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Tank 241-U-103 Tank Characterization Plan (WHC-SD-WM-TP-288)		ECN No.

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Document Title:	TANK 241-U-103 TANK CHARACTERIZATION PLAN
Release Date:	January 25, 1995

This document was reviewed following the procedures described in WHC-CM-3-4 and is:

APPROVED FOR PUBLIC RELEASE

WHC Information Release Administration Specialist:

Tamara Nohm, for — January 25, 1995

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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-103.

8. RELEASE STAMP

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WHC-SD-WM-TP-288
Revision 0

Tank 241-U-103
Tank Characterization Plan

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

by

Los Alamos Technical Associates
8633 Gage Boulevard
Kennewick, Washington 99336

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LIST OF ABBREVIATIONS

244	Lanthanum Fluoride Waste
BL	B-Plant Low-Level Waste
BNW	Battelle Northwest Laboratory Waste
CW	Coating Waste
DNFSB	Defense Nuclear Facilities Safety Board
DOE	Department of Energy
DQO	Data Quality Objective
DSSF	Double-Shell Slurry Feed
DST	Double-Shell Tank
DW	Decontamination Waste
EB	Evaporator Bottoms
EVAP	Evaporator Feed (Post 1976)
HDRL	Hanford Defense Residual Liquor
HTCE	Historical Tank Characterization Estimates
IX	Ion Exchange Waste
MW	Metal Waste

NCPLX	Non-complexed Waste
PNF	Partial Neutralized Waste
R	REDOX Waste
RCRA	Resource Conservation and Recovery Act
RIX	REDOX Ion Exchange Waste
SST	Single-Shell Tank
TCP	Tank Characterization Plan
TLM	Tank Layering Model
TOC	Total Organic Carbon
TWRS	Tank Waste Remediation System
U-103	241-U-103
WHC	Westinghouse Hanford Company

1.0 INTRODUCTION

The Defense Nuclear Facilities Safety Board (DNFSB) has advised the Department of Energy (DOE) to concentrate the near-term sampling and analysis activities on the identification and resolution of safety issues (Conway 1993). The data quality objective (DQO) process was chosen as a tool to be used to identify the 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-44-00 has been made, which states that "A Tank Characterization Plan (TCP) will also 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." This document satisfies that requirement for tank 241-U-103 (U-103) sampling activities.

2.0 DATA QUALITY OBJECTIVES APPLICABLE TO TANK U-103

The sampling and analytical needs associated with the 177 single-shell and double-shell underground storage tanks have been identified through the DQO process. DQO's identify information needed by a program group in the Tank Waste Remediation System (TWRS) concerned with safety issues, regulatory requirements, or the transporting and processing of tank waste. Present characterization objectives for fiscal year 1995 involve sampling of tanks to resolve safety issues. Other samples may be requested for ongoing waste processing and regulatory requirements in active tanks. As of January 1995, the following DQO's apply to tank U-103.

2.1 SAFETY ISSUE DATA QUALITY OBJECTIVES

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.

2.2 SAFETY SCREENING DATA QUALITY OBJECTIVES

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.

2.3 WASTE COMPATIBILITY DATA QUALITY OBJECTIVES

The *Data Quality Objectives for the Waste Compatibility Program* (Carothers 1994) identified 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 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 exist to judge compatibility with safety and operations decision rules.

2.4 WATCH LIST DATA QUALITY OBJECTIVES

Based on the current classification of the tank, the Watch List DQO's applicable to tank U-103 are: *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al. 1994) and *Flammable Gas Safety Program: Data Requirements for the Flammable Gas Safety Issue Developed through the Data Quality Objectives (DQO) Process* (McDuffie and Johnson 1994).

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.

The flammable gas DQO effort focuses on DST's that contain or may contain, based on safety screening, flammable gases above the established decision thresholds. Data from core samples are needed to provide an understanding of the mechanisms for gas generation, conditions which cause gas retention, the source terms for dose consequence calculations, and to support tank behavior models needed to 1) develop mitigation methods and 2) make appropriate safety analysis decisions on future operations to prevent the creation of additional flammable gas tanks. To achieve this, a multitude of chemical and radionuclide composition and physical property analyses are needed along with supporting operational data. The most reliable information can be obtained from one complete core.

