-64-1	58	13.6	1.161	448	1.040	401		11
Trichlorofluoromethane	75-69-4	137	14.1	0.188	31	0.176	29		6
Pentane	109-66-0	72	14.9	0.010	3.1	0.010	3.0	J	4
Methylene Chloride	75-09-2	85	15.7	0.041	11	0.045	12	J	9
Propanenitrile	107-12-0	55	17.0	0.265	108	0.270	110		2
Propanol	71-23-8	60	17.0	0.068	25	0.063	23		7
2-Butanone	78-93-3	72	18.6	0.093	29	0.082	25		13
Hexane	110-54-3	86	19.8	0.004	1.1	0.004	1.1	J	5
Butanenitrile	109-74-0	69	21.8	0.124	40	0.111	36		11
1-Butanol	71-36-3	74	22.3	0.019	5.6	0.017	5.2	J	8
Benzene	71-43-2	78	22.8	0.001	0.40	0.006	1.7	J	
Pyridine	110-86-1	79	26.5	0.021	6.0	0.022	6.2	J	4
Pentanenitrile	110-59-8	83	27.4	0.013	3.5	0.014	3.7		5
Toluene	108-88-3	92	28.5	0.003	0.63	0.004	0.90	J	35
Hexanenitrile	628-73-9	97	32.8	0.010	2.4	0.011	2.5	J	6
Undecane	1120-21-4	156	44.4	0.009	1.3	0.011	1.5	J	16
Dodecane	112-40-3	170	48.4	0.015	2.0	0.011	1.4	J	36
Tridecane	629-50-5	184	52.2	0.030	3.6	0.016	2.0	J	58
Tetradecane	629-59-4	198	55.7	0.008	0.93	0.013	1.5	U	

Data Quality Flags

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

(a) Detected target analytes.

(b) WHC sample identification number.

(c) Relative percent differences (RPDs) based on mg/m³ values.

Table 3.9 Organic Compound Concentrations in Triple Sorbent Trap Blank Samples Associated with the Sampling of Tank C-107 on 9/5/96

Target Analytes ^(a)	CAS	MW	Ret Time	S6091-A17.1205 ^(b) VSS FB #1		S6091-A18.1206 ^(b) VSS FB #2		S6091-A19.1207 ^(b) VSS TB #		S6091-A20.1208 ^(b) VSS TB #2					
				(mg/m ³)	(ppbv)	Flag	(mg/m ³)	(ppbv)	Flag	(mg/m ³)	(ppbv)	Flag	(mg/m ³)	(ppbv)	Flag
Acetone	67-64-1	58	13.6	0.007	2.8	U	0.008	3.0	J	0.007	2.8	U	0.008	3.2	J
Methylene Chloride	75-09-2	85	15.7	0.054	14	J,B	0.031	8.3	J,B	0.035	9.3	J,B	0.038	10	J,B
Tetradecane	629-59-4	198	55.7	0.013	1.5	U	0.019	2.1	J	0.013	1.5	U	0.013	1.5	U

Data Quality Flags

- B Compound found in associated laboratory blank.
 - J Target compound detected above the IDL but below the EQL.
 - U Target compound not detected at or above the IDL.
- (a) Detected target analytes.
 (b) WHC sample identification number.

Table 3.10. Flammability Data for Tank C-107 Sampled on 9/5/96

Analyte	CAS #	Lower Flammability Limit(LFL)	Average Measured Concentrations	% of LFL ^(a)
Ammonia (ppm)	7664-41-7	150000	63	0.042
Carbon Monoxide (ppm)	630-08-0	125000	<17	<0.014
Hydrogen (ppm)	1333-74-0	40000	562	1.405
Methane (ppm)	74-82-8	50000	<25	<0.050
TNMOC (mg/m ³)		42000	3.44	0.008
Total				<1.519

(a) Less than values are calculated using the average concentration less than values. These values are summed to determine the total LFL.

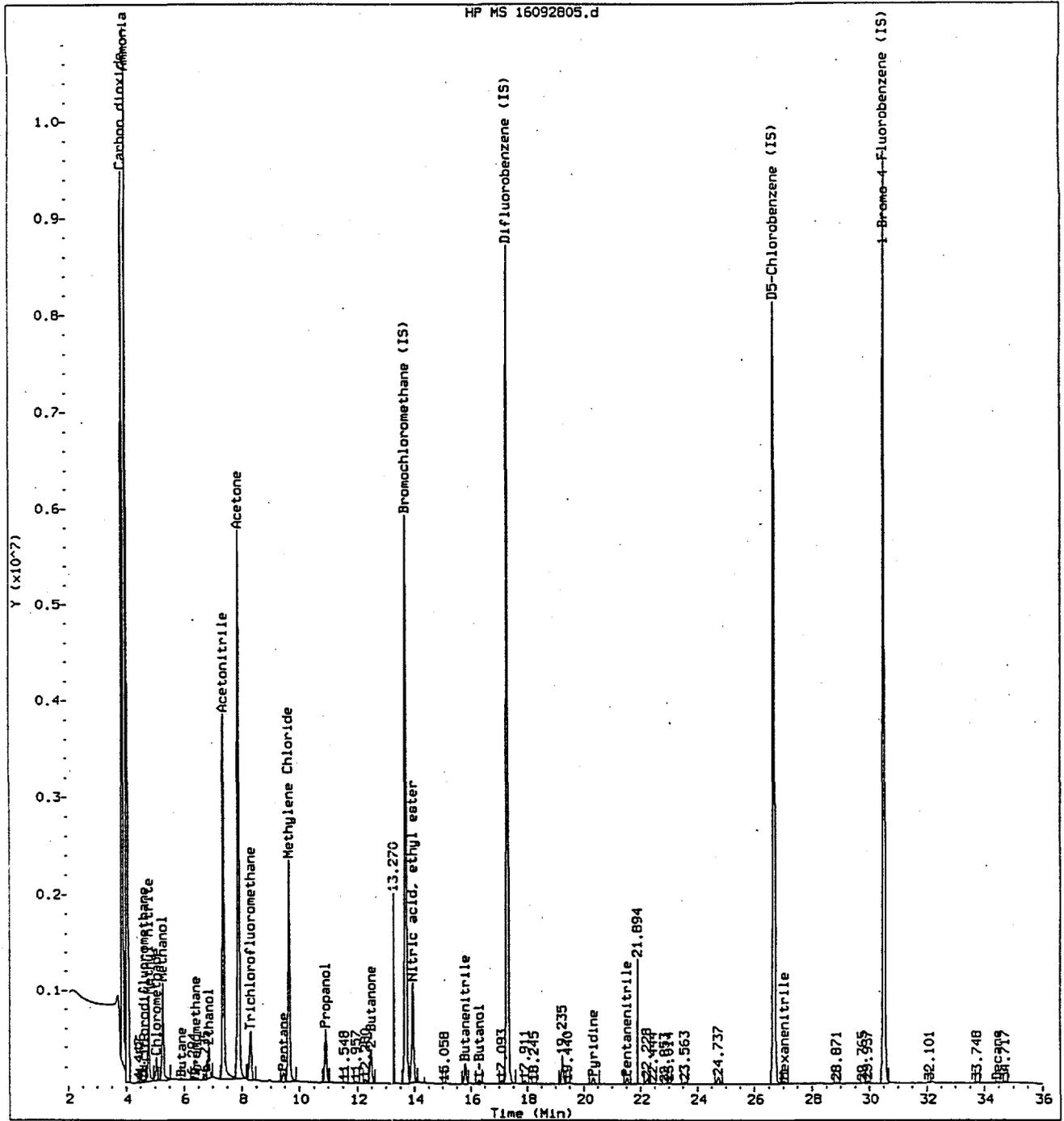


Figure 3.1a Typical Total Ion Chromatogram (2 - 36 min) for SUMMA™ Canister Samples from Tank C-107 Sampled on 9/5/96

