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7. Abstract

Significant changes have been made to all of the original vapor characterization reports. This report documents specific headspace gas and vapor characterization results for all vapor sampling events to date. In addition, changes have been made to the original vapor reports to qualify the data based on quality assurance issues associated with the performing laboratories.

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Tank 241-TY-101 Headspace Gas and Vapor Characterization Results for Samples Collected in August 1994 and April 1995

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Acronyms and Abbreviations

CES	consensus exposure standard
EPA	Environmental Protection Agency
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
ISS	<i>in situ</i> sampling
LFL	lower flammability limit
MS	mass spectrometer
NFPA	National Fire Protection Association
NPH	normal paraffinic hydrocarbon
ORNL	Oak Ridge National Laboratory
PNL	Pacific Northwest Laboratory
ppmv	parts per million by volume, 1 ppmv = 10 ⁻⁴ vol%
TST	triple sorbent trap
Vol%	percent by volume, 1 vol% = 10,000 ppmv
VSS	Vapor Sampling System
WHC	Westinghouse Hanford Company

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Tank 241-TY-101 Headspace Gas and Vapor Characterization Results

for Samples Collected in August 1994 and April 1995

1.0 INTRODUCTION

1.1 Characterization Objectives

Tank TY-101 headspace gas and vapor samples were collected and analyzed to help determine the potential risks of fugitive emissions to tank farm workers. The drivers and objectives of waste tank headspace sampling and analysis are discussed in *Program Plan for the Resolution of Tank Vapor Issues* (Osborne and Huckaby 1994). This report primarily discusses results from the April 1995 sampling event, but also includes selected results of the August 1994 sampling event. The tank TY-101 headspace was sampled in August 1994 in accordance with *Safety Assessment for Gas Sampling All Ferrocyanide Tanks* (Farley 1991), and in April 1995 in accordance with *Data Quality Objectives for Generic In-Tank Health and Safety Issue Resolution* (Osborne et al. 1994).

1.2 Characterization Data Criteria

Data Quality Objectives for Generic In-Tank Health and Safety Issue Resolution describes parameters for data collection to ensure appropriate conclusions can be drawn from the data. Tank headspace characterization data was collected to help in the evaluation of 1) headspace flammability, and 2) identification and quantification of compounds of toxicological concern.

Single Shell Tank Interim Operational Safety Requirements (Dougherty 1995) specifies that combustible constituents in tank headspaces be maintained below 25 % of the lower flammability limit (LFL). This essentially agrees with National Fire Protection Association requirements that combustible concentrations be maintained at or below 25 % of the LFL (NFPA 1992). However, current governing operating specifications for Watchlist tanks, such as tank TY-104, specify that combustible constituents be maintained at or below 20 % of the LFL (WHC 1995a).

Headspace characterization data are used by Westinghouse Hanford Company (WHC) Tank Waste Remediation Systems Industrial Hygiene as source term data in the industrial hygiene strategy to protect workers from tank fugitive emissions. Because selection of worker protective equipment must be based on industrial hygiene monitoring of the work place and not on source term data (29 CFR 1910.120), tank headspace characterization data can not be used for this purpose. Furthermore, because there are mechanisms by which headspace constituents can be either diluted or concentrated as they are released to the atmosphere, the headspace characterization data should not be considered to be representative of emissions at the point of emission.

These statements notwithstanding, the data quality objectives document specifies that the industrial hygiene group be advised if constituents with toxicological properties exceed 50 % of the appropriate consensus exposure standard (CES) for non-carcinogens, or 10 % of the appropriate CES for carcinogens. A CES is defined as the most stringent of known regulatory or recommended toxicological values for the workplace (Osborne et al. 1994).

1.3 Sampling Overview

Tank TY-101 was vapor sampled in August 1994 using the *in situ* sampling (ISS) method, and again in April 1995 using the more robust vapor sampling system (VSS) method. Because the ISS sample volume flow measurement used during the August 1994 event was inherently less accurate than that of the VSS, the sorbent trap samples from the ISS event are not considered to be equivalent to those from the VSS sampling event (Huckaby and Babad 1995). Furthermore, there are other discrepancies between results from the ISS and VSS methods that are not understood (Huckaby 1994a), and until the ISS method has been validated and the discrepancies resolved, results from early ISS events should be considered suspect.

Nevertheless, a brief description of the ISS event and hydrogen cyanide sampling results from the August 1994 ISS event are presented below, because this analyte was not sampled for during the VSS event. Huckaby and Babad (1995) describe the bases for using the ISS method for hydrogen cyanide, and why it was not sampled for during the VSS event. All other results presented here are from the April 1995 VSS sampling event.

Samples collected are thought to have been representative of the tank TY-101 headspace when the tank was sampled, and sample analyses were designed to provide a reasonably accurate and complete characterization of the significant headspace constituents (Meacham et al. 1995). No assessment has been made of how the tank TY-101 headspace composition changes with time, though studies of tank C-103 suggest that composition changes probably occur very slowly in the passively ventilated tanks (Huckaby and Story 1994).

1.4 Tank Headspace Dynamics

Tank TY-101 is the first tank in a 3-tank cascade with tanks TY-102 and TY-103. It is connected to tank TY-102 via a 7.4-cm (2.9-in.) inside diameter, 7.6-m (25-ft) long cascade line. Tanks TY-102 and TY-103 are connected by a similar cascade line. Because these cascade lines connect the headspaces of these tanks, gases and vapors originating from the wastes in tank TY-102 or tank TY-103 may be transferred to tank TY-101 (unless the cascade lines are obstructed). At this time, however, no headspace characterization data are available for tank TY-102 to assess the potential effect on tank TY-101.

The cascade of tanks TY-101, TY-102, and TY-103 is passively ventilated, which means that the tanks are allowed to exhale air, waste gases, and vapors as the barometric pressure falls, and inhale ambient air as the barometric pressure rises. Each of these tanks has its own filtered breather riser. Barometric pressure typically rises and falls on a diurnal cycle, producing an average daily exchange of air equal to about 0.46 % of each tank headspace (Huckaby 1994b). Changes in the concentrations of tank headspace constituents due to barometric pressure changes are consequently very slow.

