

# **PERMIT ATTACHMENT BB**

Waste Analysis Plan – Section 6 of the Permit  
Application;  
and  
Analytical Methods – Appendix D of the Permit  
Application

Permit Number: WA 7890008967

The following listed documents are hereby incorporated, in their entirety, by reference into this Permit. Some of the documents are excerpts from the Permittees' DBVS Facility Research, Development, and Demonstration Dangerous Waste Permit Application dated May 10, 2004 (document #04-TED-036); hereafter called the Permit Application. Ecology has, as deemed necessary, modified specific language in the attachments. These modifications are described in the permit conditions (Parts I through V), and thereby supersede the language of the attachment. These incorporated attachments are enforceable conditions of this Permit, as modified by the specific permit conditions.

**Waste Analysis Plan – Section 6 of  
the Permit Application**

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1    **6.0    WASTE ANALYSIS PLAN**

2    **6.1    GENERAL**

3    The Waste Analysis Plan (WAP) provides the basis for measuring the adequacy of waste  
4    treatment and assists in optimizing the waste treatment operation based on treated waste analysis  
5    results. It also provides information on secondary waste streams to determine the required type  
6    and level of treatment or the appropriate disposal path.

7    The WAP objective is to develop a sampling approach for the final vitrified waste form to ensure  
8    compliance with the waste acceptance criteria of the IDF or another permitted disposal facility  
9    and the land disposal restrictions listed in WAC 173-303-140. As depicted in Figure 6-1, the  
10   WAP identifies assumptions, sample points, sampling methods and frequencies, and analytical  
11   objectives.

12   **6.2    WASTE FEED CHARACTERISTICS AND SAMPLING**

13   **6.2.1   Dangerous Waste Designations**

14   Tank 241-S-109 has the dangerous waste designations listed in Table 6-1, per the SST Part A  
15   Form 3 (DOE/RL-88-21).

16   Process knowledge, process history, pertinent literature on waste chemistry and tank history, and  
17   analysis on the waste retrieved during Phase 1 and Phase 2 will be used to address the Dangerous  
18   Waste Codes D001 (Ignitability), D002 (Corrosivity), and D003 (Reactivity) before transfer to  
19   the DBVS to ensure characteristics associated with these waste codes do not exist in the waste  
20   feed. Sections 6.2.4 and 6.2.5 discusses sampling frequency in greater detail.

21   **6.2.2   Waste Physical Properties**

22   The DBVS has been designed to receive waste that has the physical properties listed in Table 6-2  
23   (RPP-17403). The waste will not contain a visible separate organic phase.

**Table 6-1. Dangerous Waste Designation and Sampling/Analysis Strategy (2 pages)**

Waste Code	Chemical/Characteristic (40 CFR 268.40)	Strategy			
		Phase 1		Phase 2	
		Waste Feed	Vitrified Waste	Waste Feed	Vitrified Waste
D001	Ignitable Characteristic Waste	√	1	2	2
D002	Corrosive Characteristic Waste	√	1	2	2
D003	Reactive Characteristic Waste	√	1	2	2
D004	Arsenic	√	√	2	3
D005	Barium	√	√	2	3
D006	Cadmium	√	√	2	3
D007	Chromium (total)	√	√	2	3
D008	Lead	√	√	2	3
D009	Mercury	√	√	2	3
D010	Selenium	√	√	2	3
D011	Silver	√	√	2	3
D018	Benzene	√	√	2	3
D019	Carbon Tetrachloride	√	√	2	3
D022	Chloroform	√	√	2	3
D028	1,2-Dichloroethane	√	√	2	3
D029	1,1-Dichloroethylene	√	√	2	3
D030	2,4-Dinitrotoluene	√	√	2	3
D033	Hexachlorobutadiene	√	√	2	3
D034	Hexachloroethane	√	√	2	3
D035	Methyl ethyl ketone	√	√	2	3
D036	Nitrobenzene	√	√	2	3
D038	Pyridine	√	√	2	3
D039	Tetrachloroethylene	√	√	2	3
D040	Trichloroethylene	√	√	2	3
D041	2,4,5-Trichlorophenol	√	√	2	3
D043	Vinyl chloride	√	√	2	3
F001 F002 F003 F004 F005	Acetone Benzene n-Butyl alcohol Carbon disulfide Carbon Tetrachloride Chlorobenzene Cresol – mixed isomers Cyclohexanone o-Dichlorobenzene Ethyl Acetate Ethyl Benzene Ethyl ether Isobutyl alcohol Methanol Methylene chloride Methyl ethyl ketone Methyl isobutyl ketone Nitrobenzene	√	√	2	3

**Table 6-1. Dangerous Waste Designation and Sampling/Analysis Strategy (2 pages)**

		Strategy			
	Pyridine Tetrachloroethylene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane 1,1,2-Trichloro-1,2,2-trifluoroethane Trichloroethylene Trichloromonofluoromethane Xylenes – mixed isomers				
WP01	Persistent Dangerous Waste	4	4	4	4
WP02	Extremely Persistent Dangerous Waste	4	4	4	4
WT01	Toxic Dangerous Waste	5	5	5	5
WT02	Extremely Toxic Dangerous Waste	5	5	5	5

<sup>1</sup> Analyze if exists in waste feed

<sup>2</sup> Analyze first waste feed tank

<sup>3</sup> Analyze first ten containers, then randomly

<sup>4</sup> Book designate per WAC 173-303-100(6)

<sup>5</sup> Book designate per WAC 173-303-100(5)

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**Table 6-2. Waste Feed Physical Properties**

Property	Value
Density	1.2 – 1.3 g/ml (10.0 – 10.8 lb/gal)
Viscosity	10 cP Maximum at 25 °C (77 °F)
Percent Solids	<3%

3  
4

### 6.2.3 Waste Feed Chemical and Radiochemical Properties

#### 6.2.3.1 Saltcake Key Chemical and Radiological Contaminants.

6 The average concentrations of major constituents important for glass performance and key  
7 contaminants in the Tank 241-S-109 saltcake waste are shown in Tables 6-3 and 6-4. The major  
8 constituents listed in Table 6-3 are important to ensure proper glass processing and good glass  
9 performance. The key contaminants indicated in Table 6-3, column 3, are important to ensure  
10 compliance with LDRs listed in 40 CFR 268 and to determine the compliance with performance  
11 assessment objectives. Concentrations have been normalized to 5 M sodium, the sodium  
12 concentration in the expected feed to the bulk vitrification process. Note that not all of these  
13 constituents will be present in the retrieval stream, since some of the solids (in particular the  
14 metals and transuranics) have very low solubility and will mostly be left in the tank or removed  
15 via a solids/liquid hydroclone separator.

