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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-BX-104.

8. RELEASE STAMP

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WHC-SD-WM-TP-296
Revision 0

Tank 241-BX-104 Tank Characterization Plan

MASTER

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

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LIST OF ACRONYMS

BX-104	Tank 241-BX-104
DQO	data quality objective
DST	double-shell tank
FIC	Food Instrument Corporation
GC/FID	Gas chromatograph/flame ionization detector
GC/MS	Gas chromatograph/mass spectrometry
NIOSH	National Institute of Occupational Safety and Health
ppmv	parts per million by volume
PUREX	Plutonium-Uranium Extraction plant
RCRA	Resource Conservation and Recovery Act
SST	single-shell tank
TOC	total organic carbon
TO-12	EPA task order protocol 12
TO-14	EPA task order protocol 14
TWRS	WHC Tank Waste Remediation System
WHC	Westinghouse Hanford Company

TANK 241-BX-104 TANK CHARACTERIZATION PLAN

1.0 SPECIFIC TANK CHARACTERIZATION OBJECTIVES

This Tank Characterization Plan will identify characterization objectives for tank BX-104 pertaining to sample collection, sample 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 applicable Data Quality Objectives identified in the following section. In addition, the current status (Section 2) and contents estimate (Section 3) of the tank is given.

1.1 Data Quality Objectives: Applicable to Tank BX-104

The sampling and analytical needs associated with the 51 Hanford Site underground storage tanks classified on one or more of the four Watch Lists (ferrocyanide, organic, flammable gas, and high heat), and the safety screening of all 177 tanks have been identified through the Data Quality Objective (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.

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 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.

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. Since tank BX-104 is identified as a non-Watch List tank, only the safety screening DQO applies to current sampling and analytical requirements.

DQO's concerned with fugitive vapor emissions from tank BX-104 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). 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.

2.0 RELEVANT SAFETY INFORMATION

Resolution of tank vapor safety issues involve the identification of potential flammable and fugitive vapor emissions from tanks which could become worker health and safety hazards.

2.1 Tank Status

Single-shell tank BX-104 is classified as a non-Watch List tank. The tank is sound, and was interim stabilized in 1989. To prevent further waste addition, intrusion prevention was completed. The tank is out of compliance with regard to waste temperature monitoring since internal tank temperature cannot be measured with present equipment. The last temperature measurement of 31°C was recorded in October, 1980 (Hanlon 1994). Recent waste level measurements have maintained a consistent level of 80.3 to 81.3 cm (Brevick et al. 1994).

This tank is estimated to contain 375,000 L (99,000 gallons) of non-complexed waste. The waste is comprised of 364,000 L (96,000 gallons) of sludge containing 113,000 L (30,000 gallons) of drainable interstitial liquid. The remaining 11,000 L (3,000 gallons) is supernatant liquid (Hanlon 1994).

2.2 Tank Monitoring Activities

Waste level measurements are taken on a daily basis through riser 8 using an automatic FIC gauge. Internal tank temperature is not available. Six active dry wells monitor radiation in the surrounding soil (Hanlon 1994).

3.0 SUMMARY OF HISTORICAL INFORMATION FOR TANK BX-104

Included in this section are a physical description of tank BX-104, its process history, and recorded sampling events.