2.5 FUGITIVE VAPOR EMISSION DATA QUALITY OBJECTIVES

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 headspace 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 U-103 HISTORICAL INFORMATION

This section summarizes the available information on tank U-103. Included are the present status and physical description of the tank, its age, process history, and the expected contents of the tank based on historical information. The fill history information is available in *A History of the 200 Area Tank Farms* (Anderson 1990) and *Historical Tank Content Estimate for the Northwest Quadrant of the Hanford 200 West Area* (Brevick 1994).

3.1 JANUARY 1995 TANK STATUS

Tank U-103 is on the Organic and the Hydrogen Watch Lists. It was added to the Organic Watch List in May 1994 and to the Hydrogen Watch List in January 1991.

This tank currently contains non-complexed waste (NCPLX) with an estimated total waste volume of 1,770 kL (468 kgal), which is equivalent to 419 cm (165 in) of waste as measured from the baseline of the tank. The surface level, as monitored with a Food Instrument Corporation gauge, has remained steady with the readings ranging between 423.7 cm (166.8 in) and 421.1 cm (165.8 in). The waste is comprised of 49.2 kL (13 kgal) of supernate, 1,600 kL (423 kgal) of saltcake, and 121 kL (32 kgal) of sludge. A portion of the solids volume consists of 666 kL (176 kgal) drainable interstitial liquid (Hanlon 1994).

3.2 TANK CONFIGURATION

Tank U-103 is one of 16 single-shell tanks in the 200 West Area U Farm which was constructed between 1946 and 1949. It is 23 m (75 ft) in diameter with a concave-shaped base and has a 2,010 kL (530 kgal) tank capacity. Tank U-103 is third in a three-tank cascade series with tanks 241-U-101 and 241-U-102.

3.3 TANK HISTORY

Tank U-103, constructed between 1943 and 1944 as the last in a three tank cascade, entered service in February 1947 and contained metal waste from 1947 until 1956. In 1956 the tank was sluiced out and declared empty. The tank received REDOX waste from 1957 until 1975. During the second quarter of 1975 the tank received 242-T bottoms and recycle waste. From 1978 until 1980, the tank received partial neutralized feed waste. Between 1974 and 1976 the tank received waste water. During 1977 the tank received HNO_3 / $KMnO_4$ solution. The tank was removed from service and labeled inactive in 1978. Presently the waste is classified as non-complex waste (Brevick, 1994). Tank U-103 has been declared sound and was partially isolated in December 1982. The tank level was adjusted in December 1977 and April 1982.

From July 1987 to present, the median temperature is 30 °C (86 °F) with a minimum of 22.3 °C (72.2 °F) and a maximum of 37 °C (99 °F).

The last solids volume update was obtained April 28, 1982, and the last photo was taken September 13, 1988 (Hanlon 1994). The photograph shows that the waste surface is partially covered with a floating saltcake and most of the tank is filled with a dark grey liquid. Figure 1 summarizes the fill history of tank U-103 from 1947 to the present (Brevick 1994).

3.4 EXPECTED TANK CONTENTS

Tank U-103 is expected to contain four primary layers of waste. The top layer consists of supernate, followed by a salt slurry waste layer which accumulated during the 242-S evaporator/crystallizer campaign from 1977 until 1980. The third layer, the largest portion of the waste volume, is a saltcake waste layer generated from the 242-S evaporator/crystallizer from 1973 until 1976. The bottom layer consists of metal waste. A summary of the analytical data from Brevick (1994) is given in the second column of Table 2. Although the total organic carbon (TOC) content of tank U-103, as obtained from the *Historical Tank Characterization Estimates* (HTCE) (Brevick 1994), is listed in the table as 0.07 wt%, Turner (1994) reports a higher laboratory measurement of 3.38 wt% TOC (wet basis) for a tank U-103 "salt" sample. Converting this number to a "dry basis," as used for the 3.0 wt% TOC Watch List criterion, would yield a yet higher value. Development and refinement of the Tank Layering Model (TLM), which is the basis of the HTCE estimates, is continuing. At this point, the uncertainty of the TLM estimates is unknown.

In addition, calculation models have been developed using vapor liquid equilibria, tank farm measurements, and empirical correlations to estimate tank farm vapor space composition. By using the available WHC analytical data on the waste composition and volume, a model input has been developed and implemented. The resulting estimated vapor composition for tank U-103 (Scott 1994) is summarized in the last column of Table 2.

