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7. Abstract

Tank 241-C-104 headspace gas and vapor samples were collected and analyzed to help determine the potential risks to tank farm workers due to fugitive emissions from the tank. 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 241-C-104 was vapor sampled in accordance with "Data Quality Objectives for Generic In-Tank Health and Safety Issue Resolution (Osborne et al., 1994).

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Tank 241-C-104 Vapor Sampling and Analysis Tank Characterization Report

X.0 INTRODUCTION

Tank C-104 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 C-104 was vapor sampled in accordance with *Data Quality Objectives for Generic In-Tank Health and Safety Issue Resolution* (Osborne et al. 1994). Results presented here represent the best available data on the headspace constituents of tank C-104.

Gas and vapor concentrations in tank C-104 are thought to be strongly affected by active ventilation of the headspace. Though no exhauster is used directly on tank C-104, it is connected via an underground cascade line to tank C-105, which is actively ventilated. Monthly waste tank summary reports list tank C-104 as an actively vented tank (Hanlon 1995). Though it has not been proven, it is assumed that the cascade line connection to tank C-105 is open and tank C-104 is actively ventilated.

The active ventilation of tank C-104 has 2 potential effects on the gas and vapor data presented below. First, most passively ventilated waste tanks exchange gases and vapors with the atmosphere so slowly that the headspace vapors are virtually in equilibrium with waste liquids. Active ventilation of tank C-104, however, may preclude vapor-liquid equilibrium and complicate the interpretation of vapor data. And second, diffusion and convection are thought to make the headspaces of passively ventilated tanks homogeneous, and representative samples can be drawn from any central region of the headspace. Though mixing is generally more vigorous in an actively ventilated tank headspace (other factors being equal), the discrete sources of fresh air and the single outlet would produce concentration gradients within the headspace. This would make gas and vapor concentrations a function of location, and limit the value of samples collected from a single arbitrary location, as are those discussed below.

The degree to which active ventilation affects the gas and vapor data depends most directly on 1) the ventilation rate relative to the headspace volume, and 2) the locations of air in-leakage points relative to the cascade line. Unfortunately, neither the ventilation rate nor all of its in-leakage points are known.

X.1 SAMPLING EVENT

Headspace gas and vapor samples were collected from tank C-104 using the vapor sampling system (VSS) on March 3, 1994 by WHC Sampling & Mobile Laboratories

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(WHC 1995). Sample collection and analysis were performed as directed by the sample and analysis plan (WHC 1995, Appendix A). The tank headspace temperature was determined to be 28 °C. Air from the C-104 headspace was withdrawn via a 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. Sampling media were prepared and analyzed by WHC, Oak Ridge National Laboratories (ORNL), Pacific Northwest Laboratories (PNL), and Oregon Graduate Institute of Science and Technology (OGIST) through a contract with Sandia National Laboratories.

Tank C-104 was the fourth tank to be sampled using the VSS, and was sampled only about 5 weeks after the first use of the VSS on a tank. Because the methods and equipment were relatively new, problems with sample handling (e.g., chain-of-custody and shipping) were encountered (WHC 1995, Lucke et al. 1995a). The precision of the sampling results, however, is generally good. Sorbent trap collection problems noted in a subsequent sampling event[1] do not appear to have occurred when tank C-104 was sampled.

The 46 tank air samples and 6 ambient air control samples collected are listed in Table X-1 by analytical laboratory. Table X-1 also lists the 8 trip blanks and 3 spiked blanks provided by the laboratories. A general description of vapor sampling and sample analysis methods is given by Huckaby (1994). 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 1995 and references therein.

X.2 INORGANIC GASES AND VAPORS

Analytical results of sorbent trap and SUMMA™,[2] canister tank air samples for selected inorganic gases and vapors are given in Table X-2 in parts per million by volume (ppmv). Inorganic analyte sorbent traps were prepared and analyzed by PNL. SUMMA™ canisters were analyzed for inorganic analytes by OGIST. Reports by PNL (Lucke et al. 1995a) and OGIST (Rasmussen 1994a) describe sample preparation and analyses.

The relative standard deviations of the inorganic gas and vapor results are given in the last column in Table X-2. Except for the measurement of sulfur oxides, the precision of reported results is good. Relative standard deviations range from about 4 % for nitrous oxide and water vapor results, to 50 % for sulfur oxides results. The precision reported depends both on sampling parameters (e.g., sample flow rate and flow time for sorbent traps)

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[1] Because of a valve-sequencing error, sample air volumes through specific sampling ports were not properly measured during sample job 7A of tank C-103. The error resulted in obvious and significant differences in sorbent trap results, but had no effect on SUMMA™ samples.

[2] SUMMA is a trademark of Molectrics, Inc., Cleveland, Ohio.

and analytical parameters (e.g., sample preparation, dilutions, etc.), and the small relative standard deviations suggest adequate controls were maintained both in the field and in the laboratories.

X.2.1 Ammonia, Hydrogen, and Nitrous Oxide

Ammonia, hydrogen, and nitrous oxide are commonly detected in the waste tanks. Thought to be products of radiolysis and oxidation-reduction reactions of the waste, they appear ubiquitously with the high-level waste. These headspace constituents have been the most abundant waste artifacts in virtually every tank headspace sampled to date.

The reported ammonia concentration, 44 ppmv, is above the National Institute of Occupational Safety and Health (NIOSH) 8-hr recommended exposure limit (REL) of 25 ppmv (NIOSH 1995). Ammonia has been observed in virtually all of the waste tanks sampled to date, at concentrations ranging from about 3 ppmv in tank C-108 (Lucke et al. 1995b), to 1040 ppmv in BY-108 (McVeety et al. 1995). Typically the ammonia concentration of a waste tank can be correlated to its organic vapor concentration. As is discussed below, the organic vapor concentration is tank C-104 is relatively high, and the ammonia concentration might be expected to be higher than measured. Active exhausting of the tank C-104 headspace probably accounts for the relatively low ammonia concentration in tank C-104.

