PNNL-11151
UC-606
2030

Pacific Northwest National Laboratory

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

TANK VAPOR CHARACTERIZATION PROJECT

Headspace Vapor Characterization of Hanford Waste Tank 241-BY-108: Results from Samples Collected on 01/23/96

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

PNNL-11151

PACIFIC NORTHWEST NATIONAL LABORATORY
operated by
BATTELLE MEMORIAL INSTITUTE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

Printed in the United States of America

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

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

PNNL-11151
UC-606

TANK VAPOR CHARACTERIZATION PROJECT

Headspace Vapor Characterization of Hanford Waste Tank 241-BY-108: Comparison Study Results from Samples Collected on 1/23/96

K. H. Pool
J. C. Evans
B. L. Thomas
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
under Contract DE-AC06-76RLO 1830

Pacific Northwest National Laboratory
Richland, Washington 99352

Summary

This report describes the analytical results of vapor samples taken from the headspace of waste storage tank 241-BY-108 (Tank BY-108) at the Hanford Site in Washington State. The results described in this report 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
ICB	initial calibration blank
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 quantitation limit
VAL	Vapor Analytical Laboratory
VSS	vapor sampling system
WHC	Westinghouse Hanford Company

Contents

Tables

Tables (Cont)

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-BY-108 (Tank BY-108) 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 S6004, S6005, and S6006. Samples were collected by WHC on January 23, 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 25 TSTs for organic analytes (17 samples, 6 field blanks, and 2 trip blanks). The samples and controls were provided to WHC on January 11, 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),

---

(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 issues, and detailed sample results are provided for each category of samples and analyses in Appendices A, B, C, D, and E. Appendix F contains a listing of all target analytes measured during the analysis of samples from this Tank BY-108 comparison study. Appendix G contains the completed COC forms.

2.0 Analytical Results

Samples obtained by WHC from the headspace of Tank BY-108 on January 23, 1996, (Sample Jobs S6004, S6005, and S6006) were analyzed in the PNNL VAL. Summarized results are described in this section. Details of samples, analyses, and data tables are provided in the appendices.

2.1 Water and Ammonia

The complete results of the water and ammonia analysis of Tank BY-108 for the 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 13.7 mg/L in the ISVS samples without HEPA filtration to 14.4 mg/L in the VSS samples. The mean H_2O concentration value for the ISVS samples with HEPA filtration was 14.1 mg/L. Mean NH_3 concentration values ranged from 801 parts per million by volume (ppmv) for the VSS samples to 810 ppmv for the ISVS samples with HEPA filtration. The mean NH_3 concentration value for the ISVS samples without HEPA filtration was 806 ppmv.

Table 2.1. Comparison of Water and Ammonia Mean Values for Samples Collected from the Headspace of Tank BY-108 Using VSS and ISVS With and Without Particulate Filtration

	VSS	ISVS With Filtration	ISVS Without Filtration
Water (mg/L)	14.4	14.1	13.7
Ammonia (ppmv)	801	810	806

2.2 Permanent Gases

The complete results of the permanent gas analysis of Tank BY-108 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. Two permanent gases, hydrogen (H_2) and nitrous oxide (N_2O), were measured above the analytical method estimated quantitation limit (EQL). Methane (CH_4) was observed above the IDL, but below the EQL. Carbon monoxide (CO) was below the IDL in all the samples analyzed. Little differences in H_2, N_2O or CH_4 mean values for the three different sampling methods were found; however, differences were observed for CO_2. The mean CO_2 concentration in the ISVS samples with HEPA filtration were observed at 30 ppmv, significantly higher than observed in either the VSS samples or ISVS samples without HEPA filtration. The difference in CO_2 concentration is thought to be related to an improper connection between the transfer tubing and sampling ports resulting in an ambient air leak.

Table 2.2. Comparison of Permanent Gas Mean Values for Samples Collected from the Headspace of Tank BY-108 Using VSS and ISVS With and Without Particulate Filtration

	VSS	ISVS With Filtration	ISVS Without Filtration
H_2 (ppmv)	361	346	359
CO_2 (ppmv)	12 J	30	17 J
N_2O (ppmv)	469	447	461
CH_4 (ppmv)	15 J	15 J	16 J
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 BY-108 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 189 mg/m^3 for the ISVS samples with HEPA filtration to 222 mg/m^3 for the VSS samples. The average value for the ISVS samples without HEPA filtration was 203 mg/m^3.

Table 2.3. Comparison of TO-12 Mean Values for Samples Collected from the Headspace of Tank BY-108 Using VSS and ISVS With and Without Particulate Filtration

	VSS	ISVS With Filtration	ISVS Without Filtration
TO-12 (mg/m^3)	222	189	203

2.4 Organic Compounds from SUMMA™ Canisters

The complete set of SUMMA™ volatile organic analysis results for Tank BY-108 is found in Appendices D and F of this report. Table 2.4 presents a summary of these data. Some target compounds from the VSS samples, the ISVS samples with HEPA filtration, and the ISVS samples without HEPA filtration exceeded the upper quantitation limit (UQL) for the original analyses. Subsequent analysis runs were diluted (2X, 40X) and appropriate results were reported.

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

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

In summary, 1-butanol, 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 BY-108. Based on the average values for each of the sampling methods, the highest concentrations of methanol, propanol, tetrahydrofuran, 1-butanol, dodecane, tridecane, and tetradecane were observed in the VSS samples. The highest concentrations of ethanol, acetonitrile, and acetone were observed in the ISVS samples with HEPA filtration. The highest concentration of hexane was observed in the ISVS samples without HEPA filtration. Many of the differences in average concentration values may not be significant for the different sampling methods.

The results of the trip and field blank sample analyses indicated 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 BY-108 on 1/23/96

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	2067	Y	1158	Y	131	2723	464	747	1071	18901	732	545	459	Z
ST DEV	132		365		27	1098	35	82	52	2270	87	95	133	
% RSD	6		29		20	38	7	10	5	11	12	16	27	
ISVS with HEPA														
Average	2004	Y	952	Y	126	1989	446	701	965	17726	513	272	90	Z
ST DEV	138		76		10	179	44	85	48	1033	79	72	52	
% RSD	7		8		7	9	10	12	5	6	14	24	53	
ISVS without HEPA														
Average	2042	Y	969	Y	147	2434	476	749	1066	18670	637	354	176	Z
ST DEV	168		96		47	1076	27	61	50	2051	76	70	103	
% RSD	8		9		30	41	6	8	5	11	13	20	55	

Data Qualifier Flags

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

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

Table 2.5 Summary of Triple Sorbent Trap Sample Results for Samples Collected from the Headspace of Tank BY-108 on 1/23/96

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	2731 E,Y	2301 E,Y	200	2072 E	442	755	1102	13086	623	628	445 E	< 0.8 Y
ST DEV	554	470	78	219	55	109	145	785	91	82	173	
% RSD	19	20	36	10	11	13	12	6	13	12	36	
ISVS with HEPA												
Average	2375 E,Y	2351 E,Y	264	2101 E	414	729	1143 E	11828	438	436	261 J	< 0.8 Y
ST DEV	1027	886	40	157	79.3	48	105	1471	71	82	70	
% RSD	33	37	14	7	18	6	9	12	19	21	29	
ISVS without HEPA												
Average	2055 E,Y	2219 E,Y	195.1	1850 E	340	594	1212 E	9897	500	477	283 E	< 0.8 Y
ST DEV	972	927	72	459	124.3	159	157	1832	186	201	134	
% RSD	42	40	35	23	33	24	13	16	34	38	43	

Data Qualifier Flags

E Flag denotes target compound detected above upper calibration standard

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

Y Initial calibration was performed; however, a CCV was not performed. Concentration is considered an estimate.

< Denotes compound not detected at or above the LLS.

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 BY-108 sampled on January 23, 1996. WHC sample job numbers were S6004, S6005, and S6006. 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

---

(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

---

(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 twenty-three 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 100017, 100020, and 100023. The samples were analyzed for water on February 1 and 2, and for ammonia on February 12, 1996.

Table A.2 lists results of the water and ammonia analysis from samples collected from the headspace of Tank BY-108. These samples were collected through the VSS and through the ISVS with and without HEPA filtration. A total of six samples each were collected with the three different sampling methods. Mean water concentration values were 13.7 mg/L, 14.1 mg/L, and 14.4 mg/L for the ISVS samples without HEPA filtration, ISVS samples with HEPA filtration, and VSS samples, respectively. Results show water concentrations were comparable for the three methods.

Mean ammonia concentration values ranged from a low of 801 ppmv for the VSS samples to the high of 810 ppmv for the ISVS samples with HEPA filtration. Mean ammonia concentration value for the ISVS samples without HEPA filtration was 806 ppmv.

Table A.2 Water and Ammonia Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 1/23/96

VSS Truck Samples	H_2O mg/L		NH_3 ppmv
S6004-A18.46S	14.3		800
S6004-A19.47S	14.3		789
S6004-A20.48S	14.6		809
S6004-A21.49S	14.4		815
S6004-A22.50S	14.5		809
S6004-A23.51S	14.4		783
Average	**14.4**		**801**
% RSD	**0.8**		**1.6**
ISVS with HEPA			
S6005-A42.55S	14.3		822
S6005-A43.56S	22.2	S	781
S6005-A44.57S	63.6	S	825
S6005-A51.58S	13.7		817
S6005-A52.59S	14.2		819
A6005-A53.60S	14.2		793
Average	**14.1**		**810**
% RSD	**1.9**		**2.2**
ISVS without HEPA			
S6006-A66.63S	13.6		785
S6006-A67.64S	13.9		823
S6006-A68.65S	13.5		804
S6007-A69.66S	13.6		779
S6007-A70.67S	13.0		844
S6007-A71.68S	14.6		800
Average	**13.7**		**806**
% RSD	**3.9**		**3.0**

S Data suspect; values not used in calculation of average value or % RSD.

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 instrument detection limit (IDL) study was conducted and performance data are presented in Table B.1. The EQL for the method has also been established as 10 times the IDL. Before and after each sample analysis set, a gas standard was run to evaluate system performance and to measure system accuracy. The calculated concentration of the individual gases in the standards fell within $\pm$ 25% of the expected concentrations. One sample was run in duplicate to provide a measure of method precision. Results of the replicate analysis are presented in Table B.2. An N_2 reagent blank, an ambient-air sample collected $\sim$10 m upwind of Tank BY-108, 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 of the permanent gas analysis from samples collected from the headspace of Tank BY-108 and ambient air collected near Tank BY-108 on January 23, 1996. These samples were collected through the VSS and through the ISVS with and without HEPA filtration. A total of six samples each were collected with the three different sampling methods. The samples were analyzed on January 31, 1996. Replicate analysis on single SUMMA™ canisters were conducted on three samples within each sampling method set. Hydrogen (H_2) and nitrous oxide (N_2O) were observed above the EQL in all the tank headspace samples. Average H_2 concentrations ranged from 346 ppmv in the ISVS samples with HEPA filtration to 361 ppmv in the VSS samples. Average N_2O concentrations ranged from 447 ppmv in the ISVS samples with HEPA filtration to 469 ppmv in the VSS samples. Average carbon dioxide (CO_2) concentrations ranged from 12 ppm (below the EQL) in the VSS samples to 30 ppmv in the ISVS samples with HEPA filtration. Carbon dioxide concentrations in the VSS samples and the ISVS samples without HEPA filtration were less than the EQL, but greater than the IDL. Carbon dioxide concentrations in the ISVS samples with HEPA

filtration were significantly higher than either the VSS samples or ISVS samples without HEPA filtration. The difference in CO_2 concentration is thought to be related to an improper connection between the transfer tubing and sampling ports resulting in an ambient air leak. Average methane (CH_4) concentrations appeared to be similar for all three sampling methods, ranging from 15 ppmv (below the EQL) to 16 ppmv (below the EQL). Carbon monoxide (CO) concentrations were all below the IDL.

Table B.2 Permanent Gas Analysis Results for Samples Collected from the Headspace of Tank BY-108 and Ambient Air near Tank BY-108 on 1/23/96

(VSS Truck)	H_2 (ppmv)		CO_2 (ppmv)		N_2O (ppmv)		CH_4 (ppmv)		CO (ppmv)	
S6004-A01.002 (Ambient)	8	J	391		2	U	4	U	3	U
S6004-A02.013 (Ambient)	8	J	394		2	U	4	U	3	U
S6004-A03.029	364		14	J	465		13	J	3	U
S6004-A04.050	362		10	J	481		14	J	3	U
S6004-A15.054	362		12	J	472		16	J	3	U
S6004-A16.055	354		9	J	472		14	J	3	U
S6004-A27.067	352		11	J	461		19	J	3	U
S6004-A28.080	372		13	J	466		15	J	3	U
Average	**361**		**12**	**J**	**469**		**15**	**J**	**3**	**U**
% RSD	**2.0**				**1.5**					
S6004-A03.029 (Rep)	369		24	J	474		15	J	3	U
S6004-A15.050 (Rep)	359		13	J	468		16	J	3	U
S6004-A27.067 (Rep)	357		11	J	458		17	J	3	U
ISVS with HEPA										
S6005-A33.094 (Ambient)	10	J	392		2	U	4	U	3	U
S6005-A34.099 (Ambient)	10	J	393		2	U	4	U	3	U
S6005-A36.114	345		32		453		15	J	3	U
S6005-A37.116	348		29		445		17	J	3	U
S6005-A38.138	338		27		444		12	J	3	U
S6005-A45.143	339		29		440		18	J	3	U
S6005-A46.145	358		30		453		16	J	3	U
S6005-A47.154	351		32		445		15	J	3	U
Average	**346**		**30**		**447**		**15**	**J**	**3**	**U**
% RSD	**2.2**		**6.0**		**1.2**					
S6005-A36.114 (Rep)	350		29		449		13	J	3	U
S6005-A45.143 (Rep)	333		28		438		19	J	3	U
S6005-A46.145 (Rep)	353		29		450		12	J	3	U
ISVS without HEPA										
S6006-A35.161 (Ambient)	10	J	436		2	U	4	U	3	U
S6006-A54.182	364		19	J	456		14	J	3	U
S6006-A55.208	358		17	J	470		20	J	3	U
S6006-A56.211	357		16	J	455		16	J	3	U
S6006-A57.228	356		17	J	466		17	J	3	U
S6006-A58.323	367		19	J	464		12	J	3	U
S6006-A59.324	355		14	J	455		18	J	3	U
Average	**359**		**17**	**J**	**461**		**16**	**J**	**3**	**U**
% RSD	**1.3**				**1.4**					
S6006-A54.182 (Rep)	361		21	J	457		11	J	3	U
S6006-A57.228 (Rep)	352		17	J	465		16	J	3	U
S6006-A59.324 (Rep)	347		13	J	453		16	J	3	U

Data Qualifier Flags

J Flag to denote target compound detected but, quantitated amount below Estimated Quantitation Limit (EQL).

U Flag to denote target compound not detected above Instrument Detection Limit (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:

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

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 of the TO-12 gas analysis from samples collected from the headspace of Tank BY-108 and ambient air collected near Tank BY-108 on January 23, 1996. These samples were collected through the VSS and through the ISVS with and without HEPA filtration. A total of six samples each were collected with the three different sampling methods. The samples were analyzed on February 6 and 7, 1996. Replicate analyses on single SUMMA™ canisters were conducted on three samples within each sampling method set. Concentrations in the five ambient air samples ranged from 0.34 mg/m^3 (below the EQL) to 0.91 mg/m^3. Average concentrations ranged from 189 mg/m^3 in the ISVS samples with HEPA filtration to 222 mg/m^3 in the VSS samples. The average concentration in the ISVS samples without HEPA filtration was measured at 203 mg/m^3.