2.0 SAMPLING EVENT

2.1 August 1994 *In Situ* Sampling Event

Tank TY-101 was sampled using ISS methods on August 4, 1994 by WHC Sampling and Mobile Laboratories. This sampling was conducted to satisfy requirements of *Safety Assessment for Gas Sampling All Ferrocyanide Tanks* (Farley 1991). Samples for hydrogen cyanide were collected from a point approximately 7.6 m below the top of the flange on riser 3, between 2:00 p.m. and 3:30 p.m.

Huckaby and Babad (1995) give a general description of the ISS method and equipment. Pingel (1994) provides field sampling information for the tank TY-101 August 1994 ISS event, and Mahon (1995) provides revised sample volume measurements. In addition to the hydrogen cyanide results presented below, Pool et al. (1995) provide sample preparation and analysis results for selected organic and inorganic vapors.

2.2 April 1995 Vapor Sampling System Sampling Event

Headspace gas and vapor samples were collected from tank TY-101 using the VSS on April 6, 1995 by WHC Sampling and Mobile Laboratories (WHC 1995b). Sample collection and analysis were performed as directed by *Tank 241-TY-101 Tank Characterization Plan* (Homi 1995). The tank headspace temperature was determined to be 15.6 °C. Air from the tank TY-101 headspace was withdrawn from a single elevation via a 5.5-m long heated sampling probe mounted in riser 3, and transferred via heated tubing to the VSS sampling manifold. All heated zones of the VSS were maintained at approximately 40 °C. All tank air samples were collected between 10:14 a.m. and 1:59 p.m., with no anomalies noted.

Sampling media were prepared and analyzed by WHC, Oak Ridge National Laboratories (ORNL), and Pacific Northwest Laboratories (PNL). The 40 tank air samples and 2 ambient air control samples collected are listed in Table 2-1 by analytical laboratory. Table 2-1 also lists the 14 trip blanks and 14 field blanks provided by the laboratories.

A general description of vapor sampling and sample analysis methods is given by Huckaby and Babad (1995). The sampling equipment, sample collection sequence, sorbent trap sample air flow rates and flow times, chain of custody information, and a discussion of the sampling event itself are given in WHC (1995b).

3.0 INORGANIC GASES AND VAPORS

Analytical results of sorbent trap and SUMMA^{TM,1} canister tank air samples for selected inorganic gases and vapors are given in Table 3-1 in parts per million by volume (ppmv) in dry air. The concentration of water vapor given in Table 3-1 has been adjusted to tank conditions given in Section 3.3. Inorganic analyte sorbent traps and SUMMATM canisters were prepared and analyzed by PNL (Pool et al. 1995, Klinger et al. 1995).

Analyses of the VSS event sorbent traps were performed within 13 days of sample collection, analyses of VSS event SUMMATM canisters for inorganic compounds were performed 12 days after sample collection, and analyses of the ISS event hydrogen cyanide sorbent traps were performed within 35 days of sample collection (Ligotke 1995). This satisfied the 60-day holding time requirement of the WHC quality assurance project plan (Keller 1994). Though no holding time study has been performed to address the stability of compounds in SUMMATM canisters in the chemical matrix of the tank samples, these inorganic compounds (i.e., the permanent gases) would be expected to be very stable in the SUMMATM canisters, and the results may not have been affected even if the 60 day holding time requirements had been exceeded. Sample preparation and analysis were performed at PNL quality assurance impact level 2, except for the hydrogen cyanide results, which were produced at PNL quality assurance impact level 3.

3.1 Ammonia, Hydrogen, and Nitrous Oxide

The concentration of ammonia in the headspace of TY-101 was measured to be 16 ppmv. Ammonia has been observed in virtually all of the passively ventilated waste tanks that have been sampled, at concentrations ranging from about 3 ppmv in tank C-108 (Lucke et al. 1995), to 1040 ppmv in BY-108 (McVeety et al. 1995). Given the LFL of ammonia in air is about 15 % by volume (vol%), the measured 16 ppmv corresponds to about 0.01 % of the LFL, and does not contribute appreciably to the flammability of the headspace.

The concentration of hydrogen in tank TY-101 was determined to be < 93 ppmv in each of the 3 SUMMATM canister samples. Hydrogen in the waste tanks is of concern as a fuel. Given that the LFL for hydrogen in air is about 4 vol% , a 93 ppmv hydrogen concentration in tank TY-101 would correspond to about 0.23 % of its LFL. At this concentration, hydrogen is not a flammability concern in tank TY-101.

The reported nitrous oxide concentration in tank TY-101, 98 ppmv, is typical of other passively ventilated tanks that have been sampled. Under the proper conditions, nitrous oxide can serve as an oxidizer to support combustion. However, Cashdollar et al. (1992) found that nitrous oxide had no significant effect on the flammability of hydrogen and air mixtures for hydrogen concentrations less than 20 vol%, and that "small amounts of nitrous oxide (relative to air) do not appear to have much effect on the flammability". Their results suggest the measured nitrous oxide concentration is much too low to have a significant effect on the flammability of the tank TY-101 headspace.

3.2 Carbon Monoxide and Carbon Dioxide

Carbon monoxide in the tank TY-101 headspace was measured to be < 12 ppmv in each of the 3 SUMMATM canister samples. In ambient air it typically ranges from 0.05 to 0.15 ppmv. Because different analytical methods have been used to measure carbon monoxide in the waste tanks, the information on carbon monoxide has varied from tank to tank. However, elevated waste tank headspace carbon monoxide

¹SUMMA is a trademark of Molectrics, Inc., Cleveland, Ohio.

concentrations are common, and are thought to be due to the decomposition of organic waste in the tanks. Carbon monoxide has not been measured at very high levels in any of the waste tanks, and the highest level measured to date was [26.7 ppmv] in tank C-103² (Huckaby and Story 1994).