The concentration of hydrogen in tank C-104 was determined to be 68 ppmv. Hydrogen in the waste tanks is of concern as a fuel. Given that the lower flammability limit (LFL) for hydrogen in air is about 4 % by volume, the 68 ppmv hydrogen concentration in tank C-104 corresponds to about 0.2 % of its LFL, and indicates hydrogen is not a flammability concern in tank C-104.

The nitrous oxide concentration in tank C-104, 64 ppmv, is above the NIOSH 8-hr REL of 25 ppmv (NIOSH 1995). Nitrous oxide, also known as laughing gas, has been detected in other waste tanks at concentrations as low as about 12 ppmv in tank TX-105 (Klinger 1995), and as high as about 800 ppmv in tank C-103 (Huckaby and Story 1994).

Neither hydrogen nor nitrous oxide is highly soluble in the aqueous wastes of the tanks, so except for situations where bubbles are trapped within the waste and released episodically, the gases are released as they are generated. The concentration of these gases in the tank headspaces is consequently determined by a simple balance between their overall generation rate and the rate at which they are vented to the atmosphere. If the tank C-104 headspace is actively exhausted by its connection to tank C-105 (as is thought), the measured concentrations of hydrogen and nitrous oxide may actually equate to relatively high gas generation rates, despite the fact that the gas concentrations are lower than in many passively ventilated waste tanks.

X.2.2 Carbon Monoxide, Nitric Oxide, Nitrogen Dioxide, Water and Tritium

Carbon monoxide in the tank C-104 headspace, measured to be 2 ppmv in SUMMA™ samples (Rasmussen 1994a), is above its concentration in ambient air, where it typically ranges from 0.05 to 0.15 ppmv. Ambient air samples collected at the beginning of the tank C-104 vapor sampling event were measured to have < 1 ppmv. Elevated waste tank headspace carbon monoxide concentrations are common (e.g., carbon monoxide concentration in tank C-103 was 26.7 ppmv, Huckaby and Story 1994), and are thought to be due to the decomposition of organic waste in the tanks. The 2 ppmv of carbon monoxide in tank C-104 is much less than the NIOSH 8-hr REL of 35 ppmv.

Nitric oxide and nitrogen dioxide concentrations in the tank C-104 headspace were determined to be 0.37 ppmv and ≤ 0.02 ppmv, respectively. These are both acid gases that would have very low equilibrium concentrations above the high pH sludge in tank C-104. The measurable presence of nitric oxide may be due to its formation from oxygen and nitrogen in the radiation field of the headspace. The NIOSH 8-hr REL is 25 ppmv for nitric oxide, and the 15-minute short term exposure limit (STEL) for nitrogen dioxide is 1 ppmv.

The water vapor concentration of tank C-104 was determined to be about 12.9 mg/L, at the tank headspace temperature of 28 °C and pressure of 1001 mbar (750.5 torr), (WHC 1995). This corresponds to a water vapor partial pressure of 17.8 mbar (13.4 torr), to a dew point of 15.7 °C, and to a relative humidity of 47 %. The water vapor content of the tank C-104 headspace is significantly lower than that measured in other sludge-containing 241-C farm tanks, which more typically have relative humidities of about 85 %. This may be an indication that the tank is indeed actively exhausted via its cascade line to tank C-105.

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. Analysis of the silica gel, which would have trapped approximately 44 mg of water vapor, indicated the total activity of the sample to be below the method detection limit of 50 pCi (WHC 1995).

X.3 ORGANIC VAPORS

Organic vapors in the tank C-104 headspace were sampled using SUMMA™ canisters, which were analyzed by OGIST and PNL, Occupational Safety and Health Administration (OSHA) versatile samplers (OVSs), which were analyzed by PNL, and triple sorbent traps (TSTs), which were analyzed by ORNL. PNL, OGIST, and ORNL used gas chromatographs (GCs) equipped with mass spectrometer (MS) detectors to separate, identify, and quantitate organic vapors. A quantitative measurement of the total organic vapor concentration by the U.S.

Environmental Protection Agency (EPA) task order 12 (TO-12) method was also performed by OGIST (EPA 1988, Rasmussen 1994a). Descriptions of sample device cleaning, sample preparations, and analyses are given by Jenkins et al. (1994), Rasmussen (1994a), and Lucke et al. (1995a).

SUMMA^{TM} sample results should be considered to be the primary organic vapor data for tank C-104. ORNL analyses of TST samples from this and other waste tanks generally agree with, support, and augment the SUMMA^{TM} 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.

The TST samples from tank C-104 were inadvertently stored at room temperature for several days. Mistakenly sent to PNL, the sealed TSTs were allowed remain in an ice-cooled shipping container over a weekend, during which time the ice melted and the samples reached room temperature. Though there was no apparent loss of analytes or contamination, ORNL had specified that the samples be stored and shipped at 4 °C.

X.3.1 Positively Identified Organic Analytes

Table X-3 lists the organic compounds positively identified and quantitated in SUMMA^{TM} and OVS samples by PNL. SUMMA^{TM} analyses were performed according to the EPA TO-14 methodology (EPA 1988, Lucke et al. 1995a). Only 3 of the 40 TO-14 target analytes were observed to be above the 0.002 ppmv quantitation limit of the analyses (Lucke et al. 1995a provide the complete TO-14 analyte list). Averages reported are from analyses of 3 SUMMA^{TM} canister samples.

