

Pacific Northwest National Laboratory

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TANK VAPOR CHARACTERIZATION PROJECT

Tank 241-U-104 Headspace Gas and Vapor Characterization Results from Samples Collected on July 16, 1996

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August 1997

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Prepared for Lockheed Martin Hanford
under a Related Services Agreement
with the U.S. Department of Energy
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TANK VAPOR CHARACTERIZATION PROJECT

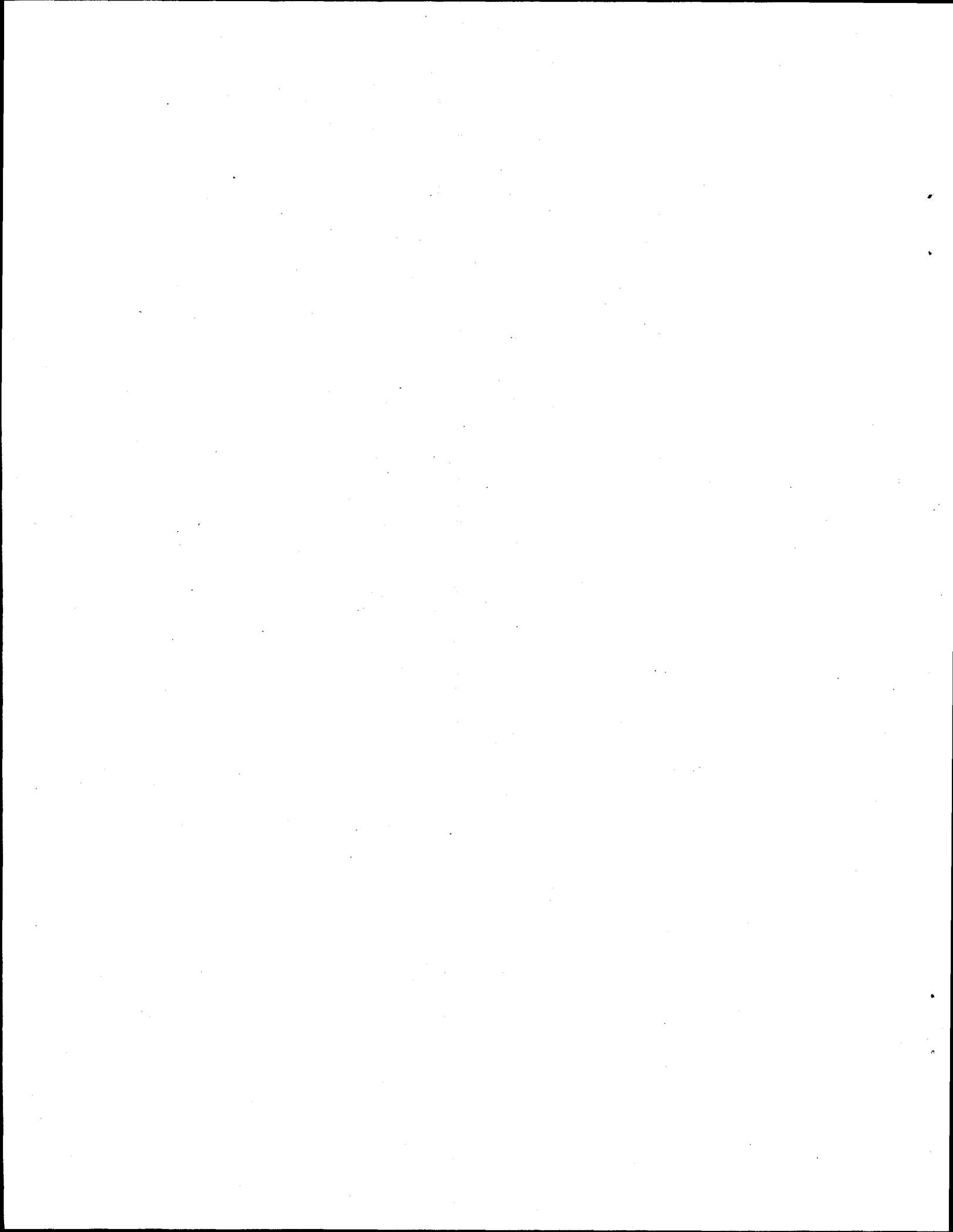
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Richland, Washington 99352



Summary

This report presents the results from analyses of samples taken from the headspace of waste storage tank 241-U-104 (Tank U-104) at the Hanford Site in Washington State. Tank headspace samples collected by Westinghouse Hanford Company (WHC) were analyzed by Pacific Northwest National Laboratory (PNNL) to determine headspace concentrations of selected non-radioactive analytes. Analyses were performed by the Vapor Analytical Laboratory (VAL) at PNNL. Vapor concentrations from sorbent trap samples are based on measured sample volumes provided by WHC.

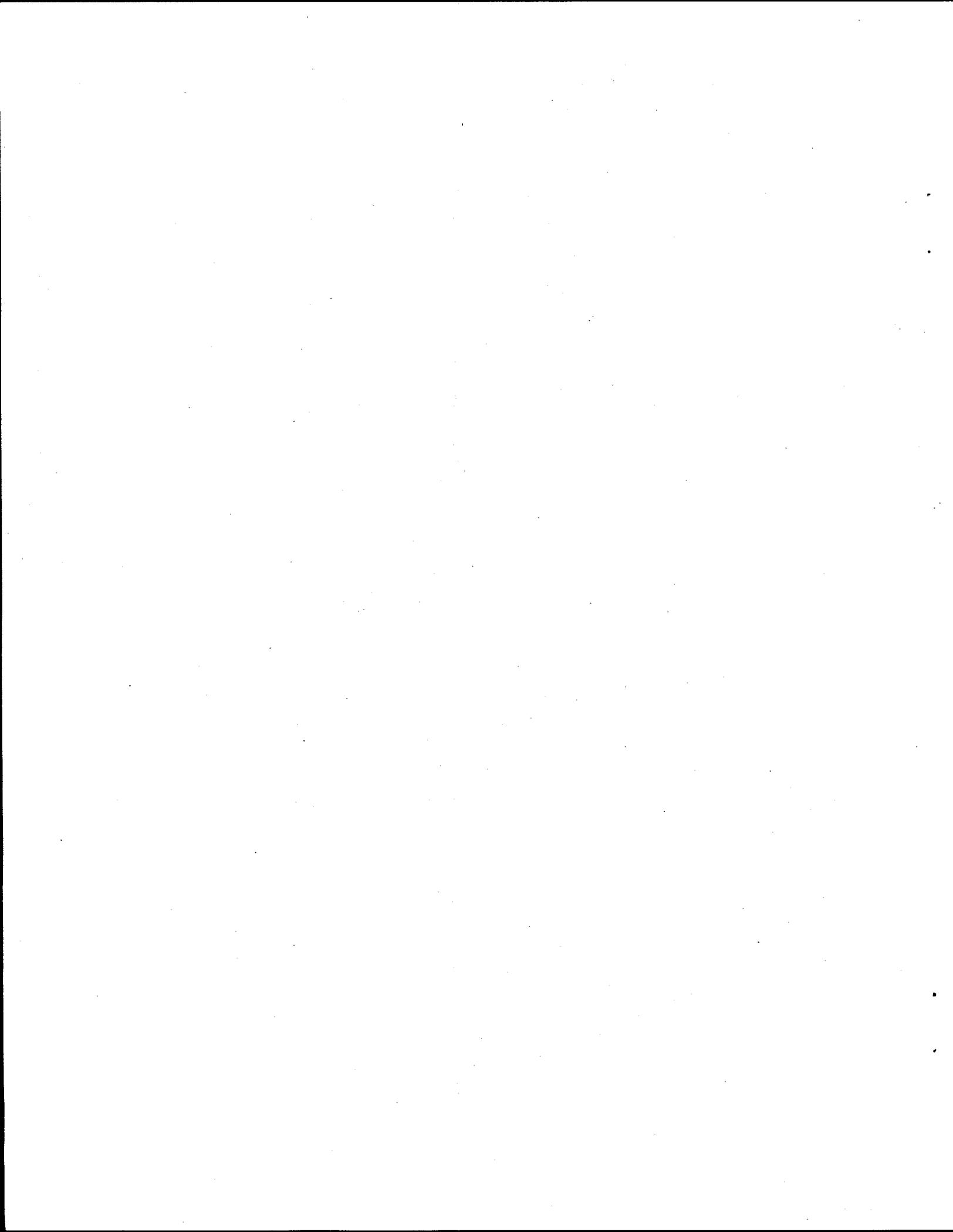
No analytes were determined to be above the immediate notification limits specified by the sampling and analysis plan (SAP) (Buckley 1996). None of the flammable constituents were present at concentrations above the analytical instrument detection limits. Total headspace flammability was estimated to be <0.108% of the lower flammability limit (LFL).