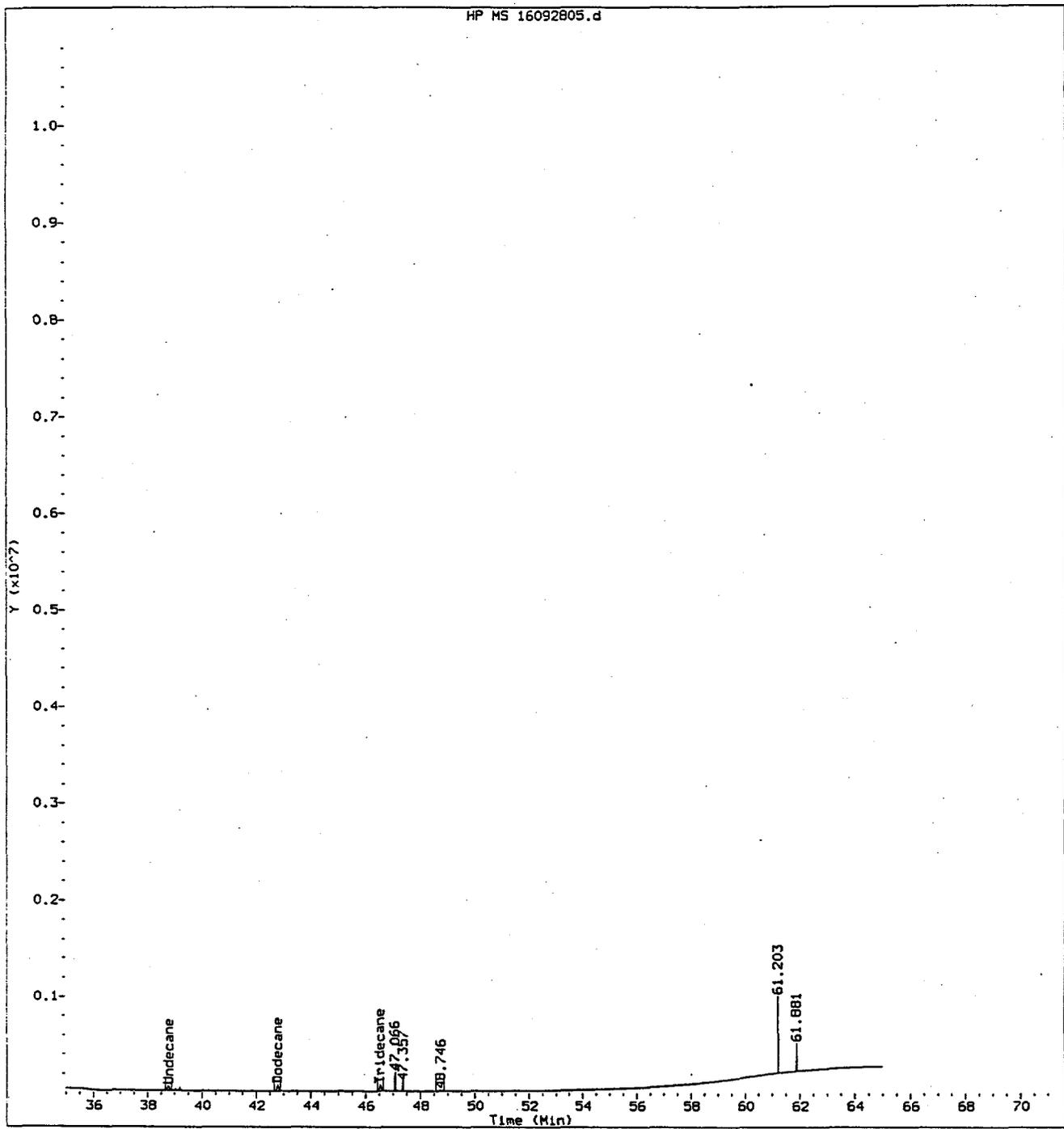


Figure 3.1b Typical Total Ion Chromatogram (36 - 70 min) for SUMMA™ Canister Samples from Tank C-107 Sampled on 9/5/96

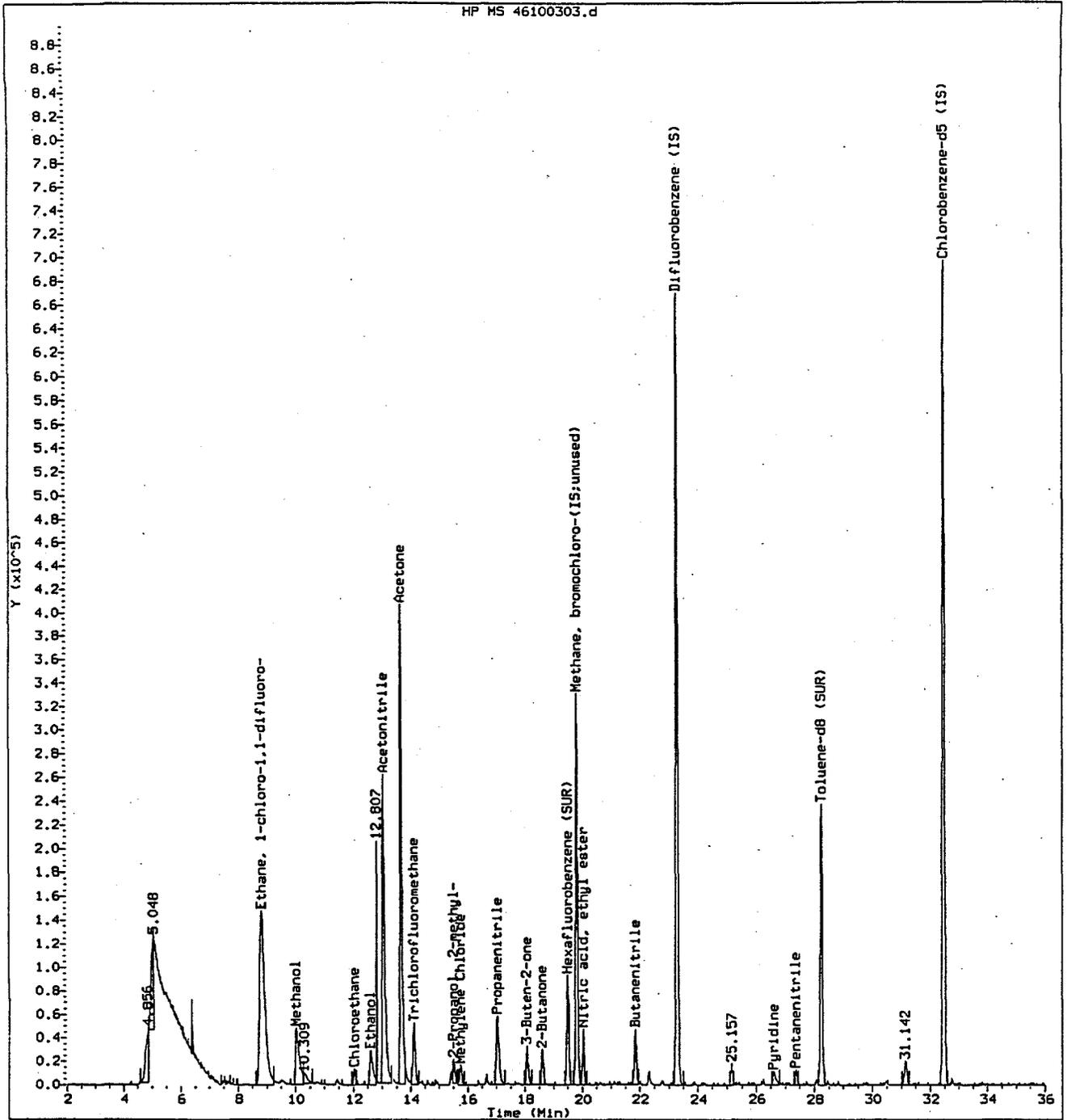


Figure 3.2a Typical Total Ion Chromatogram (2 - 36 min) for Triple Sorbent Trap Samples from Tank C-107 Sampled on 9/5/96