OVSs were used to sample the tank air for the following semivolatile normal paraffinic hydrocarbons (NPHs): n-Undecane; n-dodecane; n-tridecane; n-tetradecane; and n-pentadecane. Unlike TST samples or SUMMA^{TM} canisters, the analytical preparation of OVSs involves liquid extraction, and larger sample volumes are required for comparable detection limits. The OVS results given in Table X-3 are from a single sample because the NPHs were below detection limits in the other 2 OVSs. It is not clear why the NPH analytes were not detected in the other 2 OVSs; whether errors were made in sample collection or analysis is not clear. Two of the NPH target analytes, n-undecane and n-pentadecane, were below detection limits in all 3 OVSs.

Table X-4 lists the organic compounds positively identified and quantitated in SUMMA^{TM} samples by OGIST. SUMMA^{TM} analyses were performed according to the EPA TO-14 methodology (EPA 1988, Rasmussen 1994a). Averages reported are from analyses of 3 SUMMA^{TM} samples. The OGIST and PNL results are in fair agreement, the most significant difference being that OGIST reported an average of 0.036 ppmv of trichlorofluoromethane, while PNL reported an average of 0.0089 for this analyte.

Jenkins et al. (1994) report the positive identification of 24 of 26 target analytes in TST samples. The target analyte list was based on advice from a panel of toxicology experts (Goheen 1993). The average concentrations of 22 target analytes, from the analysis of 3 TSTs, are given in Table X-5. Two target analytes, dichloromethane and 1,1-dichloroethene, were below detection limits. Two other target analytes, tributyl phosphate (TBP) and dibutylbutyl phosphonate (DBBP), were positively identified, but not quantitatively measured. TBP was estimated to be present at 0.025 ppmv (0.3 mg/m^3) and DBBP was estimated to be present at 0.006 ppmv (0.07 mg/m^3).

Dichloromethane, benzene, and toluene were common to both the TST and SUMMA^{TM} analyses. Dichloromethane was not detected in either TST or PNL SUMMA^{TM} samples, but 0.0025 ppmv was reported in OGIST SUMMA^{TM} samples. The reported average concentrations of benzene, 0.0099 ppmv, and toluene, 0.082 ppmv, in TST samples are higher than in either the PNL or OGIST SUMMA^{TM} samples. The average 0.082 ppmv toluene concentration found in TST samples is well below the 100 ppmv NIOSH 8-hr REL for toluene. The average 0.082 ppmv benzene concentration found in TST samples, however, is about the same as the 0.1 ppmv NIOSH 8-hr REL for benzene.

The most abundant analytes in Tables X-3 through X-5 are 1-butanol, n-tridecane, and n-dodecane. At the reported concentrations, the target analytes do not individually or collectively represent a flammability hazard.

X.3.2 Tentatively Identified Organic Analytes

In addition to targeted analytes, the OGIST, ORNL, and PNL analytical procedures allow the tentative identification of other organic vapors. By the nature of the samples and their analysis, virtually all 3- to 15-carbon organic compounds present in the tank headspace above analytical detection limits are observable. The PNL list of tentatively identified compounds, with estimated concentrations, is given in Table X-6, and the ORNL list of tentatively identified compounds, and their estimated concentrations, is given in Table X-7. Estimated concentrations are in mg/m^3, based on dry air at 0 °C and 1.01 bar.

OGIST provided total-ion chromatograms for each of the tank C-104 SUMMA^{TM} samples and assigned tentative identities to the significant peaks, but did not estimate their concentrations. Table X-8 lists the analytes tentatively identified by OGIST, and designates which analytes were detected in each of the 3 SUMMA^{TM} samples. As a rough guide, the analytes listed in Table X-8 were probably detected at greater than 0.001 ppmv. It should also be noted that some peaks in the total-ion chromatograms were not labeled, despite being relatively high, because confidence in assigning a chemical identity was very low.

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. Entries in Tables X-6 and X-7, particularly near the bottom of the tables where the analytes have higher molecular weights, illustrate this.

The ORNL and PNL methods used to tentatively identify and estimate concentrations are described by Jenkins et al. (1994) and Lucke et al. (1995a), respectively, and should be reviewed before this data is used for decision making. Results in Tables X-6 and X-7 are presented in terms of observed peaks, and are not adjusted for the occurrence of split chromatographic peaks (e.g., Cmpd # 1, 2, and 3 in Table X-7) or the assignment of the same identity to different peaks (e.g., Cmpd # 68 and 87 in Table X-7). In these instances, the estimated concentration of a compound appearing as a doublet or triplet is simply the sum of the individual peak estimates.

Concentrations given in Tables X-6 and X-7 should be considered rough estimates. The proper quantitation of all observed analytes is outside the scope and budget of these analyses, and the estimation of concentrations involves several important assumptions. The validity of each assumption depends on the analyte, and such factors as the specific configuration of the analytical instrumentation.

X.3.3 Total Nonmethane Organic Compounds

OGIST measured the total nonmethane organic compound (TNMOC) concentration in 3 SUMMA^{TM} canister samples using the EPA TO-12 method (Rasmussen 1994a). The sample mean was 28.1 mg/m^3, with a standard deviation of 0.7 mg/m^3. Though data on other tanks is very limited, this value is higher than average for the waste tanks sampled to date. It is particularly high if indeed the headspace of tank C-104 is actively ventilated.

