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Results from Analysis of a Hanford Tank 241-AN-102 Crucible-Scale Glass Waste Form

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SAVANNAH RIVER TECHNOLOGY CENTER

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Acronyms

BNI.....	Bechtel National, Inc.
BWXT	Babcock and Wilcox Technology
CLP.....	Contract Laboratory Program
COC.....	Chain of Custody
GC-MS	Gas Chromatography-Mass Spectrometry
HpCDD.....	Heptachlorodibenzo-p-Dioxin
HpCDF	Heptachlorodibenzofuran
HxCDD.....	Hexachlorodibenzo-p-Dioxin
HxCDF	Hexachlorodibenzofuran
IC	Ion Chromatography
ICP-MS.....	Inductively-Coupled Plasma-Mass Spectrometry
LAW	Low-Activity Waste
LCS.....	Laboratory Control Sample
LRM	Low-Activity Reference Material
MS	Matrix Spike
MSD	Matrix Spike Duplicate
ND	Not Determined
NF.....	Not Flammable
OCDD.....	Octachlorodibenzo-p-Dioxin
OCDF	Octachlorodibenzofuran
PCB	Polychlorinated Biphenyl
PeCDD	Pentachlorodibenzo-p-Dioxin
PeCDF	Pentachlorodibenzofuran
QA	Quality Assurance
QAPjP.....	Quality Assurance Project Plan
QC	Quality Control
RCRA	Resource Conservative and Recovery Act
RPD	Relative Percent Deviation
RPP.....	River Protection Project
SOP.....	Standard Operating Procedure
SRS.....	Savannah River Site
SRTC	Savannah River Technology Center
TCDD	Tetrachlorodibenzo-p-Dioxin
TCDF.....	Tetrachlorodibenzofuran
TCLP	Toxicity Characteristic Leaching Procedure
THF	Tetrahydrofuran
TIC.....	Tentatively-Identified Compound
USDOE.....	United States Department of Energy
USEPA	United States Environmental Protection Agency
UTS	Universal Treatment Standard
WTP	Waste Treatment Plant

1.0 Executive Summary

Analysis has been completed of Low-Activity Waste (LAW) glass samples that were prepared from pretreated Hanford Waste Tank AN-102 supernate. Concentrations of the primary components were within 10 % of the target composition except sulfur. The total beta activity in the sample was 1.9×10^3 nCi/g or 5.5 Ci/m^3 (primarily strontium-90 and its daughter, yttrium-90), and total alpha activity was 21 nCi/g (primarily americium-241 and curium-244). These results met the River Protection Project-Waste Treatment Project Department of Energy/Office of River Protection contract radionuclide specifications for these radionuclides (20 Ci/m^3 for strontium-90 and 100 nCi/g for alpha-emitting transuranic radionuclides). The density of the waste form was determined to be 2.9 g/cc.

The waste form sample did not show any characteristics of Hazardous Waste.⁸ The sample was not ignitable, and the cyanide concentration was shown to be well below the 40CFR268 land disposal restriction limits. In addition, the waste form sample was shown to meet Universal Treatment Standards for RCRA metals. No organic compounds were detected when the samples were analyzed for volatile and semivolatile organic compounds, dioxins and furans, pesticides, and polychlorinated biphenyls (PCBs).

2.0 Introduction and Background

Bechtel National, Inc. (BNI) has been awarded a contract from the United States Department of Energy's (USDOEs) Office of River Protection to develop, design, construct, and startup the River Protection Project Waste Treatment Plant (RPP-WTP). The RPP-WTP will pretreat and immobilize the radioactive waste that is being stored in underground storage tank at the USDOE Hanford site outside Richland, Washington. As part of the RPP-WTP, BNI and USDOE have contracted Westinghouse Savannah River Company's Savannah River Technology Center (SRTC) to perform process demonstrations using nonradioactive samples (waste stream simulants) and radioactive samples (from the Hanford Tank Farm). The work reported in this report is therefore being performed by SRTC under the technical direction of BNI.

As part of these demonstrations, SRTC is to have key samples analyzed according to USEPA SW-846 protocol.^{1,2,3} This report is a summary of analysis results of the glass waste form that was produced from pretreatment and vitrification of an Envelope C supernate sample taken from Hanford Tank 241-AN-102. The "241" is used to designate all Hanford radioactive underground waste storage tanks. Throughout the rest of this report the sample will be referred as "AN102".

The results in this report are from analysis of a glass waste form that was produced by adding the waste stream and glass-forming minerals to crucibles and melting the mixture in a furnace. The details surrounding these furnace demonstrations (including the composition of the waste stream and glass-forming minerals) have been described elsewhere.⁴ The waste stream used in this study was taken from the evaporator feed samples described in Reference 10. Reference 10 includes results from the USEPA SW-846 analyses of this waste stream.

SRTC personnel collected samples of the glass waste form. SRTC personnel also performed some of the sample preparation. Babcock and Wilcox Technology (BWXT) Services Inc. performed the sample analyses. BWXT Services Inc. is qualified to perform analyses according to the USEPA protocol specified for these samples.⁸

Results from all regulatory analyses of the 102-AN glass waste form have been presented in this report. Although some of the field blank results have also been presented here, a complete presentation of results from the field blanks, trip blanks, laboratory method blanks, and standards have not been included here. Instead, the nine-volume data package is being issued independently.⁵

This report has been organized to make it as compatible as possible with the basis documents.^{1,2} Chapter **3.0. Experimental** has been organized chronologically into sections describing the sample collection, preparation and analysis. Chapter **4.0 Results** has been organized into a separate section for each of the tables in Reference 1.

This document is being issued as **Revision 1** of **SRT-RPP-2001-00013**. **Revision 0** of this document was issued when data was in the preliminary validation stages and was issued as a progress report. Because changes to this document have been substantial, editorial bars have not been used to identify changes. The entire document should therefore be considered to be a new entity.

3.0 Experimental

This chapter is a description of the methods used to collect, prepare, and analyze the samples. Descriptions of the sample collection and sample preparation methods have been organized according to how the samples were generated. Sample analyses have been presented in the same order as the analytes listed in Tables 1 through 5 of Reference 1.

A. Sample Collection

Two furnace tests were performed each producing a 110-gram “supersample” of glass waste form material. The term “supersample” is being used to describe the batch of glass that was produced during each furnace test. The term “sample” is being used to describe an aliquot from one of the supersamples that was submitted for analysis. The supersamples were

- AN-102 samples for volatile organic compound analysis.
- AN-102 samples to be ground prior to analysis.

The supersamples collected for volatile organic compound analysis were used to meet some of the characterization needs specified in Reference 1 for supporting a delisting petition and for supporting permitting. The other supersamples were used for the remainder of the delisting and permitting analyses and all analyses specified for tank waste, radionuclide, and physical property characterization.

Sampling of these waste forms was performed between 1 and 2 weeks after the glass waste forms were produced. The sampling process was a simple transfer of the glass waste forms from their storage containers to a glass jar that had been certified as clean. The storage containers were either another certified clean glass jar or the crucible in which the glass was made.

Field blanks were collected immediately after the glass waste form was sampled. The field blanks were a standard low-activity reference material (LRM) glass generated in the same fashion as the waste form supersamples. To generate the field blanks, LRM glass was transferred to sample containers in the same location immediately after sampling of the glass waste forms. For the AN-102 supersamples that were used for volatile organic compounds characterization, the supersamples were collected on different days. Therefore, a separate field blank was generated for each of these supersamples. In addition, LRM glass was used for trip blanks.

B. Sample Preparation

Each supersample was subjected to size reduction and divided into samples for preparation and analysis. Table III.1 is a summary of the samples, blanks, standards, and preparation and analysis methods. For each sample, some of the sample preparation was performed by SRTC, and the remainder was performed by subcontractor prior to analysis.

Size reduction was performed at SRS. For samples and blanks analyzed for volatile organics, an agate mortar and pestle were used to break the material into pieces that fit into a standard forty-milliliter vial. For the other supersamples, the agate mortar and pestle were used to grind samples and blanks into particles with diameters of less than 0.9 cm. Some of this material was ground further using an agate ball pulverizing mill and dissolved by either an acid dissolution, a sodium peroxide fusion with an acid uptake, or a sodium peroxide fusion with a water uptake.

The resulting samples were sent to BWXT Services, Inc. where additional pretreatment was performed as shown in Table III.1 (column labeled "Lab."). The extraction indicated in Table III.1 for the IC was a water extraction. This and the other preparation steps indicated in Table III.1 were performed by BWXT Services, Inc.