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**Table 6-3. Chemical Constituents/LDR Contaminants in Average Tank 241-S-109 Saltcake Waste**

Constituent/ Contaminant	Average Saltcake Normalized to 5 M Na <sup>1</sup> (µg/ml)	Key Contaminants Land Disposal Restriction Level)
Aluminum	1300	
Calcium	49	
Chloride	270	
Total Chromium	770	√ (0.75 mg/L TCLP)
Fluoride	110	
Iron	270	
Potassium	170	
Manganese	8	
Nickel	6	√ <sup>2</sup>
Nitrite <sup>3</sup>	3000	√ <sup>2</sup>
Nitrate <sup>3</sup>	290000	√ <sup>2</sup>
Lead	29	√ (0.75 mg/L TCLP)
Phosphate	5500	
Silica	160	
Sulfate	3700	
Total Inorganic Carbon as CO <sub>3</sub>	11000	
Total Organic Carbon	320	
Total Uranium	28	√ <sup>2</sup>

<sup>1</sup>The composition is based on the saltcake portion of the Tank 241-S-109 Best Basis Inventory (BBI 2001), normalized to 5 M Na (115000 µg/ml)

<sup>2</sup>Not listed as an LDR contaminant

<sup>3</sup> Destroyed or removed in the vitrification process

TCLP = Toxic Characteristic Leaching Procedure

**Table 6-4. Key Radionuclide Contaminants in Average Tank 241-S-109 Saltcake Waste**

Contaminant	Average Saltcake Normalized to 5 M Na <sup>1</sup> (μCi/ml)
TRU (total)	3.7 x 10 <sup>-3</sup>
Cesium-137	6.2 x 10 <sup>0</sup>
Strontium-90	2.3 x 10 <sup>0</sup>
Technetium-99	4.7 x 10 <sup>-2</sup>
Cobalt-60	5.5 x 10 <sup>-3</sup>
Europium-154	2.5 x 10 <sup>-2</sup>
Iodine-129	9.1 x 10 <sup>-5</sup>

<sup>1</sup>The composition is based on the saltcake portion of the Tank 241-S-109 Best Basis Inventory (BBI, 2001), normalized to 5 M Na (115000 μg/ml)

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**6.2.3.2 Expected Concentrations of Retrieved Waste Streams**

The composition of the waste retrieved during different phases of RD&D operations depends on the relative amounts of interstitial liquid and the dissolution brine retrieved. The interstitial liquid is the liquid phase that currently exists in the tank and contains the highly soluble components including the bulk of the Cs-137. The composition of this liquid is constant and established by analysis of saltwell grab samples. The dissolution brine is the liquid phase formed as the solid saltcake is dissolved through the addition of water and is composed of the relatively soluble components in the salt phase. The composition of the dissolution brine is established through modeling and changes over the course of the retrieval process. The exact ratio of these liquids retrieved in the different phases is not known but an approximate composition can be established via a general understanding of the effects of dissolution on the waste.

Retrieval operations that attempt to remove cesium from Tank 241-S-109 and route it to the DST system will focus on removing the interstitial liquid. These operations will maximize removal of the liquor and minimize the addition of water that might create dissolution brine that will dilute the liquor. The interstitial liquid concentrations for any contaminant that might end up in the glass waste form (e.g., all contaminants other than NO<sub>2</sub> and NO<sub>3</sub> which are destroyed or removed during vitrification) are bounded by the concentrations in the interstitial liquid.

The Phase 1 and Phase 2 retrieval operations will obtain an acceptable feed for the DBVS. These retrieval phases will maximize the quantity of dissolution brine retrieved while minimizing the incorporation of the interstitial liquid. This strategy will minimize the concentration of all key contaminants other than NO<sub>2</sub> and NO<sub>3</sub> that are collected as the salt dissolves but are destroyed or removed during vitrification. In all cases, the contaminant concentrations of all key contaminants listed in Tables 6-3 and 6-4 (other than NO<sub>2</sub> and NO<sub>3</sub>) will be lower than those in the interstitial liquid.

1 **6.2.3.3 Compositions of Interstitial Liquid and Dissolution Brine.** Tables 6-5 and 6-6 show  
2 the concentrations of key constituents/contaminants that are expected for the interstitial liquid  
3 and the dissolution brine at three different points in the retrieval process. The brine and liquor  
4 have been normalized to 5 M sodium. The interstitial liquid composition is based on grab  
5 samples taken in the saltwell.

6 The dissolution brine compositions are based on the Environmental Simulation Program (ESP), a  
7 chemical thermodynamic model, which used the Tank 241-S-109 Best Basis Inventory  
8 (BBI 2001) as input. The contaminants F, NO<sub>3</sub>, PO<sub>4</sub>, SO<sub>4</sub>, CO<sub>3</sub>, and TOC (in the form of  
9 oxalate) are present with sodium primarily in the soluble solids fraction. Sodium nitrate  
10 (NaNO<sub>3</sub>) is the dominant solid and produces most of the solute in the dissolution brine. Because  
11 sodium phosphate, sulfate, and carbonate are present in small quantity, they are entirely  
12 dissolved early in the dissolution process, subsequently washing out of the waste. Sodium  
13 fluoride and oxalate salts are also present in small quantity, but their dissolution is effectively  
14 suppressed by the other salts until middle or late dissolution, so their concentrations rise later in  
15 the process. Contaminants that do not dissolve in water are excluded from the dissolution brine,  
16 as are contaminants that are present entirely in dissolved form in the original waste. The key  
17 contaminants excluded because the solid forms have very low solubility in water are Al, Ca,  
18 Cr(III), Fe, Mn, Ni, Pb, Si, U, TRU, Sr-90, Co-60, and Eu-154. The key contaminants excluded  
19 because they are present completely in dissolved form in the original waste are Cl, Cr(VI), K,  
20 NO<sub>2</sub>, Cs-137, Tc-99, and I-129.

#### 21 **6.2.3.4 Waste Acceptance Criteria**

22 Other waste feed characteristics have a role in determining how the waste feed will be handled in  
23 the DBVS, but do not represent limiting specifications that would prevent the tank waste from  
24 being processed to generate the data necessary to determine if bulk vitrification is a viable  
25 production process. As an RD&D facility, it is important to maintain the flexibility to accept and  
26 test a wide range of feed compositions and to adequately challenge the process. Waste feed  
27 variations can be accommodated through blending of the waste with chemical simulant,  
28 adjusting waste loading, or through processing modifications. The limiting specifications for  
29 waste feed from Tank 241-S-109 to the DBVS are:

- 30 1. Cesium concentration must be less than 0.05 Ci/L (on a 7 M sodium basis),
- 31 2. The average solids concentration must be less than 3%,
- 32 3. TRU concentration must be less than 100 nCi/g.

1

**Table 6-5. Key Chemical Constituents/Contaminants in Interstitial Liquid and Dissolution Brine Fractions of Tank 241-S-109 Retrieval Stream**

Contaminant	In Interstitial Liquid Normalized to 5 M Na <sup>1</sup> (µg/ml)	In Dissolution Brine at 5 M Na (µg/ml)		
		During Early Dissolution	During Middle Dissolution	During Late Dissolution
Al	24000	Low Sol	Low Sol	Low Sol
Ca	25	Low Sol	Low Sol	Low Sol
Cl	5000	In Interstitial Liquid	In Interstitial Liquid	In Interstitial Liquid
total Cr	4700	Cr III Low Sol Cr VI in Interstitial Liquid	Cr III Low Sol Cr VI in Interstitial Liquid	Cr III Low Sol Cr VI in Interstitial Liquid
F	39	75	280	21
Fe	15	Low Sol	Low Sol	Low Sol
K	1200	In Interstitial Liquid	In Interstitial Liquid	In Interstitial Liquid
Mn	3	Low Sol	Low Sol	Low Sol
Ni	4	Low Sol	Low Sol	Low Sol
NO <sub>2</sub>	45000	In Interstitial Liquid	In Interstitial Liquid	In Interstitial Liquid
NO <sub>3</sub>	69000	186000	281000	301000
Pb	21	Low Sol	Low Sol	Low Sol
PO <sub>4</sub>	840	17000	3200	1100
Si	71	Low Sol	Low Sol	Low Sol
SO <sub>4</sub>	900	8000	5400	750
TIC as CO <sub>3</sub>	5100	34000	6200	2100
TOC	580	38	99	120
Total U	1	Low Sol	Low Sol	Low Sol

<sup>1</sup>The interstitial liquid composition is based on grab-samples taken in the saltwell. The dissolution brine compositions are based on runs of the ESP code using the Tank 241-S-109 Best Basis Inventory (BBI, 2001) as input  
 Early dissolution: 1 part water has been added to 4 parts waste  
 Middle: 1 part water has been added to 1.6 parts waste  
 Late: 1 part water has been added to 1 part waste  
 Low Sol: Low solubility in water  
 In Interstitial Liquid: As modeled in ESP, not present in dissolution brine because 100% is in the interstitial liquid  
 TIC: total inorganic carbon  
 TOC: total organic carbon

