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Document Number:	WHC-SD-WM-TP-317, REV 0
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1. Total Pages 29

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TANK 241-S-111 TANK CHARACTERIZATION PLAN	WHC-SD-WM-TP-317	0

5. Key Words

CHARACTERIZATION, DQO, HEALTH AND SAFETY VAPOR ISSUE, FLAMMABLE, ORGANIC, QUALITY CONTROL, SINGLE-SHELL TANK, VAPOR SAMPLING, ANALYSIS, TANK CHARACTERIZATION PLAN

6. Author

Name: C. S. HOMI

Signature 3/6/95

Organization/Charge Code 71520/N4168

7. Abstract

This document is a plan which serves as the contractual agreement between the Characterization Program, Sampling Operations, Oak Ridge National Laboratory, and PNL tank vapor program. The scope of this plan is to provide guidance for the sampling and analysis of vapor samples from tank 241-S-111.

8. RELEASE STAMP

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WHC-SD-WM-TP-317
Revision 0

Tank 241-S-111
Tank Characterization Plan

Prepared for the U.S. Department of Energy
Office of Environmental Restoration
and Waste Management

by

Los Alamos Technical Associates
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Kennewick, Washington 99336

DLC

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LIST OF ABBREVIATIONS

DQO	Data Quality Objective
DST	Double-Shell Tank
HEPA	High Efficiency Particulate
NCPLX	Non-Complexed Waste
RCRA	*Resource Conservation and Recovery Act of 1976*
S-111	Tank 241-S-111
SST	Single-Shell Tank
TWRS	Tank Waste Remediation System
USQ	Unreviewed Safety Question
WHC	Westinghouse Hanford Company

1.0 INTRODUCTION

The Defense Nuclear Facilities Safety Board (DNFSB) has advised the U.S. Department of Energy (DOE) to concentrate the near term sampling and analysis activities on identification and resolution of safety issues (Conway 1993). The data quality objective (DQO) process was chosen as a tool to identify sampling and analytical needs for the resolution of safety issues. As a result, a revision in the Federal Facility Agreement and Consent Order (Tri-Party Agreement) milestone M-44 has been made, which states that "A Tank Characterization Plan (TCP) will be developed for each double-shell tank (DST) and single-shell tank (SST) using the DQO process ... Development of TCPs by the DQO process is intended to allow users (e.g., Hanford Facility user groups, regulators) to ensure their needs will be met and that resources are devoted to gaining only necessary information" (Ecology et al. 1994). This document satisfies that requirement for tank 241-S-111 (S-111) sampling activities.

2.0 DATA QUALITY OBJECTIVES APPLICABLE TO TANK S-111

The sampling and analytical needs associated with the Hanford Site underground storage tanks on one or more of the four Watch Lists (ferrocyanide, organic, flammable gas, and high heat) and the safety screening of all 177 tanks have been identified through the DQO process. A DQO identifies the information needed by a program group concerned with safety issues, regulatory requirements, tank waste processing, or the transport of tank waste. The DQOs that have been completed and apply to tank S-111 are discussed in the following paragraphs.

Both Watch List and non Watch List tanks will be sampled and evaluated to classify waste tanks in one of three categories (SAFE, CONDITIONALLY SAFE, or UNSAFE). The safety issue DQOs identify the guidelines to determine to which classification a tank belongs based on analyses that indicate if certain measurements are within established parameters. If a specified parameter is exceeded, further analyses may be required to classify a tank. A tank can be removed from a Watch List if it is classified as SAFE. Tank S-111 is on the Organic and the Flammable Gas Watch List.

2.1 SAFETY SCREENING DATA QUALITY OBJECTIVES

The *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994) describes the sampling and analytical requirements that are used to screen waste tanks for unidentified safety issues. This DQO requires that a vertical profile of the tank waste be obtained from at least two widely spaced risers. This vertical profile may be obtained using core, auger, or grab samples. The primary analytical requirements for the safety screening of a tank are energetics, total alpha activity, moisture content, and flammable gas concentration. The safety screening analyses shall be applied to all core samples, DST Resource Conservation and Recovery Act (RCRA) samples, and all auger samples, except those taken exclusively to assess the flammable gas crust burn issue.

2.2 WASTE COMPATIBILITY DATA QUALITY OBJECTIVES

The *Data Quality Objectives for the Waste Compatibility Program* (Carothers 1994) identifies four safety-related decision elements (criticality, flammable gas accumulation, energetics, and corrosivity), needed to determine potential incompatibility of wastes that may occur from routine waste transfers into and within a DST. A routine transfer has the appropriate historical data necessary for the Waste Compatibility Program to determine the acceptability of the transfer from an engineering process control perspective.

Four operations-related decision elements have been identified for a non-routine transfer; separation of transuranic waste from non-transuranic waste, limits on heat generation, segregation of complexant waste, and ensuring pumping system capabilities. A non-routine transfer includes waste that has unique chemical and/or physical properties for which no historical data exists to judge compatibility with safety and operations decision rules.

2.3 FUGITIVE VAPOR EMISSION DATA QUALITY OBJECTIVES

The Tank Vapor Issue Resolution Program was initiated in 1992 to resolve the health and safety issues associated with the high level waste tanks at the Hanford site. The two main issues related to this program are 1) an insufficient understanding of reported exposures of tank farm personnel to unacceptable levels of noxious vapors and 2) the risks to worker health and safety can not be determined until the vapors in the waste tanks are well characterized. Westinghouse Hanford Company (WHC) standard safety practices dictate that any flammable components in the headspace of any Watch List tank must be determined and quantified before intrusive work can be conducted on these tanks. The DQO applicable to head space vapor sampling is *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issue Resolution* (Osborne et al. 1994).

A nitrogen gas purge will be used to clear and cool the drill bit during rotary core sampling. This purge gas exhausts into the waste tank head space resulting in an uncontrolled release of pollutants. A portable modular unit has been developed to exhaust the tank head space during rotary core sampling. This modular unit will remove airborne particles through high efficiency particulate (HEPA) filters, but is not designed or equipped to treat or remove toxic vapors. It is equipped with instruments to monitor and alarm for total organic carbon (TOC) and ammonia vapors. The tank head space must be characterized to confirm that the modular unit can be safely started and to establish acceptable TOC and ammonia levels for safe operation. The applicable DQO for rotary core sampling is the *Rotary Sampling Core Vapor Sampling Data Quality Objective* (Price 1994)

2.4 ORGANIC SAFETY ISSUE DATA QUALITY OBJECTIVES

The *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al. 1994) focuses on SSTs that contain or may contain, based on safety screening, organic compounds above the established decision thresholds. It identifies the information needed to determine if tanks should be added, removed or remain on the Organic Watch List. The sampling and analyses prescribed by this DQO will allow tanks to be classified as safe, conditionally safe, or unsafe. This DQO requires that at least two core samples be taken from risers separated radially to the maximum extent possible by the existing risers.

The primary analytical requirements for the organic DQO are energetics, moisture content, total organic carbon, and the presence of a free organic liquid phase.

