

# Pacific Northwest National Laboratory

Operated by Battelle for the  
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## TANK VAPOR CHARACTERIZATION PROJECT

### Headspace Vapor Characterization of Hanford Waste Tank 241-BY-108: Second Comparison Study Results from Samples Collected on 03/28/96

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Prepared for Westinghouse Hanford Company  
under a Related Services Agreement  
with the U.S. Department of Energy  
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### **Headspace Vapor Characterization of Hanford Waste Tank 241-BY-108: Second Comparison Study Results from Samples Collected on 3/28/96**

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## Summary

This report describes the analytical results of vapor samples taken from the headspace of waste storage tank 241-BY-108 (Tank BY-108) 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 ( $H_2O$ ) and ammonia ( $NH_3$ ), 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.



## **Acknowledgments**

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## 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



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## 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-BY-108 (Tank BY-108) 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 non-methane organic compounds (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 S6021 and S6022. Samples were collected by WHC on March 28, 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 16, 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),

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(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 BY-108 comparison study. Appendix G contains the completed COC forms.

## 2.0 Analytical Results

Samples obtained by WHC from the headspace of Tank BY-108 on March 28, 1996, (Sample Jobs S6021 and S6022) 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 BY-108 for the two sampling methods can be found in Appendix A. Table 2.1 presents the mean concentration values for these two analytes. Mean water concentration values ranged from 13.7 mg/L in the ISVS samples to 14.3 mg/L in the VSS samples. Mean  $\text{NH}_3$  concentration values ranged from 821 ppmv in the ISVS samples to 822 ppmv in the VSS samples.

**Table 2.1.** Comparison of Water and Ammonia Mean Values for Samples Collected from the Headspace of Tank BY-108 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
Water (mg/L)	14.3	13.7
Ammonia (ppmv)	822	821

### 2.2 Permanent Gases

The complete results of the permanent gas analyses of Tank BY-108 for the two sampling methods can be found in Appendix B. Table 2.2 presents the mean concentration values for the five permanent gases measured. Hydrogen ( $\text{H}_2$ ) and nitrous oxide ( $\text{N}_2\text{O}$ ) were measured above the analytical method estimated quantitation limit (EQL). Carbon dioxide ( $\text{CO}_2$ ) and methane ( $\text{CH}_4$ ) were observed just above the IDL for these gases. Carbon monoxide ( $\text{CO}$ ) was not observed in any of the samples. No significant differences were found in the mean concentrations of  $\text{H}_2$  and  $\text{N}_2\text{O}$  for the two different sampling methods.

**Table 2.2.** Comparison of Permanent Gas Mean Values for Samples Collected from the Headspace of Tank BY-108 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
H <sub>2</sub> (ppmv)	352	351
CO <sub>2</sub> (ppmv)	18 J	7.7 J
N <sub>2</sub> O (ppmv)	505	512
CH <sub>4</sub> (ppmv)	8.5 J	9.0 J
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 BY-108 can be found in Appendix C. A summary of those results can be found in Table 2.3. The TNMOC average concentrations ranged from 219 mg/m<sup>3</sup> in the ISVS samples to 243 mg/m<sup>3</sup> in the SUMMA™ samples.

**Table 2.3.** Comparison of TO-12 Mean Values for Samples Collected from the Headspace of Tank BY-108 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
TO-12 (mg/m <sup>3</sup> )	243	219

### 2.4 Organic Compounds from SUMMA™ Canisters

The complete results of the organic vapor analyses from SUMMA™ canisters from Tank BY-108 can be found in Appendices D and F. A summary of those results can be found in Table 2.4.

In summary, 1-butanol and methanol were the most abundant compounds identified in each of the SUMMA™ canister samples. 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, acetone, propanol, hexane, and 1-butanol were observed in the ISVS samples. The highest concentrations of acetonitrile, tetrahydrofuran, dodecane, tridecane, and tetradecane were observed in the VSS samples.

### 2.5 Organic Compounds from Triple Sorbent Traps

The complete results of the organic vapor analyses from TSTs from Tank BY-108 can be found in Appendices E and F. A summary of those results can be found in Table 2.5.

In summary, 1-butanol and methanol were the most abundant compounds identified in each of the trap samples. Tributyl phosphate was not observed in any samples from Tank BY-108. Based on the average values for each of the sampling methods the highest concentrations of methanol, ethanol, acetonitrile, propanol, hexane, and 1-butanol were observed in the ISVS samples. The highest concentrations of acetone, tetrahydrofuran, dodecane, tridecane and tetradecane were observed in the VSS samples.

**Table 2.4** Summary of SUMMA™ Sample Results for Samples Collected from the Headspace of Tank BY-108 on 3/28/96

	METHANOL		ETHANOL		ACETONITRILE		ACETONE		PROPANOL		TETRAHYDROFURAN		HEXANE		1-BUTANOL		DODECANE		TRIDECANE		TETRADECANE		TBP	
VSS Truck Samples	(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)	
Average	1722	Y	920	Y	131		1383		304		894		972		11859		503		572		356		Z	
ST DEV	1068		864		118		744		90		98		77		3116		90		110		152			
% RSD	62		94		90		54		30		11		7.9		26		18		19		43			
<b>ISVS</b>																								
Average	2133	Y	1115	Y	91		2465		396		893		1267		12778		350		349		155		Z	
ST DEV	818		595		46		1425		124		170		132		4480		59		96		38			
% RSD	38		53		50		58		31		19		10		35		17		28		25			

**Data Qualifier Flag**

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 2.5 Summary of Triple Sorbent Trap Sample Results for Samples Collected from the Headspace of Tank BY-108 on 3/28/96

VSS Truck Samples	ISVS			Data Qualifier Flag		
	Average	ST DEV	% RSD	Average	ST DEV	% RSD
METHANOL (ppbv)	1580 Y	253	16	1951 Y	66	3.4
ETHANOL (ppbv)	1169 Y	180	15	1289 Y	97	7.6
ACETONITRILE (ppbv)	268	36	14	286	26	9.0
ACETONE (ppbv)	1789 E	47	2.6	1709 E	107	6.3
PROPANOL (ppbv)	388	32	8.3	418	22	5.1
TETRAHYDROFURAN (ppbv)	665	12.2	1.8	617	12	1.9
HEXANE (ppbv)	1021	94	9.2	1041	48	4.6
1-BUTANOL (ppbv)	7227 E	144	2.0	7504 E	783	10
DODECANE (ppbv)	538	86	16	147	32	22
TRIDECANE (ppbv)	686 E	199	29	203	66	32
TETRADECANE (ppbv)	628 E	189	27	176 E	57	33
TBP (ppbv)	<0.8 Z			<0.8 Z		

E Target compound exceeds upper quantification limit (UQL).  
J Target compound detected above the IDL but below the FQL.  
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.



### 3.0 Conclusions

The air concentrations of  $\text{H}_2\text{O}$  and  $\text{NH}_3$ , permanent gases, total non-methane organic compounds, and organic vapors were determined from samples from the headspace of Tank BY-108 sampled on March 28, 1996. WHC sample job numbers were S6021 and S6022. The gas and vapor concentrations were based either on whole-volume samples (SUMMA<sup>TM</sup> 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

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(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  $\text{NH}_3$ -selective sorbent traps was placed into labeled 20-mL glass scintillation vials. Vials containing front-, or primary-, section sorbent material were treated with 10.0 mL of deionized water (DIW), and vials containing back-up-section sorbent material were treated with 5.0 mL of DIW. After extraction, the  $\text{NH}_3$  sorbent traps were analyzed using the selective ion electrode procedure PNL-ALO-226 Rev. 0<sup>(a)</sup>. Briefly, this method includes 1) preparing a 1000- $\mu\text{g/mL}$  (ppm)  $\text{NH}_3$  stock standard solution from dried reagent-grade  $\text{NH}_4\text{Cl}$  and DIW, 2) preparing 0.1-, 0.5-, 1.0-, 10-, and 100-ppm  $\text{NH}_3$  working calibration standards by serial dilution of the freshly made stock standard, 3) generating an initial calibration curve from the measured electromotive force signal versus  $\text{NH}_3$  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  $\text{NH}_4\text{Cl}$  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  $\text{NH}_3$  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 including PNL-MA-70 (Part 3), PNL-ALO-212, PNL-ALO-226, and Quality Assurance Plan ETD-002. The samples were analyzed following PNNL Impact Level II. 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 3 L and a desorption-solution volume of 3 mL (10 mL for  $\text{NH}_3$ ).

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

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(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  $\text{NH}_3$  analyses, the accuracy of laboratory measurements by selective ion electrode was estimated to be  $\pm 5\%$  relative, independent of concentration at  $1 \mu\text{g/mL}$  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>

Analyte	Formula	Procedure	EQL <sup>(b)</sup> ( $\mu\text{g}$ )	EQL <sup>(b)</sup> (ppmv)	Notification Level <sup>(c)</sup> (ppmv)
Ammonia	$\text{NH}_3$	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  $\pm 0.1 \text{ mg}$ , 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  $\pm 1 \text{ mg}$  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 BY-108. These samples were collected through the VSS and through the ISVS systems. A total of 11 samples were collected with the two different sampling methods. The samples were analyzed for ammonia and water on April 25, 1996. Mean water concentration values ranged from  $13.7 \text{ mg/L}$  in the ISVS samples to  $14.3 \text{ mg/L}$  in the VSS samples. Mean ammonia concentration values ranged from 821 ppmv in the ISVS samples to 822 ppmv in the VSS samples.

**Table A.2** Water and Ammonia Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 3/28/96

VSS Truck Samples	H <sub>2</sub> O mg/m <sup>3</sup>	NH <sub>3</sub> ppmv
S6021-A17.S27	14.2	814
S6021-A18.S28	14.2	821
S6021-A19.S29	14.4	821
S6021-A20.S30	14.3	830
Average	14.3	822
% RSD	0.7	0.8

<b>ISVS</b>		
S6022-A34.S33	13.6	835
S6022-A35.S34	13.7	806
S6022-A36.S35	13.9	822
Average	13.7	821
% RSD	1.1	1.8

## 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.

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(a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, 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 BY-108, 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 BY-108 and ambient air collected near Tank BY-108. 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 19 and 22, 1996. Replicate analyses on SUMMA™ canisters were conducted on one sample within each sampling method set. Hydrogen and nitrous oxide were observed above the EQL in all of the tank headspace samples. Average hydrogen concentrations ranged from 351 ppmv in the ISVS samples to 352 ppmv in the VSS samples. Average nitrous oxide concentrations ranged from 505 ppmv in the VSS samples to 512 ppmv in the ISVS samples. Carbon monoxide concentrations were below the IDL in all of the tank samples. Carbon dioxide and methane concentrations were just above the IDL in several tank samples.

**Table B.2** Permant Gas Analysis Results for Samples Collected from the Headspace of Tank BY-108 and Ambient Air Collected Near Tank BY-108 on 3/28/96

VSS Truck Samples	H <sub>2</sub> (ppmv)	Flag	CO <sub>2</sub> (ppmv)	Flag	N <sub>2</sub> O (ppmv)	Flag	CH <sub>4</sub> (ppmv)	Flag	CO (ppmv)	Flag
S6021-A03.062 (Ambient)	3.1	U	350		2.0	U	4.3	U	3.2	U
S6021-A04.157 (Ambient)	3.1	U	346		2.0	U	4.3	U	3.2	U
S6021-A05.212	353		26		499		7.8	J	3.2	U
S6021-A06.215	352		20	J	502		4.3	U	3.2	U
S6021-A07.220	352		8.0	J	514		9.2	J	3.2	U
Average	352		18	J	505		8.5	J	3.2	U
% RSD	0.1		50		1.6		NA			
S6021-A05.212 (REP)	351		23	J	504		4.3	U	3.2	U
<b>ISVS</b>										
S6022-A27.227 (Ambient)	3.1	U	365		2.0	U	4.3	U	3.2	U
S6022-A30.247	345		7.2	J	513		4.3	U	3.2	U
S6022-A29.244	355		7.9	J	515		9.5	J	3.2	U
S6022-A28.232	352		7.9	J	510		4.3	U	3.2	U
Average	351		7.7	J	512		9.0	J	3.2	U
% RSD	1.5		5.7		0.5		NA			
S6022-A29.244 (REP)	356		6.9	J	515		4.3	U	3.2	U

**Data Qualifier Flags**

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

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 compound (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.

