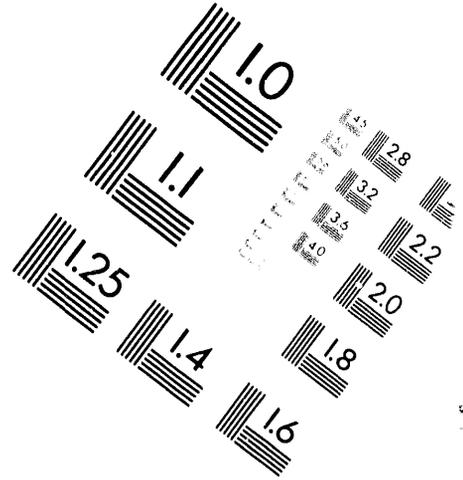
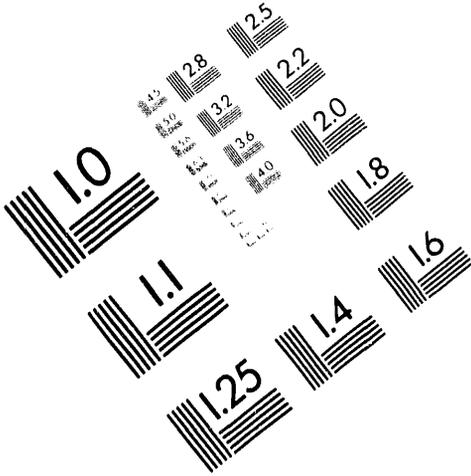




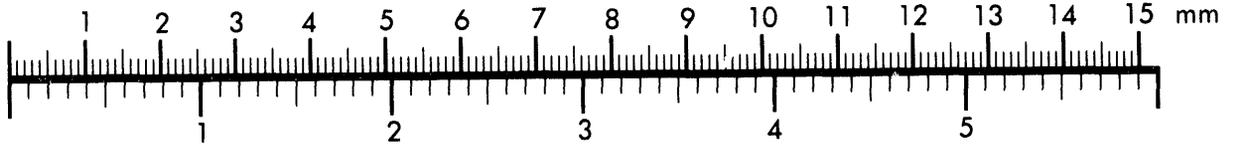
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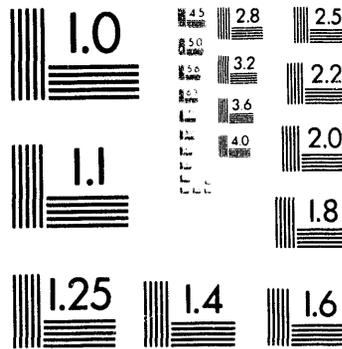
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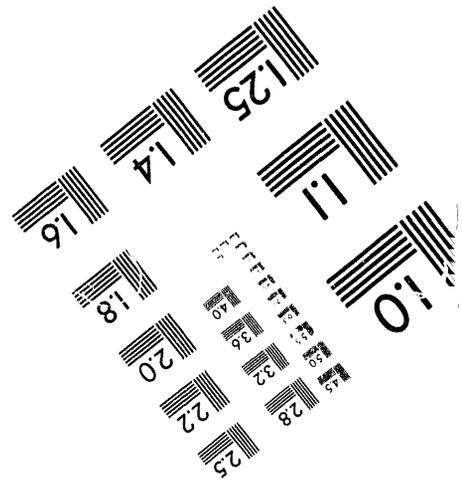
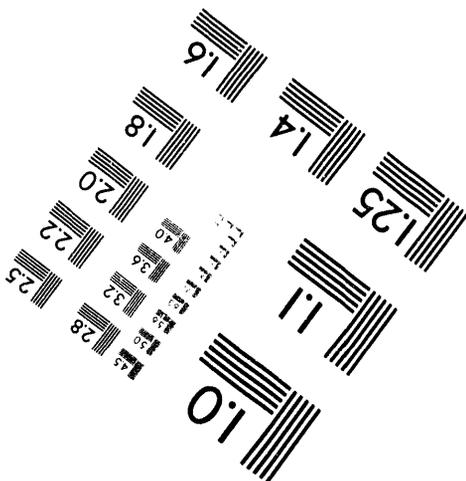
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Vapor Characterization of Tank 241-C-103

J. L. Huckaby
Westinghouse Hanford Company

M. S. Story
Northwest Instrument Systems Inc.

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**Westinghouse
Hanford Company**

P.O. Box 1970
Richland, Washington 99352

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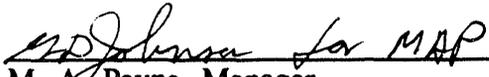
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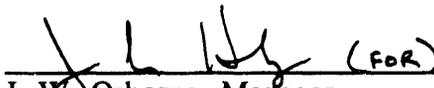
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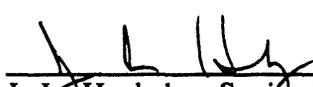
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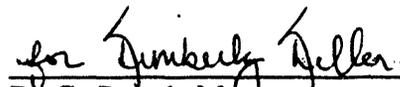
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Approved by:  6/28/94
M. A. Payne, Manager Date
Waste Tank Safety Programs

Approved by:  (FOR) 6/28/94
J. W. Osborne, Manager Date
Tank Vapor Issue Resolution Program

Prepared/
Approved by:  6/28/94
J. L. Huckaby, Senior Engineer Date
Tank Vapor Issue Resolution Program

Reviewed/
Approved by:  6/20/94
H. Babad, Advisory Chemist Date
Waste Tank Safety Programs

Reviewed by:  6/28/94
D. C. Board, Manager Date
Tank Waste Quality Engineering

EXECUTIVE SUMMARY

The Westinghouse Hanford Company Tank Vapor Issue Resolution Program has developed, in cooperation with Northwest Instrument Systems, Inc., Oak Ridge National Laboratory, Oregon Graduate Institute of Science and Technology, Pacific Northwest Laboratory, and Sandia National Laboratory, the equipment and expertise to characterize gases and vapors in the high-level radioactive waste storage tanks at the Hanford Site in south central Washington State. This capability has been demonstrated by the characterization of the tank 241-C-103 headspace. This tank headspace is the first, and for many reasons is expected to be the most problematic, that will be characterized (Osborne 1992).

Results from the most recent and comprehensive sampling event, sample job 7B, are presented for the purpose of providing scientific bases for resolution of vapor issues associated with tank 241-C-103. This report is based on the work of Clauss et al. 1994, Jenkins et al. 1994, Ligothke et al. 1994, Mahon et al. 1994, and Rasmussen and Einfeld 1994. No attempt has been made in this report to evaluate the implications of the data presented, such as the potential impact of headspace gases and vapors to tank farm workers' health. That and other issues will be addressed elsewhere.

Key to the resolution of worker health issues is the quantitation of compounds of toxicological concern. The Toxicology Review Panel, a panel of Pacific Northwest Laboratory experts in various areas of toxicology, has chosen 19 previously identified compounds as being of potential toxicological concern. During sample job 7B, the sampling and analytical methodology was validated for this preliminary list of compounds of toxicological concern. Validation was performed according to guidance provided by the Tank Vapor Conference Committee, a group of analytical chemists from academic institutions and national laboratories assembled and commissioned by the Tank Vapor Issue Resolution Program.

Sampling results for the 19 preliminary compounds of toxicological concern are summarized in Table ES-1. The table lists the compounds, sampling method, number of samples, the average or range of measured concentration, and estimated uncertainties. Three analytes previously identified in the tank 241-C-103 headspace are listed in the table, although they were not detected in sample job 7B. These analytes are: nitrogen dioxide, sulfur oxides, and vinylidene chloride. Other compounds quantitatively measured in the tank headspace include hydrogen, water, carbon monoxide, and selected ketones, nitriles, and aromatics. Many other detected compounds were tentatively identified, and their concentrations estimated.

The tank 241-C-103 headspace was also determined to be nearly saturated with water vapor, having a dewpoint of about 36.3 °C, while the headspace itself is about 38 °C. Gas

and vapor samples taken from 3 elevations of the headspace show no indication of vertical stratification. Comparisons of results from a 6-month period indicate that the headspace composition does not change significantly over short (24-hr) or long (90-day) periods.

This report and the data on which it is based will be reviewed by the Tank Vapor Conference Committee. The Toxicology Review Panel will also review this report and issue a final list of analytes of toxicological concern for tank 241-C-103.

In addition to specifically characterizing the tank 241-C-103 headspace, results discussed in this report indicate the general applicability of the sampling and analytical methodology. Validation study results indicate the methodology used can account for even the complex tank 241-C-103 chemical matrix, and that it may apply by extension to the many waste tanks with similar but less concentrated constituents.

Table ES-1. Preliminary Compounds of Toxicological Concern

Compound	Sample Type	n ¹	Mean or Range (ppmv ²)	σ^3 (ppmv)
Acetone	SUMMA ⁴	10	19.2 - 19.4	2.0
	TST ⁵	4	8.8	3.9
Acetonitrile	SUMMA TM	10	12.7 - 13.2	1.2
	TST	4	9.1	2.2
Ammonia	Sorbent Trap	35	304	11
Benzene	SUMMA TM	21	<0.01 - 0.33	0.11
	TST		0.08	0.03
1,3-Butadiene	SUMMA TM	10	<0.05 - 0.060	0.020
Butanal	SUMMA TM	10	4.4 - 4.7	0.7
	TST	4	1.2	0.8
n-Butanol	SUMMA TM	10	13.1	2.5
	TST	4	28.4	6.1
n-Dodecane	OVS ⁶	29	36.2 - 46.4	-
	CCT ⁷	10	40.3	7.2
n-Hexane	SUMMA TM	10	0.71 - 0.72	0.1
	TST	4	0.80	0.06
2-Hexanone	SUMMA TM	10	0.57 - 0.59	0.05
	TST	4	0.51	0.12
Methylene Chloride	SUMMA TM	21	<0.02 - 0.061	0.030
	TST	4	1.62	1.47
Nitric Oxide	Sorbent Trap	8	1.5	0.3
Nitrogen Dioxide	Sorbent Trap	18	<0.06	-
Nitrous Oxide	SUMMA TM	37	763	51
Propanenitrile	SUMMA TM	10	5.1 - 5.3	0.7
	TST	4	3.3	0.3
Sulfur Oxides	Sorbent Trap	3	<0.02	-
Tributyl Phosphate	CCT	5	0.51	0.05
n-Tridecane	OVS	29	40.1 - 63.0	-
	CCT	10	52.0	3.7
Vinylidene Chloride	SUMMA TM	21	<0.02	-
	TST	4	<0.009	-

¹n = Number of samples

²Parts per million by volume.

³ σ = Standard deviation

⁴SUMMA is a registered trademark of Molexics, Inc., Cleveland, Ohio. (SUMMA canisters are evacuated containers that are filled with an air sample.) Analyzed at Oregon Graduate Institute of Science and Technology.

⁵Triple sorbent trap. Prepared and analyzed at Oak Ridge National Laboratory.

⁶OSHA versatile sampler. Analyzed at Pacific Northwest Laboratory.

⁷Carbotrap/Carbotrap C sorbent trap. Prepared and analyzed at Oak Ridge National Laboratory.

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LIST OF TERMS

CCT	Carbotrap/Carbotrap C
DAAMS	Depot Area Air Monitoring System
DBBP	Dibutylbutyl Phosphonate
DOE	U.S. Department of Energy
EPA	Environmental Protection Agency
FID	Flame Ionization Detector
GC	Gas Chromatograph
HEPA	High-Efficiency Particulate Air (Filter)
MS	Mass Spectrometer
NPH	Normal Paraffinic Hydrocarbons
OGIST	Oregon Graduate Institute of Science and Technology
ORNL	Oak Ridge National Laboratory
OVS	OSHA Versatile Sampler
PNL	Pacific Northwest Laboratory
ppmv	Parts Per Million by Volume
PUREX	Plutonium-Uranium Extraction (Process)
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
TBP	Tributyl Phosphate
TRP	Toxicology Review Panel
TST	Triple Sorbent Trap
TVCC	Tank Vapor Conference Committee
USQ	Unreviewed Safety Question
Vapor Program	Tank Vapor Issue Resolution Program
VSS	Vapor Sampling System
WHC	Westinghouse Hanford Company

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The authors wish to acknowledge the hard work and dedication of the many people who participated in the collection and analysis of samples. Among those deserving special recognition are the lead samplers, R. D. Mahon and C. M. Jones, who are directly responsible for the high quality of the samples discussed in this report. Sample analysis was given high priority and special attention by the participating laboratories, Oak Ridge National Laboratory, Oregon Graduate Institute of Science and Technology, Pacific Northwest Laboratory, and Sandia National Laboratory. Our thanks to the many analysts and their leaders for the high quality work performed.

1.0 INTRODUCTION

1.1 PURPOSE

This document describes and summarizes available data on vapors and gases above the high-level radioactive waste stored in tank 241-C-103 at the Hanford Site in south central Washington State. The data are presented for the purpose of providing a scientific basis for the resolution of worker health and safety issues associated with tank 241-C-103.

1.2 SCOPE

This document reports the best available data on vapors and gases in the headspace of tank 241-C-103. Implications of the data presented, such as the potential impact of headspace gases and vapors to tank farm workers' health, or chemical reactions that may occur in the vapor phase, are not within the scope of this document.

1.3 BACKGROUND

1.3.1 Tank Description and Contents

Tank 241-C-103 is a 2,017-kL (533-kgal) underground storage tank at the Hanford Site in south central Washington State. It is a reinforced-concrete tank with a steel liner covering the waste-bearing surfaces. The dome of the tank is covered by about 2 m of soil, and supports 10 vertical steel pipes (risers) through which the tank contents may be accessed.