A summation of concentrations of target and tentatively identified compounds in TST samples by GC/MS indicated the total organic vapor concentration to be about 25 mg/m^3, in excellent agreement with the EPA TO-12 method results. TO-12 method TNMOC measurements of other waste tanks have ranged from as high as 5,000 mg/m^3 in tank C-103 (Rasmussen and Einfeld 1994), to as low as 0.18 mg/m^3 in tank C-111 (Rasmussen 1994b), while the TNMOC concentration of clean ambient air ranges from about 0.03 to 0.1 mg/m^3.

X.3.4 Discussion of Organic Analytes

The mixture of organic vapors in a waste tank provides a signature of the volatile and semivolatile organic wastes stored in that tank. The organic vapor signature of tank C-104 is similar in many ways to that of other 241-C farm tanks, particularly tank C-103. A convenient way to consider the organic compounds listed in Tables X-3 through X-8 is to separate them into 2 categories: 1) organic compounds added to tank C-104 as waste that are still evaporating, and 2) organic compounds that have been generated by reactions of the original waste.

The first category encompasses all organic compounds that were placed into the tank as waste. It includes TBP, DBBP, and the semivolatile branched alkanes and NPHs, all of which ostensibly came from the Plutonium-Uranium Extraction (PUREX) process. It also includes the volatile halogenated compounds, such as chloromethane, dichlorodifluoromethane, and trichlorofluoromethane, which may have been used as solvents and were sent to the waste tanks when contaminated.

Tank C-104 is believed to have contained about a 7.5 cm (3 in.) floating layer of TBP and NPH diluent in the early 1970s. Hall (1972) reported the tank contained about 30,000 L (8,000 gal) of "TBP-NPH", and provides results from a radiochemical analysis of organic liquid samples apparently collected in January 1972. Anderson (1990) reports that various types and amounts of wastes were pumped into and out of the tank between 1972 and 1976, when tank C-104 became inactive. The pumpable supernatant liquids remaining in tank C-104 were removed in 1989, when the tank was interim stabilized (Boyles 1992).

While the amount of organic liquid that could have been removed during the various waste transfers from tank C-104 is uncertain, it is unlikely that it was all removed. The organic vapor signature of tank C-104 supports this premise. Though the semivolatile NPH concentrations reported in Tables X-3 and X-5 are lower than would be expected if the vapor phase were in equilibrium with a PUREX process solution of NPH and TBP, there are at least 2 reasons for this. First, preferential evaporation of the NPHs from an NPH-TBP solution would, during 20-odd years of storage, reduce the partial pressure of the NPHs. And second, as discussed above, if the headspace of tank C-104 is indeed actively ventilated, it is unlikely that vapor-liquid equilibrium would be maintained.

One type of semivolatile compound commonly detected in the NPH-rich waste tanks, but not in tank C-104, is a group of semivolatile bicyclic alkanes. Decahydronaphthalene and several methyl-substituted decahydronaphthalenes have been detected in several of the 241-BY and 241-C farm tanks, notably tank C-103. Table X-7 does identify several such compounds (e.g., Cmpd # 59 and 78), but their concentrations relative to other semivolatiles are much lower than in other NPH-rich tanks. This may be an indication that the organic waste in tank C-104 originated during a period when the PUREX process was using a relatively pure NPH diluent.

The second category includes all organic compounds that were not placed into the tank as waste, but instead have been generated via radiolytic and chemical reactions of the waste. The majority of compounds listed in Tables X-3 through X-8 fall into this category, including the alcohols, aldehydes, ketones, nitriles, alkenes, alkyl nitrates, and volatile alkanes, all of which have been associated with the degradation of the NPHs. By far the most abundant of these in the headspace are the short-chain volatile compounds, however, some long-chain low volatility compounds detected may also be waste reaction products.

Samples from tank C-104 were found to have several alkyl nitrates. Though it is reasonable to expect alkyl nitrates to be produced via chemical and radiolytic processes of the NPH with other waste, the solubility of alkyl nitrates in the aqueous waste supernates would also be expected to significantly reduce their vapor-phase concentrations. Indeed, their presence in other NPH-rich tanks has been minimal. That several alkyl nitrates are at detectible levels in tank C-104 may indicate dry conditions where they are formed.

The tank C-104 samples were tentatively determined to contain the following homologous series of straight-chain compounds:

1) nitriles from ethanenitrile (acetonitrile) through nonanenitrile;

2) alkyl nitrates from methyl to hexyl nitrate;

3) alkanes from propane through n-nonane[3];

4) 2-ketones from propanone (acetone) through 2-decanone;

5) aldehydes from ethanal (acetaldehyde) through decanal;

6) 1-alkanols from ethanol through 1-hexanol;

7) 1-alkenes from propene through 1-nonene;

as well as several partially complete series of other functionally related organic compounds. As has been noted in other NPH-rich waste tanks, many of the volatile species (presumed to be degradation products of the NPHs) have functional groups on the molecule's first or second carbon atom.

1-Butanol is prominent amongst the alcohols detected. It is known to be a degradation product of TBP, and has been found in virtually all of the waste tanks sampled to date.

In summary, there is evidence that concentrations of compounds in the tank C-104 headspace are strongly affected by the use of an exhauster on tank C-105. The constituents of the tank C-104 are typical of most NPH-rich tank headspaces, except that the alkyl nitrate signature is stronger than typical.

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[3] n-Decane through n-pentadecane were also detected, however, these are principally constituents of the original waste and not waste reaction products.

