PNL-10468
UC-606

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January 1995

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 Laboratory
Operated for the U.S. Department of Energy
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Pacific Northwest Laboratory
Richland, Washington 99352

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Summary

This report describes inorganic and organic analyses results from samples obtained from the headspace of the Hanford waste storage Tank 241-BY-107 (referred to as Tank BY-107). The results described here were obtained to support safety and toxicological evaluations. A summary of the results for inorganic and organic analytes is listed in Table 1. Detailed descriptions of the results appear in the text.

Quantitative results were obtained for the inorganic compounds ammonia (NH_3), nitrogen dioxide (NO_2), nitric oxide (NO), and water (H_2O). Sampling for hydrogen cyanide (HCN) and sulfur oxides (SO_X) was not requested. Organic compounds were also quantitatively determined. Sixty eight organic tentatively identified compounds (TICs) were observed above the detection limit of (ca.) 10 ppbv, but standards for most of these were not available at the time of analysis, and the reported concentrations are semiquantitative estimates. In addition, we looked for the 40 TO-14 plus an additional 14 analytes. Of these, 3 were observed above the 5-ppbv detection limit. The 10 organic analytes with the highest estimated concentrations are listed in Table 1 and account for approximately 57% of the total organic components in Tank BY-107. Two permanent gases, carbon dioxide (CO_2) and nitrous oxide (N_2O), were also detected.

Table 1. Summary Results of Inorganic and Organic Samples Collected from the Headspace of Tank BY-107 on 10/26/94

Category	Analyte	Vapor[(a)] Concentration	Units
Inorganic	NH_3	972 ± 6	ppmv
	NO_2	≤ 0.02	ppmv
	NO	0.13 ± 0.03	ppmv
	H_2O	15 ± 1	mg/L
Organic	Acetone	16.71	mg/m^3
	1-Butanol	5.33	mg/m^3
	Propanol	4.71	mg/m^3
	Tridecane	3.96	mg/m^3
	2-Butanone	3.56	mg/m^3
	Hexane	3.02	mg/m^3
	Dodecane	2.95	mg/m^3
	Unknown Alkane	2.74	mg/m^3
	Unknown C_{15} Alkane	2.39	mg/m^3
	Tetradecane	2.24	mg/m^3

(a) Vapor concentrations were determined using sample-volume data provided by Westinghouse Hanford Company and are based on averaged data.

Acknowledgments

The authors gratefully acknowledge the support of other project staff at Pacific Northwest Laboratory who contributed to the successful completion of this sampling and analysis activity. Jeff Edwards served as the PNL single-point-of-contact and coordinated sample handling and communications with Westinghouse Hanford Company. K. B. Olsen assisted in preparing the organic portion of thiis report. Sally Slate, May-Lin Thomas and Karen Schielke analyzed inorganic samples, and Gary Dennis prepared the solid-sorbent sample trains. Bonnie L. Lehrman provided word processing support.

Abbreviations

CAS	Chemical Abstracts Service
COC	chain of custody
C_v	concentration by volume
DIW	deionized water
emf	electromotive force
EPA	U.S. Environmental Protection Agency
GC/MS	gas chromatography/mass spectrometry
GC/TCD	gas chromatography/thermal conductivity detection
HP	Hewlett Packard
IC	ion chromatography
IL	impact level
IS	internal standard
MDL	minimum detection limit
NIST	National Institute for Standards and Technology
NPH	normal paraffin hydrocarbon
OGI	Oregon Graduate Institute
OSHA	Occupational Safety and Health Administration
PFA	perfluoroalkoxy
PNL	Pacific Northwest Laboratory
ppbv	part per billion by volume
ppmv	part per million by volume
QA	quality assurance
REL	recommended exposure limit
SCIC	suppressed-conductivity ion chromatography
SIE	selective ion electrode
SRM	standard reference material
STP	standard temperature and pressure
TEA	triethanolamine
TIC	tentatively identified compound
VSS	vapor sampling system
WHC	Westinghouse Hanford Company

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1.0 Introduction

This report describes results of the analyses of tank-headspace samples taken from the Hanford waste Tank 241-BY-107 (referred to as Tank BY-107). Pacific Northwest Laboratory (PNL)[a] contracted with Westinghouse Hanford Company (WHC) to provide sampling devices and to analyze inorganic and organic analytes collected from the tank headspace and ambient air near the tank. The organic analytes for TO-14 compounds were extended to include 15 analytes identified by the Toxicological Review Panel for Tank C-103 and reported in *Toxicological Evaluation of Analytes from Tank 241-C-103 PNL-10189* (Mahlum et al. 1994). While these analytes (shown in Table 3.1) are only of toxicological concern for Tank C-103, program management included these analytes for future tank analyses as identified in the fiscal year work plan. This plan is attached to a letter dated 9/30/94 and addressed to Mr T. J. Kelly of WHC. The plan also requires PNL to analyze for the permanent gases as shown in Table 3.5. The sample job was designated S4077, and samples were collected by WHC on October 26, 1994, using the vapor sampling system (VSS).

Sampling devices, including six sorbent trains (for inorganic analyses), and six SUMMA™ canisters (for organic analyses) were supplied to the WHC sampling staff on October 24. Samples were taken (by WHC) from the tank headspace on October 26 and were returned to PNL from the field on November 8. Inorganic (sorbent trap) samples were delivered to PNL on chain of custody (COC) 008071 (see Figure 1.1a). The SUMMA™ canisters were delivered on COC 008070 (see Figure 1.1b). Three SUMMA™ canister samples were stored at the PNL 326/23B laboratory pending further instruction from WHC to send them to the Oregon Graduate Institute (OGI) for analysis.

The samples were inspected upon delivery to the 326/23B laboratory and logged into PNL record book 55408 as described in PNL Technical Procedure PNL-TVP-07[b]. Custody of the sorbent traps was transferred to PNL personnel performing the inorganic analysis and stored at refrigerated ($\leq$ 10°C) temperature until the time of analysis. The canister was stored in the 326/23B laboratory at ambient (25°C) temperature until time of analysis. Access to the 326/23B laboratory is limited to PNL personnel working on the waste-tank safety program. Analyses described in this report were performed at PNL in the 300 area of the Hanford Reservation. Analytical methods that were used are described in the text. In summary, sorbent traps for inorganic analyses containing sample materials were either weighed (for water analysis) or weighed and desorbed with the appropriate aqueous solutions {for ammonia (NH_3), nitrogen dioxide (NO_2), and nitric oxide (NO) analyses}. The aqueous extracts were analyzed by either selective electrode or ion chromatography (IC). Organic analyses were performed using cryogenic preconcentration followed by gas chromatography/mass spectrometry (GC/MS).

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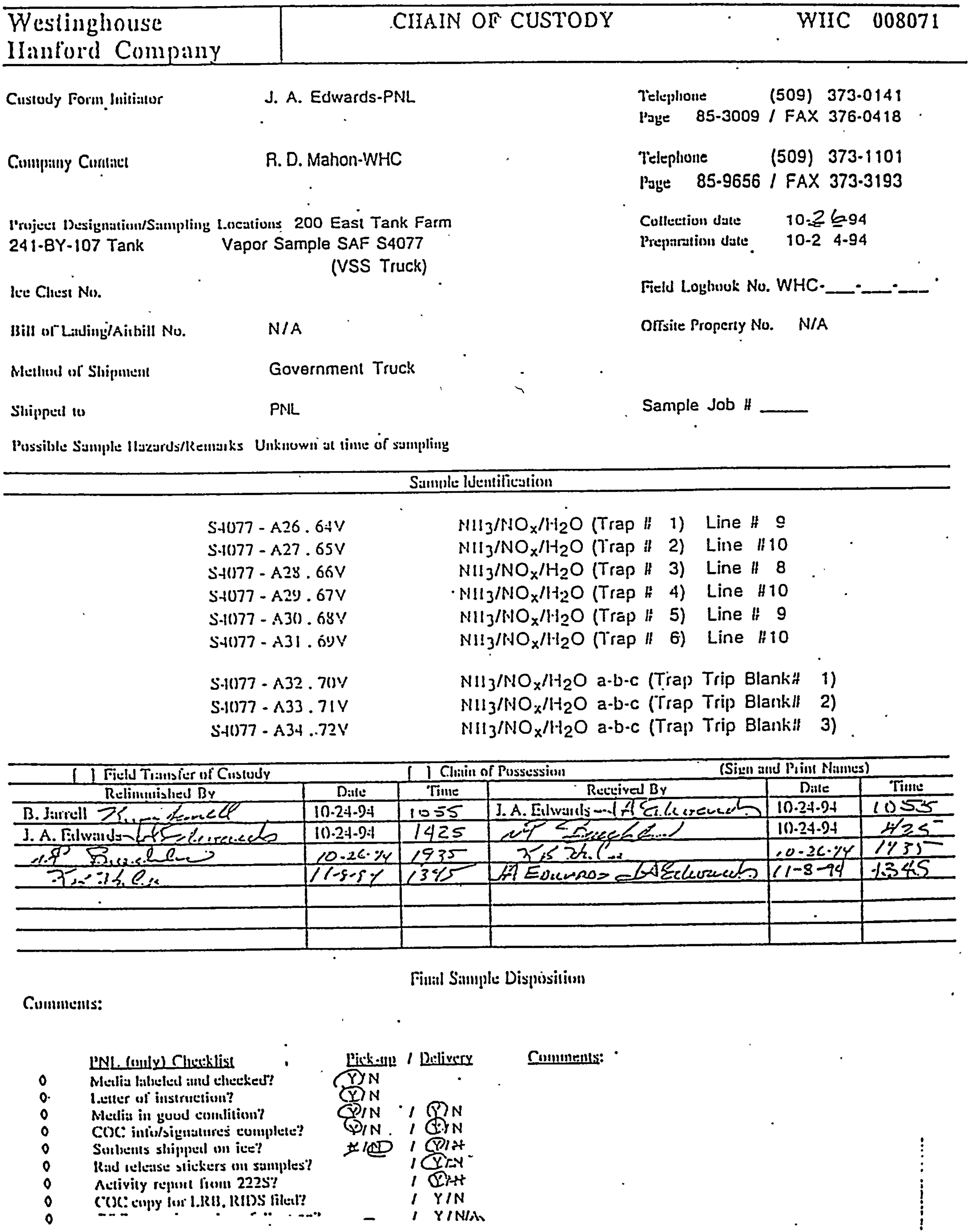
[a] Pacific Northwest Laboratory is operated for the U. S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

[b] PNL-TVP-07, Rev. 0, October 1994, *Sample Shipping and Receiving Procedure for PNL Waste Tank Samples*, PNL-Technical Procedure, Tank Vapor Project, Richland, Washington.



