

# Pacific Northwest National Laboratory

Operated by Battelle for the  
U.S. Department of Energy

## TANK VAPOR CHARACTERIZATION PROJECT

### Headspace Vapor Characterization of Hanford Waste Tank 241-C-107: Second Comparison Study Results from Samples Collected on 03/26/96

J. C. Evans  
K. H. Pool  
B. L. Thomas

K. B. Olsen  
J. S. Fruchter  
K. L. Silvers

RECEIVED  
APR 28 1997  
OSTI

January 1997

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

MASTER

PNNL-11269

## DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY

*operated by*

BATTELLE MEMORIAL INSTITUTE

*for the*

UNITED STATES DEPARTMENT OF ENERGY

*under Contract DE-AC06-76RLO 1830*

Printed in the United States of America

Available to DOE and DOE contractors from the  
Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831;  
prices available from (615) 576-8401.

Available to the public from the National Technical Information Service,  
U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161



This document was printed on recycled paper.

## **TANK VAPOR CHARACTERIZATION PROJECT**

### **Headspace Vapor Characterization of Hanford Waste Tank 241-C-107: Second Comparison Study Results from Samples Collected on 3/26/96**

J. C. Evans  
K. H. Pool  
B. L. Thomas  
K. B. Olsen  
J. S. Fruchter  
K. L. Silvers

January 1997

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

Pacific Northwest National Laboratory  
Richland, Washington 99352

**MASTER**

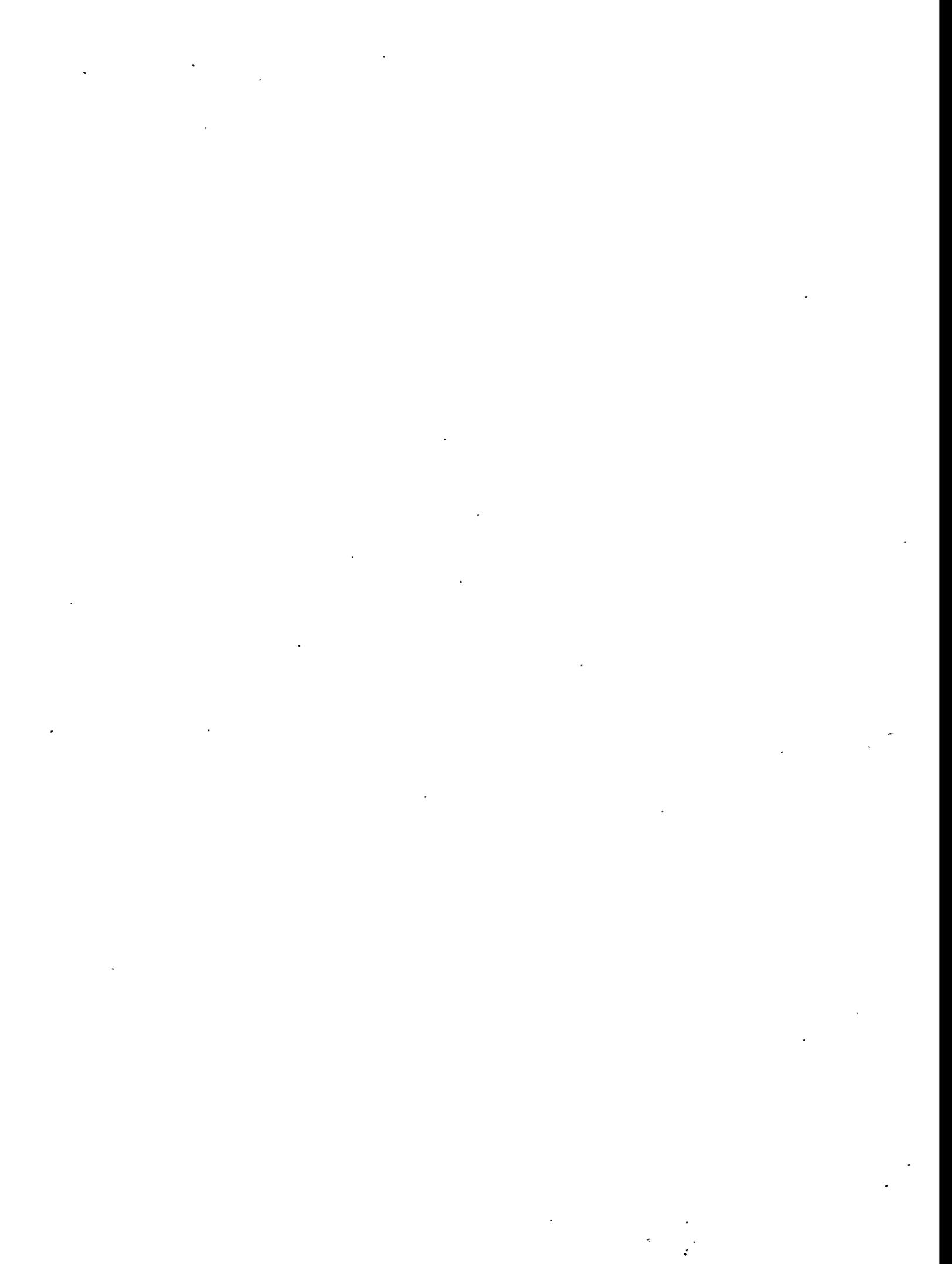
HH  
DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

NOTES

1. The first part of the notes is devoted to a discussion of the

## Summary

This report describes the analytical results of vapor samples taken from the headspace of waste storage tank 241-C-107 (Tank C-107) at the Hanford Site in Washington State. The results described in this report is the second in a series comparing vapor sampling of the tank headspace using the Vapor Sampling System (VSS) and In Situ Vapor Sampling (ISVS) system without high efficiency particulate air (HEPA) prefiltration. The results include air concentrations of water (H<sub>2</sub>O) and ammonia (NH<sub>3</sub>), permanent gases, total non-methane organic compounds (TO-12), and individual organic analytes collected in SUMMA™ canisters and on triple sorbent traps (TSTs). Samples were collected by Westinghouse Hanford Company (WHC) and analyzed by Pacific Northwest National Laboratory (PNNL). Analyses were performed by the Vapor Analytical Laboratory (VAL) at PNNL. Analyte concentrations were based on analytical results and, where appropriate, sample volume measurements provided by WHC.

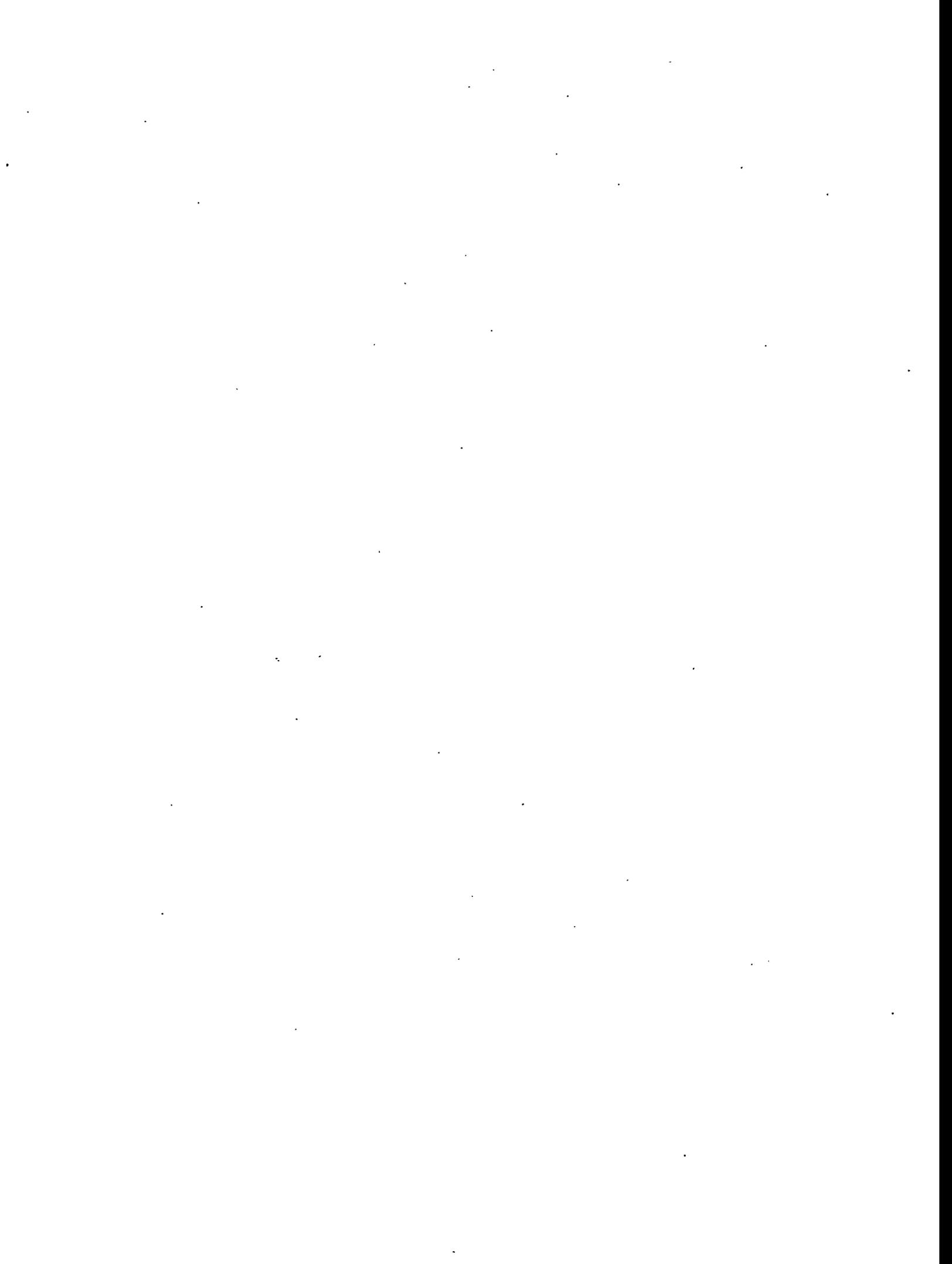


**DISCLAIMER**

**Portions of this document may be illegible  
in electronic image products. Images are  
produced from the best available original  
document.**

## **Acknowledgments**

The authors gratefully acknowledge the support of other project staff at Pacific Northwest National Laboratory (PNNL) who contributed to the successful completion of this sampling and analysis activity. J.A. Edwards served as the PNNL single-point-of-contact for sample preparation and shipping. J.L. Julya assisted with organic laboratory work. S.O. Slate, K.P. Schielke, L.M.P. Thomas, and G.W. Dennis supported inorganic laboratory work.



## Glossary

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
HEPA	high efficiency particulate air
IDL	instrument detection limit
IS	internal standard
ISVS	In Situ Vapor Sampling
LLS	low level standard
MDL	method detection limit
NIST	National Institute for Standards and Technology
% D	percent difference
PNL	previous designation for Pacific Northwest 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
QC	quality control
RPD	relative percent difference
RSD	relative standard deviation
SAP	sample and analysis plan
ST DEV	standard deviation
STP	standard temperature and pressure
SUMMA™	stainless steel, passivated interior canister
TBP	tributyl phosphate
TIC	tentatively identified compound
TNMOC	total non-methane organic compounds
TST	triple sorbent trap
UHP	ultra high purity
VAL	Vapor Analytical Laboratory
VSS	vapor sampling system
WHC	Westinghouse Hanford Company



# Contents

Summary .....	iii
Acknowledgments .....	v
Glossary .....	vii
1.0 Introduction .....	1
2.0 Analytical Results .....	3
2.1 Water and Ammonia .....	3
2.2 Permanent Gases .....	3
2.3 Total Non-Methane Organic Compounds .....	4
2.4 Organic Compounds from SUMMA™ Canisters .....	4
2.5 Organic Compounds from Triple Sorbent Traps .....	5
3.0 Conclusions .....	9
4.0 Reference and Further Reading .....	11
Appendix A: Tank Vapor Characterization: Water and Ammonia .....	A.1
Appendix B: Tank Vapor Characterization: Permanent Gases .....	B.1
Appendix C: Tank Vapor Characterization: Total Non-Methane Organic Compounds .....	C.1
Appendix D: Tank Vapor Characterization: Organic Compounds from SUMMA™ Canisters ..	D.1
Appendix E: Tank Vapor Characterization: Organic Compounds from Triple Sorbent Traps ..	E.1
Appendix F: Tank Vapor Characterization: Target Analytes Measured .....	F.1
Appendix G: Tank Vapor Characterization: Chain of Custody Sample Control Forms .....	G.1

## Tables

2.1.	Comparison of Water and Ammonia Mean Values for Samples Collected from the Headspace of Tank C-107 Using VSS and ISVS	3
2.2	Comparison of Permanent Gas Mean Values for Samples Collected from the Headspace of Tank C-107 Using VSS and ISVS	4
2.3	Comparison of TO-12 Mean Values for Samples Collected from the Headspace of Tank C-107 Using VSS and ISVS	4
2.4	Summary of SUMMA™ Sample Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96	6
2.5	Summary of Triple Sorbent Trap Sample Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96	7
A.1	Analytical Procedures, Quantitation Limits, and Notification Levels for Selected Inorganic Analytes	A.4
A.2	Water and Ammonia Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96	A.5
B.1	Analytical Procedures and Detection Limits for Permanent Gases	B.2
B.2	Permanent Gas Analysis Results for Samples Collected from the Headspace of Tank C-107 and Ambient Air Collected Near Tank C-107 on 3/26/96	B.3
C.1	TO-12 Analysis Results for Samples Collected from the Headspace of Tank C-107 and Ambient Air Near Tank C-107 on 3/26/96	C.4
D.1	Reported Organic Analytes of Interest	D.2
D.2	SUMMA™ Sample Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96	D.6
D.3	Replicate Analysis of SUMMA™ Canisters for Samples Collected from the Headspace of Tank C-107 on 3/26/96	D.7
D.4	SUMMA™ Blank Sample Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96	D.8
E.1	Reported Organic Analytes of Interest	E.2
E.2	Triple Sorbent Trap Sample Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96	E.7

## Tables (Cont)

E.3	Replicate Analysis of Triple Sorbent Trap Samples Collected from the Headspace of Tank C-107 on 3/26/96	E.8
E.4	Triple Sorbent Trap Blank Sample Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96	E.9
F.1	SUMMA™ Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank C-107 on 3/26/96	F.1
F.2	Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank C-107 on 3/26/96	F.3
F.3	SUMMA™ Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank C-107 on 3/26/96	F.5
F.4	Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank C-107 on 3/26/96	F.7
F.5	SUMMA™ Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank C-107 on 3/26/96	F.9
F.6	Triple Sorbent Trap Replicate Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank C-107 on 3/26/96	F.11
F.7	SUMMA™ Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank C-107 on 3/26/96	F.13
F.8	Triple Sorbent Trap Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank C-107 on 3/26/96	F.15

## 1.0 Introduction

This report describes the results of vapor samples obtained to compare vapor sampling of the tank headspace using the Vapor Sampling System (VSS) and In Situ Vapor Sampling System (ISVS) without particulate prefiltration. Samples were collected from the headspace of waste storage tank 241-C-107 (Tank C-107) at the Hanford Site in Washington State. Pacific Northwest National Laboratory (PNNL)<sup>(a)</sup> was contracted by Westinghouse Hanford Company (WHC) to provide sampling devices and analyze samples for water, ammonia, permanent gases, total nonmethane hydrocarbons (TNMOCs, also known as TO-12), and organic analytes in samples collected in SUMMA™ canisters and on triple sorbent traps (TSTs) from the tank headspace. The analytical work was performed by the PNNL Vapor Analytical Laboratory (VAL) by the Tank Vapor Characterization Project. Work performed was based on a sampling and analysis plan (SAP) prepared by WHC. The SAP provided job-specific instructions for samples, analyses, and reporting. The SAP for this sample job was "Sampling and Analysis Plan for Tank Vapor Sampling Comparison Test" (Homi 1996), and the sample jobs were designated S6019 and S6020. Samples were collected by WHC on March 26, 1996, using the VSS, a truck-based sampling method using a heated probe; and the ISVS without particulate prefiltration.

Sampling devices and controls provided for this job included 11 sorbent trains for water and ammonia (seven sample trains and four field blanks); nine SUMMA™ canisters for permanent gases, TO-12 and volatile organic analytes (six samples and three ambient canisters); and 13 TSTs for organic analytes (seven samples, four field blanks, and two trip blanks). The samples and controls were provided to WHC on March 25, 1996. Exposed samples and controls were returned to PNNL on April 2, 1996. Samples and controls were handled, stored, and transported using chain-of-custody (COC) forms to ensure sample quality was maintained.

Samples and controls were handled and stored as per PNNL technical procedure PNL-TVP-07<sup>(b)</sup>, and upon return to PNNL, were logged into PNNL Laboratory Record Book 55408. Samples were stored at the VAL under conditions (e.g., ambient, refrigerated) required by technical procedures. Access to the samples was controlled and limited to PNNL staff trained in the application of specific technical procedures to handle samples for the tank vapor characterization project. Analyses were performed in the 300 Area at Hanford. Specific analytical methods are described in the text.

Tank headspace samples were analyzed for

- *water and ammonia* using weight gain for water and ion-specific electrode for ammonia,
- *permanent gases* using gas chromatography/thermal conductivity detection (GC/TCD),

---

(a) Pacific Northwest National Laboratory is operated for the U. S. Department of Energy by Battelle under Contract DE-AC06-76RLO 1830. The previous name for the laboratory was Pacific Northwest Laboratory (PNL), which is used when previously published documents are cited.

(b) PNL-TVP-07, Rev. 2, December, 1995, *Sample Shipping and Receiving Procedure for PNL Waste Tank Samples*, PNL Technical Procedure, Tank Vapor Project, Pacific Northwest Laboratory, Richland, Washington.

- *total non-methane organic compounds* using cryogenic preconcentration followed by gas chromatography/flame ionization detection (GC/FID), and
- *organic vapors* using cryogenic preconcentration followed by gas chromatography/mass spectrometer (GC/MS) detection.

This report provides summary and detailed analytical information related to the samples and controls. Section 2.0 provides a summary of analytical results. Section 3.0 provides conclusions. Descriptions of samples, analytical methods, quality assurance (QA) and quality control issues, and detailed sample results are provided for each category of samples and analyses in Appendices A, B, C, D, and E. Appendix F contains a listing of all target analytes measured during the analysis of samples from this Tank C-107 comparison study. Appendix G contains the completed COC forms.

## 2.0 Analytical Results

Samples obtained by WHC from the headspace of Tank C-107 on March 26, 1996, (Sample Jobs S6019 and S6020) were analyzed in the PNNL VAL. Summarized results are described in this section. Details of samples, analyses, and data tables are provided in the appendices.

### 2.1 Water and Ammonia

The complete results of the water and ammonia analysis of Tank C-107 for the two sampling methods can be found in Appendix A of this report. Table 2.1 presents the mean concentration values for these two analytes. Mean water concentration values ranged from 29.2 mg/L in the ISVS samples to 47.0 mg/L in the VSS samples. Mean NH<sub>3</sub> concentration values ranged from 47.3 ppmv in the VSS samples to 49.4 ppmv in the ISVS samples.

**Table 2.1.** Comparison of Water and Ammonia Mean Values for Samples Collected from the Headspace of Tank C-107 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
Water (mg/L)	47.0	29.2
Ammonia (ppmv)	47.3	49.4

### 2.2 Permanent Gases

The complete results of the permanent gas analyses of Tank C-107 for the two sampling methods can be found in Appendix B of this report. Table 2.2 presents the mean concentration values for the five permanent gases measured. Hydrogen (H<sub>2</sub>), carbon dioxide (CO<sub>2</sub>), and nitrous oxide (N<sub>2</sub>O) were measured above the analytical method estimated quantitation limit (EQL). Methane (CH<sub>4</sub>) and carbon monoxide (CO) were not observed in any of the samples. No significant differences were found in the mean concentrations of H<sub>2</sub>, CO<sub>2</sub>, and N<sub>2</sub>O for the two different sampling methods.

**Table 2.2.** Comparison of Permanent Gas Mean Values for Samples Collected from the Headspace of Tank C-107 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
H <sub>2</sub> (ppmv)	254	250
CO <sub>2</sub> (ppmv)	574	567
N <sub>2</sub> O (ppmv)	56	55
CH <sub>4</sub> (ppmv)	4.3 U	4.3 U
CO (ppmv)	3.2 U	3.2 U

### 2.3 Total Non-Methane Organic Compounds

The complete results of the U.S. Environmental Protection Agency (EPA) TO-12 analyses for TNMOCs in Tank C-107 can be found in Appendix C of this report. A summary of those results can be found in Table 2.3. The TNMOC average concentrations ranged from 2.8 mg/m<sup>3</sup> in the VSS SUMMA™ samples to 4.2 mg/m<sup>3</sup> in the ISVS samples.