One set of samples was characterized for cyanide, density, ignitability, ammonia, and total halides. For density, ignitability and total halide analyses, no preparation was necessary. Prior to the other analyses, the following preparation steps were performed:

- Cyanide: USEPA SW-846 Method 9010B
- Ammonia: Sample Digestion

Table III.1. Summary of Sample, Blank, and Standard Preparation and Analysis Methods

	Samples	Blanks		Standards	Preparation		Analysis***
		Field	Trip		SRS	Lab.***	
Misc. Volatiles*	4	2	1	-	Break	5035	8260B
IC – Organics*	4	2	1	-	Break	Extract	Mod. 9056
Semivolatiles	2	1	-	-	Grind	3550B	8270C
Pesticides&PCBs	2	1	-	-	Grind	3550B	8081,8082
Dioxins&Furans	2	1	-	-	Grind	8290	8290
TCLP	1	1	-	1	Grind	1311	3015,6010B
Miscellaneous	2	1	-	-	Grind	**	**
Acid Dissolution	2	1	-	2	Dissolve	Table III.2	Table III.2
Fusion & Acid	2	1	-	2	Dissolve	Table III.2	Table III.2
Fusion & H ₂ O	2	1	-	2	Dissolve	Table III.2	Table III.2

*Samples prepared by breaking of supersamples that were produced for volatile organic analyses.

**Samples analyzed for cyanide, density, oxalate, ignitability, ammonia, and total halides.

***Performed by BWXT Services, Inc.

C. Sample Analysis

The title of each subsection in this section was taken from the corresponding table in Reference 1. Titles are not meant to suggest that the designated activities are underway. The title of each section in this portion of the report was taken from the corresponding table in Reference 1.

1. Analysis to Support Delisting Petition

Analyses described in this section were performed on the last six sets of samples listed in Table III.1. The methods used for most of the analyses have been given in Table III.1. For the metals, anions and radionuclides analyses, the samples were dissolved at SRS prior to shipment to BWXT Services, Inc. for analysis. The analytical methods have been given in Table III.2 along with the preparation and analysis methods used to characterize the samples that were dissolved at SRS prior to shipment to BWXT Services, Inc.

Standard methods were used to characterize the samples for the other analytes. For cyanide and ignitability, the set of samples designated for miscellaneous analyses was used. Cyanide was determined using Method 9014. Ignitability was measured using Method 1010 modified to accommodate smaller samples. TCLP leachates were acid-digested as required by the TCLP method (Step 7.2.14) prior to analysis. Although laboratories that perform TCLP often do not perform an acid-digestion on the leachate, this practice is in direct contradiction of the TCLP procedure which states in Step 7.2.14, "...data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous." Except for the sample analyzed for mercury, Method 3015 was used to acid-digest the samples. The resulting digested extract was analyzed

using Method 6010B. For mercury, the sample was acid-digested and analyzed using Method 7470A.

Table III.2. Preparation and Analysis Methods for Dissolved Glass Samples

	Samples	Preparation	Analysis
ICP-ES	Acid, Fusion & Acid	3015	6010B
CV-Hg	Acid, Fusion & Acid	-	7470A
Gamma	Acid, Fusion & Acid	-	Gamma PHA
Transuranics	Acid, Fusion & Acid	Separation	Alpha PHA
Uranium	Acid, Fusion & Acid	Separation	Alpha PHA
C-14/H-3	Acid, Fusion & Acid	Oxidation	Scintillation
Alpha/Beta	Acid, Fusion & Acid	-	Method 9310
Sr-90	Acid, Fusion & Acid	Separation	Scintillation
I-129	Acid, Fusion & Acid	Separation	Gamma PHA
Se-79	Acid, Fusion & Acid	Separation	Scintillation
Tc-99	Acid, Fusion & Acid	Separation	Scintillation
Anions	Fusion & Water	25:1 Dilution	Method 9056

In addition, dioxins and furans were measured according to USEPA SW-846 Method 8290. This and all analytical procedures had been implemented by BWXT Services, Inc. as a standard operating procedure (SOP).

2. Analysis to Support Characterization of Immobilized Waste Form Samples

These analyses were performed on the last three sets of samples listed in Table III.1. The cyanide and metals analyses have been discussed in Section 3.C.1. The other analyses that were performed to support characterization of the tank waste samples were Method 9056 analysis for the anions, determination of ammonia, and Method 9020B for total halide determination.

Ammonium was determined using an ion-selective electrode technique. Ammonium was determined in the field blank. These results have been included with the appropriate qualifications.

3. Analysis to Support Permitting

Analyses described in this section were performed on the first six sets of samples listed in Table III.1. These analyses consist of all the organic compound analyses except the dioxins and furans. All methods used for these analyses had been discussed with RPP-WTP R&T personnel prior to the analyses. All methods had also been incorporated in the task plan.³

As indicated in Table III.1, after preparation of samples by USEPA SW-846 Method 5035, most of the volatile organic compounds specified in Reference 1 were determined by BWXT Services, Inc. using USEPA SW-846 Method 8260B.

To determine the concentration of formate, acetate, and oxalate, BWXT Services, Inc. used Method 9056 after extracting the samples with water overnight. Method 9056 was implemented by BWXT Services, Inc. as a SOP. Triethylamine was determined by a GC-MS method that used a column deactivated for amines (RTx-5 Amine). This analytical method had also been implemented at BWXT Services, Inc. as a SOP.

As indicated in Table III.1, after preparing the sample using USEPA SW-846 Method 3550B, B&W used USEPA SW-846 Method 8270C to analyze samples for semivolatile organic compounds. In accordance with Step 5.4.1 of Method 3550B, a one-to-one mixture of methylene chloride and acetone was used for extracting these samples for semivolatile analyses.

Because USEPA Method 3550B was used as a preparation step for both the pesticides and PCB analyses, a single sample was extracted, and the resulting extraction liquid was used for both analyses. In accordance with Step 5.4.2 and Step 5.4.3 of Method 3550B, a one-to-one mixture of hexane and acetone was used for extracting these samples for pesticides and PCB analyses.

After extraction of the pesticide and PCB samples using USEPA SW-846 Method 3550B, B&W used a portion of the resulting extraction liquid for pesticide analyses by USEPA SW-846 Method 8081 and another portion to analyze for PCBs by USEPA SW-846 Method 8082. All calibrations, calibration checks and recoveries were within specified limits for both of these analyses.

4. Radionuclide Analysis

These analysis methods have been summarized in Table III.2. As shown in Table V.9, detection limits for selenium-79 have been included as they were reported from the laboratory although recoveries for this species were not within the range specified in Reference 2. Recoveries could not be determined because a selenium-79 standard could not be located. Detection limits for carbon-14 were very high because of interferences. The results have been reported in this report along with the appropriate qualifications.

5. Determination of Physical Properties

Pycnometry was used to determine the density of the glass waste form samples. These analyses were performed on the set of samples labeled as "Miscellaneous" in Table III.1. Size reduction on these samples was limited to that which was necessary to provide a waste form with particles with less than a 0.9-cm diameter. This may have reduced the bulk density somewhat by producing very small particles; however, this was expected to have affected the density by less than a 10%.

4.0 Quality Assurance

As indicated previously, samples were collected, prepared, and analyzed according to USEPA SW-846 protocol to the greatest extent practical, even for target analytes not addressed in USEPA SW-846 (e.g. for radionuclides). Upon completion of the analyses, results were validated according to the general criteria given for "Definitive Data" in "Data Quality Objectives for Superfund," Interim Final, **EPA540-R-93-071**, September 1993 and "EPA National Functional Guidelines for Organic Data Review," Revised Draft, June 1991. Aspects reviewed during the validation were

Chain of Custody (COC): COC documents completed correctly and signed

Sample Documentation: All relevant documentation completed and included in data package.

Sample Preservation: Verification that samples were held at 4 °C when appropriate.

Hold Times: Samples prepared and analyzed according to hold time for analytes of interest.

Initial and Continuing Calibration: Initial and continuing calibration within appropriate range.

Analyte Identification: Analyte correctly identified (e.g. mass spectrometry tune validation).

Analyte Quantitation: Concentration correctly determined (e.g. lack of interferences).

Method Blanks: Method blank concentrations sufficiently below sample concentrations.

Matrix Spike Recoveries: Recovery within specified range and recovery precision as specified.

Surrogate, Tracer, and Carrier Recoveries: Recovery within range specified for matrix spike.

Laboratory Control Sample Recoveries: Recovery within range specified for matrix spike.

Laboratory Duplicates: Precision within range allowed for by specific USEPA SW-846 method.

Trip Blanks (where appropriate): Concentrations sufficiently below sample concentrations.