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- (a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, 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 BY-108 and ambient air collected near Tank BY-108. These samples were collected through the VSS and ISVS. A total of nine samples were collected for the two sampling methods. The samples were analyzed on May 7, 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 0.17 mg/m<sup>3</sup> to 0.49 mg/m<sup>3</sup>. Average concentrations in the tank samples ranged from 219 mg/m<sup>3</sup> in the ISVS samples to 243 mg/m<sup>3</sup> in the VSS samples.

**Table C.1** TO-12 Analysis Results for Samples Collected from the Headspace of Ta and Ambient Air Near Tank BY-108 on 3/28/96

VSS Truck Samples	TO-12 mg/m <sup>3</sup>	Flag
S6021-A03.062 (Ambient)	0.38	J
S6021-A04.157 (Ambient)	0.17	J
S6021-A05.212	245	
S6021-A06.215	245	
S6021-A07.220	239	
Average	243	
% RSD	1.5	
S6021-A05.212 (REP)	242	
<b>ISVS</b>		
S6022-A27.227 (Ambient)	0.49	J
S6022-A30.247	218	
S6022-A29.244	219	
S6022-A28.232	220	
Average	219	
% RSD	0.5	
S6022-A29.244 (REP)	218	

**Data Qualifier Flags**

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

## **Appendix D**

### **Tank Vapor Characterization:**

### **Organic Compounds from SUMMA<sup>TM</sup> 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

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- (a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, Richland, Washington.
- (b) Pacific Northwest Laboratory. 2/95. *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.

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

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(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.



**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 16, 1996, under WHC CQC numbers 100052 and 100053. Samples were analyzed on August 16, 20, and 21, and September 20, 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 from the different sampling methods 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. The compounds 1-butanol and methanol were the most abundant compounds identified in each of the SUMMA™ canister samples. 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, acetone, propanol, hexane, and 1-butanol were observed in the ISVS samples. The highest concentrations of acetonitrile, tetrahydrofuran, dodecane, tridecane, and tetradecane were observed in the VSS samples.

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 compounds consistently observed in the blank samples were ethanol and acetone. Traces of methanol, acetonitrile, propanol, tetrahydrofuran, hexane, 1-butanol, dodecane, and tridecane were observed in several of the ambient air samples. However, these levels were significantly lower than concentrations observed in the tank samples.

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

Batch #1 (File Identifier # 16081601 & 2.b) - S6021-A03.062, S6021-A04.157, S6022-A27.227, and S6021-A07.220;

Batch #2 (file Identifier # 16082001 & 2.b) - S6022-A29.244, S6021-A06.215, S6021-A05.212, and S6022-A30.247;

Batch #3 (file Identifier # 16082101 & 2.b) - S6022-A28.232, S6021-A07.220 REP, and S6022-A29.244 REP;

Batch #4 (file Identifier # 16092001 & 2.b) - S6021-A06.215, S6021-A07.220 REP, S6021-A05.212, S6022-A28.232, and S6022-A29.244.

The following procedural changes and observations were noted during the analysis of Tank BY-108:

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.

This analytical sequence was run using 20 ml volumes to quantify target compounds in each tank sample. Relatively small volumes of samples were used due to high concentration of pollutants in this tank.

Eight target compounds (dichlorodifluoroethane at 31.64%, 1,2-dichloro-1,1,2,2-tetrafluoroethane at 48.53%, ethanol at 38.04%, propanol at 34.68%, butanenitrile at 36.95%, 4-methyl-2-pentanone at 37.73%, pyridine at 45.95%, and 1,1,2,2-tetrachloroethane at 43.15%) surpassed the 30% (%RSD) acceptance criteria for the initial calibration.

The compounds 1,2-dichloro-1,1,2,2-tetrafluoroethane, pyridine and butanenitrile were found in all the samples at concentrations between the IDL and the EQL. Dichlorodifluoroethane and 4-methyl-2-pentanone were found in all samples at concentrations between the IDL and the EQL, with the exception of sample S6021-A04.157, in which dichlorodifluoroethane was found at a concentration between the EQL and the upper quantitation limit (UQL), and tank sample S6021-A07.220, in which 4-methyl-2-pentanone was found at a concentration between the EQL and the UQL. Propanol was found in samples S6021-A04.157 and S6022-A27.227 at concentrations between the IDL and the EQL and in samples S6021-A03.062 and S6021-A07.220 at concentrations between the EQL and the UQL. Ethanol and 1,1,2,2-tetrachloroethane were found in all samples at concentrations between the EQL and the UQL.

Four target compounds (1,2-dichloro-1,1,2,2-tetrafluoroethane at 32.5%, ethanol at 57.5%, 1,1,2-trichloroethane at 39.9%, and 1,1,2,2-tetrachloroethane at 48.3%) were outside the

25% difference (% D) acceptance criteria for the continuing calibration verification (CCV) sample. However, the CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of all target compounds.

The compound 1,2-dichloro-1,1,2,2-tetrafluoroethane, as was mentioned above, was found in all samples at concentrations between the IDL and the EQL. Ethanol and 1,1,2,2-tetrachloroethane, as mentioned above, were found in tank samples at concentrations between EQL and UQL. The compound 1,1,2-trichloroethane was found in all samples at concentrations between the IDL and the EQL, with the exception of tank sample S6021-A07.220, in which it was not found above the IDL.

Twenty-one target compounds (dichlorodifluoromethane, vinyl chloride, butane, ethanol, acetone, propanol, methylene chloride, 1,1-dichloroethane, 2-butanone, 1-butanol, benzene, chloroform, heptane, 1,1,2,2-tetrachloroethane, 1-ethyl-2-methylbenzene, 1,3,5-trimethylbenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2-dichlorobenzene, decane and 1,2,4-trichlorobenzene) were found in the continuing calibration blank (CCB) above its EQLs but their concentrations, with the exceptions of butane and acetone, were less than 3.6 ppbv. This contamination took place because the samples from this tank were analyzed on this system before this sequence and contained a high concentrations of some of these compounds. Target compounds butane, acetone, methylene chloride, 1-butanol, heptane, 1,1,2-trichloroethane, chlorobenzene, 1,3,5-trimethylbenzene, 1,1,2,2-tetrachloroethane, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2-dichlorobenzene, hexachloro-1,3-butadiene and 1,2,4-trichlorobenzene were found in the initial calibration blank (ICB) above the EQLs, but the concentrations were less than 2.5 ppbv.

After completing a valid bromofluorobenzene (BFB) tune, the 12-hour clock criterion for an analytical sequence was exceeded by three minutes.

The tank sample internal standard quantification area percent recoveries were within the acceptance criterion (50% to 200%) allowed by procedure PNL-TVP-03, Rev. 1, except for blank runs (16081608.d and 16081610.d).

Batch #2:

This analytical sequence was run using 20 ml volumes to quantify target compounds in each tank sample. Relatively small volume of samples was used due to high concentration of pollutants in this tank.

Eight target compounds (dichlorodifluoroethane at 31.64%, 1,2-dichloro-1,1,2,2-tetrafluoroethane at 48.53%, ethanol at 38.04%, propanol at 34.68%, butanenitrile at 36.95%, 4-methyl-2-pentanone at 37.73%, pyridine at 45.95%, and 1,1,2,2-tetrachloroethane at 43.15%) surpassed the 30% RSD acceptance criteria for the initial calibration.

Dichlorodifluoroethane, 1,2-dichloro-1,1,2,2-tetrafluoroethane, pyridine and butanenitrile were found in all the samples at concentrations between the IDL and the EQL, with the exception of butanenitrile which was not found in tank sample S6022-A29.244.

Ethanol, propanol, 4-methyl-2-pentanone and 1,1,2,2-tetrachloroethane were found in all the tank samples at concentrations between the EQL and the UQL.

Six target compounds (1,2-dichloro-1,1,2,2-tetrafluoroethane at 38.7%, ethanol at 45.3%, vinyl chloride at 34.2%, butane at 27.7%, bromomethane at 31.0%, and 1,1,2,2-tetrachloroethane at 53.1%) were outside the 25% D acceptance criteria for the CCV sample. However, the CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of all target compounds.

The compound 1,2-dichloro-1,1,2,2-tetrafluoroethane, as mentioned above, and vinyl chloride and bromomethane were found in all the samples at concentrations between the IDL and the EQL. Ethanol and 1,1,2,2-tetrachloroethane, as mentioned above, were found in all the tank samples at concentrations between the EQL and the UQL. Butane was found in all the samples at concentrations between the IDL and the EQL, with the exception of tank sample S6022-A29.244, in which it was found above the UQL.

Twenty nine target compounds (dichlorodifluoromethane, vinyl chloride, butane, acetone, methylene chloride, 1,1,2-trichloro-1,2,2-trifluoroethane, 1,1-dichloroethane, 2-butanone, chloroform, tetrahydrofuran, butanenitrile, 1,1,1-trichloroethane, 1-butanol, benzene, carbon tetrachloride, trichloroethane, heptane, 4-methyl-2-pentanone, trans-1,2-dichloropropene, pentanenitrile, 1,1,2,2-tetrachloroethane, nonane, 1-ethyl-2-methylbenzene, 1,3,5-trimethylbenzene, 1,3-dichlorobenzene, decane, 1,4-dichlorobenzene, 1,2-dichlorobenzene and 1,2,4-trichlorobenzene) were found in the CCB above the EQLs, but the concentrations, with the exception of butane (5.22 ppbv), were less than 4.0 ppbv. This contamination took place because the samples from this tank were analyzed on this system before this sequence and contained high concentrations of some of these compounds.

Fourteen target compounds (butane, acetone, methylene chloride, 1-butanol, heptane, 1,1,2-trichloroethane, chlorobenzene, 1,3,5-trimethylbenzene, 1,1,2,2-tetrachloroethane, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2-dichlorobenzene, hexachloro-1,3-butadiene and 1,2,4-trichlorobenzene) were found in the ICB above the EQLs, but the concentrations were less than 2.5 ppbv.

The internal standard quantification area percent recoveries for tank samples and blanks exceeded the acceptance criterion (50% to 200%) allowed by procedure PNL-TVP-03, Rev. 1. The changes in the internal standard areas was caused by water induced fatigue. This problem is routinely observed with the HP5972 GC/MS system because of its poor pumping capacity. 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 #3:

This analytical sequence was run using 20 ml volumes to quantify target compounds in each tank sample. Relatively small volumes of samples were used due to high concentration of pollutants in this tank.

Eight target compounds (dichlorodifluoroethane at 31.64%, 1,2-dichloro-1,1,2,2-tetrafluoroethane at 48.53%, ethanol at 38.04%, propanol at 34.68%, butanenitrile at 36.95%, 4-methyl-2-pentanone at 37.73%, pyridine at 45.95%, and

1,1,2,2-tetrachloroethane at 43.15%) surpassed the 30% RSD acceptance criteria for the initial calibration.

Dichlorodifluoroethane, 1,2-dichloro-1,1,2,2-tetrafluoroethane, pyridine and butanenitrile were found in all the tank samples at concentrations between the IDL and the EQL.

Ethanol, propanol, 4-methyl-2-pentanone and 1,1,2,2-tetrachloroethane were found in all the tank samples at concentrations between the EQL and the UQL.

