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# Quality Assurance Project Plan for Waste Tank Vapor Characterization

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## EXECUTIVE SUMMARY

This Quality Assurance Project Plan, WHC-SD-WM-QAPP-013, applies to four separate vapor sampling tasks associated with Phases 1 and 2 of the Tank Vapor Issue Resolution Program and support of the Rotary Mode Core Drilling Portable Exhauster Permit. These tasks focus on employee safety concerns and tank ventilation emission control design requirements.

Previous characterization efforts and studies are of insufficient accuracy to adequately define the problem. It is believed that the technology and maturity of sampling and analytical methods can be sufficiently developed to allow the characterization of the constituents of the tank vapor space.

The goals of this effort are as follows:

### Phase 1

1. Identify analytes collected by the sampling systems and obtain rough quantitative data describing the constituents of the vapor space of tank 241-C-103.
2. Select the best sampling and analytical process/methodology for vapor characterization.

These will be accomplished through obtaining a better understanding of the tank vapor space by applying validated sampling/analytical procedures,

identifying the performance (accuracy and precision) of those methods, understanding which of the methods are best applicable to the effort, and establishing the sampling protocol.

3. From the results of 1. (above), and in consultation with the Tank Vapor Conference and the Toxicology Panel, revise the list of analytes of concern and analytes of interest for targeted analysis.

## Phase 2

1. Fully characterize volatile components of concern and interest in the vapor phase of tank 241-C-103, both qualitatively and quantitatively.
2. Specify modifications to the characterization process/methodology that will allow the vapor spaces of all Hanford Site waste tanks to be characterized.

This will be accomplished by development of a sitewide vapor verification program, Tank Advisory Panel approval, and sufficient characterization of the tank vapor space contents (hazardous, flammable, and permanent gases/vapors) to permit the cost effective engineering of a mitigation or remediation system for tank 241-C-103. The Project shall apply validated vapor sampling and analytical procedures to specified waste tanks, and support the Rotary Mode Core Sampling program. This program requires the qualitative and quantitative characterization of analytes of concern and interest for the permitting of a portable exhauster to be operated during rotary mode core sampling.

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# LIST OF TERMS

AMS	Analytical Measurement System
DOE	U.S. Department of Energy
DQO	Data Quality Objectives
EPA	Environmental Protection Agency
FID	Flame Ionization Detection
GC	Gas Chromatography
GC/FID	Gas Chromatography/Flame Ionization Detection
GC/MS	Gas Chromatography/Mass Spectrometry
GC/TC	Gas Chromatography/Thermal Conductivity Detector
IC	Ion Chromatography
MSA	Matrix Spike Addition
MS	Mass Spectrometry
MZG	Moist Zero Gas
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Science and Technology
OGI	Oregon Graduate Institute
PNL	Pacific Northwest Laboratories
QA	Quality Assurance
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	Quality Control
RSD	Relative Standard Deviation
Sandia NL	Sandia National Laboratories
SIE	Specific Ion Electrode
TLV/TWA	Threshold Limit Value/Time Weighted Average
TQL	Target Quantitative Limit
TWRS	Tank Waste Remediation System
WHC/PAL	Westinghouse Hanford Company/Processing and Analytical Laboratories

## QUALITY ASSURANCE PROJECT PLAN FOR WASTE TANK VAPOR CHARACTERIZATION

### 1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) identifies procedures, describes applicable quality control (QC) and quality assurance (QA) methods, and documents information that will be used to support vapor sampling and analysis for the characterization of waste tank vapor spaces. The plan is limited to the developmental effort to accomplish objectives outlined in Section 2.0. Westinghouse Hanford impact level 3SQ is assigned to this effort until the evaluation of the results indicates the need for another level.

Data quality objectives (DQO) are being developed to support the decisions to be made using the data obtained from this effort. These decisions involve the chemical constituents contained in waste tank vapor spaces, and will be developed from sampling efforts performed on tank 241-C-103. The objectives do not include establishing the mechanism of vapor formation and do not include the characterization of any medium other than the tank vapor space.

The initial objectives of Phase 1 will be to select the best process/methodology of tank vapor space characterization and to obtain preliminary measurements of the vapor space constituents in the specified tanks.

The ultimate objectives of Phase 2 are to establish a tank vapor space characterization program that can be applied to any Hanford Site waste tank and to characterize the vapor space of any waste tank. Included in this characterization will be an identification of flammability potential and a health hazard determination resulting from identification of toxic and/or noxious vapor and gaseous constituents. The details of the DQOs for the vapor space characterization effort are subject to evaluation and refinement. All numerical limits contained herein are tentative and subject to change.

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## 2.0 PROJECT DESCRIPTION

### 2.1 PROJECT TASK BREAKDOWN

The purpose of this document is to provide interim QA/QC guidance for sampling and analyzing the vapor space according to the following tasks:

- Obtain triplicate SUMMA canister vapor samples for qualitative analysis of a specific set of organic analytes using a heated sample probe at a single level in tank 241-C-103 according to WHC-SD-WM-WP-198 (WHC,1993).
- Obtain SUMMA canister and solid sorbent vapor samples for the development of quantitative analysis methods using a heated probe at each of three levels in tank 241-C-103 according to WHC-SD-WM-WP-223 (WHC,1993).
- Sample and analyze the vapor in the tank C-103 according to WHC-SD-WM-WP-174 (WHC 1992).