Table X-1
Tank C-104 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	0.5 and 10	Organic vapors	10 tank air samples, + 1 trip blank
Oregon Graduate Institute of Science and Technology	SUMMA™ canister	6.0	Hydrogen, Nitrous Oxide, Carbon Monoxide, Organic vapors	3 tank air samples, 6 ambient air samples
Pacific Northwest Laboratories	Acidified Carbon Sorbent Trap	3.0	Ammonia	6 tank air samples + 1 trip blank
	Triethanolamine Sorbent Trap	3.0	Nitrogen Dioxide	6 tank air samples + 1 trip blank
	Oxidation Bed + Triethanolamine Sorbent Trap	3.0	Nitric Oxide	6 tank air samples + 1 trip blank
	Treated Carbon Bead Sorbent Trap	3.0	Oxides of Sulfur	3 tank air samples
	Silica Gel Sorbent Trap	3.0	Water vapor	5 tank air samples + 1 trip blanks
	OSHA Versatile Sampler	3.0	Semivolatile organic vapors	3 tank air samples + 3 trip blanks + 3 spiked blanks
	SUMMA™ canister	6.0	Organic vapors	3 tank air samples
WHC 222-S Laboratory	Silica Gel Sorbent Trap	3.0	Tritium-Substituted Water Vapor	1 tank air sample

Table X-2
Tank C-104 Inorganic Gas and Vapor Concentrations

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	44	< 1	--
Carbon Monoxide, CO	630-08-0	SUMMA™	3	2	< 1	--
Hydrogen, H_2	1333-74-0	SUMMA™	3	68	8.0	12
Nitric Oxide, NO	10102-43-9	Sorbent Trap	6	0.37	0.05	14
Nitrogen Dioxide, NO_2	10102-44-0	Sorbent Trap	6	≤ 0.02	--	--
Nitrous Oxide, N_2O	10024-97-2	SUMMA™	3	64	2.6	4
Oxides of Sulfur, SO_X		Sorbent Trap	3	0.4	0.2	50
Water Vapor, H_2O	7732-18-5	Sorbent Trap	5	17,800 (12.9 mg/L)	750 (0.5 mg/L)	4

1. CAS = Chemical Abstracts Service.

2. RSD = relative standard deviation.

Table X-3
Tank C-104 Positively Identified Compounds in PNL SUMMA™ and OVS Samples

Cmpd #	Compound	CAS[1] Number	Average (ppmv)	Standard Deviation (ppmv)	RSD[2] (%)
1	Dichlorodifluoromethane[3]	75-71-8	0.0016	0.00004	2
2	Chloromethane[3] (methyl chloride)	74-87-3	0.0024	0.0002	8
3	Trichlorofluoromethane[3]	75-69-4	0.0089	0.0022	25
4	Methylbenzene (toluene)[4]	108-88-3	0.0064	--	--
5	n-Undecane[5]	1120-21-4	< 0.14	--	--
6	n-Dodecane[6]	112-40-3	0.39	--	--
7	n-Tridecane[6]	629-50-5	0.52	--	--
8	n-Tetradecane[6]	629-59-4	0.11	--	--
9	n-Pentadecane[5]	629-62-9	< 0.11	--	--

1. CAS = Chemical Abstract Service.

2. RSD = relative standard deviation.

3. Results from 3 SUMMA™ samples.

4. Detected in only 1 SUMMA™ sample.

5. Result of 3 OSHA versatile sampler (OVS) samples.

6. Result of 1 OSHA versatile sampler (OVS) sample.

Table X-4
Tank C-104 Positively Identified Organic Compounds in OGIST SUMMA™ Samples

Cmpd #	Compound	CAS[1] Number	Average (ppmv)	Standard Deviation (ppmv)	RSD[2] (%)
1	Dichlorodifluoromethane	75-71-8	0.0008	0.0003	43
2	Chloromethane	74-87-3	0.0028	0.0011	39
3	1,3-Butadiene	106-99-0	0.008	0.001	17
4	Trichlorofluoromethane	75-69-4	0.036	0.009	25
5	1,1-Dichloroethene (Vinylidene chloride)	75-35-4	0.0011	0.00038	35
6	Dichloromethane (methylene chloride)	75-09-2	0.0025	0.0022	94
7	1,1,2-Trichloro-1,2,2-trifluoroethane[3]	76-13-1	0.0003	--	--
8	Trichloromethane (chloroform)[4]	67-66-3	0.00045	--	--
9	Benzene	71-43-2	0.0048	0.0016	33
10	Toluene	108-88-3	0.0024	0.0009	36
11	Ethyl benzene	100-41-4	0.00033	0.00015	46
12 & 13	m-Xylene & p-Xylene[5]	108-38-3 106-42-3	0.0008	0.0003	38
14	o-Xylene	95-47-6	0.00033	0.00088	35

1. CAS = Chemical Abstracts Service.

2. RSD = relative standard deviation.

3. Detected in only 1 sample.

4. Detected in only 2 samples.

5. These analytes coelute, so the given concentration is their sum.

Table X-5
Tank C-104 Positively Identified Organic Compounds in TST Samples

Cmpd #	Compound	CAS[1] Number	Average (ppmv)	Standard Deviation (ppmv)	RSD[2] (%)
1	Ethanenitrile (acetonitrile)	75-05-8	0.13	0.08	61
2	Propanone (acetone)	67-64-1	0.15	0.07	44
3	Propanenitrile	107-12-0	0.041	0.039	94
4	n-Hexane	110-54-3	0.038	0.001	3
5	Benzene	71-43-2	0.0099	0.0034	34
6	1-Butanol	71-36-3	2.90	0.32	11
7	Butanenitrile	109-74-0	0.044	0.002	5
8	2-Pentanone	107-87-9	0.012	0.001	8
9	n-Heptane	142-82-5	0.015	0.001	9
10	Toluene	108-88-3	0.082	0.001	14
11	Pentanenitrile	110-59-8	0.032	0.006	17
12	2-Hexanone	591-78-6	0.015	0.005	31
13	n-Octane	111-65-9	0.013	0.003	22
14	Hexanenitrile	628-73-9	0.028	0.006	20
15	2-Heptanone	110-43-0	0.014	0.002	15
16	n-Nonane	111-84-2	0.017	0.003	17
17	Heptanenitrile	629-08-3	0.021	0.004	19
18	2-Octanone	111-13-7	0.0075	0.0014	19
19	Octanenitrile	124-12-9	0.012	0.003	22
20	Nonanenitrile	2243-27-8	0.0072	0.0013	18
21	n-Dodecane	112-70-3	0.072	0.003	4
22	n-Tridecane	629-50-5	0.079	0.009	11
Sum of positively identified compounds:			12.7 mg/m^3		