3.1 Configuration

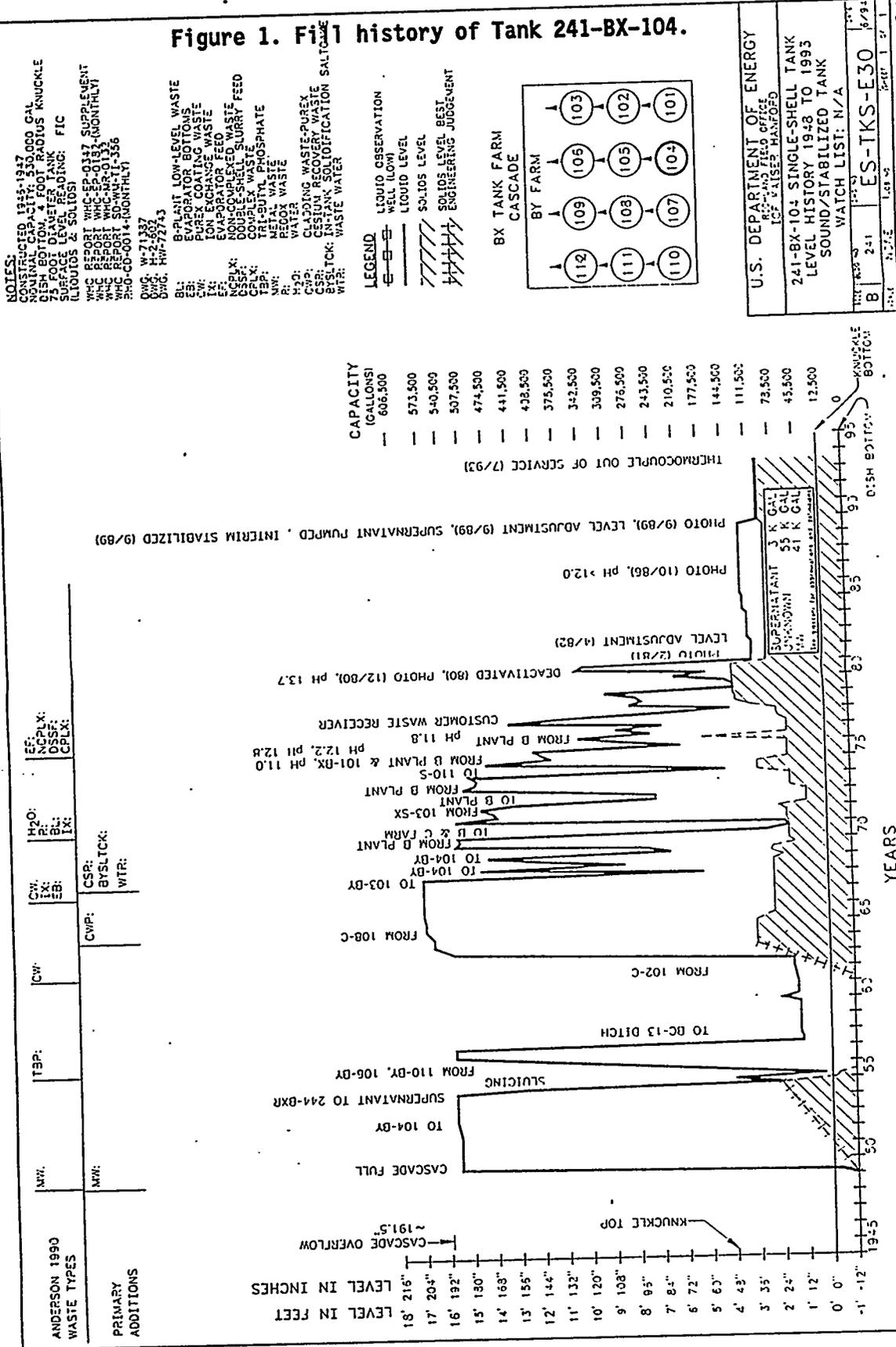
Tank BX-104 is one of 12 single-shell tanks in the 200 East area BX Farm constructed during 1946-47. It is 23 meters (75 ft.) in diameter with a concave base and has a 2.01 million liter (530,000 gal.) tank capacity. The tank was constructed as the primary tank in a cascade containing tanks BX-105 and BX-106; tank BX-106 may also cascade to primary cascade tank BY-103 (Brevick et al. 1994).

3.2 Process History

Tank BX-104, beginning service in the first quarter of 1949 as a primary cascade receiver, was filled to capacity with metal waste. After sluicing the metal waste to recover uranium in 1954, the tank may have been filled with waste from both the U Plant uranium recovery process and from ferrocyanide scavenged waste receivers BY-106 and BY-110. After a short period of settling, the supernatant was pumped to the BC-13 ditch in 1957. The tank received PUREX cladding waste in 1962-65, likely from tanks C-102 and C-108 and cesium recovery waste from B Plant in 1967-76. It also may have received saltcake waste from BY Farm in-tank solidification units. During the period 1976-80 tank BX-104 was identified as containing both evaporator feed and double-shell slurry feed waste from the 242-A evaporator. A liquid level adjustment occurred in 1980, after which the tank was deactivated. Additional liquid level adjustments occurred in 1982 and 1989. Figure 1 summarizes the fill history of tank BX-104 from 1945 to the present (Brevick et al. 1994).