Results from quality assurance and quality control efforts are presented in this chapter. Matrix spike and matrix spike duplicate results have been presented in each section of this chapter along with any incident in which one of the aspects listed above did not meet the requirements of the USEPA SW-846 method, Reference 2, and Reference 3. In most cases, these results are presented in the same order as the analytes listed in Tables 1 through 5 of Reference 1. The title of each section in this chapter was taken from the corresponding table in Reference 1.

A. Analysis to Support Dangerous Waste Form Characteristic Determinations

Results presented in this section were those specified in Table 1 of Reference 1. Except where noted below, these analyses met all QA requirements given in the QAPjP² and project task plan³. Reference 2 specified that recoveries would be between 50 and 130 % for semivolatile and volatile organic analytes. USEPA SW-846 methods provide requirements for establishing laboratory-specific acceptable recovery ranges. Prior to preparation of Reference 3, BWXT Service, Inc. indicated that 50 % recoveries would not be possible for several of the analytes. As a result of discussions with RPP personnel, the lower limit on acceptable recovery range for organics was changed from 50 to 20 %. During these discussions, a decision was also made not to add the sodium thiosulfate preservative initially specified for some of the samples. These deviations were documented in the task plan.³

1. Cyanide

Three matrix spikes were analyzed for cyanide. One was created by spiking an aliquot of the sample prior to the Method 9010B extraction. The resulting recovery was 106 %. A spike and duplicate were also created by spiking aliquots of the extractate before the Method 9014 analyses. These recoveries were 92 and 84 %. To be conservative, using the lowest and highest values to calculate a relative percent deviation (RPD), the value would be 23 %. This is slightly higher than the specified limit of 20 %.^{2,3} Cyanide analyses met all other QA specifications.

2. Dangerous Waste Characteristics

Recoveries associated with these analyses have been given in Tables IV.1 and IV.2. Analyses associated with the Table IV.1 recoveries met all QA specifications except

- Mercury was detected in two continuing calibration blanks.
- One selenium continuing calibration verification was 78.8 % (SW-846 specifies 85 to 115 %).
- Dioxin and furan recoveries were not determined for the matrix and Laboratory Control Samples.
- The dioxin and furan sample extracts reached 9 °C (instead of 4 ± 2 °C) prior to sample analysis.

As shown in Table IV.1, some matrix spike and duplicate recoveries were outside of the acceptable range. For silver, this appears to have been due to solubility issues associated with the high concentrations (1000 µg/L) used in the matrix spikes. Analytical spike samples (50 µg/L added) were within the acceptable values (recoveries of 118 and 109 % for the acid and fusion dissolutions respectively). Selenium recoveries were also low; however, selenium spike concentrations were low enough (20 µg/L) to avoid insolubility concerns.

Although one nickel and one mercury recovery were outside the acceptable range for the peroxide fusion spikes, the peroxide fusion results were not reported for these elements. This dissolution technique is not compatible with nickel determinations because a nickel crucible is used. Mercury analyses are suspect because the dissolution is performed at 700 °C in an open vessel. In such conditions, mercury losses are problematic.

Recoveries associated with analysis of the TCLP extracts have been given in Table IV.2. These analyses met all QA specifications except

- The arsenic final calibration verification was 79.1 % (SW-846 specifies 85 to 115 %).
- The selenium low-level concentration standard was 190 % (specified range for recoveries was 75 to 25 %).^{2,3}
- The extraction blank was contaminated with 1238 µg/L of barium.

Table IV.1. Recoveries for Dangerous Waste Characteristic Analyses

Characteristic	Analyte	Acceptable Values		^b Acid Dissolution			^c Peroxide Fusion		
		Recovery	RPD	MS	MSD	RPD	MS	MSD	RPD
Total Metals (%)	As	75-125	<20	119.3	119.6	0.2	^a 138.5	^a 150.3	8.2
	Ba	75-125	<20	103.5	103.2	0.3	99.6	100.7	1.1
	Cd	75-125	<20	99.5	98.4	1.1	98.2	97.4	0.9
	Cr	75-125	<20	104.4	103.5	0.8	101.7	101.7	0
	Pb	75-125	<20	99.6	100.1	0.5	100.3	102.5	2.2
	Se	75-125	<20	117.1	^a 14.4	^a 156	^a 30.7	^a 27.1	12
	Ag	75-125	<20	^a 12.5	^a 34.8	^a 85	^a 8.1	^a 10.1	17
	Ni	75-125	<20	107.2	106.2	0.9	^a 137.0	90.0	0.8
	Hg	75-125	<20	85.6	97.2	8.6	75.0	^a 74.2	1.0
^d Volatiles	-	Sect.4.C.1							
^e Semi-Volatiles	-	Sect. 4.C.2							
		Acceptable Values		Measured Recoveries					
		Recovery	RPD	Standard	Duplicate	RPD			
^f Dioxins & Furans (%)	*2,3,7,8,-TCDD	20-130	<25	75.4	73.7	2.3			
	*1,2,3,7,8-PeCDD	20-130	<25	97.6	92.5	5.4			
	**1,2,3,4,7,8-HxCDD	20-130	<25	89.9	89.0	1.0			
	*1,2,3,6,7,8-HxCDD	20-130	<25	92.9	89.0	4.3			
	1,2,3,7,8,9-HxCDD	20-130	<25	-	-	-			
	*1,2,3,4,6,7,8-HpCDD	20-130	<25	83.6	79.2	5.4			
	*OCDD	20-130	<25	82.8	78.5	5.3			
	*2,3,7,8-TCDF	20-130	<25	77.3	75.6	2.2			
	*1,2,3,7,8-PeCDF	20-130	<25	75.2	74.9	0.40			
	**2,3,4,7,8-PeCDF	20-130	<25	87.2	79.5	9.2			
	**1,2,3,4,7,8-HxCDF	20-130	<25	85.9	87.9	2.3			
	*1,2,3,6,7,8-HxCDF	20-130	<25	77.1	80.4	4.2			
	***1,2,3,7,8,9-HxCDF	20-130	<25	91.3	89.3	2.2			
	***2,3,4,6,7,8-HxCDF	20-130	<25	88.7	86.5	2.5			
	*1,2,3,4,6,7,8-HpCDF	20-130	<25	91.5	89.0	2.8			
	**1,2,3,4,7,8,9-HpCDF	20-130	<25	94.6	88.8	6.3			
	OCDF	20-130	<25	-	-	-			

*These recoveries were for the carbon-13 labeled internal standards.

**These recoveries were for the carbon-13 labeled surrogate standards.

***These recoveries were for the carbon-13 labeled surrogate “alternate” standards.

^aThese values were outside of the project specifications for acceptable recoveries.

^bResults from analysis of samples listed as “Acid Dissolution” in Table III.1.

^cResults from analysis of samples listed as “Fusion & Acid” in Table III.1.

^dResults from analysis of samples listed as “Misc. Volatiles” and “IC-Organics” in Table III.1.

^eResults from analysis of samples listed as “Semivolatiles” and “IC-Organics” in Table III.1.

^fResults from analysis of samples listed as “Dioxins&Furans” in Table III.1.

B. Analysis to Support Characterization of Immobilized Waste Form Samples

Recoveries associated with these analyses have been given in Table IV.3. Laboratory control samples were outside the specified range (75 to 125 %) ^{2,3} for platinum (3.5 %), tungsten (64 %), tantalum (13.8 %), and uranium (0 %). Several analytical spike sample recoveries were outside this range. Except arsenic (144 %), bismuth (73.0 %), and selenium (23.7 %) in the peroxide-

fusion samples, these were due to use a low spike concentration relative to sample concentration. Except the recoveries noted here and in Table IV.3, the analyses met all QA specifications.

Table IV.2. Recoveries for Toxicity Characteristic Leaching Procedure Extract Analyses

	Acceptable Values (%)		Measured Recoveries (%)		
	Recoveries	RPD	MS	MSD	RPD
Sb	75-125	<20	88.0	89.6	1.8
As	75-125	<20	106.5	110.8	3.9
Ba	75-125	<20	87.8	88.1	0.3
Be	75-125	<20	88.6	89.0	0.5
Cd	75-125	<20	86.6	83.6	3.5
Cr	75-125	<20	91.0	89.8	1.3
Pb	75-125	<20	89.0	87.2	2.1
Se	75-125	<20	96.9	104.2	6.7
Ag	75-125	<20	87.2	87.2	0
Ni	75-125	<20	92.5	92.3	0.2
Tl	75-125	<20	89.7	93.7	4.3
*Hg	75-125	<20	64.2	-	-

*Mercury matrix spike recovery was below the specified range. No duplicate was performed.