2.5 FLAMMABLE GAS SAFETY ISSUE DATA QUALITY OBJECTIVES

Flammable Gas Tank Safety Program: Data Requirements for the Core Sample Analysis Developed Through the Data Quality Objectives (DQO) Process (McDuffie and Johnson 1994) focuses on DSTs that contain or may contain, based on safety screening analyses, flammable gases above the established decision thresholds. It concluded that the most reliable information is obtained from one complete core and summarizes the analytical needs for the core sampling activities of the Flammable Gas Watch List tanks. Data from these core samples are needed to provide an understanding of the tank contents so that 1) insight may be obtained on the mechanisms for gas generation, retention and release, 2) models of the waste behavior can be developed to support safety analysis and development of mitigation methods, and 3) modeling of the flow of gases, and potential for ignition, can be done to support hazard analyses.

3.0 TANK AND WASTE INFORMATION

This section summarizes the available historical information on tank S-111. Included are the age of the tank, process history, and the expected contents of the tank based on the latest information. The fill history information is available in *A History of the 200 Area Tank Farms* (Anderson 1990).

3.1 1995 TANK STATUS

Tank S-111 is classified as sound with respect to tank integrity and is passively ventilated. The tank was primary stabilized in March 1978 and partial interim isolation was completed in december 1982. Approximately 2,260,000 liters (596,000 gallons) of waste is contained in the tank. The waste consists of 37,800 liters (10,000 gallons) of supernatant, 1,690,000 liters (447,000 gallons) of saltcake, and 526,000 liters (139,000 gallons) of sludge; of which 738,000 liters (195,000 gallons) is drainable interstitial liquid (Hanlon 1995). This volume of waste corresponds to a depth of 569 centimeters (224 inches). The highest temperature as of December 1994 is 34C (93F) (Hanlon 1995).

3.2 TANK CONFIGURATION

Single shell tank S-111 was constructed as a type III tank between 1950 and 1951 and is located in the 200 West Area. The tank is 23 meters (75 feet) in diameter and has a design capacity of 2,870,000 liters (758,000 gallons); however, safety consideration require an maximum operating capacity of 2,840,000 liters (750,000 gallons). Tank S-111 is second in a cascade flow series consisting of tanks 241-S-110, S-111, and 241-S-112. A cascade flow system consists of tanks connected in series by pipes. When the primary tank in the system became full, the waste would then flow to the secondary tanks in the system. Tank S-111 has 12 risers and two 31 centimeter (12 inch) risers, #6 and #8, are available for use.

3.3 AGE AND PROCESS HISTORY

Single-shell tank S-111 was constructed between 1950 and 1951. Placed into service in 1952, tank S-111 received waste from the REDOX Plant from the third quarter of 1952 until the fourth quarter of 1973 (Brevick 1994). During the first quarter of 1974, 1,720,000 liters (456,000 gallons) were transferred to tank 241-S-102 and 7,600 liters (2000 gallons) were transferred to tank 241-S-101. The tank received bottoms and recycle streams from the 242-S Evaporator/Crystallizer from the first quarter of 1974 until the tank was salt-filled in the third quarter of 1975. The tank was removed from service in the second quarter of 1976. During the following quarter it was labelled inactive and salt-well pumped. Tank S-111 was primary stabilized in March 1978 and partially isolated in December 1982. The tank contained partial neutralized feed waste from the first quarter of 1978 until the third quarter of 1980. The waste has been classified as non-complexed since the fourth quarter of 1980. Figure 3-1 summarizes the influx and effluent history of tank S-111 (Anderson 1990, Agnew 1994).

3.4 EXPECTED TANK CONTENTS

Tank S-111 is expected to have three primary layers. A bottom layer consisting of REDOX waste, cladding waste from the REDOX process, and unknown waste, followed by a layer of S1 saltcake, and a top layer of supernatant. The last solids update was obtained April 28, 1982, and the last photo was taken August 10, 1989 (Hanlon 1995). The photograph shows a surface that is one-third to one-half covered with solid material that appears to be floating on a liquid surface. An estimated inventory based on historical sample and analysis data is shown in Table 3-1 (Brevick 1994).