### 2.2 SAMPLE ANALYSIS TECHNOLOGIES

In general, the samples taken from these tanks will be analyzed under the QA/QC requirements described in this document. The effort will involve the use of four independent sampling and analysis techniques in Phases 1 and 2. These techniques are:

- SUMMA<sup>1</sup> canister sampling followed by GC/MS<sup>2</sup> or GC/FID<sup>3</sup> organic analysis
- Sorbent Tubes<sup>4</sup>
- GC/FID or GC/TC organic vapor space analysis either (1) after direct sampling or (2) solid sorbent sampling of the vapor

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<sup>1</sup>Stainless steel sampling container with electropolished interior walls and certified to a cleanliness of 12  $\mu\text{g}/\text{m}^3$  or 20 ppb.

<sup>2</sup>Gas chromatographic separation of organic vapors followed by mass spectrometric identification and quantification of eluting compounds.

<sup>3</sup>Gas chromatographic organic vapor separation followed by quantification of the eluting compounds using a monitor having a flame ionization detector

<sup>4</sup>Pencil-like glass or metal tubes filled with sorbent material

- Midget impingers (bubblers)<sup>1</sup> for inorganic vapor analysis.

### 2.3 QA OVERSIGHT

During sampling QA will perform surveillances, where applicable, in accordance with Section 10.4 of the Quality Assurance Manual (QAM), WHC-CM-4-2 (WHC, 1988), and MRP-5.50 of the Management Requirements and Procedures (MRP), WHC-CM-1-3 (WHC, 1988). Specific inspections by QC shall be based upon work packages and shall be documented on a QA Surveillance Plan. Additionally, a calibration check shall be performed to ensure the accuracy of the instruments being used to monitor flammable gas.

The QA requirements for all onsite laboratories will be implemented in accordance with QAMS-005/80 (EPA, 1983) and applicable QAPPs. Work performed by the subcontractors shall be implemented by a QA Program that satisfies the requirements of this QAPP. Westinghouse Hanford QA will perform oversight and surveillance of offsite subcontractor activities on vapor analysis.

It is intended that the data collection and analysis of this effort will satisfy several objectives. These include:

- Establish sampling/analysis method validation procedure(s) that will provide required vapor analysis method evaluation data.
- Allow the selection of the best process/methodology for waste tank vapor space characterization.
- Characterize vapor space of waste tank 241-C-103 per WHC-SD-WM-WP-174 (WHC, 1992), "Engineering Work Plan for Tank 241-C-103 Phase 1 Vapor Phase Characterization," including the identification of the vapor space hazards posed by flammable and noxious/toxic constituents.
- Establish a vapor space analysis program that can be modified to meet specific characterization needs in the tank farms.
- Establish analyses for the radiological release of samples.
- Modification of existing analytical methodologies to fit the targeted analytes and their concentration.

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<sup>1</sup>A glass cylinder containing a small volume (15-25 mL) of absorbing solution through which a vapor stream is bubbled.

To accomplish these objectives, the following project tasks have been identified:

- Publish work plans, quality assurance plans, and sampling and analysis procedures that are both method specific and integrated.
- Validate the sampling/analysis procedures.
- Evaluate validated sampling/analysis procedures.
- Design, fabricate, and install required sampling hardware.
- Perform sampling/analysis Readiness Review, where required.
- Sample tank vapor space.
- Ship required samples to subcontractors.
- Prepare and analyze required samples.
- Verify and report sample analysis results.
- Publish Vapor Conference Committee findings and recommendations.

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### **3.0 PROJECT ORGANIZATION AND RESPONSIBILITY**

The major organizations involved and their responsibilities in the vapor analysis project are identified below:

#### **3.1 WASTE TANK SAFETY PROGRAMS**

- Provides program direction and interfacing with customers such as, U.S. Department of Energy (DOE), Washington State, Environmental Protection Agency (EPA), etc.
- Evaluates results and determines project direction and milestone tracking for internal control
- Performs program integration with other waste tank safety/characterization projects
- Calculates and reports the results of vapor sampling projects.

#### **3.2 INDUSTRIAL HYGIENE AND SAFETY**

- Provides oversight for the health and safety of workers and guidance of the tank sampling per safety requirements.

#### **3.3 WHC PROCESSING AND ANALYTICAL LABORATORIES (PAL)**

- Recommends sampling techniques, procedures, and equipment necessary to ensure the DQOs of sampling.
- Responsible for the in-field activities including equipment assembly and testing, sample collection, chain of custody, sample transport, validation of water vapor and radiological analysis, and release for offsite analysis. Samples will be transported to the PAL, PNL 325, and PNL 329 laboratories. PAL will also have responsibility for refrigerated sample storage, refrigerated sample archiving, sample handling and disposal, procurement of new/modified sampling equipment, and training personnel on the sampler system.
- Responsible for interfacing with Tank Farm Operations & Maintenance to support required sampling equipment fabrication.
- Returns analytical results to Waste Tank Safety Programs point of contact.

### **3.4 325/329 PNL ANALYTICAL LABORATORIES**

- Performs validation studies, sample container preparation (when required), and sample analyses of specified organic and inorganic compounds. Analyses are performed to PNL impact level 2 or 3 criteria, under the the project QA Program.
- Returns analytical results to Waste Tank Safety Programs point of contact.