C. Analysis to Support Permitting

Results presented in this section are those that were specified in Table 3 of Reference 1. Except where noted below, these analyses met all QA requirements given in the QAPjP² and project task plan.³ As a result of discussions with RPP personnel, the lower limit on acceptable recovery range for organics was changed from 50 to 20 %. During these discussions, a decision was also made not to add the sodium thiosulfate preservative initially specified for some of the samples.³

1. Volatile Organic Compounds

Many of the target volatile organic analytes are not specifically addressed by any of the USEPA SW-846 methods. As a result, the methods used were not always capable of resolving one target analyte from another. This would have made quantify analytes difficult if both were present in the samples. This was not considered to have been a problem for samples in which neither of the analytes was present.

a. Volatile Organic Compounds Measured Using USEPA SW-846 Method 8260 (GC-MS)

Recoveries associated with these analysis have been presented in Table IV.4. All LCS recoveries were within the specified range (20 to 130 %). These analyses met all QA specifications except

WSRC-TR-2001-00566, Revision 0
SRT-RPP-2001-00013, Revision 1

Table IV.3. Recoveries for Analyses Used in Characterizing the Tank AN102 Waste Form

Analyte	Acceptable Values (%)		Acid Dissolution (%)			Peroxide Fusion (%)		
	Recoveries	RPD	MS	MSD	RPD	MS	MSD	RPD
Al	75-125	<20	^a b	^a *57.2	10.7	106.1	109.1	0.3
**B	75-125	<20	^a b	^a b	2.1	^a *73.5	^a *58.5	1.8
Be	75-125	<20	100.0	98.9	1.1	101.1	101.2	0.1
**Bi	75-125	<20	76.9	77.1	0.3	*66.1	*65.4	1.1
Ca	75-125	<20	^a b	^a *29.0	8.3	^a *152.0	^a *144.0	0.6
Co	75-125	<20	105.1	103.7	1.3	101.6	101.5	0.1
Cu	75-125	<20	105.8	105.4	0.4	102.5	103.0	0.5
Fe	75-125	<20	104.0	95.0	0.7	106.0	102.0	0.3
K	75-125	<20	103.8	104.0	0.2	104.5	104.8	0.3
**Li	75-125	<20	^a *61.9	79.7	4.5	108.1	111.0	0.7
Mg	75-125	<20	99.3	98.6	0.3	96.3	95.6	0.2
Mn	75-125	<20	99.8	106.2	1.1	97.4	97.3	0.1
**Mo	75-125	<20	100.7	99.3	1.4	99.2	99.9	0.6
Na	75-125	<20	87.0	^a *129.0	1.7	^a b	^a *300.0	0.3
**P	75-125	<20	98.4	98.3	0.1	96.7	96.8	0.1
**Pt	75-125	<20	113.0	108.8	3.7	117.5	105.4	10.9
**Pd	75-125	<20	^a *50.2	^a *52.0	0.9	^a *50.5	^a *50.7	0.1
**Rh	75-125	<20	104.9	104.7	0.2	100.0	99.8	0.3
**S	75-125	<20	98.5	100.5	1.6	97.8	100.5	2.3
Sb	75-125	<20	99.1	96.0	3.2	95.2	95.2	0
**Si	75-125	<20	^a *35.0	^a *445.0	7.1	^a b	^a b	0.8
**Sn	75-125	<20	102.1	101.1	1.0	99.3	100.2	0.9
**Ta	75-125	<20	89.2	91.3	2.1	*51.2	*40.2	19.7
Tl	75-125	<20	111.4	98.9	11.9	98.9	104.3	5.3
**U	75-125	<20	77.9	76.0	2.4	*68.1	84.4	*21.3
V	75-125	<20	104.7	104.0	0.7	102.5	102.7	0.2
**W	75-125	<20	96.9	94.4	2.6	87.5	82.8	5.5
**Y	75-125	<20	99.3	99.3	0.1	105.3	98.4	0.1
Zn	75-125	<20	101.0	90.8	1.5	102.9	98.1	0.7
**Zr	75-125	<20	^a *160.0	^a *154.9	1.1	^a *168.1	^a *157.0	2.5
PO ₄	75-125	<20	-	-	-	91.0	87.0	2.0
SO ₄	75-125	<20	-	-	-	77.0	80.0	1.1
	Acceptable Values (%)		Measured Recoveries (%)					
	Recoveries	RPD	MS			MSD		RPD
NH ₃	75-125	<20	100			101		2.0
⁺ Halide	75-125	<20	90			92		2.2

*Recoveries were not within the specified range for the matrix spikes or matrix spike duplicates for this analyte.

⁺Halide analysis recoveries are for the laboratory control sample. No matrix spike or duplicate was performed.

^aThese recoveries were out of specification because the sample concentration was high relative to the spike.

b indicates a calculated recovery that was less than 1.

**LCSs were prepared for these elements by spiking a blank with the solutions used to make matrix spikes.

Table IV.4. Laboratory Control Sample Recoveries for Results Determined by Method 8260B

Analyte	Recovery (%)	Analyte	Recovery (%)
Ethyl Benzene	102	cis-1,2-Dichloroethene	104
Styrene	104	Cyclopentane	-
cis-1,3-Dichloropropene	104	2-Butenaldehyde	-
trans-1,3-Dichloropropene	98	Carbon Tetrachloride	101
3-Heptanone	-	3-Methyl-2-Butanone**	-
m and p-Xylene	-	2-Hexanone	96
Ethylene Dibromide	104	Ethyl Alcohol***	-
Butane*	-	2-Propyl Alcohol***	-
1,3-Butadiene*	-	^a 2-Propanone (Acetone)	96
Acrolein	-	Chloroform	106
3-Chloropropene	-	Hexafluoroacetone**	-
1,2-Dichloroethane	104	n-Propyl Alcohol***	-
Propionitrile***	-	^b n-Butyl Alcohol	-
Acrylonitrile	-	Benzene	102
2-Pentanone***	-	1,1,1-Trichloroethane	-
4-Methyl-2-Pentanone	98	Bromomethane	85
m and p-Xylene	104	Chloromethane	104
Methylcyclohexane	-	Chloroethane	101
Toluene	100	1-Chloroethene	-
Chlorobenzene	100	Acetonitrile	-
Cyclohexanone	-	Dichloromethane	105
n-Pentane	-	Carbon Disulfide	98
Tetrahydrofuran	-	Oxirane	-
^c 5-Methyl-2-Hexanone	-	1,1-Dichloroethane	102
2-Heptanone	-	1,1-Dichloroethene	92
n-Hexane	-	Dichlorofluoromethane	-
Cyclohexane	-	Chlorodifluoromethane	-
Cyclohexene	-	2-Methyl-2-Propanol	-
n-Octane	-	Trichlorofluoromethane	99
^c n-Nonane	-	Dichlorodifluoromethane	100
4-Heptanone	-	^a 1,2,2-Trichloro-1,1,2-Trifluoroethane	-
n-Propionaldehyde	-	1,2,-Dichloro-1,1,2,2-Tetrafluoroethane	-
Acetic Acid n-Butyl Ester	-	1,2-Dichloropropane	100
1,4-Dioxane	-	1-Methylpropyl Alcohol***	-
2-Methyl-2-Propenenitrile	-	2-Butanone	96
Tetrachloroethene	102	1,1,2-Trichloroethane	106
Acetic Acid Ethyl Ester	-	1,1,2-Trichloroethylene	100
^b n-Heptane	-	1,1,2,2-Tetrachloroethane	-
Cyclopentane***	-	o-Xylene	106
trans-1,2-Dichloroethene	-		

^aAcetone and 1,2,2-trichloro-1,1,2-trifluoroethane would not have been resolved if both were present in a sample.

^bn-butyl alcohol and n-heptane would not have been resolved if both were present in a sample.

^c5-methyl-2-hexanone and n-nonane would not have been resolved if both were present in a sample.

- Parentheses indicate analytes for which laboratory control samples were not available.

*This analyte is a gas, and a standard could not be prepared. This analyte was monitored for but not seen as a TIC.

**A standard for this analyte was not detected at 20 ppm. This analyte was monitored for but not seen as a TIC.

***This analyte could only be monitored for as a TIC.

- Three pairs of analytes would have been resolved by the GC-MS (see Table IV.4).
- High-concentration points were dropped from several calibration curves due to lack of linearity.
- Approximately 80 % of the water and preservative was lost from one samples during sampling.
- Low-level tetrahydrofuran (THF) contamination was reported in the laboratory method blanks.

b. Organic Compounds Measured Using USEPA SW-846 Method 9056 (IC)

Matrix spike and matrix spike duplicate recoveries associated with the Method 9056 analyses for organic compounds have been given in Table IV.5. These analyses met all QA specifications except

- Matrix spike and duplicates were produced by spiking the extract rather than the initial sample.
- Preliminary analyses using calibration solutions with all analytes was only valid for oxalate.
- Since acetate and formate could not be resolved, separate analyses were performed.