Westinghouse Hanford Company	CHAIN OF CUSTODY	WHC 008071

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

Company Contact R. D. Mahon-WHC
Telephone (509) 373-1101
Page 85-9656 / FAX 373-3193

Project Designation/Sampling Locations 200 East Tank Farm
241-BY-107 Tank Vapor Sample SAF S4077
(VSS Truck)

Collection date 10-26-94
Preparation date 10-24-94

Ice Chest No.

Field Logbook No. WHC-___-___-___

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNL

Sample Job # _____

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S4077 - A26 . 64V	$NH_3/NO_x/H_2O$ (Trap # 1) Line # 9
S4077 - A27 . 65V	$NH_3/NO_x/H_2O$ (Trap # 2) Line #10
S4077 - A28 . 66V	$NH_3/NO_x/H_2O$ (Trap # 3) Line # 8
S4077 - A29 . 67V	$NH_3/NO_x/H_2O$ (Trap # 4) Line #10
S4077 - A30 . 68V	$NH_3/NO_x/H_2O$ (Trap # 5) Line # 9
S4077 - A31 . 69V	$NH_3/NO_x/H_2O$ (Trap # 6) Line #10
S4077 - A32 . 70V	$NH_3/NO_x/H_2O$ a-b-c (Trap Trip Blank# 1)
S4077 - A33 . 71V	$NH_3/NO_x/H_2O$ a-b-c (Trap Trip Blank# 2)
S4077 - A34 . 72V	$NH_3/NO_x/H_2O$ a-b-c (Trap Trip Blank# 3)

☐ Field Transfer of Custody ☐ Chain of Possession (Sign and Print Names)

Relinquished By	Date	Time	Received By	Date	Time
B. Jarrell	10-24-94	1055	J. A. Edwards	10-24-94	1055
J. A. Edwards	10-24-94	1425		10-24-94	1425
	10-26-94	1935		10-26-94	1935
	11-8-94	1345	J A EDWARDS	11-8-94	1345

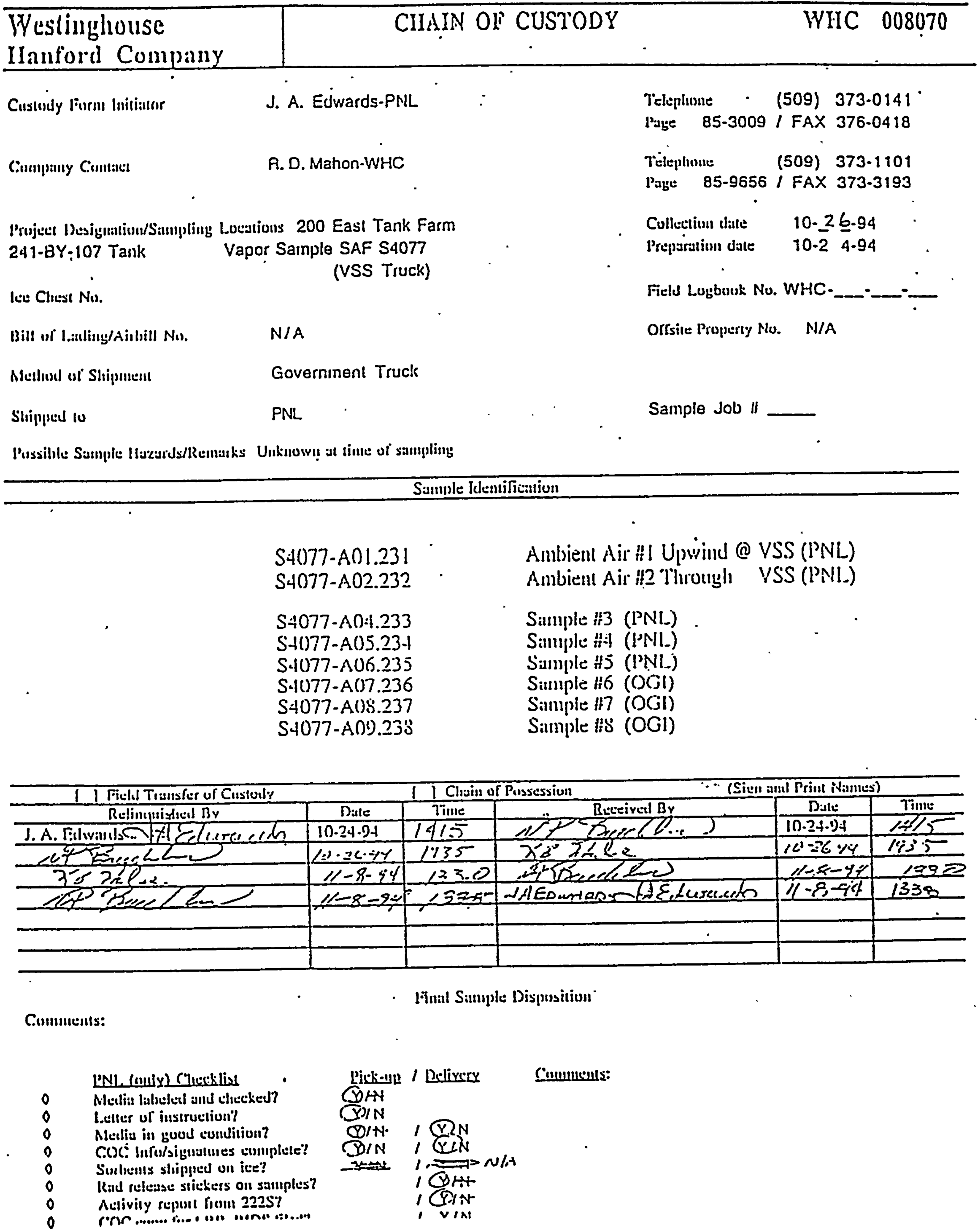
Final Sample Disposition

Comments:

PNL (only) Checklist	Pick-up / Delivery	Comments:
0 Media labeled and checked?	[✓ Y] / N	
0 Letter of instruction?	[✓ Y] / N	
0 Media in good condition?	[✓ Y] / N / [✓ Y] / N	
0 COC info/signatures complete?	[✓ Y] / N / [✓ Y] / N	
0 Sorbents shipped on ice?	~~Y~~ / [✓ N] / [✓ Y] / ~~N~~	
0 Rad release stickers on samples?	/ [✓ Y] / ~~N~~	
0 Activity report from 222S?	/ [✓ Y] / ~~N~~	
0 COC copy for LRB, RIDS filed?	/ Y / N	
0 [illegible]	— / Y / N/A	

PNL)

Figure 1.1a Chain of Custody for Inorganic Samples for Tank BY-107



Westinghouse Hanford Company CHAIN OF CUSTODY WHC 008070

Custody Form Initiator	J. A. Edwards-PNL	Telephone Page	(509) 373-0141 85-3009 / FAX 376-0418
Company Contact	R. D. Mahon-WHC	Telephone Page	(509) 373-1101 85-9656 / FAX 373-3193
Project Designation/Sampling Locations 241-BY-107 Tank	200 East Tank Farm Vapor Sample SAF S4077 (VSS Truck)	Collection date Preparation date	10-26-94 10-24-94
Ice Chest No.		Field Logbook No.	WHC-___-___-___
Bill of Lading/Airbill No.	N/A	Offsite Property No.	N/A
Method of Shipment	Government Truck		
Shipped to	PNL	Sample Job #	_____
Possible Sample Hazards/Remarks	Unknown at time of sampling		

Sample Identification

S4077-A01.231	Ambient Air #1 Upwind @ VSS (PNL)
S4077-A02.232	Ambient Air #2 Through VSS (PNL)
S4077-A04.233	Sample #3 (PNL)
S4077-A05.234	Sample #4 (PNL)
S4077-A06.235	Sample #5 (PNL)
S4077-A07.236	Sample #6 (OGI)
S4077-A08.237	Sample #7 (OGI)
S4077-A09.238	Sample #8 (OGI)

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

Relinquished By	Date	Time	Received By	Date	Time
J. A. Edwards	10-24-94	1415		10-24-94	1415
	10-26-94	1735		10-26-94	1935
	11-8-94	1330		11-8-94	1330
	11-8-94	1335	JAEDWARDS	11-8-94	1338

Final Sample Disposition

Comments:

	PNL (only) Checklist	Pick-up	/ Delivery	Comments:
0	Media labeled and checked?	[✓ Y]/~~N~~		
0	Letter of instruction?	[✓ Y]/N		
0	Media in good condition?	[✓ Y]/~~N~~	/ [✓ Y]/N	
0	COC Info/signatures complete?	[✓ Y]/N	/ [✓ Y]/N	
0	Sorbents shipped on ice?	~~Y/N~~	/ ~~Y/N~~ N/A	
0	Rad release stickers on samples?		/ [✓ Y]/~~N~~	
0	Activity report from 222S?		/ [✓ Y]/~~N~~	
0	[illegible]		/ Y/N	

Figure 1.1b Chain of Custody for Inorganic Samples for Tank BY-107

7/94 PNL)

2.0 Inorganic

Solid sorbent traps, prepared in multi-trap sampling trains, were supplied to WHC for sampling the tank headspace using the VSS. Blanks, spiked blanks (when requested), and exposed samples were returned to PNL for analysis. Analyses were performed to provide information on the tank-headspace concentration of the following analytes: NH_3, NO_2, NO, 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 samples were effectively trapping NH_3 and mass. Sample preparation, handling, and disassembly were performed as described in Technical Procedure PNL-TVP-09[(a)]. Analytical accuracy was estimated based on procedures used. Sample preparation and analyses were performed following PNL quality assurance (QA) impact level (IL) II requirements.