### **3.5 OREGON GRADUATE INSTITUTE (OGI)**

- Performs method and equipment validation studies per this document.
- Performs method and equipment modifications to analyze selected C-103 organic vapors.
- Performs survey analysis of tank vapors with best available methodologies including estimations of uncertainty.
- Document activities as required by this document (QAPP-013).
- Preparation and certification of SUMMA canisters.
- In cooperation with Sandia NL, return analytical results to Waste Tank Safety Programs point of contact.

### **3.6 TANK FARMS OPERATIONS AND MAINTENANCE**

- Responsible for the safety of in-farm activities including installation of the sampling equipment at the tank, providing approved coordination of the sampling with other in-tank activities, scheduling in-farm activities, and ensuring all documentation is in place for sampling.

### **3.7 HEALTH PHYSICS**

- Provides radiological safety and support for activities in the tank farms.

### **3.8 TWRS QUALITY ASSURANCE**

- Provides program and project support for in-field activity, plus oversight for compliance to requirements for protection of personnel, environment, and facility safety.

- Performs project assessments and documents compliance with controls to meet regulatory requirements. The QA role on in-field instruments used to monitor the gas is to ensure that their use, care, and maintenance (including calibration) comply with WHC requirements and manufacturer information.

### 3.9 ENVIRONMENTAL DIVISION

- Provides point of contact to evaluate Federal, State, and DOE environmental compliance requirements and operational guidelines.
- Submits documentation on the program for compliance to National Environmental Protection Act. Issues site-wide reports for compliance to environmental requirements.

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#### 4.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

##### 4.1 INTRODUCTION

In addition to the usual QA topics that must be addressed in a QAPP per QAMS-005/80 (EPA, 1983), the procedure validation must also be addressed. The following topics must be defined and discussed:

- Accuracy
- Precision
- Validation
- Completeness
- Comparability
- Representativeness.

Chemical analytical data can be broadly grouped into two categories; namely, quantitative, and qualitative. Qualitative data determines the presence or absence of a compound, element, or ion. Qualitative data usually connotes a bias of greater than 50%. Quantitative data defines the amount of a particular species. Modern analytical chemistry is overwhelmingly a quantitative science and will be applied to the tank targeted characterization.

Errors occur in the quantitation process. They may be bias (systematic) and affect accuracy and, if known, and are not too large and consistent, can be used to correct raw data. The errors may be random which affects the precision. Any resulting data will have an uncertainty due to this type of error. These errors can occur in sampling, sample storage or manipulation, and analytical separation and analysis. Systematic and random errors tend to increase with decreasing amounts of analyte.

Accuracy is determined by the analysis of standards. The standards used to determine accuracy shall be prepared and verified independent from those standards used for calibration purposes.

It is the object of this program to determine the vapor concentration of the targeted analytes to a known level of accuracy and precision (certainty). This means that both the random and systematic errors shall be determined, as best as practically possible, with statistical completeness.

The reduction of quantitative data to qualitative data is based on data importance and the size of both types of errors. For example, quantitative data is frequently identified with an error bias of no more than 50%. Bias can be the difference between percent recovery of a known analyte and 100. The concept of bias is different from error in that bias, when sufficiently consistent, allows the correction of raw data for this effect. Qualitative data usually connotes a bias of greater than 50%. Qualitative data allows the report of major, minor, and trace sample components. A major component would account for 10-99% of the composition. A minor component would apply to the 0.1-10% concentration range. Typically a trace component represents 1000 ppm or less. As bias approaches and increases beyond 50%, it generally becomes

less consistent and appears more like random error. In circumstances where the methods of analysis are being developed for complex matrices and/or low level target analytes, data quality parameters, developed from initial samples, shall be reported regardless of bias level.

#### 4.2 MINIMUM QUANTITATIVE LIMITS

The target quantitative limit (TQL) is the desired concentration that may be reliably quantified to meet the needs of this characterization program with regards to vapor toxicity and flammability. TQL's have been established for toxicity related compounds. Initially, this value has been set at  $(0.1) \times [\text{Threshold Limit Value}/8 \text{ hour Time Weighted Average (TLV/TWA) for that component}]$ . The value is chosen to allow a margin of error to compensate for inaccuracies and imprecision in the sample analysis and collection. Table 2A and 2B list the analytes that are currently of highest priority. The tables also show flammability, toxicity, and expected detection limits.

The actual quantitation limits will depend on the vapor characterization technologies used. At present, due to the number and type of compounds to be analyzed, three sample collection techniques will be used. These are (1) SUMMA canister, (2) sorbent tubes, and (3) midjet impingers (or bubblers). Several types of sorbent tubes are needed to collect Hydrogen Cyanide (HCN), Ammonia, and Water (both normal and tritiated) vapors. The choice of sorbent type is dependent on the component(s) of interest. Initially, an impinger collection procedure will be used to sample for HCN and Ammonia. Finally the applicable quantitation limits will depend on the measurement process used. Organics will be separated by gas chromatography (GC) followed by measurement with either flame ionization detection (FID) or mass spectrometry (MS). Furthermore, inorganic components will be measured by gravimetry, ion chromatography (IC), or specific ion electrode (SIE).

