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Pacific Northwest
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U.S. Department of Energy

TANK VAPOR CHARACTERIZATION PROJECT

Headspace Vapor Characterization of Hanford Waste Tank 241-S-102: Results from Samples Collected on 01/26/96

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

July 1996

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

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Richland, Washington 99352

Summary

This report describes the analytical results of vapor samples taken from the headspace of waste storage tank 241-S-102 (Tank S-102) at the Hanford Site in Washington State. The results described in this report were obtained to compare vapor sampling of the tank headspace using the Vapor Sampling System (VSS) and In Situ Vapor Sampling (ISVS) system with and 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 hydrocarbons (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

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

Glossary

CCV	continuing calibration verification
COC	chain of custody
DIW	deionized water
EPA	U.S. Environmental Protection Agency
EQL	estimated quantitation limit
GC/FID	gas chromatography/flame ionization detector
GC/MS	gas chromatograph/mass spectrometer
GC/TCD	gas chromatography/thermal conductivity detection
HEPA	high efficiency particulate air
IDL	instrument detection limit
IS	internal standard
ISVS	In Situ Vapor Sampling
LLS	low level standard
MDL	method detection limit
NIST	National Institute for Standards and Technology
% D	percent difference
PNL	previous designation for Pacific Northwest Laboratory
PNNL	Pacific Northwest National Laboratory
ppbv	part per billion by volume
ppm	parts per million
ppmv	part per million by volume
QA	quality assurance
QC	quality control
RPD	relative percent difference
RSD	relative standard deviation
SAP	sample and analysis plan
ST DEV	standard deviation
STP	standard temperature and pressure
SUMMA™	stainless steel, passivated interior canister
TBP	tributyl phosphate
TIC	tentatively identified compound
TNMHC	total non-methane hydrocarbons
TST	triple sorbent trap
UHP	ultra high purity
UQL	upper quantification limit
VAL	Vapor Analytical Laboratory
VSS	vapor sampling system
WHC	Westinghouse Hanford Company

Contents

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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) with and without particulate prefiltration. Samples were collected from the headspace of waste storage tank 241-S-102 (Tank S-102) at the Hanford Site in Washington State. Pacific Northwest National Laboratory (PNNL)[a] was contracted by Westinghouse Hanford Company (WHC) to provide sampling devices and analyze samples for water, ammonia, permanent gases, total nonmethane hydrocarbons (TNMHCs, 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 S6007, S6008, and S6009. Samples were collected by WHC on January 26, 1996, using the VSS, a truck-based sampling method using a heated probe; and the ISVS with and without particulate prefiltration.

Sampling devices and controls provided for this job included 25 sorbent trains for water and ammonia (18 sample trains and 7 field blanks); 23 SUMMA™ canisters for permanent gases, TO-12 and volatile organic analytes (18 samples and 5 ambient canisters); and 26 TSTs for organic analytes (18 samples, 6 field blanks, and 2 trip blanks). The samples and controls were provided to WHC on January 19 and 26, 1996. Exposed samples and controls were returned to PNNL on January 30, 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[b], 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 hydrocarbons* 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 (QC) 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 S-102 comparison study. Appendix G contains the completed COC forms.

2.0 Analytical Results

Samples obtained by WHC from the headspace of Tank S-102 on January 26, 1996, (Sample Jobs S6007, S6008, and S6009) 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 S-102 for the three sampling methods can be found in Appendix A of this report. Table 2.1 presents the mean concentration values for these two analytes. Mean water concentration values ranged from 12.9 mg/L in the ISVS samples without HEPA filtration to 14.1 mg/L in the VSS samples. The mean H_2O concentration value for the ISVS samples with HEPA filtration was 13.0 mg/L. Mean NH_3 concentration values ranged from 595 parts per million by volume (ppmv) for the ISVS samples without HEPA filtration to 622 ppmv for the VSS samples. The mean ammonia concentration value for the ISVS samples with HEPA filtration was 615 ppmv.

Table 2.1. Comparison of Water and Ammonia Mean Values for Samples Collected from the Headspace of Tank S-102 Using VSS and ISVS With and Without Particulate Filtration

	VSS	ISVS With Filtration	ISVS Without Filtration
Water (mg/L)	14.1	13.0	12.9
Ammonia (ppmv)	622	615	595

2.2 Permanent Gases

The complete results of the permanent gas analyses of Tank S-102 for the three sampling methods can be found in Appendix B of this report. Table 2.2 presents the mean concentration values for the five permanent gases measured. Methane (CH_4) and carbon monoxide (CO) were not observed in any of the samples. Only two permanent gases, hydrogen (H_2) and nitrous oxide (N_2O) were measured above the analytical method estimated quantitation limit (EQL). Average concentrations of CO_2 were below the IDL (<2 ppmv) in the VSS samples, and ranged from 7.5 ppmv in the ISVS samples without HEPA filtration to 33 ppmv in the ISVS samples without HEPA filtration. Based on a mean ambient concentration of 411 ppmv for CO_2, this indicates a 7% to 8% dilution with ambient air. This conclusion is consistent with analogous results for H_2 and N_2O in the ISVS samples with HEPA filtration also suggesting a 7% to 8% dilution with ambient air.

Table 2.2. Comparison of Permanent Gas Mean Values for Samples Collected from the Headspace of Tank S-102 Using VSS and ISVS With and Without Particulate Filtration

	VSS	ISVS With Filtration	ISVS Without Filtration
H_2 (ppmv)	737	689	725
CO_2 (ppmv)	2 U	33	8 J
N_2O (ppmv)	696	650	690
CH_4 (ppmv)	4 U	4 U	4 U
CO (ppmv)	3 U	3 U	3 U

2.3 Total Non-Methane Hydrocarbons

The complete results of the U.S. Environmental Protection Agency (EPA) TO-12 analyses for TNMHCs in Tank S-102 can be found in Appendix C of this report. A summary of those results can be found in Table 2.3. The TNMHC average concentrations ranged from 16 mg/m^3 for both the VSS samples and the ISVS samples with particulate filtration to 18 mg/m^3 for the ISVS samples without particulate filtration.

Table 2.3. Comparison of TO-12 Mean Values for Samples Collected from the Headspace of Tank S-102 Using VSS and ISVS With and Without Particulate Filtration

	VSS	ISVS With Filtration	ISVS Without Filtration
TO-12 (mg/m^3)	16	16	18

2.4 Organic Compounds from SUMMA™ Canisters

The complete set of SUMMA™ volatile organic analysis results for Tank S-102 is found in Appendices D and F of this report. Table 2.4 presents a summary of these data.

Results show methanol and ethanol to be the most abundant compounds identified in the SUMMA™ canister samples, with average values of methanol ranging from 5305 parts per billion by volume (ppbv) to 5645 ppbv and ethanol ranging from 5040 ppbv to 5428 ppbv. Concentrations of both dodecane and tetradecane were below instrument detection limit (IDL) for the canister samples, indicating an absence of these compounds. Tributyl phosphate (TBP) was not observed as a TIC in any of the SUMMA™ canister samples.

Based on the average values for each of the sampling methods, the highest concentration of acetonitrile was observed in the VSS samples. The highest concentrations of methanol, acetone, and tridecane were observed in the ISVS samples with HEPA filtration. The highest concentrations of

ethanol, hexane and 1-butanol were observed in the ISVS samples without HEPA filtration. An identical average propanol and tetrahydrofuran concentrations were observed in the ISVS samples with and without HEPA filtration. Many of the differences in average concentration values may not be significant for the different sampling methods.

2.5 Organic Compounds from Triple Sorbent Traps

Complete results for the TST volatile organic analysis of Tank S-102 are found in Appendices E and F of this report. Table 2.5 presents a summary of those results.

In summary, methanol and ethanol were the most abundant compounds identified in each of the trap samples. Tributyl phosphate was not observed in any samples from Tank S-102. Based on the average values for each of the sampling methods, the highest concentrations of methanol, ethanol and tetrahydrofuran were observed in the VSS samples. The highest concentration of acetonitrile, acetone, propanol, hexane, 1-butanol, dodecane, and tetradecane were observed in the ISVS samples without HEPA filtration. An identical average tridecane concentration was observed in the ISVS samples with and without HEPA filtration. Many of the differences in average concentration values may not be significant for the different sampling methods. The most notable differences in concentration between the different sampling methods occurred for hexane and 1-butanol. Hexane and 1-butanol concentrations for the VSS samples were significantly lower than concentrations in the ISVS samples without HEPA filtration samples. The concentrations of ISVS samples with HEPA filtration were between the concentrations of VSS samples and ISVS samples without HEPA filtration. The differences in the average concentrations of hexane and 1-butanol may have been caused by contamination from the tape used to wrap the sample bundles.

The results of the field and trip blank sample analysis indicate that significant contamination of the TST samples was caused by the tape used to seal the bundles. Compounds observed in the blank samples included but were not limited to methanol, 1-butanol, hexane, heptane, methyl hexane, and methyl cyclohexane.

When the ISVS samples were physically exposed to the tank vapor prior to actual sample collection, some tank specific compounds diffused in the TSTs. This problem becomes progressively worse with decreasing sample volume size; therefore, sampling should be performed as promptly as possible following insertion of the sampling bundle into the tank to minimize the effects of passive sampling.

Table 2.4 Summary of SUMMA™ Sample Results for Samples Collected from the Headspace of Tank S-102 on 1/26/96

VSS Truck Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)		TETRADECANE (ppbv)	TBP (ppbv)
Average	5392	Y E	5422	Y E	43	433	37	47	7.7	426	U	0.8	J	U	Z
ST DEV	524		789		8	120	4	3	0.5	27		0.2			
% RSD	10		14		17	26	10	6	7	6		21			
ISVS with HEPA															
Average	5645	Y E	5040	Y E	34	469	39	43	9.0	425	U	0.9	J	U	Z
ST DEV	794		945		11	176	4	3	2.6	53		0.2			
% RSD	14		17		29	36	12	5	27	12		18			
ISVS without HEPA															
Average	5305	Y E	5428	Y E	27	447	39	47	12	438	U	0.7	J	U	Z
ST DEV	929		918		5	123	5	1	2	42		0.1			
% RSD	16		16		17	25	12	1	13	9		20			

Data Qualifier Flag

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

U Target compound not detected at or above the IDL.

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

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

E Target compound exceeds upper quantification limit (UQL).

Table 2.5 Summary of Triple Sorbent Trap Sample Results for Samples Collected from the Headspace of Tank S-102 on 1/26/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
Average	4987 E Y	14710 E Y	66	259	56	54	9.3	625	1.6 J	2.3 J	1.7 J	< 0.8 Y
ST DEV	725	1552	11	14	6.7	0.8	0.5	28	0.2	0.6	0.8	
% RSD	14	10	16	5	11	1	6	5	10	24	42	
ISVS with HEPA												
Average	3602 E Y	10889 E Y	60	237	49	45	95	510	1.6 J	3.1 J	1.5 J	< 0.8 Y
ST DEV	827	2009	7.2	17	8.2	4.8	29	58	0.3	1.4	0.9	
% RSD	22	17	11	8	15	11	28	11	15	42	59	
ISVS without HEPA												
Average	4552 E Y	13894 E Y	89	315	58	52	172	907	3.5 J	3.1 J	2.3 J	< 0.8 Y
ST DEV	643	1113	9.4	26	9.0	2.7	16	61	0.2	0.8	1.1	
% RSD	13	8	10	8	14	5	9	6	7	23	46	

Data Qualifier Flag

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

3.0 Conclusions

The air concentrations of H_2O and NH_3, permanent gases, total non-methane hydrocarbons, and organic vapors were determined from samples from the headspace of Tank S-102 sampled on January 26, 1996. WHC sample job numbers were S6007, S6008, and S6009. The gas and vapor concentrations were based either on whole-volume samples (SUMMA™ canisters) or on triple sorbent traps exposed to sample flow. In the case of the canisters, the concentrations were based on analytical results of subsamples obtained directly from the canisters. In the case of the sorbent traps, concentrations were based on analyses by the VAL and sample volumes reported by WHC. Known sampling and analytical variances from established QA requirements, where significant, were documented in this report, as required by the SAP (Homi 1996).

4.0 Reference and Further Reading

Reference

Homi, C.S. 1996. *Sampling and Analysis Plan for Tank Vapor Sampling Comparison Test.* WHC-SD-WM-TSAP-073, Rev. OB, 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 with and without particulate filtration. Blanks, spiked blanks (when requested), 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 (NH_3) and water (H_2O). 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 NH_3 and mass. Samples were prepared, handled, and disassembled as described in Technical Procedure PNL-TVP-09[(a)]. 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 NH_3 and H_2O (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 NH_3 sorbent traps contained carbon beads impregnated with sulfuric acid; nominally, 500 mg were contained in the primary and 250 mg in the breakthrough sections. The NH_3 was chemisorbed as ammonium sulfate [$(NH_4)_2SO_4$]. 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°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 μmol, by the volume of the dried tank air sampled in moles. The micromolar sample mass was determined by dividing the compound mass, in μg, by the molecular weight of the compound, in g/mol. The molar sample volume was determined, excluding water vapor, by dividing the standard sample volume (at 0°C and 760 torr), in L, by 22.4 L/mol. For example, the concentration by volume of a 3.00-L sample containing 75.0 μg of NH_3 is given by

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

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°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. All were compiled in PNL-MA-599.

A.2.1 Ammonia Analysis. The sorbent material from the NH_3-selective sorbent traps was placed into labeled 20-mL glass scintillation vials. Vials containing front-, or primary-, section sorbent material were treated with 10.0 mL of deionized water (DIW), and vials containing back-up-section sorbent material were treated with 5.0 mL of DIW. After extraction, the NH_3 sorbent traps were analyzed using the selective ion electrode procedure PNL-ALO-226[(a)]. Briefly, this method includes 1) preparing a 1000-μg/mL (ppm) NH_3 stock standard solution from dried reagent-grade NH_4Cl and DIW, 2) preparing 0.1-, 0.5-, 1.0-, 10-, and 100-ppm NH_3 working calibration standards by serial dilution of the freshly made stock standard, 3) generating an initial calibration curve from the measured electromotive force signal versus 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 NH_4Cl standard from an independent source, after analyzing every five or six samples, 5) continuing this sequence until all samples of the batch have been measured, including duplicates and spiked samples, and 6) remeasuring the complete set of calibration standards (at the end of the session). Electromotive force (volts) signal measurements obtained for samples are compared to those for standards, either graphically or algebraically (using linear regression) to determine NH_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. Blanks were included to provide information on uncertainty.

A.3 Quality Assurance/Quality Control

Analytical work was performed according to quality levels identified in the project QA plan and several PNNL documents. The samples were analyzed following PNNL Impact Level II. The PNNL documents include PNL-MA-70 (Part 3), PNL-ALO-212, PNL-ALO-226, and ETD-002. A summary of the analysis procedures and limits for the target inorganic compounds is provided in Table A.1. The table also shows generic expected notification ranges and describes related target analytical precision and accuracy levels for each analyte; the information in the table is based on the data quality objective assessment by Osborne et al. (1995). From the table, it can be seen that the EQL 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 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 NH_3 analyses, the accuracy of laboratory measurements by selective ion electrode was estimated to be ± 5% relative, independent of concentration at 1 μg/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[a]

Analyte	Formula	Procedure	EQL[b] (μg)	EQL[b] (ppmv)	Notification Level[c] (ppmv)
Ammonia	NH_3	PNL-ALO-226	1.0	0.5	≥ 150
Mass (water)[d]	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 ± 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 ± 0.1 mg, or much less than 1% of the mass changes of most samples. The analytical accuracy of measurements of the change in mass of sorbent trains, based on the variability in mass change of field-blank sorbent trains, is determined for each sample job and is typically about ± 1 mg per five-trap sorbent train.

A.4 Water and Ammonia Sample Results

A total of 25 water/ammonia trap samples, consisting of 18 tank samples and seven field blank samples, were returned to the laboratory on January 30, 1996, under WHC COC numbers 100026, 100029, and 100032. The samples were analyzed for water on February 2, 1996 and for ammonia on February 13, 1996.

Table A.2 lists results of the water and ammonia analysis from samples collected from the headspace of Tank S-102. These samples were collected through the VSS and through the ISVS with and without particulate filtration. A total of six samples each were collected with the three different sampling methods. Mean water concentration values were 12.9 mg/L, 13.0 mg/L, and 14.1 mg/L in the ISVS samples without particulate filtration, ISVS samples with particulate filtration, and VSS samples, respectively. Results show water concentrations were comparable for the three methods.