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Figure 1. Fill history of Tank 241-BX-104.



3.3 Historical Sampling Events

Several supernatant samples were taken during 1974-5 in preparation for the startup of the 242-S Evaporator (Brevick et al. 1994). These were dilute aqueous solutions of mainly sodium nitrate, sodium nitrite, and sodium carbonate. Two cores, consisting of three segments (Core 1, 86 cm in length) and two segments (Core 2, 81 cm in length), were retrieved in February 1986 from risers 1 and 8, respectively when the tank contained a 102 cm level of waste with a liquid surface.

Observations made during extrusion and phase separation indicate that the top layer was primarily aqueous liquid with a small amount of solids and organic material. The middle portion contained much more solid material (>50%) and a significant fraction of organic liquid (up to 14%). The bottom layer was a thick sludge containing less than 10% drainable liquid. From each core a solids composite sample was made. In addition, a drainable liquid composite was blended from the aqueous phase of the Core 2 segments. No results, except the volumes recovered, were reported for the organic phase. The solid composites were prepared by a sequential liquid dissolution using a water leach, an acid leach, and finally a fusion dissolution. The acid leach solution was proportionally combined with the fusion dissolution sample solution prior to analysis. The analytical results for the three composites is given in Table 1 (Weiss and Schull 1988).

Table 1. Tank BX-104 Core Composite Analyses Results.

Sample ID	Core #1 Composite Solids		Core #2 Composite Solids		Result Total ^a Avg and Standard Deviation (SD)		Core #2 Liquid Comp.
Physical Properties, Direct preparation							
Appearance	Brown		Brown with small hard chunks		N/A		Gr Yellow
Bulk Density (g/ml)	1.85		1.76		1.81	0.06	1.31
pH	11.0		11.0		11.0	0	>12.0
Therm wt loss							
Amb. to 400°C	41.9%		45.6%		43.8%	2.6%	65.2%
400 to 1000°C	6.4%		5.8%		6.1%	0.4%	17.3%
Radiation mR/h	250		250		250	0	100
Chemical Constituents, µg/g							
Result	Water	Acid/fus	Water	Acid/fus	Tot. Avg	SD	Direct
Aluminum	1,140	39,600	1,250	52,800	47,400	9,330	3,430
Barium	0.634	1,200	1.33	2,090	1,650	620	<0.420
Bismuth	<11.5	1,690	<11.5	1,090	1,390	420	<9.24
Boron	5.71	93,400	5.65	0 ^b	93,600	N/A	11.3
Cadmium	<2.6	<21.7	<2.6	<22.4	<25.0 ^c	N/A	5.74
Calcium	44.3	3,840	75.8	5,730	4,840	1,360	110
Chromium	763	2,330	421	4,060	3,790	980	1,450

Table 1. Tank BX-104 Core Composite Analyses Results.