2.1 Standard Sampling Methodology

Standard glass tubes containing sorbent materials to trap vapors of selected analytes of NH_3, NO_2, NO, and H_2O (SKC Inc., Eighty Four, Pennsylvania) were obtained, prepared, and submitted for use by WHC. The sorbent traps were selected based on their use by the Occupational Safety and Health Administration (OSHA) to perform workplace monitoring, and because of available procedures and verification results associated with that particular application. 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, having 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 NO_2 traps contained a zeolite impregnated with triethanolamine (TEA), with 400 mg in the primary and 200 mg in the breakthrough sections. The NO_2 was absorbed and disproportionated to equi-molar quantities of nitrite ions (NO_2^-) and nitrate ions (NO_3^-). Glass tubes containing 800 mg of an oxidant such as chromate were used to convert NO to NO_2. The converted NO was then collected as nitrite and nitrate in an NO_2 trap. The water traps contained 300 mg of silica gel in the primary and 150 mg in the breakthrough sections.

Samples provided by PNL to trap inorganic compounds include all or some of the following: samples, spiked samples, spares, single trap blanks, and spiked blanks. The samples of each were prepared from same-lot batches, with the oxidizer sections of the NO_x sorbent trains having been stored previously in a freezer. After sample preparation, all samples, spiked samples, blanks, and spiked blanks were stored in a freezer, primarily because of handling recommendations for the oxidizer tubes attached to some samples. After receipt of exposed and radiologically cleared samples

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(a) Pacific Northwest Laboratory. 10/94. *Sorbent Trap Preparation for sampling and Analysis: Waste Tank Inorganic Vapor Samples*, PNL-TVP-09 (Rev.0), PNL Technical Procedure, Richland, Washington.

from WHC and disassembly of the sorbent trains, samples were provided to the analytical laboratory at ambient temperature, and selected oxidizer sections were returned to a freezer until completion of analyses.

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 2.4. 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 (PFA)-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 having 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-trap trains remained sealed other than during the actual sampling periods. C-Flex® tubing was provided by WHC to connect the downstream ends of the sorbent trains to the sampling exhaust manifold connections.

2.1.1 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 mol. 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 (C_v) of a 3.00-L sample containing 75.0 μg of NH_3 equals

$$C_v = \frac{75.0\ \mu g}{17\ g/mol}\left(\frac{3.00\ L}{22.4\ L/mol}\right)^{-1} = 32.9\ ppmv \qquad (2.1)$$

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.

2.2 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.

2.2.1 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 (SIE) procedure PNL-ALO-226 {*Ammonia (Nitrogen) in Aqueous Samples*}. Briefly, this method includes 1) preparing a 1000-µg/mL (ppm) NH_3 stock standard solution from dried reagent-grade NH_4Cl and DIW on the day analyses are performed; 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 (emf) signal versus NH_2 concentration data obtained for the set of working standards; 4) performing a calibration-verification check, using one of the midrange standards, after analyzing every four or five 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). Emf 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.

2.2.2 Nitrite Analysis. The sorbent traps for NO_2 and NO were desorbed in an aqueous TEA and n-butanol solution and analyzed by suppressed-conductivity ion chromatography (SCIC) for nitrite according to PNL-ALO-212, Rev. 1 (*Determination of Inorganic Anions by Ion Chromatography*) modified to obviate interferences by concentrations of non-target analytes. Specifically, the modifications used were 1) eluent 1.44 mM Na_2CO_3 + 1.8 mM $NaHCO_3$ at 2.0 mL/min, 2) one guard column (AG4A) and two separator columns (AS4A) in series instead of just one separator column, and 3) all standards, samples, and blanks injected into the IC sample loop through 0.45-µm syringe filters.

For the analysis, the sorbent materials were placed into labeled 20-mL glass scintillation vials. To each vial, 3.0 mL of desorbing solution (15 g TEA + 1 mL n-butanol in 1.0 L DIW) was added. Primary sorbent-tube sample materials and back-up (breakthrough) sorbent-trap materials were analyzed separately using identical procedures. Each analytical session was conducted as follows. Working nitrite standards (0, 0.1, 0.25, and 0.5 ppm) were prepared by diluting a stock nitrite standard with desorbing solution. An initial calibration curve was prepared from the instrument response (chromatographic peak height) versus nitrite standard concentration data for the set of working standards. A calibration verification check using one of the midrange standards was performed after the analysis of every six samples. If the instrument response indicated that sample nitrite concentration was outside the calibration range (> 0.5 ppm nitrite), the sample was diluted with desorbing solution and reanalyzed. After all samples of a batch were analyzed, the complete set of calibration standards was remeasured to verify consistent instrument response, and the analytical session was terminated.

Instrument responses (peak height) observed for samples were compared to those for standards to determine the nitrite concentration of the samples. Because NO_2 and NO converted to NO_2 were collected on the sorbent as equal quantities of nitrite and nitrate, and the analysis was specific for nitrite, the molar masses of NO_2 and NO were determined by doubling the analytically determined molar mass of nitrite.

2.2.3 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 and spiked blanks were included to provide information on uncertainty.

2.3 Quality Assurance/Quality Control

Analytical work was performed according to quality levels identified in the project QA plan and several PNL documents. The samples were analyzed following PNL IL II. The PNL documents include PNL-MA-70 (Part 2), PNL-MA-599, PNL-ALO-212, PNL-ALO-226, and MCS-033. A summary of the analysis procedures and limits for the target inorganic compounds is provided in Table 2.1. From the table, it can be seen that the minimum detection limit (MDL) required to resolve the analyte at one-tenth of the recommended exposure limit (REL) 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).

Table 2.1. Analysis Procedures and Typical Detection Limits of Target Inorganic Analytes. Not all analytes are included in every sample job.

Analyte	Formula	Procedure	REL[a] (ppmv)	0.1 x REL[a] (ppmv)	MDL[b] (ppmv)
Ammonia	NH_3	PNL-ALO-226	25	2.5	0.5
Nitrogen Dioxide	NO_2	PNL-ALO-212	1	0.1	0.02
Nitric oxide	NO	PNL-ALO-212	25	2.5	0.02
Mass (water)[c]	n/a	n/a	n/a	n/a	n/a

(a) Target analytical limits are equal to one-tenth of the REL.

(b) MDL is defined as the vapor concentration that can be detected with an uncertainty equal to about the magnitude of the measurement. The uncertainty is expected to reduce to about one-quarter of the magnitude of the measurement at a concentration of four times the MDL. The MDLs were based on the assumption that 3 L of vapor are sampled; if greater volumes of vapor are sampled, correspondingly smaller MDLs can be achieved. The MDLs were also based on desorbing-solution volumes of 10 mL for NH_3 and 3 mL for the other analytes.

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

The accuracy of concentration measurements depends on errors associated with both sampling and analysis (see Section 2.4). Sampling information, including sample volumes, was provided by WHC; sample volume uncertainty was not provided. The accuracy of analytical results depends on the method used. For NH_3 analyses, the accuracy of laboratory measurements by SIE 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. Unfortunately, no known National Institute for Standards and Technology (NIST)-traceable standard reference material (SRM) is available against which to compare working standards. Similarly, no known NIST SRM is available for nitrite analysis (for NO_2 and NO). Based on experience in comparing nitrite working standards prepared from several different sources and factors mentioned for NH_3 above, the estimated maximum bias for samples derived from sampling for NO_2 is ± 10%, and for samples derived from sampling for NO, it is ± 5% relative.

The accuracy of measurements of sample mass is ± 0.05 mg, or much less than 1% of the mass changes of most samples, and roughly 10% or less of the mass change of most blanks. The analytical accuracy of measurements of the change in mass of sorbent trains is estimated to be ± 1 mg per 5-trap sorbent train; this estimate is based largely on preliminary information that unopened field-blank sorbent trains gain 0.3 ± 0.4 mg per train.

2.4 Inorganic Sample Results

Samples were obtained by WHC from the headspace of Tank BY-107 on 10/26/94 using the VSS. The sample job designation number was S4077. Samples were prepared, submitted to WHC, and then analyzed to provide information on the concentrations of NH_3, NO_2, NO, and H_20. Sampling and analysis for hydrogen cyanide (HCN) and sulfur oxides ($S0_x$) was not requested. The inorganic samples were received from WHC on 11/8/94; the sample-volume information was received on 11/9/94.

A list of samples, sampling information, sample volumes, and gravimetric results is shown in Table 2.2. The types of sample trains used and the order of sorbent traps within each train are also shown in the table. For example, the sorbent train $NH_3/NO_x/H_2O$ contained an NH_3 trap at the inlet end, an NO_x series in the middle (Section 2.4.2), and a desiccant trap at the outlet end. Analytical mass and concentration results are shown in Table 2.3. Sample volumes were provided by WHC; sample-volume uncertainty was not provided. Tank-headspace concentration results (Table 2.3) are based on this information, and the listed uncertainties equal plus-or-minus one standard deviation of the individual results from each set of samples. Where analytical results from samples were nearly indistinguishable from those of blanks, indicating very low vapor concentrations of the analyte, the concentration results (Table 2.3) are listed as "less than or equal to" a probable maximum value determined by subtracting the average of the blanks less one standard deviation from the average of the samples plus one standard deviation. Results of control samples, such as spiked blanks, are discussed in this section. Spiked blanks, when used, were transported to the field but not opened. Spiked samples, when used, were opened in the field and used to collect tank vapors. Sample results were not corrected for the percentage recoveries of spiked blanks.