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**Table 6-6. Key Radionuclide Contaminants in Interstitial Liquid and Dissolution Brine Fractions of Tank 241-S-109 Retrieval Stream**

Contaminant	In Interstitial Liquid Normalized to 5 M Na <sup>1</sup> (μCi/ml)	In dissolution brine at 5 M Na (μCi/ml)		
		During Early Dissolution	During Middle Dissolution	During Late Dissolution
TRU (total)	7.6 x 10 <sup>-4</sup>	Low Sol	Low Sol	Low Sol
Cesium-137	1.6 x 10 <sup>2</sup>	In Interstitial Liquid	In Interstitial Liquid	In Interstitial Liquid
Strontium-90	1.4 x 10 <sup>-1</sup>	Low Sol	Low Sol	Low Sol
Technetium-99	1.6 x 10 <sup>-1</sup>	In Interstitial Liquid	In Interstitial Liquid	In Interstitial Liquid
Cobalt-60	2.0 x 10 <sup>-3</sup>	Low Sol	Low Sol	Low Sol
Europium-154	9.7 x 10 <sup>-2</sup>	Low Sol	Low Sol	Low Sol
Iodine-129	3.1 x 10 <sup>-4</sup>	In Interstitial Liquid	In Interstitial Liquid	In Interstitial Liquid

<sup>1</sup>The interstitial liquid composition is based on grab-samples taken in the saltwell. The dissolution brine compositions are based on runs of the ESP code using the Tank 241-S-109 Best Basis Inventory (BBI 2001) as input

Early dissolution: 1 part water has been added to 4 parts waste

Middle: 1 part water has been added to 1.6 parts waste

Late: 1 part water has been added to 1 part waste

Low Sol: Low solubility in water

In Interstitial Liquid: As modeled in ESP, not present in dissolution brine because 100% is in the interstitial liquid

2

### 3 6.2.4 Waste Feed Verification

4 In the course of the RD&D test project, waste feed batches will be received from the  
 5 Tank 241-S-109 WRS to the waste receipt tanks. These waste feed batches will be sampled for  
 6 constituents in the assigned waste codes for SST waste. Sampling will determine if these  
 7 constituents are detectable in the waste feed and the vitrified treated waste will not be tested for  
 8 the undetected LDR constituents. Processing will not begin until either the results of material  
 9 analyses are received and reviewed, or a determination has been made that the existing analyses  
 10 are valid. If necessary, the test plan for that campaign will include procedures to prevent the  
 11 mixing of materials not suitable for processing. The analytical methods used for measuring  
 12 concentrations will follow the analytical methods listed in Table 3.3 of the Waste Treatment  
 13 Plant Waste Analysis Plan (24590-WTP-RPT-ENV-01-003) and the analytical methods listed in  
 14 Appendix D from the *Regulatory Data Quality Objectives Optimization Report* for the WTP  
 15 (24590-WTP-RPT-MGT-04-001). Additional sampling and analyses to support risk and  
 16 performance assessment activities may be conducted and will be defined in test plans, as  
 17 applicable. Waste feed verification is part of the testing protocol to verify presence of a  
 18 bounding waste envelope.

1 The waste material composition as treated in the DBVS will represent the vitrification system  
2 waste stream provided by the WTP during full-scale operation. To ensure that the range of waste  
3 properties used during testing properly bounds the WTP waste properties, simulants will be  
4 added as required.

## 5 **6.2.5 Sampling Methods and Frequency**

6 Sampling during Phase 1 will be performed at the WRS waste staging tank prior to transfer to the  
7 DBVS waste receipt tank. The frequency of analysis of the waste during Phase 2 will be once  
8 per full DBVS waste receipt tank, unless a determination has been made that existing waste  
9 analyses are valid. Samples will be collected and analyzed consistent with the applicable  
10 portions of Section 9.0 in the *Regulatory Data Quality Objectives Optimization Report* (24590-  
11 WTP-RPT-MGT-04-001)(Appendix D), which consists of LDR and underlying hazardous  
12 constituent analytical methods.

13 **6.2.5.1 Treated Waste Sampling and LDR Compliance.** The final vitrified waste will be  
14 sampled to provide data for waste form qualification, risk assessment, performance assessment,  
15 and regulatory compliance. The vitrified waste will be tested for waste constituents on the SST  
16 Part A, which are LDR restricted for disposal in WAC 173-303-140 and 40 CFR 268.40. The  
17 constituents analyzed for are based on documented process knowledge, analysis of the waste  
18 feed, and are reasonably expected to be present in the final waste form. A composited vitrified  
19 waste core sample will be analyzed for the dangerous waste constituents that were detected in the  
20 tank waste feed to determine compliance with LDR requirements. The frequency of sampling  
21 the treated waste will be once per vitrified container of waste for an initial 10-sample set, after  
22 which random sampling will take place, as agreed to in the final test matrix.

23 Table 6-7 lists some of the physical properties that the treated waste will be analyzed for in order  
24 to determine waste form qualifications.

## 25 **6.3 SECONDARY WASTE STREAMS**

26 A variety of secondary wastes will be generated during DBVS operations. This section covers  
27 general requirements for management of expected secondary wastes.

### 28 **6.3.1 Secondary Liquid Waste**

29 Secondary liquid waste streams will be stored at the Test and Demonstration Facility area in  
30 portable tanks, prior to being disposed at the 200 Area ETF. Therefore, waste will be  
31 characterized in accordance with the waste characterization requirements specified in Section 3  
32 of the *Hanford Site Liquid Waste Acceptance Criteria* (HNF-3172). The sampling frequency  
33 will initially be once per tank. The long-term sampling frequency will be determined by the  
34 results of initial testing. The secondary liquid waste will be sampled with an appropriate  
35 sampler.

1

**Table 6-7. Physical Properties Sampling and Analysis<sup>1</sup>**

Property	Requirement	Citation
Vapor Hydration Test	Glass alteration rate shall be less than 50 grams/(m <sup>2</sup> -day) when measured using at least a seven day vapor hydration test run at 200 °C	ASTM WK84, <i>Test Method for Measuring Waste Glass Durability by Vapor Hydration Test.</i>
Compressive Strength after subjecting the samples to conditions noted:	Mean compressive strength of the waste form shall be at least 3.45E6 Pa and not less than 75% of the initial compressive strength	ASTM C39/C39M-01, <i>Standard Test Methods for Compressive Strength Specimens</i>
	Thermal Degradation - Thirty thermal cycles between a high of 60° C and a low of -40° C	ASTM B553-79, <i>Test Method for Thermal Cycling of Electroplated Plastics</i>
	Biodegradation - No evidence of culture growth when representative samples are tested	ASTM G21-96, <i>Standard Practice for Determining Resistance of Synthetic Polymeric Materials to Fungi</i> , and ASTM G22-76, <i>Standard Practice for Determining Resistance of Plastics to Bacteria</i>
Compression Testing	Each fully loaded package shall be able to withstand a compression load of 50,000 kg with the seal remaining intact	Integrated Disposal Facility Waste Acceptance Criteria

<sup>1</sup> Not all tests will be performed on all treated waste. Results from simulant tests may be used where applicable.

2 **6.3.2 Secondary Solid Waste**

3 A wide variety of solid and semisolid wastes will be generated during DBVS operation. Waste  
 4 streams include, but are not limited to, waste material residues in receipt and holding tanks,  
 5 collected air pollution control equipment dusts/sludges, discarded protective equipment, and  
 6 discarded samples taken during testing. These materials will be properly designated and  
 7 packaged per HNF-EP-0063 and managed at the appropriate TSD unit in accordance with the  
 8 unit's waste acceptance criteria.