**Table 2.3.** Comparison of TO-12 Mean Values for Samples Collected from the Headspace of Tank C-107 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
TO-12 (mg/m <sup>3</sup> )	2.8	4.2

### 2.4 Organic Compounds from SUMMA™ Canisters

The complete results of the organic vapor analyses from SUMMA™ canisters from Tank C-107 can be found in Appendices D and F of this report. A summary of those results can be found in Table 2.4.

In summary, methanol and acetonitrile were the most abundant compounds identified in each of the SUMMA™ canister samples. Tetrahydrofuran, dodecane, and tetradecane were not observed in any samples from Tank C-107. Tributyl phosphate (TBP) was measured as a tentatively identified compound (TIC) but was not found in any of the SUMMA™ canisters measured. Based on the average values for each of the sampling methods the highest concentrations of methanol, ethanol, acetonitrile, acetone, propanol, and 1-butanol were observed in the VSS samples. Traces of propanol, hexane, 1-butanol, and tridecane were observed in a single SUMMA™ ISVS sample.

## 2.5 Organic Compounds from Triple Sorbent Traps

The complete results of the organic vapor analyses from TSTs from Tank C-107 can be found in Appendices E and F of this report. A summary of those results can be found in Table 2.5.

In summary, methanol and acetonitrile were the most abundant compounds identified in each of the trap samples. Tetrahydrofuran and TBP were not observed in any samples from Tank C-107. Ethanol was detected at concentrations less than the low level standard. Based on the average values for both of the sampling methods the highest concentrations of methanol, hexane, 1-butanol, dodecane, tridecane, and tetradecane were observed in the VSS samples. The highest concentrations of acetonitrile, acetone, and propanol were observed in the ISVS samples.

Table 2.4 Summary of SUMMA™ Sample Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
Average	2298 Y	139 Y	1543 Y	1085	17	U	U	5.4 J	U	0.8 J	U	Z
ST DEV	433	35	592	409	3.1			1.6				
% RSD	18	25	38	38	16			29				
<b>ISVS</b>												
Average	1555 Y	83 Y	1167 Y	811	8.2 J	U	1.2 J	4.2 J	U	1.1 J	U	Z
ST DEV	231	11	39	69								
% RSD	14	13	3.3	8.4								

**Data Qualifier Flag**

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

U Target compound not detected at or above the IDL.

Z TBP was analyzed as a TIC; however, was not identified in the sample.

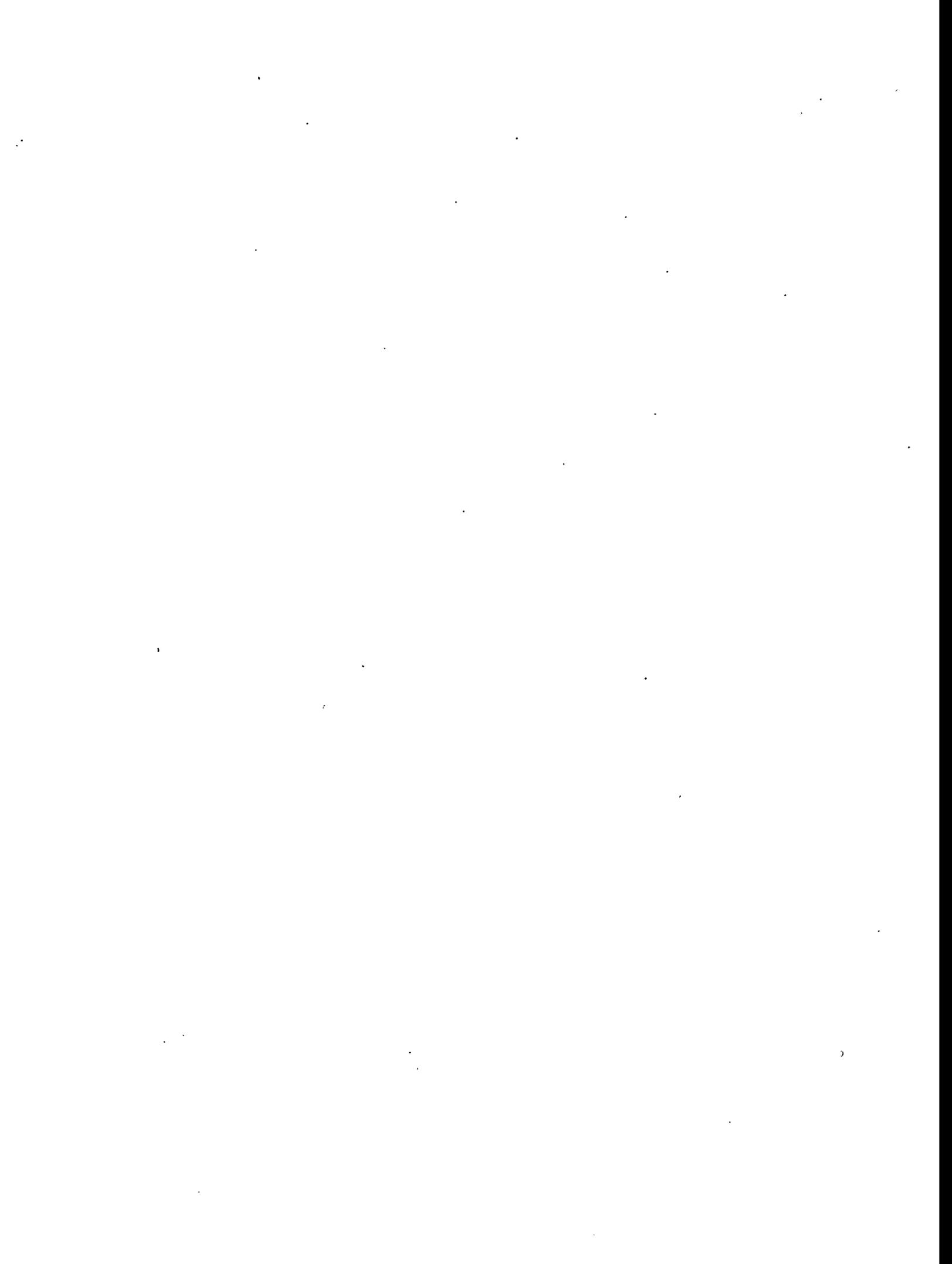
Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Table 2.5 Summary of Triple Sorbent Trap Sample Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
Average	1028 Y	<133 Y	451 Y	206	12 J	U	1.2 J	7.5 J	4.1 J	13 J	25	Z
ST DEV	400		172	31	6.9			1.6	0.2	1.7	3.9	
% RSD	39		38	15	57			21	5.9	14	16	
ISVS												
Average	634 Y	<133 Y	456	224	17 J	U	U	7.0 J	U	8.3 J	13 J	Z
ST DEV	25		9.2	6.4	0.0			0.3			12	
% RSD	3.9		2.0	2.8	0.0			4.0			94	

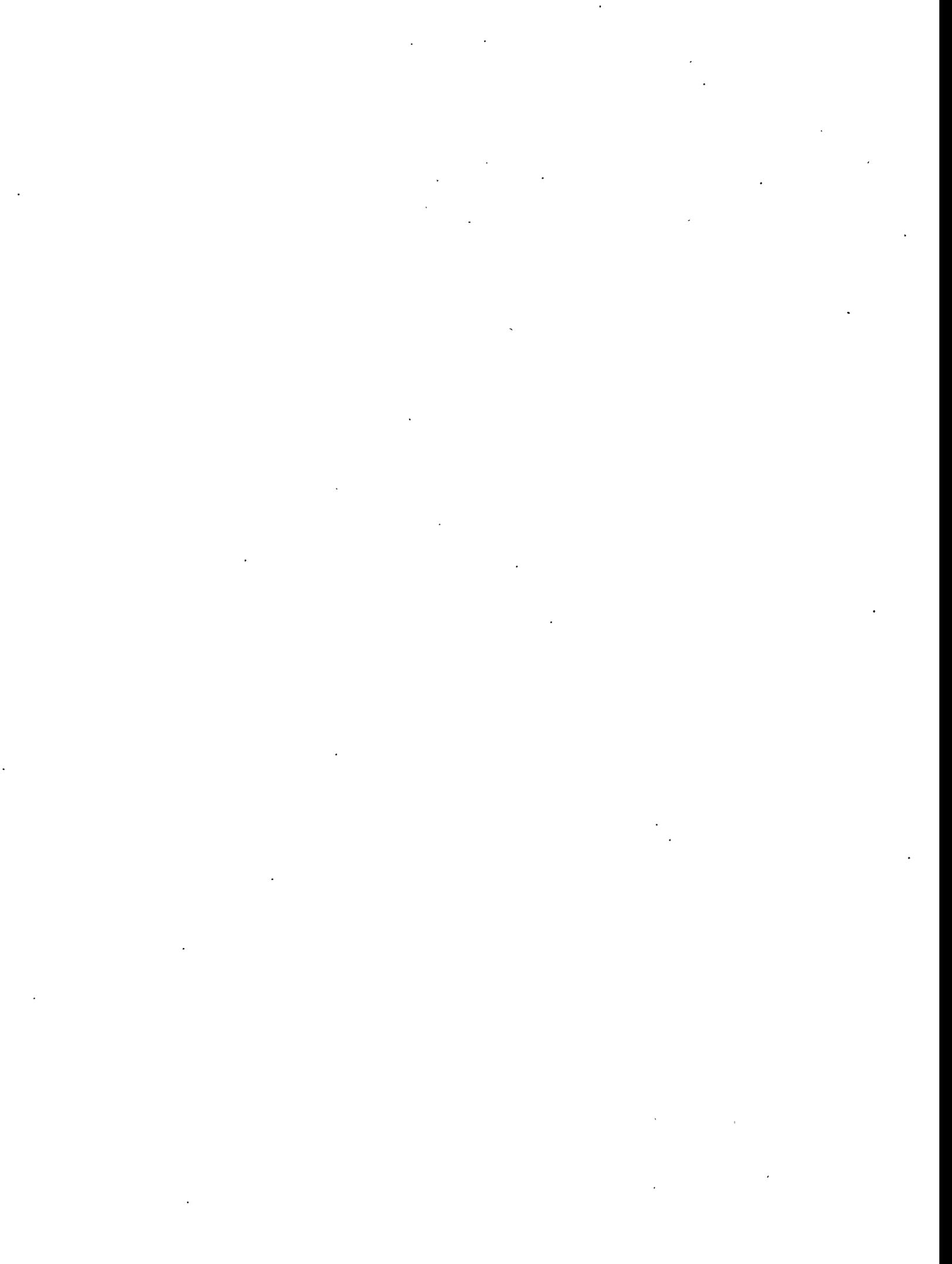
Data Qualifier Flag

- 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 E.4 for more information.



### 3.0 Conclusions

The air concentrations of H<sub>2</sub>O and NH<sub>3</sub>, permanent gases, total non-methane organic compounds, and organic vapors were determined from samples from the headspace of Tank C-107 sampled on March 26, 1996. WHC sample job numbers were S6019 and S6020. The gas and vapor concentrations were based either on whole-volume samples (SUMMA™ canisters) or on triple sorbent traps exposed to sample flow. In the case of the canisters, the concentrations were based on analytical results of subsamples obtained directly from the canisters. In the case of the sorbent traps, concentrations were based on analyses by the VAL and sample volumes reported by WHC. Known sampling and analytical variances from established QA requirements, where significant, were documented in this report, as required by the SAP (Homi 1996).



## 4.0 Reference and Further Reading

### Reference

Homi, C.S. 1996. *Sampling and Analysis Plan for Tank Vapor Sampling Comparison Test*. WHC-SD-WM-TSAP-073, Rev. 1B, Westinghouse Hanford Company, Richland, Washington.

### Further Reading

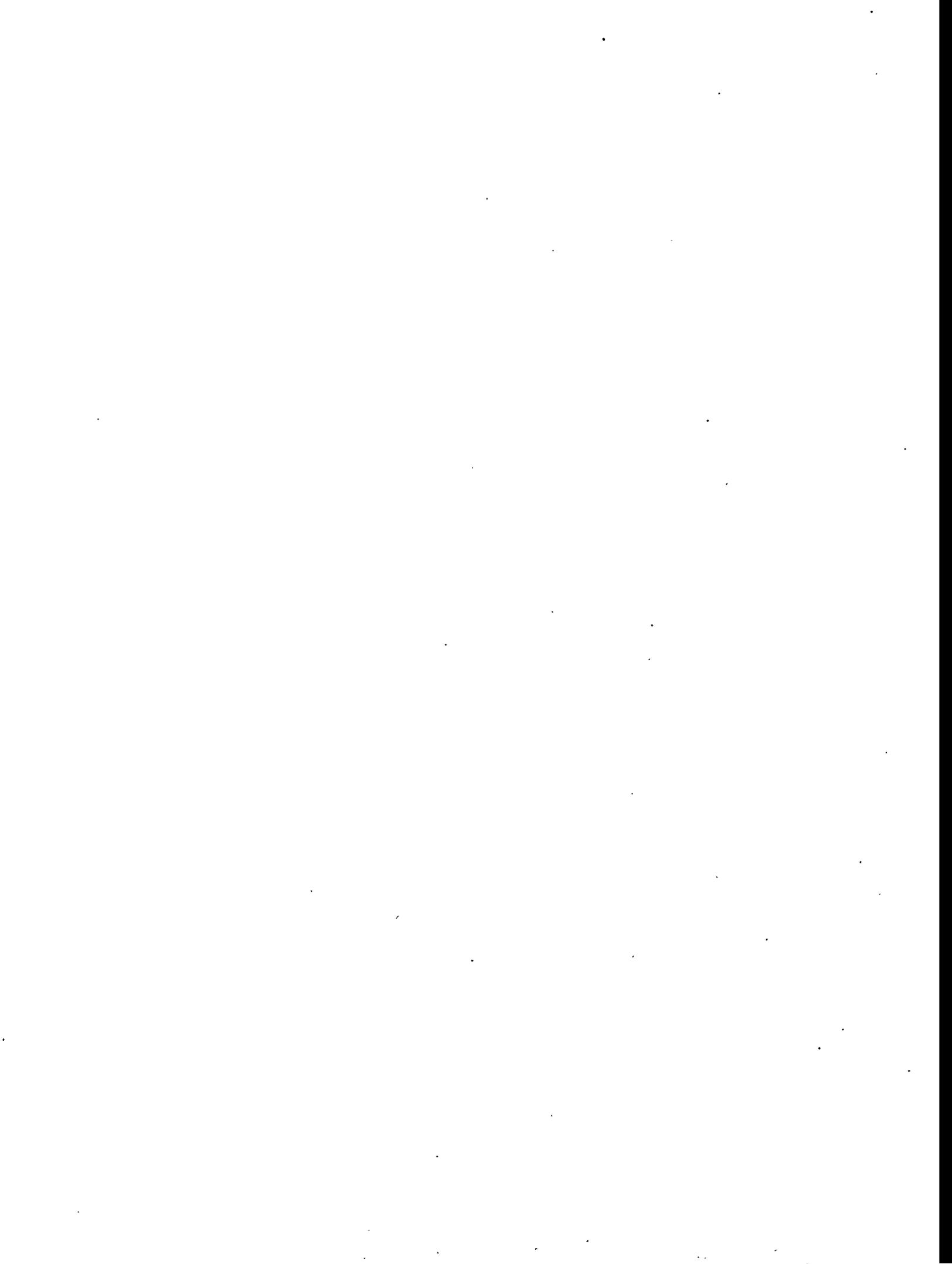
Pacific Northwest Laboratory. *Analytical Laboratory Procedure Compendium*. Procedure PNL-ALO-271. PNL-MA-599, Pacific Northwest Laboratory, Richland, Washington.

Pacific Northwest Laboratory. *Quality Assurance Manual, Part 3: Procedures for Quality Assurance Program*. PNL-MA-70, Part 3, Pacific Northwest 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. 0, Pacific Northwest National Laboratory, Richland, Washington.

U.S. Department of Energy. *Hanford Analytical Services Quality Assurance Plan (HASQAP)*. DOE/RL-94-55, Rev. 2, U. S. Department of Energy, Richland, Washington.

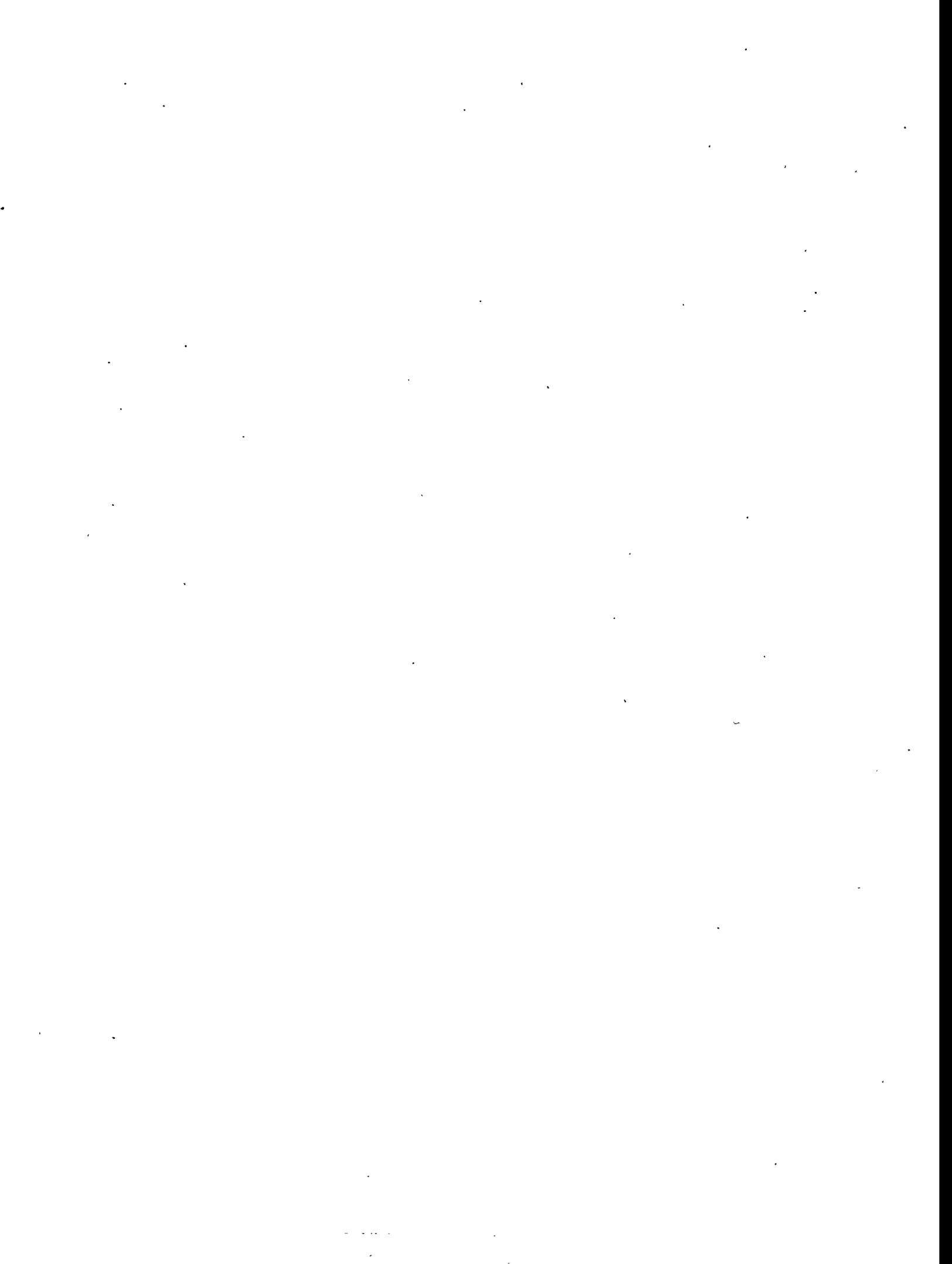
Westinghouse Hanford Company. *Quality Assurance Project Plan for Tank Vapor Characterization*. WHC-SD-WM-QAPP-013, Rev. 1, Westinghouse Hanford Company, Richland, Washington.