Table C.1 TO-12 Analysis Results for Samples Collected from the Headspace of Tank BY-108 and Ambient Air near Tank BY-108 on 1/23/96

(VSS Truck)	TO-12 mg/m3	
S6004-A01.002 (Ambient)	0.34	J
S6004-A02.013 (Ambient)	0.91	
S6004-A03.029	220	
S6004-A04.050	226	
S6004-A15.054	222	
S6004-A16.055	222	
S6004-A27.067	224	
S6004-A28.080	216	
Average	**222**	
% RSD	**1.6**	
S6004-A03.029 (Rep)	225	
S6004-A15.050 (Rep)	221	
S6004-A27.067 (Rep)	221	
ISVS with HEPA		
S6005-A33.094 (Ambient)	0.72	J
S6005-A34.099 (Ambient)	0.67	J
S6005-A36.114	184	
S6005-A37.116	190	
S6005-A38.138	192	
S6005-A45.143	177	
S6005-A46.145	185	
S6005-A47.154	205	
Average	**189**	
% RSD	**5.0**	
S6005-A36.114 (Rep)	175	
S6005-A45.143 (Rep)	176	
S6005-A46.145 (Rep)	186	
ISVS without HEPA		
S6006-A35.161 (Ambient)	0.55	J
S6006-A54.182	201	
S6006-A55.208	198	
S6006-A56.211	202	
S6006-A57.228	202	
S6006-A58.323	205	
S6006-A59.324	212	
Average	**203**	
% RSD	**2.4**	
S6006-A54.182 (Rep)	199	
S6006-A57.228 (Rep)	201	
S6006-A59.324 (Rep)	208	

Data Qualifier Flag

J Flag to denote target compound detected but, quantitated amount below Estimated Quantitation Limit (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

---

(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 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

(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.

the IS concentration. Once it is determined that the relative response is linear with increasing concentration, an average response factor is calculated for each target analyte and used to determine the concentration of target compounds in each sample. Method blanks are analyzed before and after calibration standards and tank headspace samples are analyzed.

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, and 5 ambient air samples were returned to the laboratory on January 30, 1996, under WHC COC numbers 100015, 100018, and 100021. Samples were analyzed between March 18 through March 21, 1996.

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

Table D.2 lists the quantitative results for the 12 compounds selected for this tank comparison study. Six 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. 1-Butanol, methanol, and acetone were the most abundant compounds identified in each of the SUMMA™ canister samples. Tributyl phosphate was not quantified in any of the SUMMA™ canister samples. Based on the average values for each of the sampling methods the highest concentrations of methanol, ethanol, acetone, hexane, 1-butanol, dodecane, tridecane, and tetradecane were observed in the VSS samples. The highest concentrations of acetonitrile, propanol and tetrahydrofuran were observed in the ISVS samples 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 presented in Table D.3. The RPD was calculated for analytes detected above the IDL and found in both replicates.

Results of the blank sample analyses are presented in table D.4. Low levels of acetone and 1-butanol were observed in several of the samples.

Instrument detection limits (IDLs), 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 currently not part of the operating procedure.

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

Batch #1 (file identifier 16031801.b) - S6004-A04.050, S6006-A57.228, S6006-A59.324, S6004-A04.050 REP, S6005-A46.145, S6005-A47.154, S6004-A28.080;

Batch #2 (file identifier 16031901.b) - S6006-A55.208, S6006-A58.323, S6004-A27.067, S6005-A38.138, S6004-A15.054, S6006-A58.323 REP, S6005-A45.143;

Batch #3 (file identifier 16032001.b) - S6005-A36.114, S6004-A03.029, S6005-A37.116, S6006-A54.182, S6005-A36.114 REP, S6004-A16.055, S6006-A56.211;

Batch #4 (file identifier 16032202.b) - S6004-A01.002, S6004-A02.013, S6005-A33.094, S6005-A34.099, S6006-A35.161;

Batch #5 (file identifier 16032301.b) - S6006-A55.208, S6006-A58.323, S6004-A27.067, S6005-A38.138, S6004-A15.054, S6006-A58.323 REP, S6005-A45.143;

Batch #6 (file identifier 16032401.b) - S6006-A55.208, S6004-A27.067, S6005-A38.138, S6004-A15.054, S6006-A58.323 REP, S6005-A45.143;

Batch #7 (file identifier 16032501.b) - S6005-A36.114, S6004-A03.029, S6005-A37.116, S6006-A54.182, S6005-A36.114 REP, S6004-A16.055, S6006-A56.211;

Batch #8 (file identifier 16032601.b) - S6004-A04.050, S6006-A57.228, S6006-A59.324, S6004-A04.050 REP, S6005-A46.145, S6005-A47.154, S6004-A28.080;

Batch #9 (file identifier 16032801.b) - S6004-A04.050, S6006-A57.228, S6006-A59.324, S6004-A04.050 REP, S6005-A46.145, S6005-A47.154, S6004-A28.080;

Batch #10 (file identifier 16032901.b) - S6005-A36.114, S6004-A03.029, S6005-A37.116, S6006-A54.182, S6005-A36.114 REP, S6004-A16.055, S6006-A56.211.

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

Batch #1 (normal dilution):

1. Pentane at 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 exceeding the upper quantitation limit (UQL) and flagged with an "E."

2. Four target compounds (pentane, tridecane, tetradecane, and 1,2-dichloro-1,1,2,2-tetrafluoroethane) were outside the ± 25% difference (% 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 1,2-dichloro-1,1,2,2-tetrafluoroethane was not found in the tank samples. Pentane, tridecane, and tetradecane were found in some tank samples at concentrations above the estimated quantitation limit (EQL) and for other tank samples at concentrations exceeding their UQLs which were flagged "E." Their concentration values may be over or under estimated because of the failed CCV. The concentration data flagged with an "E" will be reported in the final report using a different analytical run (samples diluted).

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 4 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. Tetradecane was found in the initial calibration blank (ICB) above the EQL; however, it was not found in the continuing calibration blank (CCB) above the EQL. Tetradecane tank results in this analysis run could be affected by carryover or system contamination.

Batch #2 (normal dilution):
1. Pentane at 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 exceeding the UQL and flagged with an "E."

2. Nine target compounds (tetrachloroethylene, chlorobenzene, ethyl benzene, toluene, octane, hexachlorobutadiene, pentane, 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. Tetrachloroethylene, chlorobenzene, ethyl benzene, toluene, octane, and hexachlorobutadiene either were not found or were found in tank samples at concentrations below the EQL. Pentane, tridecane, and tetradecane were found in some tank samples at concentrations above the EQL, and for other tank samples at concentrations exceeding their UQLs which were flagged "E." Their concentration values may be over or under estimated because of the failed CCV. The concentration data flagged with an "E" will be reported in the final report using a different analytical run (samples diluted).

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 2 hours and 2 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. Tetradecane was found in the ICB above the EQL; however, it was not found in the CCB above the EQL. Tetradecane tank results in this analysis run could be affected by carryover or system contamination.

Batch #3 (normal dilution):
1. Pentane at 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 exceeding the UQL and flagged with an "E."

2. Four target compounds (tetrachloroethylene, pentane, 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. Tetrachloroethylene was not found in the tank samples. Pentane and tetradecane were found in some tank samples at concentrations above the EQL and for other tank samples at concentrations exceeding their UQLs which were flagged "E." Their concentration values may be over or under estimated because of the failed CCV. The concentration data flagged with an "E" will be reported in the final report using a different analytical run (samples diluted).

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 1 hours and 47 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. Tetradecane was found in the ICB above the EQL; however, it was not found in the CCB above the EQL. Tetradecane tank results in this analysis run could be affected by carryover or system contamination.

Batch #4 (normal dilution):
1. Tetradecane at 36.25% surpassed the 30% RSD acceptance criteria for the initial calibration; however, tetradecane was not found in the tank samples.

2. Five target compounds (trichlorofluoromethane, pentane, dodecane, 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. None of these target compounds were found in tank samples above their EQLs.

3. Tetradecane was found in the ICB above the EQL; however, it was not found in the CCB or in tank samples above the EQL. Thus, no data results were affected.

Batch #5 (1:40 dilution):
1. Tetradecane at 36.25% surpassed the 30% RSD acceptance criteria for the initial calibration; however, this analysis run should be used only for reporting of 1-butanol.

2. Seven target compounds (trichlorofluoromethane, 4-methyl-2-pentanone, cis-1,3-dichloropropene, trans-1,3-dichloropropene, pentanenitrile, dodecane, and 1,1,2-trichloroethane) 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. This analysis run should be used only for reporting 1-butanol results.

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 13 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. Tetradecane was found in the ICB and in the CCB above the EQL. Tetradecane tank results in this analysis run could be affected by carryover or system contamination; however, this analysis run should be used only for reporting of 1-butanol.

Batch #6 (volume reduced from 100 ml to 50 ml):
1. Tetradecane at 36.25% surpassed the 30% RSD acceptance criteria for the initial calibration. Tetradecane was found in tank samples at concentrations between the EQL and UQL. Tetradecane concentration values may be over or under estimated because of the failed CCV.

2. Eight target compounds (1,2-dibromomethane, tetrachloroethylene, ethylbenzene, p/m xylene, pentanenitrile, 1-ethyl-2-methyl-benzene, cyclohexanone, and 1,1,2-trichloroethane) 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. None of these target compounds were found in tank samples above their EQLs.

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 4 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. Tetradecane was found in the ICB above the EQL, but was not found in the CCB. Tetradecane was found in tank samples at concentrations between the EQL and UQL and its concentration in this analysis run could be affected by carryover or system contamination.

Batch #7 (volume reduced from 100 ml to 50 ml):
1. Tetradecane at 36.25% surpassed the 30% RSD acceptance criteria for the initial calibration. Tetradecane was found in tank samples at concentrations between the EQL and UQL. Tetradecane concentration values may be over or under estimated because of the failed CCV.

2. Six target compounds (1,1,2-trichloroethane, pyridine, dodecane, 1-butanol, trichlorofluoromethane, and cyclohexanone) 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. Pyridine, cyclohexanone, and 1,1,2-trichloroethane were not found in tank samples at concentrations above their EQLs. The compound 1-butanol exceeded the UQL and should not be reported using this analysis run. Dodecane and trichlorofluoromethane were found in tank samples at concentrations above the EQL and 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 20 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. Tetradecane was found in the ICB above the EQL, but was not found in the CCB above the EQL. Tetradecane was found in tank samples at concentrations between the EQL and UQL and its concentration in this analysis run could be affected by carryover or system contamination.

Batch #8 (1:40 dilution):
1. Tetradecane at 36.25% surpassed the 30% RSD acceptance criteria for the initial calibration; however, this analysis run should be used only for reporting of 1-butanol.

2. Five target compounds (pyridine, cyclohexanone, alpha chloromethylbenzene, propanol, and dodecane) 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. This analysis run should be used only for reporting 1-butanol results.

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 10 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. Tetradecane was found in the ICB and in the CCB above the EQL. Tetradecane tank results in this analysis run could be affected by carryover or system contamination; however, this analysis run should be used only for reporting of 1-butanol.

Batch #9 (volume reduced from 100 ml to 50 ml):
1. Tetradecane at 36.25% surpassed the 30% RSD acceptance criteria for the initial calibration. Tetradecane was found in tank samples at concentrations between the EQL and UQL. Tetradecane concentration values may be over or under estimated because of the failed CCV.

2. Six target compounds (1,1,2-trichloroethane, dodecane, 4-methyl-2-pentanone, trans-1,3-dichloropropene, pyridine, and 1,3-dichlorobenzene) 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. Dodecane was the only target compound found in tank samples above the EQL and its 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 4 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. Tetradecane was found in the ICB and in the CCB above the EQL. Tetradecane was found in tank samples at concentrations between the EQL and UQL and its concentration in this analysis run could be affected by carryover or system contamination.

Batch #10 (1:40 dilution):
1. Tetradecane at 36.25% exceeded the 30% RSD acceptance criteria for the initial calibration; however, this analysis run should be used only for reporting of 1-butanol.

2. Five target compounds (trichlorofluoromethane, 1-ethyl-2-methyl benzene, 1,2,4-trichlorobenzene, dodecane, 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. This analysis run should be used only for reporting 1-butanol results.

3. The 12-hour clock procedure criterion for the analytical sequence was exceeded by 1 hour and 46 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. Tetradecane was found in the ICB above the EQL but was not found in the CCB above the EQL. Tetradecane tank results in this analysis run could be affected by carryover or system contamination; however, this analysis run should be used only for reporting of 1-butanol.

Table D.2 SUMMA™ Sample Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 1/23/96

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)
S6004-A03.029	2137	Y	1017	Y	147	2269	511	851	1141	20425	683	549	471	Z
S6004-A04.050	1827	Y	1900	Y	113	4255	411	651	1048	17087	662	570	643	Z
S6004-A15.054	2201	Y	1057	Y	116	1923	484	781	1030	18873	808	515	387	Z
S6004-A16.055	2070	Y	979	Y	119	3997	471	791	1133	22057	628	405	295	Z
S6004-A27.067	2141	Y	1010	Y	113	1873	467	765	1026	19238	761	530	374	Z
S6004-A28.080	2025	Y	986	Y	180	2022	438	646	1047	15728	848	699	581	Z
Average	**2067**	**Y**	**1158**	**Y**	**131**	**2723**	**464**	**747**	**1071**	**18901**	**732**	**545**	**459**	**Z**
ST DEV	**132**		**365**		**27**	**1098**	**35**	**82**	**52**	**2270**	**87**	**95**	**133**	
% RSD	**6**		**29**		**20**	**38**	**7**	**10**	**5**	**11**	**12**	**16**	**27**	
ISVS with HEPA														
S6005-A36.114	2032	Y	948	Y	114	2309	405	711	944	16885	478	221	35	Z
S6005-A37.116	1742	Y	817	Y	126	1894	504	788	942	18607	432	221	63	Z
S6005-A38.138	2126	Y	1007	Y	127	1812	499	772	954	19096	549	271	91	Z
S6005-A45.143	1990	Y	929	Y	115	1864	435	742	945	16657	425	201	47	Z
S6005-A46.145	2030	Y	984	Y	137	2017	408	588	940	16934	584	343	135	Z
S6005-A47.154	2103	Y	1028	Y	137	2035	429	607	1062	18177	610	375	166	Z
Average	**2004**	**Y**	**952**	**Y**	**126**	**1989**	**446**	**701**	**965**	**17726**	**513**	**272**	**90**	**Z**
ST DEV	**138**		**76**		**10**	**179**	**44**	**85**	**48**	**1033**	**79**	**72**	**52**	
% RSD	**7**		**8**		**7**	**9**	**10**	**12**	**5**	**6**	**14**	**24**	**53**	
ISVS without HEPA														
S6006-A54.182	1895	Y	889	Y	127	2133	491	807	1083	21106	599	298	101	Z
S6006-A55.208	2287	Y	1130	Y	122	2193	467	766	1104	17378	746	345	128	Z
S6006-A56.211	2012	Y	952	Y	125	2004	500	809	1136	21171	530	275	106	Z
S6006-A57.228	1880	Y	900	Y	242	1817	429	664	1045	16110	669	465	300	Z
S6006-A58.323	2208	Y	1036	Y	124	1849	500	761	1021	18252	680	340	104	Z
S6006-A59.324	1971	Y	904	Y	141	4610	467	688	1007	18003	601	404	316	Z
Average	**2042**	**Y**	**969**	**Y**	**147**	**2434**	**476**	**749**	**1066**	**18670**	**637**	**354**	**176**	**Z**
ST DEV	**168**		**96**		**47**	**1076**	**27**	**61**	**50**	**2051**	**76**	**70**	**103**	
% RSD	**8**		**9**		**30**	**41**	**6**	**8**	**5**	**11**	**13**	**20**	**55**	

Data Qualifier Flags

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

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

Table D.3 Replicate Analysis of SUMMA™ Canisters for Samples Collected from the Headspace of Tank BY-108 on 1/23/96

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)
S6004-A04.050	1827	Y	1900	Y	113	4255	411	651	1048	17087	662	570	643	Z
S6004-A04.050 Rep	1979	Y	958	Y	134	1919	367	622	1027	19430	773	653	614	Z
Relative Percent Difference	8		66		17	76	11	5	2	13	15	14	5	
ISVS with HEPA														
S6005-A36.114	2032		948		114	2309	405	711	944	16885	478	221	35	Z
S6005-A36.114 Rep	1627		750		116	2891	460	760	943	17713	391	165	37	Z
Relative Percent Difference	22		23		2	22	13	7	0	5	20	29	7	
ISVS without HEPA														
S6006-A58.323	2208		1036		124	1849	500	761	1021	18252	680	340	104	Z
S6006-A58.323 Rep	2252		1048		118	1903	463	728	909	18393	649	327	100	Z
Relative Percent Difference	2		1		5	3	8	4	12	1	5	4	4	

Data Qualifier Flags

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

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

Table D.4 SUMMA™ Blank Sample Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 1/23/96

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	10	J	U	U	U	2	J	U	U	U	Z
Upwind Through VSS	< 19	Y	< 13	Y	U	U		U	U	U	2	J	U	U	U	Z
Bundle A W/HEPA	< 19	Y	< 13	Y	U	12	J	U	U	U	2	J	U	U	U	Z
Bundle B W/HEPA	< 19	Y	< 13	Y	U	49		U	U	U	3	J	U	U	U	Z
Bundle C WO/HEPA	< 19	Y	< 13	Y	U	20		U	U	U	3	J	U	U	U	Z
Lab Control BLK	< 19	Y	< 13	Y	U	U		U	U	U	0.5	J	U	U	U	Z

Data Qualifier Flags

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

U Target compound not detected at or above the IDL.

