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7. Abstract
This document is to serve as a reference guide for gas and vapor sample results presented in tank characterization reports. It describes sampling equipment, devices, and protocols, and sample collection and analysis methods common to all vapor samples.
Waste Tank Headspace Gas and Vapor Characterization Reference Guide

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MASTER
1.0 INTRODUCTION

1.1 Purpose

This document describes high-level radioactive waste tank headspace gas and vapor sample collection and sample analysis methods. It is a supporting document to Tank Characterization Reference Guide (De Lorenzo 1994).

1.2 Scope

This document is to serve as a reference guide for gas and vapor sample results presented in tank characterization reports. It describes sampling equipment, devices, and protocols, and sample collection and analysis methods common to all vapor samples.

1.3 Drivers

Westinghouse Hanford Company (WHC) is characterizing the waste tank headspace gases and vapors for 3 reasons. First, there are worker health and safety issues associated with the vented gases and vapors. The WHC industrial hygiene worker protection strategy includes identifying potentially harmful conditions by determining the tank headspace concentrations of noxious gases and vapors. Second, there are headspace flammability issues because some of the gases and vapors present are flammable or are oxidizing agents. Though combustible gas meters (CGMs) are used to monitor tank headspace flammability, there are potentially conditions in the waste tanks which are not properly addressed by CGMs. And third, the possible existence of organic liquids in the waste tanks is an issue. Vapor samples may be useful in identifying which, if any, of the waste tanks contain organic liquids.
2.0 SAMPLE COLLECTION

2.1 Sample Types

Two types of sampling devices are generally used to sample waste tanks. SUMMA™ canisters are used to collect certain gases and volatile organic vapors, and sorbent traps are used to collect organic and certain inorganic gases and vapors.

2.1.1 SUMMA™ Canisters

SUMMA™ canisters are stainless steel vessels whose internal surfaces have been prepared by the SUMMA™ process. This process passivates active sites on the canister walls to minimize their adsorption of gases and vapors. SUMMA™ canisters used for waste tank sampling are cleaned and evacuated at an analytical laboratory before use. The evacuated canisters are filled with air from the tank through a valve, which is then closed to seal the sample inside. SUMMA™ canisters essentially allow collection and transfer of whole-air samples from location to an analytical laboratory where the sample can be analyzed. SUMMA™ technology is generally accepted by analytical air chemists for ambient air, and is specifically cited in the U.S. Environmental Protection Agency (EPA) Task Order 12 (TO-12) and Task Order 14 (TO-14) methods for ambient air analysis (EPA 1988).

Despite being whole-air sampling devices, SUMMA™ canisters are not appropriate sampling devices for highly polar or water soluble vapors such as ammonia and acid gases (e.g., nitric oxide, nitrogen dioxide, sulfur dioxide). The tendency of such vapors and gases to be adsorbed or even chemisorbed on the canister walls affects the quantitative recovery of these analytes from SUMMA™ canisters.

2.1.2 Sorbent Traps

Sorbent traps are used to sample organic vapors and specific inorganic gases and vapors in waste tank headspaces. Sorbent traps are typically 6 to 12-mm diameter, 50 to 75-mm long tubes that contain one or more beds of sorbent material, such as activated carbon. A known amount of sample gas is passed through the tube, which traps (by adsorption or chemisorption) virtually all of the target analyte(s). Unlike SUMMA™ canisters, sorbent traps concentrate targeted analytes by selectively removing them from the air sample, and the other constituents of the air (e.g., the oxygen, nitrogen, water, etc.) are not collected. While in principle sorbent devices could be designed to sample even gases like hydrogen, it is more

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1 SUMMA is a trademark of Molectrics, Inc., Cleveland, Ohio.
practical to employ a whole-air sampling device (e.g., SUMMATM canister) for such analytes.

After sampling is complete, sorbent traps are sealed and sent to a laboratory for analysis. At the analytical laboratory, the analyte is extracted from the sorbent media, and quantitated. Given the quantity of analyte recovered and the total volume of air sampled, the concentration of analyte in the original air sample is calculated. Sample air flow rates and the duration of flow through sorbent trap sampling are specified by the analytical laboratories which supply and later analyze the sorbent traps, and are reported by Sampling and Mobile Laboratories (S&ML).

