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CHARACTERIZATION RESULTS FOR SAMPLES COLLECTED
IN 3/1995

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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.

8. RELEASE STAMP

Tank 241-SX-103 Headspace Gas and Vapor Characterization Results for Samples Collected in March 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
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-SX-103 Headspace Gas and Vapor Characterization Results
for Samples Collected in March 1995**

1.0 INTRODUCTION

1.1 Characterization Objectives

Tank SX-103 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). Tank SX-103 was vapor sampled 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 were 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 single shell Watchlist waste tanks, such as tank SX-103, 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 headspace characterization data presented here are from a single sampling event. Samples collected are thought to have been reasonably representative of the tank SX-103 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). However, tank SX-103 is an actively ventilated tank, and the representativeness of the samples depends on such parameters as ventilation flow rate and locations of all air inlets and outlets as well as the sample collection point. A prime consideration is the ventilation rate, which is not well known. Furthermore, no assessment has been made of how the tank SX-103 ventilation rate or headspace composition changes with time.

1.3 Tank Headspace Dynamics

Tank SX-103 is the third tank in a 3-tank cascade with tanks SX-101 and SX-102. Tanks SX-101 and SX-102 are connected via underground piping, and similar piping connects tanks SX-102 and SX-103. Since the piping connects the headspaces of these tanks, gases and vapors originating from the wastes in tanks SX-102 and SX-101 may be transferred to tank SX-103 (unless the cascade lines are obstructed). By design, the ventilation of tanks SX-101 and SX-102 occurs via the underground piping and through tank SX-103. At this time, however, no headspace characterization data are available for either tanks SX-102 or SX-101 to assess their potential effect on tank SX-103.

Unlike most passively ventilated tanks, for which characterization of the headspace provides direct evidence of volatile constituents of the waste, the active ventilation of tank SX-103 and the fact that other tanks are vented through it negates direct use of the data for that purpose. The headspace characterization data are still believed to be the best available for assessing flammability and industrial hygiene concerns.

2.0 SAMPLING EVENT

Headspace gas and vapor samples were collected from tank SX-103 using the vapor sampling system (VSS) on March 23, 1995 by WHC Sampling and Mobile Laboratories (WHC 1995b). Sample collection and analysis were performed as directed by *Tank 241-SX-103 Tank Characterization Plan* (Homi 1995a). The tank headspace temperature was determined to be 18 °C. Air from the tank SX-103 headspace was withdrawn via a 7.3 m-long heated sampling probe mounted in riser 2, and transferred via heated tubing to the VSS sampling manifold. All heated zones of the VSS were maintained at approximately 50 °C. All samples were collected between 10:28 a.m. and 2:00 p.m.

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 provided by the laboratories.

A general description of vapor sampling and sample analysis methods is given by Huckaby et al. (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 as given in Section 3.3. Inorganic analyte sorbent traps were prepared and analyzed by PNL, with analyses performed within 17 days of sample collection. PNL analyses of SUMMATM canisters for inorganic compounds were performed 59 days after sample collection (Ligotke 1995), and within the 60-day holding time requirement of the WHC quality assurance project plan (Keller 1994). All sample preparation and analyses were performed at PNL quality assurance impact level 2 (Ligotke et al. 1995a).

3.1 Ammonia, Hydrogen, and Nitrous Oxide

Based on analyses of 6 ammonia sorbent trap samples, the ammonia concentration in the headspace of tank SX-103 was determined to be 77 ppmv. This is a typical ammonia concentration for the waste tanks that have been sampled. Ammonia has been observed in virtually all of the passively ventilated waste tanks sampled to date, 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 77 ppmv corresponds to about 0.05 % of the LFL, and does not contribute appreciably to the flammability of the headspace.

The concentration of hydrogen in tank SX-103 was determined by PNL to be < 23 ppmv. Given that the LFL for hydrogen in air is about 4 vol%, the < 23 ppmv hydrogen concentration in tank SX-103 corresponds to < 0.06 % of its LFL. At this level, hydrogen is not a flammability concern in tank SX-103.

The nitrous oxide concentration in tank SX-103, measured to be < 23 ppmv in each of the 3 SUMMATM samples, is relatively low compared to other waste tanks that have been sampled. Nitrous oxide has been detected in many other passively ventilated waste tanks, and has been measured at concentrations as high as 878 ppmv in tank U-103 (Ligotke et al. 1995b). 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 SX-103 headspace.