## **Appendix A**

### **Tank Vapor Characterization:**

#### **Water and Ammonia**



## Appendix A

### Tank Vapor Characterization: Water and Ammonia

Solid sorbent traps, prepared in multi-trap sampling trains, were supplied to Westinghouse Hanford Company (WHC) for sampling the tank headspace using the VSS and ISVS systems. Blanks, spiked blanks (when requested by the SAP), and exposed samples were returned to Pacific Northwest National Laboratory (PNNL) for analysis. Analyses were performed to provide information on the tank headspace concentration of ammonia ( $\text{NH}_3$ ) and water ( $\text{H}_2\text{O}$ ). Procedures were similar to those developed previously during sample jobs performed with the VSS connected to the headspace of Tank C-103 (Ligotke et al. 1994). During those sample jobs, control samples provided validation that the sorbent tubes effectively trapped  $\text{NH}_3$  and mass. Samples were prepared, handled, and disassembled as described in Technical Procedure PNL-TVP-09<sup>(a)</sup>. Analytical accuracy was estimated based on procedures used. Sample preparation and analyses were performed following PNNL quality assurance (QA) impact level II requirements.

#### A.1 Sampling Methodology

Standard glass tubes containing sorbent materials to trap vapors of  $\text{NH}_3$  and  $\text{H}_2\text{O}$  (supplied by SKC Inc., Eighty Four, Pennsylvania) were obtained, prepared, and submitted for vapor sampling. The sorbent traps were selected based on their use by the Occupational Safety and Health Administration to perform workplace monitoring and because of available procedures and verification results associated with that particular application. The typical sorbent traps used consisted of a glass tube containing a sorbent material specific to the compound of interest. In general, the tubes contained two sorbent layers, or sections; the first layer was the primary trap, and the second layer provided an indication of breakthrough. In the tubes, sorbent layers are generally held in packed layers separated by glass wool. The sorbent traps, with glass-sealed ends, were received from the vendor.

The type and nominal quantity of sorbent material varied by application. Sorbent traps were selected for the tank sample job and included the following products. The  $\text{NH}_3$  sorbent traps contained carbon beads impregnated with sulfuric acid; nominally, 500 mg were contained in the primary and 250 mg in the breakthrough sections. The  $\text{NH}_3$  was chemisorbed as ammonium sulfate [ $(\text{NH}_4)_2\text{SO}_4$ ]. The water traps contained 300 mg of silica gel in the primary and 150 mg in the breakthrough sections.

Sorbent trains provided to trap inorganic compounds included all or some of the following: samples, spiked samples, spares, blanks, and spiked blanks. Sorbent trains were prepared from same-lot batches. After sample preparation, sorbent trains were stored at  $\leq 10^\circ\text{C}$  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, Pacific Northwest Laboratory, 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 sorbent trains configured so sample flow passed in order through the traps, targeting specific analytes, and then through a desiccant trap. The specific order of traps within the various sorbent trains is described in Section A.4. The ends of the glass-tube traps were broken, and the traps were weighed and then connected to each other using uniform lengths of 3/8-in. perfluoroalkoxy-grade Teflon® tubing. The 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 that has a 3/8-in. stainless steel Swagelok® nut, sealed using a Swagelok® cap. The trailing ends of the sorbent trains (the downstream end of the traps containing silica gel) were each sealed with red plastic end caps provided by the manufacturer. The sorbent-tube trains remained sealed other than during the actual sampling periods. During vapor sampling, C-Flex® tubing was provided by WHC to connect the downstream ends of the sorbent trains to the sampling manifold exhaust connections.

**A.1.1 Concentration Calculations.** The concentrations of target compounds in the tank headspace were determined from sample results, assuming effective sample transport to the sorbent traps. Concentration, in parts per million by volume (ppmv), was determined by dividing the mass of the compound, in  $\mu\text{mol}$ , by the volume of the dried tank air sampled in moles. The micromolar sample mass was determined by dividing the compound mass, in  $\mu\text{g}$ , by the molecular weight of the compound, in  $\text{g/mol}$ . The molar sample volume was determined, excluding water vapor, by dividing the standard sample volume (at  $0^\circ\text{C}$  and 760 torr), in L, by 22.4 L/mol. For example, the concentration by volume of a 3.00-L sample containing 75.0  $\mu\text{g}$  of  $\text{NH}_3$  is given by

$$\frac{75.0 \mu\text{g}}{17.0 \text{ g/mol}} \left[ \frac{3.00 \text{ L}}{22.4 \text{ L/mol}} \right]^{-1} = 32.9 \text{ ppmv} \quad (\text{A.1})$$

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

## A.2 Analytical Procedures

The compounds of interest were trapped using solid sorbents and chemisorption (adsorption of water vapor). Analytical results were based on extraction and analysis of selected ions. Analytical procedures used are specified in the text.

**A.2.1 Ammonia Analysis.** The sorbent material from the NH<sub>3</sub>-selective sorbent traps was placed into labeled 20-mL glass scintillation vials. Vials containing front-, or primary-, section sorbent material were treated with 10.0 mL of deionized water (DIW), and vials containing back-up-section sorbent material were treated with 5.0 mL of DIW. After extraction, the NH<sub>3</sub> sorbent traps were analyzed using the selective ion electrode procedure PNL-ALO-226<sup>(a)</sup>. Briefly, this method includes 1) preparing a 1000- $\mu$ g/mL (ppm) NH<sub>3</sub> stock standard solution from dried reagent-grade NH<sub>4</sub>Cl and DIW, 2) preparing 0.1-, 0.5-, 1.0-, 10-, and 100-ppm NH<sub>3</sub> 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 NH<sub>3</sub> 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<sub>4</sub>Cl standard from an independent source, after analyzing every five or six samples, 5) continuing this sequence until all samples of the batch have been measured, including duplicates and spiked samples, and 6) remeasuring the complete set of calibration standards (at the end of the session). Electromotive force (volts) signal measurements obtained for samples are compared to those for standards, either graphically or algebraically (using linear regression) to determine NH<sub>3</sub> concentration in the samples.

**A.2.2 Mass (Water) Analysis.** Sorbent traps used to make each sample train were weighed using a semi-micro mass balance, after labeling and breaking the glass tube ends, without plastic end caps. After receipt of exposed samples, the sorbent traps were again weighed to determine the change in mass. Records of the measurements were documented on sample-preparation data sheets. The mass concentration, generally roughly equal to the concentration of water, was determined by dividing the combined change in mass from all traps in a sorbent train by the actual volume of gas sampled. Field blanks were used to correct results.

### A.3 Quality Assurance/Quality Control

Analytical work was performed according to quality levels identified in the project QA plan and several PNNL documents. The samples were analyzed following PNNL Impact Level II. The PNNL documents include PNL-MA-70 (Part 3), PNL-ALO-212, PNL-ALO-226, and ETD-002. A summary of the analysis procedures and limits for the target inorganic compounds is provided in Table A.1. The table also shows generic expected notification ranges and describes related target analytical precision and accuracy levels for each analyte; the information in the table is based on the data quality objective assessment by Osborne et al. (1995). From the table, it can be seen that the method detection limit (MDL) required to resolve the analyte at one-tenth of the recommended exposure limit for each of the target analytes is achieved using current procedures and with a vapor-sample volume of 2 L and a desorption-solution volume of 3 mL (10 mL for NH<sub>3</sub>).

The accuracy of concentration measurements depends on potential errors associated with both sampling and analysis (see Section A.4). Sampling information, including sample volumes, was provided by WHC; sample-volume uncertainty was not provided. The uncertainty of analytical results, which depends on the method used, was estimated to be within allowable tolerances (Osborne

---

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

et al. 1995; Table A.1). For NH<sub>3</sub> analyses, the accuracy of laboratory measurements by selective ion electrode was estimated to be  $\pm 5\%$  relative, independent of concentration at 1  $\mu\text{g}/\text{mL}$  or greater levels. The uncertainty includes preparation of standards, purity of the ammonium salt used to prepare standards, potential operator bias, ambient temperature variations, etc. Working standards are traceable to NIST standard reference material by using an independent calibration verification standard certified to be NIST traceable.

**Table A.1.** Analytical Procedures, Quantitation Limits, and Notification Levels for Selected Inorganic Analytes<sup>(a)</sup>

<u>Analyte</u>	<u>Formula</u>	<u>Procedure</u>	EQL <sup>(b)</sup> ( $\mu\text{g}$ )	EQL <sup>(b)</sup> (ppmv)	Notification Level <sup>(c)</sup> (ppmv)
Ammonia	NH <sub>3</sub>	PNL-ALO-226	1.0	0.7	$\geq 150$
Mass (water) <sup>(d)</sup>	n/a	PNL-TVP-09	0.6 mg	0.2 mg/L	n/a

(a) Analytical precision and accuracy targets for results in the expected ranges equal  $\pm 25\%$  and 70 to 130%, respectively (Osborne et al. 1995).

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

(c) As per Table 7-1 in Osborne et al. (1995). Notification levels require verbal and written reports to WHC on completion of preliminary analyses.

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

#### A.4 Water and Ammonia Sample Results

Table A.2 lists results of the water and ammonia analysis from samples collected from the headspace of Tank C-107. These samples were collected through the VSS and through the ISVS systems. A total of 11 samples each were collected with the two different sampling methods. The samples were analyzed for ammonia on April 15, 1996 and for water on April 12, 1996. Mean water concentration values ranged from 29.2 mg/L in the ISVS samples to 47.0 mg/L in the VSS samples. Mean ammonia concentration values ranged from 47.3 ppmv in the VSS samples to 49.4 ppmv in the ISVS samples.

**Table A.2 Water and Ammonia Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96**

<b>VSS Truck Samples</b>	<b>H<sub>2</sub>O mg/L</b>	<b>NH<sub>3</sub> ppmv</b>
S6019-A17.S16	44.3	45.2
S6019-A18.S17	47.7	51.3
S6019-A19.S18	48.3	46.1
S6019-A20.S19	47.7	46.6
<b>Average</b>	<b>47.0</b>	<b>47.3</b>
<b>% RSD</b>	<b>3.9</b>	<b>5.8</b>

<b>ISVS</b>		
S6020-A34.S22	28.7	47.4
S6020-A35.S23	28.9	47.6
S6020-A36.S24	30.0	53.2
<b>Average</b>	<b>29.2</b>	<b>49.4</b>
<b>% RSD</b>	<b>2.4</b>	<b>6.7</b>

## A.5 References

Clauss, T. W., M. W. Ligothke, B. D. McVeety, K. H. Pool, R. B. Lucke, J. S. Fruchter, and S. C. Goheen. 1994. *Vapor Space Characterization of Waste Tank 241-BY-104: Results from Samples Collected on 6/24/94*. PNL-10208. Pacific Northwest Laboratory, Richland, Washington.

Ligothke, M. W., K. H. Pool, and B. D. Lerner. 1994. *Vapor Space Characterization of Waste Tank 241-C-103: Inorganic Results from Sample Job 7B (5/12/94 - 5/25/94)*. PNL-10172, Pacific Northwest Laboratory, Richland, Washington.

Osborne, J. W., J. L. Huckaby, E. R. Hewitt, C. M. Anderson, D. D. Mahlum, B. A. Pulsipher, and J. Y. Young. 1995. *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Resolution*. WHC-SD-WM-DQO-002, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

## **Appendix B**

### **Tank Vapor Characterization:**

#### **Permanent Gases**

## Appendix B

### Tank Vapor Characterization: Permanent Gases

#### B.1 Sampling Methodology

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant-free according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-02<sup>(a)</sup>. The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis. If the canister is verified as clean by TO-12, the canister is evacuated to 5 mtorr, tagged, and stored for use in the field. Before sending the canisters out to the field for sampling, the canister vacuum is measured to determine if any leakage has occurred. If the vacuum has remained constant during storage, the canisters are prehumidified with 100 µL of distilled water and labeled with a field-sampling identification. Canisters stored more than 30 but less than 60 days are re-evacuated and rehumidified before use. If stored more than 60 days, the canisters are recleaned and validated before use.

#### B.2 Analytical Procedure

The SUMMA™ canister samples were analyzed for permanent gases according to PNNL Technical Procedure PNL-TVP-05<sup>(b)</sup> with the exceptions listed in the following text and in the quality assurance/quality control section of this report. This method was developed in-house to analyze permanent gases, defined as hydrogen (H<sub>2</sub>), carbon dioxide (CO<sub>2</sub>), carbon monoxide (CO), methane (CH<sub>4</sub>), and nitrous oxide (N<sub>2</sub>O), by gas chromatograph/thermal conductivity detection (GC/TCD). Aliquots of sampled air are drawn directly from each canister into a 5-mL gas-tight syringe and injected into a 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 CO, CO<sub>2</sub>, N<sub>2</sub>O, and CH<sub>4</sub> using Helium (He) as the carrier gas. A second GC analysis is performed for H<sub>2</sub> (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 B.1.

- 
- (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.
  - (b) Pacific Northwest Laboratory. 12/95. *Analysis Method for the Determination of Permanent Gases in Hanford Waste Tank Vapor Samples Collected in SUMMA™ Passivated Stainless Steel Canisters*, PNL-TVP-05 (Rev. 1). PNL Technical Procedure, Pacific Northwest Laboratory, Richland, Washington.

**Table B.1. Analytical Procedures and Detection Limits for Permanent Gases**

<u>Analyte</u>	<u>Formula</u>	<u>Procedure</u>	<u>Instrument Detection Limit (ppmv)</u>	<u>Estimated Quantitation Limit (ppmv)</u>
Carbon Dioxide	CO <sub>2</sub>	PNL-TVP-05	2.4	24
Carbon Monoxide	CO	PNL-TVP-05	3.2	32
Methane	CH <sub>4</sub>	PNL-TVP-05	4.3	43
Hydrogen	H <sub>2</sub>	PNL-TVP-05	3.1	31
Nitrous Oxide	N <sub>2</sub> O	PNL-TVP-05	2.0	20

### **B.3 Quality Assurance/Quality Control**

Standards for the permanent gas analysis were blended from commercially prepared and certified standards for each of the analytes reported in Table B.1. The instrument was calibrated for CO, CO<sub>2</sub>, N<sub>2</sub>O, and CH<sub>4</sub> over a range of 25 to 2100 parts per million by volume (ppmv) using standards at five different concentrations and He as a carrier gas. A similar procedure was followed for H<sub>2</sub>, except the carrier gas was changed to N<sub>2</sub>. An average response factor from the calculation was used for qualification of compound peak area.

Each analyte was quantitated by comparison of sample analyte peak area to the calibration plot generated for the compound. An instrument detection limit (IDL) study was conducted and performance data are presented in Table B.1. The EQL for the method has also been established as 10 times the IDL. 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  $\pm 25\%$  of the expected concentrations. One sample was run in duplicate to provide a measure of method precision. Results of the replicate analysis are presented in Table B.2. An N<sub>2</sub> reagent blank, an ambient-air sample collected ~10 m upwind of Tank C-107, and the ambient air collected through the VSS and ISVS were used as method blanks and used to determine the potential for analyte interferences in the samples.

### **B.4 Permanent Gases Sample Results**

Table B.2 lists results of the permanent gas analysis from samples collected from the headspace of Tank C-107 and ambient air collected near Tank C-107. These samples were collected through the VSS and ISVS systems. A total of nine samples were collected with the two different sampling methods. The samples were analyzed on April 12 and 15, 1996. Replicate analyses on SUMMA™ canisters were conducted on three samples within each sampling method set. Hydrogen, carbon dioxide, and nitrous oxide were observed above the EQL in all of the tank headspace samples. Average hydrogen concentrations ranged from 250 ppmv in the ISVS samples to 254 ppmv in the VSS samples. Average carbon dioxide concentrations ranged from 567 ppm in the ISVS samples to 574 ppmv in the VSS samples. Average nitrous oxide concentrations ranged from 55 ppmv in the ISVS samples to 56 ppmv in the VSS samples. Methane and carbon monoxide concentrations were below the IDL.

**Table B.2** Permanent Gas Analysis Results for Samples Collected from the Headspace of Tank C-107 and Ambient Air Collected Near Tank C-107 on 3/26/96

VSS Truck Samples	H <sub>2</sub> (ppmv)		CO <sub>2</sub> (ppmv)		N <sub>2</sub> O (ppmv)		CH <sub>4</sub> (ppmv)		CO (ppmv)	
S6019-A03.026 (Ambient)	3.1	U	357		2.0	U	4.3	U	3.2	U
S6019-A04.031 (Ambient)	3.1	U	343		2.0	U	4.3	U	3.2	U
S6019-A05.032	258		566		48		4.3	U	3.2	U
S6019-A07.059	251		579		60		4.3	U	3.2	U
S6019-A06.057	253		575		61		4.3	U	3.2	U
Average	254		574		56		4.3	U	3.2	U
% RSD	1.3		1.2		13		NA		NA	
S6019-A07.059 (REP)	257		577		61		4.3	U	3.2	U
<b>ISVS</b>										
S6020-A27.095 (Ambient)	3.1	U	344		2.0	U	4.3	U	3.2	U
S6020-A29.149	255		565		46		4.3	U	3.2	U
S6020-A30.150	258		580		60		4.3	U	3.2	U
S6020-A28.147	236		557		59		4.3	U	3.2	U
Average	250		567		55		4.3	U	3.2	U
% RSD	4.6		2.0		15		NA		NA	
S6020-A30.150 (REP)	254		577		63		4.3	U	3.2	U

**Data Qualifier Flags**

U Target compound not detected at or above the IDL.

NA Not applicable.



## **Appendix C**

### **Tank Vapor Characterization:**

### **Total Non-Methane Organic Compounds**

## Appendix C

### Tank Vapor Characterization: Total Non-Methane Organic Compounds

#### C.1 Sampling Methodology

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant-free according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-02<sup>(a)</sup>. The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis. If the canister is verified as clean by TO-12, the canister is evacuated to 5 mtorr, tagged, and stored for use in the field. Before sending the canisters out to the field for sampling, the canister vacuum is measured to determine if any leakage has occurred. If the vacuum has remained constant during storage, the canisters are prehumidified with 100 µL of distilled water and labeled with a field-sampling identification. Canisters stored more than 30 but less than 60 days are re-evacuated and rehumidified before use. If stored more than 60 days, the canisters are recleaned and validated before use.

#### C.2 Analytical Procedure

The SUMMA™ canister samples were analyzed according to PNNL Technical Procedure PNL-TVP-08<sup>(b)</sup>, which is similar to U.S. Environmental Protection Agency (EPA) compendium Method TO-12. The method detection limits in the sub mg/m<sup>3</sup> are required to determine total non-methane organic compounds (TNMOC) concentration in the tank samples.

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 (He). The purged TNMOCs are carried by a UHP He stream to the GC equipped with an FID where gross organic content is detected and measured.

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.

- 
- (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.
  - (b) 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, Pacific Northwest Laboratory, Richland, Washington.

Twenty-four hours before the analysis, the SUMMA™ canister samples are pressurized with purified air (supplied by Aadco Instruments, Inc., 1920 Sherwood St., Clearwater, Florida 34625). The starting pressure was first measured using a calibrated diaphragm gauge (Cole Parmer), then pressurized to a level exactly twice the original pressure. For example, if the canister had a starting pressure of 740 torr, it was pressurized to 1480 torr. The sample dilution was taken into account when calculating the analysis results.

### C.3 Quality Assurance/Quality Control

This method requires user calibration (category 2 measuring and test equipment) of the analytical system in accordance with QA plan ETD-002.

The TNMOC is calibrated by using propane as the calibration standard. The instrument calibration mixture for the PNL-TVP-08 analysis consists of National Institute for Standards and Technology (NIST) 99.999% propane analyzed using an average response factor method for calibration.

A continuing calibration verification (CCV) standard of 100 ppmv propane is analyzed to confirm acceptability of instrument performance. The initial calibration is then used to quantify the samples.

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 this 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 system should be cleaned to 0.1 mg/m<sup>3</sup> of TNMOCs. Second, an instrument continuing calibration is run using 100-mL UHP propane analyzed using the response factor followed by one blank volume of Aadco air.

**C.3.1 Quantitation Results of Target Analytes.** The mg/m<sup>3</sup> was derived from the five-point multilevel calibration curve from the propane standard using the following equation:

$$\text{mg/m}^3 = \frac{(\text{ng TNMOC}) \times (\text{dilution factor})}{\text{mL sampled volume}} \quad (\text{C.1})$$

The ng/m<sup>3</sup> concentrations are calculated from mg/m<sup>3</sup> using the equation:

$$\text{ng/m}^3 \text{ TNMOC} = \frac{(\text{ng TNMOC})}{(\text{mL sampled})} \times \text{Dilution Factor} \times \frac{(\text{mg})}{(1 \times 10^6 \text{ mL})} \times \frac{(1 \times 10^6 \text{ mL})}{(\text{m}^3)} \quad (\text{C.2})$$

#### C.4 Total Non-Methane Organic Compounds Sample Results

Table C.1 lists results of the TO-12 gas analysis from samples collected from the headspace of Tank C-107 and ambient air collected near Tank C-107. These samples were collected through the VSS and ISVS. A total of nine samples were collected by each of the two sampling methods. The samples were analyzed on April 16, 1996. Replicate analyses on SUMMA™ canisters were conducted on one sample within each sampling method set. Concentrations in the three ambient air samples ranged from 0.24 mg/m<sup>3</sup> to 0.42 mg/m<sup>3</sup>. Average concentrations ranged from 2.8 mg/m<sup>3</sup> in the VSS samples to 4.2 mg/m<sup>3</sup> in the ISVS samples.