Sample ID	Core #1 Composite Solids		Core #2 Composite Solids		Result Total ^a Avg and Standard Deviation (SD)		Core #2 Liquid Comp.
	Water	Acid/fus	Water	Acid/fus	Tot. Avg	SD	Direct
Cobalt	0 ^b	15.1	0 ^b	8.4	11.8	4.7	0.857
Copper	14.5	105	1.5	70.2	95.4	33.4	43.7
Iron	1.07	9,620	1.3	5,060	7,340	3,220	2.08
Lead	<8.5	542	<12.4	582	562	28	13.6
Magnesium	0.356	1,900	1.7	3,040	2,470	570	0.134
Manganese	<20.9	810	<20.9	1,110	960	150	<16.8
Nickel	14.2	181	19.4	95.7	155	57	47.6
Phosphorus	3,890	611	2,500	510	3,760	1,050	1,350
Potassium	492	464	511	644	1,060	140	1,510
Silicon	135	25,700	127	38,800	32,400	9,200	119
Silver	<1.1	107	<1.1	69.8	88	26	<0.840
Sodium	53,900	15,900	44,600	22,900	68,700	1,600	124,500
Strontium	0 ^b	77.1	0.112	46.5	61.9	21.6	0.160
Zinc	0 ^b	121	0 ^b	102	112	13	NR
Zirconium	<4.8	1,130	<4.8	1,360	1,250	160	<3.87
Uranium	3.80	34,800	2.83	14,000	24,400	14,700	2.56
Nitrate	41,900	NR	35,000	NR	38,500	4,900	111,000
TOC	1,780	NR	2,710	NR	2,250	660	4,721
Radionuclide Constituents, $\mu\text{Ci/g}$							
^{239,240} Pu	2.12E-3	0.339	1.33E-3	0.389	0.366	0.035	1.37E-2
14C	6.28E-4	NR	<3.5E-4	NR	6.28E-4	N/A	1.23E-3
90Sr	0.197	375	0.362	15.4	265	156	2.07
99Tc	3.35E-2	0 ^b	3.23E-2	0 ^b	3.29E-2	0.08E-2	9.32E-2
241Am	<2.05E-3	<0.225	5.75E-3	1.03	1.03	N/A	<6.2E-3
60Co	1.50E-2	6.20E-2	2.84E-2	<3.17E-2	7.70E-2	N/A	<7.6E-2
137Cs	59.2	15.9	64.3	59.5	99.6	35.6	168
129I	5.20E-5, direct		<3.50E-5, direct		5.20E-5	N/A	8.40E-5
Total Gamma	60.7	25.4	66.4	80.1	117	43	171

NR = Not reported N/A = Not Applicable

^aThe result total is the sum of the two preparation results for a core (when available), excluding results below the detection limit. The Core #1 and Core #2 result totals are then averaged.

^bReported as zero with no detection limit indicated.

^cNot an average but a maximum value.

In June of 1990, vapor samples were collected when unpleasant "fuel oil type" odors were reported emitting from tanks BX-104 and B-110. A 5-L vapor sample was drawn from each of tanks BX-104, B-110, and five other BX Farm tanks' breather exhaust ports through charcoal tubes. NIOSH Reference Methods 1003, 1500, 1501, and 1550 utilizing GC/FID were used to detect benzene and total hydrocarbons at the WHC Environmental Health Sciences laboratory. Benzene was not detected at levels above the 0.2 ppm method detection limit in any sample. Residual hydrocarbon concentrations of $\leq 500 \mu\text{g}/\text{m}^3$ were reported for all BX Farm tanks except BX-104, which was $210,000 \mu\text{g}/\text{m}^3$. The major organic components in the BX-104 sample were identified as C_{10} - C_{22} paraffins using CG/MS. Ammonia and acetone detector tube samples from tank BX-104 indicated the presence of ammonia and an absence of acetone (Wegener 1990).

In contradiction to these results, the Oregon Graduate Institute of Science and Technology analyzed vapor samples, collected in canisters on January 19, 1993, from tank BX-104. Both TO-12 and TO-14 EPA procedures were used on six tank samples, one trip blank, and one field blank. All TNMHC levels were below $10,000 \mu\text{g}/\text{m}^3$, the majority of which was identified as small-chain paraffins ($\leq \text{C}_6$). These samples were used to collect screening data and is not qualified data. Table 2 gives results for both the 1990 and 1993 (Rasmussen 1993) tank BX-104 samples. The representativeness of the samples is in question since neither sampling event used the current Type 2 or Type 3 sample collection procedures.

3.4 Expected Tank Contents

An in-tank photograph montage from September 1989 is incomplete and hazy but shows the waste surface to be a pock-marked glossy black sludge with small pools of liquid at the bases of the turbine pump and FIC gauge. The photographs were taken about the same time the tank was jet pumped and interim stabilized and may not represent the tank's current contents (Brevick et al. 1994).