Figure 1: Tank U-103 Fill History

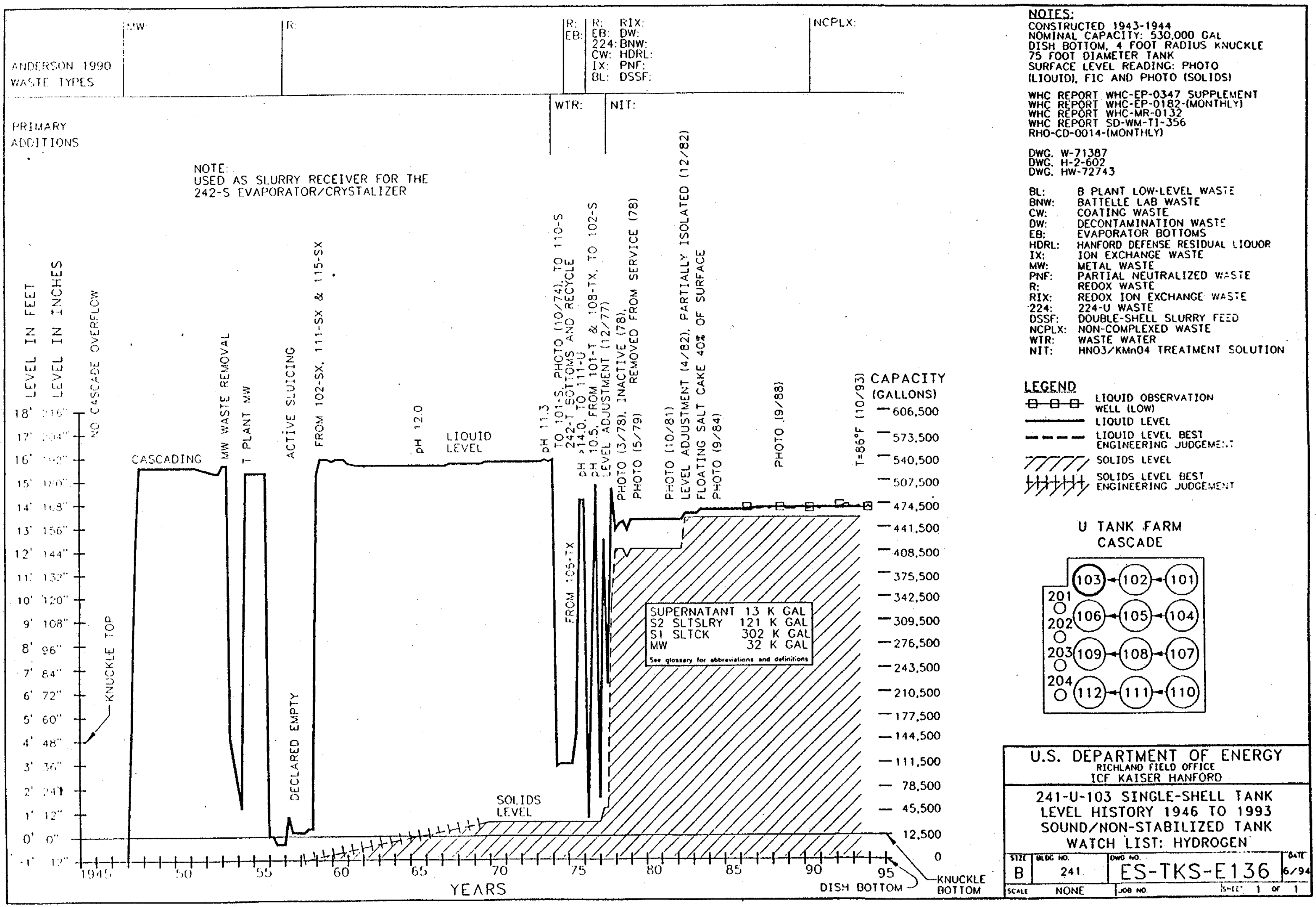

Table 1: Tank U-103 Estimated Solid[1] and Vapor[2] Compositions.