### 2.2 Sampling Apparatus

The Vapor Sampling System (VSS) is used to collect representative samples of the air, gases, and vapors from the waste tank headspaces. The VSS consists of a mobile laboratory, a hot-water jacketed stainless steel probe that is inserted into the tank headspace, and stainless steel transfer tubing that connects the mobile laboratory with the probe. Sample air is drawn from the tank headspace and through the transfer tubing and sampling manifold by an air pump. Air flow within the VSS is directed by electrically activated, pneumatically actuated valves. Instrumentation housed in the mobile laboratory provides system temperature monitoring and controlling, absolute and differential system pressure monitoring, mass flow metering and controlling, and hydrocarbon vapor monitoring. Mahon et al. (1994) describe the VSS, its performance, and its operation in detail.

A key feature of the VSS is its use of heated sample transfer tubing and a heated sampling manifold. Maintaining the system temperature at an electronically controlled, elevated temperature prevents vapor condensation and reduces vapor adsorption on surfaces exposed to sample air. Mahon et al. (1994) describe various tests and observations that indicate the VSS sample transfer efficiency is consistently high.

Sorbent trap samples are collected at the sorbent trap station of the sampling manifold. High-accuracy mass flow controllers are used to measure and control the flow rate of sample air through the sorbent traps. These are located downstream of the sorbent trap station, and downstream of in-line driers which remove water vapor from the air before it is metered. The sorbent trap station allows collection of 2 samples simultaneously, and most samples are taken in pairs.

The VSS sampling manifold also has a station for sampling air with evacuated SUMMATM canisters. To prevent condensation and minimize adsorption of vapors during SUMMATM canister sampling, the connection tubing between the sampling manifold and the
canister itself, as well as the mechanical valve on the SUMMA™, are heated with electrical heat tape.

The VSS is cleaned according to LO-450-80 (WHC 1994) before being used. A gas chromatograph (GC) with a flame ionization detector (FID) is used to confirm the VSS is free of hydrocarbons to its limit of detection. The GC/FID is part of the VSS, and may be used periodically throughout a sampling event to monitor organic vapor concentrations in the air being sampled.

2.3 Sampling Event Chronology

Heating of the transfer tubing and sampling manifold of the VSS typically begins 3 to 16 hrs before the first samples are collected. During this warm-up period the VSS sampling manifold is maintained at a partial vacuum of about 280 mbar (210 torr).

After the heated zones of the VSS have reached operating temperature, the sampling manifold is typically purged with ambient air for 30 min at a flowrate of more than 5 L/min. Two SUMMA™ canisters samples of ambient air are generally collected, one manually about 10 m upwind of the tank ventilation riser, and one using the VSS sampling manifold. The former is collected and analyzed to establish background levels of trace organic vapors, and the latter is collected and analyzed to establish the cleanliness of the sampling manifold.

A leak check of the VSS sampling manifold and transfer tubing (up to its connection to the in-tank sampling probe) is performed after ambient air samples have been collected. For this, the sampling manifold is evacuated to 280 mbar (210 torr), and the pressure of the system is monitored. Leakage resulting in less than a 3.3 mbar (2.5 torr) rise in pressure over the 15 min test is allowed. Given an estimated system volume of 8.8 L (using a typical 9.1 m transfer tube), this pressure rise corresponds to a leak rate of less than 7 mL/min at the 280 mbar (210 torr) pressure of the leak test. During the collection of sorbent traps the system pressure is typically about 987 mbar (740 torr), at which the same leak rate would be about 2 mL/min. During collection of SUMMA™ canisters most of the sampling manifold is shut off, and the pressure of the manifold in the vicinity of the canister may drop below the pressure of the system during the leak test, but only for several seconds. The potential amount of air leaked into the manifold during SUMMA™ canister filling would be very small compared to the total 6.0 L SUMMA™ canister sample.

The VSS is purged with tank headspace air for 30 min at a flow rate greater than 5 L/min. This purge is performed to flush ambient air from the system, and saturate active adsorption sites of the system. Given that the volume of the heated sampling probe, transfer tubing and sampling manifold upstream of the sampling devices is typically less than 2.5 L,
this purge provides approximately 60 air turn-overs in the system upstream of the sampling ports.

2.4 Sampling Error

Errors and uncertainties in the control of sample flow rate and duration result in sorbent trap sample volume errors and uncertainties. Mahon et al. 1994 provide a detailed discussion of sorbent trap sample volumes and estimated uncertainties. The error as determined by standards laboratory calibration is presented as uncertainty in the volume measurements for different types of samples. These ranged from 0.03% to 1.65%. This error is small compared to errors associated with sample analysis.