Figure 3-1: Fill History for Tank 241-S-111

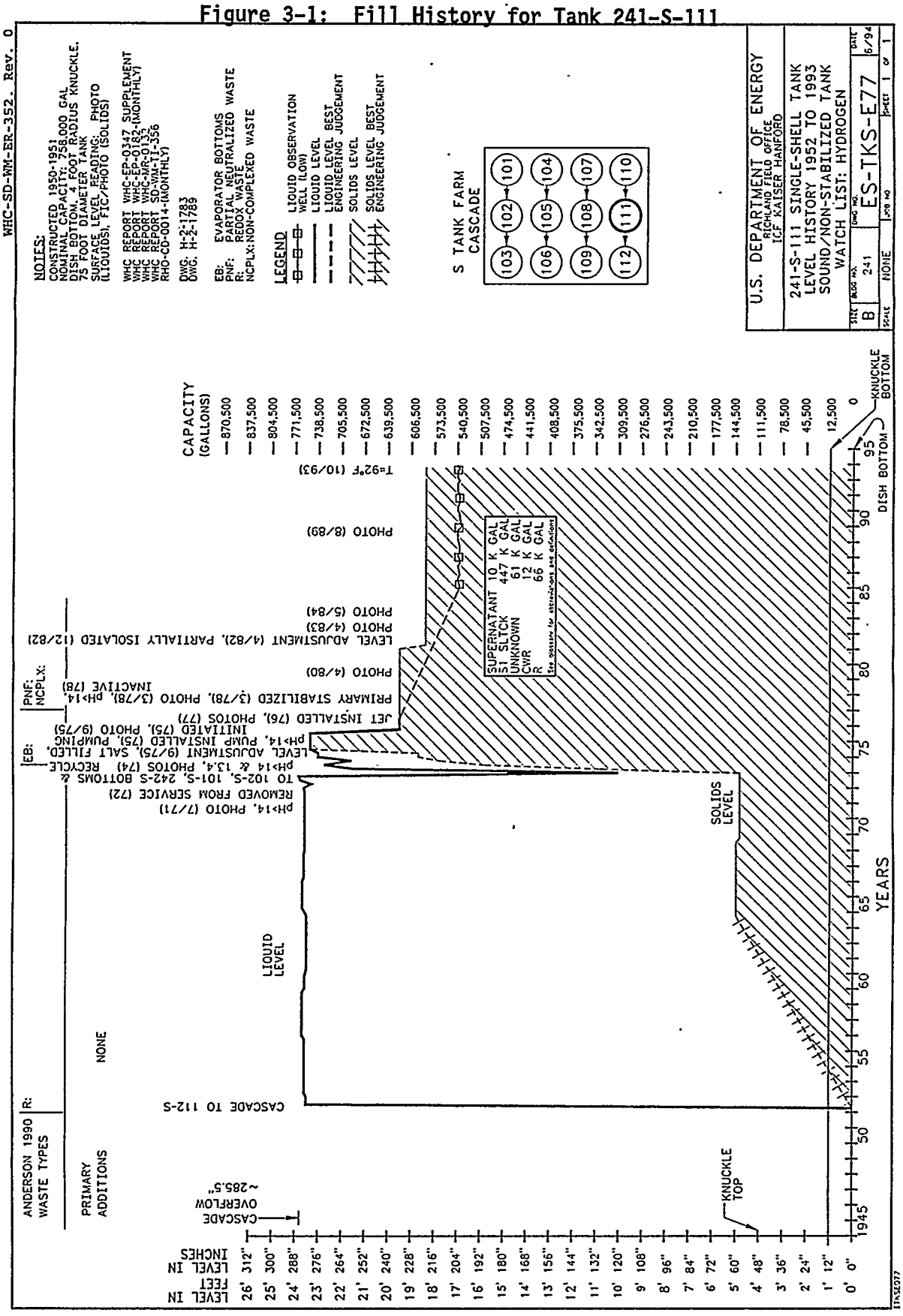

Table 3-1: Tank S-111 Solids Composite Inventory Estimate

Physical Properties			
Total Solid Waste	Mass = 3.29E+06 kg; Volume = 1,990 kL (525 kgal)		
Heat Load	4.92 kW (1.68E+04 BTU/hr)		
Bulk Density	1.66 (g/cm^3)		
Void Fraction	0.52		
Water wt%	26.08		
TOC wt% C (wet)	0.03		
Chemical Constituents	moles/L	µg/g	kg
Na^+	12.03	1.67E+05	5.50E+05
Al^{3+}	1.02	1.67E+04	5.49E+04
Fe^{3+}	1.33E-02	4.50E+02	1.48E+03
Cr^{3+}	7.73E-02	2.43E+03	7.99E+03
Ni^{2+}	4.97E-03	1.76E+02	5.80E+02
Mn^{4+}	0.13	4.45E+03	1.47E+04
K^+	7.79E-03	1.84E+02	6.05E+02
OH^-	4.11	4.22E+04	1.39E+05
NO_3^-	8.56	3.20E+05	1.05E+06
NO_2^-	0.11	3.02E+03	9.93E+03
CO_3^{2-}	0.58	2.12E+04	6.97E+04
PO_4^{3-}	0.49	2.80E+04	9.21E+04
SO_4^{2-}	0.28	1.63E+04	5.37E+04
F^-	9.03E-02	1.04E+03	3.41E+03
Cl^-	6.64E-02	1.42E+02	4.68E+02
$C_6H_5O_7^{3-}$	3.81E-03	4.35E+02	1.43E+03
$HEDTA^{3-}$	4.42E-06	0.73	2.41
$glycolate^-$	6.72E-03	3.04E+02	1.00E+03
$acetate^-$	4.26E-03	1.52E+02	5.00E+02
Radiological Constituents			
Pu	---	7.09E-02 (µCi/g)	3.89 (kg)
U	2.64E-02 (M)	3.8E+03 (µg/g)	1.25E+04 (kg)
Cs	0.32 (Ci/L)	1.95E+02 (µg/g)	6.43E+05 (Ci)
Sr	0.14 (Ci/L)	85.88 (µg/g)	2.83E+05 (Ci)

4.0 TANK S-111 SCHEDULED SAMPLING EVENTS

Three sampling events for tank S-111 are currently scheduled: a vapor sample in April 1995, a grab sample in April 1995, and a push core sample in November 1995. No other sampling is scheduled through Fiscal Year 1997 (Stanton 1995). The vapor sampling shall be performed in accordance with the two DQOs dealing with fugitive vapor emissions: *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issue Resolution* (Osborne et al. 1994) and *Rotary Sampling Core Vapor Sampling Data Quality Objective* (Price 1994). The grab sampling shall be conducted in accordance with *Data Quality Objectives for the Waste Compatibility Program* (Carothers 1994). Core sampling shall be conducted following the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994), *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al. 1994) and *Flammable Gas Tank Safety Program: Data Requirements for Core Sample Analysis Developed Through the Data Quality Objectives (DQO) Process* (McDuffie and Johnson 1994). Sampling and analytical requirements from these DQOs are identified in Table 4-1. A complete list of analytical requirements is given, as an appended revision, in the appropriate Sampling and Analysis Plan (SAP).

Table 4-1: Integrated DQO Requirements

Sampling Event	Applicable DQOs	Sampling Requirements	Analytical Requirements
Vapor	▸ Generic In-Tank Vapor Issue Resolution DQO ▸ Rotary Core Vapor Sampling DQO	3 SUMMA® canisters 12 Triple Sorbent Traps 6 Sorbent Trap Systems	Gas Flammability Gas Toxicity -Organic Vapors -Permanent Gases
Grab	▸ Compatibility DQO	3 grab samples	Energetics, Moisture, Major Anions, Cations & Radionuclides, SpG & pH, Separable Organics
Push	▸ Safety Screening DQO ▸ Flammable Gas DQO ▸ Organic DQO	2 cores from risers separated radially to the maximum extent possible	Energetics, TOC, Total Alpha, Moisture, Gas Composition, Major Anions & Cations, Radionuclides, Primary Organics & Organic Products, Physical Properties

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APPENDIX A

SAMPLING AND ANALYSIS PLAN FOR VAPOR SAMPLING IN FISCAL YEAR 1995

Tank 241-S-111

Prepared for the U.S. Department of Energy
Office of Environmental Restoration
and Waste Management

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LIST OF ACRONYMS FOR APPENDIX A

CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CGM	Combustible Gas Meter
DOT	Department of Transportation
DQO	Data Quality Objective
ECN	Engineering Change Notice
EPA	Environmental Protection Agency
ESH&QA	Environmental Safety, Health, and Quality Assurance
GC/MS	Gas Chromatography/Mass Spectrometry
HEPA	High-Efficiency Particulates Air Filters
IC	Ion Chromatography
IDLH	Immediately Dangerous to Life and Health
ISS	In Situ Sampling
LFL	Lower Flammability Limit
ORNL	Oak Ridge National Laboratory
PNL	Pacific Northwest Laboratory
ppbv	parts per billion by volume
ppmv	parts per million by volume
QA	quality assurance
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act of 1976
SAP	Sampling and Analysis Plan
SML	Sampling and Mobile Laboratories
S-111	Tank 241-S-111
SUMMA®	registered trademark for passivated stainless steel canister
TCP	Tank Characterization Plan
TRP	Toxicology Review Panel
TO-14	EPA Toxic Organic Protocol 14
TOC	Total Organic Carbon
TST	Triple Sorbent Trap
TWAP	Tank Waste Analysis Plan
TWRS	Tank Waste Remediation System
VSS	Vapor Sampling System
WHC	Westinghouse Hanford Company

A1.0 INTRODUCTION

Tank 241-S-111 (S-111) is scheduled to be sampled for flammability and toxicity of vapor in the headspace of the tank. This Appendix A is intended to address only the scheduled vapor sampling and analysis for the fiscal year 1995. Present vapor sampling systems include Type 2 (In Situ Sampling, or ISS) and Type 3 (the Vapor Sampling System, or VSS). These two sampling systems are operated by Sampling and Mobile Laboratories (SML). Both the VSS and ISS collect condensible and non-condensible gases from the tank, they just do it differently. The VSS used a heated vapor probe and collects the sample out of the tank. The ISS lowers the sorbents and TST into the tank headspace for the condensible gases and collects SUMMA®s out of the tank for permanent gases.