Nine target compounds (dichlorodifluoromethane at 49.5%, 1,2-dichloro-1,1,2,2-tetrafluoroethane at 60.6%, chloroethane at 37.3%, ethanol at 56.9%, 1-butanol at 32.6%, pyridine at 27.5%, pentanitrile at 40.0%, 1,1,2,2-tetrachloroethane at 60.7%, decane at 31.9%) were outside the 25% D acceptance criteria for the CCV sample. However, the CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of all target compounds.

Dichlorodifluoromethane, 1,2-dichloro-1,1,2,2-tetrafluoroethane and pyridine, as mentioned above, and chloroethane were found in all the samples at concentrations between the IDL and the EQL. Ethanol and 1,1,2,2-tetrachloroethane, as mentioned above, and decane were found in all the tank samples at concentrations between the EQL and the UQL. The compound 1-butanol was found in all the samples at concentrations above the UQL.

Target compounds dichlorodifluoromethane, butane, acetone, methylene chloride, 1,1,2-trichloro-1,1,2-trifluoroethane, 1,1-dichloroethane, 2-butanone, 1-butanol, benzene, heptane, trans-1,2-dichloropropene, 1,1,2-trichloroethane, 1,1,2,2-tetrachloroethane, nonane, 1-ethyl-2-methylbenzene, 1,3,5-trimethylbenzene, 1,3-dichlorobenzene, decane, 1,4-dichlorobenzene, 1,2-dichlorobenzene, and 1,2,4-trichlorobenzene were found in the CCB above the EQLs but the concentrations, with the exception of butane (7.26 ppb), were less than 4.0 ppb. This contamination took place because the samples from this tank were analyzed on this system before this sequence and contained high concentrations of some of these compounds. Target compounds (butane, acetone, methylene chloride, 1-butanol, heptane, 1,1,2-trichloroethane, chlorobenzene, 1,3,5-trimethylbenzene, 1,1,2,2-tetrachloroethane, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2-dichlorobenzene, hexachloro-1,3-butadiene and 1,2,4-trichlorobenzene) were found in the ICB above the EQLs, but its concentration was less than 2.5 ppb.

#### Batch #4:

This analytical sequence was run using 30 mL volumes of diluted 1:5 v/v samples to quantify target compounds in each tank sample. Relatively small volume of sample was used due to high concentration of pollutants in this tank.

Four target compounds (1,2,4-trichlorobenzene at 40.33%, dodecane at 39.76%, tridecane at 53.38%, and tetradecane at 69.20%) surpassed the 30% RSD acceptance criteria for the initial calibration. The compound 1,2,4-trichlorobenzene was not found in the tank samples at concentrations above the IDL. Dodecane, tridecane, and tetradecane were found in all tank samples at concentrations between the EQL and the UQL.

Seven target compounds (vinyl chloride at 39.7%, 1,3-butadiene at 30.1%, bromomethane at 68.8%, undecane at 37.7%, dodecane at 41.4%, tridecane at 58.0%, and tetradecane at 82.1%) were outside the  $\pm 25\%$  D acceptance criteria for the CCV sample. However, the CCV passed the procedural criteria requiring  $\pm 25\%$  D passage for 85% of all target compounds. Dodecane, tridecane, tetradecane, undecane, and 1,3-butadiene were found in all the tank samples at concentrations between the EQL and UQL. Vinyl chloride and bromomethane were not found in the tank samples at concentrations above their IDLs.

Target compound 1,2,4-trichlorobenzene was found in the CCB above the EQL, but as mentioned above it was not found in the tank samples. This compound was also found in the ICB above the EQL.



Table D.2 SUMMA™ Sample Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 3/28/96

VSS Truck Samples	METHANOL		ETHANOL		ACETONITRILE		ACETONE		PROPANOL		TETRAHYDROFURAN		HEXANE		1-BUTANOL		DODECANE		TRIDECANE		TETRADECANE		TBP	
	(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)	
A05.212	1091	Y	405	Y	28		529		234		917		973		13946		459		500		261		Z	
A06.215	1121	Y	438	Y	105		1736		273		978		895		13354		444		517		276		Z	
A07.220	2955	Y	1918	Y	260		1885		406		787		1048		8278		606		698		531		Z	
Average	1722	Y	920	Y	131		1383		304		894		972		11859		503		572		356		Z	
ST DEV	1068		864		118		744		90		98		77		3116		90		110		152			
% RSD	62		94		90		54		30		11		7.9		26		18		19		43			
ISVS																								
A28.232	2154	Y	1651	Y	138		3486		429		954		1181		17060		286		263		127		Z	
A29.244	2940	Y	1220	Y	90		3072		501		701		1419		13150		363		330		140		Z	
A30.247	1305	Y	474	Y	46		837		259		1025		1200		8123		401		453		199		Z	
Average	2133	Y	1115	Y	91		2465		396		893		1267		12778		350		349		155		Z	
ST DEV	818		595		46		1425		124		170		132		4480		59		96		38			
% RSD	38		53		50		58		31		19		10		35		17		28		25			

Data Qualifier Flag

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.3** Replicate Analysis of SUMMA™ Canisters for Samples Collected from the Headspace of Tank BY-108 on 3/28/96

	METHANOL		ETHANOL		ACETONITRILE		ACETONE	PROPANOL	TETRAHYDROFURAN	HEXANE	I-BUTANOL	DODECANE	TRIDECANE	TETRADECANE	TBP
VSS Truck Samples	(ppbv)		(ppbv)		(ppbv)		(ppbv)	(ppbv)	(ppbv)	(ppbv)	(ppbv)	(ppbv)	(ppbv)	(ppbv)	(ppbv)
A07.220	2955	Y	1918	Y	260		1885	406	787	1048	8278	606	698	531	Z
A07.220 REP	2113	Y	887	Y	127		3268	410	933	1074	13792	412	391	192	Z
Relative Percent Difference	33		74		69		54	1.0	17	2.5	50	38	56	94	
<b>ISVS</b>															
A29.244	2940	Y	1220	Y	90		3072	501	701	1419	13150	363	330	140	Z
A29.244 Rep	1570	Y	611	Y	110		1878	322	675	1036	13154	339	341	172	Z
Relative Percent Difference	61		67		20		48	43	3.8	31	0.0	6.8	3.3	21	

**Data Qualifier Flag**

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 Tank BY-108 on 3/28/96

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)
Upwind Ambient	<77 Y	77 Y	14 J	30 B	16 B	U	5.3 J	27 B	7.4 J	8.0 J	1.5 J	Z
Ambient Air Through VSS	<77 Y	61 Y	18 J	26 B	10 B,J	5.1	5.3 J	6.5 B,J	1.6 J	0.96 J	U	Z
ISVS Ambient Air	107 Y	77 Y	16 J	42 B	9.9 B,J	3.3 J	5.2 J	8.0 B,J	2.8 J	U	U	Z

**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.

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-d<sub>8</sub>, and bromobenzene-d<sub>5</sub>). 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™ 300 traps for sample collection and preconcentration. The traps are ground-glass tubes (11.5 cm long X 6 mm OD, 4 mm ID) containing a series of sorbents arranged in order of increasing retentivity. Each trap contains 300 mg of Carbotrap™ C, 200 mg of Carbotrap™ B, and 125 mg of Carbosieve™ S-III. The first two sorbents are deactivated graphite with limited sorption power for less volatile compounds. The final trapping stage, the Carbosieve™ S-III, is a graphitized molecular sieve used to retain the most volatile components, including some permanent gases such as Freon-12. Following sample collection and addition of 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™ 300 trap is used for repeat analysis on at least one sample per batch. Since the IS also follows the same path, quantitation may be performed directly on the repeat run without changing the calibration. Following desorption from the Carbotrap™ 300 trap, the analyte is transferred to a long, thin focusing trap filled with the same type of trapping materials as the Carbotrap™ 300 traps and in approximately the same ratios. The purpose of the focusing trap is to provide an interface to a capillary 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

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(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 4 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.

**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<sub>5</sub>, 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 seven samples, four field blanks, and two trip blanks were returned to the laboratory on April 16, 1996, under WHC COC numbers 100043 and 100044. The samples were analyzed on May 8, 10, and 13, 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 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. Six 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. The compounds 1-butanol and methanol were the most abundant compounds identified in each of the trap samples. Tributyl phosphate was not observed in any samples from Tank BY-108. Based on the average values for each of the sampling methods the highest concentrations of methanol, ethanol, acetonitrile, propanol, hexane, and 1-butanol were observed in the ISVS samples. The highest concentrations of acetone, tetrahydrofuran, dodecane, tridecane and tetradecane were observed in the VSS samples.

One triple sorbent trap sample was analyzed in replicate from the VSS and ISVS sampling methods. The RPDs were calculated and are 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. Low levels of acetone and hexane were observed in the ISVS field blanks samples. Traces of acetone and 1-butanol were observed in several 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 May 6, 1996 and subsequent sample runs quantitated against CCVs at the beginning of each daily batch.

The TST samples were analyzed in 3 batches. The sample analytical sequence runs (batches) were as follows:

Batch #1 (file identifier 46050801.d) - S6021-A10.834, S6021-A09.826, S6022-A33.857, S6022-A33.857 REP, S6022-A31.854;

Batch #2 (file identifier 46051001.d) - S6021-A08.825, S6021-A08.825 REP, S6022-A32.856, S6021-A11.836;

Batch #3 (file identifier 46051301.d) - S6021-A12.837, S6021-A13.838, S6022-A40.859, S6022-A41.868, S6021-A14.869, S6021-A15.853.

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

Batch #1:

The CCV was satisfactory with the exception of tridecane (31%) and tetradecane (63%). The CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of the target compounds. Both compounds were observed at high concentration in the samples. Due to the CCV performance, the results for these compounds have a greater uncertainty.

Batch #2:

The CCV was satisfactory with the exception of methanol (28%) and tetradecane (49%). The CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of the target compounds. Both compounds were observed at high concentration in the samples. Due to the CCV performance, the results for these compounds have a greater uncertainty. The third run (S6021-A08.825) exhibited an unusually high IS response with the repeat run immediately following showing a reduced IS response. This type of behavior is known to be associated with variations in split ratio caused by tight packing of the media in the split tube. Data associated with this run is very similar to other data from similar samples and show good agreement with the repeat run in spite of a large difference in IS responses. Surrogate recoveries were also typical for this run suggesting that the main effect of the variation in split ratio was to provide a somewhat enhanced analytical sensitivity for that sample. The VSS sample S6021-A11.836 was run in this batch, but due to a file transfer error the data for this sample were lost.

Batch #3:

The CCV was satisfactory for all target compounds with the exception of methanol (31%) and tetradecane (38%). The CCV passed the procedural criterion requiring  $\pm 25\%$  D passage for 85% of the target compounds. Trace amounts of both compounds (below EQL) were observed in at least one field blank.

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

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.

Tributyl phosphate (TBP) 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. TBP was not detected in any sample.

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

Chromatograms for both the VSS and ISVS samples showed typical normal paraffin hydrocarbon (NPH) characteristics; however, the VSS samples showed a much more pronounced NPH hump relative to the ISVS samples and high end compounds such as dodecane were noticeably higher in the VSS samples. By contrast, the more volatile compounds showed good comparability between the two sampling methods suggesting that the difference is not associated with flow control problems, but is more fundamental to the sampling process, perhaps related to the temperature of the ISVS bundle during sampling.

No TICs were detected, with the exception of 1-chloro-1,1-difluoroethane, in the field blanks suggesting that environmental contamination associated with bundle preparation, seen on a number of other ISVS runs, was relatively minimal on this job. The analytes seen in the blanks may thus be at least partially associated with passive sampling during the period the bundle is physically within the tank headspace. No target compounds were present at levels above the EQL in either of the trip blanks.