Average measured concentrations of targeted gases, inorganic vapors, and selected organic vapors are provided in Table S.1. A summary of experimental methods, including sampling methodology, analytical procedures, and quality assurance and control methods are presented in Section 2.0. Detailed descriptions of the analytical results are provided in Section 3.0.

Table S.1. Average Measured Concentrations of Gases and Inorganic and Organic Vapors in Tank U-104 Sampled on 7/16/96

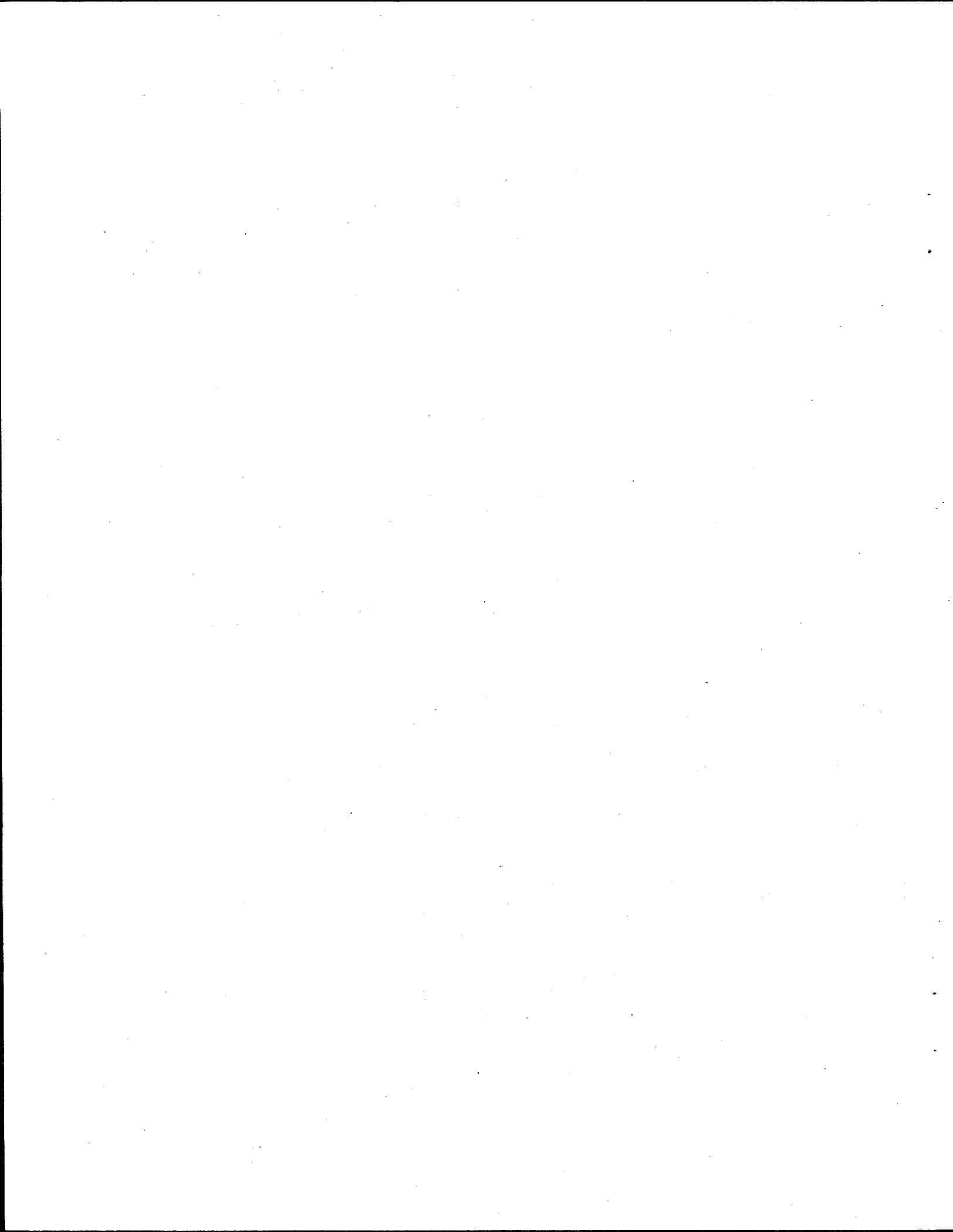
| <u>Category</u> | <u>Sample Medium</u> | <u>Analyte</u> | <u>Concentration^(a)</u> | <u>Units</u> |
|-------------------------------------|------------------------------------|-------------------------------------|------------------------------------|-------------------|
| Inorganic Vapors | Sorbent Traps | Ammonia | <0.71 | ppmv |
| | | Nitric Oxide (NO) | <0.16 | ppmv |
| | | Nitrogen Dioxide (NO ₂) | <0.16 | ppmv |
| | | Water | 17.0 | mg/L |
| Permanent Gases | SUMMA™ Canisters | Hydrogen | <17 | ppmv |
| | | Methane | <25 | ppmv |
| | | Carbon Dioxide | 652 | ppmv |
| | | Carbon Monoxide | <17 | ppmv |
| | | Nitrous Oxide (N ₂ O) | 86 | ppmv |
| Total Non-Methane Organic Compounds | SUMMA™ Canisters | Total Non-Methane Organic Compounds | <0.59 | mg/m ³ |
| Flammables | SUMMA™ Canisters and Sorbent Traps | Flammables | <0.108 | % LFL |

(a) Concentrations were determined using sample-volume data provided by Westinghouse Hanford Company and are based on averaged data from three samples. Mass concentrations are at reference temperature and pressure of 0°C and 1.013 bar.