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

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

< Denotes compound not detected at or above the LLS.

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 was performed for methanol and ethanol; however, a CCV was not performed. Therefore, concentrations reported are considered estimated for these compounds. The methanol and ethanol LLS was used as the EQL. Results below the LLS were not reported. The calibration mixture is prepared in common with the mixture used for the SUMMA™ analysis (see Section D.2). The standard calibration mix was analyzed using 4 aliquot sizes ranging from 100 mL to 1200 mL, and a response factor for each compound was calculated. Volumes of standard added to the traps are measured by pressure difference on a SUMMA™ canister of known volume. The GC/MS response for these compounds has been previously determined to be linearly related to concentration. 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 12 compounds shown in Table E.1. A gas mixture containing difluorobenzene, chlorobenzene-d_5, and 1,4 bromofluorobenzene was used as an IS for all calibration standard and sample analyses. Analyte responses from sample components, ISs, and standards were obtained from the extracted ion plot from their selected mass ion. A continuing calibration was generated by calculating the relative response ratios of the IS to calibration standard responses and plotting the ratios against the ratio of the calibration-standard concentration (in ppbv) to the IS concentration. Once it is determined that the relative response is linear with increasing concentration, an average response factor is calculated for each target analyte and used to determine the concentration of target compounds in each sample.

E.3.1 Quantitation Results of Target Analytes. The quantitative-analysis results for the target analytes were calculated directly from the calibration curve generated using the IS method described above and in PNL-TVP-10. 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:

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

E.4 Triple Sorbent Trap Volatile Organic Sample Results

Twenty-five TSTs consisting of 17 samples, 6 field blanks, and 2 trip blanks were returned to the laboratory on January 30, 1996, under WHC COC numbers 100016, 100019, and 100022. Most samples were analyzed between February 14 and 23, 1996.

The results from the GC/MS analysis of the tank headspace TST samples are presented in Table E.2. The results of replicate analyses on single TST samples are presented in Table E.3. The results of the blank samples analysis are presented in Table E.4

Table E.2 lists the quantitative results for the 12 compounds selected for this tank comparison study. Six individual TST samples were collected for each of the three different sampling methods. The individual compound values for each of the TST samples for each sampling method were averaged and a ST DEV and % RSD values were calculated. Methanol, 1-butanol, and ethanol were the most abundant compounds identified in each of the trap samples. Tributyl phosphate was not observed in any samples from Tank BY-108. Sample S6006-A39.731, representing a sample collected with the ISVS with HEPA filtration, was not included in the average value calculation because of suspect data. Based on the average values for each of the sampling methods, the highest concentrations of methanol, propanol, tetrahydrofuran, 1-butanol, dodecane, tridecane, and tetradecane were observed in the VSS samples. The highest concentrations of ethanol, acetonitrile, and acetone were observed in the ISVS samples with HEPA filtration. The highest concentration of hexane was observed in the ISVS samples without HEPA filtration. Many of the differences in average concentration values may not be significant for the different sampling methods.

Single TST samples were analyzed in replicate for each of the three different sampling methods. The RPDs were calculated and are presented in Table E.3. The RPDs were calculated for analytes detected above the IDL and found in both replicates.

The results of blank analyses are reported in Table E.4. Compounds observed in the blank samples included methanol, ethanol, acetone, tetrahydrofuran, hexane, 1-butanol, dodecane, tridecane, tetradecane, and traces of acetonitrile and propanol. The sources of these compounds in the blank samples is believed to be from the tape used to wrap the sample bundles and from passive sampling. In depth discussion of these two issues are discussed below.

Samples were collected from tank BY-108 by VSS and ISVS methods with and without HEPA filters on 1/23/96. 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 1/17/96 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 2/14/96 through 3/4/96.

Instrument detection limits, precision, and accuracy have not been experimentally evaluated for methanol, ethanol, and TBP. 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, ethanol, and TBP results falling within their 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 eight batches. The analytical sequence runs (batches) were as follows:

Batch 2/14/96 (file identifier 46021401.d) - S6004-A11.716, S6004-A12.718, S6005-A77.742, S6005-A81.743;

Batch 2/15/96 (file identifier 46021501.d) - S6006-A83.760, S6006-A84.761;

Batch 2/16/96 (file identifier 46021601.d) - S004-A13.725, S6004-A14.727, S6004-A06.708, S6005-A41.733, S6006-A63.755;

Batch 2/20/96 (file identifier 46022001.d) - S6004-A07.709, S6005-A39.731, S6004-A05.707, S6004-A05.707 REP;

Batch 2/21/96 (file identifier 46022101.d) - S6005-A49.738, S6006-A61.745, S6005-A48.734, S6005-A48.734 REP;

Batch 2/22/96 (file identifier 46022201.d) - S6004-A09.711, S6006-A62.754, S6005-A50.741, S6004-A08.710, S6004-A09.711 REANALYSIS;

Batch 2/23/96 (file identifier 46022301.d) - S6006-A65.758, S6006-A65.758 REP, S6004-A10.715, S6005-A40.732;

Batch 3/4/96 (file identifier 46030401.d) - S6004-A07.709 REANALYSIS.

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

Batch 2/14/96:
Four target compounds (acetone, pyridine, 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. Tridecane and tetradecane were not found in the samples. Acetone was found in samples S6004-A11.716 and S6004-A12.718 at a concentration between the EQL and IDL. Acetone was found in samples S6005-A77.742 and S6005-A81.743 at a concentration above the EQL. Acetone concentration values may be over or under estimated because of the failed CCV.

Batch 2/15/96:
Five target compounds (vinyl chloride, butane, acetone, pyridine, 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. Vinyl chloride was not found in the samples. Pyridine was found in the samples at a concentration between the EQL and IDL. Acetone, butane, and tetradecane were found in samples at a concentration above the EQL. These compounds concentration values may be over or under estimated because of the failed CCV.

Batch 2/16/96:
Three target compounds (acetone, pyridine, 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. Acetone and tetradecane were not found in samples S6004-A13.725 and S6004-A14.727. Acetone and tetradecane were found in the remaining samples at a concentration above the UQL. Pyridine was found in the samples at a concentration between the EQL and IDL. These compounds concentration values may be over or under estimated due to the failed CCV.

Batch 2/20/96:
Three target compounds (pyridine, 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. Pyridine was found in the samples at a concentration between the EQL and IDL. Tetradecane and tridecane were found in the samples at a concentration above the UQL. These compounds concentration values may be over or under estimated because of the failed CCV. Data for sample S6005-A39.731 is considered suspect and was not included in the preceding discussion.

One VSS sample, S6004-A07.709, required a reanalysis because of unsatisfactory IS responses. The sample split tube was rerun successfully on 3/4/96.

Batch 2/21/96:
Four target compounds (acetone, pyridine, 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. Pyridine was not found in sample S6006-A61.745. Pyridine was found in the remaining samples at a concentration between the EQL and IDL. Acetone, tridecane and tetradecane were found in the samples at a concentration above the EQL. These compounds concentration values may be over or under estimated due to the failed CCV.

Batch 2/22/96:
Three target compounds (pyridine, 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. Pyridine was found in the samples at a concentration between the EQL and IDL. Tridecane and tetradecane was found in the samples at a concentration above the UQL. These compounds concentration values may be over or under estimated because of the failed CCV.

One VSS sample, S6004-A09.711, required reanalysis because of unacceptably large IS response and low surrogate recoveries. The rerun produced abnormally low IS responses suggesting that the splitter on the instrument failed to provide a 10:1 split. The surrogate recoveries were acceptable. The data associated with this sample will be qualified as suspect.

Batch 2/23/96:
Three target compounds (pyridine, 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. Pyridine was found in the samples at a concentration between the EQL and IDL. Tridecane and tetradecane was found in the samples at

a concentration above the UQL. These compounds concentration values may be over or under estimated because of the failed CCV.

Batch 3/4/96:
Four target compounds (pyridine, decane, 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. Pyridine was found in the sample at a concentration between the EQL and IDL. Decane was found in the sample at a concentration above the EQL. Tridecane and tetradecane was found in the sample at a concentration above the UQL. These compounds concentration values may be over or under estimated because of the failed CCV.

Several issues associated with the data generated are discussed below.

Samples were analyzed under the protocols of TVP-10, Rev. 2 with initial calibration performed on January 17, 1996 and quantitated against CCVs run at beginning of each batch for the target compounds listed in the method. The samples were subjected to GC/MS analysis over the period February 14, 1996 through March 23, 1996. In addition, one VSS sample was reanalyzed on March 4, 1996 by the same protocol. The attached table details the order in which the samples were run and notes reanalysis and duplicate analysis. Minor QC problems encountered during the course of the work are noted in daily case narratives. In general, the analysis proceeded smoothly. Several specific problems are discussed below.

All field samples (VSS, ISVS, and trip blanks) contained moderately high and variable amounts of 1-chloro,1,1-difluoroethane. This compound has appeared persistently in all samples sent to the field up through January 23, 1996. It is believed to be a fugitive refrigerant. This material is never present in tubes archived for a similar amount of time in the 326 Vapor Lab or 329 Building temporary storage. The origin of the material is unclear, but since it has shown up in trip blanks (including those from this tank run) as well as field blanks, the most likely candidate is one of the refrigerators used for interim storage during sampling and radiological screening. It is worth noting, however, that the third tank job in the comparison study (Tank S-102) performed on January 26, 1996 prior to the analysis of the C-107 and BY-108 samples did not show any evidence of this material indicating that either some change in sample storage protocol has occurred or the leak has been fixed (or the source exhausted).

In addition to the fluorocarbon problem, a more serious blank problem was seen in all samples and blanks exposed to the ISVS sampling head. The compounds seen included but were not limited to methanol, 1-butanol, hexane, heptane, methyl hexane, and methyl cyclohexane. The source of the problem was traced to the abundant use of plastic adhesive tape (3M Scotch Brand 471) to bind the in situ package during insertion into the tank. The adhesive material on this tape is clearly a major source of volatile organic compounds. Analysis of a piece of the tape from the same roll used for the sampling job confirmed this supposition. A clean TST was exposed to the tape adhesive for 1 hour and analyzed by standard protocols during routine analysis of C-107 samples. 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 from C-107, BY-108, and S-102.

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 BY-108 combined with the blank problems from the tape, the passive sampling effect is difficult to accurately assess and in any case 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 acetone (15%), ethanol (18%), tetrahydrofuran (7%), and dodecane (12%). Passive sampling under the conditions used for BY-108 is thus estimated to contribute on the order of 10 to 20% for the ISVS samples without HEPA filtration and somewhat less for the ISVS samples with HEPA filtration sampling head which provides a longer flow path to the tubes. This is a considerably larger effect than was estimated from the C-107 sampling experience on January 16, 1996 and may simply reflect operational differences associated with, among other things, the length of time used for the in-tank portion of the sampling event. Sampling should be performed as promptly as possible following insertion of the sampling bundle into the tank to minimize passive sampling associated errors.

The TST analyses are performed under very prescriptive QC protocols as documented in method PNL-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 BY-108, two VSS samples (A07.709 and A09.711) were subjected to reanalysis. In both cases, unacceptably large variations in the IS responses were the cause. The first sample, A07.709, was successfully reanalyzed during a later batch and data are reported from the second analysis. The second sample A09.711 failed the IS test again on reanalysis. The problem is believed to have been erratic behavior of the sample splitter. The second analysis should be used. Except for the somewhat low IS values, all other quality indicators including surrogate recoveries and comparability with other related sample results were excellent for the reanalysis of that sample.

The total organic loading of Tank BY-108 is relatively high resulting in many of the compounds amenable to TST analysis falling outside the calibrated range. Target compounds frequently flagged as above the upper quantitation limit (E) include butane, acetone, pentane, hexane, 1-butanol, heptane, tridecane, and tetradecane. In most cases these results are close to but somewhat above the upper quantitation limit. There should be limited impact on data quality for those compounds. In the case of 1-butanol, however, the measured concentrations are more than an order of magnitude above the method upper quantitation limit and should clearly be considered suspect. 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 that procedure does not address the issue of trap saturation.

Because of problems with a lengthy procurement process, no standards were available for methanol and ethanol in the CCV at the time the samples were run and those compounds are not currently included in PNL-TVP-10. Those compounds were initially evaluated as TICs, however, it is clear that quantitation of those compounds as TICs produces an unacceptably large error for the purposes of the comparison study. Commercially calibrated vapor permeation tubes were obtained for methanol and ethanol at the end of February and added to the Kin-Tec vapor standard generation system. A new initial calibration was prepared and run on March 1, 1996. All data from Tank BY-108 was placed in a separate batch file and batch reprocessed for quantitation of methanol and ethanol as targets using the March 1, 1996 initial calibration rather than CCVs as per standard protocol. Since the calibration mixture used IS from the same batch was used for sample analysis and similar response was obtained, this procedure should be valid.

In addition to the methanol and ethanol, the compound sublist used for batch processing also included TBP processed as a target using calibrated retention time and response factors determined on October 5, 1995. Response factors were taken from a six point linear calibration curve prepared from dilutions of the neat material in methanol and injected directly onto the ends of the traps. The same lot of IS was used for this calibration as for the later sample analysis discussed here. Response factors determined on October 5, 1995 were manually entered into the method so that a single processing could be used. Full performance data has not been determined for TBP. Based on the relatively high response factor obtained, it is clear that instrument response and thus delectability 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. That number should be conservative. Tributyl phosphate was not observed in any sample using any of the three sampling methods.

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.