Mean ammonia concentration values ranged from a low of 595 ppmv for the ISVS samples without particulate filtration to a high of 622 ppmv for the VSS samples. The mean ammonia concentration for the ISVS samples with particulate filtration was 615 ppmv.

Table A.2 Water and Ammonia Analysis Results for Samples Collected from the Headspace of Tank S-102 on 1/26/96

VSS Truck Samples	H_2O mg/L	NH_3 ppmv
S6007-A18.71S	14.4	622
S6007-A19.72S	14.1	598
S6007-A20.73S	14.0	642
S6007-A21.74S	13.9	622
S6007-A22.75S	14.0	632
S6007-A23.76S	14.2	618
Average	**14**	**622**
% RSD	**1**	**2**
ISVS with HEPA		
S6008-A42.80S	12.4	588
S6008-A43.81S	12.9	616
S6008-A44.82S	12.6	614
S6008-A51.83S	13.7	621
S6008-A52.84S	12.6	622
S6008-A53.85S	13.6	627
Average	**13**	**615**
% RSD	**4**	**2**
ISVS without HEPA		
S6009-A66.88S	12.0	584
S6009-A67.89S	12.9	618
S6009-A68.90S	12.9	581
S6009-A69.91S	12.1	550
S6009-A70.92S	13.4	613
S6009-A71.93S	13.8	626
Average	**13**	**595**
% RSD	**5**	**5**

A.5 References

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

Ligotke, M. W., K. H. Pool, and B. D. Lerner. 1994. *Vapor Space Characterization of Waste Tank 241-C-103: Inorganic Results from Sample Job 7B (5/12/94 - 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[a]. The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis. 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[b] 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_2), carbon dioxide (CO_2), carbon monoxide (CO), methane (CH_4), and nitrous oxide (N_2O), 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_2, N_2O, and CH_4 using Helium (He) as the carrier gas. A second GC analysis is performed for H_2 (using nitrogen as the carrier gas) to enhance the signal sensitivity and lower the detection limit for this analyte. The permanent gases and the derived EQLs are listed in Table B.1.

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

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

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

Analyte	Formula	Procedure	Instrument Detection Limit (ppmv)	Estimated Quantitation Limit (ppmv)
Carbon Dioxide	CO_2	PNL-TVP-05	2.4	24
Carbon Monoxide	CO	PNL-TVP-05	3.2	32
Methane	CH_4	PNL-TVP-05	4.3	43
Hydrogen	H_2	PNL-TVP-05	3.1	31
Nitrous Oxide	N_2O	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_2, N_2O, and CH_4 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_2, except the carrier gas was changed to N_2. 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 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_2 reagent blank, an ambient-air sample collected $\sim$10 m upwind of Tank S-102, and the ambient air collected through the VSS 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 for the permanent gas analyses from samples collected from the headspace of Tank S-102 and ambient air collected near Tank S-102 on January 26, 1996. These samples were collected through the VSS and through the ISVS with and without HEPA filtration. A total of six tank headspace samples each were collected for the three different sampling methods. The samples were analyzed on February 7, 8, and 9, 1996. Replicate analyses of SUMMA™ canisters were conducted on three samples within each sampling method set.

Hydrogen and N_2O values were above the EQL in all the tank headspace samples. Average H_2 concentrations were 689 ppmv, 725 ppmv, and 737 ppmv for the ISVS samples with HEPA filtration, the ISVS samples without HEPA filtration, and the VSS samples, respectively. Corresponding average N_2O concentrations for the same three sampling methods were 650 ppmv, 690 ppmv, and 696 ppmv, respectively.

There was evidence that there may have been a small leak in the transfer tubing used to fill SUMMA™ canisters in the ISVS method. Specifically, SUMMA™ samples collected using the ISVS method from Tank S-102 appear to have been diluted with ambient air. The mean levels of CO_2 in the VSS samples (0 ppmv) and the ISVS samples without HEPA filtration (7.5 ppmv) are less than the EQL, while the ISVS samples with HEPA filtration had a mean concentration of 33 ppmv. Based on a mean ambient concentration of 411 ppmv for CO_2, this indicates a 7% to 8% dilution with ambient air. This conclusion is consistent with analogous results for H_2 and N_2O in the ISVS samples with HEPA filtration suggesting a 7% to 8% dilution with ambient air.

Table B.2 Permanent Gas Analysis Results for Samples Collected from the Headspace of Tank S-102 and Ambient Air Collected Near Tank S-102 on 1/26/96

VSS Truck Samples	H_2 (ppmv)		CO_2 (ppmv)		N_2O (ppmv)		CH_4 (ppmv)		CO (ppmv)	
S6007-A01.001 (Ambient)	3	U	560		2	U	4	U	3	U
S6007-A02.074 (Ambient)	3	U	406		2	U	4	U	3	U
S6007-A03.082	736		2	U	707		4	U	3	U
S6007-A04.083	740		2	U	692		4	U	3	U
S6007-A15.084	738		2	U	699		4	U	3	U
S6007-A16.097	738		2	U	700		6	J	3	U
S6007-A27.108	736		2	U	695		4	U	3	U
S6007-A28.120	736		2	U	702		4	U	3	U
S6007-A87.086	740		2	U	689		4	U	3	U
S6007-A88.089	734		2	U	689		4	U	3	U
Average	**737**		**2**	**U**	**696**		**4**	**U**	**3**	**U**
% RSD	**0**				**1**					
S6007-A15.084 (Rep)	735		2	U	705		4	U	3	U
S6007-A28.120 (Rep)	733		2	U	700		4	U	3	U
S6007-A88.089 (Rep)	730		4	J	688		4	U	3	U
ISVS with HEPA										
S6008-A33.123 (Ambient)	3	U	409		2	U	4	U	3	U
S6008-A34.127 (Ambient)	3	U	413		2	U	4	U	3	U
S6008-A36.128	679		39		646		4	U	3	U
S6008-A37.129	675		39		636		4	U	3	U
S6008-A38.134	680		39		640		4	U	3	U
S6008-A45.136	699		33		647		4	U	3	U
S6008-A46.137	697		23	J	662		4	U	3	U
S6008-A47.153	706		25		671		4	U	3	U
Average	**689**		**33**		**650**		**4**	**U**	**3**	**U**
% RSD	**2**		**22**		**2**					
S6008-A36.128 (Rep)	677		41		644		4	U	3	U
S6008-A37.129 (Rep)	665		38		646		4	U	3	U
S6008-A45.136 (Rep)	695		29		659		4	U	3	U
S6008-A38.134 (Rep)	681		41		639		4	U	3	U
ISVS without HEPA										
S6009-A35.165 (Ambient)	3	U	413		2	U	4	U	3	U
S6009-A54.167	720		9.5	J	685		4	U	3	U
S6009-A55.169	721		14	J	679		4	U	3	U
S6009-A56.170	729		6.8	J	693		4	U	3	U
S6009-A57.229	728		4.4	J	691		4	U	3	U
S6009-A58.230	721		5.2	J	695		4	U	3	U
S6009-A59.248	733		5.2	J	697		4	U	3	U
Average	**725**		**7.5**	**J**	**690**		**4**	**U**	**3**	**U**
% RSD	**1**		**49**		**1**					
S6009-A54.167 (Rep)	725		12	J	687		4	U	3	U
S6009-A56.170 (Rep)	731		6	J	688		4	U	3	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.

Appendix C

Tank Vapor Characterization:

Total Non-Methane Hydrocarbons

Appendix C

Tank Vapor Characterization: Total Non-Methane Hydrocarbons

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[a]. The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis. 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[b], which is similar to U.S. Environmental Protection Agency (EPA) compendium Method TO-12. The method detection limits in the sub mg/m^3 are required to determine total non-methane hydrocarbon (TNMHC) 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 TNMHCs are carried by a UHP He stream to the GC equipped with an FID where gross organic content is detected and measured.

The GC oven is programmed to run at a 150°C isothermal temperature. Chromatographic separation is not needed in this method since quantitation is from the entire FID response over the run time.

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

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

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

C.3 Quality Assurance/Quality Control

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

The TNMHC is calibrated by using propane as the calibration standard and using that response factor as an external standard method. The instrument calibration mixture for the PNL-TVP-08 analysis consists of National Institute for Standards and Technology (NIST) 99.999% propane analyzed using a five-point, multi-level, linear regression curve.

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^3 of TNMHCs. Second, an instrument continuing calibration is run using 100-mL UHP propane analyzed using the response factor as an external standard method followed by one blank volume of Aadco air.

C.3.1 Quantitation Results of Target Analytes. The mg/m^3 was derived from the five-point multi-level calibration curve from the propane standard using the following equation:

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

The ng/m^3 concentrations are calculated from mg/m^3 using the equation:

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

C.4 Total Non-Methane Hydrocarbons Sample Results

Table C.1 lists results for the TO-12 gas analyses from samples collected from the headspace of Tank S-102 and ambient air collected near Tank S-102 on January 26, 1996. These samples were collected through the VSS and through the ISVS with and without HEPA filtration. A total of eight headspace samples each were collected during the VSS sampling. Six samples were collected during the ISVS sampling with and without HEPA filtration. Five ambient air samples were also collected during the course of the S-102 sampling event. Replicate analyses of SUMMA™ canister samples were also conducted on one sample from each sampling method set. All samples were analyzed on February 7 and 8, 1996. For the five ambient air samples, TO-12 concentrations ranged from a low of 0.2 mg/m^3 (below the EQL) to a high of 1.4 mg/m^3. Both of these values were observed for the VSS method. Measurements for the other two methods were comparable, ranging from 0.6 mg/m^3 (below the EQL) to 0.9 mg/m^3. Average TO-12 concentrations for the tank samples were nearly identical at 16 mg/m^3, 16 mg/m^3, and 18 mg/m^3, for the VSS samples, ISVS samples with HEPA, and without HEPA filtration, respectively.

Table C.1 TO-12 Analysis Results for Samples Collected from the Headspace of Tank S-102 and Ambient Air Near Tank S-102 on 1/26/96

VSS Truck Samples	TO-12 mg/m3	
S6007-A01.001 (Ambient)	0.2	J
S6007-A02.074 (Ambient)	1.4	
S6007-A03.082	15	
S6007-A04.083	16	
S6007-A15.084	16	
S6007-A16.097	15	
S6007-A27.108	17	
S6007-A28.120	17	
S6007-A87.086	16	
S6007-A88.089	16	
Average	**16**	
% RSD	**4**	
S6007-A15.084 (Rep)	16	
S6007-A28.120 (Rep)	17	
S6007-A88.089 (Rep)	16	
ISVS Cart with HEPA		
S6008-A33.123 (Ambient)	0.6	J
S6008-A34.127 (Ambient)	0.9	
S6008-A36.128	15	
S6008-A37.129	16	
S6008-A38.134	15	
S6008-A45.136	18	
S6008-A46.137	16	
S6008-A47.153	15	
Average	**16**	
% RSD	**7**	
S6008-A36.128 (Rep)	14	
S6008-A37.129 (Rep)	14	
S6008-A45.136 (Rep)	18	
S6008-A38.134 (Rep)	15	
ISVS Cart without HEPA		
S6009-A35.165 (Ambient)	0.7	
S6009-A54.167	18	
S6009-A55.169	20	
S6009-A56.170	17	
S6009-A57.229	16	
S6009-A58.230	17	
S6009-A59.248	17	
Average	**18**	
% RSD	**9**	
S6009-A54.167 (Rep)	18	
S6009-A56.170 (Rep)	18	

Data Qualifier Flag

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

Appendix D

Tank Vapor Characterization:

Organic Compounds from SUMMA™ Canisters

Appendix D

Tank Vapor Characterization: Organic Compounds from SUMMA™ Canisters

D.1 Sampling Methodology

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant free according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-02[a]. The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis by PNNL Technical Procedure PNL-TVP-03[b], 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 μL 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-μm 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 67 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[a]. The standard calibration mix was analyzed using four aliquot sizes ranging from 30 mL to 200 mL, and a response factor for each compound was calculated. The GC/MS response for these compounds has been previously determined to be linearly related to concentration. Performance-based detection limits for the target analytes will be developed as a pool of calibration data becomes available.

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 67 volatile organic compounds. A gas mixture containing bromochloromethane, 1,4-difluorobenzene, chlorobenzene-d_5, 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

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(a) Pacific Northwest Laboratory. 8/94. *Preparation of TO-14 Volatile Organic Compounds Gas Standards,* PNL-TVP-06 (Rev. 0). PNL Technical Procedure, Richland, Washington.

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.

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^3 assumes standard temperature and pressure (STP) conditions of 760 torr and 273K and was calculated directly from the following equation:

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

D.4 Volatile Organic Sample Results

A total of 23 SUMMA™ samples, consisting of 18 tank samples and 5 ambient air samples, were returned to the laboratory on January 30, 1996, under WHC COC numbers 100024, 100027, and 100030. The majority of the samples were analyzed between March 11 and March 17, 1996.

The results from the GC/MS analyses of the tank headspace SUMMA™ samples are presented in Table D.2. The results of replicate analyses on single SUMMA™ canister samples are presented in Table D.3. The results of the blank samples 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 the 12 compounds selected for this tank comparison study. Between six and eight individual SUMMA™ canister samples were collected for each of the three different sampling methods. Individual compound values for each of the SUMMA™ canister results by sampling method were averaged and a standard deviation (ST DEV) and percent relative standard deviation (% RSD) value calculated. Methanol and ethanol were the most abundant compounds identified in each of the SUMMA™ canister samples. Dodecane and tetradecane were not observed in any samples from Tank S-102. Tributyl phosphate was not identified in any of the SUMMA™ canister samples. Based on the average values for each of the sampling methods, the highest concentration of acetonitrile was observed in the VSS samples. The highest concentrations of methanol, acetone, and tridecane were observed in the ISVS samples with HEPA filtration. The highest concentration of ethanol, hexane and 1-butanol were observed in the ISVS samples without HEPA filtration. Identical average propanol and tetrahydrofuran concentrations were observed in the ISVS samples with and without HEPA filtration. Many of the differences in average concentration values may not be significant for the different sampling methods.

Single SUMMA™ canister samples were analyzed in replicate for each of the three 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.

Instrument detection limits, precision, and accuracy have not been experimentally evaluated for methanol and ethanol. Sample results are flagged with a less-than symbol (<) when the absolute number of nanograms calculated in the sample is less than the lowest concentration standard used in the initial calibration. Results below the LLS were not reported. Methanol and ethanol results falling

within their calibration range are quantitative results as evidenced by a valid calibration for these compounds. It should be noted that these compounds are not currently part of the operating procedure.

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

Batch #1 (file identifier 16031101.b) - S6009-A55.169, S6007-A28.120, S6008-A45.136, S6009-A59.248, S6009-A55.169 REP, S6007-A04.083, S6008-A37.129;

Batch #2 (file identifier 16031201.b) - S6009-A58.230, S6008-A46.137, S6007-A03.082, S6008-A46.137 REP, S6008-A47.153, S6007-A16.097, S6009-A54.167, S6007-A88.089;

Batch #3 (file identifier 16031502.b) - S6007-A15.084, S6009-A56.170, S6008-A38.134, S6007-A27.108, S6009-A57.229, S6007-A15.084 REP, S6008-A36.128, S6007-A87.086;

Batch #4 (file identifier 16031701.b) - S6007-A01.001, S6007-A02.074, S6008-A33.123, S6008-A34.124, S6009-A35.165.

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

Batch #1:

1. Three target compounds (ethanol at 34.7%, acetonitrile at 35.9%, and tetradecane at 40.2%) surpassed the 30% RSD acceptance criteria for the initial calibration. Tetradecane low level standard failed the minimum response factor criterion of 0.05 for the initial calibration; however, tetradecane was not found in the tank samples. Ethanol data are flagged with an "E" indicating that results are above the upper quantitation limit (UQL) and cannot be considered quantitative. Acetonitrile was found in tank samples at concentrations between the EQL and UQL.

2. Four target compounds (acetonitrile, undecane, tridecane, and 1,2,4-trichlorobenzene) were outside the $\pm$ 25% difference (% D) acceptance criteria for the continuing calibration verification (CCV); however, the CCV passed the procedural criterion requiring $\pm$ 25% D passage for 85% of all target compounds. The compound 1,2,4-trichlorobenzene was not found in the tank samples. Undecane and tridecane were found in tank samples at concentrations between the EQL and IDL. Their concentration values may be over or under estimated because of the failed CCV. Acetonitrile was detected above the EQL and its concentration value may be over estimated because of the failed CCV.