1. CAS = Chemical Abstract Service.

2. RSD = relative standard deviation.

Table X-6
Tank C-104 Tentatively Identified Compounds in PNL SUMMA™ Samples

Cmpd #	Compound	CAS[1] Number	Average (mg/m^3)	Standard Deviation (mg/m^3)
1	Propene	115-07-1	0.46	0.03
2	Propane	74-98-6	0.33	0.03
3	Cyclopropane[2]	75-19-4	0.12	--
4	Ethanal (acetaldehyde)	75-07-0	0.26	0.11
5	2-Methyl-1-propene	115-11-7	0.29	0.03
6	n-Butane	106-97-8	0.59	0.06
7	Ethanenitrile (acetonitrile)	75-05-8	0.12	0.01
8	Propanone (acetone)	67-64-1	0.32	0.02
9	Pentane	109-66-0	0.26	0.02
10	Butanal	123-72-8	0.47	0.03
11	2-Butanone	78-93-3	0.32	0.01
12	1-Hexene	592-41-6	0.13	0.01
13	Tetrahydrofuran	109-99-9	0.15	0.01
14	1-Butanol	71-36-3	2.45	0.29
15	Pentanal	110-62-3	0.14	0.02
16	n-Heptane	142-82-5	0.31	0.04
17	n-Octane	111-65-9	0.21	0.01
18	3-Heptanone	106-35-4	0.41	0.05
19	n-Nonane[3]	111-84-2	0.10	--
20	n-Decane	124-18-5	0.95	0.06
21	n-Undecane	1120-21-4	4.88	0.23
22	n-Dodecane	112-40-3	6.47	0.14
23	Unknown C11 Ketone[3]		0.10	--
24	Unknown Alkane		0.11	0.01
25	n-Tridecane	629-50-5	6.22	0.13
26	n-Tetradecane	629-59-4	0.96	0.08

Sum of tentatively identified compounds:	27.13

1. CAS = Chemical Abstract Service.

2. Detected in only 2 samples.

3. Detected in only 1 sample.

Table X-7
Tank C-104 Tentatively Identified Organic Compounds in TST Samples

Cmpd #	Compound	CAS[1] Number	Average (mg/m^3)	Standard Deviation (mg/m^3)
1	Carbon Dioxide	124-38-9	0.170	0.0554
2	Carbon Dioxide	124-38-9	1.703	0.8112
3	Carbon Dioxide[2]	124-38-9	0.580	---
4	Propene	115-07-1	0.764	0.1356
5	Propene	115-07-1	0.100	0.0460
6	Cyclopropane[2]	115-07-1	0.024	---
7	1-Propene, 2-methyl-	115-11-7	0.876	0.0604
8	n-Butane	106-97-8	1.061	0.0705
9	2-Butene (trans)[2]		0.042	---
10	Methylcyclopropane		0.115	0.0138
11	2-Butene (cis)[2]		0.038	---
12	1-Pentene	109-67-1	0.412	0.0592
13	n-Butane[2]	106-97-8	0.046	---
14	n-Pentane	109-66-0	1.098	0.1476
15	Unknown[2]		0.056	---
16	Fluoroethylene[2]		0.052	---
17	1-Hexene	592-41-6	0.273	0.0497
18	3-Buten-2-one[2]	78-94-4	0.037	---
19	Butanal[2]	123-72-8	0.140	---
20	2-Butanone	789-93-3	0.282	0.0198
21	C6-Alkene[2]		0.030	---
22	Acetic acid, ethyl ester[2]	141-78-6	0.158	---
23	Acetic Acid[2]	64-19-7	0.074	---
24	Nitric acid, ethyl ester[2]	625-58-1	0.052	---
25	n-Pentane and others[2]		0.338	---
26	Tetrahydrofuran[2]	109-99-9	0.210	---
27	1-Butanol[2]		0.029	---

Cmpd #	Compound	CAS[1] Number	Average (mg/m^3)	Standard Deviation (mg/m^3)
28	Acetic Acid	64-19-7	0.624	0.2237
29	1-Octene	111-66-0	0.043	0.0121
30	Hexanal[2]	66-25-1	0.049	---
31	Acetic acid, butyl ester	123-86-4	0.106	0.0334
32	Nitric acid, butyl ester	928-45-0	0.069	0.0578
33	Pyridine, 2-methyl[2]	109-06-8	0.020	---
34	1-Hexanol	111-27-3	0.009	0.0020
35	3-Heptanone	106-35-4	0.182	0.0590
36	Pyridine, 3-methyl and 4-methyl[2]		0.013	---
37	Pyridine, 2,6-dimethyl[2]	108-48-5	0.004	---
38	1-Octene, 7-methyl	13151-06-9	0.014	0.0032
39	2 (3H)-Furanone, dihydro-	96-48-0	0.090	0.0298
40	C8-Alkanone		0.033	0.0090
41	Pyridine, 2,5-dimethyl-	589-93-5	0.089	0.0452
42	Alkyl amide		0.054	0.0250
43	C9-Alkane		0.019	0.0052
44	Cyclotetrasiloxane, octamethyl	556-67-2	0.018	0.0203
45	n-Decane	124-18-5	0.183	0.0531
46	Hexanenitrile and others[2]		0.015	---
47	C3-Pyridine and others[2]		0.002	---
48	1-Hexanol, 2-ethyl-	104-76-7	0.022	0.0084
49	Pyridine, 5-ethyl-2-methyl[2]	104-90-5	0.005	---
50	Alkyl amide		0.024	0.0106
51	n-Undecane	1120-21-4	0.902	1.1760
52	Alkane[2]		0.009	---
53	C13-Alkane		0.051	0.0048
54	Alkane[2]		0.004	---