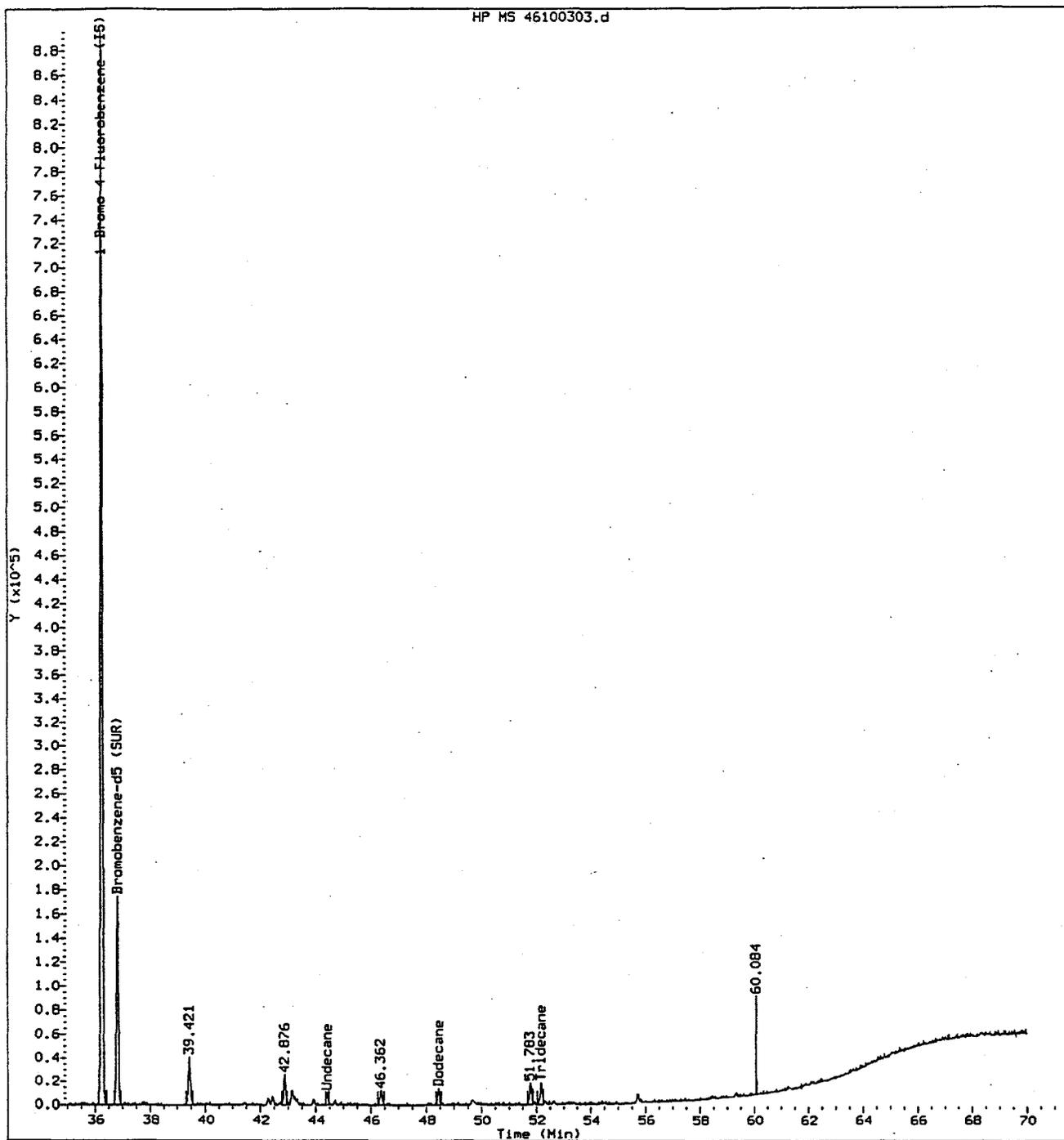


Figure 3.2b Typical Total Ion Chromatogram (36 - 70 min) for Triple Sorbent Trap Samples from Tank C-107 Sampled on 9/5/96

4.0 Conclusions

The concentrations of inorganic and organic analytes were determined from samples of the headspace of Tank C-107 on September 5, 1996 (Sample Job S6091). The vapor concentrations were based either on whole-volume samples (SUMMA™ canisters) or on sorbent traps exposed to sample flow. In the case of the canisters, the concentrations were based on analytical results and the tracking of dilution/concentration of sample volumes obtained directly from the canisters. In the case of the sorbent traps, concentrations were based on analytical results and sample volumes reported by WHC. Known sampling and analytical variances from established quality assurance requirements, where significant, were documented in this report, as required by the SAP (Buckley 1996). No immediate notifications were provided because analytical results indicated no specific analytes exceeded the notification levels; notification levels and notification procedures are described in the SAP (Buckley 1996):

5.0 References

Buckley, L. L. 1996. *Vapor Sampling and Analysis Plan*. WHC-SD-WM-TP-335, Rev. 2C, Westinghouse Hanford Company, Richland, Washington.

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Pacific Northwest National Laboratory. *Analytical Laboratory Procedure Compendium*. Procedure PNL-ALO-271. PNL-MA-599, Pacific Northwest National Laboratory, Richland, Washington.

Pacific Northwest National Laboratory. *Quality Assurance Plan for Activities Conducted by the Pacific Northwest National Laboratory Vapor Analytical Laboratory (VAL) and the Pacific Northwest National Laboratory Tank Vapor Characterization Project*. ETD-002, Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

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Westinghouse Hanford Company (WHC). 1994. *Quality Assurance Project Plan for Tank Vapor Characterization*. WHC-SD-WM-QAPP-013, Rev. 2, Westinghouse Hanford Company, Richland, Washington.

Appendix A

Chain-of-Custody Sample Control Forms

Custody Form Initiator J. A. Edwards - PNNL

Telephone (509) 373-0141
Page 85-3009 / FAX 376-0418

Company Contact R. D. Mahon - WHC

Telephone (509) 373-7437
Page 85-9656 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm
241-C-107 Tank Vapor Sample SAF S6091
Temporal VSS

Collection date 9-5-96
Preparation date 08-20-96

Ice Chest No.

Field Logbook No. WHC-N 697-10

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6091 - A07 . 94R Collect NH₃/NO_x/H₂O Sorbent Trap
S6091 - A08 . 95R Collect NH₃/NO_x/H₂O Sorbent Trap
S6091 - A09 . 96R Collect NH₃/NO_x/H₂O Sorbent Trap
S6091 - A10 . 97R Collect NH₃/NO_x/H₂O Sorbent Trap

S6091 - A15 . 98R Open, close and store NH₃/NO_x/H₂O field blank #1
S6091 - A16 . 99R Open, close and store NH₃/NO_x/H₂O field blank #2

[] Field Transfer of Custody		[X] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time
G W Dennis <i>G.W. Dennis</i>	08-21-96	1100	J A Edwards <i>J.A. Edwards</i>	08-21-96	1100
J A Edwards <i>J.A. Edwards</i>	09-03-96	1420	ET MAST <i>ET MAST</i>	09-03-96	1420
ET MAST <i>ET MAST</i>	09-05-96	0900	GS CAPRIO <i>GS CAPRIO</i>	09-05-96	0900
GS CAPRIO <i>GS CAPRIO</i>	09-16-96	0830	Rick Mahon <i>Rick Mahon</i>	09-16-96	0830
Rick Mahon <i>Rick Mahon</i>	09-16-96	1430	J A Edwards <i>J.A. Edwards</i>	09-16-96	1430
J A Edwards <i>J.A. Edwards</i>	09-17-96	0900	G.W. Dennis <i>G.W. Dennis</i>	09-17-96	0900

Final Sample Disposition

Comments:

- PNNL (only) Checklist**
- Media labeled and checked?
 - Letter of instruction?
 - Media in good condition?
 - COC info/signatures complete?
 - Rad release stickers on samples?
 - Activity report from 222S?
 - RSR/release? (a ≤100/B ≤400 pCi/g)
 - COC copy for LRB, RIDS filed?

- Pick-up / Delivery**
- Y
 - N
 - Y
 - N
 - Y
 - N
 - Y
 - N

Comments:

S6091-A07. 94R } Condensate in the form of tiny water
S6091-A08. 95R } droplets were noticed in the first
S6091-A09. 96R } sorbent tube (NH₃) of each inorgan
S6091-A10. 97R } train. The condensate started
about 1/4 inch inside the glass and
collected on the glass up to the 1st
packing in the tube. RDM 16SEP96

POC *(Signature)* POC *(Signature)*

(WHC-SD-WM-TP-335, REV. 2, Table 2b)

(Revised 05/30/96 PNNL)

Custody Form Initiator J. A. Edwards - PNNL

Telephone (509) 373-0141
Page 85-3009 / FAX 376-2329

Company Contact R. D. Mahon - WHC

Telephone (509) 373-2891
Page 85-3656 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm
241-C-107 Tank Vapor Sample SAF S6091
VSS

Collection date 09 - 5 - 96
Preparation date 09 - 03 - 96

Ice Chest No.