2.4.1 Ammonia Results. The concentration of NH_3 was 972 ± 6 ppmv, based on all six samples. The blank-corrected NH_3 quantities in the sorbent traps ranged from 129 to 130 μmol in the front sorbent sections; back sections were not analyzed. Blank corrections, ≤ 0.06 μmol in front sections, were less than 0.1% of collected quantities and were neglected. Although spiked blanks were not tested, the percentage recoveries of three sets of blanks spiked with 12.2, 22.3, and 46.4 μmol of NH_3 were 101 ± 4%, 109 ± 2%, and 104 ± 1%, respectively, during related sample jobs (Clauss et al. 1994; Ligotke et al. 1994). The analysis of one sample was duplicated and yielded a repeatability of ± 2%. One sample leachate was spiked after initial analysis with roughly the quantity of NH_3 in the sample and yielded a percentage recovery of 108%. A 5-point calibration was performed over an NH_3 range of 0.1 to 1000 μg/mL.

2.4.2 Nitrogen Oxides Results. Measurements of NO_2 and NO were made using six 5-segment $NH_3/NO_x/H_2O$ sorbent-trap trains (the NO_x trains consisted of NO_2 trap, oxidizer, and NO_2 trap). Related sample jobs, performed using the VSS in BY-104, -105, and -106 both with and without NO_x trains protected by a leading NH_3 trap (e.g., Clauss et al. 1994), indicated that the presence of the

Table 2.2 List of PNL Inorganic Samples, Controls, and Gravimetric Results Obtained From a Heated Tube Inserted into the Headspace of Tank BY-107 on 10/26/94

			Sample Port and Volume Information[a]			
Sample Number	Sorbent Type	Sample Port	Flow Rate (mL/min)	Duration (min)	Volume (L)	Mass Gain (g)
Samples:						
S4077- A26-64V	$NH_3/NO_x/H_2O$ Train	9	200	15.0	3.00	0.0473
S4077- A27-65V	$NH_3/NO_x/H_2O$ Train	10	200	15.0	3.00	0.0444
S4077- A28-66V	$NH_3/NO_x/H_2O$ Train	9	200	15.0	3.00	0.0467
S4077- A29-67V	$NH_3/NO_x/H_2O$ Train	10	200	15.0	3.00	0.0390[c]
S4077- A30-68V	$NH_3/NO_x/H_2O$ Train	9	200	15.0	3.00	0.0453
S4077- A31-69V	$NH_3/NO_x/H_2O$ Train	10	200	15.0	3.00	0.0440
S4077- A32-70V	$NH_3/NO_x/H_2O$ Blank	n/a[b]	n/a	n/a	n/a	0.0011
S4077- A33-71V	$NH_3/NO_x/H_2O$ Blank	n/a	n/a	n/a	n/a	0.0019
S4077- A34-72V	$NH_3/NO_x/H_2O$ Blank	n/a	n/a	n/a	n/a	0.0019

(a) Sampling information and dry-gas sample volumes, corrected to 0°C and 760 torr, were provided by WHC. Uncertainty values were not provided with sample-volume results.

(b) n/a = not applicable.

(c) Results suspected to be biased by loss of glass from sorbent trap.

upstream NH_3 traps resulted in NO concentrations that were about 1.3- to 1.6-fold less than those from unprotected NO_2 traps. The NO_2 concentrations were also potentially less following an NH_3 trap.

The concentrations of NO_2 and NO were ≤ 0.02 and $\leq 0.13 \pm 0.03$ ppmv, respectively. Blank-corrected NO_2^- quantities in the sorbent traps averaged ≤ 0.002 μmol (NO_2 samples) and ≤ 0.086 μmol (NO samples). Nitrite blank levels used to correct data were 0.0124 ± 0.0003 μmol in front (three of six blanks analyzed) and 0.0072 μmol in back (one of six blanks analyzed) sorbent sections. Although spiked blanks were not tested, blanks spiked with 0.0064, 0.047, 0.11, and 0.74 μmol of NO_2^- during related sample jobs yielded percentage recoveries of 153 ± 14%, 103 ± 4%, 106 ± 8%, and 111 ± 7%, respectively (Clauss et al. 1994; Ligotke et al. 1994). The analysis of one sample was duplicated and yielded a repeatability of ± 5%. One sample leachate as spiked with 0.25 ppm NO_2^- and yielded a percentage recovery of 94%. A 4-point calibration was performed over a concentration range of 0 to 0.5 μg NO_2^- per ML in the desorbing matrix.

2.4.3 Gravimetric Results. The mass concentration of material collected in the 5-trap sorbent trains, believed to be primarily water vapor, was 15 ± 2 mg/L. The result was based on an average mass gain of 45.8 mg from all six $NH_3/NO_2/H_2O$ sample trains. The blank correction applied to the results was 0.3 mg per sample train, based on mass loss of 0.3 ± 0.4 mg per three blank 5-trap sorbent trains. The blanks, first assembled as complete 5-trap sorbent trains, accompanied samples from three related VSS sample jobs in December 1994 (TX-105, TX-118, and BX-104). Although no spiked blanks were tested, the percentage recovery of mass from three blank H_2O traps spiked with 51 mg of water was 103 ± 2% during a related sample job (Clauss et al. 1994).

Table 2.3 Inorganic Vapor Sample Results Obtained From a Heated Tube Inserted into the Headspace of Tank BY-107 on 10/26/94

	Analytical Results (μmol)				
Sample	Front Section	Back Section	Total[b] Blank-Corrected	Sample Volume (L)	Vapor[a] Concentration (ppmv)
NH_3 Samples:			130[c]	3.00[c]	972 ± 6[c]
S4077- A26-64V	130	NA[d]	130	3.00	971
S4077- A27-65V	130	NA	130	3.00	971
S4077- A28-66V	129	NA	129	3.00	963
S4077- A29-67V	131	NA	131	3.00	978
S4077- A30-68V	131	NA	131	3.00	978
S4077- A31-69V	130	NA	130	3.00	971
NO_2 Samples:			≤0.002	3.00	≤ 0.02
S4077- A26-64V	0.0138	NA	n/a[d]	3.00	n/a
S4077- A27-65V	0.0096	0.0061	n/a	3.00	n/a
S4077- A28-66V	0.0136	N/A	n/a	3.00	n/a
S4077- A29-67V	0.0126	N/A	n/a	3.00	n/a
S4077- A30-68V	0.0097	N/A	n/a	3.00	n/a
S4077- A31-69V	0.0106	N/A	n/a	3.00	n/a
NO Samples:			0.0086	3.00	0.13 ± 0.03
S4077- A26-64V	0.0198	NA	0.0074	3.00	0.11
S4077- A27-65V	0.0192	NA	0.0068	3.00	0.10
S4077- A28-66V	0.0203	NA	0.0079	3.00	0.12
S4077- A29-67V	0.0249	NA	0.0125	3.00	0.19
S4077- A30-68V	0.0203	0.0077	0.0079	3.00	0.12
S4077- A31-69V	0.0213	NA	0.0089	3.00	0.13
Gravimetric Samples:			45.8 mg	3.20	15 ± 1 mg/L
S4077- A26-64V	n/a	n/a	47.6	3.00	15.9
S4077- A27-65V	n/a	n/a	44.7	3.00	14.9
S4077- A28-66V	n/a	n/a	47.0	3.00	15.7
S4077- A29-67V	n/a	n/a	39.3 x[d]	3.00	13.1 x
S4077- A30-68V	n/a	n/a	45.6	3.00	15.2
S4077- A31-69V	n/a	n/a	44.3	3.00	14.8

(a) Blank-corrected vapor concentrations were calculated using WHC-reported dry-air sample volumes (corrected to 0°C and 760 torr). In the calculation for concentration, the nitrite values (listed) were doubled to account for unanalyzed nitrate. Sample results were not corrected for percentage recovery of spiked samples or spiked blanks.

(b) Total blank-corrected analyte masses (nitrite for NO_2 and NO) were determined, when significant, by subtracting the quantity of analyte found in blanks from that found in samples. The level of analytes found in blanks is described in the subsections of Section 2.4.

(c) Underlined values represent the average of the set samples. Concentration uncertainty equals ± 1 standard deviation (absolute) for each set of samples. The use of "≤" is defined in Section 2.4.

(d) NA = not analyzed; n/a = not applicable; x=not included in determination of average concentration. Only selected back sorbent sections were analyzed. Past results have shown back sections of NH_3 samples to contain insignificant quantities of the analyte.

3.0 Organic

3.1 SUMMA™ Canister Preparation

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant free according to PNL 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 PNL Technical Procedure PNL-TVP-01[b], which is a modification of 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 30 in. Hg, tagged, and stored for use in the field. Before sending the canisters out to the field for sampling, 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.

3.2 Sample Analysis Methods

The SUMMA™ canister sample was analyzed according to PNL Technical Procedure PNL-TVP-03, *Determination of TO-14 Volatile Organic Compounds in Hanford Waste Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Gas Chromatographic-Mass Spectrometry Analysis*, which is a modified version of EPA compendium Method TO-14. The method uses an EnTech cryoconcentration system interfaced with a Hewlett Packard (HP) 5971 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 and 3-μm film thickness. The GC oven is programmed to run a temperature gradient beginning at 40°C, holding for 5 min, and ramping at 4°C per min to a final temperature of 260°C, with a 5-min hold.

The TO-14 extended analytes were prepared using permeation tubes according to *Preparation of TO-14 Volatile Organic Compounds Gas Standards* PNL Technical Procedure PNL-TVP-06. The additional analytes were blended with the TO-14 standard to make a 55-component calibration gas. The standard calibration mix was analyzed using six aliquot sizes ranging from 0.3 L to 0.005 L. Depending on the concentration of each analyte in the mixture, either five or six points were used to construct the calibration curve. Butonoic acid was added to the mixture, but was not detected in the analysis. Butanal was recovered very poorly, producing a much lower than expected response. Therefore, butanal was reported as a TIC. Detection limits for the TO-14 extended analytes have not

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

[b] Pacific Northwest Laboratory. 8/94. *Determination of TO-14 Volatile Organic Compounds in Ambient Air Using SUMMA™ Passivated Canister Sampling and Gas Chromatographic-Mass Spectrometric Analysis*, PNL-TVP-01 (Rev. 0). PNL Technical Procedure, Richland, Washington.

yet been determined. Performance-based detection limits will be developed as a pool of calibration data becomes available.