Tank 241-C-103 contains approximately 738 kL (195 kgal) of sludge and aqueous liquid high-level radioactive waste (Hanlon 1994). Floating on the aqueous waste are an estimated 15 to 21 kL (4.1 to 5.5 kgal) of organic liquid waste (Huckaby 1994a). The organic liquid waste is thought to have originated in the Plutonium Uranium Extraction (PUREX) process (Carothers 1988), and is known to be composed primarily of tributyl phosphate (TBP) and normal paraffinic hydrocarbons (NPH) in the n-dodecane to n-pentadecane series (Pool and Bean 1994).

1.3.2 Tank Vapor Issues

Tank 241-C-103 has historically been associated with nuisance odors, and 10 incidents of worker exposure to tank vapors have been associated with the tank since 1987 (Osborne and Huckaby 1994). These incidents prompted Westinghouse Hanford Company (WHC), which is responsible for work in the tank farms, to establish the Tank Vapor Issue Resolution

Program (Vapor Program) in 1992. It was determined that until the waste tank vapors and gases were adequately characterized, their potential hazard to tank farm workers could not be properly evaluated.

A second significant issue associated with tank 241-C-103 has been the suggestion that a fog of organic liquid droplets could exist in the tank headspace and constitute a flammability hazard (Trent 1990). The headspace flammability was addressed in an engineering assessment that indicated it to be an unlikely hazard (Huckaby and Estey 1992); however, the issue could not be closed without supporting characterization data.

Tank 241-C-103 was consequently chosen as the Vapor Program's highest priority tank (Osborne 1992), and to be the first tank for comprehensive headspace gas and vapor characterization. Though the Vapor Program's first priority has been worker health protection, resolution of the flammability issue associated with tank 241-C-103 was required before other intrusive tank sampling could begin.

External review and oversight of the tank 241-C-103 headspace characterization project was provided by the Tank Advisory Panel's Subpanel on Worker Health and Safety, the Waste Management External Advisory Committee, and the Defense Nuclear Facility Safety Board (Osborne and Huckaby 1994).

Two committees were formed to provide continuous, expert advice and guidance integral to the characterization strategy (Osborne and Huckaby 1994). The Tank Vapor Conference Committee (TVCC), composed of analytical chemists from academia and national laboratories, was commissioned by the Vapor Program to provide technical guidance on the sampling and analytical strategies. The Toxicology Review Panel (TRP), a panel of experts in various areas of toxicology, was established by the Vapor Program to evaluate headspace characterization data for human health hazards. The TRP is responsible for selecting analytes of toxicological concern, which comprise the list of analytes for quantitation.

1.4 HEADSPACE CHARACTERIZATION CHRONOLOGY

Little was known about the tank 241-C-103 headspace constituents in early 1992, and methods that had been used to characterize the tank had not been satisfactory (Ulbricht 1991, Story 1992). Samples of the headspace constituents were needed in order to test and optimize proposed analytical techniques, and analytical results were needed in order to design sampling equipment. The sampling equipment itself then needed to be tested at the tank, and real tank samples were needed to refine and validate the analytical techniques. Early samples indicated a large number of compounds was present in the tank headspace, and it became necessary to limit quantitation of analytes to only the most toxicologically significant compounds.

This section briefly describes the sequence of sampling events conducted to develop the sampling and analytical methodologies and then to characterize tank 241-C-103.

1.4.1 Sample Job 3

Preliminary samples of the gases and vapors in tank 241-C-103's headspace were taken from a port on the ventilation riser and through a high-efficiency particulate air (HEPA) filter in August 1992. Though the riser was purged with tank air before sampling and the sampling manifold itself was heated (Jones 1992), the samples were not considered to be representative of the headspace since condensation of vapors on the walls of the in-tank transfer tubing could not be prevented. This sampling event, sample job 3, was conducted to test sampling and analytical methods and to obtain as much qualitative and semiquantitative information as possible.

Analysis of sample job 3 SUMMA¹ canister samples at the Oregon Graduate Institute of Science and Technology (OGIST) identified 70 organic compounds (Einfeld et al. 1992a). Pacific Northwest Laboratory (PNL) analyzed cryogenically concentrated samples, positively identified 9 compounds, and tentatively identified another 10 compounds (Jones 1992). Oak Ridge National Laboratory (ORNL) supplied and analyzed several types of sorbent trap samples (Jenkins et al. 1993). Among the previously unidentified compounds observed in these samples were 8 normal aliphatic nitriles in the acetonitrile to n-nonanenitrile series.

1.4.2 Sample Jobs 4 and 5

After sample job 3 in August 1992, further tank 241-C-103 headspace characterization efforts were delayed by two significant events: First, the presence of the separable organic phase in tank 241-C-103 was declared an unreviewed safety question (USQ) in September 1992 (Richardson 1992). The USQ resulted in suspension of all intrusive activities on tank 241-C-103, including vapor sampling, until an environmental assessment was issued by the Department of Energy (DOE) in July 1993 (DOE 1993). Second, in August 1993, almost immediately after the environmental assessment was issued, an administrative hold was placed on tank farm activities to allow reassessment and improvement of tank farm worker safety (Alumkal 1993). This administrative hold was gradually lifted as Tank Farm Operations demonstrated the ability to conduct operations safely, with vapor sampling events at tank 241-C-103 in November 1993 being among the first significant jobs to occur after the hold was placed.

In November 1993, sample jobs 4 and 5 were performed to determine whether the nitriles discovered in August 1992 originated from the waste, or from the HEPA filter on the ventilation riser (Huckaby 1993). Specifically, it had been proposed that chemical or radiolytic degradation of the urethane seals in the HEPA filter could produce the nitriles, and high concentrations of nitriles were observed in the August 1992 samples because these samples had been collected downstream from the HEPA filter.

¹SUMMA is a registered trademark of Molecetrics, Inc., Cleveland, Ohio.

Samples were collected for sample job 4 from the same port of the ventilation riser as had been used in August 1992. The same number and type of samples were collected for sample job 5, but these were collected from a port on a different riser with no intervening HEPA filter. As with the August 1992 samples, samples from jobs 4 and 5 were not considered to be representative of the tank headspace with respect to condensible or condensate soluble compounds; however, they did clearly indicate that the nitriles were not associated with the HEPA filter, as have subsequent sampling results.

Sample jobs 4 and 5 presented the first opportunity to obtain samples from tank 241-C-103 since August 1992. Development of analytical methods had proceeded to its logical limit in the absence of tank samples, and sample jobs 4 and 5 provided samples needed to test these methods. Sample jobs 4 and 5 also tested the logistics of vapor sampling in tank farm 241-C, and sample handling and shipment.

1.4.3 Sample Job 6

Sample job 6, performed in December 1993, involved the first intrusive work in tank 241-C-103 allowed after declaration of the USQ. One objective of sample job 6 was the determination of NPH vapor and aerosol concentrations to assess headspace flammability. To ensure that NPH samples were unaffected by sample transport problems, the sampling devices (OSHA versatile sampler [OVS] traps) were themselves lowered into the tank headspace. Ligothke et al. 1993 documents the design and testing of the sampling system, and Ligothke et al. 1994a present and discuss results obtained from sample job 6. These results have also been incorporated in an assessment of the tank 241-C-103 headspace flammability (Huckaby 1994b), and will be briefly reviewed in Section 4.0.

Other objectives of sample job 6 were to collect aqueous and organic liquid waste samples and measure the depth of the organic layer. These objectives were met, with approximately 500 mL and 100 mL of the organic and aqueous liquids collected, respectively. The organic layer was determined to be between 3.8 and 5 cm thick (Huckaby 1994a). Analysis of the liquid samples was performed at PNL and WHC (Pool and Bean 1994). Of particular interest in the present study is the analysis of vapor above an aliquot of the organic liquid sample held at the temperature of the tank waste. Data from this analysis is in good agreement with vapor samples collected during sample job 6.

1.4.4 Sample Job 6B

The objective of sample job 6B, which occurred in January 1994, was to collect representative gas and vapor samples from the tank 241-C-103 headspace. To collect representative vapor samples from waste tanks having high water or organic vapor concentrations, the Vapor Program developed the vapor sampling system (VSS). Although

the VSS was operationally ready for use before sample jobs 4 and 5 were performed, it could not be deployed until the tank 241-C-103 headspace flammability was assessed by sample job 6.

Samples from job 6B were analyzed to obtain semi-quantitative data on targeted inorganic compounds, and a list, with estimated concentrations, of all organic compounds present in the headspace above the analytical limits of detection. The VSS was used, as depicted in Figure 1-1, to collect 97 samples from a location about 0.79 m above the waste surface.

The sample job 6B event and the analytical results were reviewed by the TVCC, which advised the Vapor Program on the adequacy of sampling and analytical methods, and suggested improvements (Story 1994). The TRP then reviewed the analytical results and the TVCC report, and determined the following preliminary list of compounds as being of potential toxicological concern to workers (Goheen 1994):

Acetone	Methylene Chloride
Acetonitrile	Nitric Oxide
Ammonia	Nitrogen Dioxide
Benzene	Nitrous Oxide
1,3-Butadiene	Propanenitrile
Butanal	Sulfur Oxides, as Sulfur Dioxide
n-Butanol	Tributyl Phosphate
n-Dodecane	n-Tridecane
n-Hexane	Vinylidene Chloride
2-Hexanone	

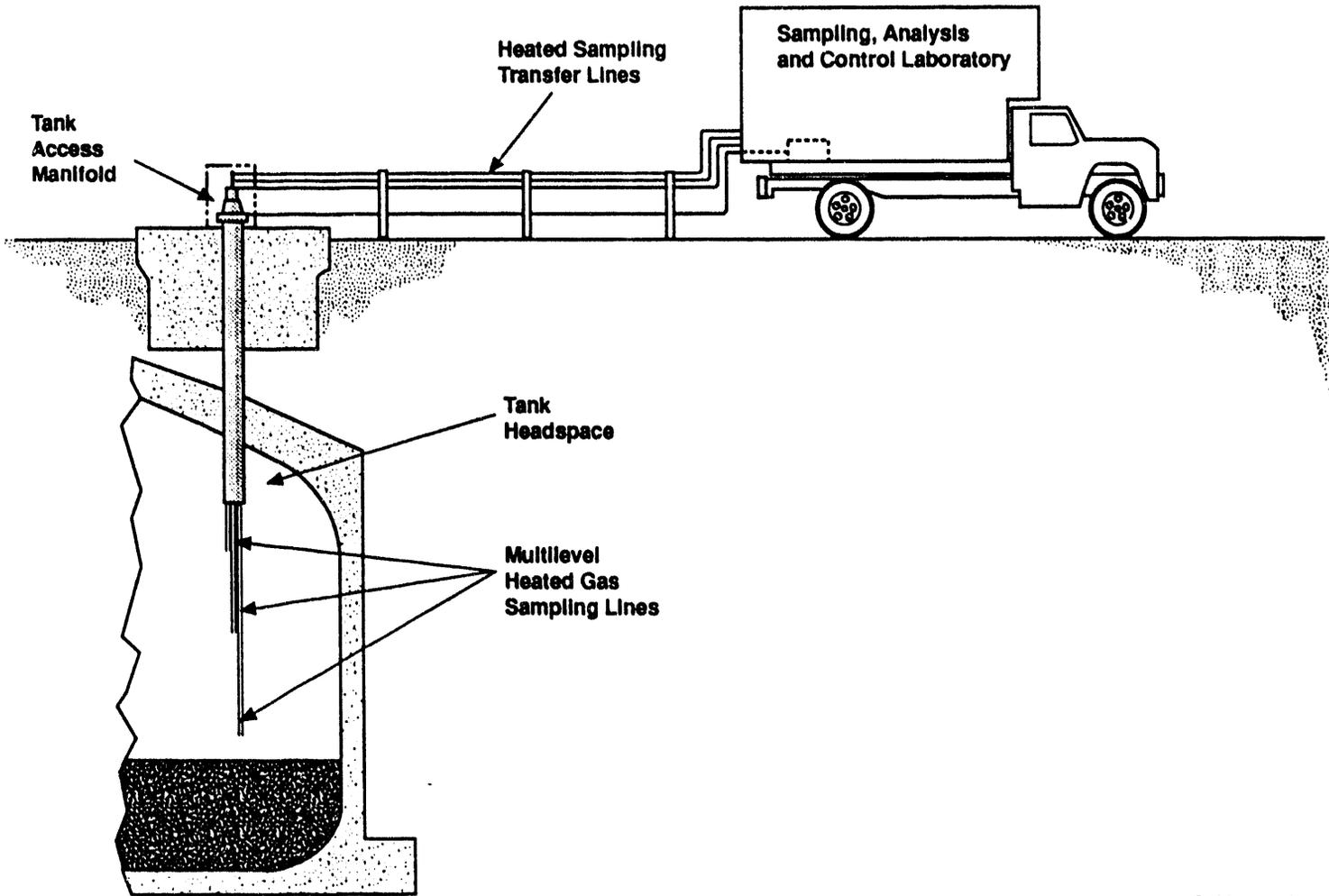
The compounds in this list became the focus of the sampling and analysis methodologies validation. This validation study was undertaken in May 1994 as sample job 7B.

While meeting its objectives, sample job 6B was also the first time that the VSS was deployed for waste tank sampling, and problems related to lack of experience were observed. Unanticipated logistical and procedural problems, for example, were encountered in sample job 6B. Also, analytical data suggested that the connection between the sampling manifold and the sampling devices needed additional heating to prevent vapor condensation in those locations (Story 1994). Minor changes to the VSS were subsequently made to eliminate these potential cold spots.

1.4.5 Sample Job 7A

Sample job 7A, which occurred in April 1994, was performed to verify that procedural and equipment modifications made following sample job 6B did indeed rectify all problems. Sample job 7A results indicated the system corrections had improved sampling

Figure 1-1. Vapor Sampling System.



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consistency; however, other procedural problems were discovered. These were addressed by introducing: 1) a sampling checklist for each sample collected; and 2) the capability to download VSS pressure and flow data to a portable computer.