Physical Properties		
Total solid waste		3.00E+06 kg (455 kgal)
Heat load		3.08 kW (1.05E+04 BTU/hr)
Bulk density		1.74 (g/cc)
Void Fraction		0.39
Water wt%		18.09
TOC wt% C(wet)		0.07
Analyte	**Solid Concentration (μg/g)**	**Estimated Vapor Concentration (ppmv)**
Na^{+}	1.99E+05	8.57E-04
Al^{3+}	1.67E+04	4.46E-05
Cr^{3+}	45.38	1.15E-06
Ni^{2+}	1.31E+02	--
Mn^{4+}	3.31E+03	8.15E-09
K^{+}	4.37E+02	2.30E-06
OH^{-}	4.37E+04	2.64E-05
NO_3^{-}	3.39E+05	5.09E-05
NO_2^{-}	7.56E+03	4.49E-06
CO_3^{2-}	3.14E+04	3.58E-05
PO_4^{3-}	3.58E+04	2.54E-05
SO_4^{2-}	2.61E+04	2.07E-07
F^{-}	2.50E+03	6.93E-09
U	1.17E+04	3.71E-09
Cl^{-}	3.50E+02	3.20E-07
$C_6H_5O_7^{3-}$	1.06E+03	--
$HEDTA^{-3}$	1.81	--
glycolate^{-}	7.23E+02	--
acetate^{-}	3.61+02	--
Radiological Constituents	**Solid Concentration (μCi/g)**	**Estimated Vapor Concentration (μCi/l)**
$^{239/240}Pu$	8.49E-03	1.67E-08
^{137}Cs	2.01E+02	2.70E-05
^{90}Sr	13.11	8.42E-08

[1](Brevick 1994); [2](Scott 1994)

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.

4.1 SAMPLING OF TANK U-103 IN THE NEAR TERM

The characterization objectives in fiscal year 1995 involve sampling of tanks to identify and resolve safety issues. Two sampling events of tank U-103 are scheduled for the 1995 fiscal year: a vapor sampling in February and a grab sampling in March (Stanton 1994). The first sampling event shall be performed in accordance with the two DQO's dealing with fugitive vapor emissions: *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). 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). The second sampling event shall be performed in accordance with the *Data Quality Objectives for the Waste Compatibility Program* (Carothers 1994).

A rotary core sampling event of tank U-103 is scheduled for the 1996 fiscal year (Stanton 1994). This sampling event shall be performed in accordance with the *Tank Safety Screening Data Quality Objective* (Redus and Babad 1994), *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al. 1994), and *Flammable Gas Safety Program: Data Requirements for the Flammable Gas Safety Issue Developed through the Data Quality Objectives (DQO) Process* (McDuffie and Johnson 1994).

4.2 INTEGRATED DQO REQUIREMENTS

Integration of sampling and analytical requirements from DQO's identified in the previous section is summarized in Table 2. These sampling and analysis events, if successful, will also satisfy the January 1995 applicable DQO requirements. A complete list of analytical requirements will be given, as a separate appendix of the sampling and analysis plan, for each sampling event.

Table 2: Integrated DQO Requirements

Sampling Event	Applicable DQO	Sampling Requirements	Analytical Requirements
Vapor Sampling	-Health & Safety Vapor Issue Resolution DQO -Rotary Sampling Core Vapor Sampling DQO	6 SUMMA® canisters 12 Triple Sorbent Traps 6 Sorbent Trap Systems	Gas Flammability Gas Toxicity -Organic Vapors -Permanent Gases
Grab Sampling	-Compatibility DQO	3 grab samples	Energetics, Moisture, Major Anions, Cations & Radionuclides, SpG & pH, Separable Organics
Rotary Sampling	-Safety Screening DQO -Organic DQO -Flammable Gas 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

TANK U-103 VAPOR SAMPLING AND ANALYSIS PLAN FOR FISCAL YEAR 1995

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CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CGM	Combustible Gas Meter
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective
ECN	Engineering Change Notice
EPA	Environmental Protection Agency
ESH&QA	Environmental Safety, Health, and Quality Assurance
FAS	Field Analytical Services
GC/MS	Gas Chromatography/Mass Spectrometry
HEPA	High-Efficiency Particulate Air Filters
IC	Ion Chromatography
IDLH	Immediately Dangerous to Life and Health
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
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
SAP	Sampling and Analysis Plan
SML	Sampling and Mobile Laboratories
SUMMA®	registered trademark for passivated stainless steel canister
TCP	Tank Characterization Plan
TRP	Toxicology Review Panel
TO-14	EPA Toxic Organics Protocol 14
TOC	Total Organic Carbon
TST	Triple Sorbent Trap
TWRS	Tank Waste Remediation System
U-103	Tank 241-U-103
VSS	Vapor Sampling System
WHC	Westinghouse Hanford Company

A1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) will identify characterization objectives for tank 241-U-103 (U-103) pertaining to sample collection, preparation and analysis, and laboratory analytical evaluation and reporting requirements in accordance with the *Tank Waste Remediation System Tank Waste Analysis Plan* (Haller 1994) and the following applicable Data Quality Objectives (DQO's): *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 samples are used to identify potential flammable and fugitive vapor emissions from the tanks which could become worker health and safety issues. Sampling and analysis of the vapor space can identify: 1) volatile compounds above the surface of the waste; and 2) gases generated by chemical or radiolytic reactions within the waste.