A2.0 SAMPLING AND ANALYSIS SCHEME

The following sections provide the methodology and procedures to be used in the preparation, retrieval, transport, analysis, and reporting of results from vapor samples retrieved from tank S-111. The requirements for this sample event, contained within this appendix of the TCP, are within the scope of work specified in the appropriate laboratory work authorizing documents. Any decisions, observations, or deviations to this sampling and analysis plan made during sample receipt, preparation and analysis shall be documented in controlled notebooks and justified in the deliverable report. The general sampling and analysis scheme for Type 3 vapor sampling is presented as a flowchart and narrative in Figure A-1.

A2.1 VAPOR SAMPLING EVENT

The responsibilities of Sampling and Mobile Laboratories (SML) to this sampling event are given in this section. For detailed information regarding applicable operating procedures for the tank S-111 vapor sampling activity refer to work package WS-95-00038. Additional quality control and deliverable requirements are given in Sections A3.0 and A6.0. of this appendix.

A2.1.1 Flammability Test

Prior to this sampling event and any intrusive work on this tank, an assessment of the flammability of the tank head space gases is required by WHC safety practices. The flammability test is performed by Industrial Hygiene Field Services using a combustible gas meter (CGM). The procedure for this flammability test is included in the sampling event work package.

- If the tank vapor fuel content is greater than or equal to 20% of the lower flammability limit (LFL) under steady state conditions, then all sampling activities must stop until further authorization is given by management.

- If CGM measures a total fuel content between 10% and 20% of the LFL, vapor sampling activity may continue under CGM monitoring to better identify the hazard level.

- If the concentration is below 10% of the LFL, the tank is not considered have a flammability hazard and all sampling work can proceed (Osborne 1994).

A2.1.2 Sample Collection Using SUMMA® Canisters And Sorbent Tubes

SML shall provide sample identification numbers to the laboratories according to the format given in Section A3.1. SML shall use labeled sample containers supplied by the laboratory (see Section A2.2.1, Preparation of Sample Media Containers) to collect vapor samples. The VSS shall be used to collect vapor from tank S-111 in accordance with laboratory operating procedure WHC-IP-1127(4.5) "Collection of SUMMA® Canisters and Sorbent Tube Sampling Using the Vapor Sampling System (VSS)". The sample type, type of collection media to be used, and the number of samples requested are given in Table A-1.

Table A-1. General Sampling Information for Tank S-111.

Sample Container	Prepared By	Preparation Procedure	Sample Type	Number of Samples
SUMMA® Canisters	PNL	PNL-TVP-02	Tank Air	3
SUMMA® Canisters	PNL	PNL-TVP-02	Ambient Air [1]	2
Triple Sorbent Traps	ORNL	AC-OP-3000907 CASD-AM-300-WP01 [2]	Tank Air	12
	ORNL	AC-OP-300-0907	Field Blank	2
	ORNL	AC-OP-300-0907	Trip Blank	2
Sorbent Trap System for NH_3, NO_2, NO, H_2O	PNL	PNL-TVP-09	Tank Air	6
	PNL	PNL-TVP-09	Trip Blank	3
Tritium Trap	WHC	LA-548-111	Tank Air	1
HEPA Filters	WHC	N/A	Tank Air	4

1 One sample taken through the VSS, one sample taken upwind of the tank.
2 Preparation procedure for samples spiked with surrogate(s).

Table A-2 provides a sequence of sampling activities along with sample collection times and the flow rates through sample collection tubes. A cleanliness check of the sampling system shall be performed in accordance with procedure WHC-IP-1127(4.5) Appendix C. A cleanliness of the VSS shall also be performed by collecting ambient air SUMMA® samples prior to sampling the tanks using the following conditions: 1) with the VSS manifold and transfer line fully heated, and 2) without the VSS, upwind of tank S-111.

Organic vapors shall be monitored using the GC/FID during the sampling event. The operating procedure for the GC/FID is provided in the procedure WHC-IP-1127(4.5) and Bellus (1993). The sampling team is responsible for documenting any problems and procedural changes affecting the validity of the sample in a field notebook.

Table A-2. List of Samples and Activities for Tank S-111.

SAMPLE CODE	SAMPLE/ACTIVITY DESCRIPTION	SAMPLER POSITION DURING COLLECTION	GAS FLOW RATE	SAMPLE DURATION
--	Adjust VSS temperature setpoint to 50°C [1]	N/A	N/A	N/A
--	Purge VSS with ambient air [2]	N/A	5,450 mL/min	30 min.
01	Collect ambient air sample SUMMA #1	Upwind of S-111	N/A	1 min.
--	Perform cleanliness check	N/A	N/A	N/A
02	Collect ambient air sample SUMMA #2	Port 15	N/A	1 min.
--	Leak test	N/A	N/A	N/A
--	Purge VSS with tank air	N/A	5,450 mL/min	30 min.
--	Measure tank pressure	N/A	N/A	N/A
03	Collect Tritium Trap	Sorbent line 8	200 mL/min	5 min.
--	Collect GC sample and initiate GC run [3]	N/A	N/A	N/A
04	Collect SUMMA #3	Port 11	N/A	1 min.
05	Collect SUMMA #4	Port 13	N/A	1 min.
06	Collect SUMMA #5	Port 15	N/A	1 min.
07	Collect Triple Sorbent Trap (TST) sample #1	Sorbent line 9	50 mL/min	4 min.
08	Collect TST sample #2	Sorbent line 10	50 mL/min	4 min.
09	Collect TST sample #3	Sorbent line 8	50 mL/min	4 min.
10	Open, close, & store TST Field Blank #1	In VSS truck	0 mL/min	N/A
11	Collect TST sample #4	Sorbent line 10	50 mL/min	4 min.
12	Collect TST sample #5	Sorbent line 9	200 mL/min	5 min.
13	Collect TST sample #6	Sorbent line 10	200 mL/min	5 min.
14	Collect TST sample #7	Sorbent line 8	200 mL/min	5 min.
15	Collect TST sample #8	Sorbent line 10	200 mL/min	5 min.
16	Collect TST sample #9	Sorbent line 9	200 mL/min	20 min.
17	Open, close, & store TST Field Blank #2	In VSS truck	0 mL/min	N/A
18	Collect TST sample #10	Sorbent line 10	200 mL/min	20 min.
19	Collect TST sample #11	Sorbent line 8	200 mL/min	20 min.
20	Collect TST sample #12	Sorbent line 10	200 mL/min	20 min.
21, 22	Store TST Trip Blanks #1 & #2	None	None	None
23	Collect NH3/NOx/H2O Sorbent Trap #1	Sorbent line 9	200 mL/min	15 min.
24	Collect NH3/NOx/H2O Sorbent Trap #2	Sorbent line 10	200 mL/min	15 min.
25	Collect NH3/NOx/H2O Sorbent Trap #3	Sorbent line 8	200 mL/min	15 min.
26	Collect NH3/NOx/H2O Sorbent Trap #4	Sorbent line 10	200 mL/min	15 min.
27	Collect NH3/NOx/H2O Sorbent Trap #5	Sorbent line 9	200 mL/min	15 min.
28	Collect NH3/NOx/H2O Sorbent Trap #6	Sorbent line 10	200 mL/min	15 min.
29, 30, 31	Store NH3/NOx/H2O Trap Trip Blanks #1, #2, & #3	None	None	None
32	Remove upstream HEPA Filter from HEPA transfer box	Upstream of box	Continuous	
33	Remove downstream HEPA Filter from HEPA transfer box	Downstream of box	Continuous	
34	Remove upstream HEPA Filter from VSS	Upstream of VSS	Continuous	
35	Remove downstream HEPA Filter from VSS	Downstream of VSS	Continuous	