**Table C.1 TO-12 Analysis Results for Samples Collected from the Headspace of Tank C-107 and Ambient Air Near Tank C-107 on 3/26/96**

<b>VSS Truck Samples</b>	<b>TO-12 mg/m3</b>
S6019-A03.026 (Ambient)	0.26 J
S6019-A04.031 (Ambient)	0.24 J
S6019-A05.032	2.9
S6019-A07.059	2.8
S6019-A06.057	2.8
<b>Average</b>	<b>2.8</b>
<b>% RSD</b>	<b>2.0</b>
S6019-A07.059 (REP)	2.8
<b>ISVS</b>	
S6020-A27.095 (Ambient)	0.42 J
S6020-A29.149	4.2
S6020-A30.150	4.1
S6020-A28.147	4.3
<b>Average</b>	<b>4.2</b>
<b>% RSD</b>	<b>2.4</b>
S6020-A30.150 (REP)	4.1

**Data Qualifier Flags**

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

## **Appendix D**

### **Tank Vapor Characterization:**

### **Organic Compounds from SUMMA™ Canisters**

## Appendix D

### Tank Vapor Characterization: Organic Compounds from SUMMA™ Canisters

#### D.1 Sampling Methodology

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant free according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-02<sup>(a)</sup>. The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis by PNNL Technical Procedure PNL-TVP-03<sup>(b)</sup>, which is a modification of the U.S. Environmental Protection Agency (EPA) compendium Method TO-14. If the canister is verified as clean, free of TO-14 and unknown contaminants to a level of 5 parts per billion by volume (ppbv), the canister is evacuated to 5 mtorr, tagged, and stored for use in the field. Before sending the canisters out to the field for sampling, the canister vacuum is measured to determine if any leakage has occurred. If the vacuum has remained constant during storage, the canisters are prehumidified with 100  $\mu$ L of distilled water and labeled with a field-sampling identification. Cleaned 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.

#### D.2 Analytical Procedure

The SUMMA™ canister sample was analyzed according to PNNL Technical Procedure PNL-TVP-03, which is a modified version of EPA compendium Method TO-14. The method uses EnTech 7000 cryoconcentration systems 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 air volume, then transfer the volume to the GC/MS for analysis. A 100-mL volume of sample is measured and analyzed from the tank headspace. The organic components in the sampled air are separated on an analytical column, J&W Scientific DB-1 phase, 60-m by 0.32-mm internal diameter with 3- $\mu$ m film thickness. 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. Twenty-four hours before the analysis, the SUMMA™ canister samples were pressurized with purified air (supplied by Aadco Instruments, Inc., 1920 Sherwood St., Clearwater, Florida 34625). The starting pressure was first measured using a calibrated diaphragm gauge (Cole Parmer), then pressurized to a level exactly twice the original pressure. For example, if the canister had a starting pressure of 740 torr, it

- 
- (a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, Richland, Washington.
- (b) Pacific Northwest Laboratory. 2/96. *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. 1), PNL Technical Procedure, Richland, Washington.

was pressurized to 1480 torr. This dilution was an effort to improve the precision of the analysis. The sample dilution was taken into account when calculating the analysis results.

The instrument calibration mixture for the PNL-TVP-03 analysis consists of 66 compounds. For this comparison study, only the 12 compounds listed in Table D.1 were considered organic analytes of interest. An initial calibration and CCV was performed for methanol and ethanol. The low level standard (LLS) was used as the EQL for these compounds. Results below the LLS were not reported. It should be noted that these two compounds are not currently part of the operating procedure. Tributyl phosphate was not analyzed as a target compound, but was evaluated as a TIC. 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<sup>(a)</sup>. The standard calibration mix was analyzed using four aliquot sizes ranging from 30 mL to 200 mL, and a response factor for each compound was calculated. The GC/MS response for these compounds has been previously determined to be linearly related to concentration. Instrument detection limits and EQLs have been determined, with the exception of methanol and ethanol as stated previously.

**Table D.1.** Reported Organic Analytes of Interest

Methanol	Acetone
Ethanol	Acetonitrile
1-Butanol	Tetrahydrofuran
Dodecane	Hexane
Tridecane	Propanol
Tetradecane	Tributyl Phosphate (TBP)

### D.3 Quality Assurance/Quality Control

Before the tank sample was analyzed, a diagnostic check was performed on the GC/MS instrument by running an instrument "high-sensitivity tune," as described in PNL-TVP-03. Upon satisfactory completion of the instrument diagnostic check, a blank volume of purified nitrogen was analyzed to check the cleanliness of the system. The instrument was then calibrated using a standard gas mixture containing 66 organic compounds. A gas mixture containing bromochloromethane, 1,4-difluorobenzene, chlorobenzene-d<sub>5</sub>, and bromofluorobenzene was used as an internal standard (IS) for all blank, calibration standard, and sample analyses. Analyte responses from sample components, ISs, and standards were obtained from the extracted ion plot from their selected mass ion. The calibration was generated by calculating the relative response ratios of the IS to calibration standard responses and plotting the ratios against the ratio of the calibration-standard concentration (in ppbv) to the IS concentration. Once it is determined that the relative response is linear with increasing concentration, an average response factor is calculated for each target analyte and used to determine

---

(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.

the concentration of target compounds in each sample. Method blanks are analyzed before and after calibration standards and tank headspace samples are analyzed.

**D.3.1 Quantitation Results of Target Analytes.** The quantitative-analysis results for the target analytes were calculated using the average response factors generated using the IS method described above and in PNL-TVP-03. The conversion from ppbv to mg/m<sup>3</sup> assumes standard temperature and pressure (STP) conditions of 760 torr and 273K and was calculated directly from the following equation:

$$\text{mg/m}^3 = \frac{(\text{ppbv}/1000) \times \text{g mol wt of compound}}{22.4 \text{ L/mol}} \quad (\text{D.1})$$

#### D.4 Volatile Organic Sample Results

Nine SUMMA™ canisters consisting of six samples and three ambient air samples were returned to the laboratory on April 2, 1996, under WHC COC numbers 100049 and 100050. Samples were analyzed on June 25 and 26, 1996.

The results from the GC/MS analysis of the tank headspace SUMMA™ samples are presented in Table D.2. The results of replicate analyses on single SUMMA™ canister samples are presented in Table D.3. The results of the blank sample analyses are presented in Table D.4. Appendix F contains a complete listing of all target analytes measured.

Table D.2 lists the quantitative results for 12 compounds selected for this tank comparison study. Six individual SUMMA™ canister samples were collected using the two different sampling methods. The individual compound values for each of the SUMMA™ canister results for each sampling method were averaged and a standard deviation (ST DEV) and % RSD value calculated. Methanol and acetonitrile were the most abundant compounds identified in each of the SUMMA™ canister samples. Tetrahydrofuran, dodecane, and tetradecane were not observed in any samples from Tank C-107. Tributyl phosphate was measured as a tentatively identified compound (TIC) but was not found in any of the SUMMA™ canisters measured. Based on the average values for each of the sampling methods the highest concentrations of methanol, ethanol, acetonitrile, acetone, propanol, and 1-butanol were observed in the VSS samples. Traces of propanol, hexane, 1-butanol, and tridecane were observed in a single SUMMA™ ISVS sample.

Single SUMMA™ canister samples were analyzed in replicate for each of the two different sampling methods. The relative percent differences (RPDs) were calculated and are presented in Table D.3. The RPDs were calculated for analytes detected above the IDL and found in both replicates.

The results of the blank analyses are reported in Table D.4. The only compound observed in the blank samples was acetone.

Methanol and ethanol are not currently included in the method performance section of the procedure for System 1; however, both 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.

The SUMMA™ canister samples were analyzed in two batches. The sample analytical sequence runs (batches) were as follows:

Batch #1 (file identifier 16062601 & 2.b) - S6019-A03.026, S6019-A04.031, S6020-A29.149, S6020-A29.149 REP, S6020-A30.150, S6019-A05.032;

Batch #2 (file identifier 16062502 & 3.b) - S6020-A27.095, S6019-A06.057, S6019-A07.059, S6020-A28.147, S6019-A06.057 REP.

Batch #1:

Eight target compounds (dichlorodifluoromethane at 32.3%, acetone at 70.2%, 1,1-dichloroethane at 41.5%, hexane at 47.9%, 1,1,1-trichloroethane at 58.3%, carbon tetrachloride at 57.0%, pyridine at 97.3%, and tetradecane at 42.2%) surpassed the 30% RSD acceptance criteria for the initial calibration. Acetone was found in the tank samples at concentrations between the EQL and upper quantitation limit. Dichlorodifluoromethane and pyridine were found in tank samples at concentrations between the IDL and the EQL. Tetradecane, 1,1-dichloroethane, carbon tetrachloride, hexane and 1,1,1-trichloroethane were not found in the tank samples. Hexane and 1,1,1-trichloroethane were found in tank samples S6020-A28.147 and S6019-A06.057 REP at concentrations between the EQL and the IDL.

Six target compounds (dichlorodifluoromethane, acetone, trichlorofluoromethane, pentane, 1,1,2-trichloro-1,2,2-trifluoromethane, and pyridine) were outside the 25% difference (% D) acceptance criteria for the CCV sample; however, the CCV passed the procedural criterion requiring +25% D passage for 85% of all target compounds. Acetone was found in tank samples at concentrations above the EQL and its concentrations may be over estimated because of the failed CCV. Dichlorodifluoromethane, trichlorofluoromethane, 1,1,2-trichloro-1,2,2-trifluoromethane and pyridine were found in tank samples at concentrations between the IDL and the EQL, except for trichlorofluoromethane in sample S6020-A27.095. Pentane was not found in any tank samples. No target compounds were found in the continuing calibration blank (CCB) above the EQL.

The tank sample internal standard quantification area percent recoveries exceeded the acceptance criterion (50% to 200%) allowed by procedure PNL-TVP-03, Rev. 1. The changes in the internal standard areas were caused by water induced fatigue. This problem is routinely observed with the HP5972 GC/MS system because of its poor pumping capacity, the high water and ammonia content in these samples. Target compounds found in these samples could be affected. This problem will continue until a larger GC/MS system is used in the analysis.

Batch #2:

Eight target compounds (dichlorodifluoromethane at 32.3%, acetone at 70.2%, 1,1-dichloroethane at 41.5%, hexane at 47.9%, 1,1,1-trichloroethane at 58.3%, carbon tetrachloride at 57.0%, pyridine at 97.3%, and tetradecane at 42.2%) surpassed the 30% relative standard deviation (%RSD) acceptance criteria for the initial calibration. Acetone was detected in tank samples at concentrations between the EQL and the upper quantitation limit

(UQL). Dichlorodifluoromethane, tetradecane, 1,1-dichloroethane, carbon tetrachloride, hexane, pyridine, and 1,1,1-trichloroethane were not found in the tank samples.

Nine target compounds (chloromethane, 1,2-dichloro-1,1,2,2-tetrafluoroethane, vinyl chloride, butane, pentane, 2-butanone, hexane, cyclohexane, and pyridine) were outside the  $\pm 25\%$  difference (% D) acceptance criteria for the continuing calibration verification (CCV) sample; however, the CCV passed the procedural criteria requiring  $\pm 25\%$  D passage for 85% of all target compounds. The compound 2-butanone was found in tank sample S6020-A30.150 at a concentration above the EQL and this concentration value may be over estimated because of the filed CCV. Chloromethane, 1,2-dichloro-1,1,2,2-tetrafluoroethane, vinyl chloride, butane, 2-butanone, pentane, hexane, pyridine, and cyclohexane were not found in any tank samples except as noted above.

The internal standard quantification area percent recoveries for several tank samples (S6019-A03.026, S6019-A04.031, S6020-A29.149, and S6020-A30.150) exceeded the acceptance criteria (50% to 200%) allowed by procedure PNL-TVP-03, Rev. 1. The changes in internal standard areas were caused by water induced instrument fatigue. This problem is routinely observed with the Hewlett Packard 5972 GC/MS system because of its poor pumping capacity, and the high water and ammonia content in these samples. Target compound results found in these samples could be affected. This problem will continue until a larger GC/MS system is used in the analysis.

Table D.2 SUMMA™ Sample Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
A05.032	2236 Y	126 Y	1140 Y	837	18	U	U	4.5 J	U	U	U	Z
A06.057	1899 Y	113 Y	1265 Y	860	14	U	U	4.5 J	U	U	U	Z
A07.059	2758 Y	179 Y	2223 Y	1557	20	U	U	7.2 J	U	0.8 J	U	Z
Average	2298 Y	139 Y	1543 Y	1085	17	U	U	5.4 J	U	0.8 J	U	
ST DEV	433	35	592	409	3.1			1.6				
% RSD	18	25	38	38	16			29				
<b>ISVS</b>												
A28.147	1521 Y	85 Y	1181 Y	864	8.2 J	U	1.2 J	4.2 J	U	1.1 J	U	Z
A29.149	1801 Y	93 Y	1198 Y	835	U	U	U	U	U	U	U	Z
A30.150	1343 Y	71 Y	1123 Y	733	U	U	U	U	U	U	U	Z
Average	1555 Y	83 Y	1167 Y	811	8.2 J	U	1.2 J	4.2 J	U	1.1 J	U	
ST DEV	231	11	39	69								
% RSD	14	13	3.3	8.4								
<b>Data Qualifier Flag</b>												
J Target compound detected above the IDL but below the EQL.												
U Target compound not detected at or above the IDL.												
Z TBP was analyzed as a TIC; however, was not identified in the sample.												
Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.												

Table D.3 Replicate Analysis of SUMMA™ Canisters for Samples Collected from the Headspace of Tank C-107 on 3/26/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
A06.057	1899 Y	113 Y	1256 Y	860	14	U	U	4.5 J	U	U	U	Z
A06.057 REP	2351 Y	134 Y	1656 Y	1207	18	0.7 J	1.1 J	6.6 J	U	U	U	Z
Relative Percent Difference	21	17	27	34	25			38				
<b>ISVS</b>												
A29.149	1801 Y	93 Y	1198 Y	835	U	U	U	U	U	U	U	Z
A29.149 Rep	1545 Y	75 Y	994 Y	689	U	U	U	U	U	U	U	Z
Relative Percent Difference	15	21	19	19								
<b>Data Qualifier Flag</b>												
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 TBP was analyzed as a TIC; however, was not identified in the sample.												

Table D.4 SUMMA™ Blank Sample Analysis Results for Samples Collected from the Headspace of C-107 on 3/26/96

Blank Samples	(ppbv)	METHANOL	(ppbv)	ETHANOL	(ppbv)	ACETONITRILE	(ppbv)	ACETONE	(ppbv)	PROPANOL	(ppbv)	TETRAHYDROFURAN	(ppbv)	HEXANE	(ppbv)	I-BUTANOL	(ppbv)	DODECANE	(ppbv)	TRIDECANE	(ppbv)	TETRADECANE	(ppbv)	TBP	(ppbv)
Upwind Through VSS	<77	Y	<53	Y	U	U	31	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	Z	Z
Upwind Through VSS	<77	Y	<53	Y	U	U	29	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	Z	Z
Upwind Through ISVS	<77	Y	<53	Y	U	U	18	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	Z	Z

**Data Qualifier Flag**

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 TBP was analyzed as a TIC; however, was not identified in the sample.

## **Appendix E**

### **Tank Vapor Characterization:**

### **Organic Compounds from Triple Sorbent Traps**

## Appendix E

### Tank Vapor Characterization: Organic Compounds from Triple Sorbent Traps

#### E.1 Sampling Methodology

Samples are collected on Supelco 300 graphite-based triple sorbent traps (TSTs). Before field deployment, each trap is heated to 380°C under inert gas flow for a minimum of 60 min. Tubes are prepared in batches with each tank sampling job constituting one batch. One tube is selected from each batch and run immediately to verify cleanliness. All remaining tubes in the batch receive equal amounts of three surrogate compounds (hexafluorobenzene, toluene-d8, and bromobenzene-d5). One per batch tube is run immediately to verify successful addition of surrogate spikes to that batch. Tubes are then placed in individually labeled plastic shipping tubes (Supelco TD<sup>3</sup>), which are sealed with gasketed end caps, thus providing a rugged, headspace-free shipping and storage medium. As a precautionary measure, sample tubes are kept in refrigerated storage before and after sampling.

#### E.2 Analytical Procedure

The Supelco 300 tubes were analyzed according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-10<sup>(a)</sup>, with the exceptions noted in Section E.4. The method employs Supelco Carbotrap<sup>™</sup> 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<sup>™</sup> C, 200 mg of Carbotrap<sup>™</sup> B, and 125 mg of Carbosieve<sup>™</sup> S-III. The first two sorbents are deactivated graphite with limited sorption power for less volatile compounds. The final trapping stage, the Carbosieve<sup>™</sup> 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 internal standard (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 collected on a second identical Carbotrap<sup>™</sup> 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<sup>™</sup> 300 trap, the analyte is transferred to a long, thin focusing trap filled with the same type of trapping materials as the Carbotrap<sup>™</sup> 300 traps and in approximately the same ratios. The purpose of the focusing trap is to provide an interface to a capillary gas chromatograph (GC) column, which may be thermally desorbed at a helium (He) flow rate compatible with the column and mass spectrometry (MS) interface (1.2 mL/min). The focusing trap is

---

(a) Pacific Northwest 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), PNL Technical Procedure, Richland, Washington.

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 MS.

The instrument calibration mixture for the TST analysis consists of 65 compounds: For this comparison study, only the 12 compounds listed in Table E.1 were considered organic analytes of interest. An initial calibration was performed for methanol and ethanol; however, a CCV was not performed. Therefore, concentrations reported are considered estimated for these compounds. The methanol and ethanol LLS was used as the EQL. Results below the LLS were not reported. The calibration mixture is prepared in common with the mixture used for the SUMMA™ analysis (see Section D.2). The standard calibration mix was analyzed using four aliquot sizes ranging from 100 mL to 1200 mL, 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 previously determined to be linearly related to concentration. Instrument detection limits and EQLs have been determined, with the exception of methanol and ethanol as stated previously.

**Table E.1.** Reported Organic Analytes of Interest

Methanol	<i>Acetone</i>
Ethanol	<i>Acetonitrile</i>
1-Butanol	Tetrahydrofuran
Dodecane	Hexane
Tridecane	Propanol
Tetradecane	Tributyl Phosphate (TBP)

*NOTE: Compounds shown in italics have an exceptionally high volatility. They are routinely included in the standard and are quantified, but have a restricted linear dynamic range because of the potential for trap breakthrough.*

### **E.3 Quality Assurance/Quality Control**

Before the tank sample was 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 tube was analyzed to check the cleanliness of the system. The instrument was then calibrated using a 300-mL volume of standard gas mixture containing 12 compounds shown in Table E.1. A gas mixture containing difluorobenzene, chlorobenzene- $d_5$ , 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 is determined that the relative response is linear with increasing concentration, an average response factor is calculated for each target analyte and used to determine the concentration of target compounds in each sample.

**E.3.1 Quantitation Results of Target Analytes.** The quantitative-analysis results for the target analytes were calculated directly from the calibration curve generated using the IS method described above and in PNL-TVP-10. The conversion from ppbv to mg/m<sup>3</sup> assumes STP conditions of 760 torr and 273K and was calculated directly from the following equation:

$$\text{mg/m}^3 = \frac{(\text{ppbv}/1000) \times \text{g mol wt of compound}}{22.4 \text{ L/mol}} \quad (\text{E.1})$$

#### **E.4 Triple Sorbent Trap Volatile Organic Sample Results**

Thirteen TSTs consisting of 7 samples, 4 field blanks, and 2 trip blanks were returned to the laboratory on April 2, 1996, under WHC COC numbers 100041 and 100042. The samples were analyzed between April 18 and April 23, 1996.

The results from the GC/MS analysis of the tank headspace TST samples are presented in Table E.2. The results of replicate analyses on TST samples are presented in Table E.3. The results of the blank sample and tape analyses are presented in Table E.4. Appendix F contains a complete listing of the 66 target analytes measured.

Table E.2 lists the quantitative results for 12 compounds selected for this tank comparison study. Five individual TST samples were analyzed and reported for the two different sampling methods. The individual compound values for each of the TST samples for each sampling method were averaged and a ST DEV and % RSD value calculated. Methanol and acetonitrile were the most abundant compounds identified in the trap samples. Tetrahydrofuran and TBP were not observed in any samples from Tank C-107. Ethanol was detected at concentrations less than the low level standard. Based on the average values for both of the sampling methods the highest concentrations of methanol, hexane, 1-butanol, dodecane, tridecane, and tetradecane were observed in the VSS samples. The highest concentrations of acetonitrile, acetone, and propanol were observed in the ISVS samples.