9 Solid waste streams that are designated as dangerous or mixed waste will be transferred to a  
 10 Hanford Site TSD unit in accordance with the current *Hanford Site Solid Waste Acceptance*  
 11 *Criteria* (HNF-EP-0063) and the waste acceptance criteria of the receiving TSD unit. The waste  
 12 will meet the acceptance criteria as outlined in HNF-EP-0063 as well as the receiving TSD unit  
 13 acceptance criteria. Process knowledge will be used to better identify the final disposal method.

14 **6.4 OFFGAS TREATMENT SYSTEM**

15 The main offgas treatment system exhaust will be monitored continuously for radionuclides  
 16 contributing greater than 0.1 mrem/year using a record sample collection system. The offgas  
 17 treatment system will also be continuously monitored for criteria pollutants (i.e., particulate  
 18 matter, CO, NO<sub>x</sub>, SO<sub>x</sub>).

## 6.5 QUALITY ASSURANCE AND QUALITY CONTROL

### 6.5.1 General

This Quality Assurance (QA) and Quality Control (QC) section is prepared to support sampling and analysis to be implemented for DBVS operations. It will be used to support verification and characterization of the waste feed, treated waste form and the characterization of secondary waste streams.

**6.5.1.1** The QA/QC program ensures that an activity or project meets a required quality standard. QA is associated with recordkeeping, tracking, audits and assessments, and involves determining the desired level of quality and setting limits in advance. The analytical methods and associated QA/QC for the constituents of concern and for supplemental analytes identified in 24590-WTP-RPT-MGT-04-001, *Regulatory Data Quality Objectives Optimization Report*, will be imposed on waste feed samples. The laboratory(s) selected to do the analyses will have QA plans approved by Ecology prior to waste sample receipt and performing the selected analytical methods.

**6.5.1.2 Chain-of-Custody.** Chain-of-custody forms are used to document the possession of samples from the time they are collected through completion of laboratory analysis. The following information will be recorded for samples of waste, treatment residuals, and secondary wastes:

- The type of waste collected
- Names and signatures of sampling personnel
- Sample number, date and time of collection, and designation (e.g., grab, core)
- Names and signatures of persons involved in transferring and analyzing samples
- If applicable, the shipping number (air bill number) for samples shipped to off-site laboratories
- Analyses to be performed.

### 6.5.2 Trip Blanks and Equipment Blanks

The trip blank will be a water sample carried during the sample collection activities to ensure that contamination is not occurring during the different steps of sample collection and transportation to the laboratory. The equipment blank is a sample of analyte-free water used to rinse the sampling equipment. It is used to document the adequate decontamination of sampling equipment. Decontamination will be performed if disposable sampling equipment cannot be used. Analysis for the trip blank and equipment blank will be the same analytical tests performed for the specified procedures.

### 6.5.3 Duplicate Samples

The duplicate sample is a second aliquot of the collected sample and is used to determine method precision. The relative percent difference of the two samples is calculated by first obtaining the

1 difference of the two samples, dividing the difference by the average of the two samples, and  
2 finally multiplying by 100.

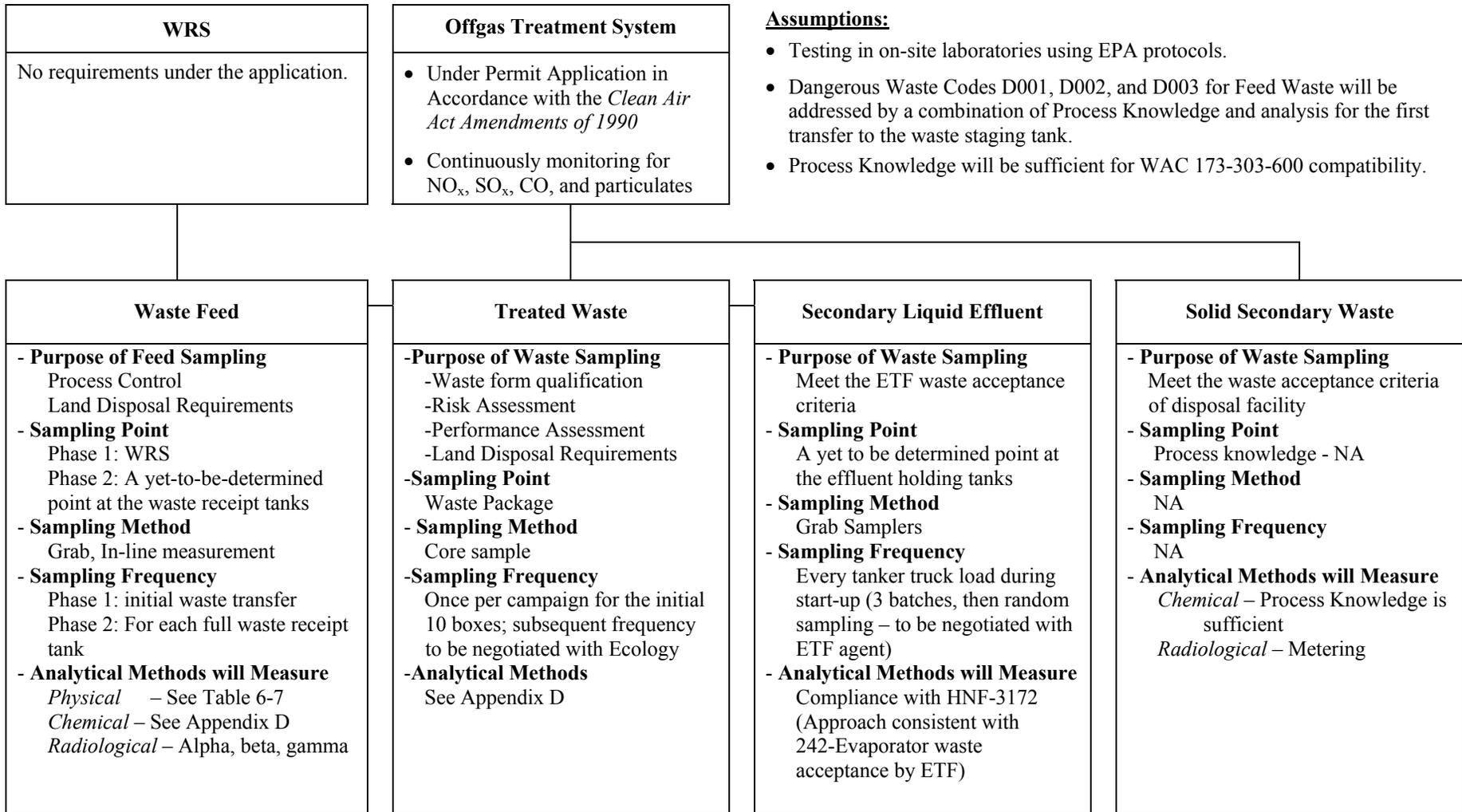
### 3 **6.5.4 Matrix Spike and Matrix Spike Duplicate Samples**

4 The matrix spike and matrix spike duplicate (MS/MSD) samples are QC samples spiked with  
5 known quantities of analytes. MS/MSD samples ensure that the analysis is testing for the  
6 specific analytes. Precision of a given sample can be calculated by the relative percent  
7 difference between the analytical results for the MS/MSD samples.

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9

**Figure 6-1. Flow Diagram for DBVS Waste Analysis Plan**



**Assumptions:**

- Testing in on-site laboratories using EPA protocols.
- Dangerous Waste Codes D001, D002, and D003 for Feed Waste will be addressed by a combination of Process Knowledge and analysis for the first transfer to the waste staging tank.
- Process Knowledge will be sufficient for WAC 173-303-600 compatibility.