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 40 minutes because WHC requested the sample analysis to be in pairs (VSS sample, ISVS sample with HEPA, and ISVS sample without HEPA) within a batch.

4. The internal standard quantification areas for the last two samples (S6007-A04.083, S6008-A37.129) of the sequence run slightly exceeded the internal standard percent recovery acceptance criteria (50% < quantification area < 200%) allowed by procedure PNL-TVP-03, Rev. 1. Although no internal standard areas percent recovery decreased to less than 45% of any CCV internal standard areas, target compound results found in these samples could be

affected. Changes in the internal standard areas were caused by water induced instrument fatigue. This problem is routinely observed with the HP5972 GC/MS system because of its poor pumping capacity and the high water and ammonia content in these samples. This instrument fatigue problem will continue until a larger GC/MS is used in the analysis.

Batch #2:

1. Three target compounds (ethanol at 34.7%, acetonitrile at 35.9%, and tetradecane at 40.2%) surpassed the 30% RSD acceptance criteria for the initial calibration. The tetradecane low level standard failed the minimum response factor criterion of 0.05 for the initial calibration; however, tetradecane was not found in the tank samples. Ethanol data are flagged with an "E" indicating the results are above the UQL and cannot be considered quantitative. Acetonitrile was found in tank samples at concentrations between the EQL and UQL.

2. Three target compounds (acetonitrile, acetone, and tridecane) were outside the $\pm$ 25% D acceptance criteria for the CCV; however, the CCV passed the procedural criterion requiring $\pm$ 25% D passage for 85% of all target compounds. Tridecane was found in tank samples at concentrations between the EQL and IDL. Acetone and tridecane concentration values may be over estimated because of the failed CCV.

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 2 hours and 39 minutes because WHC requested the sample analysis to be in pairs (VSS sample, ISVS sample with HEPA, and ISVS sample without HEPA) within a batch.

4. The internal standard quantification areas for tank samples meet the internal standard percent recovery acceptance criteria (50% $<$ quantification area $<$ 200%) allowed by procedure PNL-TVP-03, Rev. 1 except for S6007-A88.089. The percent recovery decreased to less than 46% for the third and fourth internal standard areas. The tridecane result was the only affected target compound of major interest to WHC. Changes in the internal standard areas were caused by water induced instrument fatigue. This problem is routinely observed with the HP5972 GC/MS system because of its poor pumping capacity and the high water and ammonia content in these samples. This instrument fatigue problem will continue until a larger GC/MS is used in the analysis.

Batch #3:

1. Pentane (41.7%) surpassed the 30% RSD acceptance criteria for the initial calibration. Although pentane was not one of the original target compounds of interests by WHC it was found in tank samples at concentrations between the EQL and UQL.

2. Six target compounds (acetone at 25.5%, pentane at 31.1%, methanol at 30.6%, ethanol at 28.1%, tridecane at 25.8%, and tetradecane at 41.1%) were outside the $\pm$ 25% D acceptance criteria for the CCV; however, the CCV passed the procedural criterion requiring $\pm$ 25% D passage for 85% of all target compounds. Tetradecane was not found in the tank samples. Methanol and ethanol concentration results are flagged with an "E" indicating the results exceed the UQL and cannot be considered quantitative. Acetone, pentane, and tridecane were found in tank samples at concentrations above the EQL. Their concentration values may be over or under estimated because of the failed CCV.

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 3 hours and 40 minutes because WHC requested the sample analysis to be in pairs (VSS sample, ISVS sample with HEPA, and ISVS sample without HEPA) within a batch.

4. The internal standard quantification area for all tank samples after the CCV met the internal standard percent recovery acceptance criteria (50% < quantification area < 200%) except S6008-A38.134 (second, third, and fourth internal standards); S6007-A27.108, S6007-A87.086 duplicate, and S6007-A87.086 (third internal standards) which were 7% less than the lower limit. Changes in the internal standard areas were caused by water induced instrument fatigue. This problem is routinely observed with the HP5972 GC/MS system because of its poor pumping capacity and the high water and ammonia content in these samples. 1-Butanol and tridecane results for S6008-A38.134 were the only affected target compounds of major interest to WHC. This instrument fatigue problem will continue until a larger GC/MS is used in the analysis.

5. Tetradecane was found in the Initial Calibration Blank above the EQL; however, it was not found in the Continuing Calibration Blank or in tank samples. Thus, no data results were affected.

Batch #4:

1. Pentane (41.7%) surpassed the 30% RSD acceptance criteria for the initial calibration. Although pentane was not one of the original target compounds of interest to WHC, it was found in tank samples at concentrations between the EQL and UQL.

2. Five target compounds (acetone at 92.6%, pentane at 47.1%, trichlorofluoromethane at 45.8%, ethanol at 25.3%, and tetradecane at 41.1%) were outside the ± 25% D acceptance criteria for the CCV. However, the CCV passed the procedural criterion requiring ± 25% D passage for 85% of all target compounds. Ethanol, pentane, and tetradecane were not found in the tank samples. Acetone and trichlorofluoromethane were found in tank samples at concentrations between the EQL and IDL. Their concentration values may be over or under estimated because of the failed CCV.

3. The internal standard quantification for the first two samples after the CCV met the internal standard percent recovery acceptance criteria (50% < quantification area < 200%); however, the third sample (third internal standard) and the last three samples were 13% less than the lower limit. Methanol and acetone results which had positive hits for the last three samples were the only affected target compounds of major interest to WHC. Changes in the internal standard areas were caused by water induced instrument fatigue. This problem is routinely observed with the HP5972 GC/MS system because of its poor pumping capacity and the high water and ammonia content in these samples. This instrument fatigue problem will continue until a larger GC/MS is used in the analysis.

4. Tetradecane was found in the Initial Calibration Blank above the EQL. However, it was not found in the Continuing Calibration Blank or in tank samples. Thus, no data results were affected.

Table D.2 SUMMA™ Sample Analysis Results for Samples Collected from the Headspace of Tank S-102 on 1/26/96

VSS Truck Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)		TETRADECANE (ppbv)	TBP (ppbv)
A03.082	5242	Y E	4760	Y E	32	451	34	50	7.4	464	U	0.8	J	U	Z
A04.083	6181	Y E	6154	Y E	40	252	33	43	7.9	395	U	0.7	J	U	Z
A15.084	4772	Y E	5180	Y E	38	413	41	50	8.6	431	U	0.7	J	U	Z
A16.097	4577	Y E	4146	Y E	56	595	33	48	7.3	425	U	0.9	J	U	Z
A27.108	5629	Y E	5970	Y E	50	556	41	46	7.8	418	U	0.9	J	U	Z
A28.120	5481	Y E	6457	Y E	40	276	41	48	8.0	434	U	1.1	J	U	Z
A87.086	5790	Y E	5753	Y E	41	471	37	43	7.2	384	U	0.5	J	U	Z
A88.089	5465	Y E	4958	Y E	49	452	37	48	7.2	455	U	0.9	J	U	Z
Average	**5392**	**Y E**	**5422**	**Y E**	**43**	**433**	**37**	**47**	**7.7**	**426**	**U**	**0.8**	**J**	**U**	**Z**
ST DEV	**524**		**789**		**8**	**120**	**4**	**3**	**0.5**	**27**		**0.2**			
% RSD	**10**		**14**		**17**	**26**	**10**	**6**	**7**	**6**		**21**			
ISVS with HEPA															
A36.128	6171	Y E	4403	Y E	47	534	42	42	7.1	386	U	1.1	J	U	Z
A37.129	6138	Y E	5988	Y E	25	213	34	39	10	367	U	0.7	J	U	Z
A38.134	6623	Y E	4892	Y E	32	607	46	42	7.6	478	U	1.0	J	U	Z
A45.136	5381	Y E	6429	Y E	46	280	36	46	14	379	U	0.9	J	U	Z
A46.137	4547	Y E	4203	Y E	36	564	38	42	7.6	450	U	0.7	J	U	Z
A47.153	5012	Y E	4326	Y E	20	615	40	45	7.9	487	U	0.8	J	U	Z
Average	**5645**	**Y E**	**5040**	**Y E**	**34**	**469**	**39**	**43**	**9.0**	**425**	**U**	**0.9**	**J**	**U**	**Z**
ST DEV	**794**		**945**		**11**	**176**	**4**	**3**	**2.6**	**53**		**0.2**			
% RSD	**14**		**17**		**29**	**36**	**12**	**5**	**27**	**12**		**18**			
ISVS without HEPA															
A54.167	5190	Y E	4783	Y E	23	451	34	47	13	448	U	0.7	J	U	Z
A55.169	4959	Y E	5984	Y E	33	310	45	47	13	511	U	0.9	J	U	Z
A56.170	5780	Y E	6010	Y E	25	489	41	47	13	445	U	0.7	J	U	Z
A57.229	6113	Y E	6117	Y E	34	540	41	46	11	420	U	0.5	J	U	Z
A58.230	3677	Y E	3844	Y E	26	597	32	46	10	392	U	0.6	J	U	Z
A59.248	6111	Y E	5830	Y E	23	293	39	46	10	411	U	U		U	Z
Average	**5305**	**Y E**	**5428**	**Y E**	**27**	**447**	**39**	**47**	**12**	**438**	**U**	**0.7**	**J**	**U**	**Z**
ST DEV	**929**		**918**		**5**	**123**	**5**	**1**	**2**	**42**		**0.1**			
% RSD	**16**		**16**		**17**	**25**	**12**	**1**	**13**	**9**		**20**			

Data Qualifier Flag

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

U Target compound not detected at or above the IDL.

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

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

E Target compound exceeds upper quantification limit (UQL).

Table D.3 Replicate Analysis of SUMMA™ Canisters for Samples Collected from the Headspace of Tank S-102 on 1/26/96

VSS Truck Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)		TETRADECANE (ppbv)	TBP (ppbv)
A15.084	4772	E	5180	E	38	413	41	50	8.6	431	U	0.7	J	U	Z
A15.084 Rep	5391	E	6182	E	46	513	35	44	7.8	346	U	0.7	J	U	Z
Relative Percent Difference	12		18		19	22	16	13	10	22		0			
ISVS with HEPA															
A46.137	4547	E	4203	E	36	564	38	42	7.6	450	U	0.7	J	U	Z
A46.137 Rep	5898	E	5397	E	44	458	40	50	8.6	514	U	0.7	J	U	Z
Relative Percent Difference	26		25		20	21	5	17	12	13		0			
ISVS without HEPA															
A55.169	4959	E	5984	E	33	310	45	47	13	511	U	0.9	J	U	Z
A55.169 Rep	6625	E	6396	E	31	285	37	44	12	430	U	U	J	U	Z
Relative Percent Difference	29		7		6	8	20	7	8	17					

Data Qualifier Flag

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

U Target compound not detected at or above the IDL.

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

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

E Target compound exceeds upper quantification limit (UQL).

Table D.4 SUMMA™ Blank Sample Analysis Results for Samples Collected from the Headspace of Tank S-102 on 1/26/96

Blank Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)	ACETONE (ppbv)		PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)		DODECANE (ppbv)	TRIDECANE (ppbv)		TETRADECANE (ppbv)		TBP (ppbv)
Upwind Ambient	< 19	Y	< 13	Y	U	4.0	J	U	U	U	U		U	U		U		Z
Upwind Through VSS	< 19	Y	< 13	Y	U	10		U	U	U	U		U	U		U		Z
Bundle A W/HEPA	< 19	Y	< 13	Y	U	12		U	U	U	2.0	J	U	1.0	J	U		Z
Bundle B W/HEPA	87		< 13	Y	U	19		U	U	U	2.0	J	U	1.0	J	U		Z
Bundle C WO/HEPA	< 19	Y	< 13	Y	U	7.0		U	U	U	1.0	J	U	3.0	J	2.0		Z
Lab Control BLK	< 19	Y	< 13	Y	U	1.0	J	U	U	U	0.5	J	U	0.2	J	0.5	J	Z

Data Qualifier Flag

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

U Target compound not detected at or above the IDL.

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

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

Appendix E

Tank Vapor Characterization:

Organic Compounds from Triple Sorbent Traps

Appendix E

Tank Vapor Characterization: Organic Compounds from Triple Sorbent Traps

E.1 Sampling Methodology

Samples are collected on Supelco 300 graphite-based triple sorbent traps (TSTs). Before field deployment, each trap is heated to 380°C under inert gas flow for a minimum of 60 min. Tubes are prepared in batches with each tank sampling job constituting one batch. One tube is selected from each batch and run immediately to verify cleanliness. All remaining tubes in the batch receive equal amounts of three surrogate compounds (hexafluorobenzene, toluene-d8, and bromobenzene-d5). One per batch tube is run immediately to verify successful addition of surrogate spikes to that batch. Tubes are then placed in individually labeled plastic shipping tubes (Supelco TD^3), 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[(a)], 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 graphetized 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 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.

(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.

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 and CCV were performed for methanol and ethanol. The 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. 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. Performance-based detection limits for the target analytes will be developed as a pool of calibration data becomes available.

Table E.1. Reported Organic Analytes of Interest

Methanol	*Acetone*
Ethanol	*Acetonitrile*
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 13 compounds shown in Table E.1. A gas mixture containing difluorobenzene, chlorobenzene-d_5, and 1,4 bromofluorobenzene was used as an IS for all calibration standard and sample analyses. Analyte responses from sample components, ISs, and standards were obtained from the extracted ion plot from their selected mass ion. A continuing calibration was generated by calculating the relative response ratios of the IS to calibration standard responses and plotting the ratios against the ratio of the calibration-standard concentration (in ppbv) to the IS concentration. Once it is determined that the relative response is linear with increasing concentration, an average response factor is calculated for each target analyte and used to determine the concentration of target compounds in each sample.

E.3.1 Quantitation Results of Target Analytes. The quantitative-analysis results for the target analytes were calculated directly from the calibration curve generated using the IS method described above and in PNL-TVP-10. It should be noted that the relative response factor value for tetrachloroethylene, 1-2-dibromoethane, and toluene were calculated using the first IS, not the second IS, which is nearest in retention time to these compounds. The second IS will be used to calculate the relative response factor for these compounds for subsequent analyses. The conversion from ppbv to

mg/m^3 assumes STP conditions of 760 torr and 273K and was calculated directly from the following equation:

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

E.4 Triple Sorbent Trap Volatile Organic Sample Results

Twenty-six TSTs consisting of 18 samples, 6 field blanks, and 2 trip blanks were returned to the laboratory on January 30, 1996, under WHC COC numbers 100025, 100028, and 100031. The majority of the samples were analyzed between March 5 and March 12, 1996.

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

Table E.2 lists the quantitative results for the 12 compounds selected for this tank comparison study. Six individual TST samples were collected for the VSS and ISVS with HEPA filtration sampling methods. Five TST samples were collected for the ISVS without HEPA filtration method. The individual compound values for each of the TST samples for each sampling method were averaged and a ST DEV and % RSD values calculated. Ethanol and methanol were the most abundant compounds identified in each of the TST samples. Tributyl phosphate was not observed in any samples from Tank S-102. Based on the average values for each of the sampling methods, the highest concentrations of methanol, ethanol, and tetrahydrofuran were observed in the VSS samples. The highest concentration of acetonitrile, acetone, propanol, hexane, 1-butanol, dodecane, and tetradecane were observed in the ISVS samples without HEPA filtration. An identical average tridecane concentration was observed in the ISVS samples with and without HEPA filtration. Many of the differences in average concentration values may not be significant for the different sampling methods. The most notable differences in concentration between the different sampling methods occurred for hexane and 1-butanol. Concentrations of hexane and 1-butanol were significantly lower for the VSS samples than in the ISVS samples without HEPA filtration. The ISVS samples with HEPA filtration concentration for hexane was between the VSS samples and ISVS samples without HEPA filtration concentrations. The differences in the average concentrations between the sample methods for hexane and 1-butanol may have been caused by contamination from the tape used to wrap the sample bundles.

Single TST samples were analyzed in replicate for each of the three different sampling methods. The relative percent differences (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. The results indicate significant contamination of the TST samples may have been caused by the tape used to seal the bundles. Compounds observed in the blank samples included but were not limited to methanol, 1-butanol, hexane, heptane, methyl hexane, and methyl cyclohexane. The resulting chromatogram and mass spectral identification showed a good pattern match to many of the compounds seen at similarly high levels in the ISVS samples.