Field Logbook No. WHC-21 647-10

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6091 - A01 . 275 Collect Ambient Air Sample SUMMA #1
S6091 - A02 . 283 Collect Ambient Air Sample SUMMA #2 (through tube bundle)

S6091 - A04 . 284 Collect SUMMA #3
S6091 - A05 . 285 Collect SUMMA #4
S6091 - A06 . 287 Collect SUMMA #5

[] Field Transfer of Custody		[X] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time
J A Edwards <i>J A Edwards</i>	09-3-96	1420	ES MAST <i>S/ward</i>	09-3-96	1420
ES MAST <i>E/ward</i>	09-05-96	0900	GS CAPRIO <i>GS Caprio</i>	09-05-96	0900
GS CAPRIO <i>GS Caprio</i>	09-16-96	0830	Rick Mahon <i>Rick Mahon</i>	09-16-96	0830
Rick Mahon <i>Rick Mahon</i>	09-16-96	1435	J A Edwards <i>J A Edwards</i>	09-16-96	1435

Final Sample Disposition

Comments:

PNNL (only) Checklist	Pick-up / Delivery	Comments:
◊ Media labeled and checked?	Y/N	
◊ Letter of instruction?	Y/N	
◊ Media in good condition?	Y/N	
◊ COC info/signatures complete?	Y/N	
◊ Rad release stickers on samples?	Y/N	
◊ Activity report from 222S?	Y/N	
◊ RSR/release? (a ≤100/B ≤400 pCi/g)	Y/N	
◊ COC copy for LRB, RIDS filed?	Y/N	

POC *(Signature)* POC *(Signature)*

(WHC-SD-WM-TP-335, REV. 2, Table 2b)

(Revised 05/30/96 PNNL)

Custody Form Initiator J. A. Edwards - PNL

Telephone (509) 373-0141
Page 85-3009 / P8-08 / FAX 376-0418

Company Contact R. D. Mahon - WHC

Telephone (509) 373-7437
Page 85-9656 / S3-27 / FAX 373-7076

Project Designation/Sampling Locations 200 West Tank Farm
241-C-107 Tank Vapor Sample SAF S6091
(VSS Truck)

Collection date 09 - 5 - 96
Preparation date 09 - 03 - 96

Ice Chest No.

Field Logbook No. WHC- N 647-10

Ertco Hi/Lo thermometer No. PNL-T-003

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

S6091 - A11 . 1201 . PNL Triple Sorbent Trap (TST) Sample # 1
S6091 - A12 . 1202 . PNL TST Sample # 2
S6091 - A13 . 1203 . PNL TST Sample # 3
S6091 - A14 . 1204 . PNL TST Sample # 4

S6091 - A17 . 1205 . Open, close & store TST Field Blank # 1
S6091 - A18 . 1206 . Open, close & store TST Field Blank #2

S6091 - A19 . 1207 . Store TST Trip Blank #1
S6091 - A20 . 1208 . Store TST Trip Blank #2

[] Field Transfer of Custody		[X] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time
JL Julia <i>Janet L Julia</i>	09-03-96	1200	JA Edwards <i>JA Edwards</i>	09-03-96	1200
JA Edwards <i>JA Edwards</i>	09-03-96	1420	ES Masz <i>ES Masz</i>	09-03-96	1420
ES Masz <i>ES Masz</i>	09-05-96	0900	GS Caprio <i>GS Caprio</i>	09-05-96	0900
AD Caprio <i>GS Caprio</i>	09-16-96	0830	Rick Mahon <i>Rick Mahon</i>	09-16-96	0830
Rick Mahon <i>Rick Mahon</i>	09-16-96	1435	JA Edwards <i>JA Edwards</i>	09-16-96	1435
JA Edwards <i>JA Edwards</i>	09-17-96	0930	JL Julia <i>Janet L Julia</i>	09-17-96	0930

Final Sample Disposition

Comments:

- | | |
|--|---------------------------------------|
| PNL (only) Checklist | Pick-up / Delivery |
| Media labeled and checked? | <input checked="" type="checkbox"/> N |
| Letter of instruction? | <input checked="" type="checkbox"/> N |
| Media in good condition? | <input checked="" type="checkbox"/> N |
| COC info/signatures complete? | <input checked="" type="checkbox"/> N |
| Sorbents shipped on ice? (<5°C) | <input checked="" type="checkbox"/> N |
| Hi/Lo thermometer - <u>Keep upright!</u> | <input checked="" type="checkbox"/> N |
| Hi/Lo thermometer | <input checked="" type="checkbox"/> N |
| Rad release stickers on samples? | <input checked="" type="checkbox"/> N |
| Activity report from 222S? | <input checked="" type="checkbox"/> N |
| COC copy for LRB, RIDS filed? | <input checked="" type="checkbox"/> N |

POC *(Signature)* POC *(Signature)*

Comments:

S6091-A11-1201 As the cap was being removed from the upstream side of the TST tube a chip of glass fell off of the end of the TST. RDM 16SEP96

Cooler Temperature Status	
Hi -10 °C / Lo -10 °C (pick up at PNL to WHC)	1
Hi +3 °C / Lo -6 °C (delivery at WHC from PNL)	1
Hi 6 °C / Lo -8 °C (at return to PNL from WHC)	1
Hi 6 °C / Lo +9 °C (at delivery from WHC to PNL)	1

(Revised 06/21/95 PNL)

Appendix B

Listing of All Target Compounds for Organic Compound Analysis

Table B.1 SUMMA™ Analysis Results for All Target Analytes from Tank C-107 Sampled on 9/5/96