6-13

# **Analytical Methods – Appendix D of the Permit Application**

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**APPENDIX D**  
**ANALYTICAL METHODS**

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9.8.3 Analytical Methods

The analytical methods and the target minimum reportable quantity (MRQ) ranges are indicated in Table 9-3 through Table 9-8.

Table 9-3. Methods and Target MRQ Range for Semivolatiles

CAS#	Compound	Analysis	Analytical Method <sup>(a)</sup>	Supernate Target MRQ Range (mg/L)	Sludge Target MRQ Range (mg/kg)
100-00-5	p-Nitrochlorobenzene	SVOA	8270C	0.25-5.00	1.50-5.00
100-25-4	1,4-Dinitrobenzene <sup>(d)</sup>	SVOA	8270C	0.25-25.00	1.50-5.00
106-46-7	1,4-Dichlorobenzene <sup>(b)</sup>	SVOA/VOA	8270C or 8260B	0.25-1.00	0.50-5.00
108-95-2	Phenol	SVOA	8270C	0.25-20.00	1.50-5.00
110-86-1	Pyridine <sup>(b)</sup>	SVOA/VOA	8270C or 8260B	0.25-1.00	1.50-5.00
120-82-1	1,2,4-Trichlorobenzene <sup>(b)</sup>	SVOA/VOA	8270C or 8260B	0.25-1.00	0.50-5.00
120-83-2	2,4-Dichlorophenol <sup>(c), (d)</sup>	SVOA	8270C	---	---
122-39-4	N,N-Diphenylamine	SVOA	8270C	0.25-5.00	1.50-5.00
126-73-8	Tributyl phosphate	SVOA	8270C	0.25-5.00	1.50-5.00
128-37-0	2,6-Bis(tert-butyl)-4-methylphenol	SVOA	8270C	0.25-15.00	1.50-5.00
50-32-8	Benzo(a)pyrene	SVOA	8270C	0.25-5.00 <sup>(e)</sup>	1.50-5.00 <sup>(e)</sup>
53-70-3	Dibenz[a,h]anthracene	SVOA	8270C	0.25-5.00 <sup>(e)</sup>	1.50-5.00 <sup>(e)</sup>
541-73-1	1,3-Dichlorobenzene <sup>(b)</sup>	SVOA/VOA	8270C or 8260B	0.25-1.00	0.50-5.00
59-50-7	4-Chloro-3-methylphenol <sup>(c), (d)</sup>	SVOA	8270C	---	---
627-13-4	Nitric acid, propyl ester <sup>(b)</sup>	SVOA/VOA	8270C or 8260B	0.25-1.00	2.00-5.00
62-75-9	N-Nitroso-N,N-dimethylamine <sup>(d)</sup>	SVOA	8270C	0.25-6.00	1.50-5.00
67-72-1	Hexachloroethane <sup>(b), (c), (d)</sup>	SVOA/VOA	8270C & 8260B	---	---
82-68-8	Pentachloronitrobenzene (PCNB)	SVOA	8270C	0.25-5.00	1.50-5.00
87-68-3	Hexachlorobutadiene <sup>(b)</sup>	SVOA/VOA	8270C or 8260B	0.25-1.00	0.50-5.00
87-86-5	Pentachlorophenol <sup>(d)</sup>	SVOA	8270C	0.25-35.00	1.50-5.00
88-85-7	2-sec-Butyl-4,6-dinitrophenol (Dinoseb)	SVOA	8270C	0.25-6.00	1.50-5.00
92-52-4	1,1'-Biphenyl	SVOA	8270C	0.25-5.00	1.50-5.00
95-50-1	1,2-Dichlorobenzene <sup>(b)</sup>	SVOA/VOA	8270C or 8260B	0.25-1.00	0.50-5.00
98-86-2	Acetophenone	SVOA	8270C	0.25-5.00	1.50-5.00
98-95-3	Nitrobenzene	SVOA	8270C	0.25-5.00	1.50-5.00

<sup>(a)</sup> Prep methods include SW-846 Methods 3520C and 3510C for the supernate and Methods 3540C and 3550B for the sludge. The Performance Based Measurement System should be applied as appropriate for these methods, adjusting for minor modifications required to safely handle high-level waste samples.

<sup>(b)</sup> These analytes can be determined using either the semivolatile or the volatile method. Hexachloroethane is usually included for analysis with both the semivolatile and volatile method.

<sup>(c)</sup> Hexachloroethane, 2,4-dichlorophenol, 4-chloro-3-methylphenol are not RDQO analytes, however, they are included in the list since they are currently COCs. (—) There has been no method demonstration work performed on these analytes.

<sup>(d)</sup> These analytes were identified in Table 9-2. They will be required during the analysis of regulatory compliance samples.

<sup>(e)</sup> For all analytes, EQLs ≤ target MRQ provided, except for benzo(a)pyrene and dibenz[a,h]anthracene where MDLs ≤ target MRQs.

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Table 9-4. Methods and Target MRQ Range for Volatiles

CAS#	Compound	Analysis	Analytical Method <sup>(a)</sup>	Supernate Target MRQ Range (mg/L)	Sludge Target MRQ Range (mg/kg)
100-41-4	Ethyl benzene	VOA	8260B	0.10-1.00	0.25-1.00
100-42-5	Styrene	VOA	8260B	0.10-1.00	0.25-1.00
10061-01-5	cis-1,3-Dichloropropene	VOA	8260B	0.10-1.00	0.25-1.00
10061-02-6	trans-1,3-Dichloropropene	VOA	8260B	0.10-1.00	0.25-1.00
106-35-4	3-Heptanone	VOA	8260B	0.10-1.00	0.25-8.50
106-42-3	p-Xylene & m-Xylene	VOA	8260B	0.10-1.00	0.25-1.00
106-93-4	Ethylene dibromide <sup>(d)</sup>	VOA	8260B	0.10-1.00	0.25-1.00
106-97-8	Butane	VOA	8260B	0.10-1.00	0.25-1.00
106-99-0	1,3-Butadiene	VOA	8260B	0.10-1.00	0.25-1.00
107-02-8	Acrolein	VOA	8260B	0.10-1.00	0.25-1.00
107-05-1	3-Chloropropene	VOA	8260B	0.10-1.00	0.25-1.00
107-06-2	1,2-Dichloroethane	VOA	8260B	0.10-1.00	0.25-1.00
107-12-0	Propionitrile	VOA	8260B	0.10-1.00	0.25-2.00
107-13-1	Acrylonitrile	VOA	8260B	0.10-2.00	0.25-1.00
107-87-9	2-Pentanone	VOA	8260B	0.10-5.00	0.25-1.00
108-10-1	4-Methyl-2-pentanone	VOA	8260B	0.10-1.00	0.25-1.00
108-38-3	m-Xylene (see 106-42-3)	VOA	8260B	0.10-1.00	0.25-1.00
108-87-2	Methylcyclohexane	VOA	8260B	0.10-1.00	0.25-1.00
108-88-3	Toluene	VOA	8260B	0.10-1.00	0.25-1.00
108-90-7	Chlorobenzene	VOA	8260B	0.10-1.00	0.25-1.00
108-94-1	Cyclohexanone	VOA	8260B	0.10-1.00	0.25-1.00
109-66-0	n-Pentane	VOA	8260B	0.10-1.00	0.25-1.00
109-99-9	Tetrahydrofuran	VOA	8260B	0.10-1.00	0.25-1.00
110-12-3	5-Methyl-2-hexanone	VOA	8260B	0.10-1.00	0.25-1.00
110-43-0	2-Heptanone	VOA	8260B	0.10-1.00	0.25-1.00
110-54-3	n-Hexane	VOA	8260B	0.10-1.00	0.25-1.00
110-82-7	Cyclohexane	VOA	8260B	0.10-1.00	0.25-1.00
110-83-8	Cyclohexene	VOA	8260B	0.10-1.00	0.25-1.00
111-65-9	n-Octane	VOA	8260B	0.10-1.00	0.25-1.00
111-84-2	n-Nonane	VOA	8260B	0.10-1.00	0.25-1.00
123-19-3	4-Heptanone	VOA	8260B	0.10-1.00	0.25-1.00
123-38-6	n-Propionaldehyde	VOA	8260B	0.10-1.00	0.25-8.50
123-86-4	Acetic acid n-butyl ester	VOA	8260B	0.10-1.00	0.25-1.00
123-91-1	1,4-Dioxane	VOA	8260B	0.10-1.00	0.25-5.00
126-98-7	2-Methyl-2-propenenitrile	VOA	8260B	0.10-1.00	0.25-1.00
127-18-4	1,1,2,2-Tetrachloroethene	VOA	8260B	0.10-1.00	0.25-1.00
141-78-6	Acetic acid ethyl ester	VOA	8260B	0.10-1.00	0.25-1.00
142-82-5	n-Heptane	VOA	8260B	0.10-1.00	0.25-2.00
287-92-3	Cyclopentane	VOA	8260B	0.10-1.00	0.25-1.00
4170-30-3	2-Butenaldehyde (2-Butenal)	VOA	8260B	0.10-1.00	0.25-1.00
56-23-5	Carbon tetrachloride	VOA	8260B	0.10-1.00	0.25-1.00
563-80-4	3-Methyl-2-butanone	VOA	8260B	0.10-1.00	0.25-1.00