A2.0 SCHEDULED SAMPLING EVENT

The following information provides the methodology and procedures to be used in the preparation, retrieval, transport, analysis, and reporting of results for vapor space samples retrieved from tank U-103. The requirements for the vapor sampling of tank U-103 in the 1995 fiscal year, contained within this SAP of the Tank Characterization Plan (TCP), are within the scope of work specified in the appropriate laboratory financial plans. Any decisions, observations, or deviations to this SAP made during sample receipt, preparation, and analysis shall be documented in controlled notebooks and justified in the deliverable report.

A2.1 PREPARATION OF SAMPLE MEDIA CONTAINERS

The laboratories performing the contracted analytical work shall supply prepared and labeled sample containers (SUMMA® canisters and/or selective sorbent sampling media) to Field Analytical Services (FAS) 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. FAS shall provide sample identification numbers to the laboratories for the sample media as requested following the quality assurance/quality control format given in Section A3.1.

A2.2 FLAMMABILITY OF VAPOR SPACE GASES

Prior to performing intrusive work on a tank, an assessment of the flammability of the tank vapor space gases is required by standard WHC safety practices. The flammability test procedure is included in the sampling event work package and performed by Industrial Hygiene Field Services personnel using a combustible gas meter (CGM). Under present guidelines no operational or sampling activity is permitted if a single sample of the tank vapor fuel content is greater than 20% of the lower flammability limit (LFL). If the CGM sample has a total fuel content between 10% and 20% of the LFL, a vapor sampling activity may continue under CGM monitoring to better identify the hazard level. Under 10% of the LFL the tank is not considered a flammability problem and all scheduled work can proceed (Osborne et al. 1994).

A2.3 SAMPLE COLLECTION

In fiscal year 1995, the tank U-103 vapor space shall be sampled through a heated probe in an available riser using the vapor sampling system (VSS) in accordance with laboratory operating procedure LO-080-450 "Collection of SUMMA® Canisters & Sorbent Tube Samples Using the Vapor Sampling System (VSS)". Table A-1 specifies the sample type, the type of collection media to be used, and the number of samples requested. Table A-2 provides a sequence of sampling activities and specifies the sample collection time and the flow rate through the sample collection tubes.

A cleanliness check shall be performed in accordance with procedure LO-080-450, Appendix C. Cleanliness of the VSS shall also be addressed by collecting ambient air SUMMA® samples prior to sampling the tanks using the following conditions: 1) with the VSS manifold and transfer lines fully heated; and 2) without the VSS, upwind of U-103.

The GC/FID shall be used to monitor organic vapors during the sampling event. The GC/FID shall be operated in accordance with LO-080-450, Appendix D and Bellus (1993).

Table A-1: General Sampling Information.

Sample Container	Prepared By	Preparation Procedure	Sample Type	Number of Samples
SUMMA®	PNL	PNL-TVP-02	Tank Air	3
SUMMA®	PNL	PNL-TVP-02	Ambient Air[1]	2
Triple Sorbent Traps	ORNL	AC-OP-300-0907 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).

A2.4 RADIATION SCREENING AND SAMPLE TRANSPORT

All vapor samples shall be stored under chain-of-custody requirements by FAS 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 shall be analyzed following procedures specified in a Letter of Instruction (Bratzel 1994). These procedures are referenced in Table A-4.

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 report to each analytical laboratory to identify the number of picocuries per sample (pCi/sample) for each sample that is submitted for analysis.