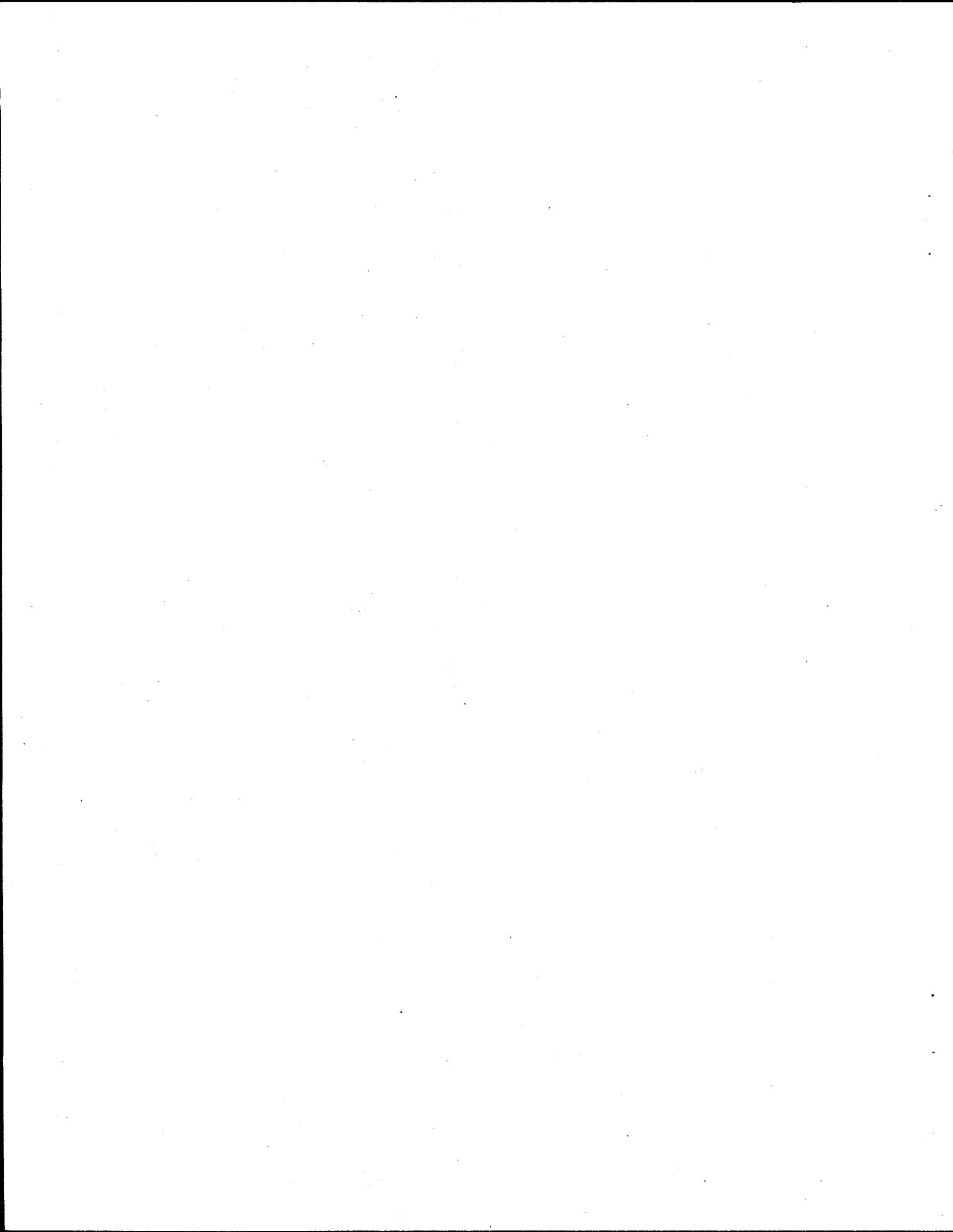
Acknowledgments

The authors gratefully acknowledge the support of other project staff at PNNL who contributed to the successful completion of this sampling and analysis activity. S. O. Slate, K. P. Schielke, L. M. P. Thomas, and G. W. Dennis supported inorganic laboratory work.



Terms and Abbreviations

| | |
|--------|---|
| % D | % Difference |
| CCV | continuing calibration verification |
| COC | chain-of-custody |
| DIW | deionized water |
| EPA | U.S. Environmental Protection Agency |
| EQL | estimated quantitation limit |
| GC/FID | gas chromatography/flame ionization detector |
| GC/TCD | gas chromatography/thermal conductivity detection |
| ICV | initial calibration verification |
| ISE | ion selective electrode |
| ISVS | In Situ Vapor Sampling System |
| LFL | lower flammability limit |
| NIST | National Institute for Standards and Technology |
| OSHA | Occupational Safety and Health Administration |
| PNL | Pacific Northwest Laboratory (previous name for the laboratory) |
| PNNL | Pacific Northwest National Laboratory |
| ppbv | part per billion by volume |
| ppm | parts per million |
| ppmv | part per million by volume |
| QA | quality assurance |
| RPD | relative percent difference |
| SAP | sample and analysis plan |
| SCIC | suppressed-conductivity ion chromatography |
| SRM | standard reference material |
| STP | standard temperature and pressure |
| SUMMA™ | process for passivating stainless steel |
| TEA | triethanolamine |
| TNMOC | total non-methane organic compound |
| UHP | ultra high purity |
| VAL | Vapor Analytical Laboratory |
| WHC | Westinghouse Hanford Company |



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1.0 Introduction

This report presents results of chemical analyses of vapor samples collected by WHC on July 16, 1996 from the headspace of waste storage tank 241-U-104 (Tank U-104) at the Hanford Site in Washington State. Pacific Northwest National Laboratory^(a) provided SUMMA™ canisters and sorbent traps for sample collection, and analyzed the samples according to instructions in the SAP (Buckley 1996). Analytical work was performed by the PNNL VAL in the 300 Area of the Hanford Site under the PNNL Tank Vapor Characterization Project.

Pacific Northwest National Laboratory provided six sets of sorbent traps for selected inorganic analytes (four samples and two field blanks) and five SUMMA™ canisters for permanent gases and organic analytes (three headspace samples and two ambient air samples). Sample devices and controls were provided to WHC on July 12, 1996 and were returned to PNNL on October 8, 1996. Westinghouse Hanford Company measured and reported to PNNL the sample volumes needed to determine headspace concentrations from sorbent trap samples.

Specific analytical methods for sample analysis are described in Section 2.0. Results and known sampling and analytical variances from established quality assurance (QA) requirements, where significant, are documented in Section 3.0. Chain-of-custody forms used to document possession and transfer of samples and controls are provided in Appendix A.

^(a) Pacific Northwest National Laboratory is operated for the U. S. Department of Energy by Battelle under Contract DE-AC06-76RLO 1830. The previous name of the laboratory was Pacific Northwest Laboratory. The former name is used when previously published documents are referenced.

2.0 Analytical Methods

Table 2.1 summarizes the analytes, sampling media, analytical methods, and laboratory procedures. Table 2.2 summarizes information regarding sample media, handling, and storage procedures.

Table 2.1. Sampling and Analysis Methods Summary

| <u>Analyte</u> | <u>Sampling Media</u> | <u>Extraction Method</u> | <u>Analysis Method</u> | <u>Procedure</u> |
|---|---|--|---|------------------|
| Ammonia | Acidified carbon bead sorbent trap | Aqueous extraction | ISE analysis | PNL-ALO-226 |
| Nitric Oxide and Nitrogen Dioxide | Triethanolamine impregnated sorbent traps | Aqueous extraction | IC | PNL-ALO-212 |
| Water Vapor | Other inorganic sorbent traps + silica gel sorbent trap | None | Gravimetric analysis (sample weight gain) | PNL-TVP-09 |
| Carbon Monoxide, Carbon Dioxide, Hydrogen, Methane, and Nitrous Oxide | SUMMA™ canisters | Analysis of SUMMA™ canister subsample | GC/TCD | PNL-TVP-05 |
| Total Non-Methane Organic Compounds | SUMMA™ canisters | Cryo-focusing of SUMMA™ canister subsample | GC/FID | PNL-TVP-08 |

Table 2.2. Sample Media Preparation, Handling, and Storage

| <u>Sampling Media</u> | <u>Supplier and Catalog Number</u> | <u>Preparation Procedure</u> | <u>Handling and Storage Procedure</u> |
|-------------------------------|--|------------------------------|---------------------------------------|
| Inorganic vapor sorbent traps | SKC No. 226-29 SKC No. 226-40-02 SKC No. 226-10-04 | PNL-TVP-09 | PNL-TVP-07 |
| SUMMA™ canisters | Scientific Instrumentation Specialist | PNL-TVP-02 | PNL-TVP-07 |

2.1 Inorganic Vapors

Solid sorbent traps, prepared in multi-trap sampling trains, were supplied to WHC for sampling the tank headspace. Blanks and exposed samples were returned to PNNL for analyses. Analyses were performed to provide information on the tank-headspace concentration of the following analytes: ammonia, nitrogen dioxide (NO₂), nitric oxide (NO), and water. Samples were prepared, handled, and disassembled as described in Technical Procedure PNL-TVP-09^(a).

2.1.1 Sampling Methodology

Standard glass tubes containing sorbent materials to trap ammonia, NO, NO₂, and water vapors were obtained, prepared, and submitted for vapor sampling. The sorbent traps were selected based on their use by the Occupational Safety and Health Administration (OSHA) to perform workplace monitoring and because of available procedures and verification results associated with that particular application. Each sorbent trap contained two sorbent sections separated by a glass wool plug. Sorbent media in the two sections were segregated and analyzed separately (except for analysis for water). Analyses of the second sorbent (breakthrough) sections were performed to demonstrate complete collection of the target analyte by the first sorbent section.

The ammonia sorbent traps contained carbon beads impregnated with sulfuric acid; nominally, 500 mg were contained in the primary and 250 mg in the breakthrough sections. The ammonia was chemisorbed as ammonium sulfate [(NH₄)₂SO₄]. The NO₂ traps contained a zeolite impregnated with triethanolamine (TEA), with 400 mg in the primary and 200 mg in the breakthrough sections. The NO₂ was absorbed and disproportionated to equi-molar quantities of nitrite ions (NO₂⁻) and nitrate ions (NO₃⁻). Glass tubes containing 800 mg of an oxidant were used to convert NO to NO₂. The converted NO was then collected as nitrite and nitrate in an NO₂ trap. The water traps contained a total of 450 mg of silica gel. All sorbent traps for a given analyte were from a single manufacturer's batch.