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Table 9-4. Methods and Target MRQ Range for Volatiles

CAS#	Compound	Analysis	Analytical Method <sup>(a)</sup>	Supernate Target MRQ Range (mg/L)	Sludge Target MRQ Range (mg/kg)
591-78-6	2-Hexanone	VOA	8260B	0.10-1.00	0.25-1.00
64-17-5	Ethyl alcohol	VOA	8260B	0.10-1.00	0.50-5.00
67-56-1	Methyl alcohol	VOA	8260B	0.10-1.00	0.50-5.00
67-63-0	2-Propyl alcohol (Isopropanol)	VOA	8260B	0.10-1.00	0.25-5.00
67-64-1	2-Propanone (Acetone)	VOA	8260B	0.10-2.00	0.25-5.00
67-66-3	Chloroform <sup>(d)</sup>	VOA	8260B	0.10-1.00	0.25-1.00
67-72-1	Hexachloroethane <sup>(b), (c), (d)</sup>	VOA/SVOA	8260B & 8270C	---	---
71-23-8	n-Propyl alcohol (1-propanol)	VOA	8260B	0.10-1.00	0.50-5.00
71-36-3	n-Butyl alcohol	VOA	8260B	0.25-1.00	0.50-5.00
71-43-2	Benzene	VOA	8260B	0.10-1.00	0.25-1.00
71-55-6	1,1,1-Trichloroethane	VOA	8260B	0.10-1.00	0.25-1.00
74-83-9	Bromomethane	VOA	8260B	0.10-1.00	0.25-1.00
74-87-3	Chloromethane	VOA	8260B	0.10-1.00	0.25-1.00
75-00-3	Chloroethane	VOA	8260B	0.10-1.00	0.25-1.00
75-01-4	1-Chloroethene	VOA	8260B	0.10-1.00	0.25-1.00
75-05-8	Acetonitrile	VOA	8260B	0.10-1.00	0.25-8.50
75-09-2	Dichloromethane (methylene chloride)	VOA	8260B	0.10-1.00	0.25-1.00
75-15-0	Carbon disulfide	VOA	8260B	0.10-1.00	0.25-1.00
75-21-8	Oxirane	VOA	8260B	0.10-1.00	0.25-5.00
75-34-3	1,1-Dichloroethane	VOA	8260B	0.10-1.00	0.25-1.00
75-35-4	1,1-Dichloroethene	VOA	8260B	0.10-1.00	0.25-1.00
75-43-4	Dichlorofluoromethane	VOA	8260B	0.10-1.00	0.25-1.00
75-45-6	Chlorodifluoromethane	VOA	8260B	0.10-1.00	0.25-1.00
75-65-0	2-Methyl-2-propanol	VOA	8260B	0.10-1.00	0.25-5.00
75-69-4	Trichlorofluoromethane	VOA	8260B	0.10-1.00	0.25-1.00
75-71-8	Dichlorodifluoromethane	VOA	8260B	0.10-1.00	0.25-1.00
76-13-1	1,2,2-Trichloro-1,1,2-trifluoroethane	VOA	8260B	0.10-1.00	0.25-1.00
76-14-2	1,2-Dichloro-1,1,2,2-tetrafluoroethane	VOA	8260B	0.10-1.00	0.25-1.00
78-87-5	1,2-Dichloropropane	VOA	8260B	0.10-1.00	0.25-1.00
78-92-2	1-Methylpropyl alcohol (2-butanol)	VOA	8260B	0.25-1.00	0.50-5.00
78-93-3	2-Butanone	VOA	8260B	0.10-1.00	0.25-5.00
79-00-5	1,1,2-Trichloroethane	VOA	8260B	0.10-1.00	0.25-1.00
79-01-6	1,1,2-Trichloroethylene	VOA	8260B	0.10-1.00	0.25-1.00
79-34-5	1,1,2,2-Tetrachloroethane	VOA	8260B	0.10-1.00	0.25-1.00
95-47-6	o-Xylene	VOA	8260B	0.10-1.00	0.25-1.00
96-22-0	3-Pentanone	VOA	8260B	0.10-1.00	0.25-1.00

<sup>(a)</sup> Prep methods include SW-846 Methods 5021 and 5030B for the supernate and Methods 5021, 5035, and 5030B for the sludge. The Performance Based Measurement System should be applied as appropriate to these methods, adjusting for minor modifications required to safely handle high-level waste samples.

<sup>(b)</sup> Hexachloroethane is not a RDQO analyte, however, it is included in the list since it is currently a constituent of concern. (---) There has been no method demonstration work performed on this analyte.

<sup>(c)</sup> Hexachloroethane is usually included for analysis with both the semivolatiles and volatile method.

<sup>(d)</sup> These analytes were identified in Table 9-2. They will be required during the analysis of regulatory compliance samples.

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Table 9-5. Methods and Target MRQ Range for PCBs

CAS#	Compound <sup>(b)</sup>	Analysis	Analytical Method <sup>(a)</sup>	Supernate Target MRQ Range (mg/L)	Sludge Target MRQ Range (mg/kg)
11096-82-5	Aroclor-1260	PCB	8082	0.025-0.05	0.10-0.25
11097-69-5	Aroclor-1254	PCB	8082	0.025-0.05	0.10-0.25
11104-28-2	Aroclor-1221	PCB	8082	0.025-0.05	0.10-0.25
11141-16-5	Aroclor-1232	PCB	8082	0.025-0.05	0.10-0.25
12672-29-6	Aroclor-1248	PCB	8082	0.025-0.05	0.10-0.25
12674-11-2	Aroclor-1016	PCB	8082	0.025-0.05	0.10-0.25
53469-21-9	Aroclor-1242	PCB	8082	0.025-0.05	0.10-0.25

<sup>(a)</sup> Prep methods include SW-846 Methods 3540C and 3510C for supernate and Methods 3540C and 3550B for sludge. The Performance Based Measurement System should be applied as appropriate to these methods, adjusting for minor modifications required to safely handle high-level waste samples.

<sup>(b)</sup> These analytes were identified in Table 9-2. They will be required during the analysis of regulatory compliance samples.