The carbon dioxide concentration in the tank TY-101 headspace, measured in the 3 SUMMA™ canister samples to be 83 ppmv, is significantly lower than it is in ambient air. Carbon dioxide introduced by air exchange with the atmosphere is readily absorbed by caustic supernatant and interstitial liquids of the waste tanks, and converted to carbonate in solution. Carbon dioxide is normally present in the ambient air at a concentration of 350 to 400 ppmv, and is typically lower than ambient in the waste tank headspaces. The 2 ambient air samples collected at the start of the tank TY-101 April 1995 sampling event, for example, were measured to have an average 374 ppmv of carbon dioxide.

3.3 Nitric Oxide, Nitrogen Dioxide, Water and Tritium

Nitric oxide and nitrogen dioxide concentrations in the tank TY-101 headspace were determined to be 0.11 ppmv and ≤ 0.04 ppmv, respectively. These are acid gases that would have very low equilibrium concentrations above the high pH sludge in tank TY-101. The measurable presence of nitric oxide is not uncommon in the waste tank headspaces, and may be due to its formation from oxygen and nitrogen in the radiation field of the headspace. These constituents could potentially serve as oxidizers to support combustion, but at the measured concentrations would have a negligible effect on the flammability of the tank TY-101 headspace.

The water vapor concentration of tank TY-101 was determined to be about 10.3 mg/L, at the measured tank headspace temperature of 15.6 °C and pressure of 982.8 mbar (737.3 torr), (WHC 1995b). This corresponds to a water vapor partial pressure of 13.7 mbar (10.3 torr), to a dew point of 11.7 °C, and to a relative humidity of 77 %.

Silica gel sorbent traps were used to test for tritium. It is assumed that tritium produced by the waste combines with hydroxide ions to form tritium-substituted water. Evaporation of the tritium-substituted water would then result in airborne radioactive contamination. Silica gel sorbent traps adsorb virtually all (normal and tritium-substituted) water vapor from the sampled tank air, and are analyzed at the WHC 222-S laboratory. Radiochemical analysis of the silica gel trap indicated the total activity of the headspace to be less than 50 pCi/L (WHC 1995b).

3.4 Hydrogen Cyanide

Analysis of the hydrogen cyanide specific sorbent traps indicated the concentration of this analyte to be below 0.01 ppmv in all 3 samples. The absence of hydrogen cyanide at measurable concentrations is consistent with the expectation that an acid gas, such as hydrogen cyanide, would not exist at significant concentrations above the caustic waste in tank TY-101. No hydrogen cyanide has been detected in any of the 10 waste tank headspaces sampled for this analyte.

² The carbon monoxide measurement in tank C-103 was made by Oregon Graduate Institute of Science and Technology, and placed in brackets to emphasize it should be considered secondary data.

3.5 Discussion of Inorganic Gases and Vapors

Aside from water vapor and carbon dioxide, the most abundant waste constituents in the tank TY-101 headspace are nitrous oxide and ammonia. These compounds, along with hydrogen, have usually been the most abundant waste species. The concentration of nitrous oxide in tank TY-101 is typical of the passively ventilated tanks that have been sampled, but the ammonia concentration in TY-101 is relatively low.

The relative standard deviations of the inorganic gas and vapor results given in the last column of Table 3-1 are good for the methods used. Relative standard deviations range from 1.3 % for water vapor to about 18 % for nitric oxide results. The relatively poor precision of the nitric oxide results is due to the variability of background contamination present in these sorbent traps. Because the precision reported depends both on sampling parameters (e.g., sample flow rate and flow time for sorbent traps) and analytical parameters (e.g., sample preparation, dilutions, etc.), small relative standard deviations suggest proper control was maintained both in the field and in the laboratories.

4.0 ORGANIC VAPORS

Organic vapors in the tank TY-101 headspace were sampled using SUMMA™ canisters, which were analyzed by PNL, and triple sorbent traps (TSTs), which were analyzed by ORNL. Both PNL and ORNL used a gas chromatograph (GC) equipped with a mass spectrometer (MS) detector to separate, identify, and quantitate the analytes. Descriptions of sample device cleaning, sample preparations, and analyses are given by Jenkins et al. (1995a) and Klinger et al. (1995).

SUMMA™ sample results should be considered to be the primary organic vapor data for tank TY-101. PNL results were produced at PNL quality assurance impact level 2. All PNL SUMMA™ canister analyses were performed within 13 days after sample collection (Ligotke 1995), well within the specified 60-day holding time (Keller 1994).

ORNL analyses of TST samples from this and other waste tanks generally agree with, support, and augment the SUMMA™ sample results. However, because certain WHC quality assurance requirements were not satisfied by ORNL, the quality assurance assessment of ORNL by Hendrickson (1995) should be reviewed before results unique to the TST samples are used for decision making.

All TSTs prepared by ORNL had 3 surrogate compounds added to evaluate sample matrix effects, potential handling, storage, and shipment problems, and analytical instrumentation performance (Jenkins et al. 1995a). ORNL evaluated the surrogate recoveries using a statistical approach similar to that prescribed by *SW 846 Method 8260A Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Technique* (EPA 1992). Using this approach, ORNL reported that all surrogates had standard deviation values within the 95 % confidence interval for variance, indicating that no bias was introduced in the measurement of analyte quantities (Jenkins 1995a).

4.1 Positively Identified Organic Compounds

Positive identification of organic analytes using the methods employed by PNL and ORNL involves matching the GC retention times and MS data from a sample with that obtained by analysis of standards. The concentration of an analyte in the sample is said to be quantitatively measured if the response of the GC/MS has been established at several known concentrations of that analyte (i.e., the GC/MS has been calibrated for that analyte), and the MS response to the analyte in the sample is between the lowest and highest responses to the known concentrations (i.e., the analyte is within the calibration range).