2. Semi-Volatile Organic Compounds

Matrix spike and matrix spike duplicate recoveries associated with the semivolatile organic compound analyses have been given in Table IV.6. These analyses met all QA specifications except

- One of the eight internal standards was low for the Method 8270C matrix spike duplicate.
- The instrument tune could not be evaluated for the triethylamine analysis.
- The internal standard was outside of the QC limits for the field blank and one sample.

3. Pesticides

Matrix spike and matrix spike duplicate recoveries associated with the pesticide analysis have been given in Table IV.7. One pesticide sample was lost during the sample preparation. Duplicate results for these analyses were taken from an extra ion chromatography sample that was shipped along with the pesticide samples. In addition, one of the continuing calibration factors for octachloronaphthalene and one of the continuing calibration factors for isodrin were slightly high (16.1 and 15.2 % respectively) compared to the Method 8081A criterion (< 15 %).

Table IV.5. Matrix Spike and Duplicate Recoveries for Organic Compounds Determined by IC

Analyte	Acceptable Values (%)		Measured Recoveries (%)		
	Recoveries	RPD	MS	MSD	RPD
Formate	20-130	<25	98.0	98.0	0
Acetate	20-130	<25	89.6	91.7	2.3
Oxalate (oxalate alone)	20-130	<25	101.9	104.2	2.2
Oxalate (oxalate and formate)	20-130	<25	99.5	100.6	0.5
Oxalate (oxalate and acetate)	20-130	<25	98.3	97.8	1.1

Table IV.6. Matrix Spike and Duplicate Recoveries for Semivolatile Organic Compounds

Analyte	Acceptable Values (%)		Measured Recoveries (%)		
	Recoveries	RPD	MS	MSD	RPD
Phenol	20-130	<25	78	65	18
1,4-Dichlorobenzene	20-130	<25	76	63	19
1,2,4-Trichlorobenzene	20-130	<25	88	71	21
2,4-Dinitrotoluene	20-130	<25	71	68	4
Pentachlorophenol	20-130	<25	74	73	1

Table IV.7. Matrix Spike and Duplicate Recoveries for Pesticide Analyses

Analyte	Acceptable Values (%)		Measured Recoveries (%)		
	Recoveries	RPD	MS	MSD	RPD
Aldrin	20-130	<25	104	103	1
gamma-BHC (Lindane)	20-130	<25	102	103	1
4,4'-DDT	20-130	<25	106	108	2
Dieldrin	20-130	<25	106	108	2
Endrin	20-130	<25	*131	123	6
Heptachlor	20-130	<25	101	103	2

*This recovery was outside of project specifications but met SW-846 Method 8081A QC limits.

4. Polychlorinated Biphenyls (PCBs)

Matrix spike and matrix spike duplicate recoveries associated with the polychlorinated (PCB) analyses have been given in Table IV.8. These analyses met all QA specifications except

- One of the extra ion chromatography samples was analyzed as the duplicate for these analyses.
- The matrix spike and duplicate were created from the ion chromatography sample duplicate.
- All results were taken from Column 1 of the GC-MS because some of the Column 2 continuing calibration factors did not meet the SW-846 specifications.

Table IV.8. Matrix Spike and Duplicate Recoveries for Polychlorinated Biphenyl Analyses

Analyte	Acceptable Values (%)		Measured Recoveries (%)		
	Recoveries	RPD	MS	MSD	RPD
Aroclor-1016	20-130	<25	84	80	5
Aroclor-1260	20-130	<25	58	55	5

*Detection limits were 2×10^2 and $50 \mu\text{g/kg}$ for the samples and field blank respectively.

**Detection limits were 3×10^2 and $1 \times 10^2 \mu\text{g/kg}$ for the samples and field blank respectively.

D. Radionuclides

Recoveries associated with the radionuclide analyses have been presented in Table IV.9. These analyses met all QA specifications except

- No standard was available for selenium-79. In addition, selenium recoveries were low.
- Carbon-14 analysis showed substantial interferences from other beta-emitting species.
- Radium-226, europium-154, cobalt-60, and cesium-134 were “nondetects”.
- Two of eight uranium-234 results and one of five curium-244 results were “nondetects”.

Table IV.9. Matrix Spike and Laboratory Control Sample Radionuclide Recoveries

Analyte	Acceptable Values (%)		Measured Recoveries (%)			
	Recoveries	RPD	MS	LCS	LCSD	RPD
Am-241	70-125	<25	-	92	-	-
Total Alpha	70-125	<25	*32	104	-	-
Total Beta	70-125	<25	*160	110	-	-
Sr-90 (Y-90)	70-125	<25	-	103	-	-
Tc-99	70-125	<25	-	*66	-	-
Pu-239/240	70-125	<25	-	113	-	-
Pu-241	70-125	<25	-	*59	-	-
H-3	70-125	<25	89	100	92	8.3
I-129	70-125	<25	-	94	83	12
U-234	70-125	<25	-	109	-	-
U-235	70-125	<25	-	99.95	-	-
U-238	70-125	<25	-	112	-	-
C-14	70-125	<25	-	73	70	4.2

*These recoveries were not within the specified range.

E. Density and Ignitability

No matrix spikes, matrix spike duplicates, or laboratory control spikes are required for these methods. For the density determination, water was used to calibrate the method. No calibration was need for the ignitability test. With the exception of sample sizes for the ignitability tests, these tests were performed according to the standard methods and were implemented by BWXT as standard operating procedures (SOPs).

5.0 Results

Results from the sample analyses are presented in this chapter. In most cases, these results are presented in the same order as the analytes listed in Tables 1 through 5 of Reference 1. The title of each section in this chapter was taken from the corresponding table in Reference 1.

A. Analysis to Support Dangerous Waste Form Characteristics Determination

Results presented in this section were those specified in Table 1 of Reference 1. All results showed the glass waste form did not have any characteristics of Hazardous Waste. Specifically, the waste form was not ignitable, not reactive, not toxic for RCRA metals, and did not contain cyanide concentrations above those specified in the Code of Federal Regulations.

1. Cyanide

As expected, cyanide was not detected in the samples or in the blanks. Any cyanide that may have been present in the feed was expected to have been converted to carbon dioxide (which would have been released to the off gas) and a nitrogen oxide (most of which would have been released to the off gas). Results for the cyanide analyses were <0.1 and <0.2 mg/kg for the duplicate samples and <0.1 mg/kg for the field blank. Detection limits were limited by the quantity of sample available for the analyses. The results showed cyanide concentrations in the glass to be well below the land disposal restriction limits given in 40CFR268 for total cyanide (590 mg/kg) and for amenable cyanide (30 mg/kg). Cyanide matrix spike and duplicate recoveries were between 82 and 106 %.

2. Dangerous Waste Characteristics

Results from the RCRA metals analyses and TCLP leachate analyses have been given in Table V.1 and Table V.3. All target TCLP analyte results were well below any of the universal treatment standard (UTS) limits. Even barium was approximately one-seventh the UTS limit. From the results in Table V.1 for the total metals, only cadmium, chromium, silver, and lead would have failed TCLP even if all of these species were to leach from the glass waste form. Matrix spike and duplicate recoveries that did not meet the specified values have been noted in the tables.

Detection and quantitation limits for these analyses have been given in Table V.2. Quantitation limits for the metal analytes were estimated quantitation limits (EQLs) and were 10 times the detection limits. The overall dilution factors for these analytes came from the sample dissolution (nominally 2×10^2 L/kg) and the acid digestion (a factor of 20 except mercury which was a factor of 2). Quantitation limits for the dioxins and furans were the minimum method calibration limits (MCLs). The dilution factors for the dioxin and furan analyses were from the sample extraction.

In addition to the results given in Table V.1, reagents were added to empty dissolution vessels and subjected to each of the dissolution procedures in parallel to the samples. Results from these method blanks were used to determine the concentration of analytes that would have been present had a sample of 0.5 g (nominal mass used) been dissolved. The method blank results for the peroxide fusion were similar to the analytical samples for arsenic (3.2 mg/kg), lead (2.2×10^2 mg/kg), and mercury (0.12 mg/kg). In addition, the peroxide fusion method blank chromium

results were 61 mg/kg which was 25 % of the sample result. For the acid method blank, mercury results were 0.40 which was similar to the sample results as shown in Table V.1.