1.4.6 Sample Job 7B

The objectives of sample job 7B were to characterize the tank 241-C-103 headspace with known standard additions to the samples, and to determine whether the headspace constituents were vertically stratified. Sample job 7B was performed in May, and required seven days of sampling spread over a two-week period. At the start of sample job 7B, samples were collected to test the procedural and equipment changes that followed sample job 7A. Spiked samples from ORNL, PNL, and OGIST were then collected to validate the recovery of the TRP analytes of concern from the chemical matrix of real tank samples. Headspace stratification was addressed by collecting ammonia, hydrogen, nitrous oxide, and NPH samples from three different elevations of the headspace (see Figure 1-1). Mahon et al. 1994 reports the sample job 7B event sequence and sampling data.

The sampling and analytical methodology is described in Section 3.0. In this section, the principles, preparation, handling, and analysis of sampling devices used in sample job 7B are discussed. Analytical results from sample job 7B are given and summarized in Sections 4.0 and 5.0, respectively.

1.5 DATA QUALITY OBJECTIVES

Objectives of the sampling and sample analyses of tank 241-C-103 headspace gases and vapors are specified in *Tank 241-C-103 Vapor and Gas Sampling DQOs* (Osborne et al. 1994). That document provides guidelines for the number and type of samples required to resolve headspace flammability and vapor toxicity issues. Data from sample jobs 4, 5, and 6 have been used to address the flammability issue (Huckaby 1994b), and selected data are presented in Section 4.0. Data from sample jobs 6B, 7A, and 7B, presented and discussed in this report, will be used to address vapor toxicity issues. It is not within the scope of this report to assess the adequacy of the results for resolving vapor toxicity issues, and will be the subject of future documentation.

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2.0 DATA QUALITY

Quality assurance guidelines for the tank 241-C-103 gas and vapor sampling and analysis are specified in *Quality Assurance Project Plan for Waste Tank Characterization* (Suydam 1993). This quality assurance project plan (QAPjP) governs the developmental phase of waste tank gas and vapor sampling and analysis by the Vapor Program, and specifically includes tank 241-C-103. In addition to general quality assurance (QA) guidance, the QAPjP establishes QA objectives for validation of this sampling and analysis technology.

Because tank 241-C-103 was the first tank to be characterized, it was used to test, refine, and validate the equipment and methods developed. The QAPjP discusses the following 6 QA objectives for validation of the sampling and analysis methodology:

- Accuracy
- Precision
- Validation
- Representativeness
- Completeness
- Comparability

Accuracy, precision, validation, and sample representativeness as QA objectives are discussed in the following sections. Completeness, in this context, refers to the ratio of the number of valid reported data to the number of expected data. Establishing an appropriate completeness is an aspect of the methodology development, but will not be addressed here because of the developmental nature of the task. Comparability, as a QA objective, refers to the degree that results from different sampling or analytical methods, or laboratories, agree. While certain organic vapor data comparisons can be and are made in Section 4.0, redundant methods and laboratory capabilities have not been developed because of time constraints.

2.1 ACCURACY

Analytical accuracy is defined as the nearness of a measurement to the true value and is evaluated by analysis of suitable reference standards (Suydam 1993). Suitable reference standards are reference materials that contain the analytes of interest at the concentration of interest and in the same chemical matrix as the real sample. In lieu of suitable reference standards, analytical accuracy can be determined from the analysis of spiked tank headspace samples (i.e., tank samples spiked with analytes or surrogates).

It is critical that, before analysis of unknown samples, the analytical measurement system is capable of measuring, with acceptable accuracy, a mixture of known compounds. This mixture may consist of either target analytes or a mixture of surrogate compounds.

Surrogates, in this context, are compounds that have a predictable behavior in the measurement process. Thus, for analyte quantitation, the analytical system response to target analytes must be within a known range.

2.2 PRECISION

Precision is the agreement between a set of measurements, and is a fundamental criteria used to evaluate the usefulness of data. Sampling and analytical precision are addressed by collecting replicate samples of all analytes. When possible, replicate analyses of individual samples are performed to address analytical method precision.

2.3 VALIDATION

Validation of the vapor sampling and analysis methodology is accomplished by determining certain sampling and analysis parameters. When these parameters are known, and determined to be satisfactory, the results are considered to be validated.

The following sample composition parameters are addressed by the sampling strategy and appropriate laboratory analyses:

- Proof that samples are not contaminated, through the use of trip blanks;
- Loss of analytes from samples, through the use of spiked blanks;
- Sorbent trap sampling efficiency, through the use of breakthrough sorbent sections;
- Sample holding times and conditions, as specified or reported;
- Sample chain-of-custody requirements;
- Sample composition as a function of time;
- Sample composition as a function of sample location;
- VSS calibration and performance verification.

The following sample analysis parameters are determined and reported by the analytical laboratories:

- Accuracy, in terms of analyte recovery from laboratory blanks, spiked samples, and/or surrogate spiked samples;
- Precision, in terms of the relative standard deviation of either replicate field samples or spike addition samples;
- Detection limit for each analyte;
- Useful concentration range for each analyte;
- Adjustments for blank results;
- Sample recovery from the sampling device.

Validation of the tank 241-C-103 headspace characterization data is based on the evaluation of the parameters listed above. Section 3.0 discusses these parameters in the context of the various sampling devices and their analysis.

Analytical accuracy is affected by poor desorption or extraction of analytes from the sampling device. The spike addition method is used to establish analytical accuracy by determining the recovery of selected analytes from sorbent traps and SUMMA™ canisters. This assesses the potential adverse effects of the other tank 241-C-103 headspace constituents.

In the spike addition method, an appropriate and known quantity of analyte(s) is added to a set of sampling devices (e.g., SUMMA™ canisters, sorbent traps). Tank samples are then collected with unspiked and these spiked sampling devices. The amount of analyte measured in the unspiked sample is then compared to the amount found in the spiked sample to determine if analysis has been affected by the chemical matrix.

The spike addition method used does not, by itself, establish either sampling efficiency (i.e., how much of the analyte present in the sampled air was collected by the sampling device), or analytical adequacy (i.e., accuracy and precision of quantitation). The sampling and analytical methods are considered validated for tank 241-C-103 if: 1) spike recovery from tank samples is consistently acceptable; 2) the accuracy of the analytical system is demonstrated by consistently acceptable recoveries of target analytes or surrogates from spiked laboratory blanks; and 3) the analytical results from duplicate field samples are acceptably repeatable.

2.4 SAMPLE REPRESENTATIVENESS

Evidence is needed that the gas and vapor samples collected from the tank headspace are representative of the headspace. The three questions to be answered are: 1) are samples collected through VSS affected by the VSS itself? 2) does the headspace composition change with time at a rate that negates data usefulness? and 3) is the headspace composition significantly different at different locations in the headspace?

The VSS sample transfer adequacy is addressed by: 1) performing a cleanliness test; 2) purging the VSS with tank air; and 3) demonstrating sample consistency over relatively short and long time periods as well as at different manifold temperatures.

Samples collected from three headspace elevations are used to determine if the headspace gases and vapors are vertically stratified. This data may also be used to evaluate the possibility of lateral concentration gradients.

3.0 SAMPLING AND ANALYTICAL METHODOLOGY

3.1 DESIGN BASIS

Characterizing the gas and vapor samples from the tank 241-C-103 headspace differs from characterizing ambient air in three important ways. First, the sampling and analytical strategy must address the fact that the headspace is nearly saturated with vapors that will condense in unheated sample transfer tubing and even in unheated sampling devices. Second, the sample collecting devices and analytical methods must be effective despite potential interferences from the diverse mixture of chemical compounds present in wide ranges of concentrations and volatilities. And third, the potential radioactive contamination of headspace samples must be addressed. Inasmuch as waste tank headspace sampling and analysis differs from ambient air sampling and analysis, techniques developed for characterizing ambient air may not be valid for characterizing the tank headspace. The following subsections discuss each of these issues in detail.

3.1.1 Vapor Condensation

Data indicate the tank 241-C-103 headspace is nearly saturated with both water and organic vapors from the waste, as has been expected (Huckaby and Estey 1992). Measurements of water vapor indicate the headspace dewpoint was approximately 36.3 °C, when the headspace temperature was 38 °C (see Section 4.3.1). If an unheated tube is used to draw an air stream from the headspace for remote sampling, condensation of vapors will occur wherever tube wall temperatures drop below the 36.3 °C dewpoint of the headspace. Given that ambient temperatures are generally lower than the headspace dewpoint, an unheated tube will not faithfully transfer condensible vapors, nor any gases that are soluble in the condensate.

In addition to remote sampling via the VSS, some vapor samples were collected by lowering sampling devices directly into the headspace of tank 241-C-103. This technique avoided the problem of vapor condensation in transfer tubing by simply eliminating the transfer tubing. These samples were collected in December 1993, and are discussed in Section 4.4.6.

3.1.2 Interferences and Concentration Ranges

High concentrations of such compounds as water and NPH in the tank 241-C-103 headspace can potentially reduce the validity of sampling and analysis of other compounds. The knowledge, experience, and judgement of analytical chemists associated with the Vapor Program were employed to identify potential interference problems.

Sampling interference issues have generally been avoided by selecting methods less subject to interference problems. However, laboratory experiments have been performed to resolve certain issues. For example, concern was raised that adsorption of organic vapors on the carbon-based sorbent media of the ammonia sorbent traps would interfere with the adsorption of ammonia. This particular issue was addressed by preloading the ammonia trap with NPH, and then demonstrating that gaseous ammonia was still effectively adsorbed and extracted (Lerner and Pool 1993). Additional discussions of some specific issues are given by Ligothke et al. 1993, and Ligothke et al. 1994b.

Large quantities of matrix hydrocarbons (e.g., NPH) may significantly affect adsorption efficiency of organic vapor sorbent traps. The extraction yield from SUMMA™ canisters may also be affected. These issues are addressed by spiking samples with additional analyte, as discussed in Section 2.3.

The types and relative amounts of analytes that can be introduced and successfully analyzed by any analytical instrument; e.g., gas chromatograph/mass spectrometer (GC/MS), gas chromatograph/flame ionization detector (GC/FID) is limited. The presence of large quantities of matrix chemicals (e.g., water vapor, NPH) tends to limit the capability of analytical instruments to detect and quantitate analytes. Generally, modifications of the analytical technique can be made to compensate for any given chemical matrix, but these require either extensive preparation and analysis of standards or real matrix samples for method development. Methods so developed must then be validated with real samples.

3.1.3 Sample Radioactivity

Gas and vapor samples from tank 241-C-103 were neither expected nor found to be radioactive (Mahon et al. 1994). Nevertheless, it has been the responsibility of WHC to demonstrate that all samples were indeed non-radioactive before they could be shipped to laboratories for analysis. All gas and vapor samples collected from tank 241-C-103 have been cleared as unconditionally free of radioactive contamination by WHC 222-S Laboratory.

Samples collected with the VSS were protected from radioactive particulates by two HEPA filters placed in line ahead of the sampling manifold. Both HEPA filters were removed at the end of each sampling session and assayed at the 222-S laboratory for total alpha and beta emissions, and a gamma energy analysis. Samples were considered free of radioactive particulates if the second HEPA filter of the series was determined to have less than detectable. The results of all HEPA filter analyses are given by Mahon et al. 1994.

Potential for tritium contamination was tested by examining water vapor samples for tritium-substituted water. Water vapor from a known volume of tank air was collected with a silica gel sorbent trap, and this was analyzed for tritium by the 222-S laboratory. Observed tritium levels, given by Mahon et al. 1994, were within limits established by WHC 1993a for shipment of samples.

3.2 VAPOR SAMPLING SYSTEM

Figure 1-1 illustrates the use of the VSS on tank 241-C-103. Gases and vapors are drawn from the waste tank through the VSS by an air pump mounted on the mobile laboratory truck. The system uses a heated sampling probe and heated sample transfer lines to transfer gases and vapors from the waste tank headspace to a heated sampling manifold housed inside the mobile laboratory. By maintaining the temperature of all surfaces exposed to the sample stream higher than the tank headspace temperature, vapor condensation is prevented. VSS temperatures are monitored and a feedback control system is used to maintain desired temperatures.

The majority of data discussed in this report are derived from samples collected using the VSS. Detailed descriptions of the VSS, its purpose, features, operation, and use in the characterization of tank 241-C-103 are given by DeFord 1993, and by Mahon et al. 1994.

3.2.1 Instrumentation and Control

Currently, absolute and differential pressures of the VSS are measured, displayed visually, and electronically recorded at 2- to 3-second intervals. Flow control within the manifold and through sampling devices is accomplished with National Institute of Science and Technology traceable mass flow controllers. Flow measurements are also displayed and electronically recorded at 2- to 3- second intervals. Pressure, temperature, and flow measurement systems were calibrated by the WHC Standards Laboratory. Mahon et al. 1994 provides calibration dates relevant to sample job 7B and indicates that the flow controlling and pressure monitoring instruments were recalibrated just before sample job 7B. The VSS is also equipped with a GC/FID for monitoring changes in organic vapor concentrations, assessing system cleanliness and adequacy of purge, and diagnosing sampling or analytical anomalies.

3.2.2 Sample Transfer Adequacy

The VSS sample transfer adequacy was addressed by: 1) demonstrating sample transfer efficiency before sample job 7B; 2) performing a cleanliness test; 3) purging the VSS with tank air; and 4) demonstrating sample composition consistency over short and long time periods.

Sample transfer integrity was evaluated before conducting sample job 7B by sampling vapor standards using the GC/FID of the VSS through the VSS sampling manifold, and also independent of the sampling manifold. Mahon et al. 1994 discusses this test and reports that the VSS transferred tested vapors with a 100% ($\pm 6\%$) efficiency.