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

**Table E.2 Triple Sorbent Trap Sample Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 3/28/96**

VSS Truck Samples	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)
A08.825	1700 Y	1355 Y	299	1766 E	418	657	926	7206 E	443	459 E	413 E	<0.8
A09.826	1289 Y	996 Y	228	1759 E	354	659	1024	7095 E	559	768 E	700 E	<0.8
A10.834	1751 Y	1155 Y	276	1843 E	391	679	1114	7381 E	611	832 E	770 E	<0.8
Average	1580 Y	1169 Y	268	1789 E	388	665	1021 E	7227 E	538	686 E	628 E	<0.8
ST DEV	253	180	36	47	32	12	94	144	86	199	189	
% RSD	16	15	14	2.6	8.3	1.8	9.2	2.0	16	29	27	
<b>ISVS</b>												
A31.854	1883 Y	1235 Y	269	1641 E	439	629	1018	7100 E	163.0	239	212 E	<0.8
A32.856	1955 Y	1401 Y	316	1833 E	396	616	1096 E	8406 E	110.0	127	110	<0.8
A33.857	2014 Y	1230 Y	274	1654 E	419	606	1010 E	7005 E	168.0	242	206 E	<0.8
Average	1951 Y	1289 Y	286	1709 E	418	617	1041 E	7504 E	147	203	176 E	<0.8
ST DEV	66	97	26	107	22	12	48	783	32	66	57	
% RSD	3.4	7.6	9.0	6.3	5.1	1.9	4.6	10	22	32	33	

**Data Qualifier Flag**

- E Target compound exceeds upper quantification limit (UQL).
- 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 E.3 Replicate Analysis of Triple Sorbent Trap Samples Collected from the Headspace of Tank BY-108 on 3/28/96

VSS Truck Samples	METHANOL	ETHANOL	ACETONITRILE	ACETONE	PROPANOL	TETRAHYDROFURAN	HEXANE	1-BUTANOL	DODECANE	TRIDECANE	TETRADECANE	TBP
A08.825	1700 Y	1355 Y	299	1766 E	418	657	926	7206 E	443	459	413 E	<0.8 Z
A08.825 Rep	1353 Y	1145 Y	285	2059 E	383	719	1267 E	10110 E	624 E	655 E	506 E	<0.8 Z
Relative Percent Difference	23	17	4.8	15	8.7	9.0	31	34	34	35	20	
ISVS												
A33.857	2014 Y	1230 Y	274	1654 E	419	606	1010 E	7005 E	168	242	206 E	<0.8 Z
A33.857 Rep	1795 Y	1138 Y	301	1583 E	415	606	1043 E	7159 E	170	243	205 E	<0.8 Z
Relative Percent Difference	11	7.8	9.4	4.4	1.0	0.0	3.2	2.2	1.2	0.4	0.5	

**Data Qualifier Flag**

- E Target compound exceeds upper quantification limit (UQL).
- 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 E.4** Triple Sorbent Trap Blank Sample Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 3/28/96

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	19 J	2.0 U	1.1 U	0.32 U	12 J	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	0.32 U	5.6 J	3.4 U	6.6 U	1.5 U	<0.8 Z
Trip Blank #1	<192 Y	<133 Y	1.8 U	21 J	2.0 U	1.1 U	0.32 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	0.32 U	2.3 U	3.4 U	6.6 U	1.5 U	<0.8 Z
Field Blank #3 ISVS	<192 Y	<133 Y	1.8 U	39	2.0 U	1.1 U	19	11 J	3.4 U	6.6 U	1.5 U	<0.8 Z
Field Blank #4 ISVS	<192 Y	<133 Y	4.5 J	35	2.0 U	1.1 U	16	13 J	3.4 U	6.6 U	2.1 J	<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 BY-108 on 3/28/96

Target Analytes	CAS No.	S6021-A05.212		S6021-A06.215		S6021-A07.220		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
DICHLORODIFLUOROMETHANE	75-71-8	1.7	B,J	2.1	B,J	16	B,J	6.5	7.9
CHLOROMETHANE	74-87-3	1.1	U	2.3	J	28	J	10	15
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	22	J	22	
METHANOL	67-56-1	1091	Y	1121	Y	2955	Y	1722	1067
VINYL CHLORIDE	75-01-4	1.5	B,J	1.8	B,J	22	B,J	8.5	12
BUTANE	106-97-8	699		736		3400		1612	1549
BROMOMETHANE	74-83-9	1.3	J	1.8	J	20	J	7.8	11
CHLOROETHANE	75-00-3	2.3	J	2.6	J	9.4	U	2.4	0.23
ETHANOL	64-17-5	405	Y	438	Y	1918	Y	920	864
ACETONITRILE	75-05-8	28		105		260		131	118
ACETONE	67-64-1	529		1736		1885		1383	744
TRICHLOROFLUOROMETHANE	75-69-4	17		20		28	J	21	5.7
PENTANE	109-66-0	838		886		1676		1133	471
1,1-DICHLOROETHENE	75-35-4	2.7	J	3.4	J	4.4	U	3.0	0.45
METHYLENE CHLORIDE	75-09-2	7.0	B	7.7	B	39	B	18	18
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	3.1	B,J	3.0	B,J	7.5	J	4.5	2.6
PROPANOL	71-23-8	234		273		406		304	90
PROPANENITRILE	107-12-0	14	J	15	J	50	J	26	20
1,1-DICHLOROETHANE	75-34-3	2.8	B,J	3.0	B,J	2.3	U	2.9	0.11
2-BUTANONE	78-93-3	442		441		384		422	33
CIS-1,2-DICHLOROETHENE	156-59-2	4.3	J	4.3	J	3.9	U	4.3	0.00
HEXANE	110-54-3	973		895		1048		972	77
CHLOROFORM	67-66-3	4.0	B,J	4.0	B,J	15	B,J	7.6	6.2
TETRAHYDROFURAN	109-99-9	917		978		787		894	97
1,2-DICHLOROETHANE	107-06-2	3.6	J	3.7	J	2.9	U	3.6	0.10
BUTANENITRILE	109-74-0	5.8	B,J	6.9	B,J	27	J	13	12
1,1,1-TRICHLOROETHANE	71-55-6	3.1	B,J	3.0	B,J	10	J	5.4	4.1
1-BUTANOL	71-36-3	13946	*	13354	*	8278		11859	3116
BENZENE	71-43-2	28	B	27	B	29		28	1.1
CARBON TETRACHLORIDE	56-23-5	3.0	B,J	2.9	B,J	9.1	J	5.0	3.6
CYCLOHEXANE	110-82-7	119		107		156		127	26
1,2-DICHLOROPROPANE	78-87-5	4.8	J	4.5	J	13	J	7.6	5.1
TRICHLOROETHENE	79-01-6	3.5	B,J	4.0	B,J	12	J	6.5	4.6
HEPTANE	142-82-5	544		560		464		522	51
4-METHYL-2-PENTANONE	108-10-1	43	B	46	B	64		51	11
CIS-1,3-DICHLOROPROPENE	10061-01-5	3.5	J	3.8	J	10	J	5.8	3.8
PYRIDINE	110-86-1	7.0	J	8.0	J	18	J	11.0	6.0
TRANS-1,3-DICHLOROPROPENE	10061-02-6	3.0	B,J	3.6	B,J	10	J	5.6	3.9

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**Table F.1.** SUMMA™ Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	S6021-A05.212		S6021-A06.215		S6021-A07.220		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
PENTANENITRILE	110-59-8	18	B	19	B	34	J	24	9.2
1,1,2-TRICHLOROETHANE	79-00-5	6.5		1.9	J	2.4	U	4.2	3.2
TOLUENE	108-88-3	36		38		29		34	4.5
1,2-DIBROMOETHANE	106-93-4	4.3	J	4.5	J	11	J	6.6	3.9
OCTANE	111-65-9	132		143		102		126	21
TETRACHLOROETHYLENE	127-18-4	5.6	J	6.1		11	J	7.7	3.2
CHLOROBENZENE	108-90-7	7.7		8.6		15	J	10	3.8
HEXANENITRILE	628-73-9	5.3	J	7.5	J	23	J	12	9.8
ETHYLBENZENE	100-41-4	5.9		6.7		14	J	9.0	4.6
P/M-XYLENE	106-42-3	16	J	19	J	29	J	21	7.0
CYCLOHEXANONE	108-94-1	25		26		36	J	29	6.0
STYRENE	100-42-5	3.6	J	4.3	J	13	J	6.9	5.2
1,1,2,2-TETRACHLOROETHANE	79-34-5	10	B	11	B	55	B	26	26
O-XYLENE	95-47-6	7.9		8.7		18	J	12	5.9
NONANE	111-84-2	64		61		69		65	4.1
1-ETHYL-2-METHYL BENZENE	611-14-3	3.3	B,J	3.6	B	17	J	7.8	7.6
1,3,5-TRIMETHYLBENZENE	108-67-8	2.8	B,J	3.6	B	15	J	7.1	6.8
1,2,4-TRIMETHYLBENZENE	95-63-6	3.2	J	3.9	J	15	J	7.4	6.6
DECANE	124-18-5	64		58		107		76	26
1,3-DICHLOROBENZENE	541-73-1	3.1	B	4.1	B	14	B	7.1	6.2
1,4-DICHLOROBENZENE	106-46-7	3.2	B	4.1	B	14	B,J	6.9	5.7
1,2-DICHLOROBENZENE	95-50-1	3.0	B	4.1	B	14	B,J	6.9	5.9
UNDECANE	1120-21-4	150		126		192		156	33
1,2,4-TRICHLOROBENZENE	120-82-1	4.0	B	5.2	B	12	B,J	7.1	4.4
DODECANE	112-40-3	459		444		606		503	90
HEXACHLORO-1,3-BUTADIENE	87-68-3	3.5	J	4.9		12	J	6.8	4.6
TRIDECANE	629-50-5	500		517		698		572	110
TETRADECANE	629-59-4	261		276		531		356	152
1,3-BUTADIENE	106-99-0	<34	Y	<34	Y	<34	Y		

**Data Qualifier Flags**

B Compound found in associated laboratory blank.

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

U Target compound not detected at or above the IDL.