Table V.1. Analyses to Determine if Samples Have Dangerous Waste Characteristics
(note that units have been given in the “Characteristic” column)

Characteristic	Analyte	Samples				Field Blank	
^a Ignitability	-	*N F		*N F		*N F	
		^b Acid		^c Fusion		^b Acid	^c Fusion
Total Metals (mg/kg)	As	<2	<2	***<2	***<2	<2	***<4
	Ba	(38)	(38)	(31)	(36)	(14)	(10)
	Cd	(26)	(26)	(26)	(27)	1.4x10 ³	1.5x10 ³
	Cr	2.2x10 ²	2.3x10 ²	2.3x10 ²	2.5x10 ²	1.4x10 ³	1.5x10 ³
	Pb	(1.5x10 ²)	(1.1x10 ²)	<1.1x10 ²	(2.0x10 ²)	(9.7x10 ²)	(1.0x10 ³)
	Se	^R <5	^R <5	^R <5	^R <5	^R <5	^R <10
	Ag	^J 1.0x10 ²	^J 1.1x10 ²	^J 99	^J 1.1x10 ²	^J 24	^J 45
	Ni	(2.0x10 ²)	(1.8x10 ²)	-	-	1.5x10 ³	-
	Hg	0.43	0.45	-	-	0.49	-
^d Volatiles	-	Sect. 5.C.1		Sect. 5.C.1		Sect. 5.C.1	
^e Semi-Volatiles	-	Sect. 5.C.2		Sect. 5.C.2		Sect. 5.C.2	
^f Dioxins & Furans (µg/kg)	2,3,7,8,-TCDD	<0.1		<0.09		<0.05	
	1,2,3,7,8-PeCDD	<0.1		<0.09		<0.05	
	1,2,3,4,7,8-HxCDD	<0.1		<0.1		<0.06	
	1,2,3,6,7,8-HxCDD	<0.1		<0.1		<0.06	
	1,2,3,7,8,9-HxCDD	<0.1		<0.1		<0.06	
	1,2,3,4,6,7,8-HpCDD	<0.2		<0.2		<0.09	
	**OCDD	^{J_B} <0.47		^{J_B} 0.70		^{J_B} <0.54	
	2,3,7,8-TCDF	<0.09		<0.07		<0.04	
	1,2,3,7,8-PeCDF	<0.1		<0.07		<0.05	
	2,3,4,7,8-PeCDF	<0.1		<0.07		<0.04	
	1,2,3,4,7,8-HxCDF	<0.09		<0.07		<0.04	
	1,2,3,6,7,8-HxCDF	<0.09		<0.07		<0.04	
	1,2,3,7,8,9-HxCDF	<0.1		<0.07		<0.04	
	2,3,4,6,7,8-HxCDF	<0.1		<0.09		<0.05	
	1,2,3,4,6,7,8-HpCDF	<0.1		<0.09		<0.05	
	1,2,3,4,7,8,9-HpCDF	<0.1		<0.1		<0.07	
	OCDF	<0.3		<0.2		<0.01	

<These analyte concentrations were less than the MDL. This is commonly designated with a “U” data qualifier.

()These analytes were detected but at less than the EQL. This is commonly designated with a “J” data qualifier.

^BThis analyte was detected in the laboratory method blank at a concentration of 1.4 ppt.

^RThese results are “unusable”. A determination cannot be made as to whether the analyte was present at the reported MDL.

^JTreat these results as estimates. The analyte is regarded as detected, but its reported concentration is uncertain.

*N F indicates a “not flammable” result in the ignitability test (Method 1010 modified for smaller sample size).

**Results are to be interpreted as nondetects based on application of the CLP 5X rule to the method blank. See footnote B.

***Matrix spike and matrix spike duplicate recoveries were greater than the specified limit of 125 %. See Table V.A.

^aResults from analysis of samples and blanks listed as “Miscellaneous” in Table III.1.

^bResults from analysis of samples and blanks listed as “Acid Dissolution” in Table III.1.

^cResults from analysis of samples and blanks listed as “Fusion & Acid” in Table III.1.

^dResults from analysis of samples and blanks listed as “Misc. Volatiles” and “IC-Organics” in Table III.1.

^eResults from analysis of samples and blanks listed as “Semivolatiles” and “IC-Organics” in Table III.1.

^fResults from analysis of samples and blanks listed as “Dioxins&Furans” in Table III.1.

Table V.2. Detection and Quantitation Limits for Dangerous Waste Characteristics Tests

Characteristic	Analyte	Dilution (L/kg)		Instrument Limits (µg/L)			Method Limits (mg/kg)			
Total Metals				Detection		Quantitation	Detection		Quantitation	
	As	4.0 x 10 ³		0.52		5.2	2.1		21	
	Ba	4.0 x 10 ³		1.0		10	4.0		40	
	Cd	4.0 x 10 ³		1.2		12	4.8		48	
	Cr	4.0 x 10 ³		2.5		25	10		1.0 x 10 ²	
	Pb	4.0 x 10 ³		28		280	1.1 x 10 ²		1.1 x 10 ³	
	Se	4.0 x 10 ³		1.3		13	5.2		52	
	Ag	4.0 x 10 ³		4.1		41	16		1.6 x 10 ²	
	Ni	4.0 x 10 ³		6.45		64.5	26		2.6 x 10 ²	
	Hg	4.0 x 10 ²		0.05		0.5	0.02		0.2	
^d Volatiles	-	Sect. 5.C.1		Sect.5.C.1			Sect.5.C.1			
^e Semi-Volatiles	-	Sect. 5.C.2		Sect. 5.C.2			Sect. 5.C.2			
^f Dioxins & Furans				Instrument (µg/L)			Method (µg/kg)			
				Detection		Quantitation	Detection		Quantitation	
	Sample Number	1	2	1	2	-	1	2	1	2
	2,3,7,8,-TCDD	43.4	42.2	0.1	0.09	0.50	4.3	3.8	22	21
	1,2,3,7,8-PeCDD	43.4	42.2	0.1	0.09	2.5	4.3	3.9	109	106
	1,2,3,4,7,8-HxCDD	43.4	42.2	0.1	0.1	2.5	4.3	4.2	109	106
	1,2,3,6,7,8-HxCDD	43.4	42.2	0.1	0.1	2.5	4.3	4.2	109	106
	1,2,3,7,8,9-HxCDD	43.4	42.2	0.1	0.1	2.5	4.3	4.2	109	106
	1,2,3,4,6,7,8-HpCDD	43.4	42.2	0.2	0.2	2.5	8.7	8.4	109	106
	**OCDD	43.4	42.2	0.47	0.47	5.0	20	20	217	211
	2,3,7,8-TCDF	43.4	42.2	0.09	0.07	0.50	3.9	3.0	22	21
	1,2,3,7,8-PeCDF	43.4	42.2	0.1	0.07	2.5	4.3	3.0	109	106
	2,3,4,7,8-PeCDF	43.4	42.2	0.1	0.07	2.5	4.3	3.0	109	106
	1,2,3,4,7,8-HxCDF	43.4	42.2	0.09	0.07	2.5	3.9	3.0	109	106
	1,2,3,6,7,8-HxCDF	43.4	42.2	0.09	0.07	2.5	3.9	3.0	109	106
	1,2,3,7,8,9-HxCDF	43.4	42.2	0.1	0.07	2.5	4.3	3.0	109	106
	2,3,4,6,7,8-HxCDF	43.4	42.2	0.1	0.09	2.5	4.3	3.8	109	106
	1,2,3,4,6,7,8-HpCDF	43.4	42.2	0.1	0.09	2.5	4.3	3.8	109	106
	1,2,3,4,7,8,9-HpCDF	43.4	42.2	0.1	0.1	2.5	4.3	4.2	109	106
		OCDF	43.4	42.2	0.3	0.2	5.0	13	8.4	217

**The detection limits reported here are concentration detected in the method blank.

^bResults from analysis of samples and blanks listed as "Acid Dissolution" in Table III.1.

^cResults from analysis of samples and blanks listed as "Fusion & Acid" in Table III.1.

^dResults from analysis of samples and blanks listed as "Misc. Volatiles" and "IC-Organics" in Table III.1.

^eResults from analysis of samples and blanks listed as "Semivolatiles" and "IC-Organics" in Table III.1.

^fResults from analysis of samples and blanks listed as "Dioxins&Furans" in Table III.1.

Detection and quantitation limits for the TCLP leachates have been given in Table V.4. The dilution factors were from the leachate acid digestion. Laboratories commonly do not perform this digestion prior to analysis; however, SW-846 Method 1311 requires an acid digestion. Quantitation limits were estimated quantitation limits (EQL) defined as 10 times the detection limits.