The SUMMA™ canister samples were analyzed for permanent gases according to PNL Technical Procedure PNL-TVP-05, *Analysis Method for the Determination of Permanent Gases in Hanford Waste Tank Vapor Samples Collected in SUMMA™ Passivated Stainless Steel Canisters*, 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 for the analysis of permanent gases defined as hydrogen, (H_2), carbon dioxide (CO_2), carbon monoxide (CO), methane (CH_4), and nitrous oxide (N_2O) by gas chromatography-thermal conductivity detection (GC-TCD) and is not validated in any other laboratory. No previous work up of the sample canister is necessary before permanent gas analysis. Aliquots of sampled air are manually drawn from each canister into a 5-mL gas-tight syringe and directly injected into a GC-TCD fitted with a 0.5-mL injection loop. An aliquot of 5 mL is used so that the injection loop is flushed and filled with sample air, ensuring that no dilution of sample concentration takes place within the injection loop. One set of GC conditions are used to analyze 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 increase the detection limit for this analyte. A total of 30 to 60 mL may be drawn from each canister sample, depending upon the number of repeat analyses performed.

3.3 Quality Assurance/Quality Control

Before the tank sample was analyzed, a diagnostic check was performed on the GC/MS instrument by running an instrument "quick 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 over 5 to 6 data points ranging from 5 ppbv to 300 ppbv, using a standard gas mixture containing 40 volatile organic compounds listed in EPA compendium Method TO-14 and an additional 14 tank-related compounds (referred to as TO-14 extended analytes). A gas mixture containing bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5 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 curve 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. A least-squares linear-regression routine was applied to the data set to generate the best-fit line for each compound. The equation for that line was then used to quantify the TO-14 compounds found in the tank samples.

Standards for the permanent gas analysis were blended from commercially prepared and certified standards for each of the analytes reported in Table 3.5. The instrument was calibrated over three data points for CO, CO_2, N_2O, and CH_4 using He as a carrier gas, and then the samples were analyzed. The carrier was changed to N_2, the calibration was performed for H_2 only, and the samples were reanalyzed. A least-squares linear-regression routine was applied to the data set to generate the best fit for each compound. The quantitation for each analyte was performed by direct comparison of sample analyte peaks to the plot generated for the compound. The lowest calibration standard for each analyte is reported as the method detection limit. An MDL for the instrument has not been determined. A nitrogen reagent blank was not analyzed with this set of samples. The ambient air sample collected ~10 m upwind of BY-107 and the ambient air collected through the VSS were used as method blanks and to determine the potential for analyte interferences in the samples. Continuing calibration standards for this sample set fell within ± 25% of the expected concentrations for the analytes observed in the samples.

3.3.1 Quantitation Results of TO-14 Extended Analytes. The quantitative-analysis results for the TO-14 extended analyte volatile organic compounds were calculated directly from the calibration curve 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:

$$\text{mg/m}^3 = \frac{(\text{ppbv}/1000) \times \text{g mol wt of compound}}{22.4 \text{ L/mol}} \tag{3.1}$$

3.3.2 Identification and Quantitation of Tentatively Identified Compounds. The tentatively identified compounds (TICs) are determined by mass-spectral interpretation and comparison of the spectra with the EPA/NIST/WILEY Library, which is a part of the HP 5971 instrument operating system. Chromatographic peaks with an area count greater than, or equal to, one half of the total area count of the chlorobenzene-d_5 IS peak at the 20-ppbv calibration level are tentatively identified and quantitatively estimated. This standard was chosen to determine the integration cutoff as it is in the middle of the chromatographic range and not in a region typically affected by coelution of other compounds. The quality of the mass-spectral searches was then reviewed by the principal investigators before the identification was assigned to each chromatographic peak.

The concentration of each TIC was estimated using a relative response factor calculated using a corrected total peak area for the IS chlorobenzene-d_5. Specifically, the total integrated area for the chlorobenzene-d_5 peak had to be corrected for possible coeluting compounds before calculating the response factor. The corrected total peak area for the IS was calculated by multiplying the IS quantitation ion by a correction factor based on the ratio of the total integrated peak area to the quantitation ion as measured in blank runs. The corrected peak area was then used to calculate a response factor using the IS concentration in mg/m^3:

$$\text{Response Factor} = \frac{\text{IS conc. (mg/m}^3\text{)}}{\text{IS peak area}} \tag{3.2}$$

The calculated response factor was then multiplied by the TIC peak area to give an estimated concentration for that compound. For butane, the total peak area was multiplied by the response factor for chlorobenzene-d_5 to give an estimated concentration of 1.7 mg/m^3. Internal standards bromochloromethane and difluorobenzene were not used to quantitate the TICs because coeluting compounds appeared to have greatly altered the signal of the quantitation ions for those two ISs.

The ppbv concentrations are calculated from mg/m^3 and the molecular weight of the analyte.

$$\text{TIC in ppbv} = \frac{\text{TIC (mg/m}^3\text{)} \times 22.4 \text{ L/mol} \times 1000}{\text{TIC g mol wt}} \tag{3.3}$$

The IS level added to all blank, standard, and sample injections was 18.3 ppbv for bromochloromethane, 20.3 ppbv for 1,4-difluorobenzene, and 18.2 ppbv for chlorobenzene-d_5. The IS concentrations were converted from ppbv to mg/m^3 at STP using a molecular weight of 129.39 (g/mol) for bromochloromethane, 114.09 for 1,4-difluorobenzene, and 117.6 for chlorobenzene-d_5.

3.4 Analysis Results

The results from the GC/MS analysis of the tank-headspace samples are presented in Tables 3.1 and 3.2. The results of GC/MS analysis of ambient air sample collected upwind of Tank BY-107 and through the VSS near Tank BY-107 are presented in Tables 3.3 and 3.4. The GC/MS analysis results for permanent gases for both the ambient samples and from the tank headspace are presented in Table 3.5. A representative total ion chromatogram showing the identity of major constituents is given in Figure 3.1.

Table 3.1 lists the quantitative results for compounds listed in Method TO-14 extended analytes. Acetone was approximately 52% of the total mass of the target analytes.

Table 3.2 lists the semi-quantitative results for the TICs. The predominant species observed in this sample were 1-butanol, tridecane, and dodecane. The normal paraffin hydrocarbons (NPH), defined as n-alkanes from C_{11} to C_{15}, were observed at trace levels. It should be noted that because the SUMMA™ canisters were not heated at the time of analysis, the NPH concentrations listed after the retention time of decane may not be a true accounting of all the NPH in the sample. Similarly, polar compounds, which may adhere to the inside surface of the canister, may also be under represented in this analysis. The total concentration of the TIC compounds was found to be 51.19 mg/m^3.

Tables 3.3 and 3.4 list results of TO-14 extended analyte analyses for ambient air collected upwind of Tank BY-107 and ambient air collected through the VSS. Acetaldehyde, ethanol, methylcyclopentane, and acetone were detected in the upwind sample. Acetaldehyde and acetone were observed through the VSS.

Table 3.5 lists results of permanent gas analysis from samples collected from the headspace of Tank BY-107, ambient air collected ~10 m upwind of the tank, and ambient air collected through the VSS. Permanent gases observed in the headspace were CO_2 and N_2O. Carbon dioxide in the headspace was at a lower concentration than in ambient air. Nitrous oxide was not detected either in the ambient air collected ~10 m upwind of the Tank BY-107 or ambient air collected through the VSS. A replicate analysis was performed on one of the samples collected from this tank; however, only the results from the first analysis are reported. A replicate H_2 analysis for one of the tank samples resulted in a concentration approximately 39% higher than the first analysis. Since the level of H_2 detected in these samples is much lower than the 20% flammability limit for the waste tanks, the sample set was not reanalyzed and only the first analysis results are reported for H_2.

4.0 Conclusions

The concentrations of selected inorganic and organic compounds were determined from samples of the headspace of Tank BY-107 on 10/26/94. Sampling and analysis methods followed those described by Ligotke et al. (1994) and Clauss et al. (1994) for samples obtained from C-103, a tank containing a relatively complex headspace composition. Method-validation measurements during that study did appear to validate the trapping and analysis of NH_3, but did not eliminate the possibility of interferences that could affect NO_x results. It is recommended that additional control samples be obtained if a tank is discovered in the future to contain significant quantities of NO_x. In the current sample job, NO_x samples were obtained after first passing the sample flow through an NH_3 trap. The average and standard deviation of the concentration results from inorganic sorbent trains were 972 ± 6 ppmv (NH_3), $\leq$0.02 ppmv (NO_2), 0.13 ± 0.03 ppmv (NO), and 15 ± 1 mg/L (vapor-mass concentration). The vapor-mass concentration is expected to consist largely of water vapor. The major organic constituent in the samples collected from the headspace of Tank BY-107 was acetone at 16.7 mg/m^3, approximately 52% of the total organic mass. Normal paraffin hydrocarbons were observed in trace amounts. The average level of CO_2 in the headspace was 94 mg/m^3 (below the level of CO_2 in air).

5.0 References

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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.

6.0 Further Reading

Pacific Northwest Laboratory. Analytical Laboratory Procedure Compendium. Procedures PNL-ALO-212, -226, -271. PNL-MA-599, Richland, Washington.

Pacific Northwest Laboratory. Quality Assurance Manual, Part 2: Good Practices Standard. PNL-MA-70, Part 2, Richland, Washington.

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Pacific Northwest Laboratory. 1994. *Determination of TO-14 Volatile Organic Compounds in Hanford Waste Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Gas Chromatographic-Mass Spectrometry Analysis*, PNL-TVP-03 (Rev. 0), PNL Technical Procedure, Richland, Washington.