3.2 Carbon Monoxide and Carbon Dioxide

Carbon monoxide was determined to be < 23 ppmv in the 3 SUMMATM samples from tank SX-103. 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 sampled to date, the information on carbon monoxide has varied from tank to tank. However, in the waste tank headspaces elevated carbon monoxide 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, the highest level measured to date was [26.7 ppmv] in tank C-103² (Huckaby and Story 1994).

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

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.

The carbon dioxide concentration in the tank SX-103 headspace, measured to be an average 193 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 SX-103 gas and vapor sampling event, for example, were measured to have an average 353 ppmv of carbon dioxide (Ligotke et al. 1995a).

3.3 Nitric Oxide, Nitrogen Dioxide, Water, and Tritium

Nitric oxide and nitrogen dioxide concentrations in the tank SX-103 headspace were determined to be ≤ 0.06 and ≤ 0.09 ppmv, respectively. These are acid gases that would have very low equilibrium concentrations above the high pH sludge in tank SX-103. 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 SX-103 headspace.

The water vapor concentration of tank SX-103 was determined to be about 12.6 mg/L, at the measured tank headspace temperature of 18 °C and pressure of 976.0 mbar (732.2 torr), (WHC 1995b). This equates to a water vapor partial pressure of 17.0 mbar (12.7 torr), to a dew point of 14.9 °C, and to a relative humidity of 82 %.

Silica gel sorbent traps were used to sample 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 Discussion of Inorganic Gases and Vapors

Aside from water vapor and carbon dioxide, the most abundant waste constituent in the tank SX-103 headspace is ammonia. Ammonia has been detected in all tank headspaces sampled to date. Compared to other passively ventilated waste tanks that have been sampled, tank SX-103 has low concentrations of both hydrogen and nitrous oxide.

The relative standard deviations of the inorganic gas and vapor results given in the last column in Table 3-1 are good for the methods used at the concentrations reported. Relative standard deviations range from less than 2 % for the measurement of water vapor, to about 8 % for the measurement of carbon dioxide. 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 SX-103 headspace were sampled using SUMMA™ canisters, which were analyzed at PNL, and triple sorbent traps (TSTs), which were analyzed by ORNL. Both laboratories 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).

PNL SUMMA™ sample results should be considered to be the primary organic vapor data for tank SX-103. All PNL sample preparation and analyses were performed at PNL quality assurance impact level 2. Analyses of SUMMA™ canisters for organic vapors by PNL was completed within 59 days of sample collection (Ligotke 1995). It should be noted that though this satisfied the administratively chosen 60-day holding time (Keller 1994), no holding time study has been performed to address the stability of organic compounds in SUMMA™ canisters in the likely chemical matrix of the waste tanks.

ORNL analyses of TST samples from this and other waste tanks generally agree with, support, and augment the SUMMA™ sample results. However, because analyses at ORNL were not performed under a WHC approved quality assurance plan, documentation of the quality assurance measures is less rigorous. Results of a 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 both means and standard deviations 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 from the 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 from tank C-103.

Table 4-1 lists the organic compounds positively identified and quantitated in SUMMA™ samples. Methane was analyzed for along with the inorganic gases (and not by GC/MS). Other SUMMA™ analyses were performed according to the EPA TO-14 methodology by PNL (EPA 1988, Klinger et al. 1995). Of the 53 target analytes, only trichlorofluoromethane and pyridene were positively identified and quantitatively

measured above the 0.005 ppmv quantitation limit of the analyses. Averages reported are from analyses of 3 SUMMA™ canister samples.

Jenkins et al. (1995a) report the positive identification of 24 of 27 target analytes in TST samples. 1,1-Dichloroethene, tributyl phosphate, and dibutyl butylphosphonate were the only TST target analytes not detected in the TST samples. Only 1 analyte, 1-butanol, was present at a high enough concentration to allow quantitation in all 4 TST samples. Tables 4-2 and 4-3 give the 4-TST average concentrations of the positively identified compounds. Compounds for which the ORNL practical holding time was exceeded are listed in Table 4-3. Data in Tables 4-2 and 4-3 may not be accurate to within $\pm 30\%$ as specified by Burnum (1995).

Based on a practical holding time study performed by ORNL, the reported concentrations of propanone, butanal, 1-butanol, 2-pentanone, and n-pentanenitrile may have been affected by the 49 day period between sample collection and analysis (Jenkins 1995b). These analytes are reported in Table 4-3. The 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 (Jenkins et al 1995b).

