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5. Key Words

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

6. Author

Name: B. C. CARPENTER

Brad Carpenter
Signature

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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-U-111.

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TANK 241-U-111 TANK CHARACTERIZATION PLAN

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

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LIST OF ABBREVIATIONS

DQO	data quality objective
DST	double-shell tank
ppmv	parts per million by volume
RCRA	Resource Conservation and Recovery Act
REDOX	Reduction-Oxidation process
SST	single-shell tank
TOC	total organic carbon
U-111	Tank 241-U-111
TWRS	WHC Tank Waste Remediation System
WHC	Westinghouse Hanford Company

1.0 INTRODUCTION

Sampling and analysis of the waste stored in underground storage tanks in Hanford is being done to characterize the waste for resolving various issues related to waste management and safety. Tank Characterization Plan (TCP) development by the data quality objective (DQO) process is intended to ensure users (e.g., Hanford Facility user groups, regulators) needs will be addressed and resources are devoted to gaining only necessary information (Federal Facility Agreement and Consent Order (TPA) milestone M-44-00). This Tank Characterization Plan will identify characterization objectives for tank U-111 pertaining to sample collection, sample preparation and analysis, and reporting requirements in accordance with applicable DQOs and the Tank Waste Remediation System Tank Waste Analysis plan (Haller 1994).

2.0 DATA QUALITY OBJECTIVES APPLICABLE TO TANK U-111

Waste sampling and analytical needs associated with the tank U-111 have been identified through the Data Quality Objective (DQO) process. DQO's identify information needed by a program group in the Tank Waste Remediation System concerned with safety issues, regulatory requirements, or the transporting and processing of tank waste.

Both Watch List and non-Watch List tanks will be sampled and evaluated to classify the waste tanks into one of three categories: SAFE, CONDITIONALLY SAFE, or UNSAFE following safety parameters related to the four Watch-List and other safety issues. A tank can be removed from a Watch List if it is classified as SAFE. The Watch List and other safety issue DQO's identify the requirements used to determine which classification to place a tank, based on analyses that indicate if certain measures are above or below established thresholds. The measures begin with the determination of the concentration of primary analytes which have been determined indicators of potentially unsafe conditions within a tank. If a specific criteria level on one of these items is exceeded, further analysis and a possible change in tank classification, may be required. The following sections summarize the applicable DQOs, completed as of January 1995, that were applied to meet the tank U-111 characterization objectives.

2.1 Tank Safety Screening DQO

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

2.2 Organic Fuel Rich Tank Safety Issue DQO

SSTs that carry waste with a total organic carbon (TOC) content equal or exceeds 3 wt% (dry basis) are placed in the Organic Watch List. The *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al.,1994) describes the sampling and analytical requirements, associated with Organic Watch List tanks, necessary to place the tank in an appropriate classification (SAFE, CONDITIONALLY SAFE, UNSAFE). The sampling requirements of the organic fuel rich DQO effort are for a minimum of two widely spaced core samples. Analyses employed are organic carbon, presence of a free organic liquid phase, moisture content, tank temperature, major organic species, certain oxidizing agents, hydroxide level, and radiochemical species.

2.3 Fugitive Vapor Emission DQOs

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 and have identified and placed 36 tanks on the "Suspect Tank List" (Osborne and Huckaby 1994b). DQO's concerned with fugitive vapor emissions from tank U-103 are: *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issue Resolution* (Osborne et al. 1994a) and *Rotary Sampling Core Vapor Sampling Data Quality Objective* (Price 1994). Characterization of the tank headspace is needed to: 1) identify those tanks which can safely be sampled with intrusive equipment without risk of gas ignition; 2) identify and estimate concentrations of toxicologically significant compounds present in the tank headspace to establish worker safety precautions; and 3) support the startup and operation of the portable exhauster used during rotary mode core sampling.