Target Analytes	CAS	MW	Ret Time	Ambient Air		Ambient Air through VSS		S6091-A01.275 VSS		S6091-A02.283 VSS		S6091-A04.284 VSS		S6091-A05.285 VSS		S6091-A05.285 VSS RE		S6091-A06.287 VSS			
				(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	Fla	Fla	(mg/m ³)	(ppbv)	Fla	Fla	(mg/m ³)	(ppbv)	Fla	Fla	(mg/m ³)	(ppbv)	Fla	Fla
Dichlorodifluoromethane	75-71-8	120.91	4.571	0.0036	0.66	0.0029	0.54	J	0.0032	0.59	J	0.0031	0.58	J	0.0029	0.53	J	0.0033	0.61	J	
Chloromethane	74-87-3	50.49	4.947	0.0024	1.06	0.0024	1.06	U	0.0086	3.82	J	0.0081	3.59	J	0.0086	3.82	J	0.0077	3.44	J	
1,2-dichloro-1,1,2,2-tetrafluoroethane	76-14-2	170.92	5.163	0.013	1.7	0.013	1.7	U	0.013	1.7	U	0.013	1.7	U	0.013	1.7	U	0.013	1.7	U	
Methanol	67-56-1	32.04	5.238	<0.110	<7.7	Y	<7.7	Y	0.8723	609.85	Y	1.008	704.71	Y	1.0442	730.06	Y	0.9886	691.16	Y	
Vinyl Chloride	75-01-4	62.5	5.443	0.0016	0.57	U	0.0016	0.57	U	0.0016	0.57	U	0.0016	0.57	U	0.0016	0.57	U	0.0016	0.57	U
1,3-Butadiene	106-99-0	54	5.744	<0.083	<3.4	Y	<0.083	<3.4	Y	<0.083	<3.4	Y	<0.083	<3.4	Y	<0.083	<3.4	Y	<0.083	<3.4	Y
Butane	106-97-8	58.12	5.873	0.0024	0.92	U	0.0024	0.92	U	0.0076	2.93	J	0.0071	2.75	J	0.0063	2.44	J	0.007	2.7	J
Bromomethane	74-83-9	94.94	6.347	0.0052	1.23	U	0.0052	1.23	U	0.0215	5.06	J	0.0208	4.9	J	0.0212	4.99	J	0.0194	4.57	J
Chloroethane	75-00-3	64.51	6.702	0.0054	1.88	U	0.0054	1.88	U	0.0054	1.88	U	0.0054	1.88	U	0.0054	1.88	U	0.0054	1.88	U
Ethanol	64-17-5	46.01	6.842	<0.11	<5.3	Y	<0.11	<5.3	Y	0.1209	58.85	Y	0.1294	62.98	Y	0.1235	60.14	Y	0.1248	60.74	Y
Acetonitrile	75-05-8	41.05	7.338	0.004	2.18	U	0.004	2.18	U	1.1516	628.39	Y	1.1991	654.32	Y	1.2102	660.39	Y	1.212	661.37	Y
Acetone	67-64-1	58.08	7.844	0.016	6.15	J	0.0176	6.77	J	1.3685	527.78	Y	1.392	536.84	Y	1.3964	538.56	Y	1.4015	540.51	Y
Trichlorofluoromethane	75-69-4	137.37	8.253	0.0048	0.78	U	0.0048	0.78	U	0.1605	26.18	U	0.164	26.75	U	0.1569	25.59	U	0.1636	26.67	U
Pentane	109-66-0	72.15	8.931	0.0038	1.18	U	0.0038	1.18	U	0.0038	1.18	U	0.0038	1.18	U	0.0038	1.18	U	0.0038	1.18	U
1,1-Dichloroethene	75-35-4	96.94	9.48	0.0038	0.88	U	0.0038	0.88	U	0.0038	0.88	U	0.0038	0.88	U	0.0038	0.88	U	0.0038	0.88	U
Methylene Chloride	75-09-2	84.93	9.663	0.0143	3.77	J	0.0143	3.76	J	0.0154	4.07	J	0.0149	3.94	J	0.0152	4	J	0.0151	3.98	J
1,1,1-Trichloroethane	76-13-1	187.38	10.169	0.004	0.48	U	0.004	0.48	U	0.004	0.48	U	0.004	0.48	U	0.004	0.48	U	0.004	0.48	U
Propanol	71-23-8	60.1	10.891	0.0028	1.04	U	0.0028	1.04	U	0.0306	11.4	U	0.0302	11.25	U	0.0302	11.27	U	0.0292	10.87	U
Propanenitrile	107-12-0	55.08	10.934	0.0038	1.55	U	0.0038	1.55	U	0.2475	100.67	U	0.2578	104.83	U	0.2606	106	U	0.2569	104.48	U
1,1-Dichloroethane	75-34-3	98.96	11.816	0.002	0.45	U	0.002	0.45	U	0.002	0.45	U	0.002	0.45	U	0.002	0.45	U	0.002	0.45	U
2-Butanone	78-93-3	72.11	12.462	0.0095	2.94	J	0.0095	2.94	J	0.0951	29.55	U	0.0899	27.94	U	0.0869	27.01	U	0.0785	24.38	U
cis-1,2-Dichloroethene	156-59-2	96.94	13.345	0.0034	0.79	U	0.0034	0.79	U	0.0034	0.79	U	0.0034	0.79	U	0.0034	0.79	U	0.0034	0.79	U
Hexane	110-54-3	86.18	13.722	0.0032	0.83	U	0.0032	0.83	U	0.0032	0.83	U	0.0032	0.83	U	0.0032	0.83	U	0.0032	0.83	U
Chloroform	67-66-3	119.38	13.884	0.0026	0.49	U	0.0026	0.49	U	0.0026	0.49	U	0.0026	0.49	U	0.0026	0.49	U	0.0026	0.49	U
Tetrahydrofuran	109-99-9	72.11	14.637	0.0016	0.5	U	0.0016	0.5	U	0.0016	0.5	U	0.0016	0.5	U	0.0016	0.5	U	0.0016	0.5	U
1,2-Dichloroethane	107-06-2	98.96	15.305	0.0026	0.59	U	0.0026	0.59	U	0.0026	0.59	U	0.0026	0.59	U	0.0026	0.59	U	0.0026	0.59	U
Butanenitrile	109-74-0	69.11	15.811	0.0034	1.1	U	0.0034	1.1	U	0.1287	41.72	U	0.1373	44.49	U	0.1364	44.21	U	0.1297	42.04	U
1,1,1-Trichloroethane	71-55-6	133.4	15.822	0.0032	0.54	U	0.0032	0.54	U	0.0032	0.54	U	0.0032	0.54	U	0.0032	0.54	U	0.0032	0.54	U
1-Butanol	71-36-3	74.12	16.295	0.0099	2.99	J	0.0042	1.28	J	0.0153	4.64	J	0.0159	4.81	J	0.0151	4.57	J	0.0128	3.87	J
Benzene	71-43-2	78.11	16.715	0.0016	0.46	U	0.0016	0.46	U	0.0016	0.46	U	0.0016	0.46	U	0.0016	0.46	U	0.0016	0.46	U
Carbon Tetrachloride	56-23-5	153.82	17.006	0.0034	0.5	U	0.0034	0.5	U	0.0034	0.5	U	0.0034	0.5	U	0.0034	0.5	U	0.0034	0.5	U
Cyclohexane	110-82-7	84.16	17.286	0.0036	0.96	U	0.0036	0.96	U	0.0036	0.96	U	0.0036	0.96	U	0.0036	0.96	U	0.0036	0.96	U
1,2-Dichloropropane	78-87-5	112.99	18.298	0.003	0.59	U	0.003	0.59	U	0.003	0.59	U	0.003	0.59	U	0.003	0.59	U	0.003	0.59	U
Trichloroethene	79-01-6	131.39	18.739	0.0024	0.41	U	0.0024	0.41	U	0.0024	0.41	U	0.0024	0.41	U	0.0024	0.41	U	0.0024	0.41	U
Heptane	142-82-5	100.2	19.245	0.0018	0.4	U	0.0018	0.4	U	0.0018	0.4	U	0.0018	0.4	U	0.0018	0.4	U	0.0018	0.4	U
cis-1,3-Dichloropropene	10061-01-5	110.97	20.43	0.0024	0.48	U	0.0024	0.48	U	0.0024	0.48	U	0.0024	0.48	U	0.0024	0.48	U	0.0024	0.48	U
4-Methyl-2-Pentanone	108-10-1	100.16	20.462	0.0034	0.76	U	0.0034	0.76	U	0.0034	0.76	U	0.0034	0.76	U	0.0034	0.76	U	0.0034	0.76	U
Pyridine	110-86-1	79.1	20.613	0.0036	0.96	U	0.0036	0.96	U	0.0036	0.96	U	0.0036	0.96	U	0.0036	0.96	U	0.0036	0.96	U
trans-1,3-Dichloropropene	10061-02-6	110.97	21.506	0.0018	0.36	U	0.0018	0.36	U	0.0018	0.36	U	0.0018	0.36	U	0.0018	0.36	U	0.0018	0.36	U
Pentanenitrile	110-59-8	83.13	21.592	0.0042	1.13	U	0.0042	1.13	U	0.0042	1.13	U	0.0042	1.13	U	0.0042	1.13	U	0.0042	1.13	U
1,1,2-Trichloroethane	79-00-5	133.4	21.959	0.0028	0.47	U	0.0028	0.47	U	0.0028	0.47	U	0.0028	0.47	U	0.0028	0.47	U	0.0028	0.47	U
Toluene	108-88-3	92.14	22.605	0.002	0.49	U	0.002	0.49	U	0.002	0.49	U	0.002	0.49	U	0.002	0.49	U	0.002	0.49	U

Table B.1 (Cont'd) SUMMA™ Analysis Results for All Target Analytes from Tank C-107 Sampled on 9/5/96