Table V.3. Results from TCLP, Toxicity Characteristic Leaching Procedure

Analyte	UTS Limit (µg/L)	Samples (µg/L)		Field Blanks (µg/L)	
Sb	1.15×10^3	$<2 \times 10^2$	$<2 \times 10^2$	$<2 \times 10^2$	$<2 \times 10^2$
As	5.0×10^3	^j <5	^j <5	^j <5	^j <5
Ba	2.1×10^4	^u 2.9×10^3	^u 3.0×10^3	^u 2.9×10^3	^u 3.1×10^3
Be	1.22×10^3	<2	<2	<2	<2
Cd	1.1×10^2	<12	<12	<12	<12
Cr	6.0×10^2	<25	<25	<25	<25
Pb	7.5×10^2	$<3 \times 10^2$	$<3 \times 10^2$	$<3 \times 10^2$	$<3 \times 10^2$
Se	5.7×10^3	(15)	<13	(14)	(17)
Ag	1.4×10^2	<41	<41	<41	<41
Ni	1.1×10^4	<64	(65)	<64	<64
Tl	2.0×10^2	$<6 \times 10^2$	$<6 \times 10^2$	$<6 \times 10^2$	$<6 \times 10^2$
**Hg	25	^j <0.1	^j <0.1	^j <0.1	^j <0.1

<These analyte concentrations were less than the MDL (commonly designated with a “U” data qualifier).

()These analytes were detected but at less than the EQL (commonly designated with a “J” data qualifier).

^jThese analytes were regarded as not detected, but the reported detection limit was uncertain.

^uThis analyte was regarded as not detected. The detection limit should be considered the value given.

**Matrix spike recovery for mercury was 64 % (not within the specified range of 75 to 125 %).

B. Analysis to Support Characterization of Immobilized Waste Form Samples

Results from elemental and anion analyses have been presented in Table V.5. These results show the composition of the glass waste form produced in this study was very close to the target composition. The target is the composition that was expected based on analysis of the feed material and the composition of the glass-forming minerals added to the feed. The ratio of feed to glass-forming minerals was provided to SRTC by the Vitreous State Laboratory. A mass balance can be evaluated by comparing the target and the measured waste form compositions.

Detection and quantitation limits for these analyses have been given in Table V.6. Quantitation limits for metal and anion analytes were EQLs (10 times the detection limits). Ammonia quantitation limits were minimum MCLs. No detection limit was reported for ammonia. The dilution factors for metal and anion analytes were the product of the dissolution factor (nominally 2×10^2 L/kg) and subsequent pretreatment factors (20 for metals and 10 for anions). The ammonia dilution factor was the ratio of sample mass to volume of the test solution.

Thallium detection limits were higher than expected. The results given in Table V.3 and V.5 could not be used to show the thallium concentration in the TCLP leachate would be below the UTS limit. Although thallium was not one of the target analytes for the TCLP leachate, it is a RCRA metal. Although thallium was also not one of the analytes in the analyses described in Reference 4, unpublished inductively-coupled plasma-mass spectrometry (ICP-MS) data showed that the concentration of thallium (mass 205) in the glass waste form was well below concentrations that would be of regulatory concern. Palladium concentrations were higher than

expected in the samples and in the LRM glass that was submitted with the analyses. This suggested that the reported palladium was due to the preparation and analysis of the samples. In addition, palladium matrix spike and matrix spike duplicate recoveries were outside of the range specified in Reference 2.

Table V.4. Detection and Quantitation Limits for TCLP Leachates

Analyte	Dilution	Instrument Limits (µg/L)		Method Limits (µg/L)	
		Detection	Quantitation	Detection	Quantitation
Sb	10	19	1.9×10^2	1.9×10^2	1.9×10^3
As	10	0.52	5.2	5.2	52
Ba	10	1.0	10	10	1.0×10^2
Be	10	0.2	2	2	20
Cd	10	1.2	12	12	1.2×10^2
Cr	10	2.5	25	25	2.5×10^2
Pb	10	30	3.0×10^2	3.0×10^2	3.0×10^3
Se	10	1.3	13	13	1.3×10^2
Ag	10	4.1	41	41	4.1×10^2
Ni	10	6.45	64.5	64.5	6.45×10^2
Tl	10	61	6.1×10^2	6.1×10^2	6.1×10^3
Hg	2	0.05	0.5	0.1	1

Most primary component concentrations (concentrations >1000 mg/kg) were close to the target values. Measured concentrations of manganese were significantly higher than the expected 0.77 mg/kg. The target value was based on the measured concentration of manganese in the pretreated feed. Manganese in the glass-forming minerals was not taken into account. The higher measured concentrations are expected to be caused by the presence of manganese in wollastonite.

C. Analysis to Support Permitting

Results presented in this section are those that were specified in Table 3 of Reference 1. As a result of conversations with RPP personnel, ammonium perfluorooctanoate, methyl isocyanate, and picric acid were removed from the target analyte list. Accordingly, these analytes were excluded from the target analyte lists in the project QAPjP² and in the resulting task plan.³

1. Volatile Organic Compounds

All volatile organic results should be considered “nondetects”. With four exceptions, none of the target analytes were detected. These four exceptions were acetone, chlorodifluoromethane, 2-methyl-2-propanol, and tetrahydrofuran. These four analytes were detected in the samples and were also shown to be present in the field blanks. Application of the USEPA Contract Laboratory Program (CLP) 5x rule would dictate these results be interpreted as “nondetects”.

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Table V.5. Results (mg/kg) in Support of Characterizing Tank AN102 Waste Form Samples

	Sample (mg/kg)					Field Blank (mg/kg)		
	Acid		Fusion		#Target	Acid	Fusion	##Actual
Al	3.4x10 ⁴	3.3x10 ⁴	3.3x10 ⁴	3.4x10 ⁴	3.3x10 ⁴	5.3x10 ⁴	5.4x10 ⁴	5.1x10 ⁴
B	ND	ND	3.0x10 ⁴	3.0x10 ⁴	3.1x10 ⁴	ND	2.3x10 ⁴	2.4x10 ⁴
Be	<0.9	<0.9	<0.9	<0.9	-	<0.9	<2	-
Bi	<1.9x10 ²	<2x10 ²	^J <2x10 ²	^J <2x10 ²	-	<2x10 ²	^J <4x10 ²	-
Ca	4.7x10 ⁴	4.5x10 ⁴	4.6x10 ⁴	4.9x10 ⁴	4.6x10 ⁴	3.7x10 ³	5.4x10 ³	3.8x10 ³
Co	<10	<10	(95)	1.0x10 ²	-	<10	3.3x10 ²	3.3x10 ²
Cu	(64)	(71)	1.0x10 ²	1.2x10 ²	3.6	6.7x10 ²	8.6x10 ²	8.6x10 ²
Fe	4.5x10 ⁴	4.4x10 ⁴	4.4x10 ⁴	4.6x10 ⁴	4.5x10 ⁴	1.0x10 ⁴	1.1x10 ⁴	9.6x10 ³
K	(9.6x10 ²)	(1.0x10 ³)	(1.4x10 ³)	(2.0x10 ³)	7.5x10 ²	1.2x10 ⁴	1.4x10 ⁴	1.2x10 ⁴
Li	^J 1.3x10 ⁴	^J 1.2x10 ⁴	1.3x10 ⁴	1.3x10 ⁴	1.4x10 ⁴	^J 4.6x10 ²	5.0x10 ²	5.2x10 ²
Mg	9.2x10 ³	9.2x10 ³	9.0x10 ³	9.5x10 ³	9.1x10 ³	(6.0x10 ²)	(5.1x10 ³)	6.2x10 ²
Mn	2.1x10 ²	2.1x10 ²	6.8x10 ²	7.3x10 ²	0.77	(5.7x10 ²)	2.4x10 ³	6.1x10 ²
Mo	<56	<57	<56	<56	17	6.6x10 ²	6.8x10 ²	6.7x10 ²
Na	9.2x10 ⁴	9.0x10 ⁴	ND	ND	8.8x10 ⁴	1.5x10 ⁵	ND	1.5x10 ⁵
P	(6.2x10 ²)	(5.0x10 ²)	(5.6x10 ²)	(5.8x10 ²)	5.6x10 ²	2.1x10 ³	2.1x10 ³	2.4x10 ³
Pt	^J <1x10 ²	^J <1x10 ²	^J <1x10 ²	^J (1.4x10 ²)	-	^J <1x10 ²	^J <2x10 ²	-
Pd	^J 1.1x10 ⁴	^J 1.1x10 ⁴	^J 1.0x10 ⁴	^J 1.1x10 ⁴	-	^J (3.4x10 ³)	^J (3.7x10 ³)	-
Rh	<2x10 ³	<2x10 ³	<2x10 ³	<2x10 ³	-	<2x10 ³	<4x10 ³	-
S	1.8x10 ³	1.7x10 ³	1.8x10 ³	1.9x10 ³	1.4x10 ³	9.5x10 ²	(1.1x10 ³)	1.6x10 ³
Sb	<77	<77	<77	<77	-	<77	<2x10 ²	-
Si	2.2x10 ⁵	2.0x10 ⁵	1.9x10 ⁵	2.0x10 ⁵	2.2x10 ⁵	2.3x10 ⁵	2.3x10 ⁵	2.5x10 ⁵
Sn	<2x10 ²	<2x10 ²	<2x10 ²	<2x10 ²	8.7	<2x10 ²	<3x10 ²	4.1x10 ²
Ta	^J (8.2x10 ²)	^J (9.4x10 ²)	^J (7.9x10 ²)	^J (9.1x10 ²)	-	^J <6x10 ²	^J <1x10 ³	-
Tl	<2x10 ²	<2x10 ²	<2x10 ²	<2x10 ²	-	<2x10 ²	<5x10 ²	-
U	^J <3x10 ³	^J <3x10 ³	^J <3x10 ³	^J <3x10 ³	-	^J <3x10 ³	^J <5x10 ³	-
V	(60)	(61)	(73)	(82)	0.56	<18	<35	-
W	^J <3x10 ²	^J <3x10 ²	^J <3x10 ²	^J <3x10 ²	-	^J <3x10 ²	^J <5x10 ²	-
Y	(34)	<32	(38)	<32	-	<32	<64	-
Zn	2.4x10 ⁴	2.3x10 ⁴	2.3x10 ⁴	2.4x10 ⁴	2.4x10 ⁴	(1.1x10 ²)	<30	-
Zr	^J 2.3x10 ⁴	^J 2.3x10 ⁴	^J 2.3x10 ⁴	^J 2.4x10 ⁴	2.2x10 ⁴	^J 7.2x10 ³	^J (7.4x10 ³)	6.9x10 ³
Cl	ND	ND	^J <78	^J <79	1.2x10 ³	ND	^J <2x10 ²	1.2x10 ³
F	ND	ND	^J <52	^J <52	6.0x10 ²	ND	^J <100	8.1x10 ³
PO ₄	ND	ND	1.9x10 ³	1.7x10 ³	-	ND	4.8x10 ³	3.7x10 ⁴
SO ₄	ND	ND	3.4x10 ³	3.5x10 ³	-	ND	1.8x10 ³	3.9x10 ³
NH ₃	18.3		18.3		-	45		-
Halide	99		53		-	41		-