#### 4.3 PROCEDURE VALIDATION

Whenever a sampling and/or an analysis procedure is proposed for a new application, there is a chance that the procedure may not provide the same quality data as in a proven application. Different sample matrices can adversely affect sampling and analytical measurement efficiencies. Thus, procedures need to be validated for new applications. This project has adopted the validation procedure used by the National Institute for Occupational Safety and Health (NIOSH). This procedure examines the accuracy and precision values provided by the sampling/analytical procedure for 4-5 concentrations within a specified vapor concentration range for a fixed set of sampling conditions. Ideally, the accuracy and precision values are consistent over the tested concentration range. They all meet prescribed acceptance criteria. NIOSH insists that their procedures, purporting to provide quantitative results, furnish results that are within 25% of the true concentration for 95% of the measurements. To meet this accuracy objective, the precision of the measurements must have a Relative Standard Deviation (RSD) of less than 0.105. To develop the required statistics, a minimum of triplicate samples are run at each concentration level tested.

Table 2-A. Analytes of Concern.

Compound	LFL <sup>a</sup> (ppm)	TLV/TWA <sup>b</sup> (ppm)	MDL <sup>c</sup> (ppm)
Dodecane	-	-	-
Tridecane	-	-	-
Ammonia	16000	25	2.5
NO, N <sub>2</sub> O, and NO <sub>2</sub>	-	-	-
Hexone	14000	50	5.0
Vinyl Acetate	26000	10	10
Methylene Chloride	8100	50	1.0
1,1 Dichloroethane	-	200	20
Tributyl Phosphate	-	-	-
Toluene	12000	100	100
Benzene	14000	0.1	5.0
Aliphatic Nitriles	-	-	-
1-Butanol	14000	50	5.0
Acetone	21500	750	75

<sup>a</sup>LFL = Lower Flammability Limit<sup>b</sup>TLV = Toxicity based on Threshold Limit Value<sup>c</sup>MDL = Method Detection Limit

Table 2-B. Analytes of Interest.

Compounds	LFL (ppm)	TLV/TWA (ppm)	MDL (ppm)
Water	N/A	N/A	N/A
HCN	-	-	-
Carbon Tetrachloride	N/A	5	0.5
Hydrogen	-	-	-
Tritium	N/A	N/A	N/A

Sampling efficiency will be determined separately from analytical measurement efficiency. Sampling efficiency includes both sample collection/sorption efficiency and sample desorption efficiency. The Vapor Issue Resolution Program initially expects a consistent minimum of 75% sampling efficiency. In addition, and where justified, sample results shall be corrected for sampling efficiencies that are less than 90%.

#### 4.4 ACCURACY

SW-846, (EPA, 1986) defines accuracy in terms of the nearness of a measurement to a true value and evaluates accuracy by determining the percent recovery of a reference sample. Although there have been several attempts to provide simulated reference standards for mixed waste samples, a suitable reference standard does not exist. Thus, accuracy must be evaluated differently. For the Vapor Issue Resolution Program, accuracy is evaluated implicitly. By using simple reference standards, relevant analytical measurement systems are proven to be as error-free as is practically possible. This error-free condition of the analytical measurement system is documented for each batch of samples. No vapor samples may be processed unless the analytical measurement system (AMS) has been proven "in control" by providing an acceptable percent recovery of a simple reference standard. AMS control must be documented for each batch of samples. Analytical accuracies for some methods will be ensured by a Method of Standard Addition which ideally eliminates matrix interferences. When used, the spike added must increase the concentration of the selected analytes to a concentration that is (1) approximately double the original analyte concentration and (2) within the linear range of measurement. The minimization of assignable laboratory errors shall be used to control the analytical data error. In the data quality evaluation, the error attributable to matrix interferences shall be reported in terms of a matrix spike analysis.

There is a special QA concern associated with solid sorbent vapor sampling. Unless care in testing design is taken, it is easy to exceed the capacity of a sorbent tube and lose sample due to breakthrough. Because of this each sample in the test array will have a back-up sorbent tube in series with the primary sample tube to prove the adequacy of the test design. The test design is evaluated by measuring the breakthrough of the analyte in the back-up tube relative to the total amount of analyte recovered. It is

expected that no more than 20% of each analyte shall be found in the backup tube. The breakthrough shall be documented as part of the data quality evaluation.

#### 4.5 PRECISION

SW-846, (EPA, 1986) defines precision as the agreement between a set of replicate measurements and evaluates precision using duplicate or replicate sample analysis. The concept of precision acknowledges that several measurements of a single concentration are expected to occur over a range of values. Ideally, the range of values will be narrow - the more narrow the range, the more precise the measurement. As discussed above in Section 4.2, this requires limiting the RSD to about 0.1 or the Relative Percent Difference (RPD) to 20.

Each test design will include at least one replicate sample which will document precision for the test. Some designs require triplicate samples, others require duplicate samples. Where vapor samples are collected at different levels within a tank, replicate samples will be taken for each level. Wherever possible replicate samples will be collected in parallel, i.e., at the same time, or immediately after one another, at the very least.