Both PNL and ORNL report target analyte concentrations in ppmv of analyte in dry air. The results given may be corrected for the measured water vapor content of tank SX-103 to obtain concentration in ppmv of analyte in moist tank air by multiplying the dry-air ppmv concentrations by 0.985.

Table 4-4 lists the 11 target analytes that were common to both TST and SUMMA™ analyses, with their reported average concentrations. Both sampling and analysis methods indicate that only trace levels of these constituents were present in the tank SX-103 headspace, and differences are not significant to the headspace flammability or industrial hygiene concerns.

The most abundant analyte in Tables 4-1 through 4-3 is 1-butanol, which was measured to be an average 0.13 ppmv in the 4 TST 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 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 SUMMA™ 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 (i.e., molecules with more than about 15 carbon atoms) and some polar compounds with moderate volatility (i.e., butanal) from SUMMA™ samples has been problematic.

The list of tentatively identified compounds recovered from SUMMA™ samples, with estimated concentrations, is given in Table 4-5. Only 2 organic compounds were tentatively identified in SUMMA™

canister samples, methanol and 1-butanol. The list of tentatively identified compounds detected in TST samples, and their estimated concentrations, is given in Table 4-6. The values given in Table 4-6 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/m^3 , based on dry air at 0°C and 1.01 bar.

The ORNL and PNL methods used to tentatively identify and estimate concentrations are described by Jenkins et al. (1995a) and Ligotke et al. (1995a), respectively, and should be reviewed before this data are used for decision making. Concentrations given in Tables 4-5 and 4-6 should be considered rough estimates. Results in Table 4-6 are presented in terms of observed chromatographic peaks, and are not adjusted for the occurrence of split peaks or the assignment of the same identity to different peaks (e.g., Cmpd # 41 and 42). In these instances, the estimated concentration of a compound appearing in more than 1 peak is simply the sum of the individual peak estimates.

4.3 Discussion of Organic Compounds

The organic compounds listed in Tables 4-1 through 4-6 may be classified as either compounds added to the waste tanks as waste that are still evaporating, or compounds that have been generated by reactions of the original waste. Because tanks SX-101 and SX-102 are ventilated through tank SX-103, the vapors identified in the tank SX-103 headspace may have originated in any of the 3 tanks.

The only organic compounds identified in the tank SX-103 headspace known to have come directly from the Hanford site chemical processes are the semivolatile straight-chain alkanes. These were used as diluents of tributyl phosphate in various plutonium extraction processes. The semivolatile straight-chain 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). Samples from tank SX-103 contain only trace levels of the more volatile NPHs.

Though tributyl phosphate was not directly detected in the tank SX-103 samples, its existence in the waste (probably at trace levels) can be inferred from the existence of the NPHs and 1-butanol. The 1-butanol detected in ORNL TST samples is a product of the hydrolysis of tributyl phosphate, and is virtually always detected when the NPHs are present.

The origin of trichlorofluoromethane is unknown, but may be associated with urethane foams used to seal pit covers to reduce fugitive emissions. This particular compound is commonly detected as headspace constituents by both TST and SUMMATM sample analyses.

The tentatively identified cyclosiloxanes (i.e., Cmpd # 22 and 28 in Table 4-6) may also be in this category. Small quantities of siloxanes may have been introduced to the waste tank through their use as process surfactants or the disposal of contaminated hydraulic fluids. They may also be present in the headspace due to their use in liquid traps at the tank's breather riser. These compounds are frequently detected at very low levels in blanks, so it is also possible these are laboratory or sampling contaminants.

The majority of volatile compounds listed in Tables 4-1 through 4-6 are thought to have been generated by chemical or radiolytic processes of the waste. The alcohols, aldehydes, ketones, nitriles, alkenes, and volatile alkanes have all been associated with the degradation of the semivolatile alkanes.

Ambient air SUMMATM samples collected at the start of the tank SX-103 sampling event indicate propanone (acetone) may have been present at trace levels in the heated VSS manifold. This contaminant was measured to be present in air that had been drawn through the manifold external to the tank headspace at

0.013 ppmv (Ligotke et al. 1995a). No adjustment of reported concentrations have been made for this observation.