Data are needed to identify and quantify constituents of the tank headspaces to address potential vapor flammability and toxicity. Resolution of these two issues involves a sequence of sampling events. The first step is an assessment of a tank's headspace vapor flammability. Following resolution of the flammability issue, tank headspace samples will be taken to assess vapor toxicity. Samples are removed from a single location at or near the midpoint of the tank's headspace. Tanks that are actively ventilated will have samples removed at the exhaust header.

3.0 TANK AND WASTE INFORMATION

3.1 Configuration of Tank 241-U-111

Tank 241-U-111 (known hereafter as tank U-111) is located in the 200 West Area U Farm and is of one sixteen single-shell tanks constructed during 1946 - 47. Tank U-111 has a diameter of 22.9 meter (75 ft) and a capacity of 2,010,000 L (530 Kgal). The tank cross section and riser locations are shown in Figure 1. Tank U-111 is equipped to cascade to tank U-112 and is second to primary cascade receiver tank U-110 in a three tank cascade series. Four (three 12 inch and one 4 inch) of the twelve risers are available for use (Brevick 1994a).

3.2 January 1995 Tank Status

Tank U-111 was added to the Organic Watch List in August 1993 (Fulton 1993). Historical records indicate the TOC content of interstitial liquor in tank U-111 is approximately 14 wt% (dry basis) (Hanlon, 1993). The tank is sound and has been partially interim stabilized. It has been inactive since 1980 and is passively ventilated. The tank is estimated to contain 1,250,000 L (329 Kgal) of waste classified as double shell slurry feed (Hanlon 1994). The waste is comprised of 1,150,000 L (303 kgal) of salt cake and 98,400 L (26,000 gal) of sludge. Although there is no measurable supernate in the tank, there is 375,000 L (99 kgal) of pumpable liquid contained within the waste. The waste depth is 3.25 M (10.6 ft) and is monitored with a Food Instrument Corporation gauge through riser 8. Internal temperature is monitored from 12 thermocouple probes on a single thermocouple tree in riser 1. The temperature on August 22, 1994 of the tank was 26.7^{0}C (Hanlon,1994)

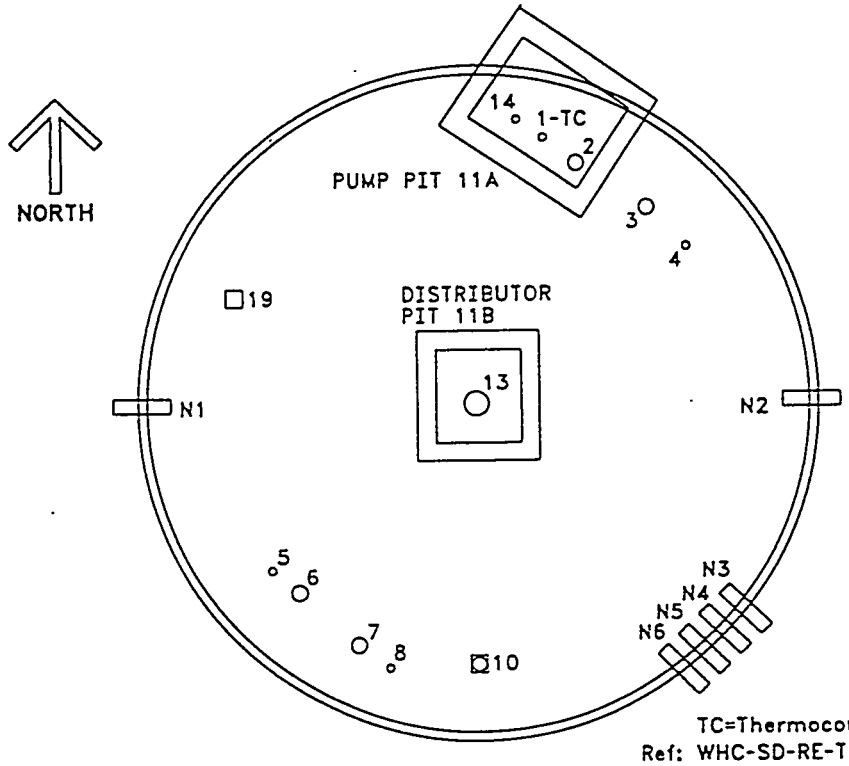
3.3 Age And Process History Of Tank 241-U-111