Table A-2: List of Samples and Activities

SAMPLE CODE	SAMPLE/ACTIVITY DESCRIPTION	SAMPLER POSITION DURING COLLECTION	GAS FLOW RATE	SAMPLE DURATION
--	Adjust VSS temperature setpoint to 60°C	N/A	N/A	N/A
--	Purge VSS with ambient air[1]	N/A	5,450 mL/min	30 min.
01	Collect ambient air sample SUMMA #1	Upwind of U-103		1 min.
--	Perform cleanliness check			
02	Collect ambient air sample SUMMA #2	Port 15		1 min.
--	Leak test	N/A		
--	Purge VSS with tank headspace 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[2]			
04	Collect SUMMA #3	Port 11		1 min.
05	Collect SUMMA #4	Port 13		1 min.
06	Collect SUMMA #5	Port 15		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	
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	
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]Not required if ambient air purge incorporated in VSS setup.
[2]Additional GC runs may be performed to obtain organic data and to assure cleanliness of 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.

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.

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.

A2.5 TANK-SPECIFIC ANALYTICAL PROCEDURES

A flowchart and narrative showing the sample collection, isolation, and analysis scheme is presented as Figure A-1. All samples are to be prepared and analyzed in accordance with this scheme. Sample receipt, custody, preparation, and analysis shall be performed in accordance with approved procedures.

Sample material retrieved from the tank U-103 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, which contain analyte-specific sorbent media, 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, shall be identified and include documented justification in the appropriate data report. If there are insufficient samples to perform all requested analyses, a partial listing of the analyses in Table A-4 that could be performed with available samples will be developed by Tank Vapor Issue Resolution Program personnel. The laboratory shall proceed with these analyses.

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 Field Analytical Services.

•Step 2 Labs: Ship containers to Field Analytical Services at least 48 hours in advance of scheduled sampling event. Receipt and control of containers shall be guided by WHC procedure LO-080-450 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 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 A2.4).

•Step 6 SML: If determined to be acceptable by offsite laboratory requirements and WHC-CM-2-14, ship samples and blanks following DOT requirements. If not acceptable to ship, maintain samples in storage and contact J. W. Osborne of Vapor Issue Resolution Program for further direction.

•Step 7 Labs: Perform laboratory analyses.

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 Vapor Issue Resolution Safety Program.

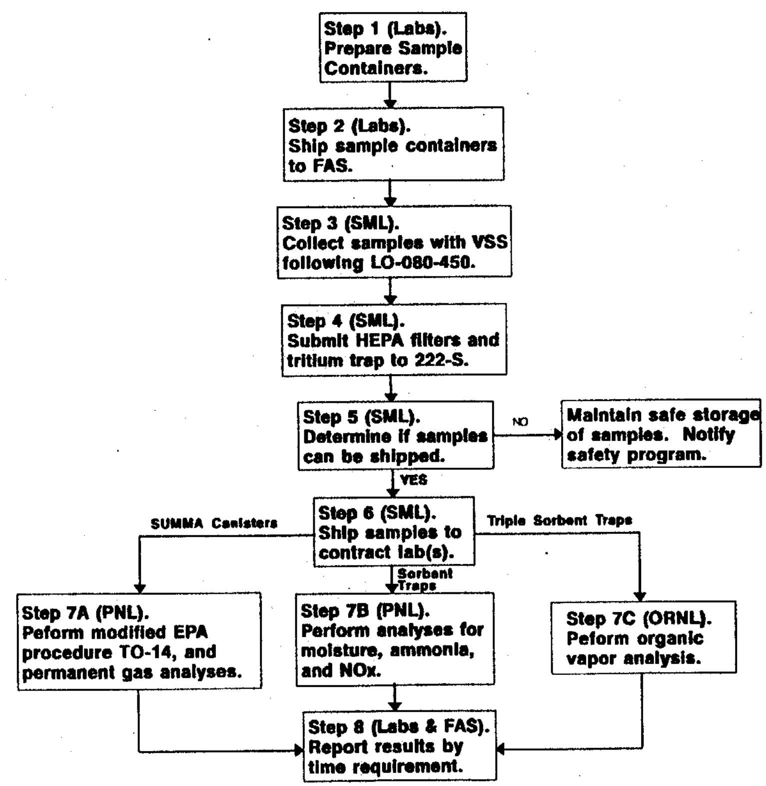

Table A-4. B-103 Sample Chemical, Physical, and Radiological Analytical Requirements