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

\* Flag to denote diluted value was reported for target compound in table

Table F.2. Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	S6021-A08.825		S6021-A09.826		S6021-A10.834		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
DICHLORODIFLUOROMETHANE	75-71-8	0.79	U	0.79	U	0.79	U		
CHLOROMETHANE	74-87-3	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		
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U	1.1	U		
METHANOL	67-56-1	1700	Y	1289	Y	1751	Y	1580	253
BUTANE	106-97-8	2199	E	2136	E	2433	E	2256	157
CHLOROETHANE	75-00-3	1.4	U	1.4	U	1.4	U		
ETHANOL	64-17-5	1355	Y	996	Y	1155	Y	1169	180
ACETONITRILE	75-05-8	299		228		276		267	36
ACETONE	67-64-1	1766	E	1759	E	1843	E	1789	47
TRICHLOROFLUOROMETHANE	75-69-4	21		20		19		20	1.1
PENTANE	109-66-0	1871	E	1864	E	2037	E	1924	98
1,1-DICHLOROETHENE	75-35-4	0.47	U	0.47	U	0.47	U		
METHYLENE CHLORIDE	75-09-2	164		12	J	18	J	65	86
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.36	U	0.36	U	0.36	U		
PROPANENITRILE	107-12-0	27		23		25		25	1.8
PROPANOL	71-23-8	418		354		391		388	32
1,1-DICHLOROETHANE	75-34-3	0.32	U	0.32	U	0.32	U		
2-BUTANONE	78-93-3	233		210		199		214	17
CIS-1,2-DICHLOROETHENE	156-59-2	0.58	U	0.58	U	0.58	U		
HEXANE	110-54-3	926		1024	E	1114	E	1021	94
CHLOROFORM	67-66-3	0.53	U	0.53	U	0.53	U		
TETRAHYDROFURAN	109-99-9	657		659		679		665	12
1,2-DICHLOROETHANE	107-06-2	0.25	U	0.25	U	0.25	U		
BUTANENITRILE	109-74-0	23		26		21		23	2.8
BENZENE	71-43-2	42		28		29		33	7.9
1,1,1-TRICHLOROETHANE	71-55-6	0.44	U	0.44	U	0.44	U		
1-BUTANOL	71-36-3	7206	E	7095	E	7381	E	7227	144
CARBON TETRACHLORIDE	56-23-5	0.20	U	0.20	U	0.20	U		
CYCLOHEXANE	110-82-7	168		161		185		171	12
1,2-DICHLOROPROPANE	78-87-5	0.38	U	0.38	U	0.38	U		
TRICHLOROETHENE	79-01-6	2.2	J	0.61	U	0.61	U	2.2	
HEPTANE	142-82-5	563		556		609		576	28
4-METHYL-2-PENTANONE	108-10-1	70		60		69		66	5.5
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.25	U	0.25	U	0.25	U		
PYRIDINE	110-86-1	12	J	11	J	12	J	12	0.63
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.53	U	0.53	U	0.53	U		
PENTANENITRILE	110-59-8	26		0.22	U	0.22	U	26	

**Table F.2.** Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	S6021-A08.825		S6021-A09.826		S6021-A10.834		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
1,1,2-TRICHLOROETHANE	79-00-5	0.68	J	0.26	U	0.26	U	0.68	
TOLUENE	108-88-3	41		38		37		39	2.1
1,2-DIBROMOETHANE	106-93-4	0.28	U	0.28	U	0.28	U		
OCTANE	111-65-9	221		205		210		212	8.2
TETRACHLOROETHYLENE	127-18-4	0.47	J	0.34	U	0.34	U	0.47	
CHLOROBENZENE	108-90-7	0.22	U	0.22	U	0.22	U		
HEXANENITRILE	628-73-9	4.5	J	0.66	U	0.66	U	4.5	
ETHYLBENZENE	100-41-4	5.1		4.5		4.8		4.8	0.30
P/M-XYLENE	106-42-3	16		14		15		15	0.94
CYCLOHEXANONE	108-94-1	2.9	U	12	J	2.9	U	12	
STYRENE	100-42-5	0.39	J	0.33	U	0.36	J	0.38	0.021
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.77	U	0.77	U	0.77	U		
O-XYLENE	95-47-6	7.9		7.0		7.2		7.4	0.44
NONANE	111-84-2	98		87		95		94	5.6
1-ETHYL-2-METHYL BENZENE	611-14-3	0.67	J	0.44	U	0.44	U	0.67	
1,3,5-TRIMETHYLBENZENE	108-67-8	0.52	J	0.44	J	0.47	J	0.48	0.04
1,2,4-TRIMETHYLBENZENE	95-63-6	1.4	J	1.2	J	1.3	J	1.3	0.12
DECANE	124-18-5	138		138		150		142	7.1
1,3-DICHLOROBENZENE	541-73-1	0.48	U	0.48	U	0.48	U		
1,4-DICHLOROBENZENE	106-46-7	0.43	U	0.43	U	0.43	U		
1,2-DICHLOROBENZENE	95-50-1	0.72	U	0.72	U	0.72	U		
UNDECANE	1120-21-4	280		303		326		303	23
1,2,4-TRICHLOROBENZENE	120-82-1	1.9	U	1.9	U	1.9	U		
DODECANE	112-40-3	443		559	E	611	E	538	86
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.8	U	1.8	U	1.8	U		
TRIDECANE	629-50-5	459	E	768	E	832	E	686	200
TETRADECANE	629-59-4	413	E	700	E	770	E	628	189
TBP	126-73-8	<0.83	Z	<0.83	Z	<0.83	Z		

**Data Qualifier Flags**

- E Target compound exceeds upper quantification limit (UQL).
- 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 BY-108 on 3/28/96

Target Analytes	CAS No.	S6022-A28.232		S6022-A29.244		S6022-A30.247		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
DICHLORODIFLUOROMETHANE	75-71-8	4.1	B,J	3.4	B,J	11	B,J	6.3	4.4
CHLOROMETHANE	74-87-3	4.3	J	1.1	U	9.6	J	6.9	3.8
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	5.7	J	3.0	J	9.5	J	6.1	3.2
METHANOL	67-56-1	2154	Y	2940	Y	1305	Y	2133	818
VINYL CHLORIDE	75-01-4	3.4	J	3.2	B,J	8.5	B,J	5.0	3.0
BUTANE	106-97-8	1532		2776	*	1168		1825	843
BROMOMETHANE	74-83-9	3.2	J	3.1	J	8.2	J	4.8	2.9
CHLOROETHANE	75-00-3	4.1	J	4.4	J	9.4	U	4.2	0.23
ETHANOL	64-17-5	1651	Y	1220	Y	474	Y	1115	595
ACETONITRILE	75-05-8	138		90		46	J	91	46
ACETONE	67-64-1	3486	*	3072	*	837		2465	1425
TRICHLOROFLUOROMETHANE	75-69-4	29		34		29	J	31	2.9
PENTANE	109-66-0	2905	*	2328	*	1288		2174	819
1,1-DICHLOROETHENE	75-35-4	3.5	J	3.7	J	14	J	7.1	6.1
METHYLENE CHLORIDE	75-09-2	8.7	B	10	B	34	B	17	14
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	5.9	B	7.1	B	17	B,J	10	6.4
PROPANOL	71-23-8	429		501		259		396	124
PROPANENITRILE	107-12-0	18		22		41	J	27	12
1,1-DICHLOROETHANE	75-34-3	4.1	B,J	4.9	B	13	B,J	7.3	4.9
2-BUTANONE	78-93-3	474		686		428		529	138
CIS-1,2-DICHLOROETHENE	156-59-2	4.4	J	5.6	J	18	J	9.2	7.3
HEXANE	110-54-3	1181		1419	*	1200		1267	133
CHLOROFORM	67-66-3	4.9		6.2	B	16	B,J	9.2	6.3
TETRAHYDROFURAN	109-99-9	954		701	*	1025		893	170
1,2-DICHLOROETHANE	107-06-2	5.8	J	6.5		14	J	8.9	4.7
BUTANENITRILE	109-74-0	9.1	J	1.1	U	21	B,J	15	8.6
1,1,1-TRICHLOROETHANE	71-55-6	5.1	J	5.3	B,J	15	B,J	8.6	5.9
1-BUTANOL	71-36-3	17060	*	13150	*	8123		12778	4480
BENZENE	71-43-2	33		39		49		40	7.9
CARBON TETRACHLORIDE	56-23-5	4.7	J	5.2	B	14	B,J	8.1	5.5
CYCLOHEXANE	110-82-7	168		201		152		173	25
1,2-DICHLOROPROPANE	78-87-5	5.6	J	0.59	U	20	J	13	10
TRICHLOROETHENE	79-01-6	5.3		4.4	B	16	B,J	8.5	6.4
HEPTANE	142-82-5	760		749		603		704	87
4-METHYL-2-PENTANONE	108-10-1	74		71		57		67	8.6
CIS-1,3-DICHLOROPROPENE	10061-01-5	4.9		3.7	J	14	J	7.4	5.5
PYRIDINE	110-86-1	27	J	13	J	18	J	19	7.1
TRANS-1,3-DICHLOROPROPENE	10061-02-6	4.8	B	3.6	B,J	13	B,J	7	4.9

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Table F.3. SUMMA™ Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	S6022-A28.232		S6022-A29.244		S6022-A30.247		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
PENTANENITRILE	110-59-8	28		22	B	33	B,J	28	5.6
1,1,2-TRICHLOROETHANE	79-00-5	8.3	B	9.4		24		14	8.7
TOLUENE	108-88-3	31		38		56		41	13
1,2-DIBROMOETHANE	106-93-4	3.8	J	3.5	J	20	J	9.2	9.7
OCTANE	111-65-9	150		166		178		165	14
TETRACHLOROETHYLENE	127-18-4	4.7	J	5.7	J	24	J	12	11
CHLOROBENZENE	108-90-7	7.9		8.4		25	J	14	9.7
HEXANENITRILE	628-73-9	9.1	J	8.0	J	23	J	13	8.5
ETHYLBENZENE	100-41-4	6.8		7.1		20	J	11	7.5
P/M-XYLENE	106-42-3	17	J	19	J	50	J	28	18
CYCLOHEXANONE	108-94-1	32		39		44	J	38	6.1
STYRENE	100-42-5	5.4		3.1	J	15	J	7.8	6.2
1,1,2,2-TETRACHLOROETHANE	79-34-5	15	B	12	B	38	B	22	14
O-XYLENE	95-47-6	8.3		8.2		22	J	13	7.9
NONANE	111-84-2	73		72		81		76	4.8
1-ETHYL-2-METHYL BENZENE	611-14-3	4.8	B	3.0	B,J	15	B,J	7.5	6.4
1,3,5-TRIMETHYLBENZENE	108-67-8	4.3	B	2.9	B,J	14	B,J	7.1	6.0
1,2,4-TRIMETHYLBENZENE	95-63-6	5.1		3.6	J	14	J	7.7	5.8
DECANE	124-18-5	79		66		90		78	12
1,3-DICHLOROBENZENE	541-73-1	4.1	B	3.2	B	17	B	8.2	8.0
1,4-DICHLOROBENZENE	106-46-7	4.1	B	3.1	B	16	B	7.9	7.4
1,2-DICHLOROBENZENE	95-50-1	4.8	B	3.4	B	17	B	8.4	7.5
UNDECANE	1120-21-4	129		121		141		130	9.9
1,2,4-TRICHLOROBENZENE	120-82-1	7.8	B	5.5	B	19	B	11	7.2
DODECANE	112-40-3	286		363		401		350	59
HEXACHLORO-1,3-BUTADIENE	87-68-3	4.3		4.1		17	J	8.4	7.3
TRIDECANE	629-50-5	263		330		453		348	96
TETRADECANE	629-59-4	127		140		199		155	38
1,3-BUTADIENE	106-99-0	<34	Y	<34	Y	<34	Y		

**Data Qualifier Flags**

B Compound found in associated laboratory blank.

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

U Target compound not detected at or above the IDL.