Tank U-111, used as a secondary cascade tank, was filled in 1947 with first-cycle wash waste from the bismuth phosphate process. The tanks entire contents were pumped to an unknown location in 1951. In 1952, the tank received a 53,000 L (14 Kgal) transfer of bismuth phosphate first-cycle sludge. REDOX waste began cascading into the tank in 1953 and continued until 1975. From 1975 to 1980, tank U-111 was the principle route for waste leaving the U Farm destined for the 242-S Evaporator. Used not only as a evaporator feed tank, it also received concentrated waste bottoms and recycled waste from the evaporator. During this period tank U-111 experienced frequent transfer activity. The tank was removed from service in 1980 and partially isolated in December 1982. The last transaction occurred in early 1980, when approximately 640,000 L of waste was removed. See Figure 2 for the level history of the tank (Brevick 1994a).

Figure 1. Configuration of Tank 241-U-111 (Brevick 1994).

241-U-111

2,008,700 Liters
[530,000 Gallons]



TC=Thermocouple
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TANK RISER LOCATION

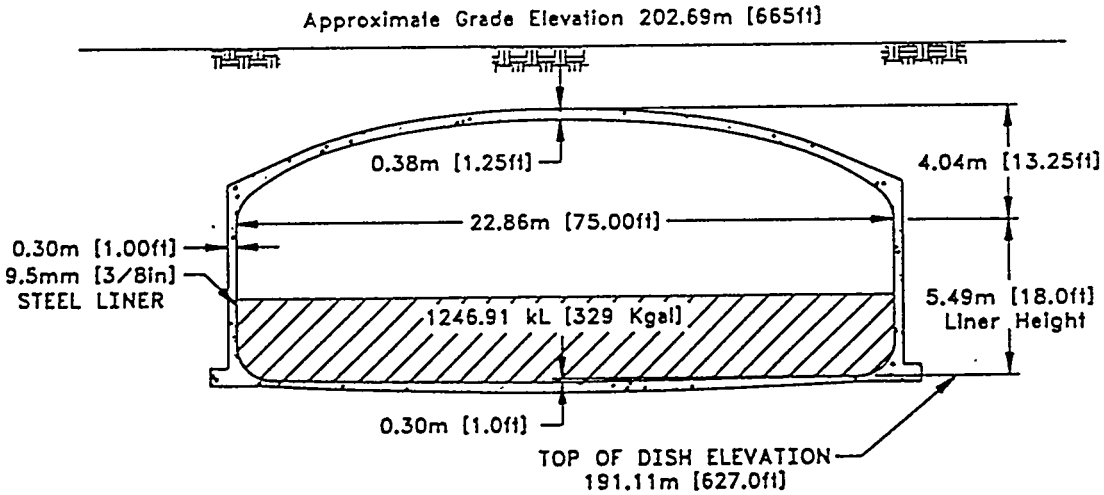

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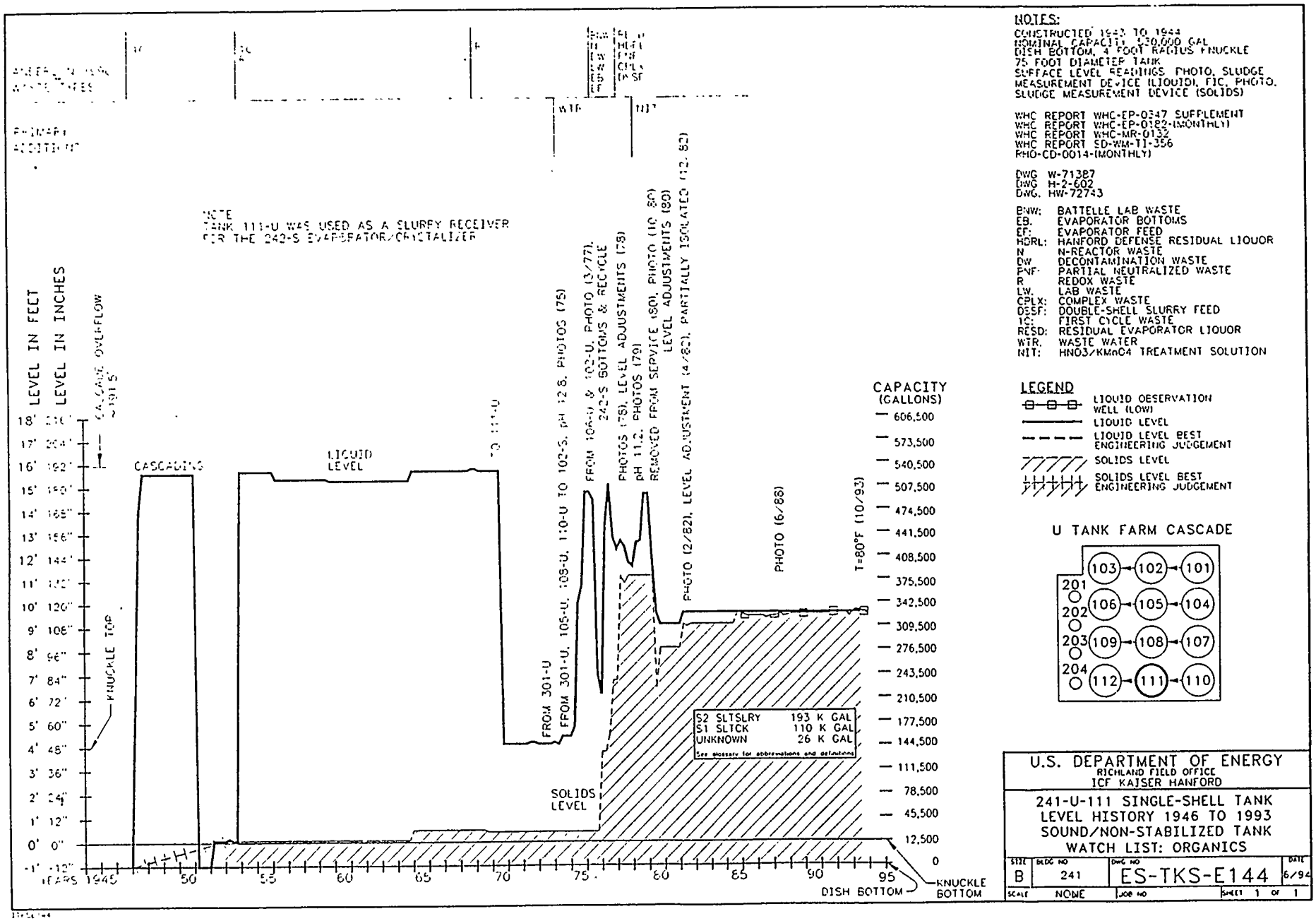
Figure 2. Level History of Tank 241-U-111 (Brevick 1994).



3.4 Expected Tank Contents

Tank U-111 stores 1,250,000 L (329 Kgal) of waste classified as double shell slurry feed. Tank U-111 was sampled in 1980. Two slurry samples were retrieved in 1980, one was a surface sample and other was from one foot above the sludge layer. The physical properties of the 1980 slurry samples indicate the top layer of the waste to be a viscous fluid slurry containing crystalline solids. Transfer of waste in the early 1980 from this tank may have occurred before or after the samples were taken. No sample date is available to verify this. The laboratory report is dated September 1980, several months after the current waste level was established, sometime during the second quarter of 1980. In 1975, both a supernate and sludge sample were obtained and analyzed (Brevick 1994b). The latter samples were taken prior to the numerous waste transactions that occurred to and from tank U-111 to support the 242-S Evaporator campaign. A summary of the analytical results is provided in Table 1.

Also included in the last column of Table 1 are solid waste analyte estimates from the Tank Layering Model (TLM). Development of the TLM is continuing. The uncertainty of the TLM estimates is unknown. A photograph taken in 1988 shows the waste surface as a somewhat dried dark colored sludge or slurry containing small patches of white solids (Brevick 1994a).