Triple sorbent trap samples were analyzed in replicate for the two different sampling methods. The RPDs were calculated and presented in Table E.3. The RPDs were calculated for analytes detected above the IDL and found in both replicates.

The results of the blank analyses are reported in Table E.4. Methanol was observed only in the ISVS and trip blanks samples, and traces of acetone were observed in all of the blank samples.

All standards, blanks and samples were analyzed under the protocols of procedure PNL-TVP-10, Rev. 2 with the initial calibration performed on April 8, 1996 and subsequent sample runs quantitated against CCVs at the beginning of each daily batch.

The TST samples were analyzed in 4 batches. The sample analytical sequence runs (batches) were as follows:

Batch #1 (file identifier 46041801.d) - S6019-A10.802, S6020-A32.820,  
S6020-A32.820 REP, S6019-A09.801;

Batch #2 (file identifier 46041901.d) - S6019-A11.803, S6020-A31.816, S6019-A08.800, S6019-A08.800 REP, S6019-A11.803 REANALYSIS;

Batch #3 (file identifier 46042201.d) - S6019-A12.805, S6019-A13.812, S6020-A40.861, S6020-A41.862, S6019-A14.813, S6019-A15.815;

Batch #4 (file identifier 46042301.d) - S6019-A09.801 REANALYSIS, S6019-A10.802 REANALYSIS.

The following discussion provides details regarding quality control (QC) criterion failures for each batch.

Batch #1:

The first CCV was unacceptable according to the procedure and a second CCV was run. The second CCV was satisfactory with the exception of tetradecane (70%). The CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of the target compounds. The tank samples contained only trace levels of tetradecane which values may be over or under estimated. Due to a major perturbation at the front end of the chromatograms believed to be from water condensation in the tubes, sample splits for S6019-A09.801 and S6019-A10.802 were retained and rerun (file identifier 46042301.d) in order to improve the data quality in the front end of the chromatogram.

Batch #2:

The CCV was satisfactory with the exception of pyridine (26%), tridecane (48%) and tetradecane (77%). The CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of the target compounds. Since these compounds were detected at most trace levels, the uncertainty associated with the CCV for these compounds has limited impact. Due to a major perturbation at the front end of the chromatograms for the VSS samples, believed to be from water condensation in the tubes, sample split for S6019-A11.803 was rerun in order to improve the data quality in the front end of the chromatogram. The data from this rerun will be reported.

Batch #3:

The CCV was satisfactory for all target compounds with the exception of pyridine (28%) and tetradecane (68%). The CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of the target compounds. Since these compounds were not detected in the trip or field blank samples, the uncertainty associated with the CCV for these compounds has no impact.

Batch #4:

The CCV was satisfactory with the exception of pyridine (33%), tridecane (49%) and tetradecane (86%). The CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of the target compounds. Since these compounds were detected at most trace levels, the uncertainty associated with the CCV for these compounds has limited impact. Tetradecane was present in the CCB above the EQL. Sample results for tetradecane are flagged with a

"B" to indicate the compound was detected in the laboratory blank at greater than twenty times the concentration in the samples. The two samples in this batch were reruns of earlier samples which showed considerably improved chromatography. The data from these reruns will be reported.

Several observations and comments associated with the data generated are discussed below.

1. Several compounds have consistently tended to exhibit less than average precision as demonstrated through comparison of daily CCVs with the initial calibration. These have included methanol, ethanol, pyridine, and tetradecane. Tridecane has also exhibited marginal behavior for some runs. It is believed that the problem with the first three compounds is associated with their high degree of polarity which evidently causes problems making reproducible vapor standards extracted from Summa canisters. Tridecane and tetradecane may have volatilities too low to produce reliable vapor standards by what are essentially volatile compound handling techniques.
2. The VSS samples from Tank C-107 showed a major perturbation of the front end of the chromatograms similar to the effect seen on the previous VSS sampling of this tank. The split samples were retained and were rerun successfully in order to improve the data quality in that region. This effect is believed to be associated with water condensation on the tubes. The effect is not present on the ISVS samples from Tank C-107 and has not been observed for VSS jobs on other tanks. Tank C-107 is known to have an exceptionally high absolute humidity in the vapor headspace. Only data from the rerun split samples should be used.
3. Methanol and ethanol are not currently included in procedure PNL-TVP-10; however, both compounds were analyzed per 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.
4. Tributyl phosphate is included in the analysis 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 0.8 ppbv (based on 200 mL sample) was possible. Tributyl phosphate was not detected in the tank samples.
5. Sample volumes for all ISVS samples have been corrected to STP from the 21°C calibration used on the sampling cart flowmeters. The correction was included in the reported data to provide seamless compatibility with the VSS data that were collected using a 0°C calibration.
6. Very narrow air spikes occasionally are present in chromatograms from this period. This problem was traced to high frequency air leakage through the diffusion pump o-ring and had been corrected for some time but has again reappeared. It has no known impact on data quality but the spikes do appear as features on the total ion chromatogram.

7. All field samples including field and trip blanks contained minor amounts of the TIC 1-chloro-1,1-difluoroethane. This compound has appeared persistently in most samples sent to the field in the past but was absent in the previous set of samples from Tank S-102. It is believed to be a fugitive refrigerant. That 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 in the field part of the chain-of-custody.

8. Target compounds observed in the VSS samples at significant levels (above EQL) included: methanol, ethanol, acetonitrile, acetone, propanenitrile, 2-butanone, butanenitrile, 1-butanol, 4-methyl-2-pentanone, and pentanenitrile. Several other target analytes were observed in trace amounts. A very limited number of TICs were detectable (acetaldehyde, bromomethane, and ethyl nitrate). The ISVS sample showed a similar list of targets and TICs; however, in addition, minor amounts of aromatics, cycloalkanes and other compounds were observed which appear to be environmental contamination associated with preparation of the ISVS bundle. Several of the indicator compounds associated with the 3M Scotch Brand 471 tape which caused major problems with the earlier Tank C-107 sampling job were also present but at much lower levels than previously. These included heptane, toluene, and methyl cyclohexane.

No target compounds were observed in the VSS field blanks or trip blanks at significant levels (above EQL). The ISVS field blanks showed acetone, toluene, and p/m xylene above the EQL. Those compounds are likely to be associated with environmental contamination picked up during the assembly of the ISVS bundle. Passive sampling inside the tank may have also contributed to the acetone, but in general, passive sampling effects appear to be very limited for this sampling job.

9. Four VSS and three ISVS TST tubes were sent to the field for sampling. All four of the VSS samples were successfully analyzed and reanalyzed a second time on the split tubes. One of the ISVS tubes was damaged in the field and disposed of at that time with the discrepancy noted on the COC form.

Table E.2 Triple Sorbent Trap Sample Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
A11.803	949 Y	<133 Y	503	220	15 J	U	1.2 J	5.8 J	4.4 J	11 J	20	Z
A09.801	936 Y	<133 Y	521	200	9.1 J	U	U	9.4 J	4.0 J	14 J	29	Z
A08.800	1588 Y	<133 Y	197	166	4.1 J	U	U	8.2 J	3.9 J	14 J	24	Z
A10.802	637 Y	<133 Y	581	237	20	U	U	6.6 J	3.9 J	11 J	27	Z
Average	1028 Y	<133 Y	451	206	12 J	U	1.2 J	7.5 J	4.1 J	13 J	25	Z
ST DEV	400		172	31	6.9			1.6	0.2	1.7	3.9	
% RSD	39		38	15	57			21	5.9	14	16	
ISVS												
A31.816	616 Y	<133 Y	449	219	17 J	U	U	7.2 J	U	8.3 J	21	Z
A32.820	651 Y	<133 Y	462	228	17 J	U	U	6.8 J	U	U	4.2	Z
Average	634 Y	<133 Y	456	224	17 J	U	U	7.0 J	U	8.3 J	13	Z
ST DEV	25		9.2	6.4	0.0			0.3			12	
% RSD	3.9		2.0	2.8	0.0			4.0			94	

Data Qualifier Flag

- 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.
- E Target compound exceeds upper quantification limit (UQL).
- 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 E.4 for more information.

Table E.3 Replicate Analysis of Triple Sorbent Trap Samples Collected from the Headspace of Tank C-107 on 3/26/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
A08.800	1588 Y	<133 Y	197 Y	166	4 J	U	U	8.2 J	3.9 J	14 J	24	<0.83 Z
A08.800 Rep	929 Y	<133 Y	539 Y	209	16 J	U	1.2 J	6.0 J	4.3 J	14 J	20	<0.83 Z
Relative Percent Difference	52		93	23	118			31	10	0.0	18	
<b>ISVS</b>												
A32.820	651 Y	<133 Y	462 Y	228	17 J	U	U	6.8 J	U	U	4.2 J	<0.80 Z
A32.820 Rep	550 Y	<133 Y	426 Y	218	14 J	U	U	6.5 J	U	U	3.3 J	<0.80 Z
Relative Percent Difference	17		8.1	4.5	19			4.5			24	

E. 8

**Data Qualifier Flag**

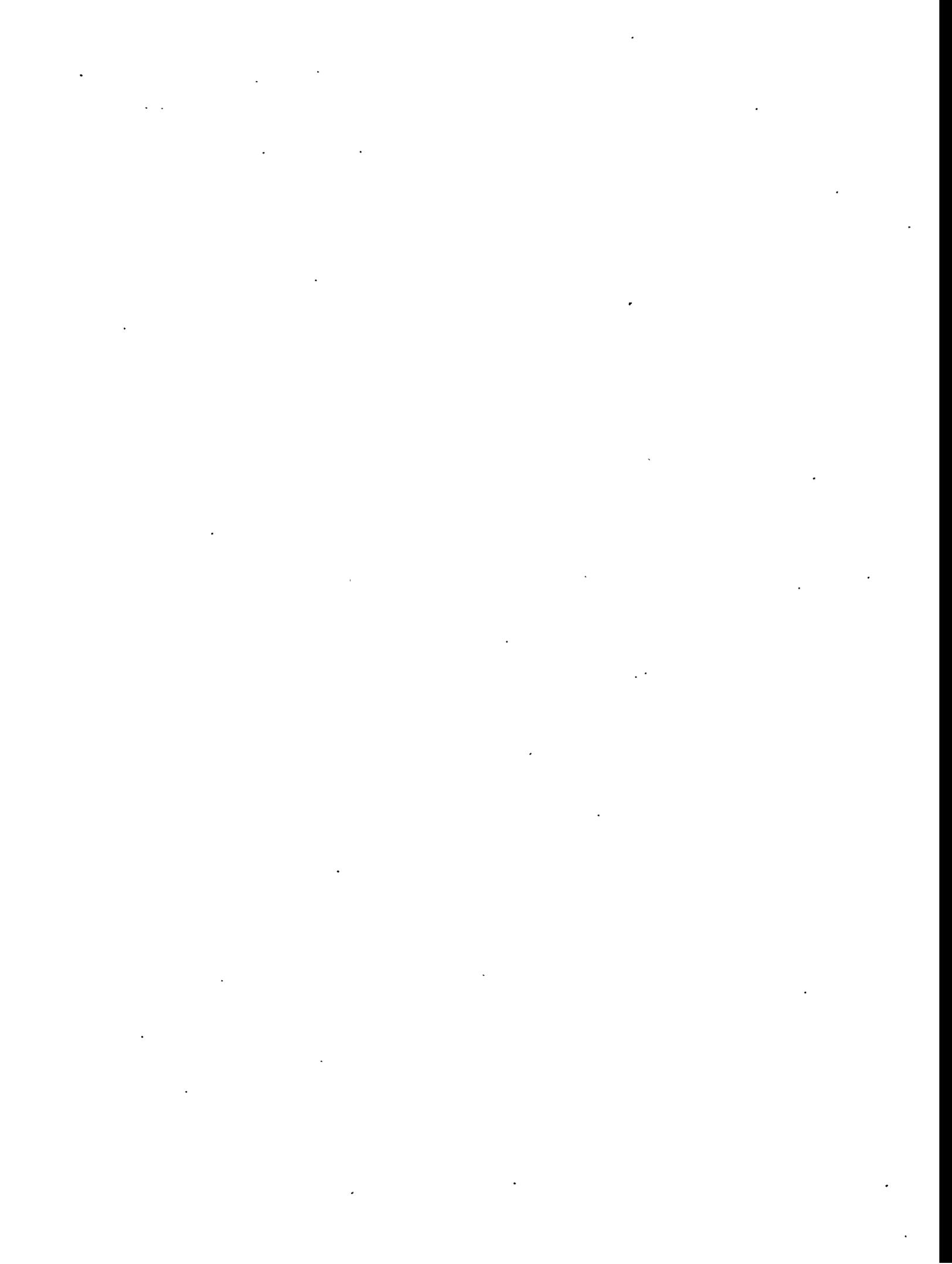
- J Target compound detected above the IDL but below the EQL.
- U Target compound not detected at or above the IDL.
- Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.
- Z TBP was analyzed as a positively identified compound (PIC) however, the analyte was not part of the current operating procedure.

Table E.4 Triple Sorbent Trap Blank Sample Analysis Results for Samples Collected from the Headspace of Tank C-107 on 3/26/96

Blank Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
Field Blank # 1 VSS	<192 Y	<133 Y	1.8 U	17 J	2.0 U	1.1 U	0.3 U	2.3 U	3.4 U	6.6 U	1.5 U	<0.8 Z
Field Blank # 2 VSS	<192 Y	<133 Y	1.8 U	18 J	2.0 U	1.1 U	0.3 U	2.3 U	3.4 U	6.6 U	1.5 U	<0.8 Z
Field Blank #3 ISVS	<192 Y	<133 Y	10 J	33	2.0 U	1.1 U	0.3 U	2.3 U	3.4 U	6.6 U	1.5 U	<0.8 Z
Field Blank #4 ISVS	<192 Y	<133 Y	10 J	30	2.0 U	1.1 U	2.0 J	2.3 U	3.4 U	6.6 U	1.5 U	<0.8 Z
Trip Blank #1	<192 Y	<133 Y	1.8 U	19 J	2.0 U	1.1 U	0.3 U	2.3 U	3.4 U	6.6 U	1.5 U	<0.8 Z
Trip Blank #2	<192 Y	<133 Y	1.8 U	17 J	2.0 U	1.1 U	0.3 U	2.3 U	3.4 U	6.6 U	1.5 U	<0.8 Z

**Data Qualifier Flag**

- 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 E.4 for more information.



## **Appendix F**

### **Tank Vapor Characterization:**

### **Target Analytes Measured**

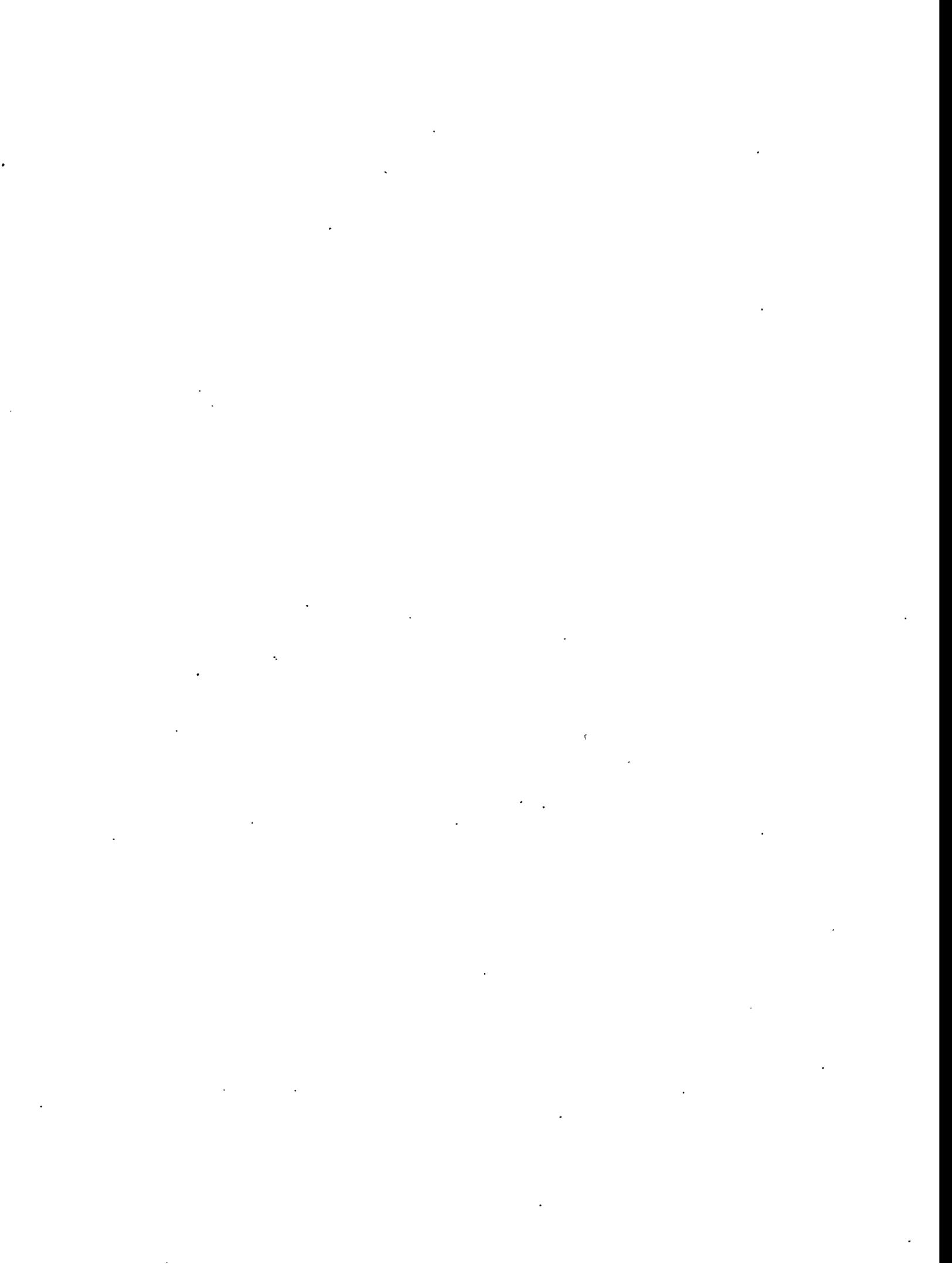


Table F.1. SUMMA Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6019-A05.032 (ppbv)	Flag	S6019-A06.057 (ppbv)	Flag	S6019-A07.S9 (ppbv)	Flag	Mean	St. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	0.52	U	1.8	J	3.4	J	2.6	1.2
CHLOROMETHANE	74-87-3	1.1	U	3.2	J	4.3	J	3.7	0.8
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	1.7	U		
VINYL CHLORIDE	75-01-4	0.57	U	0.57	U	0.57	U		
METHANOL	67-56-1	2236	Y	1899	Y	2758	Y	2297	433
BUTANE	106-97-8	0.92	U	5.6	J	3.5	J	4.6	1.5
BROMOMETHANE	74-83-9	1.2	U	1.2	U	1.2	U		
CHLOROETHANE	75-00-3	1.9	U	1.9	U	1.9	U		
ETHANOL	64-17-5	126	Y	113	Y	179	Y	139	35
ACETONITRILE	75-05-8	1140		1265		2223		1543	593
ACETONE	67-64-1	837		860		1557		1084	409
TRICHLOROFLUOROMETHANE	75-69-4	5.8	J	8.7		10		8.3	2.4
PENTANE	109-66-0	1.2	U	1.2	U	1.2	U		
1,1-DICHLOROETHENE	75-35-4	0.88	U	0.88	U	0.88	U		
METHYLENE CHLORIDE	75-09-2	0.53	U	3.0	J	3.9	J	3.5	0.63
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.48	U	0.48	U	0.48	U		
PROPANENITRILE	107-12-0	140		136		199		159	35
PROPANOL	71-23-8	18		14		20		17	3.1
1,1-DICHLOROETHANE	75-34-3	0.45	U	0.45	U	0.45	U		
2-BUTANONE	78-93-3	0.87	U	23		28		26	3.8
CIS-1,2-DICHLOROETHENE	156-59-2	0.79	U	0.79	U	0.79	U		
HEXANE	110-54-3	0.83	U	0.83	U	0.83	U		
CHLOROFORM	67-66-3	0.49	U	0.49	U	0.57	J	0.57	0.74
TETRAHYDROFURAN	109-99-9	0.50	U	0.50	U	0.74	J	0.74	
1,2-DICHLOROETHANE	107-06-2	0.59	U	0.59	U	0.59	U		
BUTANENITRILE	109-74-0	1.1	U	32		42		37	7.2
BENZENE	71-43-2	0.46	U	1.4	J	2.2	J	1.8	0.59
1,1,1-TRICHLOROETHANE	71-55-6	0.54	U	0.54	U	0.54	U		
1-BUTANOL	71-36-3	4.5	J	4.5	J	7.2	J	5.4	1.6
CARBON TETRACHLORIDE	56-23-5	0.50	U	0.50	U	0.50	U		
CYCLOHEXANE	110-82-7	0.96	U	0.96	U	0.96	U		
1,2-DICHLOROPROPANE	78-87-5	0.59	U	0.59	U	0.59	U		
TRICHLOROETHENE	79-01-6	0.41	U	0.41	U	0.41	U		
HEPTANE	142-82-5	0.40	U	0.40	U	0.85	J	0.85	
4-METHYL-2-PENTANONE	108-10-1	0.76	U	0.76	U	2.8	J	2.8	
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.48	U	0.48	U	0.48	U		
PYRIDINE	110-86-1	5.3	U	5.5	J	7.3	J	6.4	1.2
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.36	U	0.36	U	0.36	U		