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

\* Flag to denote diluted value was reported for target compound in table

Table F.4. Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	S6022-A31.854		S6022-A32.856		S6022-A33.857		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
DICHLORODIFLUOROMETHANE	75-71-8	0.84	U	0.85	U	0.76	U		
CHLOROMETHANE	74-87-3	1.5	U	1.5	U	1.4	U		
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.44	U	0.44	U	0.40	U		
METHANOL	67-56-1	1883	Y	1955	Y	2014	Y	1951	66
VINYL CHLORIDE	75-01-4	1.2	U	1.2	U	1.0	U		
BUTANE	106-97-8	2108	E	2216	E	2069	E	2131	76
CHLOROETHANE	75-00-3	1.5	U	1.5	U	1.4	U		
ETHANOL	64-17-5	1235	Y	1401	Y	1230	Y	1289	97
ACETONITRILE	75-05-8	269		316		274		286	26
ACETONE	67-64-1	1641	E	1833	E	1654	E	1710	107
TRICHLOROFLUOROMETHANE	75-69-4	31		24		24		26	4.2
PENTANE	109-66-0	1761	E	1934	E	1713	E	1803	117
1,1-DICHLOROETHENE	75-35-4	0.51	U	0.51	U	0.46	U		
METHYLENE CHLORIDE	75-09-2	7.7	J	7.3	J	5.9	J	7.0	0.91
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.38	U	0.38	U	0.35	U		
PROPANOL	71-23-8	439		396		419		418	22
PROPANENITRILE	107-12-0	16		18		17		17	1.2
1,1-DICHLOROETHANE	75-34-3	0.34	U	0.34	U	0.76	J	0.76	
2-BUTANONE	78-93-3	239		218		230		229	10
CIS-1,2-DICHLOROETHENE	156-59-2	0.62	U	0.62	U	0.56	U		
HEXANE	110-54-3	1018		1096	E	1010	E	1041	47
CHLOROFORM	67-66-3	0.57	U	0.57	U	0.52	U		
TETRAHYDROFURAN	109-99-9	629		616		606		617	12
1,2-DICHLOROETHANE	107-06-2	0.27	U	0.27	U	0.24	U		
BUTANENITRILE	109-74-0	20		14		17		17	3.0
1,1,1-TRICHLOROETHANE	71-55-6	0.48	U	0.48	U	0.43	U		
1-BUTANOL	71-36-3	7100	E	8406	E	7005	E	7504	782
BENZENE	71-43-2	26		29		26		27	1.8
CARBON TETRACHLORIDE	56-23-5	0.22	U	0.22	U	0.20	U		
CYCLOHEXANE	110-82-7	146		149		143		146	2.8
1,2-DICHLOROPROPANE	78-87-5	0.40	U	0.40	U	0.36	U		
TRICHLOROETHENE	79-01-6	0.66	U	0.66	U	0.59	U		
HEPTANE	142-82-5	496		529		498		508	19
4-METHYL-2-PENTANONE	108-10-1	53		55		53		54	1.1
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.27	U	0.27	U	0.24	U		
PYRIDINE	110-86-1	8.4	J	9.0	J	9.0	J	8.8	0.32
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.57	U	0.58	U	0.52	U		
PENTANENITRILE	110-59-8	19		23		0.21	U	21	2.9

Table F.4. Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	S6022-A31.854		S6022-A32.856		S6022-A33.857		Mean	St. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
1,1,2-TRICHLOROETHANE	79-00-5	0.28	U	0.28	U	1.7	J	1.7	
TOLUENE	108-88-3	71		80		68		73	6.4
1,2-DIBROMOETHANE	106-93-4	0.30	U	0.30	U	0.27	U		
OCTANE	111-65-9	138		141		142		140	2.2
TETRACHLOROETHYLENE	127-18-4	0.36	U	0.36	U	0.33	U		
CHLOROBENZENE	108-90-7	0.23	U	0.23	U	0.21	U		
HEXANENITRILE	628-73-9	2.7	J	2.2	J	2.4	J	2.4	0.21
ETHYLBENZENE	100-41-4	6.4		7.0		6.3		6.6	0.34
P/M-XYLENE	106-42-3	23		25		23		23	1.2
CYCLOHEXANONE	108-94-1	3.1	U	7.3	J	2.8	U	7.3	
STYRENE	100-42-5	1.7	J	2.0	J	1.6	J	1.8	0.20
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.83	U	0.83	U	0.75	U		
O-XYLENE	95-47-6	8.3		8.8		8.2		8.4	0.29
NONANE	111-84-2	39		36		41		39	2.5
1-ETHYL-2-METHYL BENZENE	611-14-3	1.5	J	1.6	J	1.5	J	1.5	0.047
1,3,5-TRIMETHYLBENZENE	108-67-8	1.4	J	1.5	J	1.5	J	1.5	0.021
1,2,4-TRIMETHYLBENZENE	95-63-6	4.2	J	4.2	J	4.2	J	4.2	0.021
DECANE	124-18-5	43		34		45		41	5.7
1,3-DICHLOROBENZENE	541-73-1	0.51	U	0.52	U	0.59	J	0.59	
1,4-DICHLOROBENZENE	106-46-7	0.46	U	0.47	U	0.59	J	0.59	
1,2-DICHLOROBENZENE	95-50-1	0.77	U	0.77	U	0.69	U		
UNDECANE	1120-21-4	78		60		80		73	11
1,2,4-TRICHLOROBENZENE	120-82-1	2.1	U	2.1	U	1.9	U		
DODECANE	112-40-3	163		110		168		147	32
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.9	U	1.9	U	1.7	U		
TRIDECANE	629-50-5	239		127		242		203	65
TETRADECANE	629-59-4	212	E	110		206	E	176	57
TBP	126-73-8	<0.83	Z	<0.83	Z	<0.83	Z		

**Data Qualifier Flags**

E Target compound exceeds upper quantification limit (UQL).

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 BY-108 on 3/28/96

Target Analytes	CAS No.	VSS				ISVS			
		S6021-A07.220				S6022-A29.244			
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	16	B,J	3.2	B,J	3.4	B,J	3.0	B,J
CHLOROMETHANE	74-87-3	28	J	5.6	J	1.1	U	3.9	J
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	22	J	3.4	J	3.0	J	3.1	J
METHANOL	67-56-1	2955	Y	2113	Y	2940	Y	1570	Y
VINYL CHLORIDE	75-01-4	22	B,J	3.4	J	3.2	B,J	3.0	J
BUTANE	106-97-8	3400		1599		2776	*	1602	
BROMOMETHANE	74-83-9	20	J	3.1	J	3.1	J	3.0	J
CHLOROETHANE	75-00-3	9.4	U	3.9	J	4.4	J	4.2	J
ETHANOL	64-17-5	1918	Y	887	Y	1220	Y	611	Y
ACETONITRILE	75-05-8	260		127		90		110	
ACETONE	67-64-1	1885		3268	*	3072	*	1878	
TRICHLOROFLUOROMETHANE	75-69-4	28	J	29		34		29	
PENTANE	109-66-0	1676		2258	*	2328	*	1369	
1,1-DICHLOROETHENE	75-35-4	4.4	U	3.5	J	3.7	J	3.6	J
METHYLENE CHLORIDE	75-09-2	39	B	9.5	B	10	B	9.2	B
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	7.5	J	3.8	B,J	7.1	B	5.8	B
PROPANOL	71-23-8	406		410		501		322	
PROPANENITRILE	107-12-0	50	J	15	J	22		14	J
1,1-DICHLOROETHANE	75-34-3	2.3	U	4.3	B,J	4.9	B	3.7	B,J
2-BUTANONE	78-93-3	384		456		686		375	
CIS-1,2-DICHLOROETHENE	156-59-2	3.9	U	4.5	J	5.6	J	3.9	J
HEXANE	110-54-3	1048		1074		1419	*	1036	
CHLOROFORM	67-66-3	15	B,J	5.0		6.2	B	4.6	J
TETRAHYDROFURAN	109-99-9	787		933		701	*	675	
1,2-DICHLOROETHANE	107-06-2	2.9	U	5.4	J	6.5		0.59	U
1-BUTANOL	71-36-3	8278		13792	*	13150	*	13354	*
BUTANENITRILE	109-74-0	27	J	5.3	J	1.1	U	4.6	J
1,1,1-TRICHLOROETHANE	71-55-6	10	J	5.1	J	5.3	B,J	4.3	J
BENZENE	71-43-2	29		31		39		29	
CARBON TETRACHLORIDE	56-23-5	9.1	J	4.6	J	5.2	B	3.9	J
CYCLOHEXANE	110-82-7	156		187		201		159	
1,2-DICHLOROPROPANE	78-87-5	13	J	5.5	B,J	0.59	U	4.9	B,J
TRICHLOROETHENE	79-01-6	12	J	4.8		4.4	B	4.8	
HEPTANE	142-82-5	464		720		749		630	
4-METHYL-2-PENTANONE	108-10-1	64		67		71		51	
CIS-1,3-DICHLOROPROPENE	10061-01-5	10	J	4.6	J	3.7	J	4.5	J
PYRIDINE	110-86-1	18	J	7.9	J	13	J	6.2	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10	J	4.2	B	3.6	B,J	4.3	B
PENTANENITRILE	110-59-8	34	J	26		22	B	26	
1,1,2-TRICHLOROETHANE	79-00-5	2.4	U	7.5	B	9.4		6.2	B
TOLUENE	108-88-3	29		29		38		31	
1,2-DIBROMOETHANE	106-93-4	11	J	3.3	J	3.5	J	4.0	J
OCTANE	111-65-9	102		143		166		132	
TETRACHLOROETHYLENE	127-18-4	11	J	4.3	J	5.7	J	4.6	J
CHLOROBENZENE	108-90-7	15	J	6.6		8.4		7.2	
HEXANENITRILE	628-73-9	23	J	3.8	J	8.0	J	6.2	J
ETHYLBENZENE	100-41-4	14	J	5.8	J	7.1		6.5	
P/M-XYLENE	106-42-3	29	J	14	J	19	J	16	J
CYCLOHEXANONE	108-94-1	36	J	28		39		26	
STYRENE	100-42-5	13	J	2.7	J	3.1	J	3.8	J
1,1,2,2-TETRACHLOROETHANE	79-34-5	55	B	12	B	12	B	12	B

**Table F.5. SUMMA™ Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank BY-108 on 3/28/96**

Target Analytes	CAS No.	VSS				ISVS			
		S6021-A07.220				S6022-A29.244			
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
O-XYLENE	95-47-6	18	J	6.8		8.2		7.6	
NONANE	111-84-2	69		68		72		61	
1-ETHYL-2-METHYL BENZENE	611-14-3	17	J	3.3	B,J	3.0	B,J	4.6	B
1,3,5-TRIMETHYLBENZENE	108-67-8	15	J	3.0	B,J	2.9	B,J	4.1	B
1,2,4-TRIMETHYLBENZENE	95-63-6	15	J	3.3	J	3.6	J	4.5	J
DECANE	124-18-5	107		78		66		66	
1,3-DICHLOROBENZENE	541-73-1	14	B	2.5	B,J	3.2	B	3.7	B
1,4-DICHLOROBENZENE	106-46-7	14	B,J	2.5	B,J	3.1	B	3.7	B
1,2-DICHLOROBENZENE	95-50-1	14	B,J	2.5	B,J	3.4	B	3.8	B
UNDECANE	1120-21-4	192		147		121		122	
1,2,4-TRICHLOROBENZENE	120-82-1	12	B,J	2.5	B,J	5.5	B	4.0	B
DODECANE	112-40-3	606		412		363		339	
HEXACHLORO-1,3-BUTADIENE	87-68-3	12	J	2.9	J	4.1		4.2	
TRIDECANE	629-50-5	698		391		330		341	
TETRADECANE	629-59-4	531		192		140		172	
1,3-BUTADIENE	106-99-0	<34	Y	<34	Y	<34	Y	<34	Y

**Data Qualifier Flags**

B Compound found in associated laboratory blank.

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

U Target compound not detected at or above the IDL.