Table 9-6. Methods and Target MRQ Range for Pesticides

CAS#	Compound	Analysis	Analytical Method <sup>(a)</sup>	Supernate Target MRQ Range (mg/L)	Sludge Target MRQ Range (mg/kg)
118-74-1	Hexachlorobenzene <sup>(b)</sup>	Pesticides	8081A	0.05-0.07	0.01-0.07
2234-13-1	Octachloronaphthalene	Pesticides	8081A	0.05-0.07	---
1321-64-8	Pentachloronaphthalene <sup>(c)</sup>	Pesticides	8081A	0.05-2.00	---
1335-87-1	Hexachloronaphthalene <sup>(c)</sup>	Pesticides	8081A	---	---
1335-88-2	Tetrachloronaphthalene <sup>(c)</sup>	Pesticides	8081A	0.05-2.00	0.05-0.07
309-00-2	Aldrin	Pesticides	8081A	0.025-0.05	0.05-0.07
319-84-6	alpha-BHC	Pesticides	8081A	0.025-0.05	0.05-0.07
319-85-7	beta-BHC	Pesticides	8081A	0.025-0.05	0.05-0.07
465-73-6	Isodrin	Pesticides	8081A	0.05-0.5	0.01-0.07
58-89-9	Gamma-BHC (Lindane)	Pesticides	8081A	0.025-0.05	0.05-0.07
60-57-1	Dieldrin	Pesticides	8081A	0.05-0.07	0.01-0.07
72-20-8	Endrin	Pesticides	8081A	0.05-0.07	0.01-0.07
76-44-8	Heptachlor <sup>(b)</sup>	Pesticides	8081A	0.025-0.05	0.05-0.07
8001-35-2	Toxaphene	Pesticides	8081A	0.05-0.07	0.10-0.50

<sup>(a)</sup> Prep methods include SW-846 Methods 3540C and 3510C for supernate and Methods 3540C and 3550B for sludge. The Performance Based Measurement System should be applied as appropriate to these methods, adjusting for minor modifications required to safely handle high-level waste samples.

<sup>(b)</sup> These analytes were identified in Table 9-2. They will be required during the analysis of regulatory compliance samples.

<sup>(c)</sup> Halowax 1014 (CAS#: 12616-36-3) contains the congener mixtures of penta-, hexa-, and tetra-polychlorinated naphthalenes. The analysis of these compounds is dependent on standards availability. The method demonstration was performed on supernate simulat and waste using Halowax 1013 (CAS#: 12616-35-2), which contains the congener mixtures of penta-, tetra-, and tri-polychlorinated naphthalenes. (---) Reporting limits have not been established for this analyte.

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Table 9-7. Methods and Target MRQ Range for Organic Acids

CAS#	Compound	Analysis	Analytical Method <sup>(a)</sup>	Supernate Target MRQ Range (mg/L) <sup>1</sup>	Sludge Target MRQ Range (mg/kg) <sup>(b)</sup>
144-62-7	Oxalic acid	Organic Acid	9056 (IC)	4000-5500	2000-3500
64-18-6	Formic acid	Organic Acid	9056 (IC)	6500-8000	2000-3500
64-19-7	Acetic acid	Organic Acid	9056 (IC)	6500-8000	2000-3500
79-10-7	2-Propenoic acid	Organic Acid	9056 (IC)	6500-8000	5000-6500

<sup>(a)</sup> The preparation for these samples in supernate is included in SW-846 Method 9056 and for the sludge were included in EPA Method 300.0 and ASTM Method D3987-85. The Performance Based Measurement System should be applied as appropriate to these methods, adjusting for minor modifications required to safely handle high-level waste samples.

<sup>(b)</sup> These reporting limits are greater than 1000, however, they are still within limits that support data needs for these analytes.

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Table 9-8. Methods and Target MRQ Range for Inorganics and Metals

CAS#	Compound	Analytical Method <sup>(a)</sup>	Target MRQ Range (mg/L)	Target MRQ Range (mg/kg)
7440-22-4	Ag <sup>(d)</sup>	6010B or (7761)	2.0-7.5 or (0.8-2.0)	25.0-80.0
7429-90-5	Al	6010B	20.0-45.0	70.0-600
7440-38-2	As <sup>(d)</sup>	6010B or (7060A)	10.0-15.0 or (0.6-2.0)	175-250 or (6.0-10.0)
7440-39-3	Ba <sup>(d)</sup>	6010B	0.55-1.75	6.00-25.0
7440-41-7	Be <sup>(d)</sup>	6010B	0.05-0.25	1.50-5.00
7440-43-9	Cd <sup>(d)</sup>	6010B	1.75-2.50	10.0-25.0
7440-48-4	Co	6010B	2.50-5.00	9.00-35.0
7440-47-3	Cr <sup>(d)</sup>	6010B	0.75-8.50	25.0-110
7440-50-8	Cu <sup>(d)</sup>	6010B	2.00-8.50	40.0-75.0
7439-89-6	Fe	6010B	2.00-20.0	100-200
7439-93-2	Li	6010B	1.50-10.0	35.0-150
7439-95-4	Mg	6010B	9.50-30.0	150-400
7439-96-5	Mn	6010B	0.55-1.75	1.00-15.0
7439-98-7	Mo	6010B	3.00-15.0	40.0-150
7440-23-5	Na	6010B	60.0-250	600-750
7440-02-0	Ni <sup>(d)</sup>	6010B	6.50-15.0	45.0-100
7723-14-0	P	6010B	15.0-80.0	200-250
7439-92-1	Pb <sup>(d)</sup>	6010B	15.0-45.0	100-450
7440-16-6	Rh (ICP-MS) <sup>(b)</sup>	6020	15.0-20.0	0.25-10.00
7704-34-9	S	6010B	50.0-75.0	150-300
7440-36-0	Sb <sup>(d)</sup>	6010B	15.0-20.0	210-250
7782-49-2	Se <sup>(d)</sup>	6010B or (7740)	20.0-30.0 or (2.5-5.0)	175-400 or (26-50)
7440-31-5	Sn	6010B	60.0-100	250-2500 <sup>(c)</sup>
7440-25-7	Ta (ICP-MS) <sup>(b)</sup>	6020	0.25-5.00	8.50-15.0
7440-28-0	Tl <sup>(d)</sup>	6010B	5.50-15.0	175-200
7440-61-1	U	6010B	250-525	1800-3200 <sup>(e)</sup>
7440-62-2	V <sup>(d)</sup>	6010B	2.50-5.00	25.0-50.0
7440-33-70	W	6010B	7.00-125	125-1100 <sup>(c)</sup>
7440-65-5	Y	6010B	1.00-6.00	7.00-100
7440-66-6	Zn <sup>(d)</sup>	6010B	3.50-5.50	15.0-100
7440-67-7	Zr	6010B	2.50-125	15.0-550
7439-97-6	Hg <sup>(d)</sup>	7470A or (7471A)	0.025-1.00	(0.10-3.50)
57-12-5	CN	9010B/9014 or 9012A	2.50-10.00	0.50-3.50

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Table 9-8. Methods and Target MRQ Range for Inorganics and Metals

CAS#	Compound	Analytical Method <sup>(a)</sup>	Target MRQ Range (ug/L)	Target MRQ Range (mg/kg)
7664-41-7	NH3	SM-4500-NH3-F or EPA Method 350.3	0.08-15.0	0.75-3.50
---	pH <sup>(d)</sup>	Std Method	---	---
16984-48-8	Fluoride	9056 <sup>(e)</sup>	150-500	2.50-25.0
16887-00-6	Chloride	9056 <sup>(e)</sup>	100-500	2.50-25.0
14797-65-0	Nitrite	9056 <sup>(e)</sup>	2600-10000 <sup>(c)</sup>	2.50-50.0
24959-67-9	Bromide	9056 <sup>(e)</sup>	150-500	2.50-25.0
14797-55-8	Nitrate	9056 <sup>(e)</sup>	2600-10000 <sup>(c)</sup>	2.50-50.0
14265-44-2	Phosphate	9056 <sup>(e)</sup>	150-500	2.50-50.0
14808-79-8	Sulfate	9056 <sup>(e)</sup>	150-500	25.0-50.0

<sup>(a)</sup> Prep methods include SW-846 Methods 3010A, 3005A, and 3015 for the supernate and Methods 3050B and 3015 for the sludge. Fusion was also used in the methods demonstration for metals preparation. The Performance Based Measurement System should be applied as appropriate to these methods, adjusting for minor modifications required to safely handle high-level waste samples.