5.0 SUMMARY

The tank SX-103 headspace was sampled in March 1995 for gases and vapors to address flammability and industrial hygiene concerns. Sampling and analytical results 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 1995b).

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Table 2-1
Tank SX-103 Gas and Vapor Sample Type and Number

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 SX-103 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	77	3	4
Carbon Dioxide, CO ₂	124-38-9	SUMMA™	3	193	2	8
Carbon Monoxide, CO	630-08-0	SUMMA™	3	< 23	--	--
Hydrogen, H ₂	1333-74-0	SUMMA™	3	< 23	--	--
Nitric Oxide, NO	10102-43-9	Sorbent Trap	6	≤ 0.03	--	--
Nitrogen Dioxide, NO ₂	10102-44-0	Sorbent Trap	6	≤ 0.01	--	--
Nitrous Oxide, N ₂ O	10024-97-2	SUMMA™	3	< 23	--	--
Water Vapor, H ₂ O	7732-18-5	Sorbent Trap	6	17,400 (12.7 mg/L)	300 (0.2 mg/L)	1.8

1 CAS = Chemical Abstracts Service.

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

Table 4-1
Tank SX-103 Quantitatively Measured Organic Compounds in SUMMA™ Samples --
Analyses by Pacific Northwest Laboratory

Cmpd #	Compound	CAS ¹ Number	Average ² (ppmv)	Standard Deviation ³ (ppmv)	RSD ⁴ (%)
1	Trichlorofluoromethane	75-69-4	0.011	0.001	6
2	Pyridine	110-86-1	0.019	0.018	97
3	Methane	74-82-8	< 23	—	—

1 CAS = Chemical Abstract Service.

2 Average of 3 samples.

3 When the analyte was detected in only 2 samples, the entry is the relative difference (i.e., their difference divided by 2).

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

Table 4-2
Tank SX-103 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	Ethanenitrile (acetonitrile)	75-05-8	0.0079	0.0019	23
2	Dichloromethane (methylene chloride)	75-09-2	0.00040	0.00089	200
3	Propanenitrile	107-12-0	0.00080	0.00057	73
4	n-Hexane	110-54-3	0.00090	0.00025	29
5	Benzene	71-43-2	0.00070	0.00019	28
6	n-Butanenitrile	109-74-0	0.0013	0.0004	27
7	n-Heptane	142-82-5	0.00050	0.00010	22
8	Toluene	108-88-3	0.0012	0.0003	24
9	2-Hexanone	591-78-6	0.00030	0.00008	30
10	n-Octane	111-65-9	0.00040	0.00013	35
11	n-Hexanenitrile	628-73-9	0.00010	0.00012	200
12	2-Heptanone	110-43-0	0.00020	0.00006	23
13	n-Nonane	111-84-2	0.00030	0.00015	52
14	n-Heptanenitrile	629-08-3	0.00010	0.00017	200
15	2-Octanone	111-13-7	0.00020	0.00015	74
16	n-Decane	124-18-5	0.00020	0.00037	157
17	n-Undecane	1120-21-4	0.00030	0.00033	112
18	n-Dodecane	112-40-3	0.00020	0.00015	69
19	n-Tridecane	629-50-5	0.00050	0.00036	71

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 4-3
Tank SX-103 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	Propanone (acetone) ⁵	67-64-1	0.010	0.001	14
2	Butanal ⁵	123-72-8	0.022	0.002	10
3	1-Butanol ⁶	71-36-3	0.13	0.01	5
4	2-Pentanone ⁵	107-87-9	0.00050	0.00017	36
5	n-Pentanenitrile ⁵	110-59-8	0.00010	0.00016	200

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.

6 The concentration of this analyte is quantitatively measured (as defined in Section 4.1).

Table 4-4
Tank SX-103 Comparison of Organic Compounds in TST and SUMMA™ Samples --
Analyses by Oak Ridge National Laboratory
and Pacific Northwest National Laboratory

Compound	CAS ¹ Number	TST Average ² (ppmv)	SUMMA™ Average ³ (ppmv)	PRD ⁴ (%)
1,1-Dichloroethene (vinylidene chloride)	75-35-4	< 0.00058	< 0.005	--
Dichloromethane (methylene chloride)	75-09-2	0.00040	< 0.005	--
Propanone (acetone)	67-64-1	0.010	< 0.005	> 67
Ethanenitrile (acetonitrile)	75-05-8	0.0079	< 0.005	> 45
n-Propanenitrile	107-12-0	0.00080	< 0.005	--
n-Butanenitrile	109-74-0	0.0013	< 0.005	--
Benzene	71-43-2	0.00070	< 0.005	--
Toluene	108-88-3	0.0012	< 0.005	--
n-Hexane	110-54-3	0.00090	< 0.005	--
n-Heptane	142-82-5	0.00050	< 0.005	--
n-Decane	124-18-5	0.00020	< 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.