VSS cleanliness was tested by collecting a SUMMA™ canister ambient air sample using the sampling manifold at its set point temperature. A SUMMA™ canister ambient air sample was also collected upwind (without the VSS) of tank 241-C-103 for comparison. Test results are discussed in Section 4.2.

The VSS was purged with gases and vapors from the tank 241-C-103 headspace before samples were collected. A tank air purge was also performed each time the VSS was reconfigured to sample from a different elevation within the headspace. Mahon et al. 1994 describes the conditions, duration, and frequency of tank air purges performed during sample job 7B.

Consistency of samples, determined both by the GC/FID and samples collected for later analysis, was used to evaluate the adequacy of the sample transfer. Results are discussed in Section 4.2.

3.2.3 Sample Volume Uncertainty

Errors in the control of sample flow rate and duration result in sorbent trap sample volume uncertainties. Mahon et al. 1994 provides a detailed discussion of sorbent trap sample volumes and estimated uncertainties. The percent uncertainty in the volume measurements for different types of samples ranges from 0.03% to 1.65% due to flow control uncertainties. Sorbent trap volume uncertainties are combined with analytical uncertainties in results presented below.

Errors and uncertainties in the control of sample flow rate and duration result in sorbent trap sample volume errors and uncertainties. Mahon et al. 1994 provides a detailed discussion of sorbent trap sample volumes and estimated uncertainties. The error as determined by standards laboratory calibration is presented as uncertainty in the volume measurements for different types of samples. These range from 0.03% to 1.65%. Sorbent trap sample volume uncertainties contribute to the analytical uncertainties in the results presented below.

3.3 SUMMA™ CANISTER SAMPLES

SUMMA™ canisters were used to sample volatile organic vapors and certain gases in tank 241-C-103. SUMMA™ canisters are stainless steel vessels whose internal surfaces have been prepared by the SUMMA™ process. This process passivates active sites on the canister walls to minimize their adsorption of gases and vapors. SUMMA™ canisters are filled with the air to be analyzed through a valve, which is then closed to seal the sample inside. SUMMA™ canisters essentially allow collection and transfer of whole-air samples from location to an analytical laboratory where the sample can be analyzed. SUMMA™

technology is generally accepted by analytical air chemists for ambient air, and is specifically cited as the preferred sampling method in the U.S. Environmental Protection Agency (EPA) TO-12 and TO-14 methods for ambient air analysis (EPA 1988).

SUMMA™ canister samples collected during sample job 7B were either 5.8- or 6-L, single-valve canisters. Canisters were cleaned, certified as clean using EPA TO-12 methods, and evacuated by the supplying laboratory. The canisters were then connected to the heated sampling manifold of the VSS, and filled by opening the appropriate valves. All surfaces contacted by the sample between the tank headspace and the SUMMA™ canister itself (including the valve on the SUMMA™) were heated to reduce the loss of vapors to internal surfaces.

All tank 241-C-103 SUMMA™ canister samples from sample job 7B were analyzed at OGIIST. The canisters were pressurized with ultra-clean helium or oxygen to facilitate sample removal and transfer. The pressurized canisters were allowed to equilibrate at laboratory temperature, and the helium dilution ratio was determined by a gas chromatograph with a thermal conductivity detector (GC/TCD).

Two ambient air SUMMA™ canister samples were also collected at the start of sample job 7B, before the VSS was purged with air from the tank headspace. One of these was collected manually by a technician upwind of tank 241-C-103, and the other through the fully-heated VSS sampling manifold. These ambient samples were analyzed at PNL (McVeety et al. 1994) to verify the cleanliness of the VSS manifold, and are discussed in Section 4.0.

Method development was minimized by applying, and modifying as needed, methods originally developed for ambient air analysis. Application of these methods has required adjustments for the relatively high concentrations of analytes present in samples from tank 241-C-103, and extension of the methods for analysis of certain polar organic compounds. Applicability of the methods has been considered elsewhere (Einfeld et al. 1992b, Story 1994).

3.3.1 Permanent Gases

SUMMA™ canister samples were analyzed for hydrogen, carbon monoxide, methane, carbon dioxide, and nitrous oxide. Hydrogen and carbon monoxide were measured using a gas chromatograph equipped with a mercuric oxide gas reduction detector. Methane in the sample was measured by a GC/FID. Carbon dioxide was measured by a gas chromatograph equipped with a thermal conductivity detector. Nitrous oxide was measured by a gas chromatograph equipped with an electron capture detector.

3.3.2 Speciation of Organic Vapors

Chemical speciation of the organic compounds in SUMMA™ canister samples was performed using the EPA TO-14 methodology. As with the TO-12 method, the TO-14 methodology uses a cryogenically cooled loop to condense non-methane organic gases and vapors, which is then ballistically heated to evaporate the condensed compounds into a carrier gas stream. In the TO-14 method, however, the analytes and carrier gas are introduced to a gas chromatograph with a mass spectrometer (GC/MS), with the goals of optimized analyte separation via gas chromatography, followed by analyte identification and quantitation using the mass spectrometer detector (EPA 1988). A more detailed description of the technique used at OGIIST is given by Rasmussen and Einfeld 1994.

3.4 SORBENT TRAP SAMPLES

Sorbent traps were used to sample organic vapors and specific inorganic gases from tank 241-C-103. Unlike SUMMA™ canisters, sorbent traps concentrate targeted analytes by selectively removing them from the air sample, and the other constituents of the air (e.g., the oxygen, nitrogen, water, etc.) are not collected.

Sorbent traps are tubes containing one or more beds of sorbent media. Air to be sampled is drawn through the tube, and the targeted analytes are adsorbed by the sorbent media. By using a suitable, known flow rate for a known length of time, the sample will not saturate the sorbent trap, and the total volume of air sampled can be calculated. Under proper conditions, the sorbent media collects essentially all of the target analytes in the air stream. Determination of adsorption efficiency is part of the validation process. After sampling is complete, the tube containing the sorbent media is sealed and sent to a laboratory for analysis. At the analytical laboratory, the analyte is extracted from the sorbent media, and quantitated. Given the quantity of analyte recovered and the total volume of air sampled, the concentration of analyte in the original air sample is calculated.

Sorbent trap sampling efficiency was validated by using, when possible, two sections of sorbent media in series. If sampling efficiency is high, the first section of sorbent media collects nearly all of analyte, and the second section (the breakthrough section) collects very little analyte. Analysis of breakthrough sections are discussed by Ligothke et al. 1994, and Clauss et al. 1994.

All sample job 7B sorbent trap samples were collected using the VSS. The sorbent trap station of the VSS allows collection of two sorbent traps simultaneously, and most samples were taken in pairs. Flow rate data and small system pressure effects on the volume of air, gases, and vapors passed through each sorbent trap in sample job 7B are provided by Mahon et al. 1994.

3.4.1 Organic Vapors

Three types of sorbent traps were used to characterize organic vapors in the tank 241-C-103 headspace in sample job 7B. These were the OVS trap, triple sorbent traps (TSTs), and Carbotrap/Carbotrap C² traps (CCTs). During sample jobs 3, 4, 5, and 6B, Depot Area Air Monitoring System (DAAMS) traps were also used to address phosphorous containing compounds.

3.4.1.1 OSHA Versatile Sampler Traps. OVS traps are designed to trap aerosol particles and organic vapors. They consist of a glass tube in which a glass fiber filter (for collecting aerosol particles), and two separated sections of sorbent media are housed. In this study, OVS traps were used to sample only semivolatile organic vapors. The organic analytes adsorbed by the sorbent media were desorbed by liquid solvent extraction, and the extracted solution analyzed by GC/MS. Tests performed by PNL demonstrated the OVS to be an effective device for initial characterization of the semivolatile organic vapors and aerosol thought to exist in the tank 241-C-103 headspace. These tests and the analytical quantitation method are described by Ligothke et al. 1993.

OVS traps were analyzed for NPH and other semivolatile hydrocarbons. The OVS was split into two sections for analysis: 1) the glass fiber filter and the front sorbent bed; and 2) the second sorbent bed along with both sections of polyurethane foam. Analytes were desorbed from these sections by liquid carbon disulfide extraction, the extract was concentrated, diluted, or left neat, and analyzed by direct injection into a GC/MS. Details of the positive identification, extraction efficiency, and quantitation of each individual NPH were reported by Ligothke et al. 1993. Clauss et al. 1994 describes techniques used to validate the OVS traps with surrogate addition of deuterated n-dodecane and n-tridecane.

3.4.1.2 Triple Sorbent Traps. TSTs were fabricated, conditioned, and analyzed at Oak Ridge National Laboratory (ORNL). As suggested by their name, TSTs contain three types of sorbent media, each segregated as a bed. The first bed consists of Carbotrap CTM, which effectively adsorbs semivolatile organic vapors having carbon chain lengths of eight or greater. The second bed consists of CarbotrapTM, which adsorbs most organic vapors less volatile. The third bed consists of Carbo sieve IIITM, which is a molecular sieve capable of trapping virtually all organic vapors and gases.

Maintaining an appropriate air flow in the specified direction through a TST results in a distribution of compounds among the three beds. Analytes are extracted from the TST by thermal desorption into a gas stream flowing backwards through the trap. This ensures that, for example, semivolatiles trapped on the first bed are never exposed to the second or third beds, from which they are not easily desorbed. Jenkins et al. 1994 provides detailed information on the preparation, conditioning, validation, and analysis of TSTs.

²Carbotrap, Carbotrap C and Carbo sieve III are registered trademarks of Suppelco, Bellefonte, Pennsylvania.

TST sampling efficiency has been discussed by Jenkins et al. 1994. The design of these traps is such that there is little doubt that all target analytes are trapped; the more pertinent question is whether all trapped analytes are indeed desorbed.

TST analyte desorption efficiency was addressed by spiking the sorbent traps with surrogates. A surrogate, in this context, is a compound having similar or even identical chemical properties as the analyte. Deuterated dodecane, for example, served as a surrogate spike for dodecane in OVS trap sampling. All TSTs (and certain OVS traps) were spiked with surrogate compounds not expected to be found in tank 241-C-103 (e.g., deuterated bromobenzene) in known quantities before being used to collect samples (Jenkins et al. 1994, Clauss et al. 1994). Desorption efficiency is calculated by comparing the amount of surrogate recovered with the amount spiked.

The accuracy of TST analyses was further addressed by using internal standards during analysis. This assures that instrument response factors are acceptable and that they do not change during multi-sample analysis.

3.4.1.3 Carbotrap/Carbotrap C™ Traps. CCTs were also made, conditioned, and analyzed at ORNL. CCTs were validated specifically to quantitate tributyl phosphate (TBP) and dibutylbutyl phosphonate (DBBP) in the tank 241-C-103 headspace. CCTs consist of two beds of sorbent media, Carbotrap C™ and Carbotrap™, and are analyzed by the same methods as TSTs. Jenkins et al. 1994 provides relevant information on the preparation, validation, and analysis of CCTs.

CCTs from sample job 7B were analyzed for TBP and DBBP. TBP and DBBP were extracted from the sorbent media by thermal desorption. The CCT itself was ballistically heated and purged with a zero air carrier gas stream flowing in the opposite direction of sampling. The carrier gas and desorbed analytes were then cryogenically cooled to condense the analytes in a loop of the chromatographic column. This loop was then itself heated and purged slowly with carrier gas. Analyte separation, identification, and quantitation were performed using a gas chromatograph equipped with both a nitrogen-phosphorous detector and a flame ionization detector (GC/NPD/FID). This technique provides highly specific verification and good quantitation of TBP and DBBP.

CCTs were spiked with the same surrogates as TSTs. Jenkins et al. 1994 discusses differences between CCT and TST analytical quality control.

3.4.2 Inorganic Gases and Water

The tank 241-C-103 headspace was sampled for ammonia, nitric oxide, nitrogen dioxide, oxides of sulfur, and hydrogen cyanide using sorbent traps. A different trap or system of traps was used for each of these inorganic gases.

All inorganic gas sorbent traps were supplied and analyzed by PNL. Desorption of ammonia, nitrogen dioxide, nitric oxide, hydrogen cyanide, and sulfur oxides from their respective sorbent traps was accomplished by liquid (water) extraction. The primary and back-up sections of all traps were physically separated before extraction, and analyzed separately. Ammonia was analyzed by the ion selective electrode method. Nitrogen dioxide, nitric oxide, hydrogen cyanide, and sulfur oxides were analyzed by ion chromatography. Ligothke et al. 1994b discusses the methods and associated uncertainties.

3.4.2.1 Water. The water vapor concentration of the headspace was established using gravimetric analysis of sorbent traps used for other analytes. Ammonia sorbent traps, for example, were prepared with a silica gel sorbent trap for water vapor connected to the outlet end. This ammonia/water vapor sorbent trap system was weighed before and after sampling, and the observed gain in weight was used to calculate the amount of water in the sample. Using this technique, 50 usable water vapor measurements of tank 241-C-103 were made during sample job 7B without the need for dedicated water vapor samples.

3.4.2.2 Ammonia. Ammonia was collected on a sulfuric acid-impregnated carbon bead medium. This sorbent medium adsorbs the ammonia and converts it to the very stable and nonvolatile ammonium sulfate salt. The traps contained two sections of this sorbent material, separated by a plug of glass wool. The larger, primary section of sorbent material effectively trapped all of the ammonia, while the smaller, back-up section of sorbent material was analyzed to determine whether any ammonia broke through the front bed.

3.4.2.3 Nitrogen Dioxide and Nitric Oxide. Nitrogen dioxide and nitric oxide were collected at the same time using three traps in series. The first tube in the series contained two sections (primary and back-up sections) of a triethanol amine-impregnated sorbent material known to be effective at adsorbing nitrogen dioxide. The second tube in the series contained an oxidizing agent that converts nitric oxide to nitrogen dioxide, and the third tube, which was identical to the first, collected the nitrogen dioxide produced in the second tube.