<sup>(b)</sup> Although the ICP-AES methodology (6010B) can be applied to the determination of rhodium and tantalum, the ICP-MS technique (6020) has been demonstrated to achieve reporting limits that better support data needs.

<sup>(c)</sup> These reporting limits are greater than 1000, however, they are still within limits that support data needs for these analytes.

<sup>(d)</sup> These analytes were identified in Table 9-2. They will be required during the analysis of regulatory compliance samples.

<sup>(e)</sup> Prep methods for the inorganic anions were SW-846 Method 9056 for the supernate and EPA Method 300.0 and ASTM Method D3987-85 for the sludge.

( ) - Target MRQ ranges given in parentheses correspond to the methods given in parentheses.

#### 9.8.4 Analytical Method Guidelines

Per the guidelines established using the PBMS and safe handling procedures required to address ALARA concepts, sample sizes may be reduced from those recommended in SW-846. The adjustments to the sample sizes applied to the SW-846 methods and the appropriate scaling of reagents is not considered a modification or a deviation from SW-846. The sample size reduction is typical for the analysis of radioactive samples to ensure safety. The selection of acids, solvents, and surrogates may also be adjusted to address matrix interferences and are within the PBMS guidelines. Minor modifications to SW-846 methods are discussed in Table 9-9 and Table 9-10.

Table 9-9. SW-846 Guidelines and Handling Hanford Tank Waste

SW-846 Methods Guidelines	Procedures for Performing Analysis on Hanford Tank Waste
SW-846 provides recommendations for sample sizes applied to each method.	Sample size reduction, the associated scaling of reagents, and the selection of container sizes applied during sample preparation are not considered deviations from SW-846. This is required to ensure safe handling of the radioactive samples and minimize waste generation.
In some methods, SW-846 describes specific containers or vessels for application of the method and means for transferring materials (for example, pouring).	In cases where the container type may impact ability to safely handle a radioactive sample or where the sample matrix may be affected by the container material, a different container type may be specified for safe handling in laboratory procedures. Procedures may require minor adjustments for safety (for example, using a syringe to transfer the sample rather than pouring the sample). These are considered minor modifications.
SW-846 provides recommended wavelengths for ICP-AES and alternate isotopes for ICP-MS	Adjustments to wavelengths for ICP-AES and selection of alternate isotopes for ICP-MS are not considered deviations from SW-846. This is required to address complex matrix interferences and improve analytical accuracy.

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Table 9-10. Summary of Method Modifications

Analytes	Determinative	Preparation	Modifications <sup>(a)</sup>
SVOA	8270C	3520C, 3510C, 3540C, 3550B	If matrix interferences affect the recoveries of the SW-846 recommended surrogates, additional surrogates may be added to the method surrogate list for the 8270C analysis. If necessary, this should be included in the TSAPs.
VOA	8260B	5021, 5030B, 5021, 5035, 5030B	Minor <sup>(a)</sup>
PCB	8082	3540C, 3510C, 3550B	Minor <sup>(a)</sup>
Pesticides	8081A	3540C, 3510C, 3550B	Minor <sup>(a)</sup>
Organic Acids and Inorganic Anions	9056 (IC)	9056	Organic acid salts are not included in the SW-846 9056 method; however, the IC technique and column selection can be adjusted to determine these analytes and to reduce interferences from the anions and acid salts present in the tank waste.
		EPA Method 300.0 (EPA, 1989)	EPA Method 300.0 is not an SW-846 method.
		ASTM D3987-85 (1999) Shake extraction of solid waste with water	ASTM D3987-85 is not an SW-846 method. An ultrasonic bath rather than shaker may be applied to the preparation of solids if this facilitates proper extraction.
Metals	6010B (ICP-AES) 6020 (ICP-MS)	3010A, 3005A, 3015, 3050B, 3015 (Note: acid digestion methods generally preferred over fusion)	Heat source alternatives (for example, heating block) and solvent selection may be adjusted based on matrix interferences and safe sample handling practices. See Table 9-9.
Silver Arsenic Selenium	7761 (AA) 7060A (AA) 7740 (AA)	ASTM D4503-86 (1998) Dissolution of solid waste by fusion	Not an SW-846 method. The modified ASTM method uses KOH, which supports a broader analyte list, rather than lithium metaborate. ASTM methods are recognized by EPA as equivalent standards.
Mercury	7470A 7471A	NA	Minor <sup>(a)</sup>
Cyanide - CN	9010B/9014 9012A	NA	Selection of distillation apparatus may be adjusted to safely perform distillation.
Ammonia - NH <sub>3</sub>	SM-4500-NH <sub>3</sub> -F	NA	SM-4500-NH <sub>3</sub> -F (Standard Method, 1992) is not an SW-846 method, but is considered equivalent by EPA.
	EPA Method 350.3	NA	EPA Method 350.3 (EPA, 1989) is not an SW-846 method.
pH	Standard Method	NA	Application of laboratory standard pH measurement techniques are considered equivalent by EPA and can be applied to this determination.

NA - not applicable. The preparation procedure for these analytes is included in the determinative method.  
AA - atomic absorption spectrometry

<sup>(a)</sup> - The Performance Based Measurement System should be applied as appropriate to these methods, adjusting for minor modifications required to safely handle high-level waste samples. See also the discussion in Table 9-9.

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**9.8.5 Analytical Quality Control Parameters**

The QC parameters in Table 9-11 have been provided with input from a performance assessment of method evaluation and demonstration activities. These performance criteria apply for the analytes and methods described in Table 9-3 through Table 9-8.

**Table 9-11. Quality Control Parameters for SW-846 Methods**

Analytes	Method	QC Acceptance Criteria			
		LCS % Recovery	Spike % Recovery	MSD/Dup RPD	Replicate % RSD
Metals	ICP-AES-6010B	80-120 %	75-125 %	< 20 %	< 20 %
Metals	ICP-MS-6020	80-120 %	75-125 %	< 20 %	< 20 %
Metals	AA-7060A; AA-7740 or equiv	80-120 %	75-125 %	≤ 20 %	≤ 20 %
Hg	CVAA-7470A; 7471A	80-120 %	75-125 %	≤ 20 %	≤ 20 %
Organic Acids and Anions	IC-9056	80-120 %	75-125 %	≤ 20 %	≤ 20 %
pH	pH (Std Method)	± 0.1 pH units	NA	NA	NA
SVOA	8270C	70-130 %	50-150 % <sup>(a)</sup>	< 30 %	< 30 %
VOA	8260B	70-130 %	50-150 %	< 30 %	< 30 %
PCBs	8082	70-130 %	50-150 % <sup>(a)</sup>	< 30 %	< 30 %
Pesticides	8081A	70-130 %	50-150 % <sup>(a)</sup>	< 30 %	< 30 %

<sup>(a)</sup> Control chart limits should be identified for these analyses as applied to the recoveries associated with the high-level waste matrices as appropriate. The SW-846 Method 8270C acknowledges poor recoveries of phenols and other semivolatiles and recommends expanding the recovery limits to approximately D-175 % for many of these analytes. (D- applies to any result detected above zero at the instrument).

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