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

Table 4-5
Tank SX-103 Tentatively Identified Organic Compounds in SUMMA™ Samples –
Analyses by Pacific Northwest Laboratory

Cmpd #	Compounds	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
1	Methanol (methyl alcohol)	67-56-1	0.18	0.04
2	1-Butanol	71-36-3	0.47	0.06

1 CAS = Chemical Abstract Service.

2 Average of 3 samples, values listed are estimates.

Table 4-6
Tank SX-103 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	Carbon dioxide	124-38-9	0.0025	0.0050
2	Carbon dioxide	124-38-9	0.030	0.059
3	Methanol and acetone		0.0096	0.0191
4	1-Propene, 2-methyl-	115-11-7	0.0041	0.0029
5	Acetaldehyde	75-07-0	0.027	0.019
6	Ethanol	64-17-5	0.024	0.012
7	Trichloromonofluoromethane	75-69-4	0.058	0.091
8	Silane, chlorotrimethyl-	75-77-4	0.0030	0.0036
9	Isopropyl Alcohol	67-63-0	0.0065	0.0047
10	1-Propanol	71-23-8	0.0050	0.0035
11	Silanol, trimethyl-	1066-40-6	0.0011	0.0021
12	Butane, 2-methyl-	78-78-4	0.00088	0.00176
13	Furan, tetrahydro-	109-99-9	0.0051	0.0039
14	Pyrazine	290-37-9	0.017	0.001
15	N-Nitrosodimethylamine	62-75-9	0.0037	0.0026
16	Cyclohexene, 1-ethyl-6-ethylidene	61141-57-9	0.00059	0.00118
17	Propane, 2-methyl-2-nitro-	594-70-7	0.0020	0.0039
18	Benzeneacetic acid, alpha, -hydroxy	20698-91-3	0.0020	0.0039
19	Phenol, 4-propyl-	645-56-7	0.00078	0.00155
20	Benzothiazole, 2-phenyl-	883-93-2	0.00067	0.00134
21	2,5-Furandione, dihydro-	108-30-5	0.0044	0.0069
22	Cyclotrisiloxane, hexamethyl-	541-05-9	0.032	0.031
23	p-Xylene	106-42-3	0.0016	0.0011
24	Propane, 2-[(1,1-dimethylethyl) sulfonyl]-2-methyl	1886-75-5	0.0035	0.0029
25	Butyrolactone and others		0.00066	0.00132
26	Alkenol		0.0010	0.0020

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Cmpd #	Compounds	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
27	3-Heptanone	106-35-4	0.00061	0.00122
28	Cyclotetrasiloxane, octamethyl-	556-67-2	0.014	0.011
29	1-Hexanol, 2-ethyl-	104-76-7	0.0032	0.0022
30	Nonanal	124-19-6	0.0031	0.0016
31	Benzoic acid, 2-[(tri-methylsilyl) oxy]-, trimethylsilyl ester	3789-85-3	0.0017	0.0034
32	p-Trimethylsilyloxyphenyl-bis(trimethyl-silyloxy)ethane		0.00086	0.00173
33	Decanal	112-31-2	0.00062	0.00123
34	Isopropyl Myristate	110-27-0	0.0020	0.0039
35	C6-Benzenesulfamide		0.00095	0.00190
36	Alkane		0.0070	0.0089
37	Heptadecane	629-78-7	0.00069	0.00138
38	Benzenesulfonamide, n-butyl-	3622-84-2	0.039	0.027
39	1-Heptadecene	6765-39-5	0.0020	0.0039
40	Alkane		0.0013	0.0026
41	Isopropyl Palmitate	142-91-6	0.015	0.025
42	Isopropyl Palmitate	142-91-6	0.0061	0.0122
43	Alkane		0.0016	0.0033
Sum of tentatively identified compounds:			0.35	

1 CAS = Chemical Abstract Service.

2 Based on analyses of 3 samples, values listed are estimates.