Approximately 83,000 L of supernatant have been removed since the 1986 core sampling event. Therefore, the analytical results of the solids composites presented in Table 1 should be representative of the present contents of the tank after consideration of radionuclide decay, chemical reactivity, and remaining interstitial liquid. The tank is expected to contain a large amount of insoluble aluminum and other metal silicates, uranium oxide, and soluble nitrates and phosphates. The majority of the organic liquid, expected to be present as a floating layer, was obtained from the middle portion of the 1986 cores. Therefore, this organic fluid may be a contaminate originating from core sampling equipment or the tank may contain pockets of interstitial organic liquid.

Organic vapors, detected as odors in the air near tank BX-104 by field workers and at $210,000 \mu\text{g}/\text{m}^3$ in the tank's exhaust port from a charcoal tube sampler in the spring of 1990, provide evidence that organic material is present within tank BX-104. The waste in tank BX-104 had been disturbed by a jet pump only a few months before these odors were detected and may have exposed organic liquids to waste surface. Increasing ambient surface and seasonal internal tank temperatures would then cause an increase in waste surface volatility. A much lower concentration of organic vapors was measured in the winter of 1993.

Table 2. Tank BX-104 Vapor Sample Analyses Results.

Organics, $\mu\text{g}/\text{m}^3$ unless noted otherwise	1990 Vapor Sample	1993 Vapor Samples	
	Result	Result Avg	Standard Deviation
NIOSH residual hydrocarbons (GC/FID)	210,000	NR	NR
TNMHC by T0-12	NR	8,900	440
TNMHC by T0-14	NR	6,810	190
Benzene	≤ 0.2 ppm	5.3	0.5
Ammonia	Detected	NR	
Acetone	Not Detected	NR	
Ethane	NR	770	19
Ethylene	NR	1,041	26
Propane	NR	998	22
Propene	NR	236	10
i-Butane	NR	115	3
i-Butene	NR	191	7
1, 3-Butadiene	NR	8.5	0.5
n-Butane	NR	929	20
i-Pentane	NR	174	13
n-Pentane	NR	471	16
2-Methylpentane	NR	181	7
n-Hexane	NR	166	7
Toluene	NR	11	2
Alkanes (Paraffins)	NR	4,000	80
Alkenes (Olefins)	NR	1,450	630
Aromatics	NR	16.7	2.0
Total Identified Hydrocarbons	NR	5,720	120
Total Unidentified Hydrocarbons	NR	1,090	250

NR = Not reported

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APPENDICES

TANK 241-BX-104 SAMPLING AND ANALYSIS PLAN

SAMPLE EVENT A

**VAPOR SAMPLING
IN FISCAL YEAR 1995**

SAMPLE EVENT A: VAPOR SAMPLING IN FISCAL YEAR 1995

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LIST OF ACRONYMS

BX-104	Tank 241-BX-104
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CGM	combustible gas meter
DOT	Department of Transportation
DQO	data quality objective
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
IC	ion chromatography
IDLH	immediately dangerous to life and health
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
RCRA	Resource Conservation and Recovery Act
SML	Sampling and Mobile Laboratories
SUMMA®	registered trademark for passivated stainless steel canister
TCP	Tank Characterization Plan
TNMHC	Total Non-Methane Hydrocarbons
TO-14	EPA task order protocol 14
TOC	total organic carbon
TWRS	Tank Waste Remediation System
VSS	vapor sampling system
WHC	Westinghouse Hanford Company

TANK 241-BX-104 VAPOR SPACE SAMPLING AND ANALYSIS PLAN

1.0 INTRODUCTION

Vapor samples are used to identify potential flammable and fugitive vapor emissions from the tanks which could become worker health and safety issues. Sampling of the vapor space can identify: 1) volatile compounds above the surface of the waste; and 2) the amount of gases generated by chemical or radiolytic reactions within the waste.

2.0 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 BX-104. 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 TCP made during sample receipt, preparation, and analysis shall be documented and justified in the deliverable report.