Table F.1. SUMMA Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6019-A05.032 (ppbv)	Flag	S6019-A06.057 (ppbv)	Flag	S6019-A07.59 (ppbv)	Flag	Mean	St. Dev.
PENTANENITRILE	110-59-8	1.1	U	3.2	J	4.4	J	3.8	0.8
1,1,2-TRICHLOROETHANE	79-00-5	0.47	U	0.47	U	0.47	U		
TOLUENE	108-88-3	0.49	U	0.49	U	0.49	U		
1,2-DIBROMOETHANE	106-93-4	0.62	U	0.62	U	0.62	U		
OCTANE	111-65-9	0.47	U	0.47	U	0.47	U		
TETRACHLOROETHYLENE	127-18-4	0.57	U	0.57	U	0.57	U		
CHLOROBENZENE	108-90-7	0.52	U	0.52	U	0.52	U		
HEXANENITRILE	628-73-9	2.0	J	3.1	J	3.9	J	3.0	0.98
ETHYLBENZENE	100-41-4	0.59	U	0.59	U	0.59	U		
P-M-XYLENE	106-42-3	2.7	U	2.7	U	2.7	U		
CYCLOHEXANONE	108-94-1	1.3	U	1.3	U	1.3	U		
STYRENE	100-42-5	0.47	U	0.47	U	0.47	U		
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.56	U	0.56	U	0.60	J	0.60	
O-XYLENE	95-47-6	0.59	U	0.59	U	0.59	U		
NONANE	111-84-2	0.31	U	0.31	U	0.31	U		
1-ETHYL-2-METHYLBENZENE	611-14-3	0.34	U	0.34	U	0.34	U		
1,3,5-TRIMETHYLBENZENE	108-67-8	0.34	U	0.34	U	0.34	U		
1,2,4-TRIMETHYLBENZENE	95-63-6	0.48	U	0.48	U	0.48	U		
DECANE	124-18-5	0.47	U	0.63	J	0.47	U	0.63	
1,3-DICHLOROBENZENE	541-73-1	0.27	U	0.27	U	0.27	U		
1,4-DICHLOROBENZENE	106-46-7	0.27	U	0.27	U	0.27	U		
1,2-DICHLOROBENZENE	95-50-1	0.27	U	0.27	U	0.27	U		
UNDECANE	1120-21-4	0.37	U	1.0	J	1.2	J	1.1	0.1
1,2,4-TRICHLOROBENZENE	120-82-1	0.35	U	0.35	U	0.35	U		
DODECANE	112-40-3	1.3	U	1.3	U	1.3	U		
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.40	U	0.40	U	0.40	U		
TRIDECANE	629-50-5	0.73	U	0.73	U	0.81	J	0.81	
TETRADECANE	629-59-4	0.47	U	0.47	U	0.47	U		

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 F.2. Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6019-A08.800 (ppbv) Flag	S6019-A09.801 (ppbv) Flag	S6019-A10.802 (ppbv) Flag	S6019-A11.803 (ppbv) Flag	Mean	St. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	0.79 U	0.79 U	0.79 U	0.79 U		
CHLOROMETHANE	74-87-3	1.4 U	1.4 U	1.4 U	1.4 U		
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.41 U	0.41 U	0.41 U	0.41 U		
VINYL CHLORIDE	75-01-4	1.1 U	1.1 U	1.1 U	1.1 U		
METHANOL	67-56-1	1588 Y	936 Y	637 Y	949 Y	1027	401
BUTANE	106-97-8	1.4 U	1.4 U	3.4 J	3.3 J	3.4	0.13
CHLOROETHANE	75-00-3	9.2 J	24	8.1 J	8.6 J	13	7.8
ETHANOL	64-17-5	<133 Y	<133 Y	<133 Y	<133 Y		
ACETONITRILE	75-05-8	197	521	581	503	450	173
ACETONE	67-64-1	166	200	237	220	206	30
TRICHLOROFLUOROMETHANE	75-69-4	3.8 J	4.4 J	4.5 J	4.1 J	4.2	0.30
PENTANE	109-66-0	0.89 U	0.89 U	0.89 U	0.89 U		
1,1-DICHLOROETHENE	75-35-4	0.47 U	0.47 U	0.47 U	0.47 U		
METHYLENE CHLORIDE	75-09-2	11 J	38 J	25 J	29 J	26	11
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.36 U	0.36 U	0.36 U	0.36 U		
PROPANENITRILE	107-12-0	105	85	98	83	93	10
PROPANOL	71-23-8	4.1 J	9.1 J	20	15	12	6.9
1,1-DICHLOROETHANE	75-34-3	0.32 U	0.32 U	0.32 U	0.32 U		
2-BUTANONE	78-93-3	14	14	11	13	13	1.4
CIS-1,2-DICHLOROETHENE	156-59-2	0.58 U	0.58 U	0.58 U	0.58 U		
HEXANE	110-54-3	0.32 U	0.32 U	0.32 U	1.2 J	1.2	
CHLOROFORM	67-66-3	0.53 U	0.53 U	0.53 U	0.53 U		
TETRAHYDROFURAN	109-99-9	1.1 U	1.1 U	1.1 U	1.1 U		
1,2-DICHLOROETHANE	107-06-2	0.25 U	0.25 U	0.25 U	0.25 U		
BUTANENITRILE	109-74-0	36	34	36	32	34	2.0
BENZENE	71-43-2	3.3 J	6.4	2.7 J	7.4	5.0	2.3
1,1,1-TRICHLOROETHANE	71-55-6	0.44 U	0.44 U	0.44 U	0.44 U		
1-BUTANOL	71-36-3	8.2 J	9.4 J	6.6 J	5.8 J	7.5	1.6
CARBON TETRACHLORIDE	56-23-5	0.20 U	0.20 U	0.20 U	0.20 U		
CYCLOHEXANE	110-82-7	1.1 U	1.1 U	1.1 U	1.1 U		
1,2-DICHLOROPROPANE	78-87-5	0.38 U	0.38 U	0.38 U	0.38 U		
TRICHLOROETHENE	79-01-6	0.61 U	0.61 U	0.61 U	0.61 U		
HEPTANE	142-82-5	0.35 U	0.35 U	0.35 U	0.35 U		
4-METHYL-2-PENTANONE	108-10-1	1.4 J	3.0 J	6.5	5.1	4.0	2.3
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.25 U	0.25 U	0.25 U	0.25 U		
PYRIDINE	110-86-1	6.1 U	6.1 U	6.1 U	6.1 U		
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.53 U	0.53 U	0.53 U	0.53 U		
PENTANENITRILE	110-59-8	2.6	2.6	4.4	3.5	3.2	0.85
1,1,2-TRICHLOROETHANE	79-00-5	0.26 U	0.26 U	0.26 U	0.26 U		
TOLUENE	108-88-3	0.80 J	1.3 J	0.84 J	1.3 J	1.0	0.26

Table F.2. Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	C.A.S No.	S6019-A08.800 (ppbv) Flag	S6019-A09.801 (ppbv) Flag	S6019-A10.802 (ppbv) Flag	S6019-A11.803 (ppbv) Flag	Mean	St. Dev.
1,2-DIBROMOETHANE	106-93-4	0.28 U	0.28 U	0.28 U	0.28 U		
OCTANE	111-65-9	0.52 U	0.52 U	0.52 U	0.52 U		
TETRACHLOROETHYLENE	127-18-4	0.34 U	0.34 U	0.34 U	0.34 U		
CHLOROBENZENE	108-90-7	0.22 U	0.22 U	0.22 U	0.22 U		
HEXANENITRILE	628-73-9	3.2 J	3.3 J	3.4 J	2.9 J	3.2	0.23
ETHYLBENZENE	100-41-4	0.30 U	0.30 U	0.30 U	0.30 U		
P/M-XYLENE	106-42-3	0.63 U	0.63 U	0.63 U	0.63 U		
CYCLOHEXANONE	108-94-1	2.9 U	2.9 U	2.9 U	2.9 U		
STYRENE	100-42-5	0.33 U	0.33 U	0.33 U	0.33 U		
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.77 U	0.77 U	0.77 U	0.77 U		
O-XYLENE	95-47-6	0.40 U	0.40 U	0.40 U	0.40 U		
NONANE	111-84-2	0.22 U	0.22 U	0.22 U	0.22 U		
1-ETHYL-2-METHYL BENZENE	611-14-3	0.44 U	0.44 U	0.44 U	0.44 U		
1,3,5-TRIMETHYLBENZENE	108-67-8	0.44 U	0.44 U	0.44 U	0.44 U		
1,2,4-TRIMETHYLBENZENE	95-63-6	0.47 U	0.47 U	0.47 U	0.47 U		
DECANE	124-18-5	0.47 U	0.47 U	1.1 J	1.5 J	1.3	0.32
1,3-DICHLOROBENZENE	541-73-1	0.48 U	0.48 U	0.48 U	0.48 U		
1,4-DICHLOROBENZENE	106-46-7	0.43 U	0.43 U	0.43 U	0.43 U		
1,2-DICHLOROBENZENE	95-50-1	0.72 U	0.72 U	0.72 U	0.72 U		
UNDECANE	1120-21-4	2.6 J	0.63 U	3.1 J	3.3 J	3.0	0.40
1,2,4-TRICHLOROBENZENE	120-82-1	1.9 U	1.9 U	1.9 U	1.9 U		
DODECANE	112-40-3	3.9 J	4.0 J	3.9 J	4.4 J	4.0	0.27
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.8 U	1.8 U	1.8 U	1.8 U		
TRIDECANE	629-50-5	14 J	14 J	11 J	11 J	12	1.7
TETRADECANE	629-59-4	24	29 B	27 B	20	25	3.9
TBP	126-73-8	<0.83 Z	<0.83 Z	<0.83 Z	<0.83 Z		

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.

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 E.4 for more information.

Table F.3. SUMMA Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6020-A28.147		S6020-A29.149		S6020-A30.150		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
DICHLORODIFLUOROMETHANE	75-71-8	2.3	J	0.52	U	0.52	U	2.3	
CHLOROMETHANE	74-87-3	1.1	U	1.1	U	1.1	U		
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	1.7	U		
VINYL CHLORIDE	75-01-4	0.57	U	0.57	U	0.57	U		
METHANOL	67-56-1	1521	Y	1801	Y	1343	Y	1555	231.
BROMOMETHANE	74-83-9	1.2	U	1.2	U	1.2	U		
BUTANE	106-97-8	5.3	J	0.92	U	0.92	U	5.3	
CHLOROETHANE	75-00-3	1.9	U	1.9	U	1.9	U		
ETHANOL	64-17-5	85	Y	93	Y	71	Y	83	12
ACETONITRILE	75-05-8	1181		1198		1123		1167	40
ACETONE	67-64-1	864		835		733		810	69
TRICHLOROFUOROMETHANE	75-69-4	8.4		6.8	J	4.4	J	6.5	2.0
PENTANE	109-66-0	1.2	U	1.2	U	1.2	U		
1,1-DICHLOROETHENE	75-35-4	0.88	U	0.88	U	0.88	U		
METHYLENE CHLORIDE	75-09-2	3.3	J	0.53	U	0.53	U	3.3	
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.48	U	0.48	U	0.48	U		
PROPANENITRILE	107-12-0	117		142		128		129	13
PROPANOL	71-23-8	8.2	J	1.0	U	1.0	U	8.2	
1,1-DICHLOROETHANE	75-34-3	0.45	U	0.45	U	0.45	U		
2-BUTANONE	78-93-3	19		0.87	U	46		33	20
CIS-1,2-DICHLOROETHENE	156-59-2	1.3	J	0.79	U	0.79	U	1.3	
HEXANE	110-54-3	1.2	J	0.83	U	0.83	U	1.2	
CHLOROFORM	67-66-3	0.49	U	0.49	U	0.49	U		
TETRAHYDROFURAN	109-99-9	0.50	U	0.50	U	0.50	U		
1,2-DICHLOROETHANE	107-06-2	0.59	U	0.59	U	0.59	U		
BUTANENITRILE	109-74-0	30		1.1	U	1.1	U	30	
BENZENE	71-43-2	1.6	J	0.46	U	0.46	U	1.6	
1,1,1-TRICHLOROETHANE	71-55-6	0.57	J	0.54	U	0.54	U	0.57	
1-BUTANOL	71-36-3	4.2	J	0.97	U	0.97	U	4.2	
CARBON TETRACHLORIDE	56-23-5	0.50	U	0.50	U	0.50	U		
CYCLOHEXANE	110-82-7	27		0.96	U	0.96	U	27	
1,2-DICHLOROPROPANE	78-87-5	0.59	U	0.59	U	0.59	U		
TRICHLOROETHENE	79-01-6	0.41	U	0.41	U	0.41	U		
HEPTANE	142-82-5	8.4		0.40	U	6.4		7.4	1.4
4-METHYL-2-PENTANONE	108-10-1	1.9	J	0.76	U	0.76	U	1.9	

Table F.3. SUMMA Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6020-A28.147 (ppbv)	Flag	S6020-A29.149 (ppbv)	Flag	S6020-A30.150 (ppbv)	Flag	Mean	St. Dev.
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.48	U	0.48	U	0.48	U		
PYRIDINE	110-86-1	5.8	J	5.3	U	5.3	U	5.8	
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.36	U	0.36	U	0.36	U		
PENTANENITRILE	110-59-8	2.9	J	1.1	U	1.1	U	2.9	
1,1,2-TRICHLOROETHANE	79-00-5	0.47	U	0.47	U	0.47	U		
TOLUENE	108-88-3	2.2	J	2.3	J	2.3	J	2.3	0.056
1,2-DIBROMOETHANE	106-93-4	0.62	U	0.62	U	0.62	U		
OCTANE	111-65-9	0.47	U	0.47	U	0.47	U		
TETRACHLOROETHYLENE	127-18-4	0.57	U	0.57	U	0.57	U		
CHLOROBENZENE	108-90-7	0.52	U	0.52	U	0.52	U		
HEXANENITRILE	628-73-9	2.8	J	2.2	J	2.7	J	2.6	0.34
ETHYLBENZENE	100-41-4	0.59	U	0.59	U	0.59	U		
P/M-XYLENE	106-42-3	2.7	U	2.7	U	2.7	U		
CYCLOHEXANONE	108-94-1	1.3	U	1.3	U	1.3	U		
STYRENE	100-42-5	0.47	U	0.47	U	0.47	U		
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.56	U	0.56	U	0.56	U		
O-XYLENE	95-47-6	0.59	U	0.59	U	0.59	U		
NONANE	111-84-2	0.31	U	0.31	U	0.31	U		
1-ETHYL-2-METHYL BENZENE	611-14-3	0.39	J	0.34	U	0.34	U	0.39	
1,3,5-TRIMETHYLBENZENE	108-67-8	0.34	U	0.34	U	0.34	U		
1,2,4-TRIMETHYLBENZENE	95-63-6	0.48	U	0.48	U	0.48	U		
DECANE	124-18-5	3.8	J	1.1	J	0.47	U	2.4	1.9
1,3-DICHLOROBENZENE	541-73-1	0.33	J	0.27	U	0.27	U	0.33	
1,4-DICHLOROBENZENE	106-46-7	0.31	J	0.27	U	0.27	U	0.31	
1,2-DICHLOROBENZENE	95-50-1	0.36	J	0.27	U	0.27	U	0.36	
UNDECANE	1120-21-4	3.9		0.37	U	0.37	U	3.9	
1,2,4-TRICHLOROBENZENE	120-82-1	0.43	J	0.35	U	0.35	U	0.43	
DODECANE	112-40-3	1.3	U	1.3	U	1.3	U		
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.40	U	0.40	U	0.40	U		
TRIDECAENE	629-50-5	1.1	J	0.73	U	0.73	U	1.1	
TETRADECANE	629-59-4	0.47	U	0.47	U	0.47	U		

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 F.4 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6020-A31.816		S6020-A32.820		Mean
		(ppbv)	Flag	(ppbv)	Flag	
DICHLORODIFLUOROMETHANE	75-71-8	0.83	U	0.8	U	
CHLOROMETHANE	74-87-3	1.48	U	1.42	U	
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.44	U	0.42	U	
VINYL CHLORIDE	75-01-4	1.14	U	1.1	U	
METHANOL	67-56-1	616	Y	651	Y	634
BUTANE	106-97-8	3.3	J	3.3	J	3.3
CHLOROETHANE	75-00-3	16		14	J	15
ETHANOL	64-17-5	<133	Y	<133	Y	
ACETONITRILE	75-05-8	449		462		456
ACETONE	67-64-1	219		228		224
TRICHLOROFLUOROMETHANE	75-69-4	4.5	J	4.9	J	4.7
PENTANE	109-66-0	0.94	U	0.91	U	
1,1-DICHLOROETHENE	75-35-4	0.5	U	0.48	U	
METHYLENE CHLORIDE	75-09-2	11	J	19	J	15
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.38	U	0.37	U	
PROPANENITRILE	107-12-0	88		88		88
PROPANOL	71-23-8	17	J	17	J	17
1,1-DICHLOROETHANE	75-34-3	0.34	U	0.32	U	
2-BUTANONE	78-93-3	15		15		15
CIS-1,2-DICHLOROETHENE	156-59-2	0.61	U	0.59	U	
HEXANE	110-54-3	0.34	U	0.33	U	
CHLOROFORM	67-66-3	0.57	U	0.55	U	
TETRAHYDROFURAN	109-99-9	1.13	U	1.09	U	
1,2-DICHLOROETHANE	107-06-2	0.26	U	0.25	U	
BUTANENITRILE	109-74-0	32		32		32
1,1,1-TRICHLOROETHANE	71-55-6	0.47	U	0.45	U	
1-BUTANOL	71-36-3	7.2	J	6.8	J	7.0
BENZENE	71-43-2	2.3	J	2.5	J	2.4
CARBON TETRACHLORIDE	56-23-5	0.22	U	0.21	U	
CYCLOHEXANE	110-82-7	1.16	U	1.11	U	
1,2-DICHLOROPROPANE	78-87-5	0.4	U	0.38	U	
TRICHLOROETHENE	79-01-6	0.65	U	0.63	U	
HEPTANE	142-82-5	17		19		18
4-METHYL-2-PENTANONE	108-10-1	2.4	J	1.7	J	2.1
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.27	U	0.26	U	
PYRIDINE	110-86-1	6.4	U	6.17	U	
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.57	U	0.55	U	
PENTANENITRILE	110-59-8	3.2		3.4		3.3
1,1,2-TRICHLOROETHANE	79-00-5	0.28	U	0.27	U	
TOLUENE	108-88-3	56		68		62
1,2-DIBROMOETHANE	106-93-4	0.3	U	0.29	U	
OCTANE	111-65-9	1.7	J	0.53	U	1.1
TETRACHLOROETHYLENE	127-18-4	0.36	U	0.34	U	
CHLOROBENZENE	108-90-7	0.23	U	0.22	U	
HEXANENITRILE	628-73-9	2.0	J	2.2	J	2.1
ETHYLBENZENE	100-41-4	6.5		8.1		7.3
P/M-XYLENE	106-42-3	26		32		29
CYCLOHEXANONE	108-94-1	3.01	U	2.92	U	
STYRENE	100-42-5	2.3	J	2.7	J	2.5
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.82	U	0.79	U	
O-XYLENE	95-47-6	11		14		13
NONANE	111-84-2	2.1	J	2.6		2.4
1-ETHYL-2-METHYL BENZENE	611-14-3	3.4	J	4.7		4.1

**Table F.4 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank C-107 on 3/26/96**

Target Analytes	CAS No.	S6020-A31.816		S6020-A32.820		Mean
		(ppbv)	Flag	(ppbv)	Flag	
1,3,5-TRIMETHYLBENZENE	108-67-8	4.1	J	5.2		4.7
1,2,4-TRIMETHYLBENZENE	95-63-6	13		16		15
DECANE	124-18-5	4.9	J	6.3		5.6
1,3-DICHLOROBENZENE	541-73-1	0.51	U	0.49	U	
1,4-DICHLOROBENZENE	106-46-7	0.46	U	0.44	U	
1,2-DICHLOROBENZENE	95-50-1	0.76	U	0.73	U	
UNDECANE	1120-21-4	4.0	J	3.5	J	3.8
1,2,4-TRICHLOROBENZENE	120-82-1	2.02	U	1.95	U	
DODECANE	112-40-3	3.56	U	3.43	U	
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.86	U	1.8	U	
TRIDECANE	629-50-5	8.3	J	6.7	U	7.5
TETRADECANE	629-59-4	21		4.2	J	13
TBP	126-73-8	<0.88	Z	<0.85	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 E.4 for more information.