Cmpd #	Compound	CAS[1] Number	Average (mg/m^3)	Standard Deviation (mg/m^3)
55	3-Undecanone[2]	2216-87-7	0.005	---
56	Alkane[2]		0.002	---
57	Undecane, 5-ethyl-[2]	17453-94-0	0.008	---
58	Tridecane, 6-methyl-	13287-21-3	0.010	0.0072
59	Alkane and Alkyl-decahydronaphthalene[2]		0.002	---
60	C14-Alkene[2]		0.004	---
61	Cyclohexane, hexyl-[2]	4292-75-5	0.006	---
62	Mixture[2]		0.004	---
63	Cyclohexane, undecyl- and others[2]		0.006	---
64	C14-Alkanone		0.238	0.0251
65	C15-Alkane		0.007	0.0012
66	Alkane		0.015	0.0017
67	Alkane + Others		0.020	0.0017
68	n-Hexadecane	544-76-3	0.090	0.0062
69	Alkane		0.011	0.0014
70	C14-Alkane		0.216	0.0287
71	2(3H)-Furanone, dihydro-5-propyl[2]	105-21-5	0.002	---
72	2(3H)-Furanone, 5-hexyldihydro-[2]	706-14-9	0.002	---
73	C14-Alkanone		0.131	0.0150
74	C14-Alkane		0.035	0.0010
75	Thiophene, 2-methoxy-5-methyl	31053-55-1	0.023	0.0002
76	Alkane + Others		0.029	0.0016
77	Phenol, 2,6-bis(1,1-dimethylethyl)-4-methyl		0.010	0.0106
78	C4-Dihydronaphthalene[2]		0.003	---

Cmpd #	Compound	CAS[1] Number	Average (mg/m^3)	Standard Deviation (mg/m^3)
79	Alkane[2]		0.002	---
80	Undecane, 5-methyl[2]	1632-70-8	0.002	---
81	C16-Alkane		0.006	0.0009
82	Pyrazine, 2,5-dimethyl 3-(3-methylbutyl)-[2]	18433-98-2	0.003	---
83	Alkanol		0.004	0.0001
84	Alkane		0.011	0.0014
85	C15-Alkane		0.106	0.0029
86	Mixture[2]		0.004	---
87	n-Hexadecane	544-76-3	0.061	0.0048
88	3-Dodecanone[2]	1534-27-6	0.031	---
89	C-14-Alkanone		0.055	0.0114
90	C-14-Alkanone		0.043	0.0108
91	Alkanol[2]		0.001	---
92	Dibutyl butyl phosphate	78-46-6	0.066	0.0236
93	C14-Alkanone[2]		0.008	---
94	2-Pentene, 5-(pentyloxy)-, (E)-[2]	56052-85-8	0.008	---
95	Dodecane, 4,9-dipropyl[2]	3054-63-5	0.002	---
96	Benzene, (1-butylheptyl)-[2]	4537-15-9	0.002	---
97	Tributyl phosphate (TBP)	126-73-8	0.272	0.0573
98	Mixture[2]		0.002	---
99	(+)-(1R,4R,5R)-2-(10)-Pinen-4-ol[2]		0.002	---
100	Alkanol[2]		0.008	---
101	Undecane, 3-methyl[2]	1002-43-3	0.004	---
102	2-Decanone, 5,9-dimethyl[2]	33933-82-3	0.008	---
103	5-Tetradecene, (E)-[2]	41446-66-6	0.012	---
104	Mixture[2]		0.005	---

Cmpd #	Compound	CAS[1] Number	Average (mg/m^3)	Standard Deviation (mg/m^3)
105	Methylamine, N-(1-methylhexylidene)[2]	22058-71-5	0.004	---
106	Butyl myristate (Butyl tetradecylate)[2]		0.007	---
107	1-Dodecanol[2]	112-53-8	0.003	---
108	n-Heptadecane	629-78-7	0.019	0.0022
109	Alkyl dihydrofuranone[2]		0.006	---
110	Oxirane, 2-methyl-2-pentyl-[2]	53907-75-8	0.005	---
111	Tributyl phosphate[2]	126-73-8	0.008	---
112	Mixture[2]		0.004	---
113	Benzene, (1-methyldecyl)-	4536-88-3	0.004	0.0011
114	Undecane, 3-methyl[2]	1002-43-3	0.002	---
115	Benzene, (1-pentylheptyl)-[2]	2719-62-2	0.002	---
116	Nonane, 5-butyl-[2]	17312-63-9	0.001	---
117	Benzene, (1-propylnonyl)-[2]	2719-64-4	0.002	---
118	Nonane, 5-propyl-[2]	998-35-6	0.001	---
119	Alkane		0.005	0.0007
120	Butyl myristate (butyl tetradecylate)		0.009	0.0073
121	n-Octadecane	593-45-3	0.005	0.0039
122	2(3H)-Furanone, dihydro-5-propyl-[2]	105-21-5	0.001	---
123	Isopropyl myristate[2]		0.002	---
124	Benzene, (1-methylundecyl)-[2]		0.002	---
125	Butyl myristate (butyl tetradecylate)[2]		0.004	---
126	Alkane[2]		0.002	---
127	1,2-Benzenedicarboxylic acid, dibutyl ester[2]		0.002	---