Eighteen TST tubes were sent to the field for use as blanks or sample collection media. Of those, all VSS samples and blanks were analyzed. All ISVS samples and blanks with HEPA filtration were analyzed, however, one sample, S6005-A39.731, had analyte levels more typical of a field blank (i.e. passive sampling) than for a 200 ml sample. It appears that the sampling line was not connected during the sampling event. An examination of the field records from that day supports that supposition since the pressure drop was abnormally low for that sampling line. Two ISVS samples without HEPA filtration were lost due to breakage. Sample S6006-A64.756 was broken in the field during assembly of the bundle and was so noted on the COC. Sample S6006-A60.744 was found to be cracked during the analytical procedure and attempts to retrieve the sample were unsuccessful. The ISVS samples without HEPA filtration were taken using bare tubes while the ISVS samples with HEPA filtration bundle used tubes in commercially supplied plastic protective tubes. All future sampling should use the protective tube to minimize the potential for damage to the sampling tubes.

Data is reported for acetonitrile, however, during processing it was noted that the GC/MS peak has a substantial coeluting interferant thus rendering the data potentially suspect.

Table E.2 Triple Sorbent Trap Sample Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 1/23/96

VSS 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)
S6004-A06.708	3337 E,Y	2865 E,Y	246	2105 E	476	816	1156 E	12892 E	679 E	648 E	418 E	<0.8 Y
S6004-A07.709	3038 E,Y	2082 E,Y	266	2075 E	442	707	1131 E	11936 E	525 E	554 E	782 E	<0.8 Y
S6004-A05.707	2840 E,Y	2701 E,Y	236	2068 E	464	794	1178 E	14241 E	651 E	625 E	362 E	<0.8 Y
S6004-A09.711	2831 E,Y	1559 Y	50	1693 E	341	555	808	13495 E	496 E	538 E	293 E	<0.8 Y
S6004-A08.710	2632 E,Y	2411 E,Y	209	2378 E	497	844	1150 E	13289 E	720 E	769 E	436 E	<0.8 Y
S6004-A10.715	1710 Y	2186 E,Y	194	2117 E	430	815	1187 E	12662 E	668 E	634 E	379 E	<0.8 Y
Average	**2731.3 E,Y**	**2300.5 E,Y**	**200**	**2072 E**	**442**	**755**	**1102 E**	**13086 E**	**623 E**	**628 E**	**445 E**	**<0.8 Y**
ST DEV	**554**	**470**	**78**	**219**	**55**	**109**	**145**	**785**	**91**	**82**	**173**	
% RSD	**19**	**20**	**36**	**10**	**11**	**13**	**12**	**6**	**13**	**12**	**36**	
ISVS with HEPA												
S6005-A41.733	3683 E,Y	2537 E,Y	321	1907 E	401	671	984	9994 E	474	468 E	331 E	<0.8 Y
S6005-A39.731	545 Y,S	298 Y,S	28 S	166 S	18 J,S	32 S	124 S	1230 E,S	27 J,S	20 J,S	6 J,S	<0.8 Y,S
S6005-A49.738	3261 E,Y	3184 E,Y	239	2103 E	468	711	1213 E	12992 E	312	296	156	<0.8 Y
S6005-A48.734	3780 E,Y	3144 E,Y	272	2056 E	500	741	1213 E	13365 E	460	432 E	230	<0.8 Y
S6005-A50.741	1504 Y	1184 Y	216	2342 E	293	718	1086 E	10621 E	465	495 E	285 E	<0.8 Y
S6005-A40.732	2025 E,Y	1705 Y	274	2099 E	407	802	1220 E	12169 E	479	488 E	305 E	<0.8 Y
Average	**2375 E,Y**	**2351 E,Y**	**264**	**2101 E**	**414**	**729**	**1143 E**	**11828 E**	**438**	**436 E**	**261 E**	**<0.8 Y**
ST DEV	**1027**	**886**	**40**	**157**	**79**	**48**	**105**	**1471**	**71**	**82**	**70**	
% RSD	**33**	**37**	**14**	**7**	**18**	**6**	**9**	**12**	**19**	**21**	**29**	
ISVS without HEPA												
S6006-A63.755	1973 E,Y	2342 E,Y	121	1535	366	567	1136 E	10811 E	475	460 E	294 E	<0.8 Y
S6006-A61.745	738 Y	925 Y	146	1379	157	385	1030 E	7161 E	247	204	91	<0.8 Y
S6006-A65.758	2515 E,Y	3122 E,Y	256	2215 E	409	671	1371 E	10602 E	636 E	580 E	359 E	<0.8 Y
S6006-A62.754	2994 E,Y	2487 E,Y	258	2270 E	426	753	1310 E	11015 E	644 E	666 E	386 E	<0.8 Y
Average	**2055 E,Y**	**2219 E,Y**	**195**	**1850 E**	**340**	**594**	**1212 E**	**9897 E**	**500 E**	**477 E**	**283 E**	**<0.8 Y**
ST DEV	**972**	**926.78**	**72**	**459**	**124**	**159**	**157**	**1832**	**186**	**201**	**134**	
% RSD	**42**	**40**	**35**	**23**	**33**	**24**	**13**	**16**	**34**	**38**	**43**	

Data Qualifier Flag

S Data suspect; not used in calculation of average value, st dev, or % RSD.

E Flag denotes target compound detected above upper calibration standard

J Flag denotes target compound detected above IDL, but below Estimated Quantitation Limit (EQL)

U Flag to denote target compound not detected above Instrument Detection Limit (IDL).

Y Initial calibration was performed; however, a CCV was not performed. Concentration is considered an estimate.

< Denotes compound not detected at or above the LLS.

Table E.3 Replicate Analysis of Triple Sorbent Trap Samples Collected from the Headspace of Tank BY-108 on 1/23/96

VSS Truck Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)	ACETONE (ppbv)		PROPANENITRILE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)		1-BUTANOL (ppbv)		DODECANE (ppbv)		TRIDECANE (ppbv)		TETRADECANE (ppbv)		TBP (ppbv)	
S6004-A05.707	2840	E,Y	2701	E,Y	236	2068	E	17	464	794	1178	E	14241	E	651	E	625	E	362	E	< 0.8	Y
S6004-A05.707 Rep	2640	E,Y	2515	E,Y	244	1935	E	17	430	746	1139	E	13922	E	635	E	601	E	345	E	< 0.8	Y
Relative Percent Difference	7		7		3	7		0	7	6	3		2		3		4		5			
ISVS with HEPA																						
S6005-A48.734	3780	E,Y	3144	E,Y	272	2056	E	15	500	741	1213	E	13365	E	460		432	E	230		< 0.8	Y
S6005-A48.734 Rep	3593	E,Y	2994	E,Y	282	1950	E	15	462	699	1212	E	13812	E	461		435	E	228		< 0.8	Y
Relative Percent Difference	5		5	E	3	5		0	8	6	0	E	3	E	0		1		1			
ISVS without HEPA																						
S6006-A65.758	2515	E,Y	3122	E,Y	256	2215	E	15	409	671	1371	E	10602	E	636	E	580	E	359	E	< 0.8	Y
S6006-A65.758 Rep	2319	E,Y	2752	E,Y	264	2055	E	16	420	712	1475	E	12571	E	693	E	631	E	365	E	< 0.8	Y
Relative Percent Difference	8		13	E	3	8		3	3		7	E	17	E	9	E	8		2	E		

Data Qualifier Flag

E Flag denotes target compound detected above upper calibration standard

U Flag to denote target compound not detected above Instrument Detection Limit (IDL).

Y Initial calibration was performed; however, a CCV was not performed. Concentration is considered an estimate.

< Denotes compound not detected at or above the LLS.

Table E.4 Triple Sorbent Trap Blank Sample Analysis Results for Samples Collected from the Headspace of Tank BY-108 on 1/23/96

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	9.4 J	U	U	U	3.0 J	U	U	U	< 0.8 Y
Field Blank # 2 VSS	< 192 Y	< 133 Y	U	11 J	U	U	U	2.5 J	U	U	U	< 0.8 Y
Field Blank #3 W/HEPA	290 Y	< 133 Y	4.1 J	140	15 J	35	133	888	3.9 J	U	U	< 0.8 Y
Field Blank #4 W/HEPA	347 Y	173 Y	4.6 J	192	15 J	26	149	927	U	U	U	< 0.8 Y
Field Blank #5 WO/HEPA	504 Y	366 Y	5.7 J	250	20 J	38	442	1534 E	44	41 J	38	< 0.8 Y
Field Blank #6 WO/HEPA	508 Y	391 Y	6.2 J	272	24	46	531	2075 E	65	61 J	46	< 0.8 Y
Trip Blank #1	< 192 Y	< 133 Y	U	15 J	U	U	U	U	U	U	U	< 0.8 Y
Trip Blank #2	< 192 Y	< 133 Y	U	16 J	U	U	U	U	U	U	U	< 0.8 Y

Data Qualifier Flag

E Flag denotes target compound detected above upper calibration standard

J Flag to denote target compound detected but, quantitated amount below Estimated Quantitation Limit (EQL)

U Flag to denote target compound not detected above Instrument Detection Limit (IDL).

Y Initial calibration was performed; however, a CCV was not performed. Concentration is considered an estimate.

< Denotes compound not detected at or above the LLS.

Appendix F

Tank Vapor Characterization:

Target Analytes Measured

Table F.1 SUMMA™ Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6004-A03.029 (ppbv)	Flag	S6004-A04.050 (ppbv)	Flag	S6004-A15.054 (ppbv)	Flag	S6004-A16.055 (ppbv)	Flag	S6004-A27.067 (ppbv)	Flag	S6004-A28.080 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U		U				
CHLOROMETHANE	74-87-3	12		9.7	J	10	J	11		10	J	13		11	J	1.2
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U				
METHANOL	67-56-1	2137	Y	1827	Y	2201	Y	2070	Y	2141	Y	2025	Y	2067	Y	132
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U				
BUTANE	106-97-8	2235		1937		1854		2178		1979		1977		2027		148
BROMOMETHANE	74-83-9	U		U		U		U		U		U				
CHLOROETHANE	75-00-3	U		U		U		U		U		U				
ETHANOL	64-17-5	1017	Y	1900	Y	1057	Y	979	Y	1010	Y	986	Y	1158	Y	364
ACETONITRILE	75-05-8	147		113		116		119		113		180		131		27
ACETONE	67-64-1	2269		4255		1923		3997		1873		2022		2723		1098
TRICHLOROFLUOROMETHANE	75-69-4	46		35		32		60		31		65		45		15
PENTANE	109-66-0	1414		2034		1822		2159		1876		788		1682		506
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	1.4	J	1.4	J	1.3	J	1.4	J	1.2	J	1.1	J	1.3	J	0.1
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U				
PROPANOL	71-23-8	511		411		484		471		467		438		464		35
PROPANENITRILE	107-12-0	8.6	J	4.1	J	4.8	J	4.6	J	5.1	J	4.0	J	5.2	J	1.7
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U				
2-BUTANONE	78-93-3	348		304		361		354		382		354		351		26
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U				
HEXANE	110-54-3	1141		1048		1030		1133		1026		1047		1071		52
CHLOROFORM	67-66-3	U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	851		651		781		791		765		646		748		82
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U				
BUTANENITRILE	109-74-0	90		83		90		79		86		110		90		11
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U				
1-BUTANOL	71-36-3	20425		17087		18873		22057		19238		15728		18901		2270
BENZENE	71-43-2	21		16		20		20		20		18		19		1.8
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	143		134		137		128		133		157		139		10
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	521		442		481		452		466		553		486		43
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	17	J	15	J	14	J	14	J	13	J	14	J	15	J	1.4
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U				
PENTANENITRILE	110-59-8	7.4	J	7.1	J	19		7.5	J	7.1	J	10	J	9.7	J	4.7
1,1,2-TRICHLOROETHANE	79-00-5	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 BY-108 on 1/23/96

Target Analytes	CAS No.	S6004-A03.029 (ppbv)	Flag	S6004-A04.050 (ppbv)	Flag	S6004-A15.054 (ppbv)	Flag	S6004-A16.055 (ppbv)	Flag	S6004-A27.067 (ppbv)	Flag	S6004-A28.080 (ppbv)	Flag	Mean	Flag	Std. Dev.
TOLUENE	108-88-3	27		25		26		24		25		26		26		1.0
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U				
OCTANE	111-65-9	178		152		158		160		152		165		161		9.8
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U		U				
CHLOROBENZENE	108-90-7	1.0	J	U		U		U		0.9	J	0.7	J	0.9	J	0.2
HEXANENITRILE	628-73-9	1.5	J	1.2	J	2.4	J	1.7	J	1.2	J	1.8	J	1.6	J	0.4
ETHYLBENZENE	100-41-4	4.2	J	4.0	J	3.9	J	3.8	J	3.7	J	4.5	J	4.0	J	0.3
P/M-XYLENE	106-42-3	14	J	14	J	13	J	12	J	12	J	16	J	14	J	1.5
CYCLOHEXANONE	108-94-1	12	J	12	J	6.7	J	13	J	9.8	J	14		11	J	2.6
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	6.1		5.3	J	5.7	J	5.5	J	5.5	J	5.4	J	5.6	J	0.3
NONANE	111-84-2	85		71		76		76		74		72		76		5.0
1-ETHYL-2-METHYL BENZENE	611-14-3	0.6	J	0.7	J	0.6	J	0.6	J	0.6	J	0.6	J	0.6	J	0.04
1,3,5-TRIMETHYLBENZENE	108-67-8	0.4	J	0.5	J	0.4	J	0.4	J	0.4	J	0.6	J	0.5	J	0.1
1,2,4-TRIMETHYLBENZENE	95-63-6	1.2	J	1.2	J	1.1	J	1.1	J	1.1	J	1.1	J	1.1	J	0.1
DECANE	124-18-5	128		103		114		118		111		118		115		8.3
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		0.3	J	U		U		U		U		0.3	J	
UNDECANE	1120-21-4	270		219		243		257		259		281		255		22
1,2,4-TRICHLOROBENZENE	120-82-1	U		0.8	J	U		U		U		1.0	J	0.9	J	0.1
DODECANE	112-40-3	683		662		808		628		761		848		732		87
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		0.7	J	U		U		U		U		0.7	J	
TRIDECANE	629-50-5	549		570		515		405		530		699		545		95
TETRADECANE	629-59-4	471		643		387		295		374		581		459		133

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.