#### 4.5 COMPLETENESS

The QA requirement of completeness refers to what extent a project delivered all of the analytical data required by the statement of work for a given sampling job. The Vapor Issue Resolution Program will provide an unconditional minimum of 90% completeness. The term "unconditional" means that all conditions contributing to the failure of meeting the completeness objective must be discussed as part of the data quality evaluation.

#### 4.7 REPRESENTATIVENESS

This objective considers and uses sample extraction and sample handling procedures which minimize changes to the composition of the tank vapors between the time of sample extraction and analysis. There are practical considerations that can adversely affect the representativeness of the vapor sample. For instance, horizontal vapor composition variations will not be measured as samples will be extracted from only one riser. Since vertical vapor composition variations are more probable, early vapor samples shall be drawn from three levels in each tank and vertical variations evaluated. Time and turbulence is expected to eliminate any significant layering of the vapor.

Once extracted the vapor composition may change in the sampling medium as a function of time or temperature. This shall be evaluated as part of the method validation studies. Because of these studies, special sample holding times (between sampling and analysis) and/or sample storage conditions may need to be specified. Initially and until validation studies indicate otherwise, holding times will be set at 60 days. All sampling procedures will include provisions for adequate purging of sampling lines. Purging will ensure the sample lines contain only extracted tank vapor.

#### 4.8 COMPARABILITY

The comparability requirement assures that there will be an acceptable, minimum difference between any two organizations providing vapor sampling data from the same source. One way to evaluate comparability is to compare replicate results from the two organizations and accept some maximum RPD calculated from the mean corresponding to the replicate data. The target value for the RPD for the Vapor Sampling project is  $\pm 20$ .

Both PNL and OGI have been given responsibility for organic analytical method development. While there are no current plans to transfer analytical technology to the 222-S Laboratory, acceptable comparability is expected since (1) the program requires complete documentation of procedure validation and instructions, and (2) analytical procedures will be based on existing, standard lab measurement practices, such as Specific Ion Electrode (SIE), Ion Chromatography (IC), etc.

## 5.0 VALIDATION OF PROCEDURES

The validation test procedures must be considered separately for each of the technologies proposed to satisfy the applicable scope of this QAPP. Validation test results may vary with:

- Changes in major components of the vapor
- Changes in sampling technique, i.e., impinger versus sorbent tube or SUMMA canister
- Changes in sampling conditions, i.e., sample flow rate and temperature
- Changes in the way the sample is presented to the measurement system, i.e. as a mixture versus by separated components or as a concentrated volume versus diluted by some carrier gas
- Changes in measurement system, i.e., Ion Chromatography versus Mass Spectrometry or Specific Ion Electrode
- Changes in sample manipulation, i.e., concentration or dilution, etc.

The five technologies to be discussed are as follows:

- Analysis of organic vapors
  - Sample collection by SUMMA canister followed by GC/MS or GC/FID analysis
  - Direct GC/FID or GC/TC analysis
- Analysis of inorganic vapors
  - Ammonia and HCN--Sample collection by midget bubbler followed by SIE or IC analysis
  - Water--Sample collection by sorbent trap (Silica Gel) and analysis by Gravimetry
  - Tritium--Sample collection by sorbent trap (Silica Gel) and analysis by Liquid Scintillation counting

### 5.1 ORGANIC ANALYSIS BY SUMMA CANISTER AND GC/MS OR GC/FID

The sample collection efficiency for SUMMA canisters is 100%, but there are well documented sample desorption problems. It is well known that polar compounds adsorb onto the interior surface of the SUMMA and are not quantitatively released when sample volumes are extracted. Because of this sample desorption efficiency must be addressed in the validation procedure. Another sample desorption problem is that surface adsorbed compounds have been

known to undergo chemical transformations, involving hydration and oxidation, that change the composition of the vapor as a function of time in the canister. This time effect must also be checked as part of the validation process. Because of these sample desorption problems, the required analytical quantitation shall depend on multiple Matrix Spike Addition (MSA) instead of a series of calibration gas mixtures. In this procedure, spikes of chemically representative compounds at increasing levels will be added to determine the required response factors exclusive of any sample matrix effects. Thus, Validation studies shall address the following SUMMA canister sampling and analysis effects:

- Time in the canister
- Temperature of desorption
- Water vapor content of vapor sample
- Sample concentrating methods
- Level of spike addition
- Equipment operating conditions.

## 5.2 ORGANIC ANALYSIS BY DIRECT GC/FID

There are no sample collection/desorption efficiency problems to be addressed in the validation procedure since the sampling of vapors is direct. The required analytical quantitation will depend on either MSA or by a set of simulated, multi-concentration level, vapor calibration standards. Such standards are readily prepared in special plastic sample bags using a single calibration liquid mixture and the appropriate liquid and gas mixture. Validation studies shall address the following analytical effects:

- Water vapor content of vapor sample
- Sample concentrating methods
- Level of spike addition concentration range of standards
- Instrument operating conditions.

## 5.3 AMMONIA AND HCN BY BUBBLER AND SIE

Both of these gases will be absorbed in a special sorbent solution as the vapor sample is passed through a midget impinger, or bubbler. The conditions for sample collection are part of a published NIOSH procedure. The vapor sampling for Ammonia and HCN need not be validated if the NIOSH sampling conditions and procedures are used. The required analytical quantitation will depend on MSA.