2.5 Blanks and Controls

Tank air samples are accompanied by trip blanks, field blanks, or some other control such as an ambient air sample. Trip blanks are sampling devices, identical to real samples, that accompany real samples everywhere but are not opened or exposed in the field. Trip blanks provide evidence that the samples have not been contaminated during handling and shipping. Spiked trip blanks also provide evidence that analytes have not been lost during handling or shipping. Field blanks are sampling devices that are handled in as identical manner as possible to real samples (and opened in the field), but are not used to collect samples. Field blanks provide evidence that samples are not being contaminated during shipping, handling, or by inadvertent exposure in the field.

Generally, each type of sorbent trap has at least 2 trip blanks. Sorbent trap field blanks are employed periodically. SUMMATM canister trip blanks are not employed, because contamination of a canister sample would generally not be a function of handling or shipping conditions. SUMMA™ canisters may become contaminated if a seal or valve fails, but the fact that a trip blank canister had or had not become contaminated would have no bearing on whether the sample canisters had become contaminated. For lack of a more suitable control, an ambient air SUMMA™ canister is collected.
3.0 SAMPLE ANALYSIS

The waste tank headspaces may have high concentrations of gases and vapors relative to ambient air, and standard ambient air sampling and analysis methods are not always directly applicable. Analytical procedures used are described and/or referenced in the individual analytical laboratory reports for each waste tank.

Analytes may be "positively" or "tentatively" identified. For positive identification, the analytical instrumentation must be tested with a known chemical standard of the analyte in question, and its qualitative response (e.g., elution time) to the sample analyte must be positively correlated with its response to the standard. Sample analytes are referred to as tentatively identified when there is reasonable evidence for the identification, but the qualitative response of the instrumentation to the analyte in question has not been established with a standard.

The amount of an analyte may be "quantitatively" determined, or simply estimated. For the analyte to be quantitatively measured, the analytical instrumentation must be calibrated with known quantities or concentrations of the analyte before the sample is analyzed. Usually 3 to 5 calibration points are required. The observed response of the instrument to the unknown must be within the range of (bracketed by) the calibration, and the calibration points may be required to satisfy certain linearity requirements. The concentrations of tentatively identified analytes can only be estimated. Data from the mass spectral analysis of organic analytes, for example, is frequently sufficient to tentatively identify analytes using spectral libraries. The estimation of the tentatively identified analytes' concentration is based on assumptions about such parameters as the instrument response, which result in significant errors. Before using estimated concentrations for decision making, these assumptions should be reviewed and evaluated.

3.1 Sorbent Traps for Inorganic Vapors

Sorbent traps are used to sample ammonia, NH₃, nitrogen dioxide, NO₂, nitric oxide, NO, and water vapor. These are supplied and then analyzed by PNL. Sorbent trap sampling efficiency is validated by using, when possible, two sections of sorbent media in series. If sampling efficiency is high, the first section of sorbent media collects nearly all of analyte, and the second section (the breakthrough section) collects very little analyte. Analysis of breakthrough sections is discussed in the PNL analytical reports. Brief descriptions of the traps and their analysis are given in the following subsections.

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

3.1.2 Nitrogen Dioxide and Nitric Oxide

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

3.1.3 Water Vapor

The water vapor concentration of the headspace is established using gravimetric analysis of sorbent traps used for other analytes. Ammonia sorbent traps, for example, are prepared with a silica gel sorbent trap for water vapor connected to the outlet end. This ammonia/water vapor sorbent trap system is weighed before and after sampling, and the observed gain in weight is used to calculate the amount of water in the sample.

3.2 Organic Sorbent Traps

Triple sorbent traps (TSTs) are fabricated, conditioned, and analyzed at Oak Ridge National Laboratory (ORNL). As suggested by their name, TSTs contain three types of sorbent media, each segregated as a bed. The first bed consists of Carbotrap CTM2, which effectively adsorbs semivolatile organic vapors having carbon chain lengths of eight or greater. The second bed consists of CarbotrapTM, which adsorbs most organic vapors more volatile. The third bed consists of Carbosieve III™, which is a molecular sieve capable of trapping virtually all organic vapors and gases.

Maintaining an appropriate air flow in the specified direction through a TST results in a distribution of compounds among the three beds. Analytes are extracted from the TST by thermal desorption into a gas stream flowing backwards through the trap. This ensures that,

\[^2\text{Carbotrap, Carbotrap C and Carbosieve III are registered trademarks of Suppelco, Bellefonte, Pennsylvania.}\]
for example, semivolatiles trapped on the first bed are never exposed to the second or third beds, from which they are not easily desorbed. Jenkins et al. (1994) provide detailed information on the preparation, conditioning, validation, and analysis of TSTs.