1 Current (12/19/94) waste temperature is 34.0 °C.

2 Not required if ambient air purge incorporated in VSS setup.

3 Additional GC runs may be performed to obtain organic data and to assure cleanliness of the system at the discretion of the sampling scientist and shall be identified in the deliverable report. Organic data obtained from the on-line GC is developmental.

A2.1.3 Radiation Screening and Sample Transport

All vapor samples shall be stored under chain-of-custody requirements by SML while performing a radiological survey of certain items used during sampling. Surveys are conducted to assure compliance with Department of Transportation (DOT) shipping regulations and offsite laboratory acceptance criteria. Items surveyed include four HEPA filters and one tritium trap and are analyzed following procedures specified in Table A-4 (Bratzel 1994).

The results from the radiation screening are submitted to and shall be evaluated by Sampling and Mobile Laboratories (SML) to ensure the samples meet the analytical criteria specified in Table A-3. SML shall provide a Format II report to each analytical laboratory to specify survey results (refer to Section A6.2).

Trip blanks and field blanks are to accompany the waste samples to the laboratory. For specific information concerning sample and blank handling, custody, and transport refer to quality assurance/quality control requirements in Section A3.1.

Table A-3. Limits For Acceptable Radionuclide Activity Levels.

Organization	Total α	Total β/γ	Total $\alpha/\beta/\gamma$	Units
PNL Analytical Chemistry Laboratory	$\leq$ 100	$\leq$ 400	N/A	pCi/g
Oak Ridge National Laboratory	$\leq$ 135	$\leq$ 450	N/A	pCi/g
WHC-CM-2-14	N/A	N/A	$\leq$ 2000	pCi/g

A2.2 LABORATORY ANALYSIS

The responsibilities of the analytical laboratories to this sampling event are given in this section. Additional quality control and deliverable requirements are given in Sections A3.0 and A6.0.

A2.2.1 Preparation of Sample Media Containers

The laboratory performing the contracted analytical work shall supply labeled sample containers (SUMMA® canisters and selective sorbent media) to SML at least 48 hours in advance of the scheduled sampling date. Each sample media container shall be certified as clean and prepared according to procedures called out in Table A-1.

A2.2.2 Sample Analysis

Sample material retrieved from the tank S-111 vapor space and contained within the SUMMA® canisters shall be analyzed for organic compounds following modified EPA procedure TO-14 and for permanent gases CO_2, CO, CH_4, H_2, and N_2O using gas chromatography. The sorbent traps contain analyte-specific sorbent media and shall be analyzed for these specific analytes. The triple sorbent traps contain sorbent media designed to allow a broad range of organic species to be retained. Table A-4 identifies the appropriate laboratory procedures used in each analysis.

One SUMMA® canister shall be archived at the PNL Laboratory following receipt and control procedure PNL-TVP-07 for six months or until instructed by the Tank Vapor Program to clean the canister for reuse. If necessary, requirements for further quantification and speciation shall be conveyed through a Letter of Instruction by the Characterization Program and/or revision to this Tank Characterization Plan.

Any analyses prescribed by this document, but not performed, or other deviations, shall be identified and include justification in the appropriate data report.

A2.2.3 Insufficient Samples

Unlike a solid sample which may have full or partial recovery, vapor sample media contain either good, bad, or no sample. A sample that is bad or empty may not have a proper seal. Partial recovery of a vapor sample is not an issue, however, the number of good samples may be an issue. All good samples, except the SUMMA® canister archive, shall be analyzed. If there are insufficient good samples to perform all requested analyses, the Characterization Program Office and the Tank Vapor Issue Resolution Program shall be notified. The SUMMA® canister archive shall be used if one or more of the SUMMA® canister samples is compromised.

Figure A-1. Test Plan Outline and Flowchart for Tank Vapor Space Characterization.

Step 1. (Labs) Prepare sample and blank containers at contract laboratories. Label containers using sample identification numbers and sampling data provided by SML.

Step 2. (Labs) Ship Containers to SML at least 48 hours in advance of scheduled sampling event. Shipping, receiving, and control of containers shall be guided by procedures WHC-IP-1127(1.3) (for SML), and either PNL-TVP-07 (for PNL) or CASD-AM-300-WP02 (for ORNL).

Step 3. (SML) If tank is safe with regard to flammability, set up vapor sampling system (VSS) and collect samples following procedure WHC-IP-1127(4.5) and guidelines in Table A-2.

Step 4. (SML) Move the vapor sample containers to custody locked storage. Submit the HEPA filters and Tritium trap to the 222-S Laboratory for radiological survey.

Step 5. (SML) Using radiological survey report results, determine if samples are acceptable to ship offsite (see Table A-3)

Step 6. (SML) If determined to be acceptable according to offsite laboratory requirements and WHC-CM-2-14, ship sample and blanks. If not acceptable to ship, maintain samples in storage and contact the Tank Vapor Issue Resolution Program for further direction.

Step 7. (LABS) Perform laboratory analyses (see Table A-4 for procedure numbers).

A. SUMMA® Canisters (PNL): Perform organic vapor analysis by modified EPA-TO-14. Perform permanent gas analysis for the following: H_2, CO, N_2O, CH_4, CO_2.

B. Sorbent Traps (PNL): Perform gravimetric analysis for moisture. Perform selective electrode analysis for NH_3. Analyze NO and NO_2 Traps.

C. Triple Sorbent Traps (ORNL): Perform organic vapor analysis.

Step 8. (Labs and SML): Following the Section A6.0 reporting requirements, deliver a Format VI report to the Tank Vapor Issue Resolution Safety Program.