Target Analytes	CAS	MW	Ret Time	Ambient Air		Ambient Air through VSS		S6091-A01.275 VSS		S6091-A02.283 VSS		S6091-A04.284 VSS		S6091-A05.285 VSS		S6091-A06.287 VSS		
				(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	(mg/m ³)
1,2-Dibromoethane	106-93-4	187.86	24.144	0.0052	0.62	U	0.0052	0.62	U	0.0052	0.62	U	0.0052	0.62	U	0.0052	0.62	U
Octane	111-65-9	114.23	24.682	0.0024	0.47	U	0.0024	0.47	U	0.0024	0.47	U	0.0024	0.47	U	0.0024	0.47	U
Tetrachloroethylene	127-18-4	165.83	25.156	0.0042	0.57	U	0.0042	0.57	U	0.0042	0.57	U	0.0042	0.57	U	0.0042	0.57	U
Chlorobenzene	108-90-7	112.56	26.782	0.0026	0.52	U	0.0026	0.52	U	0.0026	0.52	U	0.0026	0.52	U	0.0026	0.52	U
Hexanenitrile	628-73-9	97.13	27.062	0.0052	1.2	U	0.0102	2.36	J	0.0107	2.47	J	0.0086	1.98	J	0.0099	2.28	J
Ethylbenzene	100-41-4	106.17	27.654	0.0028	0.59	U	0.0028	0.59	U	0.0028	0.59	U	0.0028	0.59	U	0.0028	0.59	U
p/m-Xylene	106-42-3	106.17	28.117	0.013	2.74	U	0.013	2.74	U	0.013	2.74	U	0.013	2.74	U	0.013	2.74	U
Cyclohexanone	108-94-1	98.14	28.515	0.0078	1.77	J	0.0058	1.32	U	0.0058	1.32	U	0.0058	1.32	U	0.0058	1.32	U
Styrene	100-42-5	104.15	29.011	0.0022	0.47	U	0.0022	0.47	U	0.0022	0.47	U	0.0022	0.47	U	0.0022	0.47	U
1,1,2,2-Tetrachloroethane	79-34-5	167.85	29.247	0.0042	0.56	U	0.0042	0.56	U	0.0042	0.56	U	0.0042	0.56	U	0.0042	0.56	U
o-Xylene	95-47-6	106.17	29.323	0.0028	0.59	U	0.0028	0.59	U	0.0028	0.59	U	0.0028	0.59	U	0.0028	0.59	U
Nonane	111-84-2	128.26	29.753	0.0018	0.31	U	0.0018	0.31	U	0.0018	0.31	U	0.0018	0.31	U	0.0018	0.31	U
1-Ethyl-2-methylbenzene	611-14-3	120.19	32.94	0.0019	0.35	J	0.0018	0.34	U	0.0018	0.34	U	0.0018	0.34	U	0.0018	0.34	U
1,3,5-Trimethylbenzene	108-67-8	120.19	32.714	0.0019	0.36	J	0.0018	0.34	U	0.0018	0.34	U	0.0018	0.34	U	0.0018	0.34	U
1,2,4-Trimethylbenzene	95-63-6	120.19	34.211	0.0029	0.55	J	0.0026	0.48	U	0.0026	0.48	U	0.0026	0.48	U	0.0026	0.48	U
Decane	124-18-5	142.28	34.448	0.003	0.47	U	0.003	0.47	U	0.0044	0.69	J	0.0036	0.56	J	0.0032	0.51	J
1,3-Dichlorobenzene	541-73-1	147	34.706	0.0038	0.58	J	0.0018	0.27	U	0.0018	0.27	U	0.0018	0.27	U	0.0018	0.27	U
1,4-Dichlorobenzene	106-46-7	147	34.921	0.0041	0.62	J	0.0018	0.27	U	0.0018	0.27	U	0.0018	0.27	U	0.0018	0.27	U
1,2-Dichlorobenzene	95-50-1	147	36.052	0.0076	1.16	J	0.0018	0.27	U	0.0018	0.27	U	0.0018	0.27	U	0.0018	0.27	U
Undecane	1120-21-4	156.31	38.765	0.0026	0.37	U	0.0026	0.37	U	0.0117	1.68	J	0.0028	0.35	U	0.0028	0.35	U
1,2,4-Trichlorobenzene	120-82-1	181.45	42.275	0.0215	2.65	J	0.0028	0.35	U	0.0028	0.35	U	0.0028	0.35	U	0.0028	0.35	U
Dodecane	112-40-3	170.34	42.77	0.0098	1.29	J,B	0.0098	1.29	U	0.0332	4.37	J	0.0276	3.63	J	0.0153	2.01	J
Hexachloro-1,3-butadiene	87-68-3	260.76	44.094	0.0115	0.99	J	0.0046	0.4	U	0.0046	0.4	U	0.0046	0.4	U	0.0046	0.4	U
Tridecane	629-50-5	184.36	46.517	0.006	0.73	U	0.006	0.73	U	0.1054	12.8	J	0.0706	8.58	U	0.0254	3.08	J
Tetradecane	629-59-4	198.34	50.016	0.0042	0.47	U	0.0042	0.47	U	0.0239	2.7	J	0.0042	0.47	U	0.0042	0.47	U

B Compound found in associated laboratory blank.

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Table B.2 Triple Sorbent Trap Analysis Results for All Target Analytes from Tank C-107 Sampled on 9/5/96

NOTE: All data in this table is reported at 21°C and 760 mm Hg pressure. To correct to 0°C and 760 mm Hg pressure multiply by 1.077.

Target Analytes	CAS	MW	Ret Time	S6091-A12.1202 VSS (mg/m ³)	Flag	S6091-A13.1203 VSS (ppbv)	Flag	S6091-A13.1203 VSS REP (mg/m ³)	Flag	S6091-A14.1204 VSS (ppbv)	Flag
Dichlorodifluoromethane	75-71-8	120.91	7.822	0.0043	U	0.79	U	0.0043	U	0.79	U
Chloromethane	74-87-3	50.49	8.789	0.0032	U	1.4	U	0.0032	U	1.4	U
1,2-dichloro 1,1,2,2-tetrafluoroeth	76-14-2	170.92	9.522	0.0032	U	0.41	U	0.0032	U	0.41	U
Methanol	67-56-1	32.04	10.033	1.5765	Y	1164.28	Y	1.4965	Y	1139.63	Y
Vinyl Chloride	75-01-4	62.5	9.916	0.003	U	1.08	U	0.003	U	1.08	U
1,3-Butadiene	106-99-0	54	10.543	<0.208	Y	<86	Y	<0.208	Y	<86	Y
Butane	106-97-8	58.14	10.755	0.0036	U	1.39	U	0.0036	U	1.39	U
Chloroethane	75-00-3	64.51	12.052	0.0759	U	26.35	U	0.0419	U	30.71	U
Ethanol	64-17-5	46.07	12.53	0.2997	Y	145.71	Y	<0.275	Y	148.21	Y
Acetonitrile	75-05-8	41.05	13.009	0.97	U	529.33	U	1.0725	U	585.24	U
Acetone	67-64-1	58.08	13.646	0.9732	U	375.34	U	1.0404	U	401.24	U
Trichlorofluoromethane	75-69-4	137.37	14.114	0.181	U	29.52	U	0.1762	U	28.74	U
Pentane	109-66-0	72	14.901	0.0055	J	1.72	J	0.0095	J	1.826	J
1,1-Dichloroethene	75-35-4	96.94	15.496	0.0021	U	0.47	U	0.0021	U	0.5102	U
Methylene Chloride	75-09-2	84.93	15.74	0.0466	J	10.93	J	0.0451	J	0.0021	U
1,1,2-Trichloroethane	76-13-1	187.38	16.282	0.003	U	0.36	U	0.003	U	0.0407	U
Propanenitrile	107-12-0	55.08	17.005	0.2457	U	99.9	U	0.2702	U	0.003	U
Propanol	71-23-8	60.1	17.037	0.0775	U	28.9	U	0.0629	U	0.2475	U
1,1-Dichloroethane	75-34-3	98.96	17.951	0.0014	U	0.32	U	0.0014	U	0.0749	U
2-Butanone	78-93-3	72.11	18.578	0.0701	U	21.76	U	0.0816	U	0.0014	U
cis-1,2-Dichloroethene	156-59-2	96.94	19.482	0.0025	U	0.58	U	0.0025	U	0.0754	U
Hexane	110-54-3	86.18	19.843	0.0044	J	1.15	J	0.0043	J	0.0025	U
Chloroform	67-66-3	119.38	20.013	0.0029	U	0.53	U	0.0029	U	0.0039	U
Tetrahydrofuran	109-99-9	72.11	20.725	0.0035	U	1.07	U	0.0035	U	0.0029	U
1,2-Dichloroethane	107-06-2	98.96	21.384	0.0011	U	0.25	U	0.0011	U	1.07	U
Butanenitrile	109-74-0	69.11	21.831	0.1133	U	36.71	U	0.0011	U	0.0011	U
1,1,1-Trichloroethane	71-55-6	133.4	21.894	0.0027	U	0.44	U	0.0027	U	0.1125	U
1-Butanol	71-36-3	74	22.277	0.0259	J	7.84	J	0.017	J	36	J
Benzene	71-43-2	78.11	22.755	0.0057	J	1.65	J	0.0059	J	0.44	J
Carbon Tetrachloride	56-23-5	153.82	23.053	0.0014	U	0.2	U	0.0014	U	5.16	J
Cyclohexane	110-82-7	84.16	23.308	0.0041	U	1.09	U	0.0041	U	1.68	J
1,2-Dichloropropane	78-87-5	112.99	24.286	0.0019	U	0.38	U	0.0019	U	0.0014	U
Trichloroethene	79-01-6	131.39	24.711	0.0036	U	0.61	U	0.0036	U	0.0041	U
Heptane	142-82-5	100.2	25.157	0.0015	U	0.35	U	0.0015	U	0.0019	U
4-Methyl-2-Pentanone	108-10-1	100.16	26.316	0.0021	U	0.46	U	0.0021	U	0.0019	U
cis-1,3-Dichloropropene	10061-01-5	110.97	26.348	0.0013	U	0.25	U	0.0013	U	0.0015	U

Table B.2 (Cont'd) Triple Sorbent Trap Analysis Results for All Target Analytes from Tank C-107 Sampled on 9/5/96
 NOTE: All data in this table is reported at 21°C and 760 mm Hg pressure. To correct to 0°C and 760 mm Hg pressure multiply by 1.077.