Table 1. Historical Analytical Results and Model Estimates (Brevick 1994b).

Sample Date	7/8/75	8/14/75	9/23/80	9/23/80	1994 TLM Estimate[1]
Description	Supernate	Sludge	Slurry (Surface)	Slurry (1 ft above sludge)	Average of Total Solids
Total Solid Waste	NR	NR	NR	NR	1.91E+06 kg
Heat Load	NR	NR	NR	NR	2.96 kW
Density (g/ml)	1.118	0.91	NR	NR	1.54
p^H	12.8	NR	NR	NR	NR
Analyte Concentration (µg/g)					
Aluminum	1,400	87,400	96,000	66,000	36,500
TIC	1,000	NR	NR	NR	3,220
Fluoride	300	NR	NR	NR	1,210
Sodium	47,900	136,000	NR	NR	153,000
Nitrate	100,000	5,800	249,000	208,000	176,000
Nitrite	2,700	6,100	112,000	105,000	54,900
Iron	NR	3,100	NR	NR	1,370
Hydroxide	9,700	3,600	56,300	51,700	95,800
Phosphate	1,500	132,000	27,000	46,600	7,240
Silicon	NR	3,400	NR	NR	231
TOC	NR	NR	5,200	5,400	2,560
Water	806,000	404,000	391,000	336,000	450,000
Chromium	Not Reported (NR)				2,130
Bismuth					365
$ZrO(OH)_2$					92.5
Lead					4.34E-02
Nickel					277
Manganese					293
Calcium					1,270
Potassium					1,470
Sulfate					13,000
Chlorine					2,570
$C_6H_5O_7^{-3}$					4,150
$HEDTA^{-3}$					7.69
glycolate					2,340
DBP					668
Uranium					1,050
Total Pu	1.20E-10	0.293	NR	NR	3.33E-02 µCi/g
(µCi/g)					
$^{89/90}Sr$	5.69E-2	2.9	NR	NR	28.3
^{134}Cs	1.45E-2	4.2	NR	NR	290 total Cs
^{137}Cs	34.98	26.1	NR	NR	

[1]Estimated composition of solid tank waste from the Tank Layering Model (Brevick 1994a).
NR = Not Reported

4.0 STRATEGY FOR WASTE CHARACTERIZATION AND SAFETY ISSUE RESOLUTION

In this section the DQO requirements for sampling and analyses are integrated and compared with scheduled sampling and analyses activities, and recommendations to resolve discrepancies are given.

4.1 Sampling of Tank U-111 in the Near Term

In fiscal year 1995 tank U-111 will be vapor sampled to identify potential flammable and fugitive vapor emissions from the tanks. A rotary core sample is scheduled for fiscal year 1996 (Stanton 1994).

Tank U-111 is one of the 36 tanks on the "Suspect Tank List" (Osborne and Huckaby 1994b). Sampling and analysis of the tank U-111 vapor space can identify and estimate: 1) the flammable constituents in the vapor above the waste; 2) gases generated by chemical and radiolytic reactions within the waste. DQO's concerned with fugitive vapor emissions from tank U-111 are: *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). Vapor sampling will satisfy part of the requirement specified in TPA Milestone M-40-08 to complete the vapor sampling of all organic Watch List tanks (Osborne and Huckaby 1994b).

Core sampling will satisfy the sampling requirements of the *Tank Safety Screening Data Quality Objective* (Redus and Babad 1994); and The *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al.,1994). The result of integrating sampling and analytical requirements from DQOs identified in this section is given in Table 2. A complete list of analytical requirements are given, as a appended revision, in the appropriate sampling and analysis plan.

Table 2. Integrated DQO Requirements.