After sample preparation, sorbent trains were stored at ≤ 10°C because of handling recommendations for the oxidizer tubes attached to some samples. After receipt of exposed and radiologically cleared samples from WHC and disassembly of the sorbent trains, samples were provided to the analytical laboratory at ambient temperature.

The sorbent traps were prepared in multi-trap trains configured so sample air flow passed in order through the ammonia, nitrogen dioxide, oxidizer, nitric oxide, and desiccant traps. Traps were weighed and then connected to each other using uniform lengths of 3/8-in. perfluoroalkoxy-grade (PFA) Teflon® tubing. The perfluoroalkoxy-grade tubing was heated in hot air and forced over the open ends of the traps to form a tight seal. The inlets of the sorbent trains each consist of a short section of tubing having a 3/8-in. stainless steel Swagelok® nut, and sealed using a Swagelok® cap. The trailing ends of the sorbent trains were each sealed with red plastic caps provided by the manufacturer.

^(a) Pacific Northwest Laboratory. 12/95. *Sorbent Trap Preparation for Sampling and Analysis: Waste Tank Inorganic Vapor Samples*, PNL-TVP-09 (Rev. 2), PNL Technical Procedure, Richland, Washington.

2.1.1.1 Concentration Calculations. Concentration, in parts per million by volume (ppmv), was determined by dividing the amount of analyte, in μmol , by the moles of the dried tank air sampled. For example, the concentration of a 3.00-L sample containing 75.0 μg of ammonia equals

$$\frac{(75.0 \mu\text{g})(22.4 \text{ L/mol})}{(17.0 \mu\text{g}/\mu\text{mol})(3.00 \text{ L})} = 32.9 \text{ ppmv} \quad (2.1)$$

Measured sample volumes were specified by WHC at standard temperature and pressure (STP; 0°C , 1.013 bar). Because water vapor is removed as an analyte before the sample air stream passes through the mass flow meters, sample volumes exclude water vapor.

2.1.2 Analytical Procedures

2.1.2.1 Ammonia Analysis. The sorbent material from the ammonia traps was placed into labeled 20-mL glass scintillation vials. Vials containing front-section sorbent material were treated with 10.0 mL of deionized water (DIW), and vials containing breakthrough-section sorbent material were treated with 5.0 mL of DIW. Ammonia present was measured using the ion selective electrode (ISE) procedure PNL-ALO-226^(a). Briefly, the method includes 1) preparing a 1000- $\mu\text{g}/\text{mL}$ ammonia stock standard solution from dried reagent-grade NH_4Cl and DIW, 2) preparing 0.1-, 0.5-, 1.0-, 10-, and 100- $\mu\text{g}/\text{mL}$ ammonia working calibration standards by serial dilution of the freshly made stock standard, 3) generating an initial calibration curve from the measured electromotive force signal versus ammonia concentration data obtained for the set of working standards, 4) performing a calibration-verification check, using a mid-range dilution of a certified National Institute for Standards and Technology (NIST)-traceable 0.1 M NH_4Cl standard from an independent source, at a minimum of once per batch, 5) continuing this sequence until all samples of the batch have been measured, including duplicates and spiked samples, and 6) remeasuring the complete set of calibration standards (at the end of the session). Electromotive force signal measurements obtained for samples are compared to those for standards to determine ammonia concentration in the samples.

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

Primary and breakthrough section materials were analyzed separately using identical procedures. Each analytical session was conducted as follows. Working nitrite standards were prepared by diluting a stock nitrite standard with desorbing solution. An initial calibration curve was

(a) Procedure entitled "Ammonia (Nitrogen) in Aqueous Samples," PNL-ALO-226 Rev. 0, in the *Analytical Chemistry Laboratory (ACL) Procedure Compendium*, Vol. 3: Inorganic Instrumental Methods. Pacific Northwest National Laboratory, Richland, Washington.

(b) Procedure entitled "Determination of Inorganic Anions by Ion Chromatography," PNL-ALO-212, in the *Analytical Chemistry Laboratory (ACL) Procedure Compendium*, Vol. 3: Inorganic Instrumental Methods. Pacific Northwest National Laboratory, Richland, Washington.

prepared from the instrument response versus nitrite standard concentration data for the set of working standards. A calibration verification check using one of the midrange standards was performed at a minimum of once per batch. If the instrument response indicated that sample nitrite concentration was outside the calibration range, the sample was diluted with desorbing solution and reanalyzed. After all samples of a batch were analyzed, the complete set of calibration standards was remeasured to verify consistent instrument response. Instrument responses observed for samples were compared to those for standards to determine the nitrite concentration of the samples. Because the analytes were collected on the sorbent as equal quantities of nitrite and nitrate, and the analysis was specific for nitrite, the moles of NO₂ and NO were determined by doubling the analytically determined moles of nitrite.

2.1.2.3 Water Analysis. All Sorbent traps used to make each multi-trap train were weighed using a semi-micro mass balance, after labeling and breaking the glass tube ends, without plastic end caps. After receipt of exposed samples, the sorbent traps were again weighed to determine the change in mass. Mass gain was assumed to be entirely due to collection of water vapor. Field blanks were used to correct results.

2.1.3 Quality Assurance/Quality Control

Analytical work was performed according to quality levels identified in PNL-ALO-212, PNL-ALO-226, and Quality Assurance Plan ETD-002. Estimated quantitation limits (EQLs) for the inorganic vapors are given in Table 2.3.

The accuracy of concentration measurements depends on errors associated with both sampling and analysis. Ammonia results were estimated to be within 5% of their true values. The uncertainty includes preparation of standards, purity of the ammonium salt used to prepare standards, potential operator bias, ambient temperature variations, etc. Working standards are traceable to NIST standard reference material (SRM) by using an independent calibration verification standard certified to be NIST-traceable. Nitrite analyses (for NO₂ and NO) are performed using certified but not NIST-traceable SRM; this is because NIST does not make a nitrite SRM. Based on experience in comparing

Table 2.3 Quantitation Limits for Selected Inorganic Analytes

| <u>Analyte</u> | <u>Formula</u> | <u>EQL^(a)</u> <u>(ppmv)</u> |
|-----------------------------|-----------------|---|
| Ammonia | NH ₃ | 0.71 |
| Nitrogen Dioxide | NO ₂ | 0.16 |
| Nitric oxide | NO | 0.16 |
| Mass (water) ^(b) | n/a | 0.3 mg/L |

(a) The lowest calibration standard is defined as the EQL.

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

n/a = not applicable.

nitrite working standards prepared from several different sources and factors mentioned for ammonia above, the estimated maximum bias for NO₂ results is ± 10%, and for NO results it is ± 5%. The accuracy of measurements of sample mass is typically ± 0.1 mg, or less than 1% of the mass changes of most samples. The analytical accuracy of measurements of the change in mass of sorbent trains, based on the variability in mass change of field blank multi-trap trains, is determined for each sample job and is typically about ± 1 mg per five-trap sorbent train.

2.2 Permanent Gases

SUMMA™ canister samples were analyzed for carbon dioxide, carbon monoxide, hydrogen, methane, and nitrous oxide (N₂O).

2.2.1 SUMMA™ Canister Preparation

All SUMMA™ canisters are cleaned and verified contaminant-free according to PNNL Technical Procedure PNL-TVP-02^(a) before use. The cleaning procedure uses an EnTech 3000 cleaning system that alternately fills the canisters with purified humid air and evacuates them for several cycles while the canister is heated. If the canister is verified as clean, the canister is evacuated to 5 mtorr and tagged. Before sending the canisters to the field for sampling, the canister vacuum is measured to determine if any leakage has occurred. If the vacuum has remained constant during storage, the canisters are prehumidified with 100 µL of distilled water and labeled. Canisters stored more than 30 but less than 60 days are re-evacuated and rehumidified before use. If stored more than 60 days, the canisters are recleaned and validated before use. All canisters are stored at room temperature.