PROJECT	U-103 VAPOR	COMMENTS	REPORT FORMATS		NO. OF SAMPLE/BLANK CONTAINERS PROCESSED				
					Organization	WHC	PNL	ORNL	TOTAL
Plan Number	WHC-SD-WM-TP-288	Type 3 vapor sampling system (VSS) using heated vapor probes.	I	Early Notify	SUMMA® Canister		3/2		5
Tank	U-103		II	Process Control	Sorbent Trap System[b]		6/3		9
Program Contact	J. W. Osborne		III	Safety Screen					
TWRS Contact	R. D. Schreiber 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 @NL	ACCURACY @NL	
CGM	Flammability	CGIMX251 CGITMX410	N/A	N/A	N/A	1	N/A	N/A	>20% LFL	<10% LFL	N/A	±10%	I
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-ALO-226 PNL-TVP-09	PNL	H_2O Extraction	Sorbent Trap	6	none	3	≥ 250 ppmv	≥ 2 ppmv	±25%	70-130%	I, VI
GC/MS	Organics**	AC-MM-1-003153 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 ≥200 pCi/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	±25%	N/A	II
GC/FID	Organics	LO-080-450	FAS	Direct	On-line	N/A	N/A	N/A	N/A	N/A	N/A	N/A	II, VI

N/A: Not Applicable
b System contains individual sorbent media sections for NO_x, NH_3, & H_2O.
c Multiple samples and blanks are taken.
d Samples are spiked with surrogates.
e Action required if any compound exceed 50% IDLH.
f Includes two trip and two field blanks.
g Survey purposes 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 SAP 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) shall be used for quality assurance guidance.
2) Each analysis and media preparation procedure given in Tables A-1 and A-4 is documented by the laboratory and available to ESH&QA.
3) Any modifications made to, or deviations from, the prescribed procedures are documented and justified in the deliverable report.

The PNL tank vapor program is governed by an impact level II QA 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

The laboratory supplying the sample collection media shall initiate the chain-of-custody form in accordance with the laboratory operating procedure LO-090-443, "Chain-of-Custody for RCRA and CERCLA Protocol Samples" using unique sample label and identification numbers provided by FAS. 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 FAS 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. The sample collection media shall also remain in a controlled area under conditions specified on the chain-of-custody form.

Applicable operating procedures for the tank U-103 vapor space sampling activities are contained in work package WS-95-00005. 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 analytical laboratories 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 (Sampling and Mobile Laboratories) 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.

Sampling and Mobile Laboratories 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

Prepared and labeled sample collection containers, trip blanks, and field blanks are supplied by the performing laboratories to FAS. The SUMMA® canisters and Sorbent Trap Systems are prepared and certified following the laboratory quality control procedures identified in Table A-1. The laboratory supplying the sample collection media shall initiate the chain-of-custody form 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 FAS.

The sample receipt and control steps used in the PNL laboratories are identified in procedure PNL-TVP-07. Oak Ridge National Laboratory shipping and receiving is done by procedure CASD-OP-300-WP02. Analyses will be performed according to 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) are identified in Table A-4. If no criteria are provided in Table A-4, the performing laboratory shall perform to its Quality Assurance Plan(s).

Due to the developmental work being done with the analysis procedures and potential sample differences (between tanks), changes in procedures may be needed. These changes must be done following laboratory QA plans, documented in controlled notebooks, and referenced in the deliverable reports to ensure traceability.

A4.0 ORGANIZATION

The organization and responsibility of key personnel involved in this tank U-103 vapor sampling project are listed in Table A-5.

Table A-5: Tank U-103 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-103 Tank Characterization Plan Engineers
J. W. Osborne	TWRS Tank Vapor Issue Resolution Program	Vapor Issue Resolution Program Manager
H. Babad	TWRS Characterization Program	Tank Safety Screening Scientist
R. S. Viswanath	Field Analytical Services	Special Analytical Studies Vapor Program Technical Lead
R. D. Mahon	Field Analytical Services	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 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 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 one 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 FAS cognizant scientist or assisting scientific technician, lead sampler, or laboratory scientist or technician who signs the *received by* block on the chain-of-custody. Transfer of custodianship occurs when the custodian signs the *relinquished by* block on the chain-of-custody 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-103 vapors shall be reported as Format VI (Section A6.3). 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 (Section A6.1). 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 West Area Shift Manager of Tank Farm Operations at 373-3475 immediately. This verbal communication must be followed within 3 working days by written communication to the Tank Vapor Issue Resolution Program, the Industrial Hygiene and Safety Program, and 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/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 the Tank Vapor Safety Resolution Program, the Field Analytical Services representative, the Characterization 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 should 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 should include:

1) A summary of analytical results;
2) a description of sample device preparation (and manufacture 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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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*.