Samples were collected from tank S-102 by the VSS and ISVS methods (with and without HEPA filters) on 1/26/96. The samples were received in the Tank Vapor Laboratory on 1/30/96 and stored

pending analysis. Samples were analyzed under the protocols of PNL-TVP-10, Rev. 2 with the initial calibration performed 3/1/96 and quantitated against CCVs run at the beginning of each batch for the target compounds listed in the method. The samples were subjected to GC/MS analysis from 3/5/96 through 3/12/96.

Instrument detection limits, precision, and accuracy have not been experimentally evaluated for methanol, ethanol and TBP. Sample results are flagged with a "<" when the absolute number of nanograms calculated in the sample is less than the lowest concentration standard used in the initial calibration. Results below the LLS were not reported. Methanol and ethanol results falling within their calibration range are quantitative results as evidenced by a valid calibration for these compounds. Tributyl phosphate results falling within the calibration range are considered estimated, as a CCV was not performed. It should be noted that these compounds are not a part of the current operating method.

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

Batch 3/5/96 (file identifier 46030501.b) - S6009-A61.828, S6007-A10.811, S6009-A63.831, S6008-A39.817, S6008-A39.817 REP;

Batch 3/6/96 (file identifier 46030601.b) - S6007-A09.810, S6009-A62.830, S6009-A62.830 REP, S6008-A48.821, S6007-A05.806;

Batch 3/7/96 (file identifier 46030701.b) - S6007-A11.813, S6007-A12.814, S6008-A77.824, S6008-A81.826;

Batch 3/8/96 (file identifier 46030801.b) - S6009-A83.834, S6009-A84.835, S6007-A13.815, S6007-A14.816;

Batch 3/11/96 (file identifier 46031101.b) - S6008-A41.819, S6007-A07.808, S6009-A65.833, S6008-A50.823, S6007-A08.809, S6009-A61.828 REANALYSIS;

Batch 3/12/96 (file identifier 46031101.b) - S6009-A64.832, S6008-A40.818, S6008-A49.822, S6007-A06.807, S6007-A06.807 REP.

The following discussion provides details regarding QC criterion failures for each batch.

Batch 3/5/96:
Three target compounds (cyclohexanone, tridecane and tetradecane) were outside the ± 25% D acceptance criteria for the CCV; however, the CCV passed the procedural criterion requiring ± 25% D passage for 85% of all target compounds. Cyclohexanone was not found in the tank samples. Tridecane and tetradecane were found in the samples at concentrations between the EQL and IDL. Tetradecane and tridecane concentration values may be over or under estimated because of the failed CCV. Sample S6009-A61.828 showed unacceptably high internal standard response and lower than typical surrogate recoveries. This sample was reanalyzed at a later date.

Batch 3/6/96:
Two target compounds (cyclohexanone and tetradecane) were outside the ± 25% D acceptance criteria for the CCV; however, the CCV passed the procedural criterion requiring ± 25% D passage for 85% of all target compounds. Cyclohexanone was not found in the tank samples.

Tetradecane was found in the samples at a concentration between the EQL and IDL. Tetradecane concentration values may be over or under estimated because of the failed CCV.

Batch 3/7/96:
Three target compounds (ethanol, cyclohexanone, and 1,1,2,2-tetrachloroethane) were outside the ± 25% D acceptance criteria for the CCV; however, the CCV passed the procedural criterion requiring ± 25% D passage for 85% of all target compounds. Ethanol, cyclohexanone, and 1,1,2,2-tetrachloroethane were not found in the samples.

Batch 3/8/96:
Five target compounds (butanenitrile, trans-1,3-dichloropropene, cyclohexanone, 1,1,2,2-tetrachloroethane, and decane) were outside the ± 25% D acceptance criteria for the CCV; however, the CCV passed the procedural criterion requiring ± 25% D passage for 85% of all target compounds. Butanenitrile, trans-1,3-dichloropropene, cyclohexanone, and 1,1,2,2-tetrachloroethane were not found in the samples; however, decane was found in the samples at a concentration between the EQL and IDL. Tetradecane concentration values may be over or under estimated because of the failed CCV.

Batch 3/11/96:
One target compound (1,1,2,2-tetrachloroethane) was outside the ± 25% D acceptance criteria for the CCV; however, the CCV passed the procedural criterion requiring ± 25% D passage for 85% of all target compounds. This compound was not found in the samples.

Sample S6009-A61.828 was reanalyzed from a split tube due to unacceptable QC during the initial run. This reanalysis was satisfactory and the data from this run was reported.

Batch 3/12/96:
Two target compounds (1,1,2,2-tetrachloroethane and decane) were outside the ± 25% D acceptance criteria for the CCV; however, the CCV passed the procedural criterion requiring ± 25% D passage for 85% of all target compounds. The compound 1,1,2,2-tetrachloroethane was not found in the samples, however decane was found in the samples at a concentration between the EQL and IDL. Tetradecane concentration values may be over or under estimated because of the failed CCV.

Samples were analyzed under the protocols of TVP-10, Rev. 2 with initial calibration performed on March 1, 1996 and quantitated against CCVs run at beginning of each batch for the target compounds listed in the method. Minor QC problems encountered during the course of the work are noted in daily case narratives. In general, the analysis proceeded very smoothly. Several specific issues are worth noting:

1. Passive Sampling:
Since the ISVS samples are physically exposed to the tank vapor for some time prior to actual sample collection and removal, some amount of passive sampling by diffusion is to be expected. The problem becomes progressively worse with decreasing sample size. In order to preserve dynamic range, a relatively small sample volume (200 ml nominal) was requested for all three sampling jobs. Because of the high levels of some compounds in Tank S-102 combined with the blank problems from the tape, the passive sampling effect is difficult to accurately assess and is likely to vary with compound properties and operational parameters. Several compounds which were relatively high in the tank and not present at significant levels in emissions from the tape

can be used to provide an estimate of passive sampling. Compounds examined included ethanol at 1.4%, acetonitrile at 13%, and tetrahydrofuran at 7.7%. Passive sampling under the conditions used for Tank S-102 is thus estimated to contribute on the order of 10% for the ISVS method as implemented at that time. This is a larger effect than was estimated from the Tank C-107 sampling on January 16, 1996, but somewhat lower and far more variable than was seen for the Tank BY-108 sampling on January 13, 1996. The differences may simply reflect operational differences associated with, among other things, the length of time used for the in-tank portion of the sampling event as well a variations in compound properties. Sampling should be performed as promptly as possible following insertion of the sampling bundle into the tank to minimize passive sampling associated errors.

2. Data Qualification:

The TST analyses are performed under very prescriptive QC protocols as documented in method TVP-10, Rev. 2. Failure to achieve certain QC goals may require reanalysis of individual samples or the entire batch. In that event, the 90% fraction of the sample collected on the spit tube and temporarily archived after each injection is used as a backup for reanalysis. For Tank S-102, only ISVS sample A61.828 required reanalysis. An unacceptably large variation in the internal standard responses for its initial analysis on March 5, 1996 was the cause. The sample was successfully reanalyzed during analysis of a later batch on March 11, 1996 and data are reported from the second analysis only.

The total organic loading of Tank S-102, particularly for the light alcohols, is somewhat high, resulting in some compounds amenable to TST analysis falling outside the calibrated range. Target compounds consistently flagged as above the UQL (E flag) were limited to methanol and ethanol. In the case of ethanol, the measured concentrations are more than an order of magnitude above the method UQL and should be considered suspect. Measured concentrations for methanol were about three times the UQL. This problem represents a limitation on method applicability since it is not practical with currently available sampling equipment to take a smaller sample. Dilution may not be done with TSTs since the procedure does not address the issue of potential trap saturation.

The compound sublist used for batch processing included TBP processed as a target using calibrated retention time and response factors determined on October 5, 1995. Since TBP was not included in any of the CCVs, calibrated results could not be obtained for TBP; however, since TBP was not detected, exact quantitation information was not needed. Full performance data has not been determined for TBP. Based on the relatively high response factor obtained, instrument response and thus detectability for TBP is excellent. An IDL of 2 ng (0.8 ppbv in 200 ml) was assumed based on comparison with other species with similar response factors. This value should be conservative. Tributyl phosphate was not observed in any sample using any of the three sampling methods.

3. Volumetric Flow Correction:

Since sample volumes reported from the field are based on two different calibration temperatures (21°C for ISVS and 0°C for VSS), a correction has been included in the reported data to provide a common volumetric basis. All ISVS volumes were corrected to 0°C for inclusion in the final data calculations reported for the TST samples.

Table E.2 Triple Sorbent Trap Sample Analysis Results for Samples Collected from the Headspace of Tank S-102 on 1/26/96

VSS Truck Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)		TRIDECANE (ppbv)		TETRADECANE (ppbv)		TBP (ppbv)	
A05.806	3986	E Y	12428	E Y	51	243	60	53	9.5	626	1.6	J	2.2	J	1.6	J	< 0.8	Y
A06.807	5961	E Y	17192	E Y	85	276	57	53	9.0	621	1.4	J	1.8	J	1.0	J	< 0.8	Y
A07.808	4310	E Y	14204	E Y	66	266	54	54	10	574	1.7	J	2.1	J	1.6	J	< 0.8	Y
A08.809	5071	E Y	14308	E Y	69	265	43	54	9.1	647	1.5	J	1.9	J	1.2	J	< 0.8	Y
A09.810	5193	E Y	15010	E Y	63	265	61	55	9.3	653	1.6	J	2.1	J	1.6	J	< 0.8	Y
A10.811	5401	E Y	15116	E Y	62	241	59	54	8.6	629	1.9	J	3.4	J	3.2	J	< 0.8	Y
Average	**4987**	**Y**	**14710**	**E Y**	**66**	**259**	**56**	**54**	**9.3**	**625**	**1.6**	**J**	**2.3**	**J**	**1.7**	**J**	**< 0.8**	**Y**
ST DEV	**725**		**1552**		**11**	**14**	**6.7**	**0.8**	**0.5**	**28**	**0.2**		**0.6**		**0.8**			
% RSD	**14**		**10**		**15**	**5**	**11**	**1**	**6**	**5**	**10**		**24**		**42**			
ISVS with HEPA																		
A39.817	4323	E Y	13006	E Y	56	241	54	49	126	605	2.1	J	5.9	J	3.3	J	< 0.8	Y
A40.818	4247	E Y	10454	E Y	68	236	34	41	123	456	1.6	J	2.3	J	1.0	J	< 0.8	Y
A41.819	3127	E Y	12244	E Y	65	261	50	52	110	545	1.5	J	2.4	J	1.1	J	< 0.8	Y
A48.821	2900	E Y	8494	E Y	62	215	58	42	73	477	1.6	J	2.9	J	1.4	J	< 0.8	Y
A49.822	4442	E Y	12533	E Y	62	248	48	45	74	513	1.5	J	2.7	J	0.9	J	< 0.8	Y
A50.823	2573	E Y	8602	E Y	48	219	46	40	61	462	1.3	J	2.4	J	1.0	J	< 0.8	Y
Average	**3602**	**Y**	**10889**	**E Y**	**60**	**237**	**48**	**45**	**95**	**510**	**1.6**	**J**	**3.1**	**J**	**1.5**	**J**	**< 0.8**	**Y**
ST DEV	**827**		**2009**		**7.2**	**17**	**8.2**	**4.8**	**28**	**58**	**0.3**		**1.4**		**0.9**			
% RSD	**22**		**17**		**11**	**8**	**16**	**11**	**28**	**11**	**15**		**42**		**59**			
ISVS without HEPA																		
A61.828	3767	E Y	14271	E Y	104	356	70	56	170	941	3.4	J	2.9	J	1.6	J	< 0.8	Y
A62.830	5460	E Y	13597	E Y	93	310	63	51	191	981	3.7	J	4.5	J	4.2	J	< 0.8	Y
A63.831	4882	E Y	12817	E Y	85	286	60	49	185	919	3.6	J	3.0	J	2.4	J	< 0.8	Y
A64.832	4352	E Y	15631	E Y	80	320	48	52	154	828	3.2	J	2.5	J	1.4	J	< 0.8	Y
A65.833	4299	E Y	13156	E Y	85	303	51	54	162	867	3.5	J	2.7	J	1.7	J	< 0.8	Y
Average	**4552**	**Y**	**13894**	**E Y**	**89**	**315**	**58**	**52**	**172**	**907**	**3.5**	**J**	**3.1**	**J**	**2.3**	**J**	**< 0.8**	**Y**
ST DEV	**643**		**1113**		**9**	**26**	**9.0**	**2.7**	**15**	**60**	**0.2**		**0.8**		**1.1**			
% RSD	**13**		**8**		**10**	**8**	**14**	**5**	**9**	**6**	**5**		**23**		**46**			

Data Qualifier Flag

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

Table E.3 Replicate Analysis of Triple Sorbent Trap Samples Collected from the Headspace of Tank S-102 on 1/26/96

VSS Truck Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)		TRIDECANE (ppbv)		TETRADECANE (ppbv)		TBP (ppbv)	
A06.807	5961	Y	17192	E Y	85	276	57	53	9.0	621	1.4	J	1.8	J	1.0	J	< 0.8	Y
A06.807 Rep	5501	Y	17094	E Y	92	262	56	52	8.8	593	1.4	J	1.8	J	0.9	J	< 0.8	Y
Relative Percent Difference	8		1		8	5	2	2	2	5	0		0		11			
ISVS with HEPA																		
A39.817	4323	E Y	13006	E Y	56	241	54	49	126	605	2.1	J	5.9	J	3.3	J	< 0.8	Y
A39.817 Rep	3138	E Y	10552	E Y	61	231	52	49	125	532	2.1	J	5.6	J	3.5	J	< 0.8	Y
Relative Percent Difference	32		21		9	4	4	0	1	13	0		5		6			
ISVS without HEPA																		
A62.830	5460	E Y	13597	E Y	93	310	63	51	191	981	3.7	J	4.5	J	4.2	J	< 0.8	Y
A62.830 Rep	5372	E Y	13409	E Y	104	290	64	51	191	915	4.6	J	4.0	J	2.9	J	< 0.8	Y
Relative Percent Difference	2		1		11	7	2	0	0	7	22		12		37			

Data Qualifier Flag

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

Table E.4 Triple Sorbent Trap Blank Sample Analysis Results for Samples Collected from the Headspace of Tank S-102 on 1/26/96

Blank Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)		ACETONE (ppbv)		PROPANOL (ppbv)		TETRAHYDROFURAN (ppbv)		HEXANE (ppbv)		1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)	
Field Blank # 1 VSS	< 192	Y	< 133	Y	U		10	J	U		U		U		U	U	U	U	< 0.8	Y
Field Blank # 2 VSS	< 192	Y	< 133	Y	U		7.8	J	U		U		U		U	U	U	U	< 0.8	Y
Field Blank #3 W/HEPA	239	Y	228	Y	8.2	J	58		U		1.9	J	113		60	U	U	U	< 0.8	Y
Field Blank #4 W/HEPA	< 192	Y	131	Y	5.8	J	46		U		1.7	J	63		49	U	U	U	< 0.8	Y
Field Blank #5 WO/HEPA	159	Y	229	Y	9.2	J	70		U		3.6	J	150		316	U	U	U	< 0.8	Y
Field Blank #6 WO/HEPA	221	Y	221	Y	14	J	74		U		3.3	J	128		266	U	U	U	< 0.8	Y
Trip Blank #1	< 192	Y	< 133	Y	U		9.8	J	U		U		U		U	U	U	U	< 0.8	Y
Trip Blank #2	< 192	Y	< 133	Y	U		9.2	J	U		U		U		U	U	U	U	< 0.8	Y