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

\* Flag to denote diluted value was reported for target compound in table

Table F.6. Triple Sorbent Trap Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	VSS				ISVS			
		S6021-A08.825				S6022-A33.857			
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	0.79	U	0.79	U	0.76	U	0.76	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	0.40	U	0.40	U
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U	1.0	U	1.0	U
METHANOL	67-56-1	1700	Y	1353	Y	2014	Y	1795	Y
BUTANE	106-97-8	2199	E	2401	E	2069	E	2089	E
CHLOROETHANE	75-00-3	1.4	U	1.4	U	1.4	U	1.4	U
ETHANOL	64-17-5	1355	Y	1145	Y	1230	Y	1138	Y
ACETONITRILE	75-05-8	299		285		274		301	
ACETONE	67-64-1	1766	E	2059	E	1654	E	1583	E
TRICHLOROFLUOROMETHANE	75-69-4	21		21		24		22	
PENTANE	109-66-0	1871	E	2390	E	1713	E	1769	E
1,1-DICHLOROETHENE	75-35-4	0.47	U	0.47	U	0.46	U	0.46	U
METHYLENE CHLORIDE	75-09-2	164		157		5.9	J	7.3	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.36	U	0.36	U	0.35	U	0.35	U
PROPANENITRILE	107-12-0	27		25		17		19	
PROPANOL	71-23-8	418		383		419		415	
1,1-DICHLOROETHANE	75-34-3	0.32	U	0.32	U	0.76	J	0.31	U
2-BUTANONE	78-93-3	233		218		230		189	
CIS-1,2-DICHLOROETHENE	156-59-2	0.58	U	0.58	U	0.56	U	0.56	U
HEXANE	110-54-3	926		1267	E	1010	E	1043	E
CHLOROFORM	67-66-3	0.53	U	0.53	U	0.52	U	0.52	U
TETRAHYDROFURAN	109-99-9	657		719		606		606	
1,2-DICHLOROETHANE	107-06-2	0.25	U	0.25	U	0.24	U	0.24	U
BUTANENITRILE	109-74-0	23		26		17		20	
BENZENE	71-43-2	42		39		26		26	
1,1,1-TRICHLOROETHANE	71-55-6	0.44	U	0.44	U	0.43	U	0.43	U
1-BUTANOL	71-36-3	7206	E	10110	E	7005	E	7159	E
CARBON TETRACHLORIDE	56-23-5	0.20	U	0.20	U	0.20	U	0.20	U
CYCLOHEXANE	110-82-7	168		188		143		144	
1,2-DICHLOROPROPANE	78-87-5	0.38	U	0.38	U	0.36	U	0.36	U
TRICHLOROETHENE	79-01-6	2.2	J	2.0	J	0.59	U	0.59	U
HEPTANE	142-82-5	563		653	E	498		496	
4-METHYL-2-PENTANONE	108-10-1	70		63		53		52	
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.25	U	0.25	U	0.24	U	0.24	U
PYRIDINE	110-86-1	12	J	12	J	9.0	J	8.7	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.53	U	0.53	U	0.52	U	0.52	U
PENTANENITRILE	110-59-8	26		21		0.21	U	23	
1,1,2-TRICHLOROETHANE	79-00-5	0.68	J	1.5	J	1.7	J	0.25	U
TOLUENE	108-88-3	41		42		68		69	
1,2-DIBROMOETHANE	106-93-4	0.28	U	0.28	U	0.27	U	0.27	U
OCTANE	111-65-9	221		227		142		142	
TETRACHLOROETHYLENE	127-18-4	0.47	J	0.34	U	0.33	U	0.33	U
CHLOROBENZENE	108-90-7	0.22	U	0.22	U	0.21	U	0.21	U
HEXANENITRILE	628-73-9	4.5	J	4.4	J	2.4	J	2.5	J
ETHYLBENZENE	100-41-4	5.1		5.2		6.3		6.3	
P/M-XYLENE	106-42-3	16		17		23		23	
CYCLOHEXANONE	108-94-1	2.9	U	2.9	U	2.8	U	6.5	J
STYRENE	100-42-5	0.39	J	0.33	U	1.6	J	1.6	J
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.77	U	0.77	U	0.75	U	0.75	U
O-XYLENE	95-47-6	7.9		8.8		8.2		8.3	
NONANE	111-84-2	98		96		41		41	

Table F.6. Triple Sorbent Trap Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	VSS				ISVS			
		S6021-A08.825				S6022-A33.857			
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
1-ETHYL-2-METHYL BENZENE	611-14-3	0.67	J	0.95	J	1.5	J	1.5	J
1,3,5-TRIMETHYLBENZENE	108-67-8	0.52	J	0.79	J	1.5	J	1.5	J
1,2,4-TRIMETHYLBENZENE	95-63-6	1.4	J	1.7	J	4.2	J	4.2	J
DECANE	124-18-5	138		142		45		45	
1,3-DICHLOROBENZENE	541-73-1	0.48	U	0.48	U	0.59	J	0.62	J
1,4-DICHLOROBENZENE	106-46-7	0.43	U	0.43	U	0.59	J	0.61	J
1,2-DICHLOROBENZENE	95-50-1	0.72	U	0.72	U	0.69	U	0.69	U
UNDECANE	1120-21-4	280		328		80		79	
1,2,4-TRICHLOROBENZENE	120-82-1	1.9	U	1.9	U	1.9	U	1.9	U
DODECANE	112-40-3	443		624	E	168		170	
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.8	U	1.8	U	1.7	U	1.7	U
TRIDECANE	629-50-5	459	E	655	E	242		243	
TETRADECANE	629-59-4	413	E	506	E	206	E	205	E
TBP	126-73-8	0.83	U	0.83	U	0.81	U	0.81	U

**Data Qualifier Flags**

E Target compound exceeds upper quantification limit (UQL).

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 Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	S6021-A03.062		S6021-A04.157		S6022-A27.227	
		VSS Amb. Air (ppbv)	Flag	Ambient Air Thru VSS (ppbv)	Flag	ISVS Amb. Air (ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	5.1	B,J	5.7	B	4.8	B,J
CHLOROMETHANE	74-87-3	5.9	J	5.6	J	5.7	J
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	5.0	J	4.9	J	4.9	J
METHANOL	67-56-1	<77	Y	<77	Y	107	Y
VINYL CHLORIDE	75-01-4	5.4	B,J	4.7	B,J	4.5	B,J
BUTANE	106-97-8	11	B	9.9	B	9.9	B
BROMOMETHANE	74-83-9	4.0	J	4.3	J	4.5	J
CHLOROETHANE	75-00-3	11	J	8.5	J	1.9	U
ETHANOL	64-17-5	77	B,Y	61	B,Y	77	B,Y
ACETONITRILE	75-05-8	14	J	18	J	16	J
ACETONE	67-64-1	30	B	26	B	42	B
TRICHLOROFLUOROMETHANE	75-69-4	3.4	J	2.8	J	2.8	J
PENTANE	109-66-0	5.6	J	4.4	J	4.5	J
1,1-DICHLOROETHENE	75-35-4	3.6	J	3.2	J	2.8	J
METHYLENE CHLORIDE	75-09-2	8.8	B	8.8	B	8.8	B
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	2.9	J	2.7	J	5.6	
PROPANOL	71-23-8	16	B	9.5	B,J	9.9	B,J
PROPANENITRILE	107-12-0	10	J	14	J	9.2	J
1,1-DICHLOROETHANE	75-34-3	3.5	B,J	3.7	B,J	3.4	B,J
2-BUTANONE	78-93-3	14	B	16	B	14	B
CIS-1,2-DICHLOROETHENE	156-59-2	4.9	J	4.0	J	3.5	J
HEXANE	110-54-3	5.3	J	5.3	J	5.2	J
CHLOROFORM	67-66-3	3.5	B,J	3.9	B,J	3.6	B,J
TETRAHYDROFURAN	109-99-9	0.50	U	5.1		3.3	J
1,2-DICHLOROETHANE	107-06-2	0.59	U	3.6	J	3.4	J
BUTANENITRILE	109-74-0	6.3	J	7.3	J	5.4	J
1,1,1-TRICHLOROETHANE	71-55-6	3.3	J	3.0	J	3.0	J
1-BUTANOL	71-36-3	27	B	6.5	B,J	8.0	B,J
BENZENE	71-43-2	4.5	J	3.9	J	3.7	J
CARBON TETRACHLORIDE	56-23-5	2.8	J	2.7	J	2.6	J
CYCLOHEXANE	110-82-7	6.0	J	6.1	J	6.1	J
1,2-DICHLOROPROPANE	78-87-5	2.8	J	0.59	U	3.0	J
TRICHLOROETHENE	79-01-6	2.7	J	2.7	J	2.4	J
HEPTANE	142-82-5	3.1	B,J	2.9	B,J	2.4	B,J
4-METHYL-2-PENTANONE	108-10-1	6.4	J	6.2	J	5.1	J
CIS-1,3-DICHLOROPROPENE	10061-01-5	2.0	J	2.4	J	1.9	J
PYRIDINE	110-86-1	6.4	J	2.9	J	6.8	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	2.0	J	2.2	J	1.5	J
PENTANENITRILE	110-59-8	6.0	J	5.2	J	4.3	J
1,1,2-TRICHLOROETHANE	79-00-5	2.6	J	2.8	J	1.9	J
TOLUENE	108-88-3	3.4	J	3.0	J	3.0	J
1,2-DIBROMOETHANE	106-93-4	2.6	J	2.7	J	2.3	J
OCTANE	111-65-9	2.0	J	1.8	J	1.4	J
TETRACHLOROETHYLENE	127-18-4	2.7	J	2.7	J	2.4	J
CHLOROBENZENE	108-90-7	3.3	J	2.9	J	2.3	J
HEXANENITRILE	628-73-9	6.9	J	4.6	J	5.0	J
ETHYLBENZENE	100-41-4	2.9	J	2.7	J	2.2	J
P/M-XYLENE	106-42-3	5.4	J	4.9	J	3.9	J
CYCLOHEXANONE	108-94-1	5.7	J	3.1	J	1.3	U
STYRENE	100-42-5	3.0	J	3.0	J	2.6	J

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Table F.7. SUMMA™ Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank BY-108 on 3/28/96

Target Analytes	CAS No.	S6021-A03.062		S6021-A04.157		S6022-A27.227	
		VSS Amb. Air	Flag	Ambient Air Thru VSS	Flag	ISVS Amb. Air	Flag
		(ppbv)		(ppbv)		(ppbv)	
1,1,2,2-TETRACHLOROETHANE	79-34-5	10	B	10	B	12	B
O-XYLENE	95-47-6	3.0	J	3.4	J	3.4	J
NONANE	111-84-2	2.9	J	3.1	J	2.8	J
1-ETHYL-2-METHYL BENZENE	611-14-3	3.3	B,J	3.6	B	3.4	B
1,3,5-TRIMETHYLBENZENE	108-67-8	3.1	B,J	3.4	B	3.2	B,J
1,2,4-TRIMETHYLBENZENE	95-63-6	3.0	J	3.2	J	3.2	J
DECANE	124-18-5	5.7	B	6.0	B	6.0	B
1,3-DICHLOROBENZENE	541-73-1	3.0	B	3.2	B	3.2	B
1,4-DICHLOROBENZENE	106-46-7	3.0	B	3.1	B	2.9	B
1,2-DICHLOROBENZENE	95-50-1	3.3	B	3.0	B	3.3	B
UNDECANE	1120-21-4	3.2	J	2.8	J	3.0	J
1,2,4-TRICHLOROBENZENE	120-82-1	3.9	B	2.3	B,J	3.1	B,J
DODECANE	112-40-3	7.4	J	1.6	J	2.8	J
HEXACHLORO-1,3-BUTADIENE	87-68-3	2.7	J	3.0	J	4.3	
TRIDECANE	629-50-5	8.0		0.96	J	0.73	U
TETRADECANE	629-59-4	1.5	J	0.47	U	0.47	U
1,3-BUTADIENE	106-99-0	<34	Y	<34	Y	<34	Y