3.4.2.4 Sulfur Oxides. Sulfur oxide traps contain two sections of a proprietary metal hydroxide on a carbon bead matrix. Here, sulfur oxide refers to the sum of sulfur dioxide and sulfur trioxide. The acid gas forms of these gases are adsorbed by the basic sorbent material and are trapped stably as salts involving sulfite and sulfate ions. Because the sulfite is readily oxidized to sulfate by the oxygen in air, it is impossible to determine the original partition of sulfur dioxide and sulfur trioxide gases.

3.4.2.5 Hydrogen Cyanide. Hydrogen cyanide traps contain two sections of hydrated calcium oxide-sodium hydroxide (soda lime) granules. As with the other acidic inorganic gases discussed above, hydrogen cyanide is adsorbed and converted to a salt.

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4.0 RESULTS

Sample job 7B results are presented in this section. Mahon et al. 1994 compiles the documentation of the sampling event itself, and provides relevant meteorological and tank headspace temperature data. Mahon et al. 1994 also provide detailed discussions of the various tests and procedures performed to ensure the validity of samples collected with the VSS. Sample analyses are provided by Clauss et al. 1994, Jenkins et al. 1994, Ligothke et al. 1994b, and Rasmussen and Einfeld 1994. These authors also discuss laboratory quality control, reasons for omitting data, and general data quality.

Note that because water vapor represents a significant fraction of the total gas and vapor in the headspace (about 6%), it is included as a component of the air in the calculation of all mass concentrations (e.g., mg/L) and dimensionless concentrations (e.g., ppmv [parts per million by volume]) in the following sections. This is illustrated with sample calculations by Ligothke et al. 1994b.

4.1 SAMPLE JOB 7B SUMMARY

Sample job 7B was conducted between May 12 and May 25, 1994, using the VSS as depicted in Figure 1-1. Three separate sampling and analysis plans were written to specify the type and number of samples collected, the second and third being written and issued when the success of previous sampling was established (Mahon et al. 1994). Similar samples were generally collected in sets of at least five, and different sets of samples that were to be compared (e.g., the sets collected from the three headspace elevations) were collected with a minimum amount of time between the sets.

The date, time of day, flow rates, and flow durations for all samples of sample job 7B are given by Mahon et al. 1994. Flow rates measured by the mass flow meters of the VSS are reported at standard temperature and pressure (i.e., 0 °C, 760 mmHg) by that instrumentation.

Preparation of the various sampling devices was performed by the same analytical laboratories responsible for their analysis. Sample identification numbers were assigned according to guidelines set forth in the sampling and analysis plan for sample job 7B (Mahon et al. 1994). Sorbent trap samples were transported in ice chests from PNL and ORNL, and stored in a locked sample refrigerator. Chain-of-custody documentation, also originating and/or ending at the laboratory supplying the sampling devices, has been compiled by Mahon et al. 1994.

4.2 VSS PERFORMANCE

4.2.1 Temperature Measurement and Control

Mahon et al. 1994 discusses the measurement and control of the various VSS heated zones during sample job 7B. The nominal set temperature for the VSS was 75 °C. Given a maximum observed deviation of 14 °C from the set temperature, the coldest part of the VSS in contact with tank gases and vapors was estimated to be 60 °C, which corresponds to about 22 °C higher than the tank headspace.

4.2.2 Flow Control and Volume Measurement

The accuracy to which sorbent trap sample volumes are known is determined by the accuracy of the flow controllers and manual timing of valves. Errors introduced by the flow controllers were determined by calibration and experiment to be between 0.03% and 1.65% of total sorbent trap sample volume. Since this is small compared to the expected sample handling and analysis error, no corrections for volume errors have been made.

4.2.3 Chemical Analyses

The GC/FID resident in the VSS has been used to determine the cleanliness of the manifold prior to sampling. Its limit of detection is about 1 ppmv for hydrocarbons. In order to determine the cleanliness of the system below this level, an ambient air SUMMA™ canister sample was collected through the sampling manifold (while it was at set temperature), and for comparison, a second ambient air SUMMA™ canister sample was collected manually upwind of tank 241-C-103.

Analyses of these ambient air SUMMA™ canister samples was performed by PNL, and is reported by McVeety et al. 1994. Comparison of these sample analyses indicates that methylene chloride was present in the manifold at the start of sample job 7B. This is almost certainly due to the unscheduled use of a new section of heated transfer tubing at the start of sample job 7B. This tubing had been cleaned with methylene chloride, and apparently not all of this cleaning solvent had been baked out.

Tank 241-C-103 headspace concentrations of n-hexane, n-dodecane, and n-tridecane measured by GC/FID in the VSS between May 16 and 25, 1994 have relative standard deviations of 3% to 6% (Mahon et al. 1994). These analyses were performed after the initial daily purge and between sample sets to demonstrate the adequacy of purging. The good agreement between analyses performed immediately after purging and later during the day indicates purging was adequate.

The adequacy of the heating of the manifold in the VSS has also been demonstrated by GC/FID analysis of the ratio of concentrations of n-dodecane to n-tridecane. The ratio of the concentrations of these compounds would be expected to be constant in the headspace. Having almost identical chemical properties, any observed change in the ratio of their concentrations in the sampling manifold would indicate changes in the manifold temperature. Mahon et al. reports a very consistent ratio of 0.86 ± 0.02 for the 15 GC/FID analyses performed during sample job 7B, and concludes the manifold temperature was consistently high enough to prevent condensation of these semivolatile compounds.

Sample transfer integrity was evaluated by comparing periodic GC/FID analyses of tank organic vapors from the sampling manifold during sample job 7B. Mahon et al. 1994 present and discuss the results of these GC/FID analyses, and conclude that short-term (24-hour) and long-term (90-day) changes in the composition of tank air from the manifold are small. GC/FID peak areas agree to within a relative standard deviation of about 2% over 24 hours, and within about 12% over 90 days.

4.3 INORGANIC GASES AND VAPORS

4.3.1 Water Vapor

Water vapor is the most abundant vapor constituent in the tank 241-C-103 headspace. Water vapor was estimated from the gravimetric analysis of 50 inorganic gas sorbent traps to be 42.2 ($\sigma = 2.4$) mg/L, assuming the tank headspace was nominally 38 °C. This corresponds to a water partial pressure in the headspace of 45.4 mmHg, to a dewpoint of 36.3 °C, and to a relative humidity of 91%. The headspace is thus nearly saturated with water vapor.

Individual sample gravimetric results are given by Ligothke et al. 1994b, which has eliminated certain data for reasons explained. The data considered to be valid is summarized in Table 4-1, which groups samples according to date and headspace elevation.

Water vapor trip blanks indicated no adverse handling effects, and no correction was applied to account for the small deviations observed. Given the $\sigma = 2.4$ mg/L as a measure of uncertainty in the water vapor measurements, the results given in Table 4-1 are self-consistent. There is not a statistically significant difference in the headspace water vapor content at the three different sampling elevations, or over the two-week sampling period.

Table 4-1. Summary of Water Vapor Data.

Date [1994]	Elevation above waste (m)	Number of Samples	Mean (mg/L)	σ (mg/L)
May 12	0.79	9	43.2	0.6
May 19	0.79	17	41.1	2.5
May 20	2.92	5	45.2	1.3
May 20	5.05	5	44.4	1.8
May 25	0.79	14	41.0	1.7
Total:		50	42.2	2.4

4.3.2 Ammonia

Ammonia vapor in the tank 241-C-103 headspace was measured to be 304 ppmv ($\sigma = 11$ ppmv) using 35 sorbent traps. This value incorporates the results of samples collected from the three headspace elevations, as well as all spiked and unspiked samples collected during sample job 7B. Table 4-2 summarizes results from the six sets of ammonia samples. Concentrations reported in Table 4-2 have been adjusted for water vapor content. Ligothe et al. 1994b provides individual results for all samples.

Analyses of spiked and unspiked ammonia samples are also summarized in Table 4-2. The average ammonia trapped in these 15 samples was 42.6 μmol , which is between the 24.2 and 48.2 μmol spike levels. Given the uncertainties associated with the sample volumes, analytical measurements, and spike amounts, there is no significant difference in the observed ammonia concentrations in the unspiked and spiked samples. The results are in very good agreement, and clearly indicate that the extraction of ammonia is not affected by the chemical matrix of the tank headspace.

Unspiked samples were collected on May 19 and 20, 1994, to determine if ammonia was subject to vertical stratification within the headspace. As indicated in Table 4-2, the differences between ammonia concentrations observed at 0.79, 2.92, and 5.05 m above the waste surface are statistically indistinguishable and suggest the tank headspace is vertically mixed.

Table 4-2. Summary of Ammonia Analyses.

Date [1994]	Elevation above waste (m)	Spike (ppmv of sample ¹)	Number of Samples	Mean (ppmv)	σ (ppmv)
May 12	0.79	0	10	310	9
May 19	0.79	170	5	300	19
May 19	0.79	338	5	300	16
May 19	0.79	0	5	296	7
May 20	2.92	0	5	307	3
May 20	5.05	0	5	307	5
Total:			35	304	11

¹Spike ppm = $10^6 \times (\text{volume of ammonia vapor spike}) / (\text{sample volume})$.

4.3.3 Hydrogen

Hydrogen gas in the tank 241-C-103 headspace was measured to be 782 ppmv ($\sigma = 63$ ppmv) from 35 SUMMA™ canister samples. This value incorporates the results of samples collected from the three headspace elevations, and the results of six samples collected during sample job 7A. Table 4-3 summarizes results from the six sets of hydrogen samples. Individual sample results are given by Einfeld 1994 and Rasmussen and Einfeld 1994.

The sample means, as reported in Table 4-3 for May 19 and 20, indicate no statistically significant stratification of hydrogen over the three elevations of headspace sampled.

4.3.4 Nitrous Oxide

Nitrous oxide vapor in the tank 241-C-103 headspace was measured to be 763 ppmv ($\sigma = 51$ ppmv) from 37 SUMMA™ canister samples. This value incorporates the results of samples collected from the three headspace elevations, and the results of six samples collected during sample job 7A. Table 4-4 summarizes results from the 6 sets of nitrous oxide samples. Individual sample results are given by Einfeld 1994 and Rasmussen and Einfeld 1994.

Table 4-3. Summary of Hydrogen Analyses.

Date [1994]	Elevation above waste (m)	Number of Samples	Mean (ppmv)	σ (ppmv)
April 7 ¹	0.79	6	724	7
May 16	0.79	4	803	26
May 19	0.79	3	718	11
May 20	2.92	3	732	49
May 20	5.05	3	714	26
May 25	0.79	16	833	43
Total:		35	782	63

¹Sample job 7A, Rasmussen and Einfeld 1994.

Table 4-4. Summary of Nitrous Oxide Analyses.

Date [1994]	Elevation above waste (m)	Number of Samples	Mean (ppmv)	σ (ppmv)
April 7 ¹	0.79	6	772	12
May 16	0.79	6	737	14
May 19	0.79	3	710	18
May 20	2.92	3	696	6
May 20	5.05	3	686	6
May 25	0.79	16	805	34
Total:		37	763	51

¹Sample job 7A, Rasmussen and Einfeld 1994.

Comparison of nitrous oxide concentration averages at the 3 headspace elevations is consistent with the previously discussed comparisons of ammonia and hydrogen; no statistically significant vertical stratification of nitrous oxide is observed.

4.3.5 Carbon Monoxide

CO gas in the tank 241-C-103 headspace was measured to be 26.7 ppmv ($\sigma = 2.3$ ppmv) from 36 SUMMA™ canister samples. This value incorporates the results of samples collected from the three headspace elevations, and the results of six samples collected during sample job 7A. Table 4-5 summarizes results from the six sets of CO samples. Individual sample results are given by Einfeld 1994 and Rasmussen and Einfeld 1994.

Table 4-5. Summary of Carbon Monoxide Analyses.

Date [1994]	Elevation above waste (m)	Number of Samples	Mean (ppmv)	σ (ppmv)
April 7 ¹	0.79	6	22.3	0.5
May 16	0.79	5	26.3	0.6
May 19	0.79	3	26.7	0.6
May 20	2.92	3	26.3	0.6
May 20	5.05	3	26.3	0.6
May 25	0.79	16	28.6	0.6
Total:		36	26.7	2.3

¹Sample job 7A, Rasmussen and Einfeld 1994.

Comparison of nitrous oxide concentration averages at the three headspace elevations is consistent with the previously discussed comparisons of ammonia, hydrogen, and nitrous oxide; no statistically significant vertical stratification of carbon monoxide is observed.

4.3.6 Nitrogen Dioxide and Nitric Oxide

Neither a concentration nor an upper concentration limit for nitrogen dioxide in the tank 241-C-103 has been established. The weight of evidence suggests that nitrogen dioxide concentrations are indeed low, but Ligothke et al. 1994b has raised a sampling interference issue, suggesting that the nitrogen dioxide sorbent trap samples may be affected by the presence of ammonia. Furthermore, Ligothke et al. 1994b explains that inconsistent levels of nitrite, the ion that is extracted from the sorbent traps and related to nitrogen dioxide in the sample, were measured in the trip blanks carried on May 25, 1994.

Nitrogen dioxide sorbent trap samples collected during sample job 7A (April 7) were downstream of ammonia sorbent traps, and were consequently protected from potential interferences from ammonia. However, water vapor condensation in the ammonia sorbent traps (upstream of the nitrogen dioxide/nitric oxide traps) may have absorbed nitrogen dioxide. Analysis of these nitrogen dioxide samples, as indicated in Table 4-6, suggests the concentration of nitrogen dioxide is <0.04 ppmv.

Table 4-6. Summary of Nitrogen Dioxide Analyses.

Date [1994]	Number of Samples	Mean (ppmv)
April 7 ¹	5	<0.04
May 12	3	<0.004
May 25	10	<0.06

¹Sample job 7A.