2.2.2 Analytical Procedure

The SUMMA™ canister samples were analyzed for five permanent gases by gas chromatography/thermal conductivity detection (GC/TCD). Aliquots of sampled air (undiluted) are drawn directly from each SUMMA™ canister into a 5-mL gas-tight syringe and injected into a Hewlett-Packard 5890 GC/TCD fitted with a loop injector valve and a column switching valve. An aliquot of 5 mL is used so that the 1.0-mL injection loop is completely purged with sample air, ensuring that no dilution of the sample takes place within the injection loop. One set of GC conditions is used to analyze for carbon monoxide, carbon dioxide, N₂O, and methane using helium as the carrier gas. A second GC/TCD analysis is performed for hydrogen using nitrogen as the carrier gas to enhance the signal sensitivity and lower the detection limit for this analyte. The permanent gases and the derived EQLs are listed in Table 2.4.

Table 2.4 Quantitation Limits for Permanent Gases

| <u>Analyte</u> | <u>Formula</u> | <u>EQL (ppmv)</u> |
|-----------------|------------------|-------------------|
| Carbon Dioxide | CO ₂ | 17 |
| Carbon Monoxide | CO | 17 |
| Methane | CH ₄ | 25 |
| Hydrogen | H ₂ | 17 |
| Nitrous Oxide | N ₂ O | 17 |

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

2.2.3 Quality Assurance/Quality Control

Standards for the permanent gas analyses were blended from commercially prepared and certified standards. The instrument was calibrated at five different concentrations for methane over a range of 25 to 2100 ppmv, calibrated for carbon monoxide, carbon dioxide, and N₂O over a range of 17 to 2100 ppmv, and calibrated for hydrogen over a range of 17 to 2120 ppmv. An average response factor from the calibration was used for quantification.

Each analyte was quantitated by comparison of sample analyte peak area to the calibration plot generated for the compound. The EQL for the method has been established as the low level calibration standard. Before and after each sample analysis set, a gas standard was run to evaluate system performance and to measure system accuracy. The calculated concentration of the individual gases in the standards fell within $\pm 25\%$ of the expected concentrations.

2.3 Total Non-Methane Organic Compounds

2.3.1 Analytical Procedure

SUMMA™ canister samples were analyzed for total non-methane organic compounds (TNMOCs) according to PNNL Technical Procedure PNL-TVP-08^(a), which is similar to U.S. Environmental Protection Agency (EPA) Task Order 12 (TO-12). Twenty-four hours before analysis, SUMMA™ canister samples are pressurized with purified air (Aadco Instruments, Inc., 1920 Sherwood St., Clearwater, Florida 34625). The original pressure is first measured using a calibrated diaphragm gauge (Cole Parmer), then pressurized to a level exactly twice the original pressure.

The method uses an EnTech 7000 cryoconcentration system interfaced with a Hewlett-Packard 5890 gas chromatograph/flame ionization detector (GC/FID). The EnTech concentrator is used to pull a metered volume of 50 to 100 mL of sample air from the SUMMA™ canister mounted on an EnTech 7016CA 16-canister autosampler. The sample is cryogenically concentrated, and constituents are trapped in a stainless steel tube containing glass beads and Tenax. The glass bead/Tenax trap is heated to 180°C and purged with ultra high purity (UHP) helium into the GC/FID. The GC oven is programmed to run at a 150°C isothermal temperature. Chromatographic separation is not needed in this method since quantitation is from the entire FID response over the run time.

Concentration in mg/m³ was derived from the 10-point multilevel calibration curve from the propane standard using the following equation:

$$\text{mg/m}^3 = \frac{(\text{ng TNMOC}) \times (\text{dilution factor})}{\text{mL sampled volume}} \quad (2.2)$$

2.3.2 Quality Assurance/Quality Control

^(a) Pacific Northwest Laboratory. 12/95. *Determination of TO-12 Total Nonmethane Organic Compounds in Hanford Waste Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Flame Ionization Detection*, PNL-TVP-08 (Rev. 1), PNL Technical Procedure, Richland, Washington.

The TNMOC is calibrated by using propane as the calibration standard. The instrument calibration mixture for the TNMOC analysis consists of NIST 99.999% propane analyzed using a 10-point, multilevel, linear regression curve.

Immediately before running the analysis sequence, a leak-check procedure, which includes evacuating the transfer lines and monitoring the pressure, must be performed on the sample manifold tower. The control limits on this test require that the change in pressure is <1.5 psi, and the absolute pressure after evacuation is <3 psi for each manifold position specified in the sequence table. If either criterion is not met, it must be corrected before the samples are analyzed.

Before the tank samples were analyzed, a diagnostic check was performed on the GC/FID instrument by running a system cleanliness procedure and an instrument continuing calibration as described in PNL-TVP-08. First, two blank volumes of Aadco purified air were analyzed to check the cleanliness of the system. This demonstrates through the analysis of a zero-air blank that the level of interference is acceptable in the analytical system. The analysis of purified air must be below 0.1 mg/m^3 . Second, an instrument continuing calibration is run using 100-mL UHP propane followed by one blank volume of Aadco air.

3.0 Analysis Results

Results from the sampling of the headspace of Tank U-104 on July 16, 1996 (Sample Job S6072) are provided below. SUMMA™ canister samples from Tank U-104 were received by PNNL 84 days after the sampling event. Samples were then held due to radiological tags on the SUMMA™ canisters. The presence of these tags required the lab to obtain paperwork to allow for filtering of samples prior to analysis. The radiological information required for this paperwork was received by PNNL on December 12, 1996, 70 days after the samples were received by PNNL and 154 days after the sampling event.

3.1 Inorganic Vapors

Measured vapor concentrations of ammonia, nitric oxide (NO), nitrogen dioxide (NO₂), and water are given in Table 3.1. The vapor concentration results were based on four samples for each compound except water vapor which was based on only three samples. The four inorganic vapors were collected at the same time using sorbent traps connected in series. Sample air was drawn first through an ammonia trap, then through a three-tube system that collected nitrogen dioxide and nitric oxide (described below), and then through a desiccant trap to remove any remaining water vapor.

Two field blank multi-trap trains, identical to sample multi-trap trains discussed in Section 2.1, were included in the tube bundle lowered into the headspace of Tank U-104 during Sample Job S6072. No air was pulled through these field blank multi-trap trains. Any analyte found in the field blank multi-trap trains over and above levels in unexposed tubes was attributed to passive sampling. Data in Table 3.1 have been corrected for these minor effects of passive sampling.

Results provided in Table 3.1 are estimated to be accurate to within $\pm 10\%$ and within the $\pm 30\%$ specified by the SAP. Percent relative standard deviations of the measured concentrations were $<2\%$, which is within the 25% specified by the SAP.

3.1.1 Ammonia

Ammonia analyses were performed on January 27, 1997, 195 days after sample collection. All available samples (100%) were successfully analyzed, and no deviations from the procedure were noted. One sample was not available; it had been sacrificed to radiological analysis.

The blank-corrected ammonia quantities in the sorbent traps were less than 0.06 μmol in front sections; blank corrected back sorbent section ammonia concentrations were $<0.01 \mu\text{mol}$. Blank corrections of 0.098 μmol in front and 0.069 μmol in the back sections were about 100% of collected quantities. The analysis of one sample was a duplicate and indicated a reproducibility of $\pm 2.2\%$. One blank sorbent trap was spiked with 17 ppm of ammonia and yielded a percentage recovery of 100%. One sample leachate was spiked after initial analysis with approximately the quantity of ammonia in the sample and yielded a percentage recovery of 95%. The initial and continuing calibration verification (ICV, CCV) standards, using NIST-traceable material, yielded percentage recoveries of 103% (ICV) and 104% and 105% (CCV) during the analytical session. A five-point calibration was performed over an ammonia range of 0.1 to 100 $\mu\text{g/ml}$.

3.1.2 Nitric Oxide and Nitrogen Dioxide

Nitric oxide and nitrogen oxide analyses were performed on January 23, 1997, 191 days after sample collection. All samples (100%) were successfully analyzed. No deviations from the procedure were noted.

Blank-corrected NO_2^- quantities in the sorbent traps were all $<0.013 \mu\text{mol}$. Nitrite blank levels used to correct data were $0.0060 \mu\text{mol}$ in front (four of four blanks analyzed) and $0.0054 \mu\text{mol}$ in back (two of four blanks analyzed) sorbent sections. The analyses of two samples were duplicated and yielded repeatabilities of $\pm 0\%$ and $\pm 2.7\%$. Two sample leachates were spiked with $0.125 \text{ ppm } \text{NO}_2^-$ and yielded percentage recoveries of 92% and 92% . A 4-point calibration was performed over a concentration range of 0 to $0.5 \mu\text{g/mL } \text{NO}_2^-$ in the desorbing matrix.