## 5.4 WATER BY SILICA SORPTION AND GAVIMETRY

While this is not a NIOSH published procedure, it is well documented. As long as the sampling conditions conform to established limits, no additional validation is required.

## 5.5 TRITIUM BY SILICA SORPTION AND LIQUID SCINTILATION

While this is not a NIOSH published procedure, it has been validated by the 222-S Laboratory. As long as the sampling conditions conform to established limits, no additional validation is required.

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## 6.0 SAMPLING PROCEDURES AND SAMPLE PREPARATION

### 6.1 INTRODUCTION

This section of the QAPP concerns the QA aspects of two different sampling configurations: (1) a vapor/aerosol sampling using a portable sample collection device and an in-tank bubbler array or SUMMA sample tube and (2) a mobile sampling vehicle, designated DML1, capable of collecting SUMMA, solid sorbent or bubbler samples at up to three tank vapor levels. Included in this document is the requirement for procedures that describe the specific sampling techniques outlined in Section 2.0. These procedures shall be subject to revision as knowledge and experience with the techniques is gained over time. Procedural guidance is available from NIOSH publications and ASTM ambient air sampling procedures, such as ASTM D 1357 (ASTM, 1989) and D 3614 (ASTM, 1990).

Sampling procedures will document both the pre- and post-sampling activities. The following sampling activities will be addressed for each of the sampling technologies applicable to this QAPP:

- Cleaning and cleanliness testing of SUMMA canisters
- Reagent preparation and filling of solid sorbent tubes
- Reagent preparation and filling of bubblers
- Cleaning and cleanliness testing of sampling tubing
- Vacuum testing of sampling system
- Applicable sample holding time limit prior to analysis
- Sample line flushing, testing and flushing acceptance criteria
- Sample data recording and calculation requirements
- Field and reagent blank preparation
- Set-up, sample collection, and shut-down operation of portable sample collection device
- Set-up, sample collection, and shut-down operation of DML1
- Post-sample handling, chain-of-custody activities, and sample radioactivity limit verification/documentation for all sampling events.

### 6.2 CHAIN-OF-CUSTODY

As noted above, sample custody will be verified from the sample collection in the field through receipt of the samples by using WHC approved chain-of-custody forms. These forms record the identity of persons in custody of the samples, sample identity, and time and date of transfer of custody.

The form shall accompany the sample while it is being transported. To verify custody, samples shall be within sight of the persons assigned custody unless the samples are contained in a locked or secured area.

### 6.3 FIELD BLANKS

A final sampling related QA concern must be addressed - field blanks. Field blanks document the absence of sample contamination from sample containers or reagents brought to the field. Every test plan will include at least one unexposed, composite Reagent Blank for each set of vapor samples. The term "composite" refers to the following three potential sources of contamination. A set of samples are those samples required by both the work plan and this QAPP for a given tank. The Reagent Blank is unexposed in the field. The purpose of the Reagent Blank is to demonstrate that the sorbent media used in the sampling devices is not contaminated (1) as received from the supplier, (2) by the sample container, (3) or by handling during preparation or field activities. The Reagent Blanks must be unexposed to eliminate sources of contamination either from the sample vapor or the sampling location ambient air. The Reagent Blanks for liquid sorbent media are analyzed directly without extraction. The Reagent Blank for solid sorbent media are analyzed after extraction. Thus, the expected analytical results shall be either (1) below the method detection limit for the analyte in question or (2) below some statistically derived maximum concentration. The Reagent Blank for SUMMA canisters shall also be unexposed in the field. The laboratory will be filled with moist zero gas (MZG) prior to analysis. See procedure T014 (EPA, 1988) for the definition of MZG. The maximum allowed limits for the Reagent Blank shall be statistically derived. Each reporting laboratory is required to develop, document, and apply acceptance criteria to the Reagent Blank.

## 7.0 CALIBRATION PROCEDURES AND FREQUENCY

This section addresses the QA requirements for the calibration of vapor sampling and analysis equipment. The relevant equipment includes (1) sampling temperature and pressure gages and (2) analytical measurement systems. In general all calibrations must be verified as current before sample analysis can be performed. All equipment requiring calibration will be uniquely identified and log books, or other suitable documentation, will be maintained to record calibration data and, where appropriate, the next required calibration date. Calibration frequencies for analytical measurement systems that differ from the manufacturer's recommendations will be determined by a statistical analysis of calibration check sample results. As long as check standards are within the pre-established acceptance limits, the previous calibration is valid. Reporting laboratories are required to develop, document, and apply acceptance criteria to calibration check results. Any time the system configuration changes for maintenance, the system calibration will be repeated and documented.

Procedures T012 (EPA, 1988) and T014 (EPA, 1988) provide useful guidance for GC/MS and GC/FID calibration information. Applicable calibration, calibration checking, and calculation procedure guidance is also contained in ASTM (ASTM, 1986) and NIOSH publications such as (Taylor, Kupel, 1977), (Busch, Taylor, 1981).

The purity of water and reagents for standards and working solutions should be described in standard operating procedures, or as part of the analysis report. Data producers are required to purchase standards traceable to the National Institute of Science and Technology (NIST), where possible, and to have on file certificates of traceability, where available.

Standards from commercial sources shall be replaced no later than the expiration date supplied by the manufacturer. Reporting laboratories shall develop, document, and apply shelf life and storage limitations to all calibration standards.