ORNL and PNL were assigned different lists of organic compounds, or target analytes, to positively identify and measure quantitatively. The ORNL target analyte list was derived from a review of the tank C-103 headspace constituents by a panel of toxicology experts (Mahlum et al. 1994). The PNL target analyte list included 39 compounds in the Environmental Protection Agency (EPA) task order 14 (TO-14) method, which are primarily halocarbons and common industrial solvents (EPA 1988), plus 14 analytes selected mainly from the toxicology panel's review of vapor data on tank C-103.

Table 4-1 lists the organic compounds positively identified and quantitated in SUMMA™ samples. All compounds in Table 4-1 were analyzed according to the TO-14 methodology, except for the methane, which was analyzed with the inorganic gases (Klinger et al. 1995). Only 2 of the 39 TO-14 target analytes (trichlorofluoromethane and tetrachloromethane) and only 4 of the 14 additional target analytes were measured to be above the 0.005 ppmv detection limit of the analyses. Averages reported are from analyses of 3 SUMMA™ canister samples.

Jenkins et al. (1995a) report the positive identification of 23 of the 27 target analytes in TST samples. Dichloromethane, n-heptane, dibutyl butylphosphonate, and tributyl phosphate were the only TST target

analytes not detected. The average concentrations of the detected target analytes, from the analysis of 4 TSTs, are given in Tables 4-2, 4-3, and 4-4. Table 4-2 lists the positively identified, quantitated analytes, Table 4-3 lists the positively identified analytes which were not quantitated (because the measurement was outside the calibration range for at least 1 sample), and Table 4-4 lists the positively identified analytes which exceeded their practical holding times. Tables 4-2, 4-3, and 4-4 are mutually exclusive, so that no analyte appears in more than 1 of these tables. Despite calibration of the instrument over about a 20-fold concentration range, 17 of the 23 compounds positively identified in TST samples were not quantitated.

The ORNL practical holding time is defined as the holding time for which there is a 15 % risk that the concentration of an analyte in the sample will be below its initial concentration. As indicated in Table 4-4, butanal and 1-butanol exceeded their practical holding times, and may have been affected by the 25 day period between sample collection and analysis (Jenkins 1995b). Jenkins et al. (1995b) describe the ORNL practical holding time study, and report the practical holding times of butanal, and 1-butanol are 1 and 17 days, respectively. It is worth noting that the contractual holding time for the TST samples was 60 days.

Both PNL and ORNL report target analyte concentrations in ppmv of analyte in dry air. To correct for the measured water vapor content of tank TY-101 and obtain concentration in ppmv of analyte in moist tank air, multiply the dry-air ppmv concentrations by 0.986.

Eleven target analytes were common to both TST and SUMMATM analyses. Table 4-5 lists these, and their reported average concentrations in TST and SUMMATM samples. Results from these 2 sampling and analytical methods agree fairly well. As indicated in Table 4-5, the reported concentrations of propanone, ethanenitrile, and propanenitrile are higher in SUMMATM samples than in TST samples. However, the target analytes are generally only present at very low concentrations, and none are at levels of concern.

The only analyte in Tables 4-1 through 4-4 above 0.1 ppmv is propanone, measured to be an average 0.14 ppmv in SUMMATM samples. At the reported concentrations, the target analytes do not individually or collectively represent a flammability hazard.

4.2 Tentatively Identified Organic Compounds

In addition to the target analytes, the ORNL and PNL analytical procedures allow the tentative identification of other organic compounds. Tentative identification of analytes was performed by comparing the MS molecular fragmentation patterns with a library of known MS fragmentation patterns. This method allows an organic analyte to be identified (with reasonable certainty) as an alkane, a ketone, an aldehyde, etc., and may also determine its molecular weight. The method usually does not, however, allow the unambiguous identification of structural isomers, and this ambiguity increases with analyte molecular weight. Using this method, many analytes can be tentatively identified with reasonable confidence without having to inject standards of each into the GC/MS to determine their GC retention times or specific MS patterns.

By the nature of the sampling devices, virtually all organic vapors present in the tank headspace are collected by both TST and SUMMATM samples. Analyses of the samples are designed to recover, separate, and identify the organic vapors in the samples. TSTs are not good for collecting highly volatile compounds (i.e., molecules more volatile than propane), but are quite good for most others. In contrast, the recovery of very low volatility compounds (e.g., molecules with more than about 15 carbon atoms) and some polar compounds with moderate volatility (e.g., butanal) from SUMMATM samples has been problematic.

The sole tentatively identified compound reported in SUMMATM samples, methanol, with its estimated concentration, is given in Table 4-6. The list of tentatively identified compounds detected in TST samples, and their estimated concentrations, is given in Table 4-7. Compounds are listed in Table 4-7 according to

the order by which they eluted chromatographically. The averages reported by ORNL in Table 4-7 are all 4-sample averages, and if an analyte was not detected in a sample, its concentration in that sample was considered to be zero for averaging purposes. Estimated concentrations are in mg/m³, based on dry air at 0 °C and 1.01 bar.

Because the list of tentatively identified organic compounds in TST samples is rather long and locating any given compound may be difficult, the list has been sorted both alphanumerically by compound name (Table 4-8), and also in order of decreasing estimated concentrations (Table 4-9). Numbers in the first columns of Tables 4-8 and 4-9 (Cmpd #) identify the location of the compound in Table 4-7.

The ORNL and PNL methods used to tentatively identify and estimate concentrations are described by Jenkins et al. (1995a) and Klinger et al. (1995), respectively, and should be reviewed before this data is used for decision making. Concentrations given in Tables 4-6 and 4-7 should be considered rough estimates.

4.3 Discussion of Organic Compounds

The organic compounds listed in Tables 4-1 through 4-9 may be classified as either 1) organic compounds added to tank TY-101 as waste that are still evaporating, or 2) organic compounds that have been generated by reactions of the original waste.