Data Qualifier Flag

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

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 S-102 on 1/26/96

Target Analytes	CAS No.	S6007-A03.082 (ppbv)	Flag	S6007-A04.083 (ppbv)	Flag	S6007-A15.084 (ppbv)	Flag	S6007-A16.097 (ppbv)	Flag	S6007-A27.108 (ppbv)	Flag	S6007-A28.120 (ppbv)	Flag	S6007-A87.086 (ppbv)	Flag	S6007-A88.089 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	1.1	J	1.3	J	1.0	J	0.9	J	1.2	J	1.1	J	1.0	J	1.0	J	1.1	J	0.1
CHLOROMETHANE	74-87-3	3.2	J	2.8	J	3.5	J	2.9	J	3.6	J	2.9	J	3.0	J	3.1	J	3.1	J	0.3
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U		U		U				
METHANOL	67-56-1	5242	E,Y	6181	E,Y	4772	E,Y	4577	E,Y	5629	E,Y	5481	E,Y	5790	E,Y	5465	E,Y	5392	E,Y	524
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U		U		U				
BUTANE	106-97-8	99		122		92		90		112		97		104		100		102		11
BROMOMETHANE	74-83-9	U		U		U		U		U		U		U		U				
CHLOROETHANE	75-00-3	U		U		U		U		U		U		U		U				
ETHANOL	64-17-5	4760	E,Y	6154	E,Y	5180	E,Y	4146	E,Y	5970	E,Y	6457	E,Y	5753	E,Y	4958	E,Y	5422	E,Y	789
ACETONITRILE	75-05-8	32		40		38		56		50		40		41		49		43		7.8
ACETONE	67-64-1	451		252		413		595		556		276		471		452		433		120
TRICHLOROFLUOROMETHANE	75-69-4	46		35		44		44		56		33		51		39		44		7.8
PENTANE	109-66-0	10		10		12		10		10		10		8.5		8.7		10		1.1
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	0.8	J	0.9	J	0.9	J	0.9	J	0.8	J	0.8	J	0.9	J	1.0	J	0.9	J	0.1
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U		U		U				
PROPANOL	71-23-8	34		33		41		33		41		41		37		37		37		3.6
PROPANENITRILE	107-12-0	3.9	J	3.5	J	4.4	J	3.7	J	4.4	J	4.2	J	4.0	J	3.8	J	4.0	J	0.3
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U		U		U				
2-BUTANONE	78-93-3	48		46		56		49		56		53		47		50		51		3.9
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U		U		U				
HEXANE	110-54-3	7.4		7.9		8.6		7.3		7.8		8.0		7.2		7.2		7.7		0.5
CHLOROFORM	67-66-3	U		U		U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	50		43		50		48		46		48		43		48		47		2.8
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U		U		U				
BUTANENITRILE	109-74-0	U		U		U		U		U		U		U		U				
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U		0.8	J	U		0.8	J	
1-BUTANOL	71-36-3	464		394		431		425		418		434		384		455		426		27
BENZENE	71-43-2	15		14		16		15		14		15		13		15		15		0.9
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	U		6.0		U		U		U		5.4		U		U		5.7		0.5
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U		U		U				
TRICHLOROETHENE	79-01-6	U		U		U		U		U		U		U		U				
HEPTANE	142-82-5	7.8		7.6		9.1		8.0		6.2		8.3		7.2		7.8		7.8		0.8
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U		U		U				
4-METHYL-2-PENTANONE	108-10-1	U		U		U		U		U		U		U		U				
PYRIDINE	110-86-1	5.7	J	5.1	J	10	J	5.8	J	5.8	J	7.5	J	6.0	J	5.7	J	6.4	J	1.6
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U		U		U				
PENTANENITRILE	110-59-8	U		U		U		U		U		U		U		U				
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		U		U		U		U				
TOLUENE	108-88-3	115		110		132		117		113		113		105		114		115		7.8
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U		U		U				
OCTANE	111-65-9	3.8		3.5		4.5		3.9		3.5		3.9		3.4		3.7		3.8		0.4
TETRACHLOROETHYLENE	127-18-4	8.4		8.0		10		8.6		8.5		8.4		7.8		8.5		8.5		0.7
CHLOROBENZENE	108-90-7	U		U		U		U		U		U		U		U				
HEXANENITRILE	628-73-9	U		U		U		U		U		U		U		U				

Table F.1 SUMMA™ Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6007-A03.082 (ppbv)	Flag	S6007-A04.083 (ppbv)	Flag	S6007-A15.084 (ppbv)	Flag	S6007-A16.097 (ppbv)	Flag	S6007-A27.108 (ppbv)	Flag	S6007-A28.120 (ppbv)	Flag	S6007-A87.086 (ppbv)	Flag	S6007-A88.089 (ppbv)	Flag	Mean	Flag	Std. Dev.
ETHYLBENZENE	100-41-4	1.0	J	0.9	J	1.2	J	1.0	J	0.9	J	1.0	J	0.9	J	1.0	J	1.0	J	0.1
P/M-XYLENE	106-42-3	3.8	J	3.2	J	4.1	J	3.7	J	3.6	J	3.6	J	3.2	J	3.7	J	3.6	J	0.3
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U		U		U				
STYRENE	100-42-5	U		U		U		U		U		U		U		U				
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U		U		U				
O-XYLENE	95-47-6	1.6	J	1.5	J	1.8	J	1.5	J	1.5	J	1.6	J	1.4	J	1.5	J	1.5	J	0.1
NONANE	111-84-2	2.0		1.8		2.4		2.0		1.8		2.0		1.7		1.9		1.9		0.2
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		U		U		U		U		U		U				
1,3,5-TRIMETHYLBENZENE	108-67-8	U		U		U		U		U		U		U		U				
1,2,4-TRIMETHYLBENZENE	95-63-6	0.3	J	U		0.4	J	0.3	J	0.3	J	0.3	J	U		0.3	J	0.3	J	0.0
DECANE	124-18-5	1.3	J	1.2	J	1.8	J	1.3	J	1.4	J	1.5	J	1.2	J	1.3	J	1.3	J	0.2
CHLOROMETHYLBENZENE, ALPHA	100-44-7	U		U		U		U		U		U		U		U				
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U		U		U		U				
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U		U		U		U				
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U		U		U				
UNDECANE	1120-21-4	1.0	J	0.8	J	1.2	J	1.0	J	U		1.0	J	0.9	J	0.8	J	0.9	J	0.1
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U		U		U				
DODECANE	112-40-3	U		U		U		U		U		U		U		U				
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U		U		U				
TRIDECANE	629-50-5	0.8	J	0.7	J	0.7	J	0.9	J	0.9	J	1.1	J	0.5	J	0.9	J	0.8	J	0.2
TETRADECANE	629-59-4	U		U		U		U		U		U		U		U				

Data Quality Flags

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

U Target compound not detected at or above the IDL.

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

E Target compound exceeds upper quantification limit (UQL).

Table F.2 Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6007 A05.806 (ppbv)	Flag	S6007 A06.807 (ppbv)	Flag	S6007 A07.808 (ppbv)	Flag	S6007 A08.809 (ppbv)	Flag	S6007 A09.810 (ppbv)	Flag	S6007 A10.811 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U		U				
CHLOROMETHANE	74-87-3	U		U		U		U		U		U				
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U				
METHANOL	67-56-1	3986	E,Y	5961	E,Y	4310	E,Y	5071	E,Y	5193	E,Y	5401	E,Y	4987	E,Y	725
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U				
BUTANE	106-97-8	63		57		63		63		59		56		60		3.3
CHLOROETHANE	75-00-3	U		U		U		U		U		U				
ETHANOL	64-17-5	12428	E,Y	17192	E,Y	14204	E,Y	14308	E,Y	15010	E,Y	15116	E,Y	14710	E,Y	1552
ACETONITRILE	75-05-8	51		85		66		69		63		62		66		11
ACETONE	67-64-1	243		276		266		265		265		241		259		14
TRICHLOROFLUOROMETHANE	75-69-4	28		30		31		29		32		27		30		1.9
PENTANE	109-66-0	15		14		15		15		15		14		15		0.5
1,1-DICHLOROETHENE	75-35-4	U		U		3.2	J	U		U		U		3.2		
METHYLENE CHLORIDE	75-09-2	11	J	9.2	J	14	J	12	J	7.3	J	8.7	J	10	J	2.4
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U				
PROPANENITRILE	107-12-0	5.1	J	5.1	J	5.1	J	5.4	J	5.5	J	5.3	J	5.2	J	0.2
PROPANOL	71-23-8	60		57		54		43		61		59		56		6.7
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U				
2-BUTANONE	78-93-3	59		59		65		65		58		56		60		3.8
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U				
HEXANE	110-54-3	9.5		9.0		10		9.1		9.3		8.6		9.3		0.5
CHLOROFORM	67-66-3	U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	53		53		54		54		55		54		54		0.8
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U				
BUTANENITRILE	109-74-0	U		U		U		U		U		U				
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U				
1-BUTANOL	71-36-3	626		621		574		647		653		629		625		28
BENZENE	71-43-2	18		18		19		18		19		18		18		0.5
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	U		U		U		U		U		U				
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U				
TRICHLOROETHENE	79-01-6	U		U		U		U		U		U				
HEPTANE	142-82-5	11		11		11		11		11		11		11		0.1
4-METHYL-2-PENTANONE	108-10-1	8.7		11		8.5		8.8		8.1		9.1		9.0		1.0
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U				
PYRIDINE	110-86-1	13	J	13	J	12	J	14	J	15	J	21	J	15	J	3.3
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U				
PENTANENITRILE	110-59-8	U		U		U		U		U		U				
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		U		U				
TOLUENE	108-88-3	121		122		123		121		123		118		121		1.9
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U				
OCTANE	111-65-9	5.0	J	4.8	J	5.1	J	5.1	J	5.1	J	4.9	J	5.0	J	0.1
TETRACHLOROETHYLENE	127-18-4	10		9.8		10		10		10		9.9		10		0.1

Table F.2 Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6007 A05.806 (ppbv)	Flag	S6007 A06.807 (ppbv)	Flag	S6007 A07.808 (ppbv)	Flag	S6007 A08.809 (ppbv)	Flag	S6007 A09.810 (ppbv)	Flag	S6007 A10.811 (ppbv)	Flag	Mean	Flag	Std. Dev.
CHLOROBENZENE	108-90-7	U		U		U		U		U		U				
HEXANENITRILE	628-73-9	U		U		U		U		U		U				
ETHYLBENZENE	100-41-4	1.2	J	1.2	J	1.1	J	1.1	J	1.2	J	1.2	J	1.2	J	0.04
P/M-XYLENE	106-42-3	3.9	J	4.2	J	3.9	J	4.0	J	3.9	J	4.8	J	4.1	J	0.4
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U				
STYRENE	100-42-5	U		1.1	J	U		U		U		0.8	J	0.9	J	0.2
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U				
O-XYLENE	95-47-6	2.0	J	1.8	J	1.8	J	1.8	J	1.9	J	1.8	J	1.9	J	0.1
NONANE	111-84-2	3.2		3.1		3.4		3.4		3.2		3.1		3.2		0.1
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		U		U		U		0.9	J	0.9	J	
1,3,5-TRIMETHYLBENZENE	108-67-8	U		U		U		U		U		0.6	J	0.6	J	
1,2,4-TRIMETHYLBENZENE	95-63-6	0.5	J	U		U		U		U		0.5	J	0.5	J	0.01
DECANE	124-18-5	2.5	J	2.4	J	2.6	J	2.4	J	2.7	J	2.5	J	2.5	J	0.1
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U		U				
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U		U				
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U				
UNDECANE	1120-21-4	1.7	J	1.4	J	1.5	J	1.4	J	1.5	J	1.6	J	1.5	J	0.1
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U				
DODECANE	112-40-3	1.6	J	1.4	J	1.7	J	1.5	J	1.6	J	1.9	J	1.6	J	0.2
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U				
TRIDECANE	629-50-5	2.2	J	1.8	J	2.1	J	1.9	J	2.1	J	3.4	J	2.3	J	0.6
TETRADECANE	629-59-4	1.6	J	1.0	J	1.6	J	1.2	J	1.6	J	3.2	J	1.7	J	0.8
TBP	126-73-8	< 0.8		< 0.8		< 0.8		< 0.8		< 0.8		< 0.8				

Data Quality Flags

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

Table F.3 SUMMA™ Analysis Results for All Target Analytes for ISVS Samples With HEPA Filtration Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6008-A36.128 (ppbv)	Flag	S6008-A37.129 (ppbv)	Flag	S6008-A38.134 (ppbv)	Flag	S6008-A45.136 (ppbv)	Flag	S6008-A46.137 (ppbv)	Flag	S6008-A47.153 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	1.1	J	1.2	J	1.2	J	1.1	J	0.8	J	0.9	J	1.1	J	0.2
CHLOROMETHANE	74-87-3	3.2	J	3.0	J	3.2	J	3.1	J	3.1	J	2.6	J	3.0	J	0.2
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U				
METHANOL	67-56-1	6171	E,Y	6138	E,Y	6623	E,Y	5381	E,Y	4547	E,Y	5012	E,Y	5645	E,Y	794
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U				
BUTANE	106-97-8	113		136		113		111		73		84		105		23
BROMOMETHANE	74-83-9	U		U		U		U		U		U				
CHLOROETHANE	75-00-3	U		U		U		U		U		U				
ETHANOL	64-17-5	4403	E,Y	5987	E,Y	4892	E,Y	6429	E,Y	4203	E,Y	4326	E,Y	5040	E,Y	945
ACETONITRILE	75-05-8	47		25		32		46		36		20		34		11
ACETONE	67-64-1	534		213		607		280		563		615		469		176
TRICHLOROFLUOROMETHANE	75-69-4	56		32		57		33		40		42		43		11
PENTANE	109-66-0	8.6		20		12		9.8		9.3		10		12		4.3
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	1.0	J	0.9	J	0.9	J	0.8	J	0.8	J	0.8	J	0.9	J	0.1
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U				
PROPANOL	71-23-8	42		34		46		36		38		40		39		4.3
PROPANENITRILE	107-12-0	4.4	J	3.3	J	4.2	J	3.8	J	3.3	J	3.5	J	3.8	J	0.5
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U				
2-BUTANONE	78-93-3	45		44		47		46		46		50		46		2.1
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U				
HEXANE	110-54-3	7.2	S	10	S	7.6	S	14	S	7.6	S	7.9	S	9.1		2.6
CHLOROFORM	67-66-3	U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	42		39		42		45		42		45		43		2.3
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U				
BUTANENITRILE	109-74-0	U		U		U		U		U		U				
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U				
1-BUTANOL	71-36-3	386	S	367	S	478	S	379	S	450	S	487	S	425		53
BENZENE	71-43-2	13		13		14		14		13		14		14		0.5
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	U		6.4	S	U		20	S	U		U		13		9.6
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U				
TRICHLOROETHENE	79-01-6	1.3	J	1.1	J	1.0	J	1.4	J	1.1	J	1.1	J	1.1	J	0.1
HEPTANE	142-82-5	8.2	S	7.8	S	7.9	S	45	S	12	S	11	S	15		15
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U				
4-METHYL-2-PENTANONE	108-10-1	U		U		U		U		U		U				
PYRIDINE	110-86-1	7.4	J	5.7	J	8.1	J	7.9	J	7.3	J	6.9	J	7.2	J	0.9
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U				
PENTANENITRILE	110-59-8	U		U		U		7.8		U		U		7.8		
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		U		U				
TOLUENE	108-88-3	112	S	103	S	122	S	124	S	108	S	114	S	114		8.1
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U				

Table F.3 SUMMA™ Analysis Results for All Target Analytes for ISVS Samples With HEPA Filtration Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6008-A36.128 (ppbv)	Flag	S6008-A37.129 (ppbv)	Flag	S6008-A38.134 (ppbv)	Flag	S6008-A45.136 (ppbv)	Flag	S6008-A46.137 (ppbv)	Flag	S6008-A47.153 (ppbv)	Flag	Mean	Flag	Std. Dev.
OCTANE	111-65-9	3.4		3.0		3.7		3.7		3.4		3.5		3.5		0.2
TETRACHLOROETHYLENE	127-18-4	7.7		6.9		8.7		7.8		7.4		7.9		7.7		0.6
CHLOROBENZENE	108-90-7	U		U		U		0.3	J	U		U		0.3	J	
HEXANENITRILE	628-73-9	U		U		U		U		U		U				
ETHYLBENZENE	100-41-4	0.9	J	0.8	J	1.0	J	1.1	J	1.0	J	1.0	J	1.0	J	0.1
P/M-XYLENE	106-42-3	3.4	J	3.0	J	3.7	J	3.7	J	3.6	J	3.7	J	3.5	J	0.3
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U				
STYRENE	100-42-5	U		U		U		U		U		U				
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U				
O-XYLENE	95-47-6	1.4	J	1.3	J	1.5	J	1.6	J	1.4	J	1.5	J	1.5	J	0.1
NONANE	111-84-2	1.5	J	1.4	J	1.8		1.9		1.6		1.7		1.7		0.2
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		U		U		U		U				
1,3,5-TRIMETHYLBENZENE	108-67-8	U		U		U		U		U		U				
1,2,4-TRIMETHYLBENZENE	95-63-6	0.3	J	U		0.4	J	0.4	J	0.4	J	0.4	J	0.4	J	0.0
DECANE	124-18-5	1.0	J	0.9	J	1.2	J	1.4	J	1.1	J	1.2	J	1.1	J	0.2
CHLOROMETHYLBENZENE, ALPHA	100-44-7	U		U		U		U		U		U				
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U		U				
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U		U				
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U				
UNDECANE	1120-21-4	0.7	J	0.7	J	0.8	J	0.9	J	0.8	J	0.8	J	0.8	J	0.1
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U				
DODECANE	112-40-3	U		U		U		U		U		U				
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U				
TRIDECANE	629-50-5	1.1	J	0.7	J	1.0	J	0.9	J	0.7	J	0.8	J	0.9	J	0.2
TETRADECANE	629-59-4	U		U		U		U		U		U				

Data Quality Flags

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

U Target compound not detected at or above the IDL.