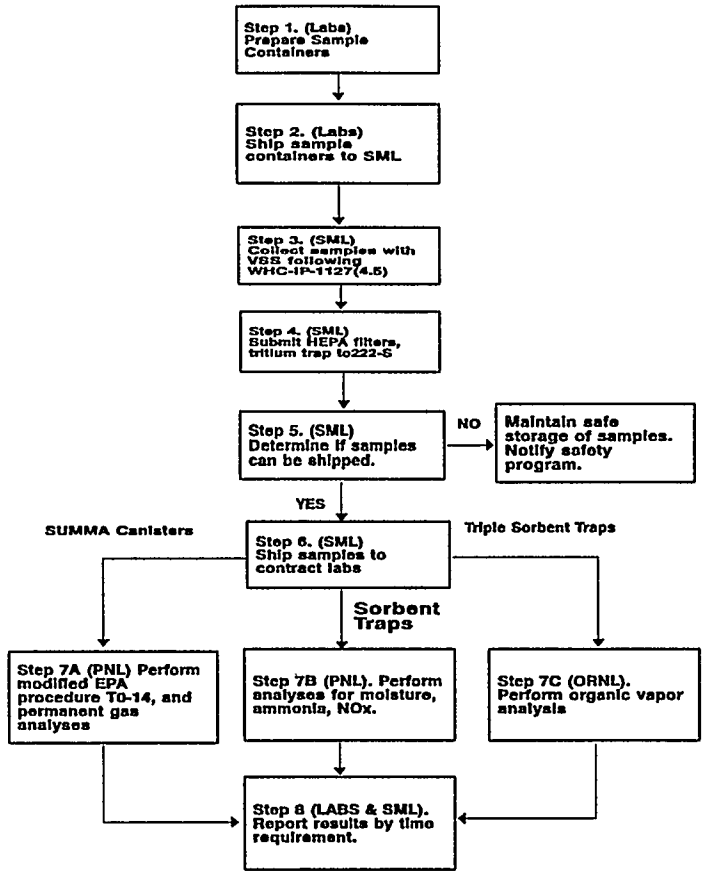

Table A-4. S-111 Sample Chemical, Physical, And Radiological Analytical Requirements

S-111 VAPOR		COMMENTS
Plan Number	WHC-SD-WM-TP-317	Type 3 vapor sampling system (VSS) using heated vapor probes.
Tank	S-111	
Program Contact	J. W. Osborne	
TWRS Contact	R.D. Schreiber C. S. Homi	
Lab Project Coordinator	S. C. Goheen (PNL) R. A. Jenkins (ORNL)	

REPORT FORMATS	
I	Early Notify
II	Process Control
III	Safety Screen
IV	Waste Management
V	RCRA Compliance
VI	Special

NO. OF SAMPLE/BLANK CONTAINERS PROCESSED				
Organization	WHC	PNL	ORNL	TOTAL
SUMMA® Canister		3[a]/2		5
Sorbent Trap System [b]		6/3		9
Triple Sorbent Trap			12/4	16
HEPA Filter	4			4
Tritium Trap	1			1

PRIMARY ANALYSES						QUALITY CONTROL[c]			CRITERIA				REPORT FORMAT
ANALYSIS METHOD	PRIMARY ANALYTE	PROCEDURE	LAB	SAMPLE PREP	SAMPLE CONTAINER	NO. OF SAMPLES	SURR SPIKE[d]	NO. OF BLANKS	NOTIFICATION LIMIT (NL)[e]	EXPECTED RANGE	PRECN at NL	ACCURACY at NL	
EPA TO-14 GC/MS	Organic* Speciation	PNL-TVP-01 PNL-TVP-02 PNL-TVP-03	PNL	Direct	SUMMA®	3	none	2	≥ 4000 ppmv n-Butanol 50% IDLH for all others*	not available	±25%	70-130%	I, VI
GC/TCD	CO_2 CO CH_4 H_2 N_2O	PNL-TVP-05 PNL-TVP-02	PNL	Direct	SUMMA®	3	none	2	N/A ≥ 20% LFL ≥ 20% LFL ≥ 20% LFL not available	not available	±25% ±25% ±25% ±25% ±25%	70-130%	VI I, VI I, VI I, VI I, VI
IC	NO NO_2	PNL-TVP-09 PNL-ALO-212	PNL	H_2O Extraction	Sorbent Trap	6	none	3	≥ 50 ppmv ≥ 25 ppmv	≥ 2 ppmv ≥ 0.1 ppmv	±25% ±25%	70-130%	I, VI I, VI
Gravimetric	H_2O	PNL-TVP-09	PNL	Direct	Sorbent Trap	6	none	3	N/A	≥ 3 mg/L	±25%	70-130%	VI
Selective Electrode	NH_3	PNL-TVP-09 PNL-ALO-226	PNL	H_2O Extraction	Sorbent Trap	6	none	3	≥ 250 ppmv	≥ 2 ppmv	±25%	70-130%	I, VI
GC/MS	Organics**	AC-MM-1-033153 CASD-OP-300-WP03 CASD-OP-300-WP04 CASD-OP-300-WP05 CASD-OP-300-WP06	ORNL	Thermal Desorption	Triple Sorbent Trap	12	all	4[f]	≥ 4000 ppmv n-Butanol, 50% IDLH for all others**	not available	±25%	70-130%	I, VI
Total α Total β Total γ	Radon Daughters	LA-508-110 LA-508-111 LA-508-162	WHC	Direct	HEPA Filter	4	N/A	N/A	≥60 pCi/g α ≥200pCi/g β ≥200 pCi/g γ	<60 pCi/g α <200 pCi/g β <200 pCi/g γ	±25% ±25% ±25%	70-130%	I, II
Liq. Scin.	Tritium [g]	LA-548-111	WHC	Direct	Tritium Trap	1	N/A	N/A	N/A	not available	N/A	N/A	II
GC/FID	Organics	WHC-IP-1127(4.5)	SML	Direct	On-line	N/A	N/A	N/A	N/A	N/A	N/A	N/A	II, VI

N/A: Not Applicable
a No extra canisters, except archive, will be stored by PNL.
b System contains individual sorbent media sections for NO_X, NH_3, & H_2O.
c Multiple samples and blanks are taken.
d Samples spiked with surrogates.
e Action required if any compound exceed 50% IDLH.
f Includes two trip and two field blanks.
g Survey purpose only.

*Acetone, acetonitrile, benzene, 1,3-butadiene, butanal, n-butanol, n-hexane, methane, propane nitrile. Other organic species detected at levels deemed sufficient by the Toxicology review Panel to be of potential toxicological concern shall be reported following Format I.

**Acetone, acetonitrile, benzene, butanol, n-dodecane, n-hexane, propane nitrile, tributyl phosphate, n-tridecane. Other organic species detected at level deemed sufficient by the Toxicology Review Panel to be of potential toxicological concern shall be reported following Format I.

A3.0 QUALITY ASSURANCE & QUALITY CONTROL

This Tank Characterization Plan and analytical laboratory operations are approved by the WHC Environmental Safety, Health, and Quality Assurance (ESH&QA) Program provided the following conditions are met.