Organic vapors in the first category, those that were placed into the tank as waste, include the semivolatile straight-chain alkanes, which were used as diluents of tributyl phosphate in various plutonium extraction processes. These alkanes (i.e., n-undecane, n-dodecane, n-tridecane, n-tetradecane, and n-pentadecane) are often referred to in Hanford site literature as the normal paraffinic hydrocarbons (NPHs). Though NPHs are positively identified in tank TY-101, their concentrations are very low compared to other NPH-rich tanks in the 241-BY and 241-C farms.

The tentatively identified cyclosiloxanes (i.e., Cmpd # 17 and 24 in Table 4-7) may also have been sent to tank TY-101 with other wastes. Small quantities of organo-silicon compounds may have been introduced to the waste tank through their use as defoaming agents, but they may also be present in the headspace due to their use in liquid traps at the tank's breather riser. Several of these have also been observed in TST blanks, however, and they may be present as artifacts of the gas chromatography process.

The absence of tributyl phosphate in the tank TY-101 samples does not necessarily indicate it is not present in the headspace or the waste. The identification of the tributyl phosphate diluents and their degradation products is reason to expect tributyl phosphate may be present in the tank waste. 1-Butanol, which was positively identified in tank TY-101 TST samples, is known to be a product of the hydrolysis of tributyl phosphate. Furthermore, informal tests by ORNL indicate that tributyl phosphate is adsorbed by the glass fiber filters used during sampling to protect the samples from radiolytic particulate contamination. Based on these considerations, the lack of tributyl phosphate in the tank TY-101 headspace samples should not be taken as proof it is not present in the headspace or the waste.

A relatively large number of halogenated organic compounds were detected in samples from tank TY-101, including 1,1,1-trichloroethane, and tetrachloroethene. These compounds may have been used as cleaning solvents and may have been sent to the waste tanks when they became radiologically contaminated. Cmpd # 40 in Table 4-7 (2,3,3',5'-tetrachloro-1,1'-biphenyl) is a polychlorinated biphenyl (PCB).

The second category includes all organic compounds that have been generated via radiolytic and chemical reactions of the waste. The majority of compounds listed in Tables 4-1 through 4-9 fall into this category,

including the alcohols, aldehydes, alkenes, ketones, nitriles, and volatile alkanes, all of which have been associated with the degradation of the NPHs.

The total organic vapor concentration of tank TY-101 was estimated by Jenkins et al. to be about 1.0 mg/m³ from the analysis of 3 TST samples by GC/MS. A similar summation of organic compounds measured in SUMMATM samples from tank TY-101 provides an estimated total organic vapor concentration of 1.65 mg/m³. This is a relatively low concentration of organic vapors compared to most other passively ventilated tanks that have been sampled.

In summary, the organic vapors in tank TY-101 clearly indicate the presence of the semivolatile NPHs and their degradation products in the tank waste, though the NPHs are at trace levels. Tributyl phosphate was not detected in any of the headspace samples, but there is strong evidence that it is also present in the waste.

5.0 SUMMARY

The tank TY-101 headspace was sampled in August 1994 and April 1995 for gases and vapors to address flammability and industrial hygiene concerns. Results unique to the August 1994 event and essentially all results from the April 1995 event have been reported. It was determined that no headspace constituents exceeded the flammability or industrial hygiene notification limits specified in the current *Vapor Sampling and Analysis Plan* (Homi 1995).

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Table 2-1
Tank TY-101 Gas and Vapor Sample Type and Number for April 1995 Sampling Event

Laboratory	Sampling Device	Nominal Sample Volume (L)	Target Analytes	Number of Samples
Oak Ridge National Laboratories	Triple Sorbent Trap	0.2	Organic vapors	4 tank air samples
		1.0	Organic vapors	4 tank air samples
		4.0	Organic vapors	4 tank air samples
				+ 2 trip blanks
				+ 2 field blanks
Pacific Northwest Laboratories	Acidified Carbon Sorbent Trap	3.0	Ammonia	6 tank air samples
				+ 3 trip blank
	Triethanolamine Sorbent Trap	3.0	Nitrogen Dioxide	6 tank air samples
				+ 3 trip blank
	Oxidation Bed + Triethanolamine Sorbent Trap	3.0	Nitric Oxide	6 tank air samples
				+ 3 trip blank
	Silica Gel Sorbent Trap	3.0	Water vapor	6 tank air samples
				+ 3 trip blanks
	SUMMA™ canister	6.0	Carbon Dioxide, Carbon Monoxide, Hydrogen, Methane, Nitrous Oxide, Organic vapors	3 tank air samples + 2 ambient air samples
WHC 222-S Laboratory	Silica Gel Sorbent Trap	1.0	Tritium-Substituted Water Vapor	1 tank air sample

Table 3-1
Tank TY-101 Inorganic Gas and Vapor Concentrations --
Analyses by Pacific Northwest Laboratory

Compound	CAS ¹ Number	Sample Type	Number of Samples	Average (ppmv)	Standard Deviation (ppmv)	RSD ² (%)
Ammonia, NH ₃	7664-41-7	Sorbent Trap	6	16	1	6
Carbon Dioxide, CO ₂	124-38-9	SUMMA TM	3	83	4	5
Carbon Monoxide, CO	630-08-0	SUMMA TM	3	< 12	--	--
Hydrogen, H ₂	1333-74-0	SUMMA TM	3	< 93	--	--
Hydrogen Cyanide ³ , HCN	74-90-8	Sorbent Trap	3	< 0.01	--	--
Nitric Oxide, NO	10102-43-9	Sorbent Trap	6	0.11	0.02	18
Nitrogen Dioxide, NO ₂	10102-44-0	Sorbent Trap	6	≤ 0.04	--	--
Nitrous Oxide, N ₂ O	10024-97-2	SUMMA TM	3	98	6	6
Water Vapor, H ₂ O	7732-18-5	Sorbent Trap	6	14,000 (10.3 mg/L)	200 (0.1 mg/L)	1.3

¹ CAS = Chemical Abstracts Service.

² RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

³ Results for hydrogen cyanide are from the August 1994 ISS event.

Table 4-1
Tank TY-101 Positively Identified Organic Compounds in SUMMA™ Samples
Analyses by Pacific Northwest Laboratory

Cmpd #	Compound	CAS ¹ Number	Average ² (ppmv)	Standard Deviation (ppmv)	RSD ³ (%)
1	Ethanenitrile (acetonitrile)	75-05-8	0.079	0.008	10
2	Propanone (acetone)	67-64-1	0.14	0.02	15
3	Trichlorofluoromethane	75-69-4	0.040	0.008	21
4	Propanenitrile	107-12-0	0.006	0.0007	12
5	Tetrachloromethane (carbon tetrachloride)	56-23-5	0.006	0.0001	2
6	2-Butanone ⁴	78-93-3	0.008	—	—
7	Methane	74-82-8	< 12	—	—
Sum of positively identified compounds:			0.86	mg/m ³	

1 CAS = Chemical Abstract Service.

2 Average of 3 samples.

3 RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

4 Detected in only 1 sample.

Table 4-2
Tank TY-101 Quantitatively Measured Organic Compounds in TST Samples –
Analyses by Oak Ridge National Laboratory¹

Cmpd #	Compound	CAS ² Number	Average ³ (ppmv)	Standard Deviation (ppmv)	RSD ⁴ (%)
1	Ethanenitrile (acetonitrile)	75-05-8	0.029	0.005	16
2	n-Dodecane	112-40-3	0.0041	0.0002	6

1 Results in this table are quantitative (as defined in Section 4.1).

2 CAS = Chemical Abstract Service.

3 Average of 4 TST samples; 3 were 4-L and 1 was 1-L.

4 RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

Table 4-3
Tank TY-101 Positively Identified Organic Compounds in TST Samples --
Analyses by Oak Ridge National Laboratory¹

Cmpd #	Compound	CAS ² Number	Average ³ (ppmv)	Standard Deviation (ppmv)	RSD ⁴ (%)
1	Propanone (acetone)	67-64-1	0.084	0.011	13
2	1,1-Dichloroethene (vinylidene chloride)	75-35-4	0.0024	0.0020	85
3	Propanenitrile	107-12-0	0.0038	0.0003	8
4	n-Hexane	110-54-3	0.00015	0.00008	57
5	Benzene	71-43-2	0.00022	0.00018	81
6	n-Butanenitrile	109-74-0	0.0012	0.0001	9
7	2-Pentanone	107-87-9	0.00064	0.00047	73
8	Toluene	108-88-3	0.00065	0.00054	82
9	n-Pentanenitrile	110-59-8	0.00036	0.00002	6
10	2-Hexanone	591-78-6	0.00033	0.00030	91
11	n-Octane	111-65-9	0.000055	0.000038	69
12	n-Hexanenitrile	628-73-9	0.00011	0.00008	67
13	2-Heptanone	110-43-0	0.00022	0.00008	38
14	n-Nonane	111-84-2	0.00011	0.00008	74
15	n-Heptanenitrile	629-08-3	0.000088	0.000059	67
16	2-Octanone	111-13-7	0.000085	0.000057	67
17	n-Decane	124-18-5	0.00015	0.00005	32
18	n-Undecane	1120-21-4	0.00036	0.00007	20
19	n-Tridecane	629-50-5	0.015	0.002	15

1 Results in this table are not quantitative (as defined in Section 4.1) because measured values in at least 1 of the samples are outside instrument calibration limits.

2 CAS = Chemical Abstract Service.

3 Average of 4 TST samples: 3 were 4-L and 1 was 1-L.

4 RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

Table 4-4
Tank TY-101 Positively Identified Compounds in TST Samples
for which Practical Holding Times were Exceeded –
Analyses by Oak Ridge National Laboratory¹

Cmpd #	Compound	CAS ² Number	Average ³ (ppmv)	Standard Deviation (ppmv)	RSD ⁴ (%)
1	Butanal ⁵	123-72-8	0.0071	0.0010	15
2	1-Butanol ⁵	71-36-3	0.0027	0.0005	19

1 Practical holding times are defined and discussed in Section 4.1.

2 CAS = Chemical Abstract Service.

3 Average of 4 TST samples: 3 were 4-L and 1 was 1-L.

4 RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

5 The concentration of this analyte was not quantitatively measured (as defined in Section 4.1), because the measured concentration was outside of the instrumental calibration limits.

Table 4-5
Tank TY-101 Comparison of Organic Compounds in TST and SUMMA™ Samples --
Analyses by Pacific Northwest Laboratory
and Oak Ridge National Laboratory

Compound	CAS ¹ Number	TST Average ² (ppmv)	SUMMA™ Average ³ (ppmv)	PRD ⁴ (%)
1,1-Dichloroethene (vinylidene chloride)	75-35-4	0.0024	< 0.005	—
Dichloromethane (methylene chloride)	75-09-2	< 0.0013	< 0.005	—
Propanone (acetone)	67-64-1	0.084	0.14	50
Ethanenitrile (acetonitrile)	75-05-8	0.029	0.079	92
Propanenitrile	107-12-0	0.0038	0.006	45
n-Butanenitrile	109-74-0	0.0012	< 0.005	—
Benzene	71-43-2	0.00022	< 0.005	—
Toluene	108-88-3	0.00065	< 0.005	—
n-Hexane	110-54-3	0.00015	< 0.005	—
n-Heptane	142-82-5	< 0.00011	< 0.005	—
n-Decane	124-18-5	0.00015	< 0.005	—