2.1 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 following the quality assurance/quality control format given in Section 3.1.

2.2 Flammability of Vapor Space Gases

Prior to this sampling event and performing any 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 is identified 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 measures 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).

2.3 Sample Collection

In fiscal year 1995, the tank BX-104 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)". The riser selected for access to the tank vapor space is identified

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in work package ES-94-904. 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 BX-104.

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	6
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 ₂ O	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

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

2.4 Radiation Screening and Sample Transport

All vapor samples shall be stored at the 222-S Laboratory Annex 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 reproduced in Table A.4.

The results from the radiation screening are submitted to and shall be evaluated by Sampling and Mobile Laboratories to ensure the samples meet the criteria specified in Table A.3. Sampling and Mobile Laboratories 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 ³	N/A	5,450 mL/min	30 min.
01	Collect ambient air sample SUMMA #1	Upwind of BX-104	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 ⁴	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 SUMMA #6	Port 12	N/A	1 min.
08	Collect SUMMA #7	Port 14	N/A	1 min.
09	Collect SUMMA #8	Port 16	N/A	1 min.
10	Collect Triple Sorbent Trap (TST) sample #1	Sorbent line 9	50 mL/min	4 min.
11	Collect TST sample #2	Sorbent line 10	50 mL/min	4 min.
12	Collect TST sample #3	Sorbent line 8	50 mL/min	4 min.
13	Open, close, & store TST Field Blank #1	In VSS truck	0 mL/min	
14	Collect TST sample #4	Sorbent line 10	50 mL/min	4 min.
15	Collect TST sample #5	Sorbent line 9	200 mL/min	5 min.
16	Collect TST sample #6	Sorbent line 10	200 mL/min	5 min.
17	Collect TST sample #7	Sorbent line 8	200 mL/min	5 min.
18	Collect TST sample #8	Sorbent line 10	200 mL/min	5 min.
19	Collect TST sample #9	Sorbent line 9	200 mL/min	20 min.
20	Open, close, & store TST Field Blank #2	In VSS truck	0 mL/min	N/A
21	Collect TST sample #10	Sorbent line 10	200 mL/min	20 min.
22	Collect TST sample #11	Sorbent line 8	200 mL/min	20 min.
23	Collect TST sample #12	Sorbent line 10	200 mL/min	20 min.
24, 25	Store TST Trip Blanks #1 & #2	None	None	None
26	Collect NH3/NOx/H2O Sorbent Trap #1	Sorbent line 9	200 mL/min	15 min.
27	Collect NH3/NOx/H2O Sorbent Trap #2	Sorbent line 10	200 mL/min	15 min.
28	Collect NH3/NOx/H2O Sorbent Trap #3	Sorbent line 8	200 mL/min	15 min.
29	Collect NH3/NOx/H2O Sorbent Trap #4	Sorbent line 10	200 mL/min	15 min.
30	Collect NH3/NOx/H2O Sorbent Trap #5	Sorbent line 9	200 mL/min	15 min.
31	Collect NH3/NOx/H2O Sorbent Trap #6	Sorbent line 10	200 mL/min	15 min.
32, 33, 34	Store NH3/NOx/H2O Trap Trip Blanks #1, #2, & #3	None	None	None
35	Remove upstream HEPA Filter from HEPA transfer box	Upstream of box	Continuous	
36	Remove downstream HEPA Filter from HEPA transfer box	Downstream of box	Continuous	
37	Remove upstream HEPA Filter from VSS	Upstream of VSS	Continuous	
38	Remove downstream HEPA Filter from VSS	Downstream of VSS	Continuous	

³Not required if ambient air purge incorporated in VSS setup.

⁴Additional 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 B/y	Tritium	Units
PNL Analytical Chemistry Laboratory	≤ 100	≤ 400	none specified	pCi/g
Oak Ridge National Laboratory	≤ 135	≤ 450	none specified	pCi/g
WHC-CM-2-14 ⁴	≤ 60	≤ 200	none specified	pCi/g

⁴ 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 3.1.