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

E Target compound exceeds upper quantification limit (UQL).

S Result affected by adhesive tape contamination.

Table F.4 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples With HEPA Filtration Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6008 A39.817 (ppbv)	Flag	S6008 A40.818 (ppbv)	Flag	S6008 A41.819 (ppbv)	Flag	S6008 A48.821 (ppbv)	Flag	S6008 A49.822 (ppbv)	Flag	S6008 A50.823 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U		U				
CHLOROMETHANE	74-87-3	U		U		U		U		U		U				
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U				
METHANOL	67-56-1	4323	E,Y	4247	E,Y	3127	E,Y	2900	E,Y	4442	E,Y	2573	E,Y	3602	E,Y	827
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U				
BUTANE	106-97-8	68		63		67		56		60		56		62		5.2
CHLOROETHANE	75-00-3	U		U		U		U		U		U				
ETHANOL	64-17-5	13006	E,Y	10454	E,Y	12244	E,Y	8494	E,Y	12533	E,Y	8602	E,Y	10889	E,Y	2009
ACETONITRILE	75-05-8	56		68		65		62		62		48		60		7.2
ACETONE	67-64-1	241		236		261		215		248		219		237		17
TRICHLOROFLUOROMETHANE	75-69-4	46		44		40		41		42		39		42		2.6
PENTANE	109-66-0	18		16		17		14		15		13		16		1.9
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	18	J	6.2	J	5.9	J	4.3	J	6.1	J	4.6	J	7.5	J	5.2
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U				
PROPANENITRILE	107-12-0	5.0	J	4.3	J	5.2	J	4.4	J	5.0	J	3.8	J	4.6	J	0.5
PROPANOL	71-23-8	54		34		50		58		48		46		48		8.2
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U				
2-BUTANONE	78-93-3	53		43		62		45		51		47		50		6.9
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U				
HEXANE	110-54-3	126	S	123	S	110	S	73	S	74	S	61	S	95		28
CHLOROFORM	67-66-3	U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	49		41		52		42		45		40		45		4.8
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U				
BUTANENITRILE	109-74-0	U		U		U		U		U		U				
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U				
1-BUTANOL	71-36-3	605	S	456	S	545	S	477	S	513	S	462	S	510		58
BENZENE	71-43-2	19		17		19		16		17		15		17		1.6
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	U		25	S	U		U		42	S	27	S	31		9.3
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U				
TRICHLOROETHENE	79-01-6	2.8	J	4.5	J	2.6	J	1.6	J	2.0	J	0.9	J	2.4	J	1.2
HEPTANE	142-82-5	218	S	177	S	168	S	212	S	214	S	187	S	196		21
4-METHYL-2-PENTANONE	108-10-1	13	S	12	S	13	S	7.7	S	10	S	7.3	S	11		2.6
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U				
PYRIDINE	110-86-1	12	J	8.7	J	13	J	11	J	14	J	9.1	J	11	J	2.1
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U				
PENTANENITRILE	110-59-8	U		U		U		U		U		U				
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		U		U				
TOLUENE	108-88-3	276	S	242	S	229	S	247	S	254	S	210	S	243		22
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U				
OCTANE	111-65-9	5.4	J	5.3	J	5.2	J	5.6		5.4	J	4.9	J	5.3	J	0.2

Table F.4 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples With HEPA Filtration Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6008 A39.817 (ppbv)	Flag	S6008 A40.818 (ppbv)	Flag	S6008 A41.819 (ppbv)	Flag	S6008 A48.821 (ppbv)	Flag	S6008 A49.822 (ppbv)	Flag	S6008 A50.823 (ppbv)	Flag	Mean	Flag	Std. Dev.
TETRACHLOROETHYLENE	127-18-4	8.8		8.6		9.5		8.1		8.3		8.0		8.5		0.6
CHLOROBENZENE	108-90-7	U		U		U		U		U		U				
HEXANENITRILE	628-73-9	U		U		U		U		U		U				
ETHYLBENZENE	100-41-4	3.7		3.1		2.8	J	3.3		3.4		2.8	J	3.2	J	0.4
P/M-XYLENE	106-42-3	14		12		10		12		13		10		12		1.5
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U				
STYRENE	100-42-5	8.3		3.0	J	2.5	J	2.9	J	3.4	J	2.5	J	3.8	J	2.3
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U				
O-XYLENE	95-47-6	5.2		4.5		4.0	J	4.6		4.8		4.0		4.5	J	0.5
NONANE	111-84-2	2.9		3.1		3.0		3.0		3.0		2.7		3.0		0.1
1-ETHYL-2-METHYL BENZENE	611-14-3	2.2	J	1.7	J	1.4	J	1.9	J	2.0	J	1.5	J	1.8	J	0.3
1,3,5-TRIMETHYLBENZENE	108-67-8	1.1	J	0.7	J	0.8	J	1.1	J	0.9	J	0.7	J	0.9	J	0.2
1,2,4-TRIMETHYLBENZENE	95-63-6	3.1	J	2.6	J	2.2	J	3.1	J	3.1	J	2.4	J	2.7	J	0.4
DECANE	124-18-5	2.7	J	2.4	J	2.5	J	2.7	J	2.4	J	2.2	J	2.5	J	0.2
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U		U				
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U		U				
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U				
UNDECANE	1120-21-4	1.6	J	1.7	J	1.8	J	1.9	J	1.8	J	1.6	J	1.7	J	0.1
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U				
DODECANE	112-40-3	2.1	J	1.6	J	1.5	J	1.6	J	1.5	J	1.3	J	1.6	J	0.3
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U				
TRIDECANE	629-50-5	5.9	J	2.3	J	2.4	J	2.9	J	2.7	J	2.4	J	3.1	J	1.4
TETRADECANE	629-59-4	3.3	J	1.0	J	1.1	J	1.4	J	0.9	J	1.0	J	1.5	J	0.9
TBP	126-73-8	< 0.8		< 0.8		< 0.8		< 0.8		< 0.8		< 0.8				

Data Quality Flags

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

S Result affected by adhesive tape contamination.

Table F.5 SUMMA™ Analysis Results for All Target Analytes for ISVS Samples Without HEPA Filtration Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6009-A54.167 (ppbv)	Flag	S6009-A55.169 (ppbv)	Flag	S6009-A56.170 (ppbv)	Flag	S6009-A57.229 (ppbv)	Flag	S6009-A58.230 (ppbv)	Flag	S6009-A59.248 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	1.0	J	0.9	J	1.1	J	1.1	J	0.8	J	1.3	J	1.0	J	0.2
CHLOROMETHANE	74-87-3	2.9	J	2.8	J	4.2	J	3.7	J	3.4	J	3.1	J	3.4	J	0.5
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U				
METHANOL	67-56-1	5190	E,Y	4959	E,Y	5780	E,Y	6113	E,Y	3677	E,Y	6110	E,Y	5305	E,Y	929
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U				
BUTANE	106-97-8	102		80		105		112		65		119		97		21
BROMOMETHANE	74-83-9	U		U		U		U		U		U				
CHLOROETHANE	75-00-3	U		U		U		U		U		U				
ETHANOL	64-17-5	4783	E,Y	5984	E,Y	6010	E,Y	6117	E,Y	3844	E,Y	5830	E,Y	5428	E,Y	918
ACETONITRILE	75-05-8	23		33		25		34		26		23		27		5.0
ACETONE	67-64-1	451		310		489		540		597		293		447		123
TRICHLOROFLUOROMETHANE	75-69-4	39		32		64		56		39		33		44		13
PENTANE	109-66-0	11		10		11		9.6		9.9		9.9		10		0.7
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	0.9	J	0.9	J	0.9	J	0.9	J	0.8	J	0.9	J	0.9	J	0.01
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U				
PROPANOL	71-23-8	34		45		41		41		32		38		39		4.6
PROPANENITRILE	107-12-0	3.7	J	3.9	J	4.0	J	4.2	J	3.6	J	4.0	J	3.9	J	0.2
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U				
2-BUTANONE	78-93-3	50		51		54		84		59		50		58		13.0
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U				
HEXANE	110-54-3	13	S	13	S	13	S	11	S	9.8	S	10	S	11		1.3
CHLOROFORM	67-66-3	U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	47		47		47		46		46		46		46		0.2
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U				
BUTANENITRILE	109-74-0	U		U		U		U		U		U				
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U				
1-BUTANOL	71-36-3	448	S	511	S	445	S	420	S	392	S	411	S	438		42
BENZENE	71-43-2	15		14		14		15		13		14		14		0.5
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	20	S	18	S	17	S	13	S	12	S	13	S	15		3.4
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U				
TRICHLOROETHENE	79-01-6	0.7	J	0.7	J	0.5	J	0.5	J	0.4	J	0.4	J	0.5	J	0.1
HEPTANE	142-82-5	44	S	36	S	35	S	25	S	23	S	23	S	31		8.5
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U				
4-METHYL-2-PENTANONE	108-10-1	U		U		U		U		U		U				
PYRIDINE	110-86-1	11	J	15	J	12	J	9.2	J	9.0	J	8.3	J	11	J	2.7
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U				
PENTANENITRILE	110-59-8	6.8		6.2		5.8		U		3.5	J	3.4	J	5.1	J	1.6
1,1,2-TRICHLOROETHANE	79-00-5	U		U		1.3	J,S	U		U		U		1.3	J	
TOLUENE	108-88-3	124	S	120	S	126	S	124	S	115	S	114	S	121		5.3
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U				

Table F.5 SUMMA™ Analysis Results for All Target Analytes for ISVS Samples Without HEPA Filtration Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6009-A54.167 (ppbv)	Flag	S6009-A55.169 (ppbv)	Flag	S6009-A56.170 (ppbv)	Flag	S6009-A57.229 (ppbv)	Flag	S6009-A58.230 (ppbv)	Flag	S6009-A59.248 (ppbv)	Flag	Mean	Flag	Std. Dev.
OCTANE	111-65-9	3.7		4.1		3.9		3.9		3.9		3.6		3.8		0.2
TETRACHLOROETHYLENE	127-18-4	8.0		8.4		8.5		8.6		7.9		7.8		8.2		0.3
CHLOROBENZENE	108-90-7	U		0.3	J	0.3	J	U		0.3	J	U		0.3	J	0.02
HEXANENITRILE	628-73-9	U		U		U		U		U		U				
ETHYLBENZENE	100-41-4	1.1	J	1.1	J	1.1	J	1.0	J	1.0	J	1.0	J	1.0	J	0.05
P/M-XYLENE	106-42-3	3.9	J	3.8	J	4.1	J	3.9	J	3.7	J	3.7	J	3.9	J	0.2
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U				
STYRENE	100-42-5	U		U		U		U		U		U				
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U				
O-XYLENE	95-47-6	1.6	J	1.6	J	1.7	J	1.7	J	1.5	J	1.6	J	1.6	J	0.1
NONANE	111-84-2	1.8		2.0		2.0		1.9		1.8		1.8		1.9		0.1
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		U		U		U		U				
1,3,5-TRIMETHYLBENZENE	108-67-8	U		U		U		U		U		U				
1,2,4-TRIMETHYLBENZENE	95-63-6	0.5	J	0.5	J	0.5	J	0.4	J	0.4	J	0.4	J	0.5	J	0.05
DECANE	124-18-5	1.2	J	1.4	J	1.4	J	1.3	J	1.3	J	1.3	J	1.3	J	0.1
CHLOROMETHYLBENZENE, ALPHA	100-44-7	U		U		U		U		U		U				
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U		U				
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U		U				
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U				
UNDECANE	1120-21-4	0.8	J	1.0	J	1.0	J	0.9	J	0.7	J	0.8	J	0.8	J	0.1
1,2,4-TRICHLOROBENZENE	120-82-1	U		0.3	J	U		U		0.2	J	U		0.3	J	0.1
DODECANE	112-40-3	U		U		U		U		U		U				
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		0.2	J	U		U		U		U		0.2		
TRIDECANE	629-50-5	0.7	J	0.9	J	0.7	J	0.5	J	0.6	J	U		0.7	J	0.2
TETRADECANE	629-59-4	U		U		U		U		U		U				

Data Quality Flags

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

S Result affected by adhesive tape contamination.

Table F.6 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Without HEPA Filtration Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6009 A61.828 (ppbv)	Flag	S6009 A62.830 (ppbv)	Flag	S6009 A63.831 (ppbv)	Flag	S6009 A64.832 (ppbv)	Flag	S6009 A65.833 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U				
CHLOROMETHANE	74-87-3	U		U		U		U		U				
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHAN	76-14-2	U		U		U		U		U				
METHANOL	67-56-1	3767	E,Y	5460	E,Y	4882	E,Y	4352	E,Y	4299	E,Y	4552	E,Y	643
VINYL CHLORIDE	75-01-4	U		U		U		U		U				
BUTANE	106-97-8	82		66		66		68		69		70		6.8
CHLOROETHANE	75-00-3	U		U		U		U		U				
ETHANOL	64-17-5	14271	E,Y	13597	E,Y	12817	E,Y	15631	E,Y	13156	E,Y	13894	E,Y	1113
ACETONITRILE	75-05-8	104		93		85		80		85		89		9.4
ACETONE	67-64-1	356		310		286		320		303		315		26
TRICHLOROFLUOROMETHANE	75-69-4	45		41		36		40		39		40		3.2
PENTANE	109-66-0	23		23		24		22		23		23		0.7
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	7.3	J	6.2	J	8.4	J	7.1	J	9.1	J	7.6	J	1.1
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U				
PROPANENITRILE	107-12-0	6.0	J	5.6	J	5.3	J	5.6	J	5.6	J	5.6	J	0.2
PROPANOL	71-23-8	70		63		60		48		51		58		9.0
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U				
2-BUTANONE	78-93-3	63		60		54		62		66		61		4.4
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U				
HEXANE	110-54-3	170	S	191	S	185	S	154	S	162	S	172		15
CHLOROFORM	67-66-3	U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	56		51		49		52		54		52		2.7
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U				
BUTANENITRILE	109-74-0	U		U		U		U		U				
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U				
1-BUTANOL	71-36-3	941	S	981	S	919	S	828	S	867	S	907		60
BENZENE	71-43-2	29		26		25		25		25		26		2.0
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U				
CYCLOHEXANE	110-82-7	185	S	U		U		121	S	U		153		46
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U				
TRICHLOROETHENE	79-01-6	1.3	J	1.8	J	1.0	J	U		1.2	J	1.3	J	0.3
HEPTANE	142-82-5	574	E,S	569	S	573	S	422	S	429	S	513		80
4-METHYL-2-PENTANONE	108-10-1	48		U		U		U		U		48		
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U				
PYRIDINE	110-86-1	15	J	13	J	16	J	14	J	15	J	15	J	1.3
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U				
PENTANENITRILE	110-59-8	U		U		U		U		U				
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		U				
TOLUENE	108-88-3	455	S	504	S	476	S	423	S	435	S	459		32

Table F.6 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Without HEPA Filtration Collected from the Headspace of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6009 A61.828 (ppbv)	Flag	S6009 A62.830 (ppbv)	Flag	S6009 A63.831 (ppbv)	Flag	S6009 A64.832 (ppbv)	Flag	S6009 A65.833 (ppbv)	Flag	Mean	Flag	Std. Dev.
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U				
OCTANE	111-65-9	7.5		7.1		6.7		6.6		6.9		7.0		0.4
TETRACHLOROETHYLENE	127-18-4	10		10		9.3		10		11		10		0.6
CHLOROBENZENE	108-90-7	U		U		U		U		U				
HEXANENITRILE	628-73-9	U		U		U		U		U				
ETHYLBENZENE	100-41-4	4.4		4.6		4.5		3.6		3.8		4.2		0.4
P/M-XYLENE	106-42-3	16		17		16		13		13		15		1.6
CYCLOHEXANONE	108-94-1	U		U		U		U		U				
STYRENE	100-42-5	U		0.9	J	0.9	J	U		U		0.9	J	0.0
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U				
O-XYLENE	95-47-6	6.1		6.4		5.9		5.1		5.4		5.8		0.5
NONANE	111-84-2	4.0		3.8		3.4		3.7		3.8		3.7		0.2
1-ETHYL-2-METHYL BENZENE	611-14-3	2.0	J	2.1	J	1.0	J	1.6	J	1.7	J	1.7	J	0.4
1,3,5-TRIMETHYLBENZENE	108-67-8	1.1	J	1.2	J	1.2	J	0.8	J	1.0	J	1.0	J	0.2
1,2,4-TRIMETHYLBENZENE	95-63-6	3.5	J	3.5	J	3.4	J	2.6	J	2.8	J	3.1	J	0.4
DECANE	124-18-5	4.1		3.8	J	3.5	J	3.3	J	3.4	J	3.6	J	0.3
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U				
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U				
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U				
UNDECANE	1120-21-4	3.4	J	3.0	J	2.5	J	2.5	J	2.8	J	2.8	J	0.4
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U				
DODECANE	112-40-3	3.4	J	3.6	J	3.7	J	3.2	J	3.5	J	3.5	J	0.2
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U				
TRIDECANE	629-50-5	2.9	J	3.0	J	4.5	J	2.5	J	2.7	J	3.1	J	0.8
TETRADECANE	629-59-4	1.6	J	2.4	J	4.2	J	1.4	J	1.7	J	2.3	J	1.1
TBP	126-73-8	< 0.8		< 0.8		< 0.8		< 0.8		< 0.8				

Data Quality Flags

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

S Result affected by adhesive tape contamination.