Cmpd #	Compound	CAS[1] Number	Average (mg/m^3)	Standard Deviation (mg/m^3)
128	1,2-Benzenedicarboxylic acid, butyl-ethylhexyl ester[2]	85-69-8	0.003	---
129	Alkane[2]		0.002	---
Sum of tentatively identified compounds:			13.13	

1. CAS = Chemical Abstract Service

2. Detected in only 1 sample.

Table X-8
Tank C-104 Tentatively Identified Compounds in OGIST SUMMA™ Samples

Cmpd #	Compound	Sample Number[1]			CAS[2] Number
		1	2	3	
1	Propene	x	x	x	115-07-1
2	Propane	x	x	x	74-98-6
3	Propadiene	x	x		463-49-0
4	Propyne		x	x	74-99-7
5	Cyclopropane	x	x	x	75-19-4
6	2-Methylpropane	x	x	x	75-28-5
7	Ethanal (acetaldehyde)	x	x	x	75-07-0
8	1-Butene	x	x	x	106-98-9
9	n-Butane	x	x	x	106-97-8
10	trans-2-Butene		x		624-64-6
11	Cyclobutane	x	x	x	287-23-0
12	cis-2-Butene		x		590-18-1
13	3-Methyl-1-butene	x	x		563-45-1
14	Ethanol	x	x	x	64-17-5
15	Ethanenitrile (acetonitrile)	x	x	x	75-05-8
16	Propanone (acetone)	x	x	x	67-64-1
17	Propanal (propionaldehyde)	x	x	x	123-38-6
18	1-Pentene	x	x	x	109-67-1
19	2-Propanol	x	x	x	67-63-0
20	n-Pentane	x	x	x	109-66-0
21	Methyl nitrate	x	x	x	598-58-3
22	Carbon disulfide	x	x	x	75-15-0
23	Propanenitrile	x	x	x	107-12-0
24	1-Propanol	x	x	x	71-23-8
25	3-Buten-2-one (methylvinyl ketone)	x	x	x	78-94-4
26	2-Methylpentane	x	x	x	107-83-5
27	Butanal	x	x	x	123-72-8

Cmpd #	Compound	Sample Number[1]			CAS[2] Number
		1	2	3	
28	2-Butanone	x	x	x	78-93-3
29	1-Hexene	x	x	x	592-41-6
30	2-Butanol	x	x	x	78-92-2
31	n-Hexane	x	x	x	110-54-3
32	Ethyl nitrate	x	x	x	625-58-1
33	Tetrahydrofuran	x	x	x	109-99-9
34	2-Butenal (crotonaldehyde)		x		4170-30-3
35	Butanenitrile	x	x	x	109-74-0
36	1-Butanol	x	x	x	71-36-3
37	2-Pentanone	x	x	x	107-87-9
38	Pentanal	x	x	x	110-62-3
39	1-Heptene	x	x	x	592-76-7
40	Propyl nitrate	x		x	627-13-4
41	n-Heptane	x	x	x	142-82-5
42	Pentanenitrile	x	x	x	110-59-8
43	1-Pentanol			x	71-41-0
44	3-Hexanone		x	x	589-38-8
45	2-Hexanone	x	x	x	591-78-6
46	Hexanal	x	x	x	66-25-1
47	1-Octene	x	x	x	111-66-0
48	n-Octane	x	x	x	111-65-9
49	Butyl nitrate	x	x	x	928-45-0
50	Hexanenitrile	x	x	x	628-73-9
51	1-Hexanol	x	x	x	111-27-3
52	4-Heptanone	x	x	x	123-19-3
53	3-Heptanone	x	x	x	106-35-4
54	2-Heptanone	x	x	x	110-43-0
55	Heptanal	x	x	x	111-71-7

Cmpd #	Compound	Sample Number[1]			CAS[2] Number
		1	2	3	
56	1-Nonene	x	x	x	124-11-8
57	n-Nonane	x	x	x	111-84-2
58	Pentyl nitrate	x	x	x	1002-16-0
59	Heptanenitrile	x	x	x	629-08-3
60	2-Octanone	x	x	x	111-13-7
61	Octanal	x	x	x	124-13-0
62	n-Decane	x	x	x	124-18-5
63	Hexyl nitrate	x	x	x	20633-11-8
64	2-Ethyl-1-hexanol		x		104-76-7
65	Octanenitrile	x	x	x	124-12-9
66	2-Nonanone	x	x	x	821-55-6
67	Nonanal	x	x	x	124-19-6
68	1-Undecene	x	x	x	821-95-4
69	n-Undecane	x	x	x	1120-21-4
70	2-Decanone	x	x	x	693-54-9
71	Decanal		x		112-31-2
72	1-Dodecene	x		x	112-41-4
73	n-Dodecane	x	x	x	112-40-3
74	2,6-Dimethylundecane	x	x	x	17301-23-4
75	n-Tridecane	x	x	x	629-50-5
76	n-Tetradecane	x	x	x	629-59-4

1. An x is placed in the column if the analyte was tentatively identified in the sample. Here 1 = sample S4004-01.C03; 2 = sample S4004-02.C03; and 3 = sample S4004-03.C03 (Rasmussen 1994a, WHC 1995).

2. CAS = Chemical Abstract Service.

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