Target Analytes	CAS	MW	Ret Time	S6091-A12.1202 VSS (mg/m ³)	S6091-A12.1202 VSS (ppbv)	Flag	S6091-A13.1203 VSS (mg/m ³)	S6091-A13.1203 VSS (ppbv)	Flag	S6091-A13.1203 VSS REP (mg/m ³)	S6091-A13.1203 VSS REP (ppbv)	Flag	S6091-A14.1204 VSS (mg/m ³)	S6091-A14.1204 VSS (ppbv)	Flag
Pyridine	110-86-1	79.1	26.518	0.0314	8.88	J	0.0211	5.98	J	0.022	6.23	J	0.0219	6.19	J
trans-1,3-Dichloropropene	10061-02-6	110.97	27.368	0.0027	0.53	U	0.0027	0.53	U	0.0027	0.53	U	0.0027	0.53	U
Pentanenitrile	110-59-8	83.15	27.368	0.0142	3.82	U	0.0129	3.48	U	0.0136	3.67	U	0.0138	3.71	U
1,1,2-Trichloroethane	79-00-5	133.4	27.815	0.0015	0.26	U	0.0015	0.26	U	0.0015	0.26	U	0.0015	0.26	U
Toluene	108-88-3	92.14	28.452	0.0023	0.55	J	0.0026	0.63	J	0.0037	0.9	J	0.0016	0.38	J
1,2-Dibromoethane	106-93-4	187.86	29.983	0.0024	0.28	U	0.0024	0.28	U	0.0024	0.28	U	0.0024	0.28	U
Octane	111-65-9	114	30.429	0.0027	0.52	U	0.0027	0.52	U	0.0027	0.52	U	0.0027	0.52	U
Tetrachloroethylene	127-18-4	165.83	30.982	0.0025	0.34	U	0.0025	0.34	U	0.0025	0.34	U	0.0025	0.34	U
Chlorobenzene	108-90-7	112.56	32.566	0.0011	0.22	U	0.0011	0.22	U	0.0011	0.22	U	0.0011	0.22	U
Hexanenitrile	628-73-9	97	32.757	0.0112	2.58	J	0.0103	2.37	J	0.0109	2.51	J	0.0114	2.63	J
Ethylbenzene	100-41-4	106.17	33.416	0.0014	0.3	U	0.0014	0.3	U	0.0014	0.3	U	0.0014	0.3	U
p/m-Xylene	106-42-3	106.17	33.841	0.003	0.63	U	0.003	0.63	U	0.003	0.63	U	0.003	0.63	U
Cyclohexanone	108-94-1	98.14	34.288	0.0125	2.85	U	0.0125	2.85	U	0.0125	2.85	U	0.0125	2.85	U
Styrene	100-42-5	104.15	34.755	0.0015	0.33	U	0.0015	0.33	U	0.0015	0.33	U	0.0015	0.33	U
1,1,2,2-Tetrachloroethane	79-34-5	167.85	34.989	0.0058	0.77	U	0.0058	0.77	U	0.0058	0.77	U	0.0058	0.77	U
o-Xylene	95-47-6	106.17	35.053	0.0019	0.4	U	0.0019	0.4	U	0.0019	0.4	U	0.0019	0.4	U
Nonane	111-84-2	128	35.414	0.0013	0.22	U	0.0013	0.22	U	0.0013	0.22	U	0.0013	0.22	U
1-Ethyl-2-methyl benzene	611-14-3	120.19	38.412	0.0024	0.44	U	0.0024	0.44	U	0.0024	0.44	U	0.0024	0.44	U
1,3,5-Trimethylbenzene	108-67-8	120.19	38.624	0.0024	0.44	U	0.0024	0.44	U	0.0024	0.44	U	0.0024	0.44	U
1,2,4-Trimethylbenzene	95-63-6	120.19	39.9	0.0025	0.47	U	0.0025	0.47	U	0.0025	0.47	U	0.0025	0.47	U
Decane	124-18-5	142.28	40.08	0.003	0.47	U	0.003	0.47	U	0.003	0.47	U	0.003	0.47	U
1,3-Dichlorobenzene	541-73-1	147	40.442	0.0032	0.48	U	0.0032	0.48	U	0.0032	0.48	U	0.0032	0.48	U
1,4-Dichlorobenzene	106-46-7	147	40.644	0.0029	0.43	U	0.0029	0.43	U	0.0029	0.43	U	0.0029	0.43	U
1,2-Dichlorobenzene	95-50-1	147	41.781	0.0047	0.72	U	0.0047	0.72	U	0.0047	0.72	U	0.0047	0.72	U
Undecane	1120-21-4	156	44.406	0.0092	1.33	J	0.009	1.3	J	0.0106	1.52	J	0.01	1.44	J
1,2,4-Trichlorobenzene	120-82-1	181.45	48.02	0.0155	1.91	U	0.0155	1.91	U	0.0155	1.91	U	0.0155	1.91	U
Dodecane	112-40-3	170	48.434	0.0146	1.92	J	0.0151	1.99	J	0.0105	1.38	J	0.0174	2.29	J
Hexachloro-1,3-butadiene	87-68-3	260.76	49.838	0.0205	1.76	U	0.0205	1.76	U	0.0205	1.76	U	0.0205	1.76	U
Tridecane	629-50-5	184	52.186	0.0213	2.6	J	0.0297	3.62	J	0.0163	1.98	J	0.0326	3.97	J
Tetradecane	629-59-4	198	55.705	0.0168	1.9	J	0.0082	0.93	J	0.013	1.47	U	0.0064	0.73	J
Tributyl Phosphate	126-73-8	266	63.1	<0.0099	<0.83	Z	<0.0099	<0.83	Z	<0.0099	<0.83	Z	<0.0099	<0.83	Z

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section 3.4.2.1 for more information.

Table B.3 Triple Sorbent Trap Analysis Results for All Target Analytes from Blank Samples Associated with the Sampling of Tank C-107 on 9/5/96
 NOTE: All data in this table is reported at 21°C and 760 mm Hg pressure. To correct to 0°C and 760 mm Hg pressure multiply by 1.077.