1 CAS = Chemical Abstract Service.

2 Average of 4 TST samples: 3 were 4-L and 1 was 1-L.

3 Average of 3 sample analyses by PNL.

4 PRD = percent relative difference. Keller (1994) requires the PRD to be less than 20 %.

Table 4-6
Tank TY-101 Tentatively Identified Organic Compounds in SUMMA™ Samples
Analyses by Pacific Northwest Laboratory

Cmpd #	Compound	CAS ¹ Number	Average (mg/m ³)	Standard Deviation (mg/m ³)
1	Methanol (methyl alcohol)	67-56-1	0.79	0.14

1 CAS = Chemical Abstract Service.

Table 4-7
Tank TY-101 Tentatively Identified Organic Compounds in TST Samples
in Order of Chromatographic Elution --
Analyses by Oak Ridge National Laboratory

Cmpd #	Compounds	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
1	Methanol	67-56-1	0.050	0.036
2	Dichlorofluoromethane	75-43-4	0.00077	0.00090
3	Ethanol	64-17-5	0.0020	0.0013
4	Trichlorofluoromethane	75-69-4	0.042	0.009
5	Silane, chlorotrimethyl	75-77-4	0.0064	0.0091
6	2-Propanol (isopropyl alcohol)	67-63-0	0.0090	0.0023
7	Ethane, 1,1,2-trichloro-1, 2,2-trifluoro-	76-13-1	0.00043	0.00086
8	Hydrazine, methyl-	60-34-4	0.0021	0.0016
9	2-Butanone	78-93-3	0.00047	0.00094
10	Trichloromethane (chloroform)	67-66-3	0.00096	0.00111
11	Ethane, 1,1,1-trichloro-	71-55-6	0.00042	0.00084
12	Nitric acid, propyl ester	627-13-4	0.00033	0.00066
13	Propylene Glycol	57-55-6	0.072	0.070
14	Furan, 2,3-dihydro-	1191-99-7	0.00039	0.00078
15	Hexanal	66-25-1	0.0017	0.0012
16	Tetrachloroethylene	127-18-4	0.0080	0.0002
17	Cyclotrisiloxane, hexamethyl	541-05-9	0.036	0.051
18	Heptanal	111-71-7	0.0036	0.0014
19	Pentanol, 5-amino-	2508-29-4	0.0016	0.0011
20	2(3H)-Furanone, dihydro-5-methyl-	108-29-2	0.00036	0.00071
21	Cyclotetrasiloxane, octamethyl	556-67-2	0.017	0.021
22	Octanal	124-13-0	0.0021	0.0015
23	1-Hexanol, 2-ethyl-	104-76-7	0.0015	0.0010
24	2(3H)-Furanone, 5-ethyldihydro-	695-06-7	0.0014	0.0009
25	Nonanal	124-19-6	0.0025	0.0017
26	Benzoic acid, 2-[(trimethylsilyl)oxy]-trimethylsilyl ester	3789-85-3	0.0062	0.0090

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Cmpd #	Compounds	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
27	3-Dodecene, (E)-	7206-14-6	0.00033	0.00067
28	Decanal	112-31-2	0.00069	0.00080
29	Methenamine	100-97-0	0.024	0.035
30	Cyclohexane, 2-butyl-1,1,3-trimethyl-	54676-39-0	0.0004	0.0007
31	2-Furancarboxylic acid, hexyl ester and others		0.0012	0.0008
32	2,2'-Bioxepane	74793-02-5	0.0015	0.0010
33	n-Tetradecane	629-59-4	0.052	0.004
34	6-Tridecanone	22026-12-6	0.0036	0.0032
35	n-Pentadecane	629-62-9	0.0039	0.0026
36	n-Hexadecane	544-76-3	0.00067	0.00077
37	Isopropyl Myristate	110-27-0	0.0066	0.0133
38	1-Pentadecanol	629-76-5	0.0053	0.0106
39	Benzenesulfonamide, N-butyl	3622-84-2	0.0079	0.0068
40	1,1'-Biphenyl, 2,3,3',5'-tetrachloro-	41464-49-7	0.0037	0.0025
41	Isopropyl Palmitate	142-91-6	0.13	0.26
42	Hexadecanoic acid	57-10-3	0.0034	0.0068
Sum of tentatively identified compounds:			0.51 mg/m ³	

1 CAS = Chemical Abstract Service.

2 Average of 4 TST samples: 3 were 4-L and 1 was 1-L, values listed are estimates.

Table 4-8
 Tank TY-101 Tentatively Identified Organic Compounds in TST Samples
 Sorted Alphanumerically --
 Analyses by Oak Ridge National Laboratory

Cmpd #	Compounds	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
38	1-Pentadecanol	629-76-5	0.0053	0.0106
23	1-Hexanol, 2-ethyl-	104-76-7	0.0015	0.0010
31	2-Furancarboxylic acid, hexyl ester and others		0.0012	0.0008
9	2-Butanone	78-93-3	0.00047	0.00094
6	2-Propanol (isopropyl alcohol)	67-63-0	0.0090	0.0023
27	3-Dodecene, (E)-	7206-14-6	0.00033	0.00067
34	6-Tridecanone	22026-12-6	0.0036	0.0032
40	1,1'-Biphenyl, 2,3,3',5'-tetrachloro-	41464-49-7	0.0037	0.0025
32	2,2'-Bioxepane	74793-02-5	0.0015	0.0010
20	2(3H)-Furanone, dihydro-5-methyl-	108-29-2	0.00036	0.00071
24	2(3H)-Furanone, 5-ethyldihydro-	695-06-7	0.0014	0.0009
39	Benzenesulfonamide, N-butyl	3622-84-2	0.0079	0.0068
26	Benzoic acid, 2-[(trimethylsilyl)oxy]-trimethylsilyl ester	3789-85-3	0.0062	0.0090
30	Cyclohexane, 2-butyl-1,1,3-trimethyl-	54676-39-0	0.0004	0.0007
21	Cyclotetrasiloxane, octamethyl	556-67-2	0.017	0.021
17	Cyclotrisiloxane, hexamethyl	541-05-9	0.036	0.051
28	Decanal	112-31-2	0.00069	0.00080
2	Dichlorofluoromethane	75-43-4	0.00077	0.00090
7	Ethane, 1,1,2-trichloro-1, 2,2-trifluoro-	76-13-1	0.00043	0.00086
11	Ethane, 1,1,1-trichloro-	71-55-6	0.00042	0.00084
3	Ethanol	64-17-5	0.0020	0.0013
14	Furan, 2,3-dihydro-	1191-99-7	0.00039	0.00078
18	Heptanal	111-71-7	0.0036	0.0014
42	Hexadecanoic acid	57-10-3	0.0034	0.0068
15	Hexanal	66-25-1	0.0017	0.0012