3.1.3 Water

Analyses for water vapor were performed on January 23, 1997, 191 days after sample collection. All available samples (100%) were successfully analyzed; one sample was not available due to loss during radiological testing.

All multi-trap sample mass gain is assumed to be due to adsorption of water. This is justified because the total mass concentration of other vapors in the headspace of Hanford waste tanks are typically two to three orders of magnitude less than the mass concentration of the water vapor found in even relatively dry tanks. Water vapor concentrations are given in Table 3.1 for both moist air at tank conditions and for dry air at STP (0°C and 1.013 bar). Because the sample volumes were measured after all water vapor was removed by the sorbent traps, the measured sample volumes are for dry air. The average water vapor concentration was 17.0 mg of water per L of dry air at STP. The result was determined from an average mass gain of 34.3 mg from three of four multi-trap trains. The blank correction applied to the results was -2.7 mg per multi-trap train. A control mass was measured and indicated a measurement accuracy of $\pm 0.1 \text{ mg}$. The average water vapor concentration corresponds to a tank headspace dew point at 17.7°C and relative humidity at 94% at the time of sampling.

3.2 Permanent Gases

Hydrogen analyses were performed on February 20, 1997, and analyses for other permanent gases were performed on February 21, 1997. All analyses exceeded the 60-day administrative holding time as specified in the WHC Tank Vapor Characterization QA Plan (WHC 1994) due to the presence of radiological tags on the SUMMA™ canisters. All samples (100%) were successfully analyzed and used in the averages. No deviations from standard procedures were noted.

Measured concentrations of carbon monoxide, carbon dioxide, hydrogen, methane, and nitrous oxide are provided in Table 3.2. Results were based on three samples for each compound. Carbon dioxide and nitrous oxide were observed above the EQL in Tank U-104.

Results provided in Table 3.2 are estimated to be accurate to within $\pm 30\%$ as specified by the SAP.

3.2.1. Procedural Deviations, Observations, and Anomalies. Samples from Tank U-104 were delivered with suspected high radiation levels in all of the canisters. Canister PNL 050 (sample S6072-A04.050) was tested as a representative sample prior to permanent gas analysis for radiation levels. This entailed equilibrating the contents of PNL 050 with an evacuated canister (PNL 049) using a collection filter between the two canisters. The equilibrated pressure of the two canisters was 349 torr. Canister PNL 050 was then diluted 2x with UHP N₂. No radiological contamination was found in PNL 050.

Hydrogen and permanent gas analyses were run using undiluted sample from canister PNL 049. Permanent gas results indicated poor data was obtained using the low pressure canister PNL 049. The sample was rerun using 2x diluted PNL 050. A repeat analysis of the same sample confirmed the result. The results from the diluted sample were consistent with the prior and previous Tank U-104 S6072 tank sample results.

3.3 Total Non-Methane Organic Compounds

Analyses for TNMOCs were performed on March 19, 1997, which exceeded the 60-day administrative holding time as specified in the WHC Tank Vapor Characterization QA Plan (WHC 1994) due to the presence of radiological tags on the SUMMA™ canisters. All three tank samples and the two ambient samples (100%) were successfully analyzed and used in the averages. No deviations from standard procedures were noted.

Table 3.3 lists results of the TO-12 analysis of SUMMA™ canister samples for TNMOCs. Results in Table 3.3 are reported in two different units; in the upper row the mass concentration (mg/m³) of non-methane organic compounds is given at STP (0°C and 1.013 bar), and in the lower row, by EPA TO-12 convention, as ppmv of carbon based on propane as the standard. The average concentration in the three tank headspace samples was below the instrument detection limit or <0.59 mg/m³ or <0.25 ppmv of carbon. Results provided in Table 3.3 are estimated to be accurate to within ± 30% as specified by the SAP.

3.3.1. Procedural Deviations, Observations, and Anomalies. Samples from Tank U-104 were delivered with suspected high radiation levels in all of the canisters. Canister PNL 050 was tested as a representative sample prior to permanent gas analysis for radiation levels. This entailed equilibrating the contents of PNL 050 with an evacuated canister (PNL 049) using a collection filter between the two canisters. The equilibrated pressure of the two canisters was 349 torr. Canisters PNL 050 and PNL 049 were diluted four times the initial canister pressure to bring the pressure to the standard dilution pressures for sample analysis. Both canisters were analyzed and yielded the same results; therefore, the results for sample S6072-A04.050 are reported.

The calibration method described in Section 2.3.2 reflects a deviation from procedure PNL-TVP-08. Refer to Deviation Report JAE082996 for further details.

3.4 Flammability

The analytical results presented above can be used to estimate the Tank U-104 headspace flammability at the time of sampling. Flammability is calculated using the ammonia concentration from the inorganic analysis, carbon monoxide, hydrogen, and methane concentrations measured from the permanent gas analysis, and the total nonmethane organic compound concentration determined from the TO-12 analysis. Table 3.4 summarizes the calculated flammability data. None of the flammable constituents were present at concentrations above the analytical instrument detection limits. Total headspace flammability was estimated to be <0.108% of the LFL.

Table 3.1 Inorganic Vapor Concentrations from Tank U-104 Sampled on 7/16/96

| Analyte | CAS Number | S6072-A07-34R | S6072-A08-35R | S6072-A09-36R | S6072-A10-37R | Average | Standard Deviation |
|-----------------------------|------------|---------------|---------------|---------------|---------------|---------|--------------------|
| Ammonia (ppmv) | 7664-41-7 | NA | <0.71 | <0.71 | <0.71 | <0.71 | |
| Nitric Oxide (ppmv) | | <0.16 | <0.16 | <0.16 | <0.16 | <0.16 | |
| Nitrogen Dioxide (ppmv) | | <0.16 | <0.16 | <0.16 | <0.16 | <0.16 | |
| Water ^(a) (mg/L) | | NA | 16.8 | 16.8 | 17.3 | 17.0 | 0.3 |
| Water ^(b) (mg/L) | | NA | 14.9 | 15.0 | 15.4 | 15.1 | 0.3 |

Footnotes

- (a) Dry air at 0°C and 1.013 bar.
- (b) Moist air at tank temperature and pressure.
- NA Not Analyzed, sample sacrificed to radiological analysis.

Table 3.2 Permanent Gas Analysis Results from Tank U-104 Sampled on 7/16/96.

| Permanent Gas Analyte | Ambient Air | | Tank Samples | | | | | Mean (ppmv) | St.Dev. |
|-----------------------|--|--|--|--|--|--|---------------------------|--------------------|--------------------|
| | Upwind S6072-A01.013 ^(a) (ppmv) | Through Bundle S6072-A02.029 ^(a) (ppmv) | S6072-A04.050 ^(a) (ppmv) | S6072-A06.099 ^(a) (ppmv) | S6072-A05.055 ^(a) (ppmv) | S6072-A05.055 ^(a/b) (ppmv) | RPD ^(c) (%) | | |
| Hydrogen | <17 | <17 | <17 ^(d) | <17 | <17 | <17 | na | <17 | <17 |
| Methane | <25 | <25 | <50 | <25 | <25 | <25 | na | <25 ^(e) | <25 ^(e) |
| Carbon Dioxide | 463 | 452 | 664 | 648 | 643 | 641 | 0.3 | 652 | 11.0 |
| Carbon Monoxide | <17 | <17 | <34 | <17 | <17 | <17 | na | <17 ^(e) | <17 ^(e) |
| Nitrous Oxide | <17 | <17 | 90 | 84 | 84 | 84 | 0.0 | 86 | 3.5 |

Footnotes

- (a) WHC sample identification number.
- (b) Replicate analysis for S6072-A05.055; results are not included in the calculation of average concentrations.
- (c) Relative percent difference (RPD) based on replicate analysis.
- (d) Result obtained from analysis of sample from canister PNL 049 (see Section 3.2.1).
- (e) Mean does not include results from sample S6072-A04.050 as this sample had to be diluted.

na Not applicable.