Table F.7 SUMMA™ Replicate Analysis Results for All Target Analytes Sampled from the Headspace of Tank S-102 on 1/26/96

		VSS				ISVS w HEPA				ISVS wo HEPA			
		S6007-A15.084		S6007-A15.084 Rep		S6008-A46.137		S6008-A46.137 Rep		S6009-A55.169		S6009-A55.169 Rep	
Target Analytes	**CAS No.**	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	1.0	J	1.1	J	0.8	J	1.2	J	0.9	J	1.3	J
CHLOROMETHANE	74-87-3	3.5	J	3.5	J	3.1	J	3.5	J	2.8	J	2.9	J
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHAN	76-14-2	U		U		U		U		U		U	
METHANOL	67-56-1	4772	E,Y	5391	E,Y	4547	E,Y	5898	E,Y	4959	E,Y	6625	E,Y
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U	
BUTANE	106-97-8	92		111		73		100		80		118	
BROMOMETHANE	74-83-9	U		U		U		U		U		U	
CHLOROETHANE	75-00-3	U		U		U		U		U		U	
ETHANOL	64-17-5	5180	E,Y	6182	E,Y	4203	E,Y	5397	E,Y	5984	E,Y	6396	E,Y
ACETONITRILE	75-05-8	38		46		36		44		33		31	
ACETONE	67-64-1	413		513		563		458		310		285	
TRICHLOROFLUOROMETHANE	75-69-4	44		55		40		47		32		33	
PENTANE	109-66-0	12		9.0		9.3		9.8		10		10	
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U	
METHYLENE CHLORIDE	75-09-2	0.9	J	0.9	J	0.8	J	0.9	J	0.9	J	0.9	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U	
PROPANOL	71-23-8	41		35		38		40		45		37	
PROPANENITRILE	107-12-0	4.4	J	4.4	J	3.3	J	4.1	J	3.9	J	3.7	J
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U	
2-BUTANONE	78-93-3	56		51		46		59		51		47	
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U	
HEXANE	110-54-3	8.6		7.8		7.6		8.6		13		12	
CHLOROFORM	67-66-3	U		U		U		U		U		U	
TETRAHYDROFURAN	109-99-9	50		44		42		50		47		44	
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U	
BUTANENITRILE	109-74-0	U		U		U		U		U		U	
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U	
1-BUTANOL	71-36-3	431		346		450		514		511		430	
BENZENE	71-43-2	16		14		13		15		14		14	
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U	
CYCLOHEXANE	110-82-7	U		U		U		U		18		17	
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U	
TRICHLOROETHENE	79-01-6	U		U		1.1	J	1.2	J	0.7	J	0.6	J
HEPTANE	142-82-5	9.1		7.5		12		13		36		34	
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U	
4-METHYL-2-PENTANONE	108-10-1	U		U		U		U		U		U	
PYRIDINE	110-86-1	10	J	5.7	J	7.3	J	8.3	J	15	J	7	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U	
PENTANENITRILE	110-59-8	U		U		U		U		6.2		5.8	
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		U		U	
TOLUENE	108-88-3	132		114		108		124		120		115	
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U	

Table F.7 SUMMA™ Replicate Analysis Results for All Target Analytes Sampled from the Headspace of Tank S-102 on 1/26/96

		VSS				ISVS w HEPA				ISVS wo HEPA			
		S6007-A15.084		S6007-A15.084 Rep		S6008-A46.137		S6008-A46.137 Rep		S6009-A55.169		S6009-A55.169 Rep	
Target Analytes	**CAS No.**	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
OCTANE	111-65-9	4.5		3.6		3.4		3.9		4.1		3.5	
TETRACHLOROETHYLENE	127-18-4	10		8.4		7.4		8.5		8.4		7.6	
CHLOROBENZENE	108-90-7	U		U		U		U		0.3	J	U	
HEXANENITRILE	628-73-9	U		U		U		U		U		U	
ETHYLBENZENE	100-41-4	1.2	J	0.9	J	1.0	J	1.1	J	1.1	J	0.9	J
P/M-XYLENE	106-42-3	4.1	J	3.3	J	3.6	J	4.2	J	3.8	J	3.6	J
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U	
STYRENE	100-42-5	U		U		U		U		U		U	
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U	
O-XYLENE	95-47-6	1.8	J	1.4	J	1.4	J	1.7	J	1.6	J	1.6	J
NONANE	111-84-2	2.4		1.6		1.6		2.0		2.0		1.7	
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		U		U		U		U	
1,3,5-TRIMETHYLBENZENE	108-67-8	U		U		U		U		U		U	
1,2,4-TRIMETHYLBENZENE	95-63-6	0.4	J	U		0.4	J	0.4	J	0.5	J	0.4	J
DECANE	124-18-5	1.8	J	1.4	J	1.1	J	1.4	J	1.4	J	1.1	J
CHLOROMETHYLBENZENE, ALPHA	100-44-7	U		U		U		U		U		U	
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U		U	
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U		U	
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U	
UNDECANE	1120-21-4	1.2	J	1.0	J	0.8	J	1.1	J	1.0	J	0.6	J
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		0.3	J	U	
DODECANE	112-40-3	U		U		U		U		U		U	
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		0.2	J	U	
TRIDECANE	629-50-5	0.7	J	0.8	J	0.7	J	0.7	J	0.9	J	U	
TETRADECANE	629-59-4	U		U		U		U		U		U	

Data Quality Flags

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

U Target compound not detected at or above the IDL.

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

E Target compound exceeds upper quantification limit (UQL).

Table F.8 Triple Sorbent Trap Replicate Analysis Results for All Target Analytes Sampled from the Headspace of Tank S-102 on 1/26/96

		VSS				ISVS w HEPA				ISVS wo HEPA			
		S6007 A06.807		S6007 A06.807 Rep		S6008 A39.817		S6008 A39.817 Rep		S6009 A62.830		S6009 A62.830 Rep	
Target Analytes	**CAS No.**	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U		U	
CHLOROMETHANE	74-87-3	U		U		U		U		U		U	
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U	
METHANOL	67-56-1	5961	E,Y	5501	E,Y	4323	E,Y	3138	E,Y	5460	E,Y	5372	E,Y
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U	
BUTANE	106-97-8	57		57		68		68		66		66	
CHLOROETHANE	75-00-3	U		U		U		U		U		U	
ETHANOL	64-17-5	17192	E,Y	17094	E,Y	13006	E,Y	10552	E,Y	13597	E,Y	13409	E,Y
ACETONITRILE	75-05-8	85		92		56		61		93		104	
ACETONE	67-64-1	276		262		241		231		310		290	
TRICHLOROFLUOROMETHANE	75-69-4	30		30		46		44		41		38	
PENTANE	109-66-0	14		12		18		16		23		23	
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U	
METHYLENE CHLORIDE	75-09-2	9.2	J	7.6	J	18	J	26	J	6.2	J	7.8	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U	
PROPANENITRILE	107-12-0	5.1	J	4.9	J	5.0	J	5.0	J	5.6	J	5.7	J
PROPANOL	71-23-8	57		56		54		52		63		64	
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U	
2-BUTANONE	78-93-3	59		58		53		49		60		57	
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U	
HEXANE	110-54-3	9		8.8		126		125		191		191	
CHLOROFORM	67-66-3	U		U		U		U		U		U	
TETRAHYDROFURAN	109-99-9	53		52		49		49		51		51	
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U	
BUTANENITRILE	109-74-0	U		U		U		U		U		U	
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U	
1-BUTANOL	71-36-3	621		593		605		532		981		915	
BENZENE	71-43-2	18		19		19		20		26		26	
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U	
CYCLOHEXANE	110-82-7	U		U		U		U		U		U	
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U	
TRICHLOROETHENE	79-01-6	U		U		2.8	J	2.9	J	1.8	J	1.6	J
HEPTANE	142-82-5	11		11		218		215		569		568	
4-METHYL-2-PENTANONE	108-10-1	11		9		13		13		U		U	
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U	
PYRIDINE	110-86-1	13	J	13	J	12	J	12	J	13	J	18	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U	
PENTANENITRILE	110-59-8	U		U		U		U		U		U	
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		U		38	
TOLUENE	108-88-3	122		122		276		275		504		503	

Table F.8 Triple Sorbent Trap Replicate Analysis Results for All Target Analytes Sampled from the Headspace of Tank S-102 on 1/26/96

		VSS				ISVS w HEPA				ISVS wo HEPA			
		S6007 A06.807		S6007 A06.807 Rep		S6008 A39.817		S6008 A39.817 Rep		S6009 A62.830		S6009 A62.830 Rep	
Target Analytes	**CAS No.**	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U	
OCTANE	111-65-9	4.8	J	4.7	J	5.4	J	5.4	J	7.1		7.5	
TETRACHLOROETHYLENE	127-18-4	9.8		10		8.8		8.9		10		9.8	
CHLOROBENZENE	108-90-7	U		U		U		U		U		U	
HEXANENITRILE	628-73-9	U		U		U		U		U		U	
ETHYLBENZENE	100-41-4	1.2	J	1.1	J	3.7		3.7		4.6		4.7	
P/M-XYLENE	106-42-3	4.2	J	4.2	J	14		14		17		17	
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U	
STYRENE	100-42-5	1.1	J	1.0	J	8.3		8.4		0.9	J	0.9	J
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U	
O-XYLENE	95-47-6	1.8	J	1.8	J	5.2		5.2		6.4		6.6	
NONANE	111-84-2	3.1		3.1		2.9		3.0		3.8		3.9	
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		2.2	J	2.1	J	2.1	J	2.2	J
1,3,5-TRIMETHYLBENZENE	108-67-8	U		U		1.1	J	1.2	J	1.2	J	1.3	J
1,2,4-TRIMETHYLBENZENE	95-63-6	U		U		3.1	J	3.3	J	3.5	J	3.4	J
DECANE	124-18-5	2.4	J	2.3	J	2.7	J	2.8	J	3.8	J	2.8	J
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U		U	
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U		U	
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U	
UNDECANE	1120-21-4	1.4	J	1.3	J	1.6	J	1.6	J	3.0	J	2.6	J
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U	
DODECANE	112-40-3	1.4	J	1.4	J	2.1	J	2.1	J	3.6	J	4.6	J
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U	
TRIDECANE	629-50-5	1.8	J	1.8	J	5.9	J	5.6	J	3.0	J	4.0	J
TETRADECANE	629-59-4	1.0	J	0.9	J	3.3	J	3.5	J	2.4	J	2.9	J
TBP	126-73-8	U		U		U		U		U		U	

Data Quality Flags

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

Table F.9 SUMMA™ Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6007-A01.001 VSS Ambient Upwind (ppbv)	Flag	S6007-A02.074 Ambient Thru VSS (ppbv)	Flag	S6008-A33.123 ISVS W HEPA Bundle A (ppbv)	Flag	S6008-A34.127 ISVS W HEPA Bundle B (ppbv)	Flag	S6009-A35.165 ISVS WO HEPA Bundle C (ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	0.5	J	0.6	J	0.6	J	1.3	J	0.8	J
CHLOROMETHANE	74-87-3	U		U		U		U		U	
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U	
METHANOL	67-56-1	< 19	Y	< 19	Y	< 19	Y	87.0	Y	19	Y
VINYL CHLORIDE	75-01-4	U		U		U		U		U	
BUTANE	106-97-8	U		U		U		U		U	
BROMOMETHANE	74-83-9	U		U		U		U		U	
CHLOROETHANE	75-00-3	U		U		U		U		U	
ETHANOL	64-17-5	< 13	Y	< 13	Y	< 13	Y	< 13	Y	< 13	Y
ACETONITRILE	75-05-8	U		U		U		U		U	
ACETONE	67-64-1	3.9	J	9.5		12		19		7.4	
TRICHLOROFLUOROMETHANE	75-69-4	U		0.4	J	1.3	J	0.9	J	0.4	J
PENTANE	109-66-0	U		U		U		U		U	
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U	
METHYLENE CHLORIDE	75-09-2	U		U		U		U		U	
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U	
PROPANOL	71-23-8	U		U		U		U		U	
PROPANENITRILE	107-12-0	U		U		U		U		U	
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U	
2-BUTANONE	78-93-3	1.5	J	5.1		2.6	J	2.9	J	6.2	
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U	
HEXANE	110-54-3	U		U		U		U		U	
CHLOROFORM	67-66-3	U		U		U		U		U	
TETRAHYDROFURAN	109-99-9	U		U		U		U		U	
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U	
BUTANENITRILE	109-74-0	U		U		U		U		U	
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U	
1-BUTANOL	71-36-3	U		U		1.8	J	1.6	J	1.4	J
BENZENE	71-43-2	U		U		U		U		U	
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U	
CYCLOHEXANE	110-82-7	U		U		U		U		U	
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U	
TRICHLOROETHENE	79-01-6	U		U		0.9	J	0.9	J	0.5	J
HEPTANE	142-82-5	U		U		U		U		U	
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U	
4-METHYL-2-PENTANONE	108-10-1	U		U		U		U		U	
PYRIDINE	110-86-1	U		U		U		U		5.4	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U	

Table F.9 SUMMA™ Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank S-102 on 1/26/96

Target Analytes	CAS No.	S6007-A01.001 VSS Ambient Upwind (ppbv)	Flag	S6007-A02.074 Ambient Thru VSS (ppbv)	Flag	S6008-A33.123 ISVS W HEPA Bundle A (ppbv)	Flag	S6008-A34.127 ISVS W HEPA Bundle B (ppbv)	Flag	S6009-A35.165 ISVS WO HEPA Bundle C (ppbv)	Flag
PENTANENITRILE	110-59-8	U		U		U		U		U	
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		U	
TOLUENE	108-88-3	0.4	J	U		2.5		3.5		2.9	
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U	
OCTANE	111-65-9	U		U		U		U		U	
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U	
CHLOROBENZENE	108-90-7	U		U		U		U		U	
HEXANENITRILE	628-73-9	U		U		U		U		U	
ETHYLBENZENE	100-41-4	U		U		U		U		U	
P/M-XYLENE	106-42-3	U		U		U		U		U	
CYCLOHEXANONE	108-94-1	U		U		U		U		U	
STYRENE	100-42-5	U		U		U		U		U	
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U	
O-XYLENE	95-47-6	U		U		U		U		U	
NONANE	111-84-2	U		U		U		U		U	
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		U		U		U	
1,3,5-TRIMETHYLBENZENE	108-67-8	U		U		U		U		U	
1,2,4-TRIMETHYLBENZENE	95-63-6	U		U		U		U		U	
DECANE	124-18-5	U		U		U		U		U	
CHLOROMETHYLBENZENE, ALPHA	100-44-7	U		U		U		U		U	
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U	
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U	
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U	
UNDECANE	1120-21-4	U		U		U		U		U	
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U	
DODECANE	112-40-3	U		U		U		U		U	
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U	
TRIDECANE	629-50-5	U		U		1.5	J	1.3	J	2.6	J
TETRADECANE	629-59-4	U		U		U		U		2.4	

Data Quality Flags

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

U Target compound not detected at or above the IDL.