Pacific Northwest Laboratory. 1994. *Sample Shipping and Receiving Procedure - DRAFT for PNL Waste Tank Samples*. PNL-TVP-07 (Rev. 0), PNL Technical Procedure, Richland, Washington.

Table 3.1 Positively Identified and Quantitated TO-14 Extended Analytes[a] Collected from the Headspace of Tank BY-107 in SUMMA™ Canisters on 10/26/94

			S4077-A04.233[b] PNL 233[c] Concentration		S4077-A05.234[b] PNL 234[c] Concentration		S4077-A06.235[b] PNL 235[c] Concentration		Means and Standard Deviations	
Analyte	CAS No.	Mol Wt	(mg/m^3)	(ppbv)	(mg/m^3)	(ppbv)	(mg/m^3)	(ppbv)	(mg/m^3)	St Dev
Dichlorodifluoromethane (FREON-12)	75-71-8	120	<0.03	<5	<0.03	<5	<0.03	<5	(d)	(d)
Methyl Chloride (Chloromethane)	74-87-3	50	<0.01	<5	<0.01	<5	<0.01	<5	(d)	(d)
1,2-Dichloro-1,1,2,2,-Tetrafluoroethane (FREON-114)	76-14-2	170	<0.04	<5	<0.04	<5	<0.04	<5	(d)	(d)
Chloroethene (Vinyl Chloride)	75-01-4	64	<0.01	<5	<0.01	<5	<0.01	<5	(d)	(d)
Methyl Bromide (Bromomethane)	74-83-9	94	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
Ethyl Chloride	75-00-3	62	<0.01	<5	<0.01	<5	<0.01	<5	(d)	(d)
Trichlorofluoromethane (FREON-11)	75-69-4	136	0.40	65.9	0.37	61.4	0.35	57.2	0.37	0.03
1,1-Dichloroethene (1,1-Dichloroethylene)	75-35-4	96	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
Dichloromethane (Methylene Chloride)	75-09-2	84	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
1,1,2-Trichloro-1,2,2-Trifluoroethane (FREON-113)	76-13-1	186	<0.04	<5	<0.04	<5	<0.04	<5	(d)	(d)
1,1-Dichloroethane	75-34-3	98	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
cis-1,2-Dichloroethene (cis-1,2-Dichloroethylene)	156-59-2	96	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
Trichloromethane (Chloroform)	67-66-3	118	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
1,2-Dichloroethane	107-06-2	98	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
1,1,1-Trichloroethane	71-55-6	132	<0.03	<5	<0.03	<5	<0.03	<5	(d)	(d)
Benzene	71-43-2	78	0.03	7.8	0.03	7.8	0.03	7.8	0.03	0.00
Carbon Tetrachloride	56-23-5	152	<0.03	<5	<0.03	<5	<0.03	<5	(d)	(d)
1,2-Dichloropropane	78-87-5	112	<0.03	<5	<0.03	<5	<0.03	<5	(d)	(d)
Trichloroethylene	79-01-6	130	<0.03	<5	<0.03	<5	<0.03	<5	(d)	(d)
cis 1,3-Dichloropropene	61-01-5	110	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
trans 1,3-Dichloropropene	61-02-6	110	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
1,1,2-Trichloroethane	79-00-5	132	<0.03	<5	<0.03	<5	<0.03	<5	(d)	(d)
Methyl Benzene (Toluene)	108-88-3	92	0.27	65.0	0.29	71.3	0.32	77.9	0.29	0.03
1,2-Dibromoethane	106-93-4	186	<0.04	<5	<0.04	<5	<0.04	<5	(d)	(d)
Tetrachloroethene (Perchloroethylene)	127-18-4	164	<0.04	<5	<0.04	<5	<0.04	<5	(d)	(d)
Chlorobenzene	108-90-7	112	<0.03	<5	<0.03	<5	<0.03	<5	(d)	(d)
Ethylbenzene	100-41-4	106	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
m-Xylene (1,3-Dimethylbenzene)[e]	108-38-3	106	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
p-Xylene (1,4-Dimethylbenzene)[e]	106-42-3	106								
Styrene	100-42-5	104	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
1,1,2,2-Tetrachloroethane	79-34-5	166	<0.02	<5	<0.02	<5	<0.02	<5	(d)	(d)
o-Xylene (1,2-Dimethylbenzene)	95-47-6	106	<0.04	<5	<0.04	<5	<0.04	<5	(d)	(d)

Table 3.1 (Contd)

Analyte	CAS No.	Mol Wt	S4077-A04.233(b) PNL 233(c) Concentration (mg/m^3)	(ppbv)	S4077-A05.234(b) PNL 234(c) Concentration (mg/m^3)	(ppbv)	S4077-A06.235(b) PNL 235(c) Concentration (mg/m^3)	(ppbv)	Means and Standard Deviations (mg/m^3)	St Dev
1,3,5-Trimethylbenzene	108-67-8	120	< 0.03	< 5	< 0.03	< 5	< 0.03	< 5	(d)	(d)
1,2,4-Trimethylbenzene	95-63-6	120	< 0.03	< 5	< 0.03	< 5	< 0.03	< 5	(d)	(d)
Chloromethylbenzene, alpha (Benzyl Chloride)	100-44-7	126	< 0.03	< 5	< 0.03	< 5	< 0.03	< 5	(d)	(d)
m-Dichlorobenzene (1,3-Dichlorobenzene)	541-73-1	146	< 0.03	< 5	< 0.03	< 5	< 0.03	< 5	(d)	(d)
p-Dichlorobenzene (1,4-Dichlorobenzene)	106-46-7	146	< 0.03	< 5	< 0.03	< 5	< 0.03	< 5	(d)	(d)
o-Dichlorobenzene (1,2-Dichlorobenzene)	95-50-1	146	< 0.03	< 5	< 0.03	< 5	< 0.03	< 5	(d)	(d)
1,2,4-Trichlorobenzene	120-82-1	180	< 0.04	< 5	< 0.04	< 5	< 0.04	< 5	(d)	(d)
Hexachloro-1,3-Butadiene	87-68-3	258	< 0.02	< 5	< 0.02	< 5	< 0.02	< 5	(d)	(d)
2-Butanone	78-93-3	72	3.73	1161	3.47	1079	3.47	1079	3.56	0.15
Acetone	67-64-1	58	18.67	7209	16.27	6282	15.20	5870	16.71	1.78
Acetonitrile	75-05-8	41	< 0.01	< 5	< 0.01	< 5	< 0.01	< 5	(d)	(d)
Heptane	142-82-5	100	0.80	179	0.88	197	0.91	203	0.86	0.06
Tetrahydrofuran	109-99-9	72	1.79	556	1.81	564	1.84	572	1.81	0.03
Pyridine	110-86-1	79	< 0.02	< 5	< 0.02	< 5	< 0.02	< 5	(d)	(d)
Butanenitrile	109-74-0	69	< 0.02	< 5	< 0.02	< 5	< 0.02	< 5	(d)	(d)
Cyclohexane	110-82-7	84	0.43	114	0.08	21.3	0.37	99.5	0.29	0.19
Decane	124-18-5	142	0.21	33.6	0.21	33.6	0.19	29.5	0.20	0.02
Hexane	110-54-3	86	3.20	833	2.93	763	2.93	764	3.02	0.16
4-Methyl-2-Pentanone	108-10-1	100	0.11	24.0	0.13	29.8	0.13	29.8	0.12	0.02
Propanenitrile	107-12-0	55	< 0.01	< 5	< 0.01	< 5	< 0.01	< 5	(d)	(d)
Cyclohexanone	108-94-1	98	0.02	5.5	0.03	6.2	<0.02	< 5	0.03	0.00
Propanol	71-23-8	60	5.33	1990	4.53	1691	4.27	1593	4.71	0.55

(a) TO-14 plus 15 additonal target analytes.
(b) WHC sample identification number.
(c) PNL canister number.
(d) Average and standard deviation are not meaningful for this analyte.
(e) m-Xylene and p-Xylene coelute; the reported concentration is the sum of these two compounds.
(f) A discussion of this analyte is included in the text.

Table 3.2 Tentatively Identified Compounds and Estimated Concentrations[(a)] from the Headspace of Tank BY-107 in SUMMA™ Canisters Collected on 10/26/94