Table F.2 Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6004-A05.707 (ppbv)	Flag	S6004-A06.708 (ppbv)	Flag	S6004-A07.709 (ppbv)	Flag	S6004-A08.710 (ppbv)	Flag	S6004-A09.711 (ppbv)	Flag	S6004-A10.715 (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	2840	E,Y	3337	E,Y	3038	E,Y	2632	E,Y	2831	E,Y	1710	Y	2731	E,Y	554
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U				
BUTANE	106-97-8	2450	E	2491	E	2305	E	2510	E	1762	E	2544	E	2344	E	297
CHLOROETHANE	75-00-3	U		U		U		U		U		U				
ETHANOL	64-17-5	2701	E,Y	2865	E,Y	2082	E,Y	2411	E,Y	1559	Y	2186	E,Y	2301	E,Y	469
ACETONITRILE	75-05-8	236		246		266		209		50		194		200		78
ACETONE	67-64-1	2068	E	2105	E	2075	E	2378	E	1693	E	2117	E	2073	E	219
TRICHLOROFLUOROMETHANE	75-69-4	25		26		24		31		22		27		26		3.1
PENTANE	109-66-0	2246	E	2239	E	2064	E	2236	E	1605	E	2292	E	2114	E	261
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	114		54		663	E	19	J	492		847	E	365	E,J	351
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U				
PROPANENITRILE	107-12-0	17		18		19		18		U		14		17		1.9
PROPANOL	71-23-8	464		476		442		497		341		430		442		55
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U				
2-BUTANONE	78-93-3	305		315		282		330		242		331		301		34
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U				
HEXANE	110-54-3	1178	E	1156	E	1131	E	1150	E	808		1187	E	1102	E	145
CHLOROFORM	67-66-3	U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	794		816		707		844		555		815		755		109
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U				
BUTANENITRILE	109-74-0	9.0		8.5		U		U		U		U		8.8		0.4
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U				
1-BUTANOL	71-36-3	14241	E	12892	E	11936	E	13289	E	13495	E	12662	E	13086	E	785
BENZENE	71-43-2	24		25		26		28		22		27		25		2.2
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	215		164		159		217		118		U		175		42
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U				
TRICHLOROETHENE	79-01-6	1.5	J	U		4.1	J	U		7.2		11		5.9	J	4.0
HEPTANE	142-82-5	584		600		535		576		387		590		545		81
4-METHYL-2-PENTANONE	108-10-1	45		47		47		46		31		47		44		6.3
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U				
PYRIDINE	110-86-1	10	J	13	J	15	J	10	J	8.0	J	10	J	11	J	2.5
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U				
PENTANENITRILE	110-59-8	U		1.3	J	1.9	J	U		U		U		1.6	J	0.4
1,1,2-TRICHLOROETHANE	79-00-5	U		8.8		U		U		U		U		8.8		
TOLUENE	108-88-3	31		29		35		33		27		37		32		3.7

Table F.2 Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6004-A05.707 (ppbv)	Flag	S6004-A06.708 (ppbv)	Flag	S6004-A07.709 (ppbv)	Flag	S6004-A08.710 (ppbv)	Flag	S6004-A09.711 (ppbv)	Flag	S6004-A10.715 (ppbv)	Flag	Mean	Flag	Std. Dev.
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U				
OCTANE	111-65-9	179		176		177		188		124		191		173		25
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U		U				
CHLOROBENZENE	108-90-7	U		U		U		U		U		U				
HEXANENITRILE	628-73-9	U		U		U		U		U		U				
ETHYLBENZENE	100-41-4	4.5		4.2		5.3		4.7		3.9		5.1		4.6		0.5
P/M-XYLENE	106-42-3	15		14		17		16		11		16		15		2.1
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U				
STYRENE	100-42-5	U		U		1.7	J	U		U		U		1.7	J	
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U				
O-XYLENE	95-47-6	6.9		6.7		8.3		7.6		4.8		7.3		6.9		1.2
NONANE	111-84-2	85		86		88		88		65		89		84		9.2
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		0.8	J	U		U		U		0.8	J	
1,3,5-TRIMETHYLBENZENE	108-67-8	1.2	J	1.2	J	0.8	J	1.4	J	U		U		1.1	J	.0.3
1,2,4-TRIMETHYLBENZENE	95-63-6	1.1	J	1.1	J	1.9	J	1.2	J	U		1.2	J	1.3	J	0.3
DECANE	124-18-5	105		103		112		109		77		108		102		13
1,3-DICHLOROBENZENE	541-73-1	U		U		0.5	J	U		U		U		0.5	J	
1,4-DICHLOROBENZENE	106-46-7	U		U		0.5	J	U		U		U		0.5	J	
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U		U		U				
UNDECANE	1120-21-4	271		282		246		284		190		278		259		36
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U				
DODECANE	112-40-3	651	E	679	E	525	E	720	E	496	E	668	E	623	E	91
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U				
TRIDECANE	629-50-5	625	E	648	E	554	E	769	E	538	E	634	E	628	E	82
TETRADECANE	629-59-4	362	E	418	E	782	E	436	E	293	E	379	E	445	E	172
TBP	126-73-8	< 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 was performed; however, a CCV was not performed. Concentration is considered an estimate.

< Denotes compound not detected at or above the LLS.

Table F.3 SUMMA™ Analysis Results for All Target Analytes for ISVS Samples With HEPA Filtration Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6005-A36.114 (ppbv)	Flag	S6005-A37.116 (ppbv)	Flag	S6005-A38.138 (ppbv)	Flag	S6005-A45.143 (ppbv)	Flag	S6005-A46.145 (ppbv)	Flag	S6005-A47.154 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U		U				
CHLOROMETHANE	74-87-3	11	J	12		10	J	9.8	J	11		12		11	J	0.9
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U		U				
METHANOL	67-56-1	2032	Y	1742	Y	2126	Y	1990	Y	2030	Y	2103	Y	2004	Y	138
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U				
BUTANE	106-97-8	2319		1902		1751		1800		2012		2018		1967		204
BROMOMETHANE	74-83-9	U		U		U		U		U		U				
CHLOROETHANE	75-00-3	U		U		U		U		U		U				
ETHANOL	64-17-5	948	Y	817	Y	1007	Y	929	Y	984	Y	1028	Y	952	Y	76
ACETONITRILE	75-05-8	114		126		127		115		137		137		126		10
ACETONE	67-64-1	2309		1894		1812		1864		2017		2035		1989		180
TRICHLOROFLUOROMETHANE	75-69-4	32		59		32		31		59		57		45		15
PENTANE	109-66-0	2008		1791		1807		1856		804		807		1512		553
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	1.1	J	1.3	J	1.4	J	1.3	J	1.1	J	1.0	J	1.2	J	0.2
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U				
PROPANOL	71-23-8	405		504		499		435		408		429		447		44
PROPANENITRILE	107-12-0	4.1	J	5.4	J	5.5	J	4.6	J	3.8	J	3.5	J	4.5	J	0.8
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U				
2-BUTANONE	78-93-3	308		284		348		321		346		367		329		30
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U				
HEXANE	110-54-3	1109		951		991		1002		1121		1062		1039		69
CHLOROFORM	67-66-3	U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	711		788		772		742		588		607		701		85
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U				
BUTANENITRILE	109-74-0	89		77		80		83		91		101		87		8.7
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U				
1-BUTANOL	71-36-3	16885		18607		19096		16657		16934		18177		17726		1033
BENZENE	71-43-2	17		19		20		19		16		17		18		1.5
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	132		128		130		131		136		144		134		5.8
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	467		451		469		462		471		512		472		21
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	16	J	15	J	13	J	13	J	12	J	25	J	16	J	4.8
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U				
PENTANENITRILE	110-59-8	U		6.6	J	7.3	J	6.9	J	7.1	J	8.7	J	7.3	J	0.8
1,1,2-TRICHLOROETHANE	79-00-5	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 BY-108 on 1/23/96

Target Analytes	CAS No.	S6005-A36.114 (ppbv)	Flag	S6005-A37.116 (ppbv)	Flag	S6005-A38.138 (ppbv)	Flag	S6005-A45.143 (ppbv)	Flag	S6005-A46.145 (ppbv)	Flag	S6005-A47.154 (ppbv)	Flag	Mean	Flag	Std. Dev.
TOLUENE	108-88-3	23		25		25		24		23		25		24		1.0
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U				
OCTANE	111-65-9	141		157		151		143		143		155		148		6.9
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U		U				
CHLOROBENZENE	108-90-7	U		0.9	J	U		U		0.9	J	U		0.9	J	0.1
HEXANENITRILE	628-73-9	U		2.0	J	1.8	J	1.5	J	2.2	J	2.2	J	2.0	J	0.3
ETHYLBENZENE	100-41-4	3.3	J	3.6	J	3.7	J	3.5	J	3.9	J	4.2	J	3.7	J	0.3
P/M-XYLENE	106-42-3	11	J	12	J	12	J	11	J	14	J	15	J	12	J	1.7
CYCLOHEXANONE	108-94-1	11	J	11	J	U		9.4	J	13	J	15		12	J	2.2
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	4.9	J	5.4	J	5.4	J	5.0	J	4.7	J	5.1	J	5.1	J	0.3
NONANE	111-84-2	66		74		72		67		62		67		68		4.3
1-ETHYL-2-METHYL BENZENE	611-14-3	0.6	J	0.6	J	0.5	J	1.0	J	0.5	J	0.6	J	0.6	J	0.2
1,3;5-TRIMETHYLBENZENE	108-67-8	0.7	J	0.4	J	0.4	J	1.1	J	0.6	J	0.4	J	0.6	J	0.3
1,2,4-TRIMETHYLBENZENE	95-63-6	1.1	J	1.1	J	1.1	J	1.0	J	1.1	J	1.1	J	1.1	J	0.05
DECANE	124-18-5	95		107		104		94		98		102		100		5.2
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	199		182		195		166		205		233		197		23
1,2,4-TRICHLOROBENZENE	120-82-1	1.0	J	0.6	J	U		0.6	J	0.7	J	0.9	J	0.8	J	0.2
DODECANE	112-40-3	478		432		549		425		584		610		513		79
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.7	J	U		U		U		U		U		0.7	J	
TRIDECANE	629-50-5	221		221		271		201		343		375		272		72
TETRADECANE	629-59-4	35		63		91		47		135		166		90		52

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.

Table F.4 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples With HEPA Filtration Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6005-A39.731 (ppbv)	Flag	S6005-A40.732 (ppbv)	Flag	S6005-A41.733 (ppbv)	Flag	S6005-A48.734 (ppbv)	Flag	S6005-A49.738 (ppbv)	Flag	S6005-A50.741 (ppbv)	Flag	Mean	Flag	Std. Dev
DICHLORODIFLUOROMETHANE	75-71-8	U	S	U		U		U		U		U				
CHLOROMETHANE	74-87-3	U	S	U		U		U		U		U				
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U	S	U		U		U		U		U				
METHANOL	67-56-1	545	Y,S	2025	E,Y	3683	E,Y	3780	E,Y	3261	E,Y	1504	E,Y	2466	E,Y	1315
VINYL CHLORIDE	75-01-4	U	S	U		U		U		U		U				
BUTANE	106-97-8	173	S	2513	E	2188	E	2335	E	2241	E	2245	E	1949		878
CHLOROETHANE	75-00-3	U	S	U		U		U		U		U				
ETHANOL	64-17-5	298	Y,S	1705	E,Y	2537	E,Y	3144	E,Y	3184	E,Y	1184	E,Y	2009	E,Y	1153
ACETONITRILE	75-05-8	28	S	274		321		272		239		216		225		103
ACETONE	67-64-1	166	S	2099	E	1907	E	2056	E	2103	E	2342	E	1779	E	802
TRICHLOROFLUOROMETHANE	75-69-4	14	S	27		26		34		36		31		28		7.9
PENTANE	109-66-0	144	S	2298	E	1877	E	2226	E	2210	E	2006	E	1794	E	823
1,1-DICHLOROETHENE	75-35-4	U	S	U		4.5	J	3.9	J	11		U		6.5	J	3.9
METHYLENE CHLORIDE	75-09-2	2666	E,S	310		270		1841	E	3595	E	936	E	1603	E	1345
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U	S	U		U		U		1.3	J	1.7	J	1.5	J	0.2
PROPANENITRILE	107-12-0	U	S	17		20		15		16		17		17		1.9
PROPANOL	71-23-8	18	J,S	407		401		500		468		293		348	J	176
1,1-DICHLOROETHANE	75-34-3	U	S	U		U		U		U		U				
2-BUTANONE	78-93-3	28	S	314		277		402		408		344		296		140
CIS-1,2-DICHLOROETHENE	156-59-2	U	S	U		U		U		U		U				
HEXANE	110-54-3	124	S	1220	E	984		1213	E	1213	E	1086	E	973	E	427
CHLOROFORM	67-66-3	U	S	U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	32	S	802		671		741		711		718		613		288
1,2-DICHLOROETHANE	107-06-2	U	S	U		U		U		U		U				
BUTANENITRILE	109-74-0	U	S	U		U		U		U		10		10		
1,1,1-TRICHLOROETHANE	71-55-6	U	S	U		U		U		U		U				
1-BUTANOL	71-36-3	1230	E,S	12169	E	9994	E	13365	E	12992	E	10621	E	10062	E	4522
BENZENE	71-43-2	16	S	35		35		36		40		37		33		8.6
CARBON TETRACHLORIDE	56-23-5	U	S	U		U		U		U		U				
CYCLOHEXANE	110-82-7	40	S,Z	189	Z	164	Z	233	Z	U		215	Z	168	Z	76
1,2-DICHLOROPROPANE	78-87-5	U	S	U		U		U		U		U				
TRICHLOROETHENE	79-01-6	40	S	4.1	J	1.3	J	32		67		9.9		26	J	26
HEPTANE	142-82-5	186	S,Z	769	E,Z	670	E,Z	908	E,Z	900	E,Z	813	E,Z	708	E,Z	270
4-METHYL-2-PENTANONE	108-10-1	8.3	S,Z	47	Z	42	Z	53	Z	56	Z	49	Z	43	Z	17
CIS-1,3-DICHLOROPROPENE	10061-01-5	U	S	U		U		U		U		U				
PYRIDINE	110-86-1	U	S	10	J	11	J	9.4	J	9.9	J	10	J	10	J	0.6
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U	S	U		U		U		U		U				
PENTANENITRILE	110-59-8	U	S	U		U		U		U		U				
1,1,2-TRICHLOROETHANE	79-00-5	5.3	S	8.7		U		U		U		U		7.0		2.4
TOLUENE	108-88-3	98	S,Z	122	Z	100	Z	188	Z	200	Z	165	Z	146	Z	45
1,2-DIBROMOETHANE	106-93-4	U	S	U		U		U		U		U				

Table F.4 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples With HEPA Filtration Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6005-A39.731 (ppbv)	Flag	S6005-A40.732 (ppbv)	Flag	S6005-A41.733 (ppbv)	Flag	S6005-A48.734 (ppbv)	Flag	S6005-A49.738 (ppbv)	Flag	S6005-A50.741 (ppbv)	Flag	Mean	Flag	Std. Dev
OCTANE	111-65-9	12	S	177		161		172		152		171		141		64
TETRACHLOROETHYLENE	127-18-4	U	S	U		U		U		U		U				
CHLOROBENZENE	108-90-7	U	S	U		U		U		U		U				
HEXANENITRILE	628-73-9	U	S	U		U		U		U		U				
ETHYLBENZENE	100-41-4	7.2	S	11		9.2		12		13		11		11		2.1
P/M-XYLENE	106-42-3	23	S	42		34		41		40		39		37		7.2
CYCLOHEXANONE	108-94-1	U	S	U		U		U		U		U				
STYRENE	100-42-5	3.6	S	2.0	J	1.6	J	3.4	J	6.2		2.2	J	3.2	J	1.7
1,1,2,2-TETRACHLOROETHANE	79-34-5	U	S	U		U		U		U		U				
O-XYLENE	95-47-6	7.7	S	15		13		15		15		15		13		2.9
NONANE	111-84-2	5.5	S	77		69		77		65		71		61		27
1-ETHYL-2-METHYL BENZENE	611-14-3	U	S	U		U		U		U		U				
1,3,5-TRIMETHYLBENZENE	108-67-8	1.5	J,S	2.2	J	1.8	J	2.2	J	2.2	J	2.2	J	2.0	J	0.3
1,2,4-TRIMETHYLBENZENE	95-63-6	5.6	S	7.9		6.5		7.9		7.8		7.7		7.2		1.0
DECANE	124-18-5	7.0	S	85		78		85		66		79		67		30
1,3-DICHLOROBENZENE	541-73-1	U	S	U		U		U		U		U				
1,4-DICHLOROBENZENE	106-46-7	U	S	U		U		U		U		U				
1,2-DICHLOROBENZENE	95-50-1	U	S	U		U		U		U		U				
UNDECANE	1120-21-4	14	S	197		201		191		131		190		154		73
1,2,4-TRICHLOROBENZENE	120-82-1	U	S	U		U		U		U		U				
DODECANE	112-40-3	27	J,S	479		474		460		312		465		370		179
HEXACHLORO-1,3-BUTADIENE	87-68-3	U	S	U		U		U		U		U				
TRIDECANE	629-50-5	20	J,S	488	E	468	E	432	E	296		495	E	367	J,E	185
TETRADECANE	629-59-4	5.5	J,S	305	E	331	E	230		156		285	E	219	J,E	122
TBP	126-73-8	< 0.8	Y,S	< 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).