Target Analytes	CAS	MW	Ret Time	Field Blank #1		Field Blank #2		S6091-A17.1205 VSS		S6091-A18.1206 VSS		S6091-A19.1207 VSS		S6091-A20.1208 VSS	
				(mg/m ³)	(ppbv)	(mg/m ³)	(ppbv)	Flag	Flag	Flag	Flag	Flag	Flag	Flag	Flag
Dichlorodifluoromethane	75-71-8	120.91	7.822	0.0043	0.79	0.0043	0.79	U	U	0.0043	0.79	U	U	0.0043	0.79
Chloromethane	74-87-3	50.49	8.789	0.0032	1.4	0.0032	1.4	U	U	0.0032	1.4	U	U	0.0032	1.4
1,2-dichloro-1,1,2,2-tetrafluoroethane	76-14-2	170.92	9.522	0.0032	0.41	0.0032	0.41	U	U	0.0032	0.41	U	U	0.0032	0.41
Methanol	67-56-1	32.04	10.033	<0.274	<192	<0.274	<192	Y	Y	<0.274	<192	Y	Y	<0.274	<192
Vinyl Chloride	75-01-4	62.5	9.916	0.003	1.08	0.003	1.08	U	U	0.003	1.08	U	U	0.003	1.08
1,3-Butadiene	106-99-0	54	10.543	<0.208	<86	<0.208	<86	Y	Y	<0.208	<86	Y	Y	<0.208	<86
Butane	106-97-8	58.14	10.755	0.0036	1.39	0.0036	1.39	U	U	0.0036	1.39	U	U	0.0036	1.39
Chloroethane	75-00-3	64.51	12.052	0.0041	1.42	0.0041	1.42	U	U	0.0041	1.42	U	U	0.0041	1.42
Ethanol	64-17-5	46.07	12.53	<0.275	<133	<0.275	<133	Y	Y	<0.275	<133	Y	Y	<0.275	<133
Acetonitrile	75-05-8	41.05	13.009	0.0033	1.8	0.0033	1.8	U	U	0.0033	1.8	U	U	0.0033	1.8
Acetone	67-64-1	58.08	13.646	0.0072	2.78	0.0072	2.78	U	U	0.0072	2.78	U	U	0.0072	2.78
Trichlorofluoromethane	75-69-4	137.37	14.114	0.0044	0.72	0.0044	0.72	U	U	0.0044	0.72	U	U	0.0044	0.72
Pentane	109-66-0	72	14.901	0.0029	0.89	0.0029	0.89	U	U	0.0029	0.89	U	U	0.0029	0.89
1,1-Dichloroethene	75-35-4	96.94	15.496	0.0021	0.47	0.0021	0.47	U	U	0.0021	0.47	U	U	0.0021	0.47
Methylene Chloride	75-09-2	84.93	15.74	0.0543	14.33	0.0314	8.29	J	J	0.0352	9.28	J	J	0.0383	10.09
1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1	187.38	16.282	0.003	0.36	0.003	0.36	U	U	0.003	0.36	U	U	0.003	0.36
Propanenitrile	107-12-0	55.08	17.005	0.0019	0.77	0.0019	0.77	U	U	0.0019	0.77	U	U	0.0019	0.77
Propanol	71-23-8	60.1	17.037	0.0053	1.99	0.0053	1.99	U	U	0.0053	1.99	U	U	0.0053	1.99
1,1-Dichloroethane	75-34-3	98.96	17.951	0.0014	0.32	0.0014	0.32	U	U	0.0014	0.32	U	U	0.0014	0.32
2-Butanone	78-93-3	72.11	18.578	0.0022	0.68	0.0022	0.68	U	U	0.0022	0.68	U	U	0.0022	0.68
cis-1,2-Dichloroethene	156-59-2	96.94	19.482	0.0025	0.58	0.0025	0.58	U	U	0.0025	0.58	U	U	0.0025	0.58
Hexane	110-54-3	86.18	19.843	0.0013	0.32	0.0013	0.32	U	U	0.0013	0.32	U	U	0.0013	0.32
Chloroform	67-66-3	119.38	20.013	0.0029	0.53	0.0029	0.53	U	U	0.0029	0.53	U	U	0.0029	0.53
Tetrahydrofuran	109-99-9	72.11	20.725	0.0035	1.07	0.0035	1.07	U	U	0.0035	1.07	U	U	0.0035	1.07
1,2-Dichloroethane	107-06-2	98.96	21.384	0.0011	0.25	0.0011	0.25	U	U	0.0011	0.25	U	U	0.0011	0.25
Butanenitrile	109-74-0	69.11	21.831	0.0014	0.45	0.0014	0.45	U	U	0.0014	0.45	U	U	0.0014	0.45
1,1,1-Trichloroethane	71-55-6	133.4	21.894	0.0027	0.44	0.0027	0.44	U	U	0.0027	0.44	U	U	0.0027	0.44
1-Butanol	71-36-3	74	22.277	0.0075	2.29	0.0075	2.29	U	U	0.0075	2.29	U	U	0.0075	2.29
Benzene	71-43-2	78.11	22.755	0.0014	0.4	0.0014	0.4	U	U	0.0014	0.4	U	U	0.0014	0.4
Carbon Tetrachloride	56-23-5	153.82	23.053	0.0014	0.2	0.0014	0.2	U	U	0.0014	0.2	U	U	0.0014	0.2
Cyclohexane	110-82-7	84.16	23.308	0.0041	1.09	0.0041	1.09	U	U	0.0041	1.09	U	U	0.0041	1.09
1,2-Dichloropropane	78-87-5	112.99	24.286	0.0019	0.38	0.0019	0.38	U	U	0.0019	0.38	U	U	0.0019	0.38
Trichloroethene	79-01-6	131.39	24.711	0.0036	0.61	0.0036	0.61	U	U	0.0036	0.61	U	U	0.0036	0.61
Heptane	142-82-5	100.2	25.157	0.0015	0.35	0.0015	0.35	U	U	0.0015	0.35	U	U	0.0015	0.35
4-Methyl-2-Pentanone	108-10-1	100.16	26.316	0.0021	0.46	0.0021	0.46	U	U	0.0021	0.46	U	U	0.0021	0.46
cis-1,3-Dichloropropene	10061-01-5	110.97	26.348	0.0013	0.25	0.0013	0.25	U	U	0.0013	0.25	U	U	0.0013	0.25

Table B.3 (Cont'd) Triple Sorbent Trap Analysis Results for All Target Analytes from Blank Samples Associated with the Sampling of Tank C-107 on 9/5/96

NOTE: All data in this table is reported at 21°C and 760 mm Hg pressure. To correct to 0°C and 760 mm Hg pressure multiply by 1.077.

Target Analytes	CAS	MW	Ret Time	Field Blank #1		Field Blank #2		Trip Blank #1		Trip Blank #2	
				(mg/m ³)	Flag						
Pyridine	110-86-1	79.1	26.518	0.0214	U	0.0214	U	0.0214	U	0.0214	U
trans-1,3-Dichloropropene	10061-02-6	110.97	27.368	0.0027	U	0.0027	U	0.0027	U	0.0027	U
Pentanenitrile	110-59-8	83.15	27.368	0.0008	U	0.0008	U	0.0008	U	0.0008	U
1,1,2-Trichloroethane	79-00-5	133.4	27.815	0.0015	U	0.0015	U	0.0015	U	0.0015	U
Toluene	108-88-3	92.14	28.452	0.0009	U	0.0009	U	0.0009	U	0.0009	U
1,2-Dibromoethane	106-93-4	187.86	29.983	0.0024	U	0.0024	U	0.0024	U	0.0024	U
Octane	111-65-9	114	30.429	0.0027	U	0.0027	U	0.0027	U	0.0027	U
Tetrachloroethylene	127-18-4	165.83	30.982	0.0025	U	0.0025	U	0.0025	U	0.0025	U
Chlorobenzene	108-90-7	112.56	32.566	0.0011	U	0.0011	U	0.0011	U	0.0011	U
Hexanenitrile	628-73-9	97	32.757	0.0029	U	0.0029	U	0.0029	U	0.0029	U
Ethylbenzene	100-41-4	106.17	33.416	0.0014	U	0.0014	U	0.0014	U	0.0014	U
p/m-Xylene	106-42-3	106.17	33.841	0.003	U	0.003	U	0.003	U	0.003	U
Cyclohexanone	108-94-1	98.14	34.288	0.0125	U	0.0125	U	0.0125	U	0.0125	U
Styrene	100-42-5	104.15	34.755	0.0015	U	0.0015	U	0.0015	U	0.0015	U
1,1,2,2-Tetrachloroethane	79-34-5	167.85	34.989	0.0058	U	0.0058	U	0.0058	U	0.0058	U
o-Xylene	95-47-6	106.17	35.053	0.0019	U	0.0019	U	0.0019	U	0.0019	U
Nonane	111-84-2	128	35.414	0.0013	U	0.0013	U	0.0013	U	0.0013	U
1-Ethyl-2-methyl benzene	611-14-3	120.19	38.412	0.0024	U	0.0024	U	0.0024	U	0.0024	U
1,3,5-Trimethylbenzene	108-67-8	120.19	38.624	0.0024	U	0.0024	U	0.0024	U	0.0024	U
1,2,4-Trimethylbenzene	95-63-6	120.19	39.9	0.0025	U	0.0025	U	0.0025	U	0.0025	U
Decane	124-18-5	142.28	40.08	0.003	U	0.003	U	0.003	U	0.003	U
1,3-Dichlorobenzene	541-73-1	147	40.442	0.0032	U	0.0032	U	0.0032	U	0.0032	U
1,4-Dichlorobenzene	106-46-7	147	40.644	0.0029	U	0.0029	U	0.0029	U	0.0029	U
1,2-Dichlorobenzene	95-50-1	147	41.781	0.0047	U	0.0047	U	0.0047	U	0.0047	U
Undecane	1120-21-4	156	44.406	0.0044	U	0.0044	U	0.0044	U	0.0044	U
1,2,4-Trichlorobenzene	120-82-1	181.45	48.02	0.0155	U	0.0155	U	0.0155	U	0.0155	U
Dodecane	112-40-3	170	48.434	0.0255	U	0.0255	U	0.0255	U	0.0255	U
Hexachloro-1,3-butadiene	87-68-3	260.76	49.838	0.0205	U	0.0205	U	0.0205	U	0.0205	U
Tridecane	629-50-5	184	52.186	0.054	U	0.054	U	0.054	U	0.054	U
Tetradecane	629-59-4	198	55.705	0.013	U	0.013	U	0.013	U	0.013	U
Tributyl Phosphate	126-73-8	266	63.1	<0.0099	Z	<0.0099	Z	<0.0099	Z	<0.0099	Z

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section 3.4.2.1 for more information.

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