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

< Denotes compound not detected at or above the LLS.

Table F.10 Triple Sorbent Trap Blank Sample Analyses Results for All Target Analytes Associated with the Headspace Sampling of Tank S-102 on 1/26/96

		S6007 A11.813 VSS FB #1		S6007 A12.814 VSS FB #2		S6007 A13.815 TB #1		S6007 A14.816 TB #2		S6008 A77.824 ISVS w/HEPA FB #3		S6008 A81.826 ISVS w/HEPA FB #4		S6009 A83.834 ISVS wo/HEPA FB #5		S6009 A84.835 ISVS wo/HEPA FB #6	
Target Analytes	**CAS No.**	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U		U		U		U	
CHLOROMETHANE	74-87-3	U		U		U		U		U		U		U		U	
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U		U		U	
METHANOL	67-56-1	< 192	Y	< 192	Y	< 192	Y	< 192	Y	239		111	J	159	Y	221	Y
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U		U		U	
BUTANE	106-97-8	U		U		U		U		33		13	J	13	J	13	J
CHLOROETHANE	75-00-3	U		U		U		U		U		U		U		U	
ETHANOL	64-17-5	< 133	Y	< 133	Y	< 133	Y	< 133	Y	228	Y	< 133	Y	229	Y	221	Y
ACETONITRILE	75-05-8	U		U		U		U		8.2	J	5.8	J	9.2	J	14	J
ACETONE	67-64-1	10	J	7.8	J	9.8	J	9.2	J	58		46		70		74	
TRICHLOROFLUOROMETHANE	75-69-4	U		U		U		U		32		20		11		11	
PENTANE	109-66-0	U		U		U		U		10		2.7	J	7.8	J	7.2	J
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		8.7		U		U		U	
METHYLENE CHLORIDE	75-09-2	U		U		U		U		21	J	5.1	J	U		U	
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U		U		U	
PROPANENITRILE	107-12-0	U		U		U		U		U		U		U		U	
PROPANOL	71-23-8	U		U		U		U		U		U		U		U	
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U		U		U	
2-BUTANONE	78-93-3	U		U		U		U		6.7	J	3.4	J	9.0		5.6	J
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U		U		U	
HEXANE	110-54-3	U		U		U		U		113		63		150		128	
CHLOROFORM	67-66-3	U		U		U		U		U		U		U		U	
TETRAHYDROFURAN	109-99-9	U		U		U		U		1.9	J	1.7	J	3.6	J	3.3	J
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U		U		U	
BUTANENITRILE	109-74-0	U		U		U		U		U		U		U		U	
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		1.4	J	U		U		U	
1-BUTANOL	71-36-3	U		U		U		U		60		49		316		266	
BENZENE	71-43-2	U		U		U		U		5.7		3.9	J	9.2		8.1	
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U		U		U	
CYCLOHEXANE	110-82-7	U		U		U		U		U		U		U		U	
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U		U		U	
TRICHLOROETHENE	79-01-6	U		U		U		U		3.5	J	1.6	J	1.5	J	1.4	J
HEPTANE	142-82-5	U		U		U		U		178		181		445		336	
4-METHYL-2-PENTANONE	108-10-1	U		U		U		U		U		7.7		U		U	
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U		U		U	
PYRIDINE	110-86-1	U		U		U		U		U		U		U		U	
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U		U		U	
PENTANENITRILE	110-59-8	U		U		U		U		U		U		U		U	
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		6.5		U		U		U	
TOLUENE	108-88-3	U		U		0.6	J	0.9	J	163		131		373		290	
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U		U		U	
OCTANE	111-65-9	U		U		U		U		U		U		2.4	J	1.8	J
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U		U		1.6	J	0.9	J
CHLOROBENZENE	108-90-7	U		U		U		U		U		U		U		U	

Table F.10 Triple Sorbent Trap Blank Sample Analyses Results for All Target Analytes Associated with the Headspace Sampling of Tank S-102 on 1/26/96

		S6007 A11.813 VSS FB #1		S6007 A12.814 VSS FB #2		S6007 A13.815 TB #1		S6007 A14.816 TB #2		S6008 A77.824 ISVS w/HEPA FB #3		S6008 A81.826 ISVS w/HEPA FB #4		S6009 A83.834 ISVS wo/HEPA FB #5		S6009 A84.835 ISVS wo/HEPA FB #6	
Target Analytes	**CAS No.**	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
HEXANENITRILE	628-73-9	U		U		U		U		U		U		U		U	
ETHYLBENZENE	100-41-4	U		U		U		U		2.5	J	1.9	J	3.2		2.3	J
P/M-XYLENE	106-42-3	U		U		U		U		8.7		7.0		11		8.1	
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U		U		U	
STYRENE	100-42-5	U		U		U		U		3.6		2.1	J	0.6	J	U	
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U		U		U	
O-XYLENE	95-47-6	U		U		U		U		3.1	J	2.2	J	4.1		2.9	J
NONANE	111-84-2	U		U		U		U		U		U		U		U	
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		U		U		1.4	J	1.1	J	1.7	J	1.2	J
1,3,5-TRIMETHYLBENZENE	108-67-8	U		U		U		U		1.6	J	0.7	J	1.0	J	0.7	J
1,2,4-TRIMETHYLBENZENE	95-63-6	U		U		U		U		2.2	J	1.7	J	2.7	J	1.8	J
DECANE	124-18-5	U		U		U		U		0.7	J	U		1.6	J	1.0	J
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U		U		U		U		U	
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U		U		U		U		U	
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U		U		U	
UNDECANE	1120-21-4	U		U		U		U		1.5	J	1.6	J	1.4	J	U	
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U		U		U	
DODECANE	112-40-3	U		U		U		U		U		U		U		U	
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U		U		U	
TRIDECANE	629-50-5	U		U		U		U		U		U		U		U	
TETRADECANE	629-59-4	U		U		U		U		U		U		U		U	
TBP	126-73-8	< 0.8	Y	< 0.8	Y	< 0.8	Y	< 0.8	Y	< 0.8	Y	< 0.8	Y	< 0.8	Y	< 0.8	Y

Data Quality Flags

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

U Target compound not detected at or above the IDL.

E Target compound exceeds upper quantification limit (UQL).

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

< Denotes compound not detected at or above the LLS.

Appendix G

Tank Vapor Characterization:

Chain of Custody Sample Control Forms

Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100026

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) 373-2891, Page 85-3152 / FAX 373-3793

Project Designation/Sampling Locations: 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6007 (VSS Truck)

Collection date: 01 - 26 - 96
Preparation date: 01 - 12 - 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		
S6007 - A18 . 71S	Collect NH_3/H_2O Sorbent Trap #1	Sorbent line 3
S6007 - A19 . 72S	Collect NH_3/H_2O Sorbent Trap #2	Sorbent line 4
S6007 - A20 . 73S	Collect NH_3/H_2O Sorbent Trap #3	Sorbent line 5
S6007 - A21 . 74S	Collect NH_3/H_2O Sorbent Trap #4	Sorbent line 6
S6007 - A22 . 75S	Collect NH_3/H_2O Sorbent Trap #5	Sorbent line 7
S6007 - A23 . 76S	Collect NH_3/H_2O Sorbent Trap #6	Sorbent line 8
S6007 - A24 . 77S	Open, close & store NH_3/H_2O field blank #1	N/A
S6007 - A25 . 78S	Open, close & store NH_3/H_2O field blank #2	N/A
S6007 - A26 . 79S	Open, close & store NH_3/H_2O field blank #2	N/A

[ ] Field Transfer of Custody [X] Chain of Possession (Sign and Print Names)

Relinquished By	Date	Time	Received By	Date	Time
G W Dennis G.W. Dennis	01-19-96	1300	J A Edwards J A Edwards	01-19-96	1300
J A Edwards J A Edwards	01-19-96	1300	T. B. Utelt / T. B. Utelt	01-19-96	1300
Tim Utelt / T. B. Utelt	1-30-96	1420	JA Edwards / JA Edwards	1-30-96	1420
JA Edwards / JA Edwards	1-31-96	0905	G.W. Dennis / G.W. Dennis	1-31-96	0905

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 / (Y)N	
◊	COC info/signatures complete?	(Y)N / (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 (E) POC (E)	

(Revised 11/30/95 PNNL)

A-6000-407 (12/92) WEF061

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Battelle Pacific Northwest Lab	CHAIN OF CUSTODY	WHC 100029

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) 373-2891
Page 85-3152 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6008
With HEPA filters *(ISVS Cart)*
Ice Chest No.

Collection date 01 - 24 - 96
Preparation date 01 12 - 96
Field Logbook No. WHC-N-617-8

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

S6008 - A42 . 80S	Collect NH_3/H_2O Sorbent Trap #7	A4
S6008 - A43 . 81S	Collect NH_3/H_2O Sorbent Trap #8	A5
S6008 - A44 . 82S	Collect NH_3/H_2O Sorbent Trap #9	A6
S6008 - A51 . 83S	Collect NH_3/H_2O Sorbent Trap #10	B4
S6008 - A52 . 84S	Collect NH_3/H_2O Sorbent Trap #11	B5
S6008 - A53 . 85S	Collect NH_3/H_2O Sorbent Trap #12	B6
S6008 - A78 . 86S	**Bundle A** Store NH_3/H_2O field blank #4	A8
S6008 - A82 . 87S	**Bundle B** Store NH_3/H_2O field blank #5	B8

[ ] Field Transfer of Custody [X] Chain of Possession (Sign and Print Names)

Relinquished By	Date	Time	Received By	Date	Time
G W Dennis G.W.D	01-19-96	1300	J A Edwards JA Edwards	01-19-96	1300
J A Edwards JA Edwards	01-19-96	1300	T. B. Utech T. B Utech	01-19-96	1300
T. B Utech T. B Utech	1-30-96	1420	JA Edwards JA Edwards	1-30-96	1420
JA Edwards JA Edwards	1-31-96	0905	G.W. Dennis G.W.D	1-31-96	0905

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 / (Y) N	
◊	COC info/signatures complete?	(Y) N / (Y) N	
◊	Rad release stickers on samples?	/ (Y) N	
◊	Activity report from 222S?	/ (Y) N	
◊	RSR/copy? (a ≤100/ß ≤400 pCi/g)	/ (Y) N	
◊	COC copy for LRB, RIDS filed?	/ (Y) N	

POC (P) POC (P)

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Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100032

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) 373-2891, Page 85-3152 / FAX 373-3793

Project Designation/Sampling Locations: 200 West Tank Farm — Collection date 01 - 26 - 96

241-S-102 Tank Vapor Sample SAF S6009 — Preparation date 01 - 12 - 96

Without HEPA filters *(ISVS Cart)*

Ice Chest No. — Field Logbook No. WHC-N-647-8

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

Method of Shipment: Government Truck

Shipped to: PNNL

Possible Sample Hazards/Remarks: Unknown at time of sampling

Sample Identification

S6009 - A66 . 88S	Collect NH_3/H_2O Sorbent Trap #13	C7
S6009 - A67 . 89S	Collect NH_3/H_2O Sorbent Trap #14	C8
S6009 - A68 . 90S	Collect NH_3/H_2O Sorbent Trap #15	C9
S6009 - A69 . 91S	Collect NH_3/H_2O Sorbent Trap #16	C10
S6009 - A70 . 92S	Collect NH_3/H_2O Sorbent Trap #17	C11
S6009 - A71 . 93S	Collect NH_3/H_2O Sorbent Trap #18	C12
S6009 - A85 . 94S	**Bundle C** Store NH_3/H_2O field blank #6	C16
S6009 - A86 . 95S	**Bundle C** Store NH_3/H_2O field blank #7	C17

[ ] Field Transfer of Custody [X] Chain of Possession (Sign and Print Names)

Relinquished By	Date	Time	Received By	Date	Time
G W Dennis	01-19-96	1300	J A Edwards	01-19-96	1300
J A Edwards	01-19-96	1300	T. B Utech	01-19-96	1300
T. B Utech	1-30-96	1470	J A Edwards	1-30-96	1420
J A Edwards	1-31-96	0905	G. W. Dennis	1-31-96	0905

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	/ (Y) N	
◊ COC info/signatures complete?	(Y) N	/ (Y) N	
◊ Rad release stickers on samples?		/ (Y) N	
◊ Activity report from 222S?		/ (Y) N	
◊ RSR/copy? (a ≤100/ß ≤400 pCi/g)		/ (Y) N	
◊ COC copy for LRB, RIDS filed?		/ (Y) N	
	POC (M)	POC (E)	

A-6000-407 (12/92) WEF061

Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100024

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) 373-2891, Page 85-9656 / FAX 373-3793

Project Designation/Sampling Locations: 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6007 (VSS Truck)

Collection date: 01 - 26 - 96
Preparation date: 01 - 18 - 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

S6007 - A01 . 001	Collect Ambient Air Sample SUMMA #1	Upwind of Tank
S6007 - A02 . 074	Collect Ambient Air Sample SUMMA #2	SUMMA Port 15
S6007 - A03 . 082	Collect SUMMA #3	SUMMA Port 13
S6007 - A04 . 083	Collect SUMMA #4	SUMMA Port 15
S6007 - A15 . 084	Collect SUMMA #5	SUMMA Port 13
S6007 - A16 . 097	Collect SUMMA #6	SUMMA Port 15
S6007 - A27 . 108	Collect SUMMA #7	SUMMA Port 13
S6007 - A28 . 120	Collect SUMMA #8	SUMMA Port 15

[ ] Field Transfer of Custody [X] Chain of Possession (Sign and Print Names)

Relinquished By	Date	Time	Received By	Date	Time
J A Edwards / JAEdwards	01-19-96	1300	T.B.Utecht / T-B Utecht	01-19-96	1300
T. B. Utecht / T- B Utecht	1-30-96	1405	JAEDWARDS / JAEdwards	1-30-96	1405

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 / (Y)N	
◊	COC info/signatures complete?	(Y)N / (Y)N	
◊	Rad release stickers on samples?	/ (Y)N	
◊	Activity report from 222S?	/ (Y)N	
◊	RSR/copy? (a ≤100/ß ≤400 pCi/g)	/ (Y)N	
◊	COC copy for LRB, RIDS filed?	/ (Y)N	
		POC (illegible) POC (illegible)	

(Revised 11/30/95 PNNL)

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Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100027

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) 373-2891 Page 85-9656 / FAX 373-3793

Project Designation/Sampling Locations: 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6008
With HEPA filters *(ISVS Cart)*

Collection date: 01 - 26 - 96
Preparation date: 01 - 18 - 96

Ice Chest No.

Field Logbook No. WHC-N -647-8

Bill of Lading/Airbill No.: N/A

Offsite Property No.: N/A

Method of Shipment: Government Truck

Shipped to: PNNL

Possible Sample Hazards/Remarks: Unknown at time of sampling

Sample Identification

S6008 - A33 . 123	Collect Ambient Air Sample SUMMA #9	AS
S6008 - A34 . 127	Collect Ambient Air Sample SUMMA #10	BS
S6008 - A36 . 128	Collect SUMMA #12	AS
S6008 - A37 . 129	Collect SUMMA #13	AS
S6008 - A38 . 134	Collect SUMMA #14	AS
S6008 - A45 . 136	Collect SUMMA #15	BS
S6008 - A46 . 137	Collect SUMMA #16	BS
S6008 - A47 . 153	Collect SUMMA #17	BS

[ ] Field Transfer of Custody [X] Chain of Possession (Sign and Print Names)

Relinquished By	Date	Time	Received By	Date	Time
J A Edwards J A Edwards	01-19-96	1300	T.B. Utley/T.B Utley	01-19-96	1300
T. B Utley/T. Utley	1-30-96	1415	J A Edwards/J A Edwards	1-30-96	1415

Final Sample Disposition

Comments:

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

Comments:
During ambient air purge and sample collection of S6008-A34.127, a pickup truck parked within 20 ft of the tube bundle may have been started. The pickup was noticed during the purge of S6009-A35.165.
RJ Mahon 29JAN96

(Revised 11/30/95 PNNL)

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