1) Each laboratory has a quality assurance program that meets the applicable requirements of DOE order 5700.6C, or United States 10 CFR 830.120. In addition, it must also meet the requirements of QAPP-013 (Keller 1994) and when implemented in August 1995, the *Hanford Analytical Services Quality Assurance Plan* (DOE 1994).

2) Each analysis and media preparation procedure given in Tables A-1 and A-4 are documented by the laboratory and available to ESH&QA.

3) Any modifications made to, or deviations from, the prescribed procedures are documented in controlled notebooks and justified in the deliverable report.

The PNL tank vapor program is governed by a QA Plan (Barnes 1995). ESH&QA will qualify laboratories for continued use by the TWRS Characterization Program after receipt of a QA plan, followed by an audit and corrective action phase.

A3.1 Sampling Operations

SML shall provide unique sample label and identification numbers to the laboratories. Each sample identification number shall have the following format:

SXXXX-WYY-LLL, where:

XXXX	=	unique number assigned to the sampling event,
W	=	a letter code indicating the day of a multi-day sampling event,
YY	=	a 2-digit sample code found in Table A-2, List of Sample and Activities, column one.
LLL	=	a special lab assigned code.

Once the sample collection media has been received by SML from the laboratory, it shall remain in the physical control of the custodian, locked in a secure area, or prepared for shipping with tamper evident tape under conditions specified on the chain-of-custody form and in accordance with laboratory operating procedure WHC-IP-1127(1.3) "Chain-of-Custody for RCRA and CERCLA Protocol Samples".

Applicable operating procedures for the tank S-111 vapor space sampling activities are contained in work package WS-95-00038. Vapor samples, trip blanks, and field blanks are to be collected in accordance with Tables A-1 and A-2 and laboratory operating procedure WHC-IP-1127(4.5) "Collection of SUMMA® Canisters & Sorbent Tube Samples Using the Vapor Sampling System (VSS)" and shipped to the laboratory in accordance with Hazardous Material Packaging and Shipping, WHC-CM-2-14.

All sampling activities shall be documented in controlled field logbooks maintained by sampling personnel (SML) and shall contain, but are not limited to:

1) identification of tank and riser number and photographs of the sample location in which the sampling is conducted,

2) if any anomalies are observed, corresponding sample identification numbers, flow rates, pressures, temperatures, and other operational parameters affecting the sample,

3) any conditions that the sampler may observe during the sampling event (i. e., odors, nearby machinery in operation, etc.),

4) names and titles of personnel involved in the field activity and their responsibilities,

5) instrument calibration dates.

SML is responsible for documenting any problems and procedural changes affecting the validity of the sample in a controlled field notebook and shall enter this information in the comment section of the chain-of-custody form for addition to the data reports.

A3.2 Laboratory Operations

The SUMMA® canisters and Sorbent Trap Systems shall be prepared, certified, and labeled by the performing laboratories following the laboratory quality control procedures identified in Table A-1. The laboratory supplying the sample collection media shall initiate the chain-of-custody in accordance with the laboratory operating procedure WHC-IP-1127(1.3), "Chain-of-Custody for RCRA and CERCLA Protocol Samples" using sample label and identification numbers provided by SML.

The sample receipt and control procedure used in the PNL laboratory is PNL-TVP-07. Oak Ridge National Laboratory shipping and receiving is done by procedure CASD-OP-300-WP02. Analyses shall be performed following the procedures in Table A-4.

Method specific quality control such as calibrations and blanks are also found in the analytical procedures. Sample quality control (duplicates, spikes, standards) specified in the applicable DQO's are identified in Table A-4. Due to the developmental work being done with the analysis procedures and potential sample differences (between tanks), changes in procedures may be needed.

A4.0 ORGANIZATION

The organization and responsibility of key personnel involved in this tank S-111 vapor sampling project are listed in Table A-5.

Table A-5. Tank S-111 Project Key Personnel List.

Individual(s)	Organization	Responsibility
S. C. Goheen	Pacific Northwest Laboratory	Project Manager for Vapor Sample Characterization
R. A. Jenkins	Oak Ridge National Laboratory	Project Manager for Vapor Sample Characterization
J. G. Kristofzski	WHC 222-S Laboratory	Project Manager for Sample Radiological Survey
C. S. Homi	TWRS Characterization Support	S-111 Tank Characterization Plan Engineers
J. W. Osborne	TWRS Tank Vapor Issue Resolution Program	Tank Vapor Issue Resolution Program Manager
H. Babad	TWRS Characterization Program	Tank Safety Screening Scientist
R. S. Viswanath	Special Analytical Studies	Vapor Program Technical Lead
R. D. Mahon	Sampling and Mobile Laboratories	Vapor Sampling Program Lead
E. H. Neilsen	Waste Tank Safety Engineering	Vapor Sampling Cognizant Engineer
D. R. Carls	Industrial Hygiene and Safety Program	Industrial Hygiene Point of Contact if Notification Limit is Exceeded (FAX 372-3522)
West Area Shift Operations Manager	Tank Farm Operations	West Tank Farm Point of Contact if Notification Limit is Exceeded (373-3475)

A5.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

Toxicology Review Panel

The Toxicology Review Panel (TRP) is a group of toxicologists, industrial hygienists, and occupational medicine physicians that convene to review quantitative vapor sample data, identify compounds of toxicological concern, and make recommendations to the WHC Tank Vapor Program Manager concerning potential impacts to worker health and safety.

Toxicological Concern

From a list of 160 analytes found in a previous study for tank C-103, the TRP identified 19 analytes of toxicological interest. These analytes and others may be identified during TRP review of qualitative GC vapor data as being of toxicological concern if they exceed recommended levels inside the tank headspace. Established guidelines for these analytes are base on Consensus Exposure Standards (CES).

Consensus Exposure Standards

A CES is generally defined as the most stringent of known regulatory or recommended toxicological values for the occupational setting including the threshold limit value (TLV), permissible exposure limit (PEL), recommended exposure limit (REL), and biological exposure limit (BEL). For those constituents with unknown toxicological values, the TRP will be responsible for development of a CES.

Trip Blanks and Field Blanks

Trip Blanks are sampling devices prepared and handled in the same manner as samples, except that they are never opened in the field. Field Blanks are sampling devices prepared and handled in the same manner as the samples, but no tank gases are drawn through them. Laboratories supplying blanks may opt to analyze only 1 trip blank unless it is determined to be contaminated, in which case all trip blanks are to be analyzed.

Sample Custodian

The sample custodian is the designated SML cognizant scientist or assisting scientific technician, lead sampler, or laboratory scientist or technician who signs the received by block on the chain-of-custody form. Transfer of custodianship occurs when the custodian signs the relinquished by block on the chain-of-custody form and releases the sample(s) to the new custodian signator.

Physical Control

Physical control of a sample includes being in the sight of the custodian, in a room which shall signal an alarm when entered, or locked in a cabinet.