Table 3.3 Total Non-Methane Organic Compound Analysis Results from Tank U-104 Sampled on 07/16/96

| | Ambient Air | | Tank Samples | | | | | | | |
|----------------------------|-------------|----------------------------|------------------------------|------------------------------|---------------------------------|------------------------------|---------------------------------|--------------------|---------------------|---------|
| | Upwind | Ambient Air Through Bundle | S6072-A01.013 ^(a) | S6072-A02.029 ^(a) | S6072-A04.050 ^{(a)(d)} | S6072-A06.099 ^(e) | S6072-A05.055 ^{(a)(b)} | RPD ^(c) | Mean ^(f) | St.Dev. |
| TO-12 (mg/m ³) | <0.59 | <0.59 | <0.59 | <0.59 | <1.18 | <0.59 | <0.59 | 0.88 | <0.59 | e |
| Total Carbon (ppmv) | | | | | | | | | | <0.25 |

Footnotes

- (a) WHC sample identification number.
- (b) Replicate analy S6072-A05. ; results are not included in the calculation of average concentrations.
- (c) Relative percent difference (RPD) based on replicate analysis.
- (d) S6072-A04.050 transferred into canister 049 for radiation screening. Canister 050 diluted 4X to 1425 Torr. Analysis of canister 049 yielded <1.18 mg/m³ at 4X dilution to 1510 Torr.
- (e) RPD and St.Dev are not meaningful for this analysis.
- (f) Mean does not include results from sample S6072-A04.050 as this sample had to be diluted.

Table 3.4 Flammability Data for Tank U-104 Sampled on 7/16/96

| Analyte | CAS # | Lower Flammibility Limit(LFL) | Average Measured Concentrations | % of LFL ^(a) |
|----------------------------|-----------|----------------------------------|---------------------------------------|-------------------------|
| Ammonia (ppm) | 7664-41-7 | 150000 | <0.71 | <0.0005 |
| Carbon Monoxide (ppm) | 630-08-0 | 125000 | <17 | <0.014 |
| Hydrogen (ppm) | 1333-74-0 | 40000 | <17 | <0.043 |
| Methane (ppm) | 74-82-8 | 50000 | <25 | <0.050 |
| TNMOC (mg/m ³) | | 42000 | <0.59 | <0.001 |
| Total | | | | <0.108 |

(a) Less than values are calculated using the average concentration less than values.
These values are summed to determine the total LFL.

4.0 Conclusions

The concentrations of inorganic and organic analytes were determined from samples of the headspace of Tank U-104 on July 16, 1996 (Sample Job S6072). The vapor concentrations were based either on whole-volume samples (SUMMA™ canisters) or on sorbent traps exposed to sample flow. In the case of the canisters, the concentrations were based on analytical results and the tracking of dilution/concentration of sample volumes obtained directly from the canisters. In the case of the sorbent traps, concentrations were based on analytical results and sample volumes reported by WHC. Known sampling and analytical variances from established quality assurance requirements, where significant, were documented in this report, as required by the SAP (Buckley 1996). No immediate notifications were provided because analytical results indicated no specific analytes exceeded the notification levels; notification levels and notification procedures are described in the SAP (Buckley 1996).

5.0 References

Buckley, L. L. 1996. *Vapor Sampling and Analysis Plan*. WHC-SD-WM-TP-335, Rev. 2C, Westinghouse Hanford Company, Richland, Washington.

Ligotke, M. W., J. S. Fruchter, J. L. Huckaby, M. B. Birn, B. D. McVeety, J. C. Evans, K. H. Pool, K. L. Silvers, and S. C. Goheen. 1995. *Waste Tank Vapor Characterization Project: Annual Status Report for FY 1995*. Pacific Northwest National Laboratory, Richland, Washington.

Pacific Northwest National Laboratory. *Analytical Laboratory Procedure Compendium*. Procedure PNL-ALO-271. PNL-MA-599, Pacific Northwest National Laboratory, Richland, Washington.

Pacific Northwest National Laboratory. *Quality Assurance Plan for Activities Conducted by the Pacific Northwest National Laboratory Vapor Analytical Laboratory (VAL) and the Pacific Northwest National Laboratory Tank Vapor Characterization Project*. ETD-002, Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

U.S. Department of Energy. *Hanford Analytical Services Quality Assurance Plan (HASQAP)*. DOE/RL-94-55, Rev. 2, United States Department of Energy, Richland, Washington.

Westinghouse Hanford Company (WHC). 1994. *Quality Assurance Project Plan for Tank Vapor Characterization*. WHC-SD-WM-QAPP-013, Rev. 2, Westinghouse Hanford Company, Richland, Washington.

Appendix A

Chain-of-Custody Sample Control Forms

Custody Form Initiator J. A. Edwards - PNNL

Telephone (509) 373-0141
Page 85-3009 / FAX 376-2329

Company Contact R. D. Mahon - WHC

Telephone (509) 373-7437
Page 85-9858 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm
241-U-104 Tank Vapor Sample SAF S6072
(ISVS Cart)

Collection date 07 - 16 - 96
Preparation date 07 - 08 - 96

Ice Chest No.

Field Logbook No. WHC-N 47 - 8

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

~~S6072 - A07 . 34R Collect NH₃/NO_x/H₂O Sorbent Trap~~ *75th 3 Oct 96*
S6072 - A08 . 35R Collect NH₃/NO_x/H₂O Sorbent Trap
S6072 - A09 . 36R Collect NH₃/NO_x/H₂O Sorbent Trap
S6072 - A10 . 37R Collect NH₃/NO_x/H₂O Sorbent Trap

S6072 - A15 . 38R Open, close and store NH₃/NO_x/H₂O field blank #1
S6072 - A16 . 39R Open, close and store NH₃/NO_x/H₂O field blank #2

| [] Field Transfer of Custody | | [X] Chain of Possession | | (Sign and Print Names) | | |
|--------------------------------|----------|-------------------------|--------------------------------|------------------------|------|--|
| Relinquished By | Date | Time | Received By | Date | Time | |
| G W Dennis <i>A.W. Dennis</i> | 07-08-96 | 1335 | J A Edwards <i>J A Edwards</i> | 07-08-96 | 1335 | |
| J A Edwards <i>J A Edwards</i> | 07-12-96 | 1025 | <i>GS CAPPIO to CD</i> | 07-12-96 | 1025 | |
| <i>GS CAPPIO to CD</i> | 07-30-96 | 1335 | Rick Mahon <i>Rick Mahon</i> | 09-30-96 | 1338 | |
| <i>Rick Mahon Rick Mahon</i> | 09-07-96 | 1345 | <i>ES MOST 9/16</i> | 29 Oct 96 | 1345 | |
| <i>ES MOST</i> | 10-29-96 | 1415 | J A Edwards <i>J A Edwards</i> | 10-29-96 | 1415 | |
| J A Edwards <i>J A Edwards</i> | 1-21-97 | 1305 | Tom Polhemus <i>UMP Thomas</i> | 1-21-97 | 1305 | |

Final Sample Disposition

Comments:

PNNL (only) Checklist

| | | | |
|---|------------------------------------|---------------------------------------|--------------------|
| 0 | Media labeled and checked? | <input checked="" type="checkbox"/> Y | Pick-up / Delivery |
| 0 | Letter of instruction? | <input checked="" type="checkbox"/> Y | |
| 0 | Media in good condition? | <input checked="" type="checkbox"/> Y | |
| 0 | COC info/signatures complete? | <input checked="" type="checkbox"/> Y | |
| 0 | Rad release stickers on samples? | <input checked="" type="checkbox"/> Y | |
| 0 | Activity report from 222S? | <input checked="" type="checkbox"/> Y | |
| 0 | RSR/release? (α ≤100/B ≤400 pCi/g) | <input checked="" type="checkbox"/> Y | |
| 0 | COC copy for LRB, RIDS filed? | <input checked="" type="checkbox"/> Y | |

Comments:

S6072-A07.34R was moved to COC 100519 to allow WSCF to perform a destructive analysis of the NH₃ and silica gel tube. The two NO_x tubes were moved to COC 100520 for return to PNNL. Room 3 Oct 96

POC *(Signature)*

POC *(Signature)*

Custody Form Initiator J. A. Edwards - PNNL Telephone (509) 373-0141
Page 85-3009 / FAX 376-2329

Company Contact R. D. Mahon - WHC Telephone (509) 373-2891
Page 85-3656 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm Collection date 07 - 16 - 96
241-U-104 Tank Vapor Sample SAF S6072 Preparation date 07 - 12 - 96
(ISVS Cart)