Table 4-6 also summarizes analytical results for sample job 7B nitrogen dioxide sorbent traps. These sorbent traps were not downstream of ammonia sorbent traps, and condensation of water vapors upstream of the sorbent media was prevented. If it is determined that ammonia does not interfere with the collection of nitrogen dioxide, and laboratory tests are planned, then the upper concentration of nitrogen dioxide in the tank 241-C-103 headspace would be established as <0.004 ppmv.

Results of the spike addition study of nitrogen dioxide are nonconclusive because: 1) trip blank contaminant nitrite concentration variations are significant; and 2) spike blank recoveries average 153%, due presumably to higher than average nitrite contamination levels.

The nitric oxide concentration in the tank 241-C-103 headspace is estimated to be 1.5 ppmv ($\sigma = 0.3$) from eight sorbent traps collected during sample job 7B. Summarized results from the two sets of nitric oxide sorbent trap samples are given in Table 4-7; individual sample results are given by Ligothke et al. 1994b.

Table 4-7. Summary of Nitric Oxide Analyses.

Date [1994]	Number of Samples	Mean (ppmv)	σ (ppmv)
May 12	3	1.33	0.06
May 25	5	1.63	0.31
Total:	8	1.5	0.3

The design of nitric oxide sorbent trap sampling is such that if ammonia interferes with the nitrogen dioxide sampling, it also interferes with nitric oxide sampling. Thus until the ammonia interference issue is resolved, the values given in Table 4-7 should be considered subject to change.

Nitric oxide sorbent trap samples collected on May 12, 1994, corrected for nitrite contamination levels, indicate incomplete adsorption of the analyte. The breakthrough sorbent section was found to contain about 7% of the analyte. Because similar data are not yet available for the May 25, 1994 samples, this correction was applied to all the results reported in Table 4-7.

Two sets of spiked nitric oxide samples were collected on May 25, 1994. These correspond to two amounts of nitrite (the adsorbed form of nitric oxide) added to the sorbent traps. The lower spike level was slightly higher than the observed tank nitric oxide concentration. Analysis of these spiked samples indicated the analytical recovery of analyte was not diminished by the sample chemical matrix. Ligotke et al. 1994b discusses this in more detail.

4.3.7 Sulfur Oxides

No significant quantities of sulfur oxides were observed in tank 241-C-103. Three sulfur oxide sorbent trap samples were collected May 12, 1994, and analyzed at PNL. Ligotke et al. 1994b reports that trace contamination of sulfur oxides was present in the sorbent media. This contamination served to validate that analytical detection limits were unaffected by vapor sampling. Analysis of the samples and blanks indicated slightly more sulfur oxides in the samples than in the blanks, but the difference was not significant given analytical standard deviations. It was determined that the sulfur oxides concentration in the tank headspace was conservatively ≤ 0.02 ppmv.

4.3.8 Hydrogen Cyanide

Five sorbent trap hydrogen cyanide samples were collected during sample job 7B. Ligothke et al. 1994b reports that the hydrogen cyanide measured in the samples was about the same as measured in the trip blanks. Conservative assumptions described in their report place the hydrogen cyanide concentration at ≤ 0.04 ppmv.

4.4 ORGANIC VAPORS

4.4.1 Aromatic Hydrocarbons

The benzene concentration in the tank 241-C-103 headspace was measured to be between <0.01 and 0.33 ppmv, from 16 SUMMA™ canister and 4 TST samples. Results of four sets of samples are presented in Table 4-8; note that the May 16, 1994 SUMMA™ canister samples were analyzed for benzene by both the EPA TO-14 methodology and a direct injection methodology.

Table 4-8. Summary of Aromatic Hydrocarbon Analyses.

Compound	Method	Date [1994]	Number of Samples	Mean (ppmv)	σ (ppmv)
Benzene	SUMMA™	April 7 ^{1,2}	6	0.33	0.11
		May 16 ²	6	0.071	0.015
		May 16 ³	6	<0.01	-
		May 25 ³	4	<0.01	-
	TST	May 16	4	0.08	0.03
Toluene	SUMMA™	April 7 ^{1,2}	6	0.057	0.016
		May 16 ²	6	0.020	0.004
	TST	May 17	4	0.03	0.02

¹Sample job 7A.

²TO-14 methodology.

³Direct injection methodology.

The toluene concentration in the tank 241-C-103 headspace was measured to be between 0.020 and 0.057 ppmv, from 12 SUMMA™ canister and 4 TST samples. Results of the three sets of samples are presented in Table 4-8.

SUMMA™ canister spike additions were used by Rasmussen and Einfeld (1994) to establish that benzene is completely recovered from SUMMA™ canisters filled with the tank 241-C-103 chemical matrix. SUMMA™ canisters were analyzed by the EPA TO-14 method for toluene (EPA 1988). Though SUMMA™ canisters were not spiked with toluene to establish recovery of toluene, toluene would be expected to behave much like benzene, and essentially total recovery of toluene from SUMMA™ canisters would be expected. Individual sample analyses and a discussion are given by Rasmussen and Einfeld 1994.

TST samples were spiked at two levels of benzene, and at two levels of toluene, however, the spike levels of both compounds were too large relative to the observed tank compound concentrations to properly address matrix interference effects. Analytical error associated with the measurement of relatively large spike amounts was essentially larger than the amounts of benzene and toluene collected from the tank sample. Rasmussen and Einfeld 1994 discusses TST and spiked TST blank analyses, as well as surrogate recoveries.

4.4.2 Butanal, n-Butanol, and 1,3-Butadiene

Table 4-9 summarizes butanal, n-butanol, and 1,3-butadiene concentrations measured in the tank 241-C-103 headspace. Butanal was measured to be between 1.2 and 4.7 ppmv, n-butanol to be between 13.1 and 28.4 ppmv, and 1,3-butadiene to be at or below 0.060 ppmv. Results are based on analysis of SUMMA™ canister and TST samples.

Tank 241-C-103 headspace SUMMA™ canister samples were spiked and analyzed to determine the recovery efficiency of these analytes from the tank chemical matrix. Recovery efficiencies are given by Rasmussen and Einfeld 1994 as 73.6% for butanal, 43.2% for n-butanol, and 86.9% for 1,3-butadiene. These average recoveries were used to correct direct injection method analyses of the May 16 and 25, 1994 SUMMA™ canister samples. Rasmussen and Einfeld 1994 provides justification and discussion of the analyses and corrections applied.

Jenkins et al. 1994 discusses the analysis of butanal and n-butanol in TST samples. TST samples were spiked with n-butanol, but the spiked amounts were not appropriate for evaluating the recovery of n-butanol.

Given that different sampling and analysis techniques as well as independent laboratories were used, the agreement between the SUMMA™ and TST results for butanal and n-butanol is very good.

4.4.3 Halogenated Compounds

Table 4-10 summarizes vinylidene chloride (1,1-dichloroethene) and methylene chloride (dichloromethane) concentrations measured in the tank 241-C-103 headspace. Vinylidene chloride was not detected in either SUMMA™ canister or TST samples, and is

conservatively estimated to be <0.02 ppmv. Averaged methylene chloride measurements are as high as 1.62 ppmv, but this is thought to be a contaminant of the VSS. The uncertainty of this highest methylene chloride measurement, as indicated by σ , is also relatively high.

Table 4-9. Butanal, Butanol, and 1,3-Butadiene Analyses.

Compound	Method	Date [1994]	Number of Samples	Mean (ppmv)	σ (ppmv)
Butanal	SUMMA™	May 16	6	4.7	0.7
		May 25	4	4.4	0.7
	TST	May 17	4	1.2	0.8
n-Butanol	SUMMA™	May 16	6	13.1	2.5
		May 25	4	13.1	2.5
	TST	May 17	4	28.4	6.1
1,3-Butadiene	SUMMA™	April 7 ¹	6	<0.020	-
		May 16 ¹	5	0.060	0.020
		May 16 ²	6	<0.05	-
		May 25 ²	4	<0.05	-

¹TO-14 methodology.

²Direct injection methodology.

Analytical limits of detection for vinylidene chloride from SUMMA™ canister samples by the direct injection method is 0.02 ppmv, and by the EPA TO-14 method is estimated by Rasmussen and Einfeld 1994 to be about 0.01 to 0.02 ppmv. The limit of detection for vinylidene chloride from TST samples is estimated by Jenkins et al. 1994 to be about 0.009 ppmv.

Methylene chloride was used as a solvent to degrease parts of the VSS sample transfer tubing and manifold. It was detected in samples from sample job 6B (it is not well detected by the GC/FID of the VSS), but was associated with its use as a cleaning solvent. As discussed in Section 4.2.3, it was observed in the cleanliness test of the VSS at the start of sample job 7B. It is believed to have been inadvertently re-introduced to the VSS when the electrically heated transfer tube between tank 241-C-103 and the mobile laboratory (which had been cleaned before the use of methylene chloride as a cleaning solvent was eliminated) was replaced at the start of sample job 7B (May 11, 1994).

Table 4-10. Summary of Halogenated Compound Analyses.

Compound	Method	Date [1994]	Number of Samples	Mean (ppmv)	σ (ppmv)
Vinylidene Chloride (1,1-Dichloroethene)	SUMMA™	April 7 ^{1,2}	6	<0.02	-
		May 16 ²	6	<0.02	-
		May 16 ³	6	<0.02	-
		May 25 ³	4	<0.02	-
	TST	May 17	4	<0.009	-
Methylene Chloride (Dichloromethane)	SUMMA™	April 7 ^{1,2}	6	0.30	0.14
		May 16 ²	6	0.061	0.030
		May 16 ³	6	<0.02	-
		May 25 ³	4	<0.02	-
	TST	May 17	4	1.62	1.47

¹Sample job 7A.

²EPA TO-14 methodology.

³Direct injection methodology.

4.4.4 Ketones

Table 4-11 summarizes the acetone (propanone), 2-pentanone, 2-hexanone, 2-heptanone, and 2-octanone concentrations measured in the tank 241-C-103 headspace. Acetone is clearly the most abundant ketone, being measured to be 8.8 and 19.4 ppmv, from TST and SUMMA™ canister samples respectively. Both acetone and 2-hexanone are on the preliminary list of compounds of toxicological concern.

Table 4-11. Summary of Ketone Analyses.

Compound	Method	Date [1994]	Number of Samples	Mean (ppmv)	σ (ppmv)
Acetone (Propanone)	SUMMA™	May 16	6	19.4	2.0
		May 25	4	19.2	2.0
	TST	May 17	4	8.8	3.9
2-Pentanone	TST	May 17	4	1.09	0.64
2-Hexanone	SUMMA™	May 16	6	0.59	0.05
		May 25	4	0.57	0.04
	TST	May 17	4	0.51	0.12
2-Heptanone	TST	May 17	4	0.56	0.10
2-Octanone	TST	May 17	4	0.25	0.04

SUMMA™ canister tank headspace samples were spiked with both acetone and 2-hexanone to establish the recovery of these compounds. Rasmussen and Einfeld 1994 reports acetone was extracted with a 69.6% efficiency, and 2-hexanone was extracted with a 50.8% efficiency. Values reported above have been corrected for these factors. The precision of SUMMA™ canister analyses is excellent; agreement between analyses of individual samples in each set is good, and the agreement between the sample set averages of May 17 and 25, 1994 is also good.

TST samples were spiked with each of the five ketones in Table 4-11. Jenkins et al. 1994 reports that only the tank headspace concentrations of 2-hexanone, 2-heptanone, and 2-octanone were properly within the range of the spike additions. For these 3 compounds, unspiked and (corrected) spiked sample results agreed well, and indicate chemical matrix effects for these compounds are small. All five ketones measured quantitatively in unspiked TST samples were within or acceptably near the instrument calibration range.

4.4.5 Nitriles

Table 4-12 summarizes the nitrile concentrations measured in the tank 241-C-103 headspace. Acetonitrile and propanenitrile are the most abundant nitriles, being measured in the 9.1 to 13.2 ppmv range and 3.3 to 5.3 ppmv range, respectively. Acetonitrile and propanenitrile are on the preliminary list of compounds of toxicological concern.

Table 4-12. Summary of Nitrile Analyses.

Compound	Method	Date [1994]	Number of Samples	Mean (ppmv)	σ (ppmv)
Acetonitrile (Ethanenitrile)	SUMMA™	May 16	6	13.2	1.2
		May 25	4	12.7	1.2
	TST	May 17	4	9.1	2.2
Propanenitrile	SUMMA™	May 16	6	5.3	0.7
		May 25	4	5.1	0.7
	TST	May 17	4	3.3	0.3
Butanenitrile	TST	May 17	4	2.4	0.8
Pentanenitrile	TST	May 17	4	1.0	0.1
Hexanenitrile	TST	May 17	4	0.79	0.10
Heptanenitrile	TST	May 17	4	0.60	0.04
Octanenitrile	TST	May 17	4	0.32	0.09
Nonanenitrile	TST	May 17	4	0.14	0.02

SUMMA™ canister tank headspace samples were spiked with acetonitrile and propanenitrile to establish the recovery of these compounds. Rasmussen and Einfeld 1994 reports acetonitrile was extracted with a 41.3% efficiency, and propanenitrile was extracted with a 64.6% efficiency. Values reported above have been corrected for these factors.

TST samples were spiked with each of the nitriles listed in Table 4-12. Jenkins et al. 1994 reports that only the tank headspace concentrations of pentanenitrile, hexanenitrile, heptanenitrile, and octanenitrile were properly within the range of the spike additions. For these four nitriles, unspiked and (corrected) spiked sample results agreed well, and indicate chemical matrix effects for these compounds are small. All nitriles measured quantitatively in unspiked TST samples were within or acceptably near the instrument calibration range.