Sampling Activity	Primary DQO Analytes	Scheduled Sampling Date
Vapor Sampling 3 SUMMA canisters 12 Triple Sorbent Traps 6 Sorbent Trap Systems	Gas Flammability Gas Toxicity	February 1995
Core Sampling, rotary mode 2 cores consisting of one partial and six complete segments.	Energetics, Moisture, Total Organic Carbon, Total Alpha, Organic Floating layer	November 1995

The scheduled sampling events, if successful, will satisfy the January 1995 applicable DQO requirements. The 1980 sample physical description and the 1988 photograph of the tank interior show that the waste surface may have fluid like properties. Since the fiscal year 1996 rotary mode core sampling may not be effective in this waste type, it is recommended that an auger sample be taken to determine surface layer physical properties prior to attempting a rotary mode core sample.

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APPENDIX A

SAMPLING AND ANALYSIS PLAN FOR VAPOR SAMPLING IN FISCAL YEAR 1995

TANK 241-U-111

CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ACRONYMS FOR APPENDIX A

U-111	Tank 241-U-111
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
OGIST	Oregon Graduate Institute of Science and Technology
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
SUMMA®	registered trademark for passivated stainless steel canister
TCP	Tank Characterization Plan
TNMHC	Total Non-Methane Hydrocarbons
TRP	Toxicology Review Panel
TO-14	EPA Toxic Organics 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-U-111 (U-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 planned 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). The ISS is a hand cart used to collect permanent (non-condensible) gas samples. The VSS is a more elaborate mobile truck sampling system which employs a heated sampling tube to collect condensible gases (i.e., moisture and organics).

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 U-111. The requirements for sample event A, contained within this appendix of the TCP, are within the scope of work specified in the appropriate laboratory financial plans. 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 U-111 vapor sampling activity refer to work package WS-94-515. 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. Flammability testing 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).

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

Step 1. (Contract Labs) Prepare sample and blank containers with sample identification numbers and sampling data provided by SML.

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 LO-090-443 (for SML), and either PNL-TVP-07 (for PNL) or CASD-AM-300-WP02 (for ORNL).

Step 2. (Industrial Hygiene Field Services) Performed the flammability test using a combustible gas meter (CGM). If the concentration is ≥ to 20% of LFL, stop sampling until further authorization.

Step 3. (SML) If tank is safe with regard to flammability, set up vapor sampling system (VSS) and collect samples following procedure LO-080-450 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 section A2.1.3)

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

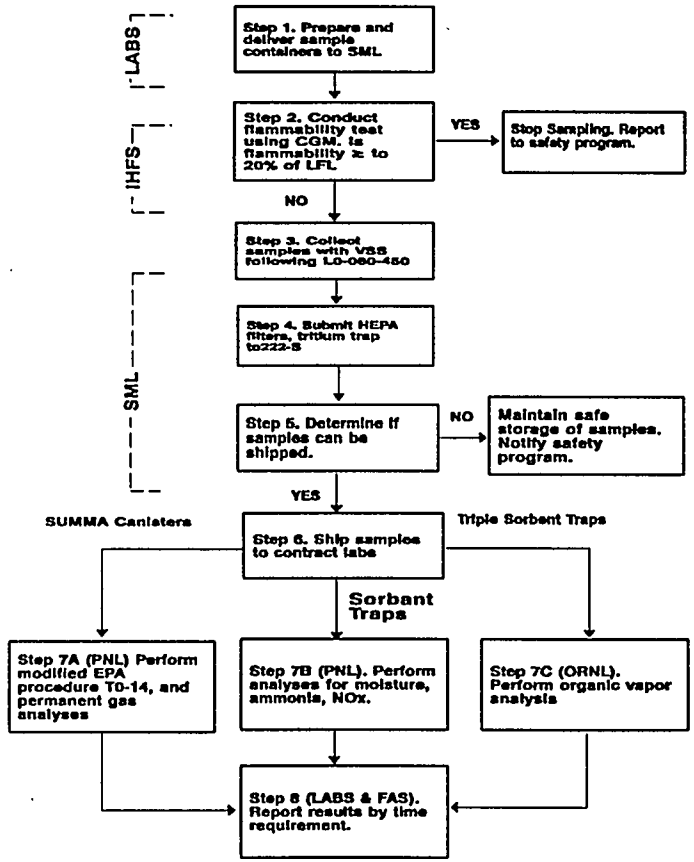
Step 7. (LABS) Perform laboratory analyses (see Table A-4 for procedure numbers).