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Cmpd #	Compounds	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
8	Hydrazine, methyl-	60-34-4	0.0021	0.0016
41	Isopropyl Palmitate	142-91-6	0.13	0.26
37	Isopropyl Myristate	110-27-0	0.0066	0.0133
1	Methanol	67-56-1	0.050	0.036
29	Methenamine	100-97-0	0.024	0.035
36	n-Hexadecane	544-76-3	0.00067	0.00077
35	n-Pentadecane	629-62-9	0.0039	0.0026
33	n-Tetradecane	629-59-4	0.052	0.004
12	Nitric acid, propyl ester	627-13-4	0.00033	0.00066
25	Nonanal	124-19-6	0.0025	0.0017
22	Octanal	124-13-0	0.0021	0.0015
19	Pentanol, 5-amino-	2508-29-4	0.0016	0.0011
13	Propylene Glycol	57-55-6	0.072	0.070
5	Silane, chlorotrimethyl	75-77-4	0.0064	0.0091
16	Tetrachloroethylene	127-18-4	0.0080	0.0002
4	Trichlorofluoromethane	75-69-4	0.042	0.009
10	Trichloromethane (chloroform)	67-66-3	0.00096	0.00111
Sum of tentatively identified compounds:			0.51 mg/m ³	

1 CAS = Chemical Abstract Service

2 Average of 4 TST samples: 3 were 4-L and 1 was 1-L, values listed are estimates.

Table 4-9
 Tank TY-101 Tentatively Identified Organic Compounds in TST Samples
 Sorted by Estimated Concentration --
 Analyses by Oak Ridge National Laboratory

Cmpd #	Compounds	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
41	Isopropyl Palmitate	142-91-6	0.13	0.26
13	Propylene Glycol	57-55-6	0.072	0.070
33	n-Tetradecane	629-59-4	0.052	0.004
1	Methanol	67-56-1	0.050	0.036
4	Trichlorofluoromethane	75-69-4	0.042	0.009
17	Cyclotrisiloxane, hexamethyl	541-05-9	0.036	0.051
29	Methenamine	100-97-0	0.024	0.035
21	Cyclotetrasiloxane, octamethyl	556-67-2	0.017	0.021
6	2-Propanol (isopropyl alcohol)	67-63-0	0.0090	0.0023
16	Tetrachloroethylene	127-18-4	0.0080	0.0002
39	Benzenesulfonamide, N-butyl	3622-84-2	0.0079	0.0068
37	Isopropyl Myristate	110-27-0	0.0066	0.0133
5	Silane, chlorotrimethyl	75-77-4	0.0064	0.0091
26	Benzoic acid, 2-[(trimethylsilyl)oxy]-trimethylsilyl ester	3789-85-3	0.0062	0.0090
38	1-Pentadecanol	629-76-5	0.0053	0.0106
35	n-Pentadecane	629-62-9	0.0039	0.0026
40	1,1'-Biphenyl, 2,3,3',5'-tetrachloro-	41464-49-7	0.0037	0.0025
18	Heptanal	111-71-7	0.0036	0.0014
34	6-Tridecanone	22026-12-6	0.0036	0.0032
42	Hexadecanoic acid	57-10-3	0.0034	0.0068
25	Nonanal	124-19-6	0.0025	0.0017
22	Octanal	124-13-0	0.0021	0.0015
8	Hydrazine, methyl-	60-34-4	0.0021	0.0016
3	Ethanol	64-17-5	0.0020	0.0013
15	Hexanal	66-25-1	0.0017	0.0012
19	Pentanol, 5-amino-	2508-29-4	0.0016	0.0011

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Cmpd #	Compounds	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
23	1-Hexanol, 2-ethyl-	104-76-7	0.0015	0.0010
32	2,2'-Bioxepane	74793-02-5	0.0015	0.0010
24	2(3H)-Furanone, 5-ethyldihydro-	695-06-7	0.0014	0.0009
31	2-Furancarboxylic acid, hexyl ester and others		0.0012	0.0008
10	Trichloromethane (chloroform)	67-66-3	0.00096	0.00111
2	Dichlorofluoromethane	75-43-4	0.00077	0.00090
36	n-Hexadecane	544-76-3	0.00067	0.00077
28	Decanal	112-31-2	0.00069	0.00080
11	Ethane, 1,1,1-trichloro-	71-55-6	0.00042	0.00084
9	2-Butanone	78-93-3	0.00047	0.00094
30	Cyclohexane, 2-butyl-1,1,3-trimethyl-	54676-39-0	0.0004	0.0007
7	Ethane, 1,1,2-trichloro-1, 2-2-trifluoro-	76-13-1	0.00043	0.00086
12	Nitric acid, propyl ester	627-13-4	0.00033	0.00066
20	2(3H)-Furanone, dihydro-5-methyl-	108-29-2	0.00036	0.00071
27	3-Dodecene, (E)-	7206-14-6	0.00033	0.00067
14	Furan, 2,3-dihydro-	1191-99-7	0.00039	0.00078
Sum of tentatively identified compounds:			0.51 mg/m ³	

1 CAS = Chemical Abstract Service.

2 Average of 4 TST samples: 3 were 4-L and 1 was 1-L, values listed are estimates.