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## 8.0 ANALYTICAL PROCEDURES

The analysis of all vapor samples within the scope of this QAPP shall be implemented in accordance with the procedures written and approved by the laboratory performing the analysis. These procedures shall meet the following general content requirements:

- Scope
- Applicable reference documents
- Method summary
- Applicability
- Interferences
- Required equipment
- Reagents and materials
- Safety precautions
- Calibration and standardization
- Analysis procedure steps
- Calculations
- Bias (from validation study) and Precision.

Laboratories and sampling operations shall maintain a list of all current procedures, either temporary or permanent, that are being used for the sampling analysis of tank vapor. Temporary procedures need not be detailed or formalized. Project reports will contain notification of procedures revised during the sampling and/or analysis of related vapor samples.

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## 9.0 DATA REDUCTION, VALIDATION, AND REPORTING

### 9.1 DATA REDUCTION

All calculations and unit conversions are confirmed by recomputation and/or review by a second, technically knowledgeable person who did not generate the data. No blank corrections, averaging of duplicates, or other manipulation of data are performed in the laboratory. Any raw laboratory data which is converted from instrument readings to concentration units, by comparison to a standard curve, is accompanied by the standard curve analysis and correlation coefficient, if used. The concentration of standard solutions and instrument readouts are correlated to the desired final report concentration units, so that a minimum of unit conversions is necessary.

Data transcriptions are reviewed and confirmed by the responsible scientist in the laboratory and/or by the manager prior to release of the final data report.

### 9.2 DATA VALIDATION

Laboratory data validation is a continuous process performed at each step of the analytical method. The analyst, responsible scientist, manager, and QA officer all participate in the data validation. The analytical data validation process starts with the verification of sample receipt and continues through approval of the final report. The general steps utilized in laboratory data validation for this project, and the responsible staff, are shown in Table 3.

The validated data report that the laboratory supplies at the completion of the sample analysis will include the information from the validation review as well as the analysis results.

### 9.3 DATA REPORTING

The report package is prepared after all laboratory analytical data has been compiled and validated. The composition of the complete data package depends on the activities assigned to the reporting unit. The original of the data package is retained by the reporting unit and a legible copy is sent to the data requestor. The composition of the data package shall include the following, as applicable:

- Chain-of-custody records and any field sampling observations and data sheets
- Summary of all non-QC sample analysis results including:
  - Sample identification
  - ppmv analyte concentration

Table 3. Laboratory Data Validation Steps.

Validation Procedure	Responsible Individual
Confirm sample identity	Analyst and scientist
Perform instrument calibration prior to analytical run	Analyst
Perform analysis of check standard and blank	Analyst
Confirms method is in control prior to analyses of samples	Analyst and scientist
Confirm method is in control periodically during long run	Analyst and scientist
For historically unstable analytical procedures, confirm method is in control at end of run, perform initial evaluation of duplicate (or matrix spike duplicate) and matrix spike and initiate re-runs as required	Analyst and scientist
Confirm data calculations and conversions	Scientist
Review data for transcription errors	Scientist and QA officer
Review accuracy, precision, completeness, holding times, detection limits, units and significant figures	Scientist and QA officer
Evaluate data for outliers	Scientist and QA officer

- Data quality report that includes the following:
  - Average matrix spike recovery for each analyte reported
  - Total analytical variance expressed as a relative standard deviation
  - Total spatial variance expressed as a relative standard deviation (where applicable)
  - Total variance of the mean expressed as a relative standard deviation

- Analyte breakthrough report
- Completeness report.
- QA/QC report confirming that:
  - Recovery of calibration check standards corresponding to reported data met established acceptance criteria.
  - Fresh calibration check standards were prepared at a frequency consistent with established criteria.
  - Calibration check standard chemicals were stored under conditions consistent with established criteria.
  - Recovery of lab control standards corresponding to reported data met established acceptance criteria.
  - Fresh lab control standards were prepared at a frequency consistent with established criteria.
  - Lab control standards chemicals were stored under conditions consistent with established criteria.
  - Blank(s) analyses met established acceptance criteria.
  - Sampling, analysis, calculations and method detection limits were performed or determined by existing (temporary or permanent) written procedures.
- Report explaining necessary procedure deviations and analytical problems
- Report listing method detection limit for each reported analyte

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## 10.0 INTERNAL QUALITY CHECKS AND CORRECTIVE ACTION

### 10.1 INTRODUCTION

Internal quality checks will be performed on the sampling and analytical systems by preparing and analyzing special samples as specified in Section 4.0 of this QAPP. These samples include field blanks, calibration checks, duplicates (or matrix spike duplicates), and matrix spikes. To promote a consistent interpretation of QC sample terminology, Table 4 lists the QC sample definitions used in this QAPP. The analytical results of these samples determine the quality of the vapor sampling results. Each set of vapor samples will have a sub-set of corresponding QC samples. The evaluation of these QC sample results determine the quality of the corresponding vapor sample results. Thus, vapor sample results may be evaluated as "quantitative" measurements or "qualitative" estimates. The vapor sample results, whose QC samples satisfy established acceptance criteria, are considered "quantitative."