2.5 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 BX-104 vapor space and contained within the SUMMA® canisters shall be analyzed for total non-methane hydrocarbons following modified EPA procedure TO-14 and the permanent gases CO₂, CO, CH₄, H₂, and N₂O 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.

Any analyses prescribed by this document, but not performed, shall be identified and justification for non-performance written 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 procedures PNL-TVP-07 and CASD-AM-300-NP02 (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 to the 222-S Laboratory, the vapor sample containers for locked storage, and the HEPA filters and Tritium Trap for radiological survey.
- Step 5
SML: Using radiological survey report results, determine if samples are acceptable to ship offsite (see Section 2.4).
- Step 6
SML: If determined to be acceptable by offsite laboratory requirements and WMC-DM-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.
Labs: Perform laboratory analyses.
- Step 7
A. SUMMA® Canisters (PNL): Perform modified full scan EPA-TO-14. Perform permanent gas analysis for the following: H₂, CO, N₂O, CH₄, CO₂.
B. Sorbent Traps (PNL): Perform gravimetric analysis for moisture. Perform selective electrode analysis for NH₃. Analyze NO and NO₂ Traps.
C. Triple Sorbent Traps (ORNL): Perform organic vapor analysis.
- Step 8
Labs and SML: Following the Section 6.0 reporting requirements, deliver a Format VI Report to the Vapor Issue Resolution Safety Program.

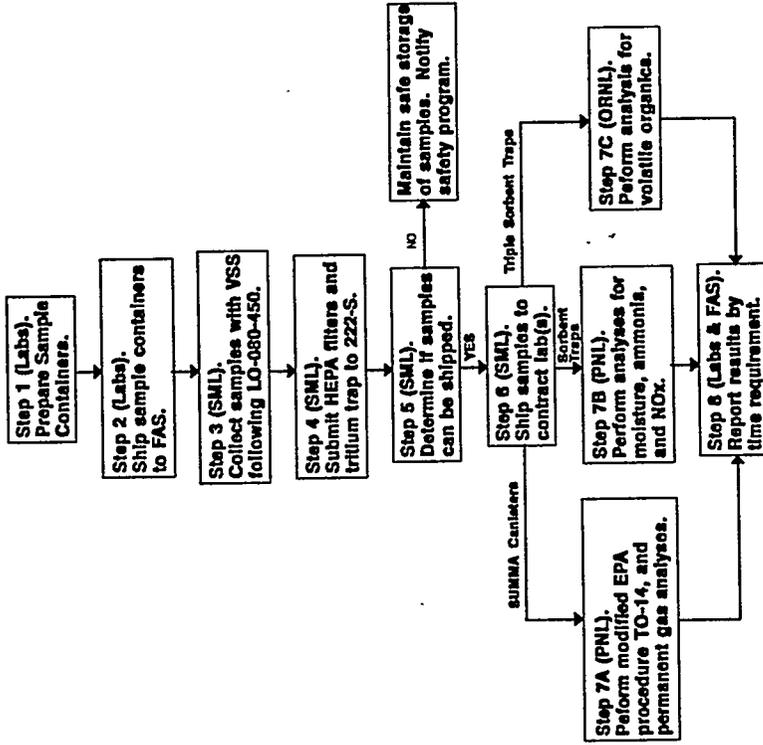


Table A.4. BX-104 Sample Chemical, Physical, and Radiological Analytical Requirements