A6.0 DELIVERABLES

The Pacific Northwest Laboratory, Oak Ridge National Laboratory, and Sampling and Mobile Laboratories VSS sampling and analyses of tank S-111 vapors shall be reported as Format VI. In addition, the analytical laboratories shall receive Format II reports from Sampling and Mobile Laboratories as described in Section A6.2. Any analyte exceeding the notification limit prescribed in Table A-4 shall be reported as Format I. Other organic species detected at levels deemed sufficient by the Toxicology Review Panel to be of potential toxicological concern shall also be reported following Format I. Additional information

regarding reporting formats is given in Schreiber (1994a, 1994b, 1994c).

A6.1 Format I Reporting

Table A-4 contains the notification limits for specific analytes. Analytes that exceed notification limits defined in the DQO processes shall be reported by the Project Manager, delegate, or Health Physics Management by calling the West Area Shift Manager of Tank Farm Operations at (509) 373-3475 immediately. This verbal communication must be followed within 3 working days by written communication to J. W. Osborne of the Tank Vapor Issue Resolution Program, D. R. Carls in the Industrial Hygiene and Safety Program, and D. R. Bratzel of the Characterization Program, documenting the observation(s). A further review of the data, including quality control results and additional analyses for verification of the exceeded analyte, may be contracted between the performing laboratory and the contacts above.

A6.2 Format II Reporting

Results of the 222-S Laboratory's radiological survey shall be reported by Sampling and Mobile Laboratories as Format II to the vapor analytical laboratories listing the picocuries per sample (pCi/g/sample) for each sample submitted for analysis. This Format II report should also provide the sample collection sequence and volumes, verification of trip and field blank use, and any anomalous sampling conditions to accompany, if possible, the shipment of samples. Alternatively, this sampling report may be transmitted by FAX to the analytical laboratories within 48 hours after the samples have been shipped.

A6.3 Format VI Reporting

All Format VI reports shall be delivered to J. W. Osborne of the Tank Vapor Safety Resolution Program, R. S. Viswanath of Special Analytical Studies, the Characterization Program Office, Analytical Services, and the Tank Characterization Resource Center.

Each analytical laboratory and SML should deliver three reports. Sampling and analytical data are requested within 5 weeks after receipt of both the samples and supporting data and shall consist of, at a minimum, data tables reporting sample collection data, industrial hygiene tank monitoring data, and radiation screening results obtained by SML, or the results of each analysis performed by the analytical laboratories. A final report shall be delivered within a nine week period after receipt of both the samples and supporting data. A cleared final report shall be delivered after it has completed the proper clearance. Final reports shall be submitted to clearance in parallel to being submitted to the WHC customers identified above.

The final sampling report from Sampling and Mobile Laboratories shall be a WHC supporting document, with sponsor-limited release. It shall include:

1) A description of sampling equipment used;

2) a description of sampling quality controls applied (e.g., leak and cleanliness tests of the sampling manifold, system temperature and pressure monitoring/alarms, instrument calibration details);

3) sampling event chronology and sample collection schedule (complete list of samples, by ID#, time collected, flow rates, etc.);

4) any industrial hygiene tank monitoring data collected before or during sampling event;

5) an evaluation of sources of sampling errors;

6) sample radiation screening results;

7) sample storage and shipment details; and

8) copies of all chain-of-custody forms.

The cleared final report from the analytical laboratories shall be acceptable for distribution to the public. To the extent possible, the final reports shall include:

1) A summary of analytical results;

2) a description of sample device preparation (and manufacturer if appropriate), citing procedures and logbooks used;

3) references providing traceability of sample device cleanliness;

4) a brief description of analytical methods, with procedures cited;

5) a brief explanation of how analytical systems control was demonstratably maintained;

6) a brief description of sample storage and shipment conditions, citing procedures and logbooks used;

7) a listing of analytes of quantitation (target analytes), with analytical method detection limit, range for which instrumentation is calibrated, number of calibration points used, and statistical data on linearity of calibration;

8) quantitative analytical results, expressed as dimensionless (ppmv or ppbv) concentration, and mass concentration ($\mu g/m^3$, mg/L, etc., calculated at 0 °C and 1 atm) of target analytes (identified by name and Chemical Abstract Service number) in each tank air sample;

9) tentative identification and semi-quantitative analytical results, expressed in both mass and dimensionless concentrations (if possible) of non-target organic analytes (identified by name and Chemical Abstract Service number) in each organic vapor sample;

10) a statistical summary (i.e., mean, standard deviation) for multiple analyses and/or multiple samples for all analytes (positively and tentatively identified compounds) in both mass and dimensionless concentrations (if possible);

11) a summary of all exceptional conditions, such as deviations from procedure or protocol, results obtained outside of instrument calibration range, sorbent trap breakthrough of analytes, or poor surrogate recoveries; and

12) Copies of all chain-of-custody forms.

A7.0 CHANGE CONTROL

Under certain circumstances, it may become necessary for the performing laboratory to make decisions concerning a sample without review of the data by the customer or the Characterization Program. These changes shall be brought to the attention of the project manager and the Characterization Program as quickly as possible and documented accordingly. Changes must be justified in their documentation and follow the protocols defined in the Quality Assurance Manual, WHC-CM-4-2, Section QR 3.0, Design Control and in Standard Engineering Practices, WHC-CM-6-2, Section EP-2.2, Engineering Document Change Control Requirements. All changes shall also be clearly documented in the final data package.

Additional analysis of sample material from this vapor space characterization project at the request of the Characterization Program shall be performed according to a revision of this Tank Characterization Plan.

A8.0 REFERENCES

Barnes, B. O., 1995, *Quality Assurance Plan for PNL TWRS Tank Vapor Program*, MCS-046, Rev. 0, Pacific Northwest Laboratory, Richland, Washington.

Bratzel, D. R., 1994, *Letter of Instruction for Radiological Analyses to Support Fiscal Year 1995 Tank Vapor Sampling*, (internal memo 74310-94-32 to J. G. Kristofzski, November 30), Westinghouse Hanford Company, Richland, Washington.

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Schreiber, R. D., 1994a, *Format I Reporting Requirement*, (internal memo 7E720-94-128 to J. G. Kristofzski, August 15), Westinghouse Hanford Company, Richland, Washington.

Schreiber, R. D., 1994b, *Revised Interim Tank Characterization Plan Guidance*, (letter 7E720-94-121 to C. S. Haller, May 13), Westinghouse Hanford Company, Richland, Washington.

Schreiber, R. D., 1994c, *Point of Contact/Distribution List*, (internal memo 7E720-94-141 to J. G. Kristofzski, October 11), Westinghouse Hanford Company, Richland, Washington.

United States Department of Energy Order 5700.6C, of 08-21-91, *Quality Assurance*.

United States Code of Federal Regulations 10 CFR, Part 830, *Nuclear Safety Management;* Section 120, *Quality Assurance Requirements*.

Whelan, T. E., 1994, *TWRS Characterization Program Quality Assurance Program Plan*, WHC-SD-WM-QAPP-025, Westinghouse Hanford Company, Richland, WA.