Table F.5. SUMMA™ Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	VSS				ISVS			
		S6019-A06.057		S6020-A29.149		S6019-A06.057		S6020-A29.149	
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	1.8	J	2.9	J	0.52	U	0.52	U
CHLOROMETHANE	74-87-3	3.2	J	4.9	J	1.1	U	1.1	U
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	1.7	U	1.7	U
VINYL CHLORIDE	75-01-4	0.57	U	0.57	U	0.57	U	0.57	U
METHANOL	67-56-1	1899	Y	2354	Y	1801	Y	1545	Y
BUTANE	106-97-8	5.6	J	7.8	J	0.92	U	0.92	U
BROMOMETHANE	74-83-9	1.2	U	1.2	U	1.2	U	1.2	U
CHLOROETHANE	75-00-3	1.9	U	1.9	U	1.9	U	1.9	U
ETHANOL	64-17-5	113	Y	134	Y	93	Y	75	Y
ACETONITRILE	75-05-8	1265		1656		1198		994	
ACETONE	67-64-1	860		1207		835		689	
TRICHLOROFLUOROMETHANE	75-69-4	8.7		9.8		6.8	J	5.1	J
PENTANE	109-66-0	1.2	U	1.2	U	1.2	U	1.2	U
1,1-DICHLOROETHENE	75-35-4	0.88	U	0.88	U	0.88	U	0.88	U
METHYLENE CHLORIDE	75-09-2	3.0	J	3.1	J	0.53	U	0.53	U
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.48	U	0.48	U	0.48	U	0.48	U
PROPANITRILE	107-12-0	136		181		142		126	
PROPANOL	71-23-8	14		18		1.0	U	1.0	U
1,1-DICHLOROETHANE	75-34-3	0.45	U	0.45	U	0.45	U	0.45	U
2-BUTANONE	78-93-3	23		25		0.87	U	0.87	U
CIS-1,2-DICHLOROETHENE	156-59-2	0.79	U	0.79	U	0.79	U	0.79	U
HEXANE	110-54-3	0.83	U	1.1	J	0.83	U	0.83	U
CHLOROFORM	67-66-3	0.49	U	0.65	J	0.49	U	0.49	U
TETRAHYDROFURAN	109-99-9	0.50	U	0.72	J	0.50	U	0.50	U
1,2-DICHLOROETHANE	107-06-2	0.59	U	0.59	U	0.59	U	0.59	U
BUTANENITRILE	109-74-0	32		43		1.1	U	1.1	U
BENZENE	71-43-2	1.4	J	2.1	J	0.46	U	0.46	U
1,1,1-TRICHLOROETHANE	71-55-6	0.54	U	0.54	U	0.54	U	0.54	U
1-BUTANOL	71-36-3	4.5	J	6.6	J	0.97	U	0.97	U
CARBON TETRACHLORIDE	56-23-5	0.50	U	0.50	U	0.50	U	0.50	U
CYCLOHEXANE	110-82-7	0.96	U	0.96	U	0.96	U	0.96	U
1,2-DICHLOROPROPANE	78-87-5	0.59	U	0.59	U	0.59	U	0.59	U
TRICHLOROETHENE	79-01-6	0.41	U	0.41	U	0.41	U	0.41	U
HEPTANE	142-82-5	0.40	U	0.98	J	0.40	U	6.2	
4-METHYL-2-PENTANONE	108-10-1	0.76	U	3.3	J	0.76	U	0.76	U
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.48	U	0.48	U	0.48	U	0.48	U
PYRIDINE	110-86-1	5.5	J	6.4	J	5.3	U	5.3	U
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.36	U	0.36	U	0.36	U	0.36	U
PENTANENITRILE	110-59-8	3.2	J	4.4	J	1.1	U	1.1	U
1,1,2-TRICHLOROETHANE	79-00-5	0.47	U	0.47	U	0.47	U	0.47	U
TOLUENE	108-88-3	0.49	U	0.49	U	2.3	J	1.6	J
1,2-DIBROMOETHANE	106-93-4	0.62	U	0.62	U	0.62	U	0.62	U
OCTANE	111-65-9	0.47	U	0.47	U	0.47	U	0.47	U
TETRACHLOROETHYLENE	127-18-4	0.57	U	0.57	U	0.57	U	0.57	U
CHLOROBENZENE	108-90-7	0.52	U	0.52	U	0.52	U	0.52	U
HEXANENITRILE	628-73-9	3.1	J	4.1	J	2.2	J	2.5	J
ETHYLBENZENE	100-41-4	0.59	U	0.59	U	0.59	U	0.59	U
P/M-XYLENE	106-42-3	2.7	U	2.7	U	2.7	U	2.7	U
CYCLOHEXANONE	108-94-1	1.3	U	1.3	U	1.3	U	1.3	U
STYRENE	100-42-5	0.47	U	0.47	U	0.47	U	0.47	U
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.56	U	0.63	J	0.56	U	0.56	U
O-XYLENE	95-47-6	0.59	U	0.59	U	0.59	U	0.59	U

Table F.5. SUMMA™ Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	VSS				ISVS			
		S6019-A06.057		S6020-A29.149					
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
NONANE	111-84-2	0.31	U	0.31	U	0.31	U	0.31	U
1-ETHYL-2-METHYL BENZENE	611-14-3	0.34	U	0.34	U	0.34	U	0.34	U
1,3,5-TRIMETHYLBENZENE	108-67-8	0.34	U	0.34	U	0.34	U	0.34	U
1,2,4-TRIMETHYLBENZENE	95-63-6	0.48	U	0.48	U	0.48	U	0.48	U
DECANE	124-18-5	0.63	J	0.82	J	1.1	J	0.47	U
1,3-DICHLOROBENZENE	541-73-1	0.27	U	0.27	U	0.27	U	0.27	U
1,4-DICHLOROBENZENE	106-46-7	0.27	U	0.27	U	0.27	U	0.27	U
1,2-DICHLOROBENZENE	95-50-1	0.27	U	0.27	U	0.27	U	0.27	U
UNDECANE	1120-21-4	1.0	J	1.2	J	0.37	U	0.37	U
1,2,4-TRICHLOROBENZENE	120-82-1	0.35	U	0.35	U	0.35	U	0.35	U
DODECANE	112-40-3	1.3	U	1.3	U	1.3	U	1.3	U
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.40	U	0.40	U	0.40	U	0.40	U
TRIDECANE	629-50-5	0.73	U	0.73	U	0.73	U	0.73	U
TETRADECANE	629-59-4	0.47	U	0.47	U	0.47	U	0.47	U

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 F.6. Triple Sorbent Trap Replicate Analysis Results for All Target Analytes for Samples Collected from from the Headspace of Tank C-107 on 3/26/96

Target Analytes	CAS No.	VSS				ISVS			
		S6019-A08.800		S6020-A32.820		S6019-A08.800		S6020-A32.820	
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	0.79	U	0.79	U	0.80	U	0.80	U
CHLOROMETHANE	74-87-3	1.4	U	1.4	U	1.4	U	1.4	U
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.41	U	0.41	U	0.42	U	0.42	U
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U	1.1	U	1.1	U
METHANOL	67-56-1	1588	Y	929	Y	651	Y	550	Y
BUTANE	106-97-8	1.4	U	2.3	J	3.3	J	3.0	J
CHLOROETHANE	75-00-3	9.2	J	8.2	J	14	J	5.4	J
ETHANOL	64-17-5	<133	Y	<133	Y	<133	Y	<133	Y
ACETONITRILE	75-05-8	197		539		462		426	
ACETONE	67-64-1	166		209		228		218	
TRICHLOROFLUOROMETHANE	75-69-4	3.8	J	3.6	J	4.9	J	4.8	J
PENTANE	109-66-0	0.89	U	0.89	U	0.91	U	0.91	U
1,1-DICHLOROETHENE	75-35-4	0.47	U	0.47	U	0.48	U	0.48	U
METHYLENE CHLORIDE	75-09-2	11	J	18	J	19	J	20	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.36	U	0.36	U	0.37	U	0.37	U
PROPANENITRILE	107-12-0	105		91		88		84	
PROPANOL	71-23-8	4.1	J	16	J	17	J	14	J
1,1-DICHLOROETHANE	75-34-3	0.32	U	0.32	U	0.32	U	0.32	U
2-BUTANONE	78-93-3	14		12		15		14	
CIS-1,2-DICHLOROETHENE	156-59-2	0.58	U	0.58	U	0.59	U	0.59	U
HEXANE	110-54-3	0.32	U	1.2	J	0.33	U	0.33	U
CHLOROFORM	67-66-3	0.53	U	0.53	U	0.55	U	0.55	U
TETRAHYDROFURAN	109-99-9	1.1	U	1.1	U	1.1	U	1.1	U
1,2-DICHLOROETHANE	107-06-2	0.25	U	0.25	U	0.25	U	0.25	U
BUTANENITRILE	109-74-0	36		32		32		30	
BENZENE	71-43-2	3.3	J	8.8		2.5	J	3.1	J
1,1,1-TRICHLOROETHANE	71-55-6	0.44	U	0.44	U	0.45	U	0.45	U
1-BUTANOL	71-36-3	8.2	J	6.0	J	6.8	J	6.5	J
CARBON TETRACHLORIDE	56-23-5	0.20	U	0.20	U	0.21	U	0.21	U
CYCLOHEXANE	110-82-7	1.1	U	1.1	U	1.1	U	1.1	U
1,2-DICHLOROPROPANE	78-87-5	0.38	U	0.38	U	0.38	U	0.38	U
TRICHLOROETHENE	79-01-6	0.61	U	0.61	U	0.63	U	0.63	U
HEPTANE	142-82-5	0.35	U	0.35	U	19		19	
4-METHYL-2-PENTANONE	108-10-1	1.4	J	2.2	J	1.7	J	1.7	J
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.25	U	0.25	U	0.26	U	0.26	U
PYRIDINE	110-86-1	6.1	U	6.1	U	6.2	U	6.2	U
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.53	U	0.53	U	0.55	U	0.55	U
PENTANENITRILE	110-59-8	2.6		3.1		3.4		3.2	
1,1,2-TRICHLOROETHANE	79-00-5	0.26	U	0.26	U	0.27	U	0.27	U
TOLUENE	108-88-3	0.80	J	1.4	J	68		68	
1,2-DIBROMOETHANE	106-93-4	0.28	U	0.28	U	0.29	U	0.29	U
OCTANE	111-65-9	0.52	U	0.52	U	0.53	U	2.1	J
TETRACHLOROETHYLENE	127-18-4	0.34	U	0.34	U	0.34	U	0.34	U
CHLOROBENZENE	108-90-7	0.22	U	0.22	U	0.22	U	0.22	U
HEXANENITRILE	628-73-9	3.2	J	3.1	J	2.2	J	2.6	J
ETHYLBENZENE	100-41-4	0.30	U	0.37	J	8.1		8.2	
P/M-XYLENE	106-42-3	0.63	U	0.63	U	32		32	
CYCLOHEXANONE	108-94-1	2.9	U	2.9	U	2.9	U	2.9	U
STYRENE	100-42-5	0.33	U	0.33	U	2.7	J	2.9	J
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.77	U	0.77	U	0.79	U	0.79	U
O-XYLENE	95-47-6	0.40	U	0.40	U	14		14	
NONANE	111-84-2	0.22	U	0.22	U	2.6		2.8	

Table F.6. Triple Sorbent Trap Replicate Analysis Results for All Target Analytes for Samples Collected from from the Headspace of Tank C-107 on 3/26/96

		VSS				ISVS			
		S6019-A08.800				S6020-A32.820			
1-ETHYL-2-METHYL BENZENE	611-14-3	0.44	U	0.44	U	4.7		4.5	
1,3,5-TRIMETHYLBENZENE	108-67-8	0.44	U	0.44	U	5.2		5.3	
1,2,4-TRIMETHYLBENZENE	95-63-6	0.47	U	0.47	U	16		16	
DECANE	124-18-5	0.47	U	0.47	U	6.3		6.1	
1,3-DICHLOROBENZENE	541-73-1	0.48	U	0.48	U	0.49	U	0.49	U
1,4-DICHLOROBENZENE	106-46-7	0.43	U	0.43	U	0.44	U	0.44	U
1,2-DICHLOROBENZENE	95-50-1	0.72	U	0.72	U	0.73	U	0.73	U
UNDECANE	1120-21-4	2.6	J	3.0	J	3.5	J	3.4	J
1,2,4-TRICHLOROBENZENE	120-82-1	1.9	U	1.9	U	2.0	U	2.0	U
DODECANE	112-40-3	3.9	J	4.3	J	3.4	U	3.4	U
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.8	U	1.8	U	1.8	U	1.8	U
TRIDECANE	629-50-5	14	J	14	J	6.7	U	6.7	U
TETRADECANE	629-59-4	24		20		4.2	J	3.3	J
TBP	126-73-8	<0.83	Z	<0.83	Z	<0.85	Z	<0.85	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 E.4 for more information.

Table F.7. SUMMA™ Blank Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6019-A03.026		S6019-A04.031		S6020-A27.095	
		Amb. Air VSS (ppbv)	Flag	Amb. Air VSS (ppbv)	Flag	Amb. Air VSS (ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	0.52	U	0.52	U	1.1	J
CHLOROMETHANE	74-87-3	1.1	U	1.1	U	1.4	J
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	0.85	U
METHANOL	67-56-1	<77	Y	<77	Y	<39	Y
VINYL CHLORIDE	75-01-4	0.57	U	0.57	U	0.29	U
BUTANE	106-97-8	0.92	U	0.92	U	0.46	U
BROMOMETHANE	74-83-9	1.2	U	1.2	U	0.61	U
CHLOROETHANE	75-00-3	1.9	U	1.9	U	0.94	U
ETHANOL	64-17-5	<53	Y	<53	Y	<27	Y
ACETONITRILE	75-05-8	2.2	U	2.2	U	1.1	U
ACETONE	67-64-1	31		29		18	
TRICHLOROFLUOROMETHANE	75-69-4	0.78	U	0.78	U	0.39	U
PENTANE	109-66-0	1.2	U	1.2	U	0.59	U
1,1-DICHLOROETHENE	75-35-4	0.88	U	0.88	U	0.44	U
METHYLENE CHLORIDE	75-09-2	0.53	U	0.53	U	2.4	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.48	U	0.48	U	0.24	U
PROPANOL	71-23-8	1.0	U	1.0	U	0.52	U
PROPANENITRILE	107-12-0	1.6	U	1.6	U	0.77	U
1,1-DICHLOROETHANE	75-34-3	0.45	U	0.45	U	0.23	U
2-BUTANONE	78-93-3	0.87	U	0.87	U	0.43	U
CIS-1,2-DICHLOROETHENE	156-59-2	0.79	U	0.79	U	0.59	J
HEXANE	110-54-3	0.83	U	0.83	U	0.42	U
CHLOROFORM	67-66-3	0.49	U	0.49	U	0.24	U
TETRAHYDROFURAN	109-99-9	0.50	U	0.50	U	0.25	U
1,2-DICHLOROETHANE	107-06-2	0.59	U	0.59	U	0.29	U
BUTANENITRILE	109-74-0	1.1	U	1.1	U	0.55	U
1,1,1-TRICHLOROETHANE	71-55-6	0.54	U	0.54	U	0.27	U
1-BUTANOL	71-36-3	0.97	U	0.97	U	0.48	U
BENZENE	71-43-2	0.46	U	0.46	U	0.85	J
CARBON TETRACHLORIDE	56-23-5	0.50	U	0.50	U	0.25	U
CYCLOHEXANE	110-82-7	0.96	U	0.96	U	0.48	U
1,2-DICHLOROPROPANE	78-87-5	0.59	U	0.59	U	0.30	U
TRICHLOROETHENE	79-01-6	0.41	U	0.41	U	0.20	U
HEPTANE	142-82-5	0.40	U	0.40	U	0.20	U
4-METHYL-2-PENTANONE	108-10-1	0.76	U	0.76	U	0.73	J
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.48	U	0.48	U	0.24	U
PYRIDINE	110-86-1	5.3	U	5.3	U	4.6	J

Table F.7. SUMMA™ Blank Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6019-A03.026		S6019-A04.031		S6020-A27.095	
		Amb. Air VSS (ppbv)	Flag	Amb. Air VSS (ppbv)	Flag	Amb. Air VSS (ppbv)	Flag
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.36	U	0.36	U	0.18	U
PENTANENITRILE	110-59-8	1.1	U	1.1	U	0.57	U
1,1,2-TRICHLOROETHANE	79-00-5	0.47	U	0.47	U	0.24	U
TOLUENE	108-88-3	0.49	U	0.49	U	0.24	U
1,2-DIBROMOETHANE	106-93-4	0.62	U	0.62	U	0.31	U
OCTANE	111-65-9	0.47	U	0.47	U	0.24	U
TETRACHLOROETHYLENE	127-18-4	0.57	U	0.57	U	0.28	U
CHLOROBENZENE	108-90-7	0.52	U	0.52	U	0.26	U
HEXANENITRILE	628-73-9	1.8	J	2.2	J	1.1	J
ETHYLBENZENE	100-41-4	0.59	U	0.59	U	0.30	U
P/M-XYLENE	106-42-3	2.7	U	2.7	U	1.4	U
CYCLOHEXANONE	108-94-1	1.3	U	1.3	U	0.66	U
STYRENE	100-42-5	0.47	U	0.47	U	0.24	U
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.56	U	0.56	U	0.28	U
O-XYLENE	95-47-6	0.59	U	0.59	U	0.30	U
NONANE	111-84-2	0.31	U	0.31	U	0.16	U
1-ETHYL-2-METHYLBENZENE	611-14-3	0.34	U	0.34	U	0.17	U
1,3,5-TRIMETHYLBENZENE	108-67-8	0.34	U	0.34	U	0.17	U
1,2,4-TRIMETHYLBENZENE	95-63-6	0.48	U	0.48	U	0.24	U
DECANE	124-18-5	0.47	U	0.47	U	0.32	J
1,3-DICHLOROBENZENE	541-73-1	0.27	U	0.27	U	0.14	U
1,4-DICHLOROBENZENE	106-46-7	0.27	U	0.27	U	0.14	U
1,2-DICHLOROBENZENE	95-50-1	0.27	U	0.27	U	0.14	U
UNDECANE	1120-21-4	0.37	U	0.37	U	0.19	U
1,2,4-TRICHLOROBENZENE	120-82-1	0.35	U	0.35	U	0.19	J
DODECANE	112-40-3	1.3	U	1.3	U	0.64	U
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.40	U	0.40	U	0.20	U
TRIDECANE	629-50-5	0.73	U	0.73	U	0.36	U
TETRADECANE	629-59-4	0.47	U	0.47	U	0.24	U