4.4.6 NPH and Alkanes

Tables 4-13 and 4-14 summarize measured tank 241-C-103 headspace concentrations of quantitated volatile alkanes and semivolatile alkanes, respectively. Only straight-chain (normal) alkanes were quantitatively analyzed; branched alkanes are discussed briefly in Section 4.4.8. n-Hexane, n-dodecane, and n-tridecane are on the preliminary list of compounds of toxicological concern.

Table 4-13. Summary of Volatile Alkane Analyses.

Compound	Method	Date [1994]	Number of Samples	Mean (ppmv)	σ (ppmv)
Methane	SUMMA ^{TM1}	Nov. 1993	18	16.0	1.6
n-Hexane	SUMMA TM	May 16	6	0.72	0.07
		May 25	4	0.71	0.1
	TST	May 17	4	0.80	0.06
n-Heptane	TST	May 17	4	0.66	0.14
n-Octane	TST	May 17	4	0.33	0.09
n-Nonane	TST	May 17	4	0.28	0.08

¹Sample jobs 4 and 5, Einfeld 1994.

Table 4-14. Semivolatile Alkane Dimensionless Concentrations.

Compound	Method	Elevation above waste (m)	Date [1994]	Number of Samples	Mean (ppmv)	σ (ppmv)
n-Decane	CCT	0.79	May 16	10	0.57	0.06
n-Undecane	OVS	0.79	May 16	9	3.7	0.1
		0.79	May 19	5	3.6	0.2
		2.92	May 20	5	3.5	0.2
		5.05	May 20	5	3.3	0.3
	CCT	0.79	May 16	10	4.6	0.7
n-Dodecane	OVS	0.63 ¹	12/2/93	9	46.4	10.4
		0.79	May 16	5	45.6	7.4
		0.79	May 19	5	40.3	3.4
		2.92	May 20	5	36.2	3.0
		5.05	May 20	5	38.0	4.8
	CCT	0.79	May 16	10	40.3	7.2
n-Tridecane	OVS	0.63 ¹	12/2/93	9	63.0	13.5
		0.79	May 16	5	53.6	10.6
		0.79	May 19	5	44.4	4.3
		2.92	May 20	5	40.1	3.6
		5.05	May 20	5	44.9	5.6
	CCT	0.79	May 16	10	52.0	3.7
n-Tetradecane	OVS	0.63 ¹	12/2/93	9	13.9	4.2
		0.79	May 16	5	5.8	1.0
		0.79	May 19	5	6.0	0.5
		2.92	May 20	5	5.6	0.3
		5.05	May 20	5	6.0	0.6
	CCT	0.79	May 16	10	10.0	1.0

¹Sample job 6, OVS lowered into headspace.

SUMMA™ canister tank headspace samples were spiked with hexane, and it was determined that hexane is extracted with a 95.4% ($\sigma = 9.3$) efficiency. The precision of SUMMA™ canister analyses of n-hexane is excellent; agreement between analyses of individual samples in each set is good, and the agreement between the sample set averages of May 17 and 25, 1994 is also good.

TST samples were spiked with n-hexane, n-heptane, n-octane, and n-nonane. Jenkins et al. 1994 reports that the tank headspace concentrations of these alkanes were all within the range of the spike additions. For these four compounds, unspiked and (corrected) spiked sample results agreed well, and indicate chemical matrix effects for these compounds are small. Each of these alkanes were within or acceptably near the instrument calibration range.

Jenkins et al. 1994 also spiked TSTs with n-dodecane and n-tridecane. The amounts of these compounds in both the unspiked and spiked TSTs were much higher than the calibration range of the instrument, however, and have not been included here. Deuterated n-dodecane and n-tridecane spiked OVS trap samples were analyzed by Clauss et al., who concluded matrix effects were not significant.

The semivolatile NPH results given in Table 4-14 are those of Jenkins et al., Clauss et al. 1994, and Ligothke et al. 1994a. The consistency of these results is very good. Measurements of the NPH using OVS samples lowered into the tank 241-C-103 headspace are similar to the measurements of both OVS trap and CCT samples collected using the VSS. From the large number and good agreement of these sample results, a high degree of confidence can be placed in the established concentrations of semivolatile NPH.

Table 4-15 presents the data from table 4-14 in mass concentrations. Table 4-16 presents the average total NPH mass concentrations from each set of samples.

Table 4-15. Semivolatile Alkane Mass Concentrations.

Compound	Method	Elevation above waste (m)	Date [1994]	Number of Samples	Mean (mg/m ³)	σ (mg/m ³)
n-Decane	CCT	0.79	May 16	10	3.06	0.32
n-Undecane	OVS	0.79	May 16	9	21.8	0.9
		0.79	May 19	5	21.7	1.2
		2.92	May 20	5	20.9	1.2
		5.05	May 20	5	19.7	2.8
	CCT	0.79	May 16	10	27.4	4.4
n-Dodecane	OVS	0.63 ¹	12/2/93	9	301	68
		0.79	May 16	5	297	49
		0.79	May 19	5	262	22
		2.92	May 20	5	236	20
		5.05	May 20	5	248	45
	CCT	0.79	May 16	10	261	47
n-Tridecane	OVS	0.63 ¹	12/2/93	9	443	95
		0.79	May 16	5	377	75
		0.79	May 19	5	314	30
		2.92	May 20	5	283	26
		5.05	May 20	5	317	52
	CCT	0.79	May 16	10	365	26
n-Tetradecane	OVS	0.63 ¹	12/2/93	9	105	32
		0.79	May 16	5	44	7.6
		0.79	May 19	5	45	3.6
		2.92	May 20	5	42	2.4
		5.05	May 20	5	46	6.8
	CCT	0.79	May 16	10	75	7.5

¹Sample job 6, OVS lowered into headspace.

Table 4-16. Average Total Semivolatile NPH Mass Concentrations.

Method	Elevation above waste (m)	Date [1994]	Number of Samples	Mean (mg/m ³)	σ (mg/m ³)
OVS	0.63 ¹	12/2/93	9	850	188
OVS	0.79	May 16	9	704	108
	0.79	May 19	5	644	55
	2.92	May 20	5	582	47
	5.05	May 20	5	630	107
CCT	0.79	May 16	10	731	85
Liquid Sample ²	-	12/15/93	1	1,260	-

¹Sample job 6, OVS traps lowered into the headspace.

²Analysis of headspace vapors above organic liquid waste sample by Pool and Bean 1994.

4.4.7 TBP and DBBP

Table 4-17 summarizes the TBP and DBBP concentrations measured in the tank 241-C-103 headspace. Results are from the analyses of CCTs collected 0.79 m above the waste surface on May 16, 1994. TBP is on the preliminary list of compounds of toxicological concern.

While the headspace concentration of TBP resulted in a sorbent trap mass loading that was within the instrument calibration range, the mass of DBBP on the sorbent traps was lower than the instrument calibration range. Jenkins et al. 1994 also notes that analyses of TBP spiked samples gave confusing results.

Table 4-17. Summary of DBBP and TBP Analyses.

Compound	Number of Samples	Mean (ppmv)	σ (ppmv)
Dibutylbutyl Phosphonate (DBBP)	5	0.107	0.011
Tributyl Phosphate (TBP)	5	0.508	0.054

4.4.8 Tentatively Identified Organic Compounds

In addition to the positively identified, quantitatively analyzed compounds discussed in the preceding sections, many other compounds were tentatively identified. Jenkins et al. 1994 and Rasmussen and Einfeld 1994 provide lists of compounds tentatively identified by GC/MS analysis of the TST and SUMMA™ canister samples, respectively. Appendix A lists, in order of chromatographic elution (retention time) for Jenkins et al. 1994, these tentatively identified compounds. The probability of correct identification varies significantly, and some, as evidenced by 2,4-dimethyl-undecane appearing at two different elution times (129 and 155), must be misidentified. Mean concentrations given in Appendix A differ from those given by Jenkins et al. 1994 because they do not include zero values obtained from some of the TSTs.

Many of the tentatively identified compounds are semivolatile branched alkanes and alkenes that were probably impurities of the PUREX process NPH diluent. Alternately, these may be products resulting from the radiolysis and oxidation of TBP and NPH, either in PUREX or in tanks. Among the compounds are many alkanes, alkenes, alcohols, ketones, aldehydes, acids, and esters.

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5.0 SUMMARY

Tank 241-C-103 headspace gas and vapor characterization data have been presented in this report. The bulk of the characterization data is derived from samples collected May 12 through 25, 1994 using heated sample transfer tubes and a heated sampling manifold. Selected data from sampling events in November and December 1993, and in April 1994 are presented for comparison or when other data are not available.

Two types of sampling devices were used, SUMMA™ canisters, which collect and store complete air samples, and sorbent traps, which selectively collect constituents from an air sample passed through them. SUMMA™ canister samples were collected and analyzed for selected inorganic and volatile organic compounds. Sorbent trap samples were collected and analyzed for inorganic and organic gases and vapors.

Certain volatile organic vapors were analyzed in both SUMMA™ and sorbent trap samples, but compounds measured from the two types of sampling devices were otherwise mutually exclusive. Agreement between SUMMA™ and TST organic vapor results is very good, given that they are fundamentally different sampling methods, and analyses were performed using distinctly different methodologies at independent laboratories. The agreement between these two sampling and analysis methods is best for nonpolar compounds, and worst for polar compounds.

Table 5-1 summarizes data on the preliminary compounds of toxicological concern. Estimated concentrations are listed according to sampling method. Reported standard deviations are also given in Table 5-1 as the measure of uncertainty. Confidence in the validity of the sampling and analysis is warranted on the bases of: 1) the good total precision of analytical results; and 2) the very favorable comparison of values obtained by different methods. More detailed results have been given in Tables 4-1 through 4-17, and discussed in Section 4.0.

Samples of ammonia, hydrogen, nitrous oxide, and semivolatile organics were collected to assess vertical stratification of gases and vapors in the tank 241-C-103 headspace. Analysis of these samples consistently indicated no significant stratification exists. Lack of vertical stratification suggests thermally driven convection is mixing the headspace, which would cause relatively uniform gas and vapor concentrations throughout the headspace (laterally as well as vertically). Based on this, sample analyses discussed in this report are thought to be representative of the predominant convective region of the headspace.

The tank 241-C-103 headspace is nearly saturated with water vapor. The dewpoint of the headspace was determined to be about 36.3 °C, and the headspace temperature itself about 38 °C. Comparison of samples collected over the period of November 1993 to May 1994 indicates that the tank 241-C-103 headspace composition does not change significantly over short (24-hour) or long (90-day) time periods.

Table 5-1. Preliminary Compounds of Toxicological Concern.

Compound	Sample Type	n	Mean or Range (ppmv)	σ (ppmv)
Acetone	SUMMA™	10	19.2 - 19.4	2.0
	TST	4	8.8	3.9
Acetonitrile	SUMMA™	10	12.7 - 13.2	1.2
	TST	4	9.1	2.2
Ammonia	Sorbent Trap	35	304	11
Benzene	SUMMA™	16	<0.01 - 0.33	0.11
	TST	4	0.08	0.03
1,3-Butadiene	SUMMA™	10	<0.05 - 0.060	0.020
Butanal	SUMMA™	10	4.4 - 4.7	0.7
	TST	4	1.2	0.8
n-Butanol	SUMMA™	10	13.1	2.5
	TST	4	28.4	6.1
n-Dodecane	OVS	29	36.2 - 4.64	-
	CCT	10	40.3	7.2
n-Hexane	SUMMA™	10	0.71 - 0.72	0.1
	TST	4	0.80	0.06
2-Hexanone	SUMMA™	10	0.57 - 0.59	0.05
	TST	4	0.51	0.12
Methylene Chloride	SUMMA™	16	<0.02 - 0.061	0.030
	TST	4	1.62	1.47
Nitric Oxide	Sorbent Trap	8	1.5	0.3
Nitrogen Dioxide	Sorbent Trap	18	<0.06	-
Nitrous Oxide	SUMMA™	37	763	51
Propanenitrile	SUMMA™	10	5.1 - 5.3	0.7
	TST	4	3.3	0.3
Sulfur Oxides	Sorbent Trap	3	<0.02	-
Tributyl Phosphate	CCT	5	0.51	0.05
n-Tridecane	OVS	29	40.1 - 63.0	-
	CCT	10	52.0	3.7
Vinylidene Chloride	SUMMA™	21	<0.02	-
	TST	4	<0.009	-

The characterization of the tank 241-C-103 headspace has demonstrated the capability of the sampling equipment developed to sample the high-level waste tanks. With few exceptions, the sampling and analytical methodology chosen has been highly successful.