### 10.2 ACCURACY RELATED QC CRITERIA

As noted above in Section 4.0 of this QAPP, prior to the analysis of any vapor sample, the analytical measurement system must be confirmed to be "in control." This is demonstrated by verifying that the analytical measurement system responds as expected to (1) lab control standard and (2) a typical calibration standard. The lab control standard is prepared in deionized (DI) water, or other suitable solvent matrix, and has no expected sample matrix interferences. The inability to obtain an acceptable recovery from this standard strongly indicates a serious problem with the analytical measurement system. Any problem(s) must be investigated and resolved prior to using the analytical measurement system to measure vapor samples. The second check standard verifies that the initial, or last, calibration is still valid. Failure to confirm the calibration status, requires recalibration of the instrument prior to quantitative analysis of vapor samples.

If the analytical measurement system can be shown to be "in control," the matrix spike recovery can be used as a measure of the method accuracy.

### 10.3 PRECISION RELATED QC CRITERIA

Data precision is the second half of data quality evaluation. Where vapor samples are extracted from several levels within the tank vapor space, replicate, even duplicate, sample data can be used to calculate (1) the total analytical variance, (2) the total spatial variance and (3) the total variance of the mean. The analytical variance is related to all the processes that take place in the field and laboratory. The spatial variance is related to any existing vapor layering in the tank. The variance of the mean is related to the combined effect of both the analytical and spatial variances. These variances need to be calculated for each reported analyte.

Table 4. QC Sample Definitions.

QC Sample Name	Definition
Reagent Blank	This is an artificial sample designed to confirm the absence of contamination introduced from the sampling container and the sampling medium. The sample is unexposed to tank vapors but is field handled during sample labeling activities. SUMMA canisters shall be filled with moist zero air prior to analysis. Each reporting lab is required to develop, document, and apply statistical acceptance criteria for the evaluation of this special sample.
Preparation Blank	This is an artificial sample designed to confirm the absence of contamination from sample preparation chemicals, i.e., solvents, reagents, etc. This sample is prepared by the analyzing laboratory and is required to develop, document, and apply statistical acceptance criteria for the evaluation of this special sample.
Field Surrogate	This is an artificial sample designed to determine the effect of time and sample storage conditions on analyte concentration once extracted from the tank vapor. The sample is prepared by the analyzing laboratory by adding a concentration of analyte to the absorbing medium and determining the recovery of the added analyte when the samples return from the field. Consistent recovery data can be used to correct calculated results.
Calibration Check Standard	This is calibration solution or gas mixture that is designed to verify that the instrument response factor for a given analyte has not changed significantly since the last complete calibration. Each reporting lab is required to develop, document, and apply statistical acceptance criteria for the evaluation of this special sample. Failure to meet the acceptance criteria requires a complete recalibration.
Lab Control Standard	This is an artificial sample designed to confirm that the analytical measurement system is "in control", i.e., can accurately measure a relevant analyte in a simple solution or gas mixture free of matrix interferences. Typically, these solutions contain one or a few analytes in a common solvent (DI Water, Methanol, air, etc.). Each reporting lab is required to develop, document and apply statistical acceptance criteria for the evaluation of this special sample. Failure to meet the acceptance criteria requires a shut down of the analytical measurement system for troubleshooting and/or repairs.
Matrix Spike	This is an artificial sample designed to measure the analytical error due to matrix interferences. The sample is prepared by adding a known amount of analyte to an aliquot of a field sample. The percent recovery of the added analyte is determined and reported. By controlling the non-matrix error (See above), the percent recovery of this special sample is used as a measure of the method accuracy.
Matrix Spike Duplicate	This is an artificial sample designed to measure the method precision. This sample is a duplicate of the matrix spike sample. A comparison of the two matrix spike percent recovery values provides the required evaluation of method precision.

#### 10.4 CONTAMINATION RELATED QC CRITERIA

Field and Preparation Blanks are prepared and analyzed to evaluate the presence of contamination from incompletely cleaned sample containers or from improperly prepared reagents. See Section 6.3 for details. By verifying the absence of contamination, via acceptable Field and Preparation Blanks, the use of Matrix Spike Recovery as a measure of method accuracy is further justified.

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## 11.0 PERFORMANCE AND SYSTEM AUDITS

Vapor sampling will be performed according to the activities found in WHC-SD-WM-WP-174 (WHC, 1992). Tank Farms Environmental or Process Engineering and QC shall have personnel periodically in the field to ensure that the sampling is performed per the procedures written as part of the experience gained in Phase 1 of this project. In addition, QA personnel may conduct additional surveillance activities for both on and offsite laboratories in accordance with CM-4-2 (WHC, 1988). The purpose of these surveillances will be to verify conformance to unreported QAPP requirements and performance according to established procedures.

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## 12.0 PREVENTATIVE MAINTENANCE

The most significant pieces of equipment involved in this effort are the gas chromatographs, the mass spectrometer, and the sampling system purge pump. The rest of the onsite equipment consists mainly of hot water heated sample tubing, electrically operated valves, calibrated gages and meters, and other equipment which can be easily repaired, fabricated, or obtained. Groups responsible for major equipment systems will develop a spare parts list and maintain the listed spares. A 48-hour availability for these spares is required.

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### 13.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quality assurance will be documented in the data report. This data will meet all data quality objectives presented in this QAPP or it will be qualified with an accompanying explanation of any variances.

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