**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.

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 BY-108 on 3/28/96

Target Analytes	CAS No.	S6021-A12.837		S6021-A13.838		S6021-A14.869		S6021-A15.853		S6022-A40.859		S6022-A41.868	
		VSS FB #1	Flag	VSS FB #2	Flag	VSS TB #1	Flag	VSS TB #2	Flag	ISVS FB #3	Flag	ISVS FB #4	Flag
		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)	
DICHLORODIFLUOROMETHANE	75-71-8	0.79	U	0.79	U	0.79	U	0.79	U	3.8	J	8.8	
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
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U
METHANOL	67-56-1	<192	Y	<192	Y	<192	Y	<192	Y	<192	Y	<192	Y
BUTANE	106-97-8	1.4	U	1.4	U	1.4	U	1.4	U	25		28	
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	4.5	J
ACETONE	67-64-1	19	J	18	J	21	J	17	J	39		35	
TRICHLOROFLUOROMETHANE	75-69-4	0.72	U	7.2	J	0.78	J	0.72	U	3.7	J	6.2	J
PENTANE	109-66-0	0.89	U	0.89	U	0.89	U	0.89	U	11		12	
1,1-DICHLOROETHENE	75-35-4	0.47	U	2.9	J	0.47	U	0.47	U	0.47	U	0.47	U
METHYLENE CHLORIDE	75-09-2	24	J	375		53		10	J	9.1	J	11	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
PROPANENITRILE	107-12-0	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U
PROPANOL	71-23-8	2.0	U	2.0	U	2.0	U	2.0	U	2.0	U	2.0	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	0.68	U	2.4	J	2.8	J	1.5	J	3.8	J	3.5	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	19		16	
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
BENZENE	71-43-2	0.40	U	0.40	U	0.40	U	0.40	U	0.40	U	0.40	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	12	J	5.6	J	2.3	U	2.3	U	11	J	13	J
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	4.0	J	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	2.3	J	2.0	J
4-METHYL-2-PENTANONE	108-10-1	0.46	U	6.3		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 BY-108 on 3/28/96

Target Analytes	CAS No.	S6021-A12.837		S6021-A13.838		S6021-A14.869		S6021-A15.853		S6022-A40.859		S6022-A41.868	
		VSS FB #1	Flag	VSS FB #2	Flag	VSS TB #1	Flag	VSS TB #2	Flag	ISVS FB #3	Flag	ISVS FB #4	Flag
		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)	
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	0.76	J	0.23	U	0.23	U	15		13	
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	144		0.66	U	0.66	U
ETHYLBENZENE	100-41-4	0.30	U	0.30	U	0.30	U	0.30	U	0.74	J	0.71	J
P/M-XYLENE	106-42-3	0.63	U	0.63	U	0.63	U	0.63	U	2.4	J	2.3	J
CYCLOHEXANONE	108-94-1	2.9	U	2.9	U	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	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	0.65	J	0.74	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	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	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	0.47	U	0.47	U
DECANE	124-18-5	0.47	U	0.47	U	0.47	U	0.47	U	0.50	J	0.47	U
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	2.1	J
TBP	126-73-8	<0.83	Z	<0.83	Z	<0.83	Z	<0.83	Z	<0.83	Z	<0.83	Z

Data Qualifier Flags

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

U Target compound not detected at or above the IDL.

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

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**

Battelle Pacific  
National Northwest Lab

# CHAIN OF CUSTODY

WHC 100047

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-BY-108 Tank Vapor Sample SAF S6021  
VSS

Collection date 03 - 28 - 96  
Preparation date 03 - 25 - 96

Ice Chest No.

Field Logbook No. WHC- N-64210

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

S6021 - A17 . S27-  
S6021 - A18 . S28-  
S6021 - A19 . S29-  
S6021 - A20 . S30-

Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap

S6021 - A21 . S31-  
S6021 - A22 . S32-

Open, close and store NH<sub>3</sub>/H<sub>2</sub>O field blank #1  
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 <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	1430	K.J. Yarn <i>K.J. Yarn</i>	03-25-96	1430	
K.J. Yarn <i>K.J. Yarn</i>	3-28-96	0700	R.D Mahon <i>R.D Mahon</i>	3-28-96	0700	
R.D Mahon <i>R.D Mahon</i>	04-16-96	0840	T.R Bickel <i>T.R Bickel</i>	4-16-96	0840	
T.R Bickel <i>T.R Bickel</i>	4-16-96	1035	J L Tiller <i>J L Tiller</i>	4-16-96	1035	
J A Edwards <i>J A Edwards</i>	4-17-96	1035	G.W.Dennis <i>G.W.Dennis</i>	4-17-96	1035	
G.W.Dennis <i>G.W.Dennis</i>	4-19-96	1330	S.O. Slate <i>S.O. Slate</i>	4-19-96	1330	

## 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)

Battelle Pacific  
National Northwest Lab

# CHAIN OF CUSTODY

WHC 100048

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-BY-108 Tank Vapor Sample SAF S6022  
Non-filtered ISVS  
Ice Chest No.

Collection date 03 - 28 - 96  
Preparation date 03 - 25 - 96

Field Logbook No. WHC-647-N-8 RMAK16  
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

S6022 - A34 . S33-  
S6022 - A35 . S34-  
S6022 - A36 . S35-

Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap  
Collect NH<sub>3</sub>/H<sub>2</sub>O Sorbent Trap

\* S6022 - A42 . S36-  
\* S6022 - A43 . S37-

Store sample bundle NH<sub>3</sub>/H<sub>2</sub>O field blank #3  
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	1430	K.J. Young <i>K.J. Young</i>	03-25-96	1430			
K.J. Young <i>K.J. Young</i>	3-28-96	0700	R D Mahon <i>R D Mahon</i>	3-28-96	0700			
R D Mahon <i>R D Mahon</i>	04-16-96	0840	T-B Utecht <i>T-B Utecht</i>	4-16-96	0840			
T-B Utecht <i>T-B Utecht</i>	4-16-96	1035	JL Tuley <i>JL Tuley</i>	4-16-96	1035			
J A Edwards <i>J A Edwards</i>	4-17-96	1035	G.W. Dennis <i>G.W. Dennis</i>	4-17-96	1035			
G.W. Dennis <i>G.W. Dennis</i>	4-19-96	1330	S.O. Slate <i>S.O. Slate</i>	4-19-96	1330			

## Final Sample Disposition

\*Comments: Tubes A42/A43 are labeled A40/A41 go by 5th

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

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

(Revised 11/30/95 PNNL)

Battelle Pacific  
National Northwest Lab

# CHAIN OF CUSTODY

WHC 100052

Today 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-BY-108 Tank Vapor Sample SAF S6021  
VSS

Collection date 03 - 28 - 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

S6021 - A03 . 062 ✓  
S6021 - A04 . 157 ✓

Collect Ambient Air Sample, Upwind #1  
Collect Ambient Air Sample, Through #2

S6021 - A05 . 212 ✓  
S6021 - A06 . 215 ✓  
S6021 - A07 . 220 ✓

Collect SUMMA #3  
Collect SUMMA #4  
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>[Signature]</i>	03-25-96	1330	<i>[Signature]</i>	03-25-96	1335
K.J. Young <i>[Signature]</i>	3-28-96	0700	R D Mahon <i>[Signature]</i>	4-28-96	0700
R D Mahon <i>[Signature]</i>	04-16-96	0840	T.B. Utter <i>[Signature]</i>	4-16-96	0840
T.B. Utter <i>[Signature]</i>	4-16-96	1035	J.L. Tuck <i>[Signature]</i>	4-16-96	1035

## Final Sample Disposition

Comments:

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

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

(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-BY-108 Tank Vapor Sample SAF S6022

Non-filtered

ISVS

Collection date 03 - 28 - 96

Preparation date 03 - 25 - 96

Ice Chest No.

Field Logbook No. WHC ~~647-N-8~~ <sup>8mm</sup> 647-N-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

S6022 - A27 . 227✓

Collect Ambient Air Sample, Upwind #1

S6022 - A28 . 232✓

Collect SUMMA #2

S6022 - A29 . 244✓

Collect SUMMA #3

S6022 - A30 . 247✓

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>J A Edwards</i>	03-25-96	1330	K.J. Yang <i>K.J. Yang</i>	03-25-96	1335	
K.J. Yang <i>K.J. Yang</i>	3-28-96	0700	R.D. Mahon <i>R.D. Mahon</i>	3-28-96	0700	
R.D. Mahon <i>R.D. Mahon</i>	04-16-96	0840	T.B. Utter <i>T.B. Utter</i>	4-16-96	0840	
T.B. Utter <i>T.B. Utter</i>	4-16-96	1035	J.L. Jurek <i>J.L. Jurek</i>	4-16-96	1035	

## Final Sample Disposition

Comments:

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

POC

POC

(Revised 11/30/95 PNNL)



Battelle Pacific  
National Northwest Lab

CHAIN OF CUSTODY

WHC 100043

Custody Form Initiator J. A. Edwards - PNL

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

Company Contact R. D. Mahon - WHC

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

Project Designation/Sampling Locations 200 West Tank Farm  
241-BY-108 Tank Vapor Sample SAF S6021  
(VSS Truck)

Collection date 03 - 28 - 96  
Preparation date 03 - 22 - 96

Ice Chest No.

Field Logbook No. WHC- N-64710

Ertco Hi/Lo thermometer No. PNL-T-00 4

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

S6021 - A08 . 825 Collect TST Sample # 1  
S6021 - A09 . 826 Collect TST Sample # 2  
S6021 - A10 . 834 Collect TST Sample # 3  
S6021 - A11 . 836 Collect TST Sample # 4

S6021 - A12 . 837 Open, close & store TST Field Blank # 1 In VSS truck  
S6021 - A13 . 838 Open, close & store TST Field Blank # 2 In VSS truck

S6021 - A14 . 869 Store TST Trip Blank # 1 N/A  
S6021 - A15 . 853 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			
J L Julva	03-25-96	1230	J A Edwards	03-25-96	1230			
J A Edwards	03-25-96	1330	K. J. Young	03-25-96	1330			
K. J. Young	3-28-96	0700	R D Mahon	3-28-96	0700			
R D Mahon	04-16-96	0840	T B Utterlitz	4-16-96	0840			
T B Utterlitz	4-16-96	1035	J L Julva	4-16-96	1035			

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

POC 1/2 POC 90

Comments:

Cooler Temperature Status  
 IHi 15 °C / Lo 15 °C (pick up at PNL to WHC)  
 IHi     °C / Lo     °C (delivery at WHC from PNL)  
 IHi     °C / Lo     °C (at return to PNL from WHC)  
 IHi     °C / Lo     °C (at delivery from WHC to PNL)

(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 373-7076Project Designation/Sampling Locations 200 West Tank Farm  
241-BY-108 Tank Vapor Sample SAF S6022  
(ISVS Cart)

Collection date 03 - 28 - 96

Preparation date 03 - 22 - 96

Ice Chest No.

Field Logbook No. WHC-147-85-8  
N-647-8

Erco Hi/Lo thermometer No. PNL-T-001

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

S6022 - A31 . 854 Collect TST Sample # 1  
S6022 - A32 . 856 Collect TST Sample # 2  
S6022 - A33 . 857 Collect TST Sample # 3

S6022 - A40 . 859 Open, close & store TST Field Blank # 3  
S6022 - A41 . 868 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	
J L Julia	03-25-96	1230	J A Edwards	03-25-96	1230	
J A Edwards	03-25-96	1330	K.J. Lewis	03-25-96	1330	
K.J. Lewis	3-28-96	0700	R.D. Mahon	3-28-96	0700	
R.D. Mahon	04-16-96	0840	T. Butler	4-16-96	0840	
T. Butler	4-16-96	1035	J L Julia	4-16-96	1035	

## Final Sample Disposition

## Comments:

PNL (only) Checklist

0	Media labeled and checked?	Y/N
0	Letter of instruction?	Y/N
0	Media in good condition?	Y/N
0	COC info/signatures complete?	Y/N
0	Sorbents shipped on ice? (<5°C)	Y/N
0	Hi/Lo thermometer - Keep upright!	Y/N
0	Hi/Lo thermometer	Y/N
0	Rad release stickers on samples?	Y/N
0	Activity report from 222S?	Y/N
0	RSR/copy? (a ≤100/B ≤400 pCi/g)	Y/N
0	COC copy for LRB, RIDS filed?	Y/N

POC [Signature]

## 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	°C/Lo	°C (at delivery from WHC to PNL)

(Revised 06/21/95 PNL)

## **Distribution List**

**PNNL-11271**

### **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