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APPENDIX A
TABLE OF TENTATIVELY IDENTIFIED COMPOUNDS

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APPENDIX A - TABLE OF TENTATIVELY IDENTIFIED COMPOUNDS

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
1	1-Butene	106-98-9	0.62	22.6	12.1
2	Butane	106-97-8		20.1	3.0
3	2-Methyl-1-Propene	115-11-7	0.85	16.1	18.2
4	(Z)-2-Butene	590-18-1	0.96	1.6	N. A.
5	Methyl Ether	115-10-6	2.55	4.2	N. A.
6	1-Pentene	109-67-1	3.45	10.1	1.1
7	Furan	110-00-9		9.7	N. A.
8	Carbon Disulfide	75-15-0	6.42	2.7	N. A.
9	4-Methyl-1-Pentene	691-37-2	8.35	1.6	N. A.
10	2-Methylpentane	107-83-5	9.25	4.7	N. A.
11	1,1-Dichloroethene	75-35-4		20.2	1.1
12	1-Hexene	592-41-6	11.37	12.6	3.3
13	3-Buten-2-one	78-94-4	11.53	3.6	N. A.
14	2-Butanone	78-93-3	12.38	42.0	11.8
15	2-Methylfuran	534-22-5	12.72	3.7	N. A.
16	Acetic Acid, ethyl ester	141-78-6	14.18	46.8	34.8
17	Tetrahydrofuran	109-99-9	14.94	15.9	3.7
18	Alkene			15.8	N. A.
19	2,5-Dihydrofuran	1708-29-8	17.05	5.7	N. A.
20	3-Methyl-2-Butanone	563-80-4	17.90	7.1	8.5
21	1,4-Butanediol, dinitrate	3457-91-8	23.22	2.1	N. A.
22	Formic acid, butyl ester	592-84-7	23.41	3.3	N. A.
23	Mixture (containing diethylmethyl borane)	1115-07-7	23.89	5.0	N. A.
24	4-Methyl-2-Pentanone	108-10-1	24.65	4.2	2.2

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
25	Mixture		25.66	1.5	N. A.
26	Acetic acid, 2-propenyl ester	591-87-7		4.6	N. A.
27	Cyclopropane, butyl	930-57-4	25.78	1.7	N. A.
28	C6-Alkene			8.7	N. A.
29	Mixture			8.3	N. A.
30	4-Methylheptane	589-53-7	26.54	2.0	N. A.
31	Alkane		27.05	1.7	N. A.
32	3-Hexanone	589-38-8	28.24	28.1	46.4
33	C6-Alkanone		28.57	18.3	N. A.
34	Hexanal	66-25-1	29.33	5.7	N. A.
35	2-Propenoic acid	79-10-7		4.4	N. A.
36	Acetic Acid, butyl ester	123-86-4	30.41	17.4	10.5
37	Tetramethylcyclotrisiloxane		30.78	4.0	N. A.
38	Hexamethyl-cyclotrisiloxane			6.3	2.2
39	Methyl pyridine		30.85	0.8	N. A.
40	Butanoic Acid	107-92-6		5.8	N. A.
41	Formic acid, 2-propenyl ester	1838-59-1	32.25	4.1	N. A.
42	Cyclopentanol	96-41-3		7.3	2.3
43	Cyclohexane	110-82-7	32.46	4.3	N. A.
44	Alkyl nitrile and others			10.5	5.3
45	C6-Alkanone			7.9	1.8
46	4-Methyl-2-Hexanone	105-42-0	32.87	5.5	N. A.
47	1-Propanol, 2,2-dimethyl	75-84-3		7.8	N. A.
48	2-Pyrrolidinone	616-45-5	33.34	1.0	N. A.
49	Mixture (containing methyl pyridine)		34.44	2.2	0.2

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
50	Alkane and C2-Benzene			2.0	1.3
51	Alkene and Alkane			1.0	N. A.
52	C7-Alkene			1.7	0.2
53	4-Heptanone	123-19-3		2.2	0.6
54	Butane, 1,1'-oxybis	142-96-1		2.3	N. A.
55	3-Heptanone	106-35-4	35.64	3.6	0.5
56	Styrene	100-42-5		1.3	N. A.
57	Pyrrolidine	123-75-1		2.1	N. A.
58	Methanamine, N,N-dimethyl-	75-50-3		1.7	N. A.
59	1-Heptanol	111-70-6		2.5	N. A.
60	4-Octanone	589-63-9	41.43	2.4	0.1
61	Butanoic acid	107-92-6		0.8	N. A.
62	Propanoic acid, butyl ester	590-01-2	37.05	1.1	0.7
63	Mixture		38.31	1.7	0.7
64	Alkanone			2.0	N. A.
65	Mixture			1.3	N. A.
66	2(3H)-Furanone, dihydro	96-48-0	38.66	3.5	4.0
67	Mixture			1.2	0.2
68	C8-Alkanone			14.6	2.2
69	6-Methyl-2-Heptanone	928-68-7	40.34	11.8	1.2
70	4-Octanone	589-63-9	41.43	2.2	0.4
71	C2-Pyrrolidine			1.3	N. A.
72	C3-Cyclohexane			1.0	0.2
73	C9-Alkenone			0.8	N. A.
74	Mixture			1.5	N. A.
75	3-Octanone	106-68-3	42.40	2.5	1.1

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
76	1-Heptanol	111-70-6		2.0	0.4
77	Butanoic acid, butyl ester	109-21-7		2.6	0.9
78	Cyclotetrasiloxane, octamethyl	556-67-2		5.4	5.1
79	Decane	124-18-5	43.13	18.4	6.5
80	Alkyl-Cyclohexane			1.8	0.5
81	Alkyl Cyclohexane and others			0.9	N. A.
82	Octanal	124-13-0		2.6	N. A.
83	C4-Cyclopentane			1.7	N. A.
84	2,4-Dimethylpyridine	108-47-4	44.03	0.5	N. A.
85	C2-Pyridine and others			0.9	N. A.
86	2,6-Dimethylnonane	17302-28-2	44.56	4.2	1.5
87	Alkene			1.0	0.1
88	1-Hexanol, 2-ethyl	104-76-7		1.5	0.4
89	Alkanol			1.7	0.4
90	alkyl-Cyclopentane			1.9	N. A.
91	Cyclohexane, (1-methylpropyl)-	7058-01-7		2.1	0.4
92	C4-Cyclohexane			1.4	N. A.
93	Alkanone			2.2	0.5
94	C4-Cyclohexane			1.2	0.6
95	C12-Alkene		46.69	3.2	0.7
96	C11-Alkane			1.2	0.0
97	C9-Alkanone			6.8	1.1
98	C8-Alkanone		47.10	4.6	1.5
99	Naphthalene, decahydro-,trans-	493-02-7	47.56	4.7	2.2
100	Alkanone			1.2	N. A.
101	Ethanone, 1-phenyl	98-86-2		2.4	0.3

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
102	C2-Benzene and others			1.6	N. A.
103	2-Nonanone	821-55-6		10.5	2.2
104	3-Nonanone	925-78-0		0.9	N. A.
105	2-Phenyl-2-Propanol	617-94-7	49.06	13.4	4.9
106	Undecane	1120-21-4	49.30	91.7	40.4
107	C11-Alkene			3.1	1.8
108	Nonanal	124-19-6		6.6	2.3
109	5-Methyl Undecane	1632-70-8		6.9	1.8
110	C11-Alkanone			6.6	N. A.
111	C12-Alkane			3.1	0.8
112	C13-Alkane			15.2	2.9
113	C12-Alkane			3.3	0.9
114	C12-Alkene			2.4	0.5
115	alkyl-Cyclohexane			4.8	1.7
116	Cyclotetradecane	295-17-0		15.8	1.2
117	Naphthalene, decahydro-2-methyl	2958-76-1	51.37	21.0	7.7
118	C12-Alkane			27.9	8.7
119	Alkane			13.8	N. A.
120	C12-Alkane			10.4	2.0
121	4-Methyl-Undecane			13.3	3.7
122	2-Methyl-Undecane	7045-71-8		28.4	8.8
123	3-Methyl-Undecane	1002-43-3		17.5	2.4
124	Butanedioic acid, diethyl ester	123-25-1		2.6	3.7
125	C12-Alkene			4.2	N. A.
126	C7-Cyclohexane			17.5	3.5

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
127	Alkane and Alkene			5.9	4.1
128	Alkane			4.0	1.5
129	2,4-Dimethyl-undecane	17312-80-0		13.8	21.2
130	2,6-Dimethylundecane	17301-23-4		41.5	14.2
131	Alkene			12.4	N. A.
132	3,7-Dimethyl-undecane	17301-29-0		24.2	7.8
133	3,7-Dimethyl-undecane	17301-29-0		2.7	N. A.
134	2,10-Dimethyl-undecane	17301-27-8		3.0	0.8
135	2,4,6-Trimethyldecane	62108-27-4		24.3	7.5
136	C13-Alkane			4.0	N. A.
137	C13-Alkane			9.1	N. A.
138	C13-Alkene			8.0	1.3
139	C7-Cyclohexane			28.5	13.9
140	C7-Cyclopentane			74.9	21.3
141	4-Methyl-dodecane	6117-97-1		19.3	2.7
142	2,10-Dimethyl-undecane	17301-27-8		42.8	8.2
143	C7-Cyclohexane			N. A.	N. A.
144	4,6-Dimethyldodecane	61141-72-8		62.2	21.7
145	Mixture			15.8	2.3
146	C14-Alkene			9.5	4.5
147	C14-Alkane			14.7	4.8
148	C14-Alkane			12.7	6.3
149	6-Methyl-tridecane	13287-21-8		29.0	14.8
150	Mixture			4.8	0.9
151	alkyl-Cyclohexane			5.3	1.0
152	C14-Alkane			10.2	1.8

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
153	C14-Alkene			5.5	0.8
154	C14-Alkane			8.4	1.2
155	2,4-Dimethyl-undecane	17312-80-0		4.6	N. A.
156	5-Methyl-tridecane	25117-31-1		12.1	N. A.
157	C7-Cyclohexane			29.7	17.5
158	C14-Alkene			N. A.	N. A.
159	2-Methyl-tridecane	1560-96-9		21.2	4.1
160	Alkanone		68.68	2.7	3.9
161	3-Methyl-tridecane	64418-41-3		12.4	0.9
162	C14-Alkane			17.9	0.9
163	C12-Alkanone			3.4	2.4
164	C15-Alkane			10.7	5.6
165	2,6,10-Trimethyl-dodecane	3891-98-3		59.0	19.6
166	2,6,11-Trimethyl-Dodecane	31295-56-4		47.9	N. A.
167	Mixture		69.08	1.9	N. A.
168	3-Tridecanone	1534-26-5		N. A.	N. A.
169	3-Dodecanone	1534-27-6		9.1	1.3
170	Tetradecane	629-59-4	65.79	94.1	34.7
171	Ascaridole	512-85-6		14.0	N. A.
172	Alkene			14.4	0.8
173	C14-Alkane			14.9	N. A.
174	Pentadecane	629-62-9		14.0	4.4
175	1,1'-Biphenyl	92-52-4		15.2	N. A.
176	1-Hexadecene	629-73-2	69.84	2.6	N. A.
177	C15-Alkane			9.4	11.9
178	C15-Alkene		67.43	20.9	1.5

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
179	C15-Alkene			2.5	0.2
180	Alkyl-cyclopentane			2.3	N. A.
181	Alkene			5.6	2.1
182	Alkene			2.4	0.4
183	Alkene			6.5	0.9
184	Decyl-cyclopentane	1795-21-7		5.3	N. A.
185	1-Tetradecene	1120-36-1		5.7	0.7
186	Alkane		68.03	1.6	N. A.
187	Alkane		68.8	7.4	N. A.
188	Hexadecane	544-76-3		38.6	6.9
189	Heptadecane	629-78-7		43.5	N. A.
190	Alkane			3.4	0.4
191	C13-Alkanone			9.8	1.6
192	Alkane			2.2	0.2
193	Alkyl ester of formic acid			3.1	0.7
194	3-Tridecanone	1534-26-5	69.40	5.4	1.0
195	C16-Alkane			30.1	2.1
196	Pentadecane	629-62-9	69.63	33.9	10.5
197	Alkanone			6.7	0.4
198	C9-Cyclohexane			2.2	0.4
199	Mixture			2.5	0.4
200	Phenol,2,4-bis(1,1-dimethylethyl)4-methyl	128-37-0	70.97	5.0	2.1
201	Alkanone			2.3	0.3
202	Alkane		71.88	1.7	1.8
203	2-Undecanone	112-12-9		2.8	N. A.

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
204	Alkane		72.22	1.0	0.1
205	Alkane			1.8	0.2
206	Alkane			N. A.	N. A.
207	C9-Cyclohexane		72.82	3.1	N. A.
208	alkyl-Cyclohexane			1.2	0.3
209	C15-Alkane			N. A.	N. A.
210	Butyl Myristate		73.22	1.9	N. A.
211	Alkanone			2.3	0.5
212	Mixture			1.9	0.8
213	3-Tetradecanone	629-23-2	73.59	1.4	0.1
214	Mixture			1.6	0.2
215	Hexadecane	544-76-3	73.73	2.9	0.6
216	Mixture			2.0	1.2
217	Alkanone		73.84	1.4	N. A.
218	Ester of Alkanoic Acid		74.03	0.5	N. A.
219	Phthalate		74.26	3.7	2.3
220	Dibutyl Butyl Phosphonate	78-46-4	74.56	7.9	2.1
221	Tributyl Phosphate	126-73-8	75.85	49.2	15.8
222	Tetradecanoic acid	544-63-8		7.5	0.6
223	Alkane			1.3	N. A.
224	Alkene			0.8	N. A.
225	Alkanol			0.8	N. A.
226	Butyl Myristate		77.21	0.6	N. A.
227	Alkanol			0.7	0.2
228	C16-Alkane			0.9	0.1
229	Alkanone			1.3	0.2

Peak #	Compound	CAS Number	Retention Time (min)	Mean (mg/m ³)	σ (mg/m ³)
230	Hexanedioic acid, dioctyl ester	123-79-5		0.9	0.0
231	Hexanedioic acid, dioctyl ester	123-79-5		1.4	N. A.
232	Tetradecanoic acid	544-63-8		1.6	0.8
233	Pentadecane	629-62-9	77.67	0.3	N. A.
234	2,3-Dihydro-farnesol			2.6	N. A.
235	Isopropyl Myristate		82.86	2.0	0.9
236	C14-Alkanoic Acid			1.1	N. A.
237	Pentadecanoic acid	1002-84-2		2.4	0.1
238	1-Hexadecanol	36653-82-4		1.6	N. A.
239	9-Hexadecanoic acid	2091-29-4		3.7	0.2
240	Alkanone			1.2	0.3
241	Alkanol			0.5	N. A.
242	Alkanone			0.6	N. A.
243	Hexadecanoic acid	57-10-3		4.2	5.5
244	Phthalate			1.5	N. A.
245	C16-Alkanoic Acid			10.8	N. A.
246	C16-Alkanoic Acid			1.9	N. A.

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