Tentatively Identified Compound[(d)]	CAS No.[(d)]	Mol Wt	Ret Time	S4077-A04.233[(b)] PNL 233[(c)]		S4077-A05.234[(b)] PNL 234[(c)]		S4077-A05.235[(b)] PNL 235[(c)]		Means and Standard Deviations			
				(mg/m^3)	(ppbv)	(mg/m^3)	(ppbv)	(mg/m^3)	(ppbv)	(mg/m^3)	St Dev	(ppbv)	St Dev
Propane	74-98-6	44	3.94	1.08	548	1.44	731	2.37	1205	1.63	0.67	828	339
Propene	115-07-1	42	3.94	2.18	1163	1.53	814	2.37	1262	2.02	0.44	1079	235
Cyclopropane	75-19-4	42	4.81	0.35	185	0.40	212	0.38	204	0.38	0.03	200	14.2
Isobutane	75-28-5	58	5.12	0.65	251	0.71	275	0.68	264	0.68	0.03	263	12.0
1-Butene	106-98-9	56	5.71	0.71	283	0.76	304	0.70	281	0.72	0.03	289	12.8
Butane	106-97-8	58	5.97	1.74	672	1.74	674	1.61	623	1.70	0.07	656	28.6
2-Methyl-1-Propene	115-11-7	56	6.62	0.32	130	0.36	144	0.31	125	0.33	0.02	132.8	10.0
Ethanol	64-17-5	46	7.49	<0.02	<10.0	0.14	68	<0.02	<10.0	(e)	(e)	(e)	(e)
Isopropyl Alcohol	67-63-0	60	9.05	0.62	231	0.61	228	0.59	221	0.61	0.01	226	5.0
Pentane	109-66-0	72	9.63	1.10	343	1.11	347	1.10	342	1.11	0.01	344	2.2
2-Methylpentane	107-83-5	86	13.12	1.51	393	1.50	392	1.53	397	1.51	0.01	394	3.1
Butanal	123-72-8	72	13.39	0.08	25	0.08	25	0.08	25	0.08	0.00	25	0.0
3-Methylpentane	96-14-0	86	13.91	0.29	77	0.31	80	0.31	81	0.30	0.01	79.2	2.3
2-Butanol	78-92-2	74	14.17	0.25	77	0.26	79	0.26	79	0.26	0.00	78.3	1.3
Methylcyclopentane	96-37-7	84	16.51	0.18	47	0.19	51	0.19	51	0.19	0.01	49.4	2.2
3-methyl-2-Butanone	563-80-4	86	17.16	0.25	64	0.25	64	0.23	60	0.24	0.01	62.9	2.1
1-Butanol	71-36-3	74	17.57	5.88	1779	5.31	1607	4.80	1454	5.33	0.54	1613	162.6
2-Pentanone	107-87-9	86	18.61	1.92	499	<0.04	<10.0	1.71	444	1.81	(e)	472	(e)
3-Methyhexane,	589-34-4	100	19.16	1.07	239	1.06	237	1.02	229	1.05	0.02	235	5.1
2-Pentanol	6032-29-7	88	19.55	0.17	44	0.17	44	0.17	42	0.17	0.00	43.4	1.1
1-Heptene	592-76-7	98	19.83	0.16	36	0.16	36	0.15	35	0.15	0.00	35.4	0.5
Unknown C7 Alkene/Cycloalkane		98	20.06	<0.05	<10.0	0.14	32	0.14	32	0.14	0.00	32.0	0.6
2-Methyl-2-Pentanol	590-36-3	102	21.55	0.20	44	0.21	46	0.20	44	0.20	0.01	44.8	1.2
Methylcyclohexane	108-87-2	98	22.06	0.35	80	0.35	81	0.34	78	0.35	0.01	79.8	1.5
3-Methyl-2-Pentanone	565-61-7	100	22.55	<0.05	<10.0	<0.05	<10.0	0.16	35	(e)	(e)	(e)	(e)
1-Pentanol	71-41-0	88	23.22	0.16	40	0.16	41	0.15	38	0.16	0.01	39.7	1.5
2-Methylheptane	592-27-8	114	24.17	0.58	114	0.62	122	0.62	122	0.61	0.02	119.5	4.4
2-Hexanone	591-78-6	100	24.29	0.62	140	0.61	136	0.64	143	0.62	0.02	139.6	3.4
Octane	111-65-9	114	25.97	0.57	112	0.59	116	0.61	119	0.59	0.02	115.8	3.5
Unknown Alkane			27.58	0.24	(f)	0.25	(f)	0.27	(f)	0.26	0.01	(e)	(e)

BY-107 Table 3.2 (Contd)

Tentatively Identified Compound[d]	CAS No.[d]	Mol Wt	Ret Time	S4077-A04.233[b] PNL 233[c] (mg/m^3)	(ppbv)	S4077-A05.234[b] PNL 234[c] (mg/m^3)	(ppbv)	S4077-A05.235[b] PNL 235[c] (mg/m^3)	(ppbv)	Means and Standard Deviations (mg/m^3)	St Dev	(ppbv)	St Dev
1,1,3-Trimethylcyclohexane	3073-66-3	126	28.45	0.20	35	0.22	39	0.23	42	0.22	0.02	38.6	3.5
3-Heptanone	106-35-4	114	29.47	0.41	81	0.44	86	0.45	88	0.43	0.02	85.1	3.7
2-Heptanone	110-43-0	114	29.60	0.46	90	0.47	92	0.47	92	0.47	0.01	91.4	1.6
Nonane	111-84-2	128	31.08	0.40	69	0.42	73	0.41	72	0.41	0.01	71.4	1.9
6-Methyl-2-Heptanone	928-68-7	128	32.86	0.75	131	0.77	135	0.77	135	0.76	0.01	133.8	2.3
C3-substituted Cyclohexane		126	33.06	0.13	23	0.14	25	0.14	25	0.14	0.01	24.3	1.0
2-Octanone	111-13-7	128	34.52	0.20	35	0.21	36	0.21	36	0.20	0.00	35.7	0.6
C4-substituted Cyclohexane		140	34.70	0.17	27	0.19	30	0.18	29	0.18	0.01	28.5	1.5
Unknown C11 Alkane		156	36.93	0.42	60	0.45	64	0.44	63	0.44	0.02	62.6	2.3
C4-substituted Cyclohexane		140	37.83	0.27	44	0.30	47	0.38	61	0.32	0.06	50.7	9.3
Unknown C11 Alkane		156	39.01	0.13	18	0.13	19	0.13	19	0.13	0.00	18.9	0.4
Undecane	1120-21-4	156	40.16	1.38	198	1.44	206	1.44	207	1.42	0.04	204	5.1
Unknown methyl decahydronaphthalene		152	41.81	0.17	25	0.24	35	0.24	36	0.22	0.04	31.8	5.9
Pentylcyclohexane,	4292-92-6	154	42.20	0.28	40	0.30	44	0.30	44	0.29	0.02	42.7	2.2
Unknown C12 Alkane		170	42.44	0.21	27	0.22	30	0.22	29	0.22	0.01	28.5	1.1
Unknown C12 Alkane		170	42.82	0.32	42	0.35	46	0.34	44	0.33	0.01	44.1	1.6
Dodecane	112-40-3	170	44.20	2.91	384	3.06	403	2.86	377	2.95	0.10	388	13.4
Decahydro-2,6-Dimethylnaphtalene	1618-22-0	166	44.70	0.17	23	0.19	25	0.19	25	0.18	0.01	24.4	1.2
Unknown C13 Alkane		184	44.84	1.75	213	1.88	229	1.75	214	1.80	0.07	219	9.1
Decahydro-2,3-Dimethylnaphthalene	1008-80-6	166	45.90	0.20	27	0.22	30	0.21	28	0.21	0.01	28.3	1.3
C7-substituted Cyclohexane		182	46.25	0.96	118	1.03	127	0.97	119	0.98	0.04	121.0	4.8
Unknown			46.51	0.32	(f)	0.35	(f)	0.32	(f)	0.33	0.02	(e)	(e)
Unknown Alkane		184	46.67	0.51	62	0.56	68	0.48	58	0.51	0.04	62.4	4.9
Unknown Alkane			46.97	0.33	(f)	0.37	(f)	0.32	(f)	0.34	0.02	(e)	(e)
Unknown Alkane			47.11	2.76	(f)	2.93	(f)	2.52	(f)	2.74	0.21	(e)	(e)
Unknown C4 Alkene/Cycloalkane		182	47.56	<0.08	< 10.0	<0.08	< 10.0	0.13	16	(e)	(e)	(e)	(e)
Tridecane	629-50-5	184	47.98	4.12	502	4.17	508	3.57	434	3.96	0.34	482	40.9
Unknown Alkene/Cycloalkane		182	48.18	0.15	19	0.17	21	0.15	18	0.16	0.01	19.4	1.3
Unknown Alkene/Cycloalkane		196	48.38	0.50	57	0.57	65	0.48	55	0.52	0.05	59.0	5.2
Unknown Alkane			48.75	0.61	(f)	0.64	(f)	0.54	(f)	0.60	0.05	(e)	(e)

BY-107 Table 3.2 (Contd)

Tentatively Identified Compound[d]	CAS No.[d]	Mol Wt	Ret Time	S4077-A04.233[b] PNL 233[c] (mg/m^3)	S4077-A04.233[b] PNL 233[c] (ppbv)	S4077-A05.234[b] PNL 234[c] (mg/m^3)	S4077-A05.234[b] PNL 234[c] (ppbv)	S4077-A05.235[b] PNL 235[c] (mg/m^3)	S4077-A05.235[b] PNL 235[c] (ppbv)	Means and Standard Deviations (mg/m^3)	St Dev	(ppbv)	St Dev
Unknown Alkene/Cycloalkane		182	50.12	0.65	80	0.70	86	0.58	72	0.65	0.06	79.6	7.3
2-Methyltridecane	1560-96-9	198	50.29	0.42	48	0.42	48	0.36	41	0.40	0.03	45.4	3.9
3-Methyltridecane	6418-41-3	198	50.57	0.24	27	0.24	27	0.21	24	0.23	0.01	25.9	1.5
Unknown C15 Alkane		212	50.86	2.51	265	2.50	264	2.16	229	2.39	0.20	253	20.9
Tetradecane	629-59-4	198	51.50	2.35	266	2.29	259	2.08	236	2.24	0.14	254	15.9
Unknown Alkene/Cycloalkane		210	52.24	0.25	27	0.27	29	0.23	24	0.25	0.02	26.7	2.5
Unknown Alkane		198	53.72	0.40	45	0.41	46	0.40	45	0.40	0.01	45.6	0.8
Pentadecane	629-62-9	212	54.81	< 0.10	< 10.0	< 0.10	< 10.0	0.12	13	(e)	(e)	(e)	(e)

(a) Semi-quantitative estimate calculated using concentration of closest eluting ID.

(b) WHC sample identification number.

(c) PNL SUMMA™ canister number.

(d) Obtained by mass spectral interpretation amd comparison with the EPA/NIST/WILEY Library.

(e) Average and standard deviation are not meaningful for this analyte.

Table 3.3 Positively Identified and Quantitated TO-14 Extended Analytes[a] for Ambient Air Collected Near Tank BY-107 in SUMMA™ Canisters on 10/26/94

			Upwind S4077-A01.231[b] PNL 231[c] Concentration		Through VSS S4077-A02.232[b] PNL 232[c] Concentration	
Analyte	CAS No.	Mol Wt	(mg/m^3)	(ppbv)	(mg/m^3)	(ppbv)
Acetone	67-64-1	58	0.06	23.0	0.15	57.1

(a) TO-14 plus 15 additonal analytes.
(b) WHC sample identification number.
(c) PNL canister number.