S Flag denotes data is suspect.

Z Results affected by adhesive tape contamination.

Y Initial calibration was performed; however, a CCV was not performed. Concentration is considered an estimate.

< Denotes compound not detected at or above the LLS.

Table F.5 SUMMA™ Analysis Results for All Target Analytes for ISVS Samples Without HEPA Filtration Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6006-A54.182 (ppbv)	Flag	S6006-A55.208 (ppbv)	Flag	S6006-A56.211 (ppbv)	Flag	S6006-A57.228 (ppbv)	Flag	S6006-A58.323 (ppbv)	Flag	S6006-A59.324 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U		U				
CHLOROMETHANE	74-87-3	12		11		11		10	J	10	J	12		11	J	0.9
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHAN	76-14-2	U		U		U		U		U		U				
METHANOL	67-56-1	1895	Y	2287	Y	2012	Y	1880	Y	2208	Y	1971	Y	2042	Y	168
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U				
BUTANE	106-97-8	2125		2121		2150		1906		1812		1828		1990		159
BROMOMETHANE	74-83-9	U		U		U		U		U		U				
CHLOROETHANE	75-00-3	U		U		U		U		U		U				
ETHANOL	64-17-5	889	Y	1130	Y	952	Y	900	Y	1036	Y	904	Y	969	Y	96
ACETONITRILE	75-05-8	127		122		125		242		124		141		147		47
ACETONE	67-64-1	2133		2193		2004		1817		1849		4610		2434		1076
TRICHLOROFLUOROMETHANE	75-69-4	60		35		57		55		31		61		50		13
PENTANE	109-66-0	1981		2161		2153		754		1916		2076		1840		541
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U				
METHYLENE CHLORIDE	75-09-2	1.4	J	1.3	J	1.5	J	1.3	J	1.3	J	1.2	J	1.3	J	0.1
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U				
PROPANOL	71-23-8	491		467		500		429		500		467		476		27
PROPANENITRILE	107-12-0	5.7	J	4.7	J	5.2	J	4.2	J	5.3	J	4.8	J	5.0	J	0.5
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U				
2-BUTANONE	78-93-3	332		329		359		309		390		370		348		30
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U				
HEXANE	110-54-3	1083		1104		1136		1045		1021		1095		1081		42
CHLOROFORM	67-66-3	U		U		U		U		U		U				
TETRAHYDROFURAN	109-99-9	807		766		809		664		761		688		749		61
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U				
BUTANENITRILE	109-74-0	90		86		91		79		89		95		88		5.4
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U				
1-BUTANOL	71-36-3	21106		17378		21171		16110		18252		18003		18670		2051
BENZENE	71-43-2	20		19		21		17		20		18		19		1.5
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U				
CYCLOHEXANE	110-82-7	141		150		143		125		136		155		142		11
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	488		534		483		436		471		547		493		41
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	147		13	J	12	J	14	J	12	J	12	J	35	J	55
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U				
PENTANENITRILE	110-59-8	7.5	J	U		7.5	J	5.3	J	7.3	J	9.2	J	7.3	J	1.4
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		1.7	J	U		1.7	J	
TOLUENE	108-88-3	37		35		34		33		35		36		35		1.4
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U				
OCTANE	111-65-9	164		152		163		149		151		174		159		9.8

Table F.5 SUMMA™ Analysis Results for All Target Analytes for ISVS Samples Without HEPA Filtration Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6006-A54.182 (ppbv)	Flag	S6006-A55.208 (ppbv)	Flag	S6006-A56.211 (ppbv)	Flag	S6006-A57.228 (ppbv)	Flag	S6006-A58.323 (ppbv)	Flag	S6006-A59.324 (ppbv)	Flag	Mean	Flag	Std. Dev.
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U		U				
CHLOROBENZENE	108-90-7	1.0	J	0.6	J	U		0.9	J	U		U		0.8	J	0.2
HEXANENITRILE	628-73-9	U		1.9	J	2.1	J	U		3.0	J	1.5	J	2.1	J	0.6
ETHYLBENZENE	100-41-4	3.8	J	3.9	J	3.9	J	4.1	J	3.8	J	4.8	J	4.0	J	0.4
P/M-XYLENE	106-42-3	13	J	12	J	13	J	14	J	12	J	16	J	13	J	1.5
CYCLOHEXANONE	108-94-1	12	J	11	J	11	J	11	J	11	J	15		12	J	1.6
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	5.6	J	5.6	J	5.6	J	5.1	J	5.5	J	6.0		5.6	J	0.3
NONANE	111-84-2	77		74		77		68		73		80		75		4.2
1-ETHYL-2-METHYL BENZENE	611-14-3	0.6	J	0.6	J	0.6	J	0.6	J	0.6	J	0.6	J	0.6	J	0.0
1,3,5-TRIMETHYLBENZENE	108-67-8	0.4	J	0.5	J	0.4	J	0.4	J	0.3	J	0.4	J	0.4	J	0.1
1,2,4-TRIMETHYLBENZENE	95-63-6	1.1	J	1.2	J	1.2	J	1.1	J	1.1	J	1.2	J	1.1	J	0.1
DECANE	124-18-5	112		108		116		99		108		121		111		7.6
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		0.3	J	U		U		U		U		0.3	J	
UNDECANE	1120-21-4	221		231		229		220		243		227		229		8.3
1,2,4-TRICHLOROBENZENE	120-82-1	0.8	J	1.0	J	0.8	J	0.5	J	U		1.0	J	0.8	J	0.2
DODECANE	112-40-3	599		746		530		669		680		601		638		76
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U				
TRIDECANE	629-50-5	298		345		275		465		340		404		355		70
TETRADECANE	629-59-4	101		128		106		300		104		316		176		103

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.

Table F.6 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Without HEPA Filtration Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6006-A61.745 (ppbv)	Flag	S6006-A62.754 (ppbv)	Flag	S6006-A63.755 (ppbv)	Flag	S6006-A65.758 (ppbv)	Flag	Mean	Flag	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U				
CHLOROMETHANE	74-87-3	U		U		U		U				
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	U		U		U		U				
METHANOL	67-56-1	738	Y	2994	E,Y	1973	E,Y	2515	E,Y	2055	E,Y	972
VINYL CHLORIDE	75-01-4	U		U		U		U				
BUTANE	106-97-8	1706	E	2346	E	1931	E	2318	E	2075.3	E	311
CHLOROETHANE	75-00-3	U		U		U		U				
ETHANOL	64-17-5	925	Y	2487	E,Y	2342	E,Y	3122	E,Y	2219	E,Y	927
ACETONITRILE	75-05-8	146		258		121		256		195		72
ACETONE	67-64-1	1379		2270	E	1535		2215	E	1850	E	459
TRICHLOROFLUOROMETHANE	75-69-4	17		27		18		35		24		8.5
PENTANE	109-66-0	1431	E	2149	E	1679	E	2130	E	1847	E	352
1,1-DICHLOROETHENE	75-35-4	11		U		U		13		12		1.4
METHYLENE CHLORIDE	75-09-2	580		379		110		6189	E	1815	E	2923
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	1.0	J	1.1	J	U		0.9	J	1.0	J	0.1
PROPANENITRILE	107-12-0	5.8	J	22		7.6	J	15		13	J	7.4
PROPANOL	71-23-8	157		426		366		409		340		124
1,1-DICHLOROETHANE	75-34-3	U		U		U		U				
2-BUTANONE	78-93-3	171		324		200		293		247		73
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U				
HEXANE	110-54-3	1030	E	1310	E	1136	E	1371	E	1212	E	157
CHLOROFORM	67-66-3	U		U		U		U				
TETRAHYDROFURAN	109-99-9	385		753		567		671		594		159
1,2-DICHLOROETHANE	107-06-2	U		U		U		U				
BUTANENITRILE	109-74-0	U		U		U		U				
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U				
1-BUTANOL	71-36-3	7161	E	11015	E	10811	E	10602	E	9897	E	1832
BENZENE	71-43-2	44		54		42		58		50		7.7
CARBON TETRACHLORIDE	56-23-5	U		U		U		U				
CYCLOHEXANE	110-82-7	U		362	Z	356	Z	405	Z	374	Z	27
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U				
TRICHLOROETHENE	79-01-6	12		6.5		U		191		70		105
HEPTANE	142-82-5	1478	E,Z	1274	E,Z	1229	E,Z	1320	E,Z	1325	E,Z	108
4-METHYL-2-PENTANONE	108-10-1	92	Z	48	Z	36	Z	88	Z	66	Z	28
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U				
PYRIDINE	110-86-1	U		15	J	8.7	J	12	J	12	J	3.2
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U				
PENTANENITRILE	110-59-8	U		U		U		U				
1,1,2-TRICHLOROETHANE	79-00-5	83		U		U		U		83		
TOLUENE	108-88-3	563	Z	466	Z	421	Z	570	Z	505	Z	73
1,2-DIBROMOETHANE	106-93-4	U		U		U		U				
OCTANE	111-65-9	86		194		144		209		158		56

Table F.6 Triple Sorbent Trap Analysis Results for All Target Analytes for ISVS Samples Without HEPA Filtration Collected from the Headspace of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6006-A61.745 (ppbv)	Flag	S6006-A62.754 (ppbv)	Flag	S6006-A63.755 (ppbv)	Flag	S6006-A65.758 (ppbv)	Flag	Mean	Flag	Std. Dev.
TETRACHLOROETHYLENE	127-18-4	U		U		U		U				
CHLOROBENZENE	108-90-7	U		U		U		U				
HEXANENITRILE	628-73-9	U		U		U		U				
ETHYLBENZENE	100-41-4	13		13		10		21		14		4.6
P/M-XYLENE	106-42-3	44		49		37		55		46		7.6
CYCLOHEXANONE	108-94-1	U		U		U		U				
STYRENE	100-42-5	8.3		6.4		5.5		19		9.8		6.2
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U				
O-XYLENE	95-47-6	16		19		14		20		17		2.8
NONANE	111-84-2	39		87		62		95		71		25
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		2.3	J	U		2.3	J	
1,3,5-TRIMETHYLBENZENE	108-67-8	2.6	J	2.6	J	1.9	J	2.6	J	2.4	J	0.3
1,2,4-TRIMETHYLBENZENE	95-63-6	8.7		8.3		6.1		8.6		7.9		1.2
DECANE	124-18-5	48		106		73		117		86		31
1,3-DICHLOROBENZENE	541-73-1	U		U		U		U				
1,4-DICHLOROBENZENE	106-46-7	U		U		U		U				
1,2-DICHLOROBENZENE	95-50-1	U		U		U		U				
UNDECANE	1120-21-4	116		272		195		294		219		81
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U				
DODECANE	112-40-3	247		644	E	475		636	E	501	E	186
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U				
TRIDECANE	629-50-5	204		666	E	460	E	580	E	478	E	201
TETRADECANE	629-59-4	91		386	E	294	E	359	E	283	E	133
TBP	126-73-8	< 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).

Z Results affected by adhesive tape contamination.

Y Initial calibration was performed; however, a CCV was not performed. Concentration is considered an estimate.

< Denotes compound not detected at or above the LLS.

Table F.7 SUMMA™ Replicate Analysis Results for All Target Analytes Sampled from the Headspace of Tank BY-108 on 1/23/96

		VSS				ISVS w HEPA				ISVS wo HEPA			
		S6004-A04.050		S6004-A04.050 Rep		S6005-A36.114		S6005-A36.114 Rep		S6006-A58.323		S6006-A58.323 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	9.7	J	11		11	J	11		10	J	10	J
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHAN	76-14-2	U		U		U		U		U		U	
METHANOL	67-56-1	1827	Y	1979	Y	2032	Y	1627	Y	2208	Y	2252	Y
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U	
BUTANE	106-97-8	1937		1916		2319		1849		1812		1852	
BROMOMETHANE	74-83-9	U		U		U		U		U		U	
CHLOROETHANE	75-00-3	U		U		U		U		U		U	
ETHANOL	64-17-5	1900	Y	958	Y	948	Y	750	Y	1036	Y	1048	Y
ACETONITRILE	75-05-8	113		134		114		116		124		118	
ACETONE	67-64-1	4255		1919		2309		2891		1849		1903	
TRICHLOROFLUOROMETHANE	75-69-4	35		61		32		56		31		30	
PENTANE	109-66-0	2034		753		2008		1884		1916		1873	
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		U		U	
METHYLENE CHLORIDE	75-09-2	1.4	J	1.3	J	1.1	J	1.4	J	1.3	J	1.3	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		U	
PROPANOL	71-23-8	411		367		405		460		500		463	
PROPANENITRILE	107-12-0	4.1	J	3.8	J	4.1	J	4.4	J	5.3	J	4.9	J
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U	
2-BUTANONE	78-93-3	304		312		308		292		390		351	
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U	
HEXANE	110-54-3	1048		1027		1109		943		1021		1016	
CHLOROFORM	67-66-3	U		U		U		U		U		U	
TETRAHYDROFURAN	109-99-9	651		622		711		760		761		728	
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U	
BUTANENITRILE	109-74-0	83		90		89		77		89		87	
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U	
1-BUTANOL	71-36-3	17087		19430		16885		17713		18252		18393	
BENZENE	71-43-2	16		16		17		19		20		19	
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U	
CYCLOHEXANE	110-82-7	134		142		132		125		136		128	
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	442		474		467		443		471		453	
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	15	J	10	J	16	J	14	J	12	J	10	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U	
PENTANENITRILE	110-59-8	7.1	J	U		U		5.9	J	7.3	J	7.1	J
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		1.7	J	U	
TOLUENE	108-88-3	25		22		23		24		35		32	
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U	
OCTANE	111-65-9	152		151		141		155		151		140	
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U		U	
CHLOROBENZENE	108-90-7	U		1.0	J	U		U		U		0.8	J

Table F.7 SUMMA™ Replicate Analysis Results for All Target Analytes Sampled from the Headspace of Tank BY-108 on 1/23/96

		VSS				ISVS w HEPA				ISVS wo HEPA			
		S6004-A04.050		S6004-A04.050 Rep		S6005-A36.114		S6005-A36.114 Rep		S6006-A58.323		S6006-A58.323 Rep	
Target Analytes	CAS No.	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
HEXANENITRILE	628-73-9	1.2	J	U		U		2.1	J	3.0	J	U	
ETHYLBENZENE	100-41-4	4.0	J	4.3	J	3.3	J	3.6	J	3.8	J	3.5	J
P/M-XYLENE	106-42-3	14	J	15	J	11	J	12	J	12	J	11	J
CYCLOHEXANONE	108-94-1	12	J	13		11	J	11	J	11	J	9.8	J
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	5.3	J	4.9	J	4.9	J	5.3	J	5.5	J	5.1	J
NONANE	111-84-2	71		67		66		71		73		68	
1-ETHYL-2-METHYL BENZENE	611-14-3	0.7	J	1.0	J	0.6	J	0.5	J	0.6	J	0.5	J
1,3,5-TRIMETHYLBENZENE	108-67-8	0.5	J	1.1	J	0.7	J	0.6	J	0.3	J	0.5	J
1,2,4-TRIMETHYLBENZENE	95-63-6	1.2	J	1.0	J	1.1	J	1.0	J	1.1	J	1.1	J
DECANE	124-18-5	103		111		95		102		108		100	
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	0.3	J	U		U		U		U		U	
UNDECANE	1120-21-4	219		240		199		178		243		221	
1,2,4-TRICHLOROBENZENE	120-82-1	0.8	J	1.0	J	1.0	J	0.7	J	U		U	
DODECANE	112-40-3	662		773		478		391		680		649	
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.7	J	U		0.7	J	U		U		U	
TRIDECANE	629-50-5	570		653		221		165		340		327	
TETRADECANE	629-59-4	643		614		35		37		104		100	

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.