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 Format VI reports.



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

SML shall provide sample identification numbers to the laboratories for the sample media according to the format given in Section A3.1. SML shall use labeled sample containers supplied by the laboratory (see Section A2.2.1 for preparation of sample media containers) to collect vapor samples. The vapor sampling system (VSS) shall be used to collect vapor from tank U-111 in accordance with laboratory operating procedure LO-080-450 "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 U-111.

Sample Container	Prepared By	Preparation Procedure	Sample Type	Number of Samples
SUMMA® Canisters	PNL	PNL-TVP-02	Tank Air	6
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 time and the flow rate through sample collection tubes. A cleanliness check of the sampling system shall be performed in accordance with procedure LO-080-450 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 U-111.

Organic vapor shall be monitored by using GC/FID during the sampling event. The operating procedure for GC/FID is provided in the procedure LO-080-450 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 U-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 U-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 (8/22/94) waste temperature is 26.7 °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	Units
PNL Analytical Chemistry Laboratory	$\leq$ 100	$\leq$ 400	pCi/g
Oak Ridge National Laboratory	$\leq$ 135	$\leq$ 450	pCi/g
WHC-CM-2-14 [1]	$\leq$ 60	$\leq$ 200	pCi/g

1 Samples above these limits may be shipped as Limited Quantity of Radioactive Material.

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 laboratories 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 U-111 vapor space and contained within the SUMMA® canisters shall be analyzed for organic compounds following modified EPA procedure TO-14, and the 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 documented 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.

Table A-4. U-111 Sample Chemical, Physical, and Radiological Analytical Requirements

U-111 VAPOR		COMMENTS	REPORT FORMATS		NO. OF SAMPLE/BLANK CONTAINERS PROCESSED				
Plan Number	WHC-SD-WM-TP-249	Type 3 vapor sampling system (VSS) using heated vapor probes.	I	Early Notify	Organization	WHC	PNL	ORNL	TOTAL
Tank	U-111		II	Process Control	SUMMA® Canister		3[a]/2		5
Program Contact	J. W. Osborne		III	Safety Screen	Sorbent Trap System[b]		6/3		9
TWRS Contact	B. C. Carpenter C. S. Homi		IV	Waste Management	Triple Sorbent Trap			12/4	16
			V	RCRA Compliance	HEPA Filter	4			4
Lab Project Coordinator	S. C. Goheen (PNL) R. A. Jenkins (ORNL)		VI	Special	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	LO-080-450	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 the vapor QAPP (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 has a Quality Assurance Plan (Barnes 1995) written to comply with 5700.6C. 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 LO-090-443 "Chain-of-Custody for RCRA and CERCLA Protocol Samples".

Applicable operating procedures for the tank U-111 vapor space sampling activities are contained in work package WS-94-515. Vapor samples, trip blanks, and field blanks are to be collected in accordance with Tables A-1 and A-2 and laboratory operating procedure LO-080-450 "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 LO-090-443, "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 laboratories 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. If no criteria are provided in Table A-4, the performing laboratory shall analyze quality control samples according to its QA Plan.

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 U-111 vapor sampling project are listed in Table A-5.

Table A-5. Tank U-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
B. C. Carpenter C. S. Homi	TWRS Characterization Support	U-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 U-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 laboratory scientist 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 East Area Shift Manager of Tank Farm Operations at (509) 373-2689 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 Field Analytical Services, the Characterizaton Program Office, Analytical Services, and the Tank Characterization Resource Center.

Each analytical laboratory and SML shall 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. Changes may be documented through the use of internal change notices or analytical deviation reports for minor, low-impact changes (no change in scope). All significant changes (such as changes in scope) shall be documented by Characterization Support via an Engineering Change Notice to this Tank Characterization Plan. 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

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