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 F.8. Triple Sorbent Trap Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6019-A12.805 VSS FB #1 (ppbv)	Flag	S6019-A13.812 VSS FB #2 (ppbv)	Flag	S6019-A14.813 VSS TB #1 (ppbv)	Flag	S6019-A15.815 VSS TB #2 (ppbv)	Flag	S6020-A40.861 ISVS FB #3 (ppbv)	Flag	S6020-A41.862 ISVS FB #4 (ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	0.79	U	0.79	U	0.79	U	0.79	U	0.79	U	0.79	U
CHLOROMETHANE	74-87-3	1.4	U	1.4	U	1.4	U	1.4	U	1.4	U	1.4	U
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.41	U	0.41	U	0.41	U	0.41	U	0.41	U	0.41	U
METHANOL	67-56-1	<192	Y	<192	Y	<192	Y	<192	Y	<192	Y	<192	Y
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U
BUTANE	106-97-8	1.4	U	1.4	U	1.4	U	1.4	U	1.4	U	1.4	U
CHLOROETHANE	75-00-3	1.4	U	1.4	U	1.4	U	1.4	U	1.4	U	1.4	U
ETHANOL	64-17-5	<133	Y	<133	Y	<133	Y	<133	Y	<133	Y	<133	Y
ACETONITRILE	75-05-8	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U
ACETONE	67-64-1	17	J	18	J	19	J	17	J	33		30	
TRICHLOROFLUOROMETHANE	75-69-4	0.72	U	0.72	U	0.72	U	0.72	U	0.72	U	0.72	U
PENTANE	109-66-0	0.89	U	0.89	U	0.89	U	0.89	U	0.89	U	0.89	U
1,1-DICHLOROETHENE	75-35-4	0.47	U	0.47	U	0.47	U	0.47	U	0.47	U	0.47	U
METHYLENE CHLORIDE	75-09-2	7.2	J	18	J	6.4	J	26	J	5.7	J	6.5	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.36	U	0.36	U	0.36	U	0.36	U	0.36	U	0.36	U
PROPANOL	71-23-8	2.0	U	2.0	U	2.0	U	2.0	U	2.0	U	2.0	U
PROPANENITRILE	107-12-0	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U
1,1-DICHLOROETHANE	75-34-3	0.32	U	0.32	U	0.32	U	0.32	U	0.32	U	0.32	U
2-BUTANONE	78-93-3	1.9	J	1.8	J	1.9	J	1.6	J	2.8	J	2.7	J
CIS-1,2-DICHLOROETHENE	156-59-2	0.58	U	0.58	U	0.58	U	0.58	U	0.58	U	0.58	U
HEXANE	110-54-3	0.32	U	0.32	U	0.32	U	0.32	U	0.32	U	0.32	U
CHLOROFORM	67-66-3	0.53	U	0.53	U	0.53	U	0.53	U	0.53	U	0.53	U
TETRAHYDROFURAN	109-99-9	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U
1,2-DICHLOROETHANE	107-06-2	0.25	U	0.25	U	0.25	U	0.25	U	0.25	U	0.25	U
BUTANENITRILE	109-74-0	0.45	U	0.45	U	0.45	U	0.45	U	0.45	U	0.45	U
1,1,1-TRICHLOROETHANE	71-55-6	0.44	U	0.44	U	0.44	U	0.44	U	0.44	U	0.44	U
1-BUTANOL	71-36-3	2.3	U	2.3	U	2.3	U	2.3	U	2.3	U	2.3	U
BENZENE	71-43-2	0.40	U	0.40	U	0.40	U	0.40	U	0.40	U	0.40	U
CARBON TETRACHLORIDE	56-23-5	0.20	U	0.20	U	0.20	U	0.20	U	0.20	U	0.20	U
CYCLOHEXANE	110-82-7	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U
1,2-DICHLOROPROPANE	78-87-5	0.38	U	0.38	U	0.38	U	0.38	U	0.38	U	0.38	U
TRICHLOROETHENE	79-01-6	0.61	U	0.61	U	0.61	U	0.61	U	0.61	U	0.61	U
HEPTANE	142-82-5	0.35	U	0.35	U	0.35	U	0.35	U	0.35	U	0.35	U
4-METHYL-2-PENTANONE	108-10-1	2.3	J	3.3	J	0.46	U	0.46	U	0.46	U	0.46	U
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.25	U	0.25	U	0.25	U	0.25	U	0.25	U	0.25	U
PYRIDINE	110-86-1	6.1	U	6.1	U	6.1	U	6.1	U	6.1	U	6.1	U
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.53	U	0.53	U	0.53	U	0.53	U	0.53	U	0.53	U

Table F.8. Triple Sorbent Trap Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank C-107 on 3/26/96

Target Analytes	CAS No.	S6019-A12.805 VSS FB #1 (ppbv)	Flag	S6019-A13.812 VSS FB #2 (ppbv)	Flag	S6019-A14.813 VSS TB #1 (ppbv)	Flag	S6019-A15.815 VSS TB #2 (ppbv)	Flag	S6020-A40.861 ISVS FB #3 (ppbv)	Flag	S6020-A41.862 ISVS FB #4 (ppbv)	Flag
PENTANENITRILE	110-59-8	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U
1,1,2-TRICHLOROETHANE	79-00-5	0.26	U	0.26	U	0.26	U	0.26	U	0.26	U	0.26	U
TOLUENE	108-88-3	0.23	U	0.23	U	0.23	U	0.23	U	0.27	U	0.29	U
1,2-DIBROMOETHANE	106-93-4	0.28	U	0.28	U	0.28	U	0.28	U	0.28	U	0.28	U
OCTANE	111-65-9	0.52	U	0.52	U	0.52	U	0.52	U	0.52	U	0.52	U
TETRACHLOROETHYLENE	127-18-4	0.34	U	0.34	U	0.34	U	0.34	U	0.34	U	0.34	U
CHLOROBENZENE	108-90-7	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U
HEXANENITRILE	628-73-9	0.66	U	0.66	U	0.66	U	0.66	U	0.66	U	0.66	U
ETHYLBENZENE	100-41-4	0.30	U	0.30	U	0.30	U	0.30	U	0.30	U	0.30	U
P/M-XYLENE	106-42-3	0.63	U	0.63	U	0.63	U	0.63	U	2.3	J	2.5	J
CYCLOHEXANONE	108-94-1	2.9	U	2.9	U	2.9	U	2.9	U	8.2	U	8.9	U
STYRENE	100-42-5	0.33	U	0.33	U	0.33	U	0.33	U	0.33	U	0.33	U
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U
O-XYLENE	95-47-6	0.40	U	0.40	U	0.40	U	0.40	U	3.2	J	3.5	J
NONANE	111-84-2	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U
1-ETHYL-2-METHYL BENZENE	611-14-3	0.44	U	0.44	U	0.44	U	0.44	U	1.8	J	1.9	J
1,3,5-TRIMETHYLBENZENE	108-67-8	0.44	U	0.44	U	0.44	U	0.44	U	0.86	J	0.91	J
1,2,4-TRIMETHYLBENZENE	95-63-6	0.47	U	0.47	U	0.47	U	0.47	U	2.7	J	3.0	J
DECANE	124-18-5	0.47	U	0.47	U	0.47	U	0.47	U	0.83	J	1.2	J
1,3-DICHLOROBENZENE	541-73-1	0.48	U	0.48	U	0.48	U	0.48	U	0.48	U	0.48	U
1,4-DICHLOROBENZENE	106-46-7	0.43	U	0.43	U	0.43	U	0.43	U	0.43	U	0.43	U
1,2-DICHLOROBENZENE	95-50-1	0.72	U	0.72	U	0.72	U	0.72	U	0.72	U	0.72	U
UNDECANE	1120-21-4	0.63	U	0.63	U	0.63	U	0.63	U	0.63	U	0.63	U
1,2,4-TRICHLOROBENZENE	120-82-1	1.9	U	1.9	U	1.9	U	1.9	U	1.9	U	1.9	U
DODECANE	112-40-3	3.4	U	3.4	U	3.4	U	3.4	U	3.4	U	3.4	U
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U
TRIDECANE	629-50-5	6.6	U	6.6	U	6.6	U	6.6	U	6.6	U	6.6	U
TETRADECANE	629-59-4	1.5	U	1.5	U	1.5	U	1.5	U	1.5	U	1.5	U
TBP	126-73-8	<0.83	Z	<0.83	Z	<0.83	Z	<0.83	Z	<0.83	Z	<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 E.4 for more information.

## **Appendix G**

### **Tank Vapor Characterization:**

### **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 S6019  
VSS

Collection date 03-26-96  
Preparation date 03-25-96

Ice Chest No.

Field Logbook No. WHC-N-547.16

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

S6019 - A17 . S16 Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
S6019 - A18 . S17 Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
S6019 - A19 . S18 Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
S6019 - A20 . S19 Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap

S6019 - A21 . S20 Open, close and store NH<sub>3</sub>/H<sub>2</sub>O field blank #1  
S6019 - A22 . S21 Open, close and store NH<sub>3</sub>/H<sub>2</sub>O 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	03-25-96	0920	J A Edwards	03-25-96	0920
J A Edwards	03-25-96	1430	K. J. Young	03-25-96	1430
K. J. Young	3-26-96	0700	R D Mahon	3-26-96	0700
R D Mahon	4-2-96	1200	T. B. White	4-2-96	1200
T. B. White	4-2-96	1505	J A Edwards	4-2-96	1505
J L. Julius	4-11-96	1130	G.W. Dennis	4-11-96	1130
G.W. Dennis	4-12-96	1130	K.H. Pool	4-12-96	1130

Comments:

Final Sample Disposition

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

POC  POC

(Revised 11/30/95 PNNL)

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 S6020  
Non-filtered ISVS  
Ice Chest No.

Collection date 03-25-96  
Preparation date 03-25-96

Field Logbook No. WHC-N-647-8

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

S6020 - A34 . S22 Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
S6020 - A35 . S23 Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
S6020 - A36 . S24 Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap

\* S6020 - A42 . S25 Store sample bundle NH<sub>3</sub>/H<sub>2</sub>O field blank #3  
\* S6020 - A43 . S26 Store sample bundle NH<sub>3</sub>/H<sub>2</sub>O field blank #4

[ ] 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>	03-25-96	0920	J A Edwards <i>J.A. Edwards</i>	03-25-96	0920
J A Edwards <i>J.A. Edwards</i>	03-25-96	1435	K.J. Yang <i>K.J. Yang</i>	03-25-96	1435
K.J. Yang <i>K.J. Yang</i>	3-26-96	0700	R.D. Mahon <i>R.D. Mahon</i>	3-26-96	0700
R.D. Mahon <i>R.D. Mahon</i>	4-2-96	1200	T. B. Utecht <i>T. B. Utecht</i>	4-2-96	1200
T. B. Utecht <i>T. B. Utecht</i>	4-2-96	1510	J.A. Edwards <i>J.A. Edwards</i>	4-2-96	1510
J.A. Edwards <i>J.A. Edwards</i>	4-11-96	1130	G.W. Dennis <i>G.W. Dennis</i>	4-11-96	1130
G.W. Dennis <i>G.W. Dennis</i>	4-12-96	1130	K.H. Pool <i>K.H. Pool</i>	4-12-96	1130

Final Sample Disposition

\* Comments: Tubes A42/A43 are labeled A40/A41 go by S#

PNNL (only) Checklist	Pick-up / Delivery	Comments:
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 / <input checked="" type="checkbox"/> Y/N	
COC info/signatures complete?	<input checked="" type="checkbox"/> N / <input checked="" type="checkbox"/> Y/N	
Rad release stickers on samples?	<input checked="" type="checkbox"/> N / <input checked="" type="checkbox"/> Y/N	
Activity report from 222S?	<input checked="" type="checkbox"/> N / <input checked="" type="checkbox"/> Y/N	
RSR/copy? (a ≤100/B ≤400 pCi/g)	<input checked="" type="checkbox"/> N / <input checked="" type="checkbox"/> Y/N	
COC copy for LRB, RIDS filed?	<input checked="" type="checkbox"/> N / <input checked="" type="checkbox"/> Y/N	

POC  POC

NEEDS RCT SURVEY  4/2/96 done 4-11-96

(Revised 11/30/95 PNNL)

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 S6019  
VSS

Collection date 03 - 26 - 96  
Preparation date 03 - 25 - 96

Ice Chest No.

Field Logbook No. WHC- N-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

S6019 - A03 . 026	Collect Ambient Air Sample, Upwind #1
S6019 - A04 . 031	Collect Ambient Air Sample, Through #2
S6019 - A05 . 032	Collect SUMMA #3
S6019 - A06 . 057	Collect SUMMA #4
S6019 - A07 . 059	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>	03-25-96	1330	<i>R.D. Mahon</i>	03-25-96	1330
<i>R.D. Mahon</i>	3-26-96	0700	<i>R.D. Mahon</i>	3-26-96	0700
<i>R.D. Mahon</i>	4-2-96	1200	<i>T.B. Utecht</i>	4-2-96	1200
<i>T.B. Utecht</i>	4-2-96	1505	<i>J.A. Edwards</i>	4-2-96	1505

Final Sample Disposition

Comments:

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

(Revised 11/30/95 PNNL)

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 Collection date 03 - 26 - 96  
241-C-107 Tank Vapor Sample SAF S6020 Preparation date 03 - 25 - 96  
Non-filtered ISVS  
Ice Chest No. Field Logbook No. WHC- N-647-8

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

S6020 - A27 . 095 Collect Ambient Air Sample, Upwind #1

S6020 - A28 . 147 Collect SUMMA #2  
S6020 - A29 . 149 Collect SUMMA #3  
S6020 - A30 . 150 Collect SUMMA #4

[ ] Field Transfer of Custody		[ X ] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time
J A Edwards <i>[Signature]</i>	03-25-96	1330	K.J. Young <i>[Signature]</i>	03-25-96	1330
K.J. Young <i>[Signature]</i>	4-2-96	1200	T.B. Utecht <i>[Signature]</i>	4-2-96	1200
T.B. Utecht <i>[Signature]</i>	4-2-96	1505	J.A. Edwards <i>[Signature]</i>	4-2-96	1505

Final Sample Disposition

Comments:

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

POC *[Signature]* POC *[Signature]*

(Revised 11/30/95 PNNL)

Custody Form Initiator J. A. Edwards - PNL  
 Company Contact R. D. Mahon - WHC  
 Project Designation/Sampling Locations 200 West Tank Farm  
 241-C-107 Tank Vapor Sample SAF S6019  
 (VSS Truck)  
 Ice Chest No.  
 Erco Hi/Lo thermometer No. PNL-T-004  
 Bill of Lading/Airbill No. N/A  
 Method of Shipment Government Truck  
 Shipped to WHC  
 Possible Sample Hazards/Remarks Unknown at time of sampling

Telephone (509) 373-0141  
 Page 85-3009 / P8-08 / FAX 376-0418  
 Telephone (509) 373-7437  
 Page 85-9656 / S3-27 / FAX 373-7076  
 Collection date 03-26-96  
 Preparation date 03-22-96  
 Field Logbook No. WHC-N-64710

Offsite Property No. N/A

Sample Identification

S6019 - A08 . 800 · Collect TST Sample # 1  
 S6019 - A09 . 801 · Collect TST Sample # 2  
 S6019 - A10 . 802 · Collect TST Sample # 3  
 S6019 - A11 . 803 · Collect TST Sample # 4  
  
 S6019 - A12 . 805 · Open, close & store TST Field Blank # 1 In VSS truck  
 S6019 - A13 . 812 · Open, close & store TST Field Blank # 2 In VSS truck  
  
 S6019 - A14 . 813 · Store TST Trip Blank # 1 N/A  
 S6019 - A15 . 815 · Store TST Trip Blank # 2 N/A

[ ] Field Transfer of Custody		[ X ] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time
JL Julva <i>Janet L Julva</i>	03-25-96	1230	J A Edwards <i>J A Edwards</i>	03-25-96	1230
J A Edwards <i>J A Edwards</i>	03-25-96	1330	K.S. Kern <i>K.S. Kern</i>	03-25-96	1330
K.S. Kern <i>K.S. Kern</i>	3-26-96	0700	R.D. Mahon <i>R.D. Mahon</i>	3-26-96	0700
R.D. Mahon <i>R.D. Mahon</i>	4-2-96	1200	J A Edwards <i>J A Edwards</i>	4-2-96	1505 <i>(1P)</i>
T.B. Utzell <i>T.B. Utzell</i>	4-2-96	1505	J A Edwards <i>J A Edwards</i>	4-2-96	1505
J A Edwards <i>J A Edwards</i>	4-12-96	0800	J.L. Julva <i>Janet L Julva</i>	4-12-96	0800 <i>6/11/96</i>

Final Sample Disposition

Comments:

- PNL (only) Checklist
- Media labeled and checked?  Y  N
  - Letter of instruction?  Y  N
  - Media in good condition?  Y  N
  - COC info/signatures complete?  Y  N
  - Sorbents shipped on ice? (<5°C)  Y  N
  - Hi/Lo thermometer - Keep upright!  Y  N
  - Hi/Lo thermometer  Y  N
  - Rad release stickers on samples?  Y  N
  - Activity report from 2225?  Y  N
  - RSR/copy? (a ≤100/B ≤400 pCi/g)  Y  N
  - COC copy for LRB, RIDS filed?  Y  N

Comments:

Cooler Temperature Status

Hi -15°C / Lo -15°C (pick up at PNL to WHC) |  
 Hi \_\_\_°C / Lo \_\_\_°C (delivery at WHC from PNL) |  
 Hi \_\_\_°C / Lo \_\_\_°C (at return to PNL from WHC) |  
 Hi +12°C / Lo -17°C (at delivery from WHC to PNL) |

OK BY RCT 4-11-96 *(initials)*

POC *(initials)* POC *(initials)*

(Revised 06/21/95 PNL)

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 Collection date 03 - 26 - 96  
241-C-107 Tank Vapor Sample SAF S6020 Preparation date 03 - 22 - 96  
(ISVS Cart)

Ice Chest No. Field Logbook No. WHC-N-647.8

Erco Hi/Lo thermometer No. PNL-T-004

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

S6020 - A31 . 816 Collect TST Sample # 1  
S6020 - A32 . 820 Collect TST Sample # 2  
S6020 - A33 . 824 Collect TST Sample # 3

S6020 - A40 . 861 Open, close & store TST Field Blank # 3  
S6020 - A41 . 862 Open, close & store TST Field Blank # 4

[ ] Field Transfer of Custody			[ X ] Chain of Possession (Sign and Print Names)		
Relinquished By	Date	Time	Received By	Date	Time
JL Julva <i>JL Julva</i>	03-25-96	1230	JA Edwards <i>JA Edwards</i>	03-25-96	1230
JA Edwards <i>JA Edwards</i>	03-25-96	1330	K.J. Young <i>K.J. Young</i>	03-25-96	1330
K.J. Young <i>K.J. Young</i>	3-26-96	0700	Rd Mahon <i>Rd Mahon</i>	3-26-96	0700
Rd Mahon <i>Rd Mahon</i>	4-2-96	1200	T. R. Lita <i>T. R. Lita</i>	4-2-96	1200
T. R. Lita <i>T. R. Lita</i>	4-2-96	1510	JA Edwards <i>JA Edwards</i>	4-2-96	1510
JA Edwards <i>JA Edwards</i>	4-12-96	0800	T. R. Lita <i>T. R. Lita</i>	4-12-96	0800

Final Sample Disposition

Comments:

- |   |                                   |  |
|---|-----------------------------------|--|
|   | PNL (only) Checklist              | Pick-up / Delivery                     |
| o | Media labeled and checked?        | <input checked="" type="checkbox"/> /N |
| o | Letter of instruction?            | Y/ <input checked="" type="checkbox"/> |
| o | Media in good condition?          | <input checked="" type="checkbox"/> /N |
| o | COC info/signatures complete?     | <input checked="" type="checkbox"/> /N |
| o | Sorbents shipped on ice? (-5°C)   | <input checked="" type="checkbox"/> /N |
| o | Hi/Lo thermometer - Keep upright! | <input checked="" type="checkbox"/> /N |
| o | Hi/Lo thermometer                 | Y/ <input checked="" type="checkbox"/> |
| o | Rad release stickers on samples?  | Y/ <input checked="" type="checkbox"/> |
| o | Activity report from 222S?        | Y/ <input checked="" type="checkbox"/> |
| o | RSR/copy? (a ≤100/B ≤400 pCi/g)   | Y/ <input checked="" type="checkbox"/> |
| o | COC copy for LRB, RIDS filed?     | Y/ <input checked="" type="checkbox"/> |

Comments:

S6020-A33.824 was broken during tube bundle disassembly and disposed of in the farm.

Cooler Temperature Status

Hi -15°C / Lo 15°C (pick up at PNL to WHC) |

Hi \_\_\_\_\_°C / Lo \_\_\_\_\_°C (delivery at WHC from PNL) |

Hi \_\_\_\_\_°C / Lo \_\_\_\_\_°C (at return to PNL from WHC) |

Hi +12°C / Lo -17°C (at delivery from WHC to PNL) |

OK BY RCT 4-11-96 (2)

POC (1/2) POC (1/2)

(Revised 06/21/95 PNL)

NEEDS RCT SURVEY @ 4/2/96

**Distribution List**

11269  
~~PNNL-11169~~

**PNNL**

Karl Pool	P8-08
Berta Thomas	P8-08
John Evans	K6-96
Khris Olsen	K6-96
Kurt Silvers	K9-08
Jon Fruchter	K6-96
Jim Huckaby	K6-80
Brenda Thornton	K6-80
Darlene Varley	K1-06
Katherine Savard	K9-04
Kris Walters	K6-80 (5 copies)

**Lockheed**

Larry Pennington	S7-21
Luther Buckley	R2-12

**DOE-RL**

Carol Babel	S7-54
Jim Thompson	S7-54