Table F.8 Triple Sorbent Trap Replicate Analysis Results for All Target Analytes Sampled from the Headspace of Tank BY-108 on 1/23/96

		VSS				ISVS w HEPA				ISVS w/o HEPA			
		S6004-A05.707		S6004-A05.707 Rep		S6005-A48.734		S6005-A48.734 Rep		S6006-A65.758		S6006-A65.758 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		30	
CHLOROMETHANE	74-87-3	U		U		U		U		U		U	
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHAN	76-14-2	U		U		U		U		U		U	
METHANOL	67-56-1	2840	E,Y	2640	E,Y	3780	E,Y	3593	E,Y	2515	E,Y	2319	E,Y
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U	
BUTANE	106-97-8	2450	E	2413	E	2335	E	2326	E	2318	E	2353	E
CHLOROETHANE	75-00-3	U		U		U		U		U		U	
ETHANOL	64-17-5	2701	E,Y	2515	E,Y	3144	E,Y	2994	E,Y	3122	E,Y	2752	E,Y
ACETONITRILE	75-05-8	236		244		272		282		256		264	
ACETONE	67-64-1	2068	E	1935	E	2056	E	1950	E	2215	E	2055	E
TRICHLOROFLUOROMETHANE	75-69-4	25		24		34		32		35		36	
PENTANE	109-66-0	2246	E	2184	E	2226	E	2245	E	2130	E	2257	E
1,1-DICHLOROETHENE	75-35-4	U		U		3.9	J	U		13		U	
METHYLENE CHLORIDE	75-09-2	114		115		1841	E	1827	E	6189	E	7842	E
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		1.0	J	0.9	J	U	
PROPANENITRILE	107-12-0	17		17		15		15		15		16	
PROPANOL	71-23-8	464		430		500		462		409		420	
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U	
2-BUTANONE	78-93-3	305		300		402		391		293		302	
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U	
HEXANE	110-54-3	1178	E	1139	E	1213	E	1212	E	1371	E	1475	E
CHLOROFORM	67-66-3	U		U		U		U		U		U	
TETRAHYDROFURAN	109-99-9	794		746		741		699		671		712	
1,2-DICHLOROETHANE	107-06-2	U		U		U		U		U		U	
BUTANENITRILE	109-74-0	9.0		9.0		U		U		U		U	
1,1,1-TRICHLOROETHANE	71-55-6	U		U		U		U		U		U	
1-BUTANOL	71-36-3	14241	E	13922	E	13365	E	13812	E	10602	E	12571	E
BENZENE	71-43-2	24		25		36		37		58		64	
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U	
CYCLOHEXANE	110-82-7	215		210		233		229		405		392	
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U	
TRICHLOROETHENE	79-01-6	1.5	J	0.9	J	32		32		191		189	
HEPTANE	142-82-5	584		568		908	E	902	E	1320	E	1396	E
4-METHYL-2-PENTANONE	108-10-1	45		44		53		53		88		87	
CIS-1,3-DICHLOROPROPENE	10061-01-5	U		U		U		U		U		U	
PYRIDINE	110-86-1	10	J	11	J	9.4	J	9.0	J	12	J	13	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	U		U		U		U		U		U	
PENTANENITRILE	110-59-8	U		1.3	J	U		U		U		U	
1,1,2-TRICHLOROETHANE	79-00-5	U		1.8	J	U		U		U		U	
TOLUENE	108-88-3	31		31		188		187		570		574	
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U	
OCTANE	111-65-9	179		178		172		171		209		210	
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U		U	

Table F.8 Triple Sorbent Trap Replicate Analysis Results for All Target Analytes Sampled from the Headspace of Tank BY-108 on 1/23/96

		VSS				ISVS w HEPA				ISVS w/o HEPA			
		S6004-A05.707		S6004-A05.707 Rep		S6005-A48.734		S6005-A48.734 Rep		S6006-A65.758		S6006-A65.758 Rep	
Target Analytes	CAS No.	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
CHLOROBENZENE	108-90-7	U		U		U		U		U		U	
HEXANENITRILE	628-73-9	U		U		U		U		U		U	
ETHYLBENZENE	100-41-4	4.5		4.5		12		12		21		21	
P/M-XYLENE	106-42-3	15		15		41		40		55		55	
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U	
STYRENE	100-42-5	U		U		3.4	J	3.4	J	19		20	
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U	
O-XYLENE	95-47-6	6.9		6.9		15		15		20		21	
NONANE	111-84-2	85		84		77		76		95		96	
1-ETHYL-2-METHYL BENZENE	611-14-3	U		U		U		U		U		U	
1,3,5-TRIMETHYLBENZENE	108-67-8	1.2	J	1.3	J	2.2	J	2.2	J	2.6	J	2.7	J
1,2,4-TRIMETHYLBENZENE	95-63-6	1.1	J	1.1	J	7.9		8.0		8.6		8.7	
DECANE	124-18-5	105		102		85		85		117		118	
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	271		261		191		191		294		306	
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U	
DODECANE	112-40-3	651	E	635	E	460		461		636	E	693	E
HEXACHLORO-1,3-BUTADIENE	87-68-3	U		U		U		U		U		U	
TRIDECANE	629-50-5	625	E	601	E	432	E	435	E	580	E	631	E
TETRADECANE	629-59-4	362	E	345	E	230		228		359	E	365	E
TBP	126-73-8	< 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 was performed; however, a CCV was not performed. Concentration is considered an estimate.

< Denotes compound not detected at or above the LLS.

Table F.9 SUMMA™ Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank BY-108 on 1/23/96

Target Analytes	CAS No.	S6004-A01.002 VSS Ambient Upwind (ppbv)	Flag	S6004-A02.013 VSS Ambient Thru VSS (ppbv)	Flag	S6005-A33.094 ISVS W/HEPA Bundle A (ppbv)	Flag	S6005-A34.099 ISVS W/HEPA Bundle B (ppbv)	Flag	S6006-A35.161 ISVS WO/HEPA Bundle C (ppbv)	Flag
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-TETRAFLUOROETHANE	76-14-2	U		U		U		U		U	
METHANOL	67-56-1	< 19	Y	< 19	Y	< 19	Y	< 19	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	10	J	U		12	J	49		20	
TRICHLOROFLUOROMETHANE	75-69-4	U		U		U		U		U	
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	1.3	J	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	30		5.3	J	2.4	J	U		2.3	J
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	1.7	J	2.1	J	1.7	J	2.9	J	2.6	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		U		U		U	
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		16	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 BY-108 on 1/23/96

Target Analytes	CAS No.	S6004-A01.002 VSS Ambient Upwind (ppbv)	Flag	S6004-A02.013 VSS Ambient Thru VSS (ppbv)	Flag	S6005-A33.094 ISVS W/HEPA Bundle A (ppbv)	Flag	S6005-A34.099 ISVS W/HEPA Bundle B (ppbv)	Flag	S6006-A35.161 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	U		U		0.9	J	U		0.8	J
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	0.4	J	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		U		U		U	
TETRADECANE	629-59-4	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.

< Denotes compound not detected at or above the LLS.

Table F.10 Triple Sorbent Trap Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank BY-108 on 1/23/96.

Target Analytes	CAS No.	S6004-A11.716 VSS FB #1 (ppbv)	Flag	S6004-A12.718 VSS FB #2 (ppbv)	Flag	S6004-A13.725 TB #1 (ppbv)	Flag	S6004-A14.727 TB #2 (ppbv)	Flag	S6005-A77.742 ISVS w/HEPA FB #3 (ppbv)	Flag	S6005-A81.743 ISVS w/HEPA FB #4 (ppbv)	Flag	S6006-A83.760 ISVS wo/HEPA FB #5 (ppbv)	Flag	S6006-A84.761 ISVS wo/HEPA FB #6 (ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	U		U		U		U		U		36		10		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	290	Y	347	Y	504	Y	508	Y
VINYL CHLORIDE	75-01-4	U		U		U		U		U		U		U		U	
BUTANE	106-97-8	U		U		U		U		135		210		148		162	
CHLOROETHANE	75-00-3	U		U		U		U		U		U		U		U	
ETHANOL	64-17-5	< 133	Y	< 133	Y	< 133	Y	< 133	Y	< 133	Y	173	Y	366	Y	391	Y
ACETONITRILE	75-05-8	U		U		U		U		4.1	J	4.6	J	5.7	J	6.2	J
ACETONE	67-64-1	9.4	J	11	J	15	J	16	J	140		192		250		272	
TRICHLOROFLUOROMETHANE	75-69-4	U		U		U		U		4.1	J	25		8.1		3.5	J
PENTANE	109-66-0	U		U		U		U		163		154		206		240	
1,1-DICHLOROETHENE	75-35-4	U		U		U		U		2.8	J	68		18		8.6	
METHYLENE CHLORIDE	75-09-2	43		14	J	27	J	157		536		5483	E	3973	E	941	E
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	U		U		U		U		U		1.5	J	U		0.6	J
PROPANENITRILE	107-12-0	U		U		U		U		U		U		U		U	
PROPANOL	71-23-8	U		U		U		U		15	J	15	J	20	J	24	
1,1-DICHLOROETHANE	75-34-3	U		U		U		U		U		U		U		U	
2-BUTANONE	78-93-3	U		U		U		U		23		32		34		36	
CIS-1,2-DICHLOROETHENE	156-59-2	U		U		U		U		U		U		U		U	
HEXANE	110-54-3	U		U		U		U		133		149		442		531	
CHLOROFORM	67-66-3	U		U		U		U		U		U		U		U	
TETRAHYDROFURAN	109-99-9	U		U		U		U		35		26		38		46	
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		2.7	J	U		U	
1-BUTANOL	71-36-3	3.0	J	2.5	J	U		U		888		927		1534	E	2075	E
BENZENE	71-43-2	U		U		U		5.6		13		22		29		32	
CARBON TETRACHLORIDE	56-23-5	U		U		U		U		U		U		U		U	
CYCLOHEXANE	110-82-7	U		U		U		U		34		85		230		220	
1,2-DICHLOROPROPANE	78-87-5	U		U		U		U		U		U		U		U	
TRICHLOROETHENE	79-01-6	U		U		U		U		6.4		52		49		15	
HEPTANE	142-82-5	U		U		U		U		215		367		832	E	1026	E
4-METHYL-2-PENTANONE	108-10-1	U		U		U		U		6.3		21		47		58	
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		36	J	37	J
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		10		12	
1,1,2-TRICHLOROETHANE	79-00-5	U		U		U		U		5.3		U		36		48	
TOLUENE	108-88-3	U		U		U		0.8	J	81		152		391		485	
1,2-DIBROMOETHANE	106-93-4	U		U		U		U		U		U		U		U	
OCTANE	111-65-9	U		U		U		U		8.1		3.0	J	15		26	
TETRACHLOROETHYLENE	127-18-4	U		U		U		U		U		U		U		U	
CHLOROBENZENE	108-90-7	U		U		U		U		U		U		U		U	
HEXANENITRILE	628-73-9	U		U		U		U		U		U		U		U	
ETHYLBENZENE	100-41-4	U		U		U		U		5.6		7.8		8.5		9.7	
P/M-XYLENE	106-42-3	U		U		U		U		21		24		28		35	
CYCLOHEXANONE	108-94-1	U		U		U		U		U		U		U		U	
STYRENE	100-42-5	U		U		U		U		1.4	J	3.9		6.7		6.6	
1,1,2,2-TETRACHLOROETHANE	79-34-5	U		U		U		U		U		U		U		U	

Table F.10 Triple Sorbent Trap Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank BY-108 on 1/23/96.

Target Analytes	CAS No.	S6004-A11.716 VSS FB #1 (ppbv)	Flag	S6004-A12.718 VSS FB #2 (ppbv)	Flag	S6004-A13.725 TB #1 (ppbv)	Flag	S6004-A14.727 TB #2 (ppbv)	Flag	S6005-A77.742 ISVS w/HEPA FB #3 (ppbv)	Flag	S6005-A81.743 ISVS w/HEPA FB #4 (ppbv)	Flag	S6006-A83.760 ISVS wo/HEPA FB #5 (ppbv)	Flag	S6006-A84.761 ISVS wo/HEPA FB #6 (ppbv)	Flag
O-XYLENE	95-47-6	U		U		U		U		6.9		7.9		8.9		11	
NONANE	111-84-2	U		U		U		U		2.2	J	0.9	J	8.4		12	
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		1.3	J	1.3	J	1.5	J	2.1	J
1,2,4-TRIMETHYLBENZENE	95-63-6	U		U		U		U		5.2		4.8		5.3		7.0	
DECANE	124-18-5	U		U		U		U		2.1	J	0.9	J	10		14	
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		2.9	J	0.7	J	19		28	
1,2,4-TRICHLOROBENZENE	120-82-1	U		U		U		U		U		U		U		U	
DODECANE	112-40-3	U		U		U		U		3.9	J	U		44		65	
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		41	J	61	J
TETRADECANE	629-59-4	U		U		U		U		U		U		38		46	
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.

Y Initial calibration was performed; however, a CCV was not performed. Concentration is considered an estimate.

< 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 100017

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-BY-108 Tank Vapor Sample SAF S6004
(VSS Truck)

Collection date: 01 - 23 - 96
Preparation date: 01 - 08 - 96

Field Logbook No. WHC-H-647-10

Ice Chest No.

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		
S6004 - A18 . 46S	Collect NH_3/H_2O Sorbent Trap #1	Sorbent line 3
S6004 - A19 . 47S	Collect NH_3/H_2O Sorbent Trap #2	Sorbent line 4
S6004 - A20 . 48S	Collect NH_3/H_2O Sorbent Trap #3	Sorbent line 5
S6004 - A21 . 49S	Collect NH_3/H_2O Sorbent Trap #4	Sorbent line 6
S6004 - A22 . 50S	Collect NH_3/H_2O Sorbent Trap #5	Sorbent line 7
S6004 - A23 . 51S	Collect NH_3/H_2O Sorbent Trap #6	Sorbent line 8
S6004 - A24 . 52S	Open, close & store NH_3/H_2O field blank #1	N/A
S6004 - A25 . 53S	Open, close & store NH_3/H_2O field blank #2	N/A
S6004 - A26 . 54S	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 GW Dennis	01-10-96	1000	J A Edwards JA Edwards	01-10-96	1000
J A Edwards JA Edwards	1-11-96	1315	T. B. Utesh / T B Utesh	1-11-96	1315
T. B. Utesh / T B Utesh	1-30-96	1430	JAEDWARDS / JA Edwards	1-30-96	1430
JAEDWARDS / JA Edwards	1-31-96	0900	G.W. Dennis / GW Dennis	1-31-96	0900

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 (JE)	POC (JE)	

(Revised 11/30/95 PNNL)

A-6000-407 (12/92) WEF061

Battelle Pacific
National Northwest Lab

CHAIN OF CUSTODY

WHC 100020

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-BY-108 Tank Vapor Sample SAF S6005
With HEPA filters *(ISVS Cart)*
Ice Chest No.