PROJECT		BX-104 VAPOR		COMMENTS		REPORT FORMATS		NO. OF SAMPLE/BLANK CONTAINERS PROCESSED					
Plan Number	MHC-SD-WM-TP-296	Type 3 vapor sampling system (VSS) using heated vapor probes.	Early Notify	Organization	WHC	PNL	ORNL	TOTAL				TOTAL	
Tank	BX-104		Process Control	SUMMA® Canister	3 ^a	3/2		8					
Program Contact	J. M. Osborne		Safety Screen	Sorbent Trap System ^b		6/3		9					
THRS Contact	B. C. Carpenter C. S. Homi		Waste Management	Triple Sorbent Trap			12/4	16					
Lab Project Coordinator	S. C. Goheen (PNL) R. A. Jenkins (ORNL)		RCRA Compliance	HEPA Filter	4			4					
			Special	Tritium Trap	1			1					
PRIMARY ANALYSES													
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	REPORT FORMAT
GC/MS	Flammability	GGIMX251 GGIMX410	N/A	N/A	N/A	1	N/A	N/A	>20% LFL	<10% LFL	N/A	N/A	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 ₂ O	PNL-TVP-05 PNL-TVP-02	PNL	Direct	SUMMA®	3	none	2	N/A ≥ 20% LFL ≥ 20% LFL not available	not available	±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 ₂ O 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 ₂ O	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 ₂ O Extraction	Sorbent Trap	6	none	3	≥ 250 ppmv	≥ 2 ppmv	±25%	70-130%	I, VI
GC/MS	Organics**	AC-MM-1-003153 AC-MM-1-003157	ORNL	Thermal Desorption	Triple Sorbent Trap	12	all	4	≥ 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	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

- a Three canisters will be archived at PNL until arrangements can be made for transport and analytical work at the OGIST laboratory.
- b System contains individual sorbent media sections for NO_x, NH₃, & H₂O.
- 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.

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

3.0 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, American Society of Mechanical Engineers NQA-1, EPA QAMS-005/80 or United States 10 CFR 830.120.
- 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 has an impact level II Laboratory Quality Assurance Plan (Barnes 1994) written to comply with 5700.6C. ESH&QA will qualify laboratories for continued use by the TWRS Characterization program after receipt of the Laboratory quality assurance plans, followed by an audit and corrective action phase.

3.1 Sampling Operations

Field Analytical Services shall provide unique sample label and identification numbers to the laboratories supplying the sample collection medium. 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 BX-104 vapor space sampling activities are contained in work package ES-94-904. 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:

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- 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.

3.2 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 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 Field Analytical Services.

The sample receipt and control steps used by the PNL laboratories are identified in procedure PNL-TVP-07. Oak Ridge National Laboratory shipping and receiving is done by procedure CASD-AM-300-WP02. The analytical procedures used are identified 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 analyze quality control samples according 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 documented in controlled notebooks and referenced in the deliverable reports to ensure traceability.

4.0 ORGANIZATION

The organization and responsibility of key personnel involved in this tank BX-104 vapor sampling project are listed in Table A.5.

Table A.5. Tank BX-104 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	BX-104 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 Sampling Technical Support
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)
East Area Shift Operations Manager	Tank Farm Operations	East Tank Farm Point of Contact if Notification Limit is Exceeded (373-2689)

5.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

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 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.

6.0 DELIVERABLES

The Pacific Northwest Laboratory, Oak Ridge National Laboratory, and Sampling and Mobile Laboratories VSS sampling and analyses of tank BX-104 vapors shall be reported as Format VI (Section 6.3). In addition, the analytical laboratories shall receive Format II reports from Sampling and Mobile Laboratories as described in Section 6.2. Any analyte exceeding the notification limit prescribed in Table A.4 shall be reported as Format I (Section 6.1). 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).

6.1 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.

6.2 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.

6.3 Format VI Reporting

All Format VI reports shall be delivered to J. W. Osborne of the Tank Vapor Safety Resolution Program, R. S. Viswaneth of Field Analytical Services, 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 demonstrably 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 ($\mu\text{g}/\text{m}^3$, 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 chain-of-custody forms attached.

7.0 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. 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.

8.0 REFERENCES

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- Bellus, T. H., 1993, *Configuration of Hewlett Packard (HP) 5890 Series II Gas Chromatograph (GC) for DML1*, (internal memo 12240-SAA93-039 to L. L. Lockrem, July 10), Westinghouse Hanford Company, Richland, Washington.
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- United States Code of Federal Regulations, 10 CFR Part 830, *Nuclear Safety Management*; Section 120, *Quality Assurance Requirements*.
- Whelan, T. E., 1994, *TWRS Characterization Program Quality Assurance Program Plan*, WHC-SD-WM-QAPP-025, Westinghouse Hanford Company, Richland, WA.