The configuration of TSTs is such that there is little doubt that all target analytes are trapped; the more pertinent question is whether all trapped analytes are indeed desorbed. TST analyte desorption efficiency is addressed by spiking the sorbent traps with surrogates. A surrogate, in this context, is a compound having similar or even identical chemical properties as the analyte. All TSTs (samples and blanks) are spiked with known quantities of surrogate compounds not expected to be found in the tank headspace before being used to collect samples. Desorption efficiency is calculated by comparing the amount of surrogate recovered with the amount spiked, and reported in the ORNL tank vapor analysis reports.

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

### 3.3 SUMMA™ Canisters

SUMMA™ canisters are cleaned, certified as clean using either the EPA TO-12 or TO-14 methods, and evacuated by the supplying laboratory. Generally, the canister is pressurized by the addition of either ultra-pure air, helium, nitrogen, or oxygen at the analyzing laboratory to facilitate sample removal, which dilutes the constituents of interest. The ratio of diluent gas + sample air to sample air (dilution ratio) may be determined from the change in canister pressure (this is the PNL method), or, if the diluent gas is not air, the concentration of diluent can be determined by measuring the helium concentration analytically (this is the OGIST method). Similarly, the volume of an aliquot taken from the canister may be measured by metering the flow (the PNL method) or measuring pressure changes (the OGIST method).

### 3.3.1 Selected Gases and Inorganic Vapors

SUMMA™ canister samples are analyzed at OGIST for hydrogen, carbon monoxide, methane, carbon dioxide, and nitrous oxide. The canisters are pressurized with ultra-clean helium or oxygen to facilitate sample removal and transfer. The pressurized canisters are allowed to equilibrate at laboratory temperature, and the dilution ratio is determined by a GC with a thermal conductivity detector (TCD). Different analytes are then measured with different instruments: Hydrogen and carbon monoxide are measured using a GC equipped with a mercuric oxide gas reduction detector; methane is measured by a GC/FID; carbon dioxide is measured by a GC/TCD; and nitrous oxide is measured by a GC equipped with an electron capture detector. Analyses are performed only after instrument calibration and
calibration curve linearity has been established with standards. The OGIST analytical reports provide further details on these methods.

### 3.3.2 Organic Vapors

Two types of analyses are performed to measure organic vapor concentrations in SUMMA™ canister samples, the EPA TO-12 and TO-14 methods (EPA 1988). Both methods use a cryogenically cooled loop of tubing to condense the nonmethane organic compounds in an aliquot of the air sample. The condensed analytes are then rapidly heated and swept into a GC with a carrier gas.

In the TO-12 method, the GC performs very little chemical separation, and a FID is used to quantitatively measure the total nonmethane organic compound (TNMOC) concentration. The TO-12 method was designed and intended for ambient air sample analysis, where TNMOC levels are relatively low, and the predominant organic vapors are volatile. Unfortunately, its accuracy and repeatability when applied to waste tank air samples decreases as the TNMOC concentration increases, and as the ratio of semivolatile to volatile vapors increases. Thus, a measured TNMOC concentration of 1000 μg/m³ is probably quite accurate and repeatable, but very high TNMOC concentrations (>10⁶ μg/m³) due to semivolatile organic vapors should be considered to be only an estimate. In an extreme case, the TNMOC concentration of tank 241-C-103 samples, which have high concentrations of the semivolatile normal paraffinic hydrocarbons, could only be determined to be between 2 x 10⁶ and 5 x 10⁶ μg/m³. The TO-12 method is still very useful for 2 reasons: First, it does provide an estimated tank TNMOC concentration for comparison with other tanks; and second, it allows the analytical laboratory to determine proper sample aliquot size for optimum TO-14 analyses.

In the TO-14 method, a capillary column is used to optimize chemical separation within the GC, and a mass spectrometer (MS) detector is used to identify and quantitate the analytes as they elute. Though the TO-14 method, as published by EPA, has a target list of only about 40 specifically named analytes, the method can be applied to identify and quantitate other analytes as well. However, most TO-14 method analyses performed to date have positively identified and quantitated only the TO-14 analytes, and other analytes have been tentatively identified and their concentrations have been estimated.
4.0 REFERENCES