Collection date: 01 - 23 - 96
Preparation date: 01 - 08 - 96

Field Logbook No. WHC-H-642-8

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

S6005 - A42 . 55S	Collect NH_3/H_2O Sorbent Trap #7	A4
S6005 - A43 . 56S	Collect NH_3/H_2O Sorbent Trap #8	A5
S6005 - A44 . 57S	Collect NH_3/H_2O Sorbent Trap #9	A6
S6005 - A51 . 58S	Collect NH_3/H_2O Sorbent Trap #10	B4
S6005 - A52 . 59S	Collect NH_3/H_2O Sorbent Trap #11	B5
S6005 - A53 . 60S	Collect NH_3/H_2O Sorbent Trap #12	B6
S6005 - A78 . 61S	Bundle A Store NH_3/H_2O field blank #4	A8
S6005 - A82 . 62S	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 GWD	01-10-96	1000	J A Edwards JAEdwards	01-10-96	1000
J A Edwards JAEdwards	1-11-96	1315	T. B Utecht / T. B Utecht	1-11-96	1315
T. B. Utecht / T. B Utecht	1-30-96	1430	JAEDWARDS / JAEdwards	1-30-96	1430
JAEDWARDS / JAEdwards	1-31-96	0900	G.W. Dennis / GWD	1-31-96	0900

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 (RZ) POC (RZ)

Comments:
S6005-A51BS - Flow was started before totalizer was zeroed. Once noticed, flow was stopped and totalizer zeroed and sorbent re-started. Estimated flow is 274 sccm for 15 seconds. Sample volume should be considered to be 2058 ± 20 sccm.
Rick Mahon 29JAN96

(Revised 11/30/95 PNNL)

A-6000-407 (12/92) WEF061

1 of 1

Battelle Pacific
National Northwest Lab

CHAIN OF CUSTODY

WHC 100023

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-BY-108 Tank · Vapor Sample SAF S6006
Without HEPA filters (ISVS Cart)
Ice Chest No.

Collection date 01 - 23 - 96
Preparation date 01 - 08 - 96

Field Logbook No. WHC-N-648

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

S6006 - A66 . 63S	Collect NH_3/H_2O Sorbent Trap #13	C7
S6006 - A67 . 64S	Collect NH_3/H_2O Sorbent Trap #14	C8
S6006 - A68 . 65S	Collect NH_3/H_2O Sorbent Trap #15	C9
S6006 - A69 . 66S	Collect NH_3/H_2O Sorbent Trap #16	C10
S6006 - A70 . 67S	Collect NH_3/H_2O Sorbent Trap #17	C11
S6006 - A71 . 68S	Collect NH_3/H_2O Sorbent Trap #18	C12
S6006 - A85 . 69S	Bundle C Store NH_3/H_2O field blank #6	C16
S6006 - A86 . 70S	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 G.W.	01-10-96	1000	J A Edwards J A Edwards	01-10-96	1000
J A Edwards J A Edwards	1-11-96	1315	T R Utech / T R Utech	1-11-96	1315
T R Utech / T R Utech	1-30-96	1430	J A Edwards / J A Edwards	1-30-96	1436
J A Edwards / J A Edwards	1-31-96	0900	G W Dennis / G.W.	1-31-96	0900

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 (VS)	POC (VS)	

(Revised 11/30/95 PNNL)

A-6000-407 (12/92) WEF061

Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100015

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-BY-108 Tank Vapor Sample SAF S6004
(VSS Truck)
Collection date 01 - 23 - 96
Preparation date 01 - 10 - 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

S6004 - A01 . 002	Collect Ambient Air Sample SUMMA #1	Upwind of Tank
S6004 - A02 . 013	Collect Ambient Air Sample SUMMA #2	SUMMA Port 15
S6004 - A03 . 029	Collect SUMMA #3	SUMMA Port 13
S6004 - A04 . 050	Collect SUMMA #4	SUMMA Port 15
S6004 - A15 . 054	Collect SUMMA #5	SUMMA Port 13
S6004 - A16 . 055	Collect SUMMA #6	SUMMA Port 15
S6004 - A27 . 067	Collect SUMMA #7	SUMMA Port 13
S6004 - A28 . 080	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 / J A Edwards	01-11-96	1325	T. B Utelt / T. Butelt	01-11-96	1325
T. B. Utelt / T. Butelt	1-30-96	1415	J A Edwards / J A Edwards	1-30-96	1415

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 (LE) POC (LE)

(Revised 11/30/95 PNNL)

A-6000-407 (12/92) WEF061

1 of 1

Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100018

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-BY-108 Tank Vapor Sample SAF S6005
With HEPA filters *(ISVS Cart)*
Ice Chest No.

Collection date 01 - 29 - 96
Preparation date 01 - 10 - 96

Field Logbook No: WHC-N-6478

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

S6005 - A33 . 094	Collect Ambient Air Sample SUMMA #9	AS
S6005 - A34 . 099	Collect Ambient Air Sample SUMMA #10	BS
S6005 - A36 . 114	Collect SUMMA #12	AS
S6005 - A37 . 116	Collect SUMMA #13	AS
S6005 - A38 . 138	Collect SUMMA #14	AS
S6005 - A45 . 143	Collect SUMMA #15	BS
S6005 - A46 . 145	Collect SUMMA #16	BS
S6005 - A47 . 154	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	01-11-96	1325	T. B Utecht/T. B Utecht	01-11-96	1325
T. B Utecht/T. B Utecht	1-30-96	1415	J A Edwards J A Edwards	1-30-96	1415

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 [signature] POC [signature]

(Revised 11/30/95 PNNL)

A-6000-407 (12/92) WEF061

Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100021

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 85-3656 / FAX 373-3793

Project Designation/Sampling Locations: 200 West Tank Farm
241-BY-108 Tank Vapor Sample SAF S6006
Without HEPA filters (ISVS Cart)

Collection date: 01 - 23 - 96
Preparation date: 01 - 10 - 96

Ice Chest No.

Field Logbook No. WHC-N-6478

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

S6006 - A35 . 161	Collect Ambient Air Sample SUMMA #11	CS
S6006 - A54 . 182	Collect SUMMA #18	CS
S6006 - A55 . 208	Collect SUMMA #19	CS
S6006 - A56 . 211	Collect SUMMA #20	CS
S6006 - A57 . 228	Collect SUMMA #21	CS
S6006 - A58 . 323	Collect SUMMA #22	CS
S6006 - A59 . 324	Collect SUMMA #23	CS

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

Relinquished By	Date	Time	Received By	Date	Time
J A Edwards JAEdwards	01-11-96	1325	T.B.Utelt/T.B.Utelt	01-11-96	1325
T. B. Utelt/T.B.Utelt	1-30-96	1415	JAEDWARDS/JA.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	POC

Comments:
S6006-A35.161 → The 1/4" swagelok nut on SUMMA valve turned while trying to mount SUMMA. This is the nut that held valve onto SUMMA. The nut was tightened before sampling.

S6006-A55.208 → While removing brass swagelok plug on SUMMA a slight hiss was heard. The SUMMA valve was checked and found closed.
Rick Mahon 29JAN96

(Revised 11/30/95 PNNL)

A-6000-407 (12/92) WEF061

Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100016

Custody Form Initiator: J. A. Edwards - PNL
Telephone (509) 373-0141
Page 85-3009 / P8-08 / FAX 376-0418

Company Contact: R. D. Mahon - WHC
Telephone (509) 373-7437
Page 85-9656 / S3-27 / FAX 373-7076

Project Designation/Sampling Locations: 200 West Tank Farm
241-BY-108 Tank Vapor Sample SAF S6004 (VSS Truck)
Collection date 01 - 23 - 96
Preparation date 01 - 08 - 96

Ice Chest No.
Field Logbook No. WHC-N-607

Erteo Hi/Lo thermometer No. PNL-T-006

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

Method of Shipment: Government Truck

Shipped to: WHC

Possible Sample Hazards/Remarks: Unknown at time of sampling

Sample Identification

S6004 - A05 . 707	Collect TST Sample # 1	Sorbent Line 3
S6004 - A06 . 708	Collect TST Sample # 2	Sorbent Line 4
S6004 - A07 . 709	Collect TST Sample # 3	Sorbent Line 5
S6004 - A08 . 710	Collect TST Sample # 4	Sorbent Line 6
S6004 - A09 . 711	Collect TST Sample # 5	Sorbent Line 7
S6004 - A10 . 715	Collect TST Sample # 6	Sorbent Line 8
S6004 - A11 . 716	Open, close & store TST Field Blank # 1	In VSS truck
S6004 - A12 . 718	Open, close & store TST Field Blank # 2	In VSS truck
S6004 - A13 . 725	Store TST Trip Blank # 1	N/A
S6004 - A14 . 727	Store TST Trip Blank # 2	N/A

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

Relinquished By	Date	Time	Received By	Date	Time
J L Julya	01-10-96	0825	J A Edwards	01-10-96	0825
J A Edwards	01-10-96	1320	T. B Ulteli / T. B Ulteli	01-10-96	1320
T. B Ulteli / T. B Ulteli	1-30-96	1425	J A Edwards / J A Edwards	1-30-96	1425
J A Edwards / J A Edwards	2-5-96	0850	Janet L Julya / Janet L Julya	2-5-96	0850

Final Sample Disposition

Comments:

PNL (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
◊ Sorbents shipped on ice? (<5°C)	(Y)/N	/ (Y)/N
◊ Hi/Lo thermometer - *Keep upright!*	(Y)/N	
◊ Hi/Lo thermometer		/ (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	(JE)	POC (JE)

Comments:

Cooler Temperature Status
Hi -16 °C / Lo -16 °C (pick up at PNL to WHC)
Hi ___ °C / Lo ___ °C (delivery at WHC from PNL)
Hi ___ °C / Lo ___ °C (at return to PNL from WHC)
Hi +6 °C / Lo 0 °C (at delivery from WHC to PNL)

(Revised 06/21/95 PNL)

A-6000-407 (12/92) WEF061

Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100019

Custody Form Initiator: J. A. Edwards - PNL
Telephone (509) 373-0141
Page 85-3009 / P8-08 / FAX 376-0418

Company Contact: R. D. Mahon - WHC
Telephone (509) 373-7437
Page 85-9656 / S3-27 / FAX 373-7076

Project Designation/Sampling Locations: 200 West Tank Farm
241-BY-108 Tank Vapor Sample SAF S6005
With HEPA filters (ISVS Cart)
Ice Chest No.

Collection date 01 - 23 - 96
Preparation date 01 - 08 - 96

Field Logbook No. WHC-H-647-8

Erteo Hi/Lo thermometer No. PNL-T-006

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment: Government Truck

Shipped to: WHC

Possible Sample Hazards/Remarks: Unknown at time of sampling

Sample Identification

S6005 - A39.731 - 1	Collect TST Sample # 7	A1
S6005 - A40.732 - 1	Collect TST Sample # 8	A2
S6005 - A41.733 - 1	Collect TST Sample # 9	A3
S6005 - A48.734 - 1	Collect TST Sample # 10	B1
S6005 - A49.738 - 1	Collect TST Sample # 11	B2
S6005 - A50.741 - 1	Collect TST Sample # 12	B3
S6005 - A77.742 - 1	Bundle A Store TST Field Blank # 3	A7
S6005 - A81.743 - 1	Bundle B Store TST Field Blank # 4	B7

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

Relinquished By	Date	Time	Received By	Date	Time
J L Julya Janet Julya	01-10-96	0825	J A Edwards J A Edwards	01-10-96	0825
J A Edwards J A Edwards	01-10-96	1320	V.B. Ukelt/T. B Ukelt 1-11-96	01-10-96	1320
T. B Ukelt/T. B Ukelt	1-30-96	1925	J A Edwards/J A Edwards	1-30-96	1925
J A Edwards/J A Edwards	2-5-96	0850	Janet L Julya/Janet L Julya	2-5-96	0850

Final Sample Disposition

Comments:

	PNL (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
◊	Sorbents shipped on ice? (<5°C)	(Y)N	/ (Y)N
◊	Hi/Lo thermometer - *Keep upright!*	(Y)N	
◊	Hi/Lo thermometer		/ (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:

Cooler Temperature Status
Hi -16 °C / Lo -16 °C (pick up at PNL to WHC)
Hi ____ °C / Lo ____ °C (delivery at WHC from PNL)
Hi ____ °C / Lo ____ °C (at return to PNL from WHC)
Hi +6 °C / Lo 0 °C (at delivery from WHC to PNL)

(Revised 06/21/95 PNL)

Battelle Pacific Northwest Lab	CHAIN OF CUSTODY	WHC 100022

Custody Form Initiator: J. A. Edwards - PNL
Telephone (509) 373-0141
Page 85-3009 / P8-08 / FAX 376-0418

Company Contact: R. D. Mahon - WHC
Telephone (509) 373-7437
Page 85-9656 / S3-27 / FAX 373-7076

Project Designation/Sampling Locations: 200 West Tank Farm
241-BY-108 Tank Vapor Sample SAF S6006
Without HEPA filters (ISVS Cart)
Collection date 01 - 23 - 96
Preparation date 01 - 08 - 96

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

Ertco Hi/Lo thermometer No. PNL-T-006

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

Method of Shipment: Government Truck

Shipped to: WHC

Possible Sample Hazards/Remarks: Unknown at time of sampling

Sample Identification

S6006 - A60.744 .1	Collect TST Sample # 13	C1
S6006 - A61.745 .1	Collect TST Sample # 14	C2
S6006 - A62.754 .1	Collect TST Sample # 15	C3
S6006 - A63.755 .1	Collect TST Sample # 16	C4
~~S6006 - A64.756 .1~~	~~Collect TST Sample # 17~~	~~C5~~
S6006 - A65.758 .1	Collect TST Sample # 18	C6
S6006 - A83.760 .1	Bundle C Store TST Field Blank # 5	C14
S6006 - A84.761 .1	Bundle C Store TST Field Blank # 6	C15

RECEIVED EMPTY TUBE (JE) 2-5-96

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

Relinquished By	Date	Time	Received By	Date	Time
J L Julya / J L Julya	01-10-96	0825	J A Edwards / J A Edwards (JE)	01-10-96	0825
J A Edwards / J A Edwards 1-11-96	01-10-96 (JE)	1320	T B Utesch / T B Utesch 1-11-96	01-10-96 (JE)	1320
T B Utesch / T B Utesch	1-30-96	1425	J A Edwards / J A Edwards	1-30-96	1425
J A Edwards / J A Edwards	2-5-96	0850	Janet L Julya / Janet L Julya	2-5-96	0850

Final Sample Disposition

Comments:

PNL (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
◊ Sorbents shipped on ice? (<5°C)	(Y) N	/ (Y) N
◊ Hi/Lo thermometer - *Keep upright!*	(Y) N	
◊ Hi/Lo thermometer		/ (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	(JE)	POC (JE)

Comments:
S6006-A64.756 → Broken during disassembly of bundle C in farm and disposed of. Rick Mahon 29JAN96

Cooler Temperature Status
Hi -16 °C / Lo -16 °C (pick up at PNL to WHC)
Hi ___ °C / Lo ___ °C (delivery at WHC from PNL)
Hi ___ °C / Lo ___ °C (at return to PNL from WHC)
Hi +6 °C / Lo 0 °C (at delivery from WHC to PNL)

(Revised 06/21/95 PNL)

A-6000-407 (12/92) WEF061

PNNL

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

Lockheed

Larry Pennington	S7-21
Luther Buckley	R2-12

DOE-RL

Carol Babel	S7-54
Jim Thompson	S7-54