Table 3.4 Tentatively Identified Compounds and Estimated Concentrations(a) for Ambient Air Collected Near Tank BY-107 in SUMMA™ Canisters on 10/26/94

				Upwind S4077-A01.231(b) PNL 231(c)		Through VSS S4077-A02.232(b) PNL 232(c)	
Tentatively Identified Compound(d)	CAS No.(d)	Mol. Wt	Ret. Time	(mg/m^3)	(ppbv)	(mg/m^3)	(ppbv)
Acetaldehyde	75-07-0	44	5.15	0.13	65.2	0.24	123.2
Ethanol	64-17-5	46	7.49	0.19	91.5	< 0.02	< 10.0
Methylcyclopentane	96-37-7	84	16.51	0.13	35.2	< 0.04	< 10.0

(a) Semi-quantitative estimate calculated using concentration of closest eluting ID.

(b) WHC sample identification number.

(c) PNL SUMMA™ canister number.

(d) Obtained by mass spectral interpretation amd comparison with the EPA/NISTand WILEY Libraries.

Table 3.5 Permanent Gas Analysis Results for Samples Collected from the Headspace of Tank BY-107 and for Ambient Air Collected Near Tank BY-107 in SUMMA™ Canisters on 10/29/94

Permanent Gas	Ambient Air Upwind S4077-A01.231[a] PNL 231[b] Concentration (ppmv)	Ambient Air Through VSS S4077-A02.232[a] PNL 232[b] Concentration (ppmv)	S4077-A04.233[a] PNL 233[b] Concentration (ppmv)	S4077-A05.234[a] PNL 234[b] Concentration (ppmv)	S4077-A06.235[a] PNL 235[b] Concentration (ppmv)
Hydrogen	<80	<80	<80	<80	<80
Methane	<20	<20	<20	<20	<20
Carbon Dioxide	398	399	111	63.8	107
Carbon Monoxide	<20	<20	<20	<20	<20
Nitrous Oxide	<20	<20	587	658	618

(a) WHC sample identification number.

(b) PNL canister number.

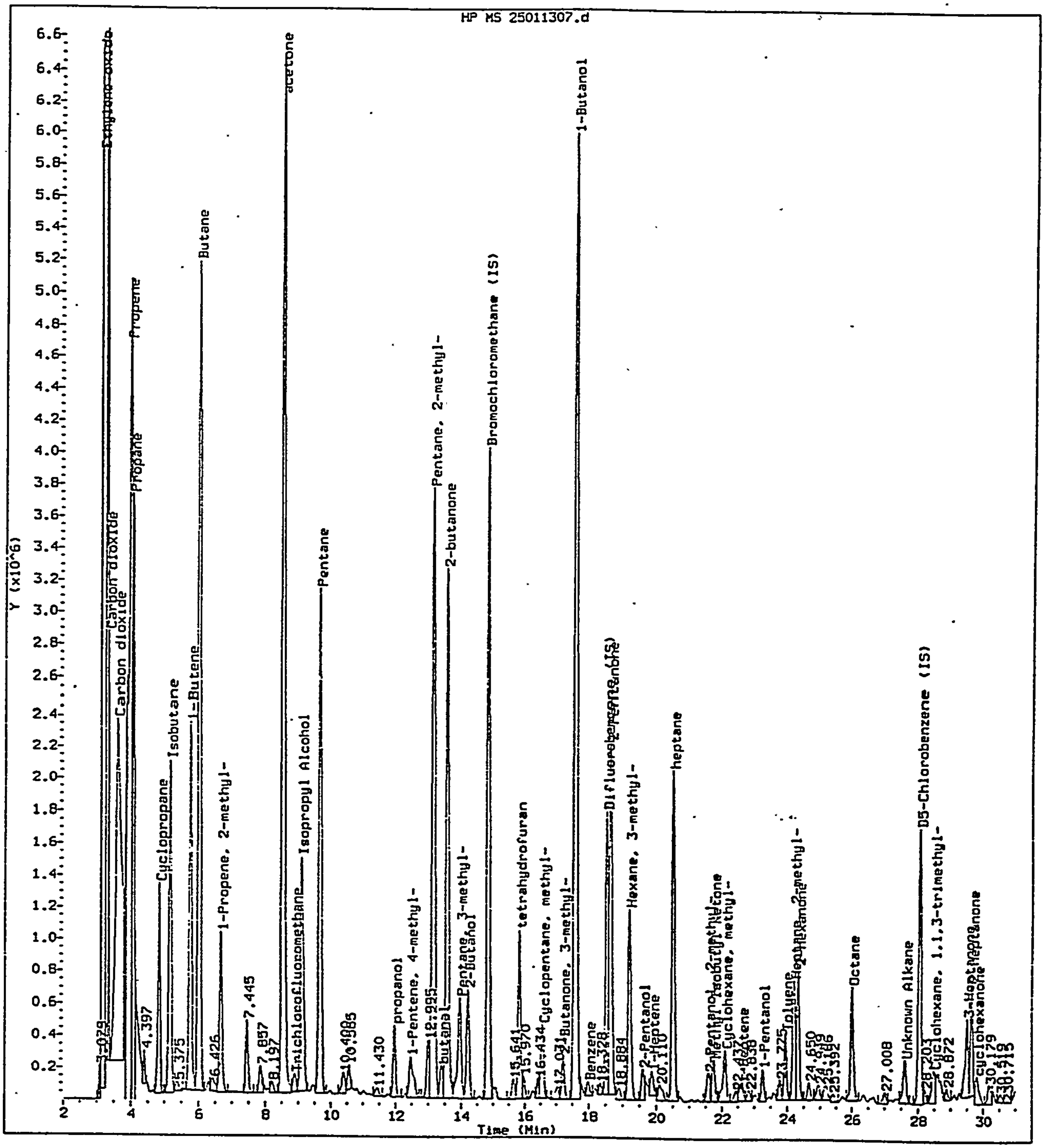

Figure 3.1a Total Ion Chromatogram (2 - 30 min) for Hanford Waste Tank BY-107 SUMMA™ Canister Sample S4077-A04-233 Collected on 10/26/94

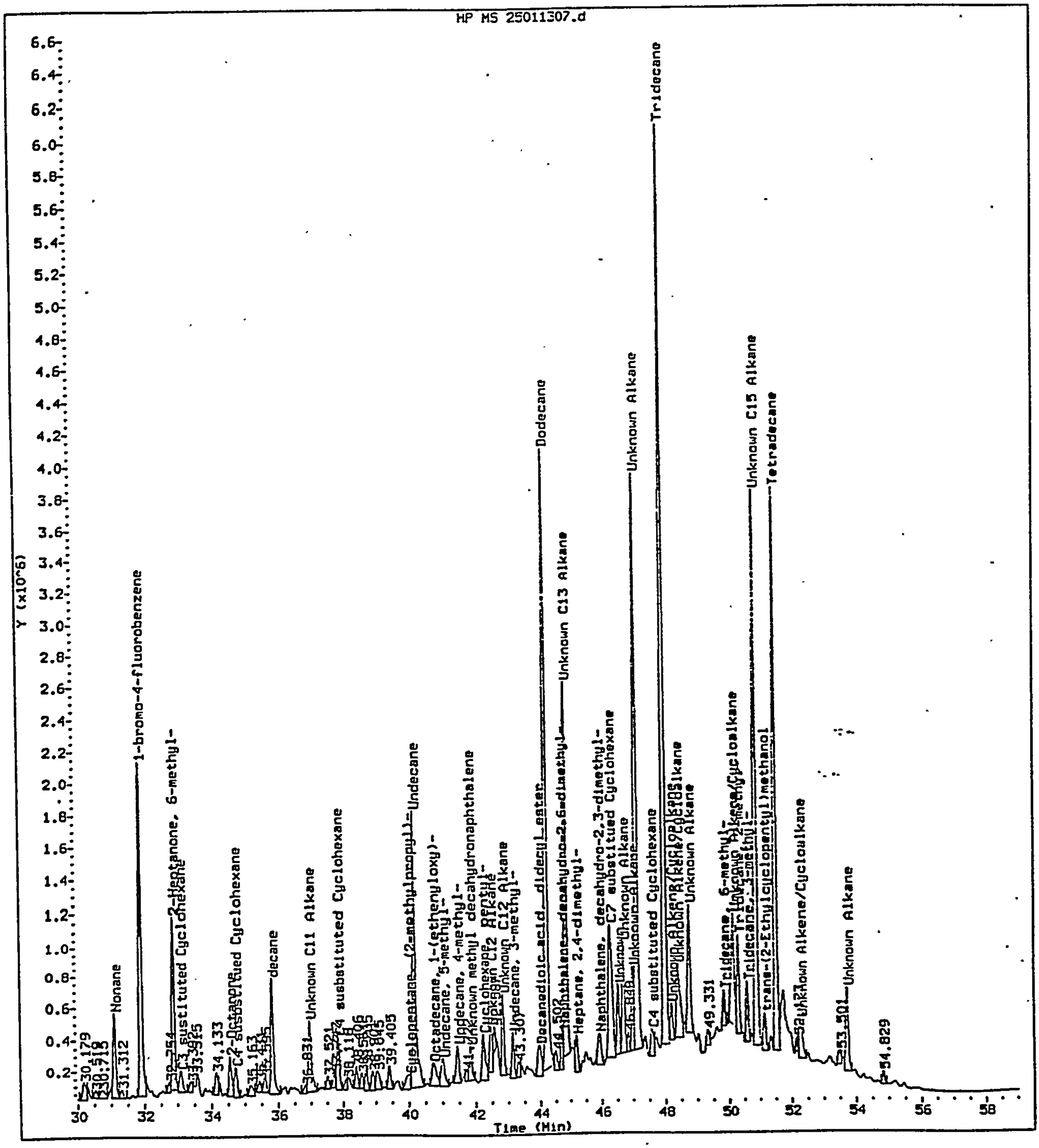

Figure 3.1b Total Ion Chromatogram (30 - 58 min) for Hanford Waste Tank BY-107 SUMMA™ Canister Sample S4007-A04-233 Collected on 10/26/94

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