Ice Chest No. Field Logbook No. WHC-N-647.8

Bill of Lading/Airbill No. N/A Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6072 - A01 . 013 Collect Ambient Air Sample SUMMA #1
S6072 - A02 . 029 Collect Ambient Air Sample SUMMA #2 (through tube bundle)

S6072 - A04 . 050 Collect SUMMA #3
S6072 - A05 . 055 Collect SUMMA #4
S6072 - A06 . 099 Collect SUMMA #5

| [] Field Transfer of Custody | | [X] Chain of Possession | | (Sign and Print Names) | | |
|--------------------------------|----------|---------------------------|--------------------------------|------------------------|------|--|
| Relinquished By | Date | Time | Received By | Date | Time | |
| J A Edwards <i>J A Edwards</i> | 07-12-96 | 1030 | GSCAPRIO <i>J A Edwards</i> | 07-12-96 | 1030 | |
| GSCAPRIO <i>J A Edwards</i> | 10-8-96 | 0950 | J A EDWARDS <i>J A Edwards</i> | 10-8-96 | 0950 | |
| | | | | | | |
| | | | | | | |

Final Sample Disposition

Comments:

- | PNNL (only) Checklist | Pick-up / Delivery | Comments: |
|------------------------------------|---|-----------|
| Media labeled and checked? | <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N | |
| Letter of instruction? | <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N | |
| Media in good condition? | <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N / <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N | |
| COC info/signatures complete? | <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N / <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N | |
| Rad release stickers on samples? | <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N / <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N | |
| Activity report from 222S? | <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N / <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N | |
| RSR/release? (a ≤100/B ≤400 pCi/g) | <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N / <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N | |
| COC copy for LRB, RIDS filed? | <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N / <input checked="" type="checkbox"/> Y / <input type="checkbox"/> N | |

POC *[Signature]* POC *[Signature]*

(WHC-SD-WM-TP-335, REV. 2, Table 2b)

(Revised 05/30/96 PNNL)

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

| | | | | | | | | |
|---|------------|--|------------|-------------------|---|-------------|-----------------|------------------------------------|
| Westinghouse Hanford Company | | C.O.C.# /00519 | | | | | | |
| Collector: Glenn Caprio/ Rick Mahon | | Page 1 of 1 | | | | | | |
| SAF Number S6072 | | Tel. No. 373-7437 MSIN 33-27 FAX 373-7076 | | | | | | |
| Project Title: <i>Upper Sampling</i> | | Purchase Order/Charge Code E46353 | | | | | | |
| Shipped to (Lab) HSCF | | Ice Chest# NA Temp. MA | | | | | | |
| Project: None | | Date of Loading/Air Bill No. NA | | | | | | |
| Method of Shipment: Government Vehicle | | Offsite Property No. NA | | | | | | |
| Data Turnaround: 24 hr. | | Preservative | | | | | | |
| Sample No. | Lab. ID | Date | Time | No/Type Container | Sample Analysis | | | |
| S6072-AD734R | | 16JUL96 | 1100 | (1) | AT/TB/GEA | | | |
| S6072-AD734R | | 16JUL96 | 1100 | (1) | Tritium | | | |
| NA | NA | NA | NA | () | NOTE: These samples were moved from COC 100255. This sample is listed as one entry on the original COC but is listed here as two separate samples because the tubes were separated from each other. | | | |
| | | | | () | The PFA connections were cut off to allow USCF to analyze. | | | |
| | | | | () | The N/A's and silica gel tubes separately. The NOX portion of the sample was moved to COC 100520. ADM 30876 | | | |
| | | | | () | | | | |
| | | | | () | | | | |
| | | | | () | | | | |
| | | | | () | | | | |
| | | | | () | | | | |
| | | | | () | | | | |
| POSSIBLE SAMPLE HAZARDS/REMARKS List all known wastes. | | | | | | | | |
| SPECIAL INSTRUCTIONS Please fax the results with the time and day of analysis to Rick Mahon at 373-7076. Thank You. | | | | | | | | |
| Relinquished By | Print | Sign | Date/Time | Received By | Print | Sign | Date/Time | Matrix* |
| Rick Mahon | Rick Mahon | Rick Mahon | 70076/7/27 | TANIA BATES | TANIA BATES | JANIA BATES | 10-7-96 (f.i.s) | S SE SO SL W O A |
| Relinquished By | | | Date/Time | Received By | | | Date/Time | DL T WT L V X |
| Relinquished By | | | Date/Time | Received By | | | Date/Time | DS DL T WT L V X |
| Relinquished By | | | Date/Time | Received By | | | Date/Time | DS DL T WT L V X |
| FINAL SAMPLE DISPOSITION Disposal Method: <input type="checkbox"/> Return to customer, per lab procedure, used in process. | | | | | | Disposed By | | |

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

Westinghouse Hanford Company

COC# 100520

Collector: Rick Mahon
 SAF Number: S6072
 Project Title: Va per Sampling
 Shipped To (Lab): PNNL
 Protocol: RCRA/CECLA

Contact/Requestor: Rick Mahon
 Sample Origin: U-104 ISVS
 Logbook #: WHC-N-647-8
 Method of Shipment: Government Vehicle
 Data Turnaround: See SAP

Tel. No. 373-7437 MSIN 53-27 FAX 373-7076
 Purchase Order/Charge Code: See Larry Remington
 Ice Chest #: NA Temp: NA
 Bill of Lading/Air Bill No.: NA
 Offsite Property No.: NA

| Sample No. | Lab ID | Date | Time | No/Type Container | Sample Analysis | Preservative |
|--------------|--------|-----------|------|-------------------|---|--------------|
| 3486 | | 16 JUL 96 | 1155 | (1) | See SAP | NA |
| S6072-A07348 | | 16 JUL 96 | 1155 | (1) | See SAP | |
| 3487 | | 16 JUL 96 | 1155 | (1) | See SAP | |
| S6072-A07348 | | NA | NA | (1) | NA | |
| NA | | | | (1) | NOTE: These samples were moved from COC 100255. This sample is listed as one entry on the original COC but it listed here as three separate samples because the tubes were separated from each other. The PFA connections were cut off to allow WCF to analyze the NH3 and silica gel tubes separately. The NH3 samples will be returned to PNNL. The NH3 and silica gel were moved to COC 100579. RDM 30E796 | |
| | | | | (1) | | |
| | | | | (1) | | |

POSSIBLE SAMPLE HAZARDS/REMARKS: MSDS Yes No X SPECIAL INSTRUCTIONS: Held Time

List all known wastes.

Received By: Rick Mahon Date/Time: 29 OCT 96 / 1345
 Signature: Rick Mahon
 Received By: E Mc S mart Date/Time: 29 OCT 96 / 1430
 Signature: Jeff Edwards
 Received By: Jeff Edwards Date/Time: 10/29/96 @ 1430
 Signature: Jeff Edwards
 Received By: LMP Thomas Date/Time: 1-21-97 @ 1305
 Signature: LMP Thomas

Matrix: S - Soil, SE - Sediment, SO - Solid, SL - Sludge, W - Water, O - Oil, A - Air, DS - Dried Solids, DL - Dried Liquids, T - Tissue, WI - Waste, L - Liquid, V - Vegetation, X - Other

FINAL SAMPLE DISPOSITION: Disposed Method: e.g. Return to customer, per lab procedure, used in process. Disposed By: Date/Time:

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin. A-6001-500 (07/95)

Distribution List

PNNL-11266

PNNL

| | |
|------------------|------------------|
| J. C. Evans | K6-96 |
| K. H. Pool | P8-08 |
| J. C. Hayes | K6-96 |
| A. V. Mitroshkov | K6-96 |
| J. A. Edwards | P8-08 |
| J. L. Julya | K6-75 |
| B. M. Thornton | K6-80 (3 copies) |
| J. S. Fruchter | K6-96 |
| K. L. Silvers | K9-08 |
| J. L. Huckaby | K6-80 |
| D. A. Varley | K1-06 |

LMHC

| | |
|------------------|-------|
| L. D. Pennington | S7-21 |
| L. L. Buckley | R2-12 |

DOE-RL

| | |
|----------------|-------|
| C. A. Babel | S7-54 |
| J. F. Thompson | S7-54 |