ND analyte could not be determined in the sample indicated. Neither a concentration nor a detection limit is available.

<These analyte concentrations were less than the MDL (commonly designated with a "U" data qualifier).

()These analytes were detected but at less than the EQL. This is commonly designated with a "J" data qualifier.

^JTreat these results as estimates. Matrix spike, matrix duplicate or laboratory control sample recovery was unacceptable.

[#]This was the target waste form composition agreed upon by the Vitreous State Laboratory, RPP and SRTC personnel.

^{##}This is the actual composition of LRM glass.⁹ This was not a true field blank. Some of the analytes were known to be present.

Recoveries were within the specified limit^{2, 3} (between 20 and 130 % with relative deviations of less than 20 %).

Table V.6. Detection and Quantitation Limits Tank AN102 Waste Form Characterization

Analyte	Dilution (L/kg)	Instrument Limits (µg/L)		Method Limits (mg/kg)	
		Detection	Quantitation	Detection	Quantitation
Al	4.0 x 10 ³	12	1.2 x 10 ²	48	4.8 x 10 ²
B	4.0 x 10 ³	4.9	49	20	2.0 x 10 ²
Be	4.0 x 10 ³	0.2	2	0.8	8
Bi	4.0 x 10 ³	49	4.9 x 10 ²	2.0 x 10 ²	2.0 x 10 ³
Ca	4.0 x 10 ³	53	5.3 x 10 ²	2.1 x 10 ²	2.1 x 10 ³
Co	4.0 x 10 ³	2.6	26	10	1.0 x 10 ²
Cu	4.0 x 10 ³	2.7	27	10	1.0 x 10 ²
Fe	4.0 x 10 ³	29	2.9 x 10 ²	1.1 x 10 ²	1.1 x 10 ³
K	4.0 x 10 ³	56	5.6 x 10 ²	2.2 x 10 ²	2.2 x 10 ³
Li	4.0 x 10 ³	6.1	61	24	2.4 x 10 ²
Mg	4.0 x 10 ³	20	2.0 x 10 ²	80	8.0 x 10 ²
Mn	4.0 x 10 ³	1.1	11	4.4	44
Mo	4.0 x 10 ³	14	1.4 x 10 ²	64	6.4 x 10 ²
Na	4.0 x 10 ³	22	2.2 x 10 ²	88	8.8 x 10 ²
P	4.0 x 10 ³	99	9.9 x 10 ²	4.0 x 10 ²	4.0 x 10 ³
Pt	4.0 x 10 ³	28	2.8 x 10 ²	1.1 x 10 ²	1.1 x 10 ³
Pd	4.0 x 10 ³	1.5 x 10 ²	1.5 x 10 ³	6.0 x 10 ²	6.0 x 10 ³
Rh	4.0 x 10 ³	4.9 x 10 ²	4.9 x 10 ³	2.0 x 10 ³	2.0 x 10 ⁴
S	4.0 x 10 ³	21	2.1 x 10 ²	84	8.4 x 10 ²
Sb	4.0 x 10 ³	19	1.9 x 10 ²	76	7.6 x 10 ²
Si	4.0 x 10 ³	1.6 x 10 ²	1.6 x 10 ³	6.4 x 10 ²	6.4 x 10 ³
Sn	4.0 x 10 ³	42.5	4.25 x 10 ²	1.7 x 10 ³	1.7 x 10 ⁴
Ta	4.0 x 10 ³	1.5 x 10 ²	1.5 x 10 ³	6.0 x 10 ²	6.0 x 10 ³
Tl	4.0 x 10 ³	61	6.1 x 10 ²	2.4 x 10 ²	2.4 x 10 ³
U	4.0 x 10 ³	6.6 x 10 ²	6.6 x 10 ³	2.6 x 10 ³	2.6 x 10 ⁴
V	4.0 x 10 ³	4.4	44	18	1.8 x 10 ²
W	4.0 x 10 ³	67	6.7 x 10 ²	2.7 x 10 ²	2.7 x 10 ³
Y	4.0 x 10 ³	8.0	80	32	3.2 x 10 ²
Zn	4.0 x 10 ³	3.7	37	15	1.5 x 10 ²
Zr	4.0 x 10 ³	1.2 x 10 ²	1.2 x 10 ³	4.8 x 10 ²	4.8 x 10 ³
Cl	2.0 x 10 ³	39.5	3.95 x 10 ²	79	7.9 x 10 ²
F	2.0 x 10 ³	26	2.6 x 10 ²	52	5.2 x 10 ²
PO ₄	2.0 x 10 ³	91	9.1 x 10 ²	1.8 x 10 ²	1.8 x 10 ³
SO ₄	2.0 x 10 ³	41	4.1 x 10 ²	82	8.2 x 10 ²
NH ₃	83	-	100	-	83

Note that although Method 8000B of USEPA SW-846 indicates use of 70 to 130 % until acceptance criteria are established for an analyte, acceptance criteria below the lower limit of 70 % had already been established for some of the target analytes. In addition, some of the nonroutine target analytes were known to experience poor recoveries and were expected to fail the 70 % limitation. Therefore, as a result of conversations with RPP personnel, the specified range was extended to between 20 and 130 in Attachment 1 of the project task plan.³

a. Volatile Organic Compounds Measured Using USEPA SW-846 Method 8260 (GC-MS)

Results from the Method 8260B analysis have been presented in Table V.7. Acetone, chlorodifluoromethane, 2-methyl-2-propanol, and tetrahydrofuran were detected in the samples at low concentrations (between 3 and 19 µg/kg). These analytes were also detected in the field blanks at similar concentrations (between 4 and 16 µg/kg). In addition, acetone and tetrahydrofuran were detected in the trip blank at 6.7 and 9.1 µg/kg respectively.

The minimum method quantitation levels (MQLs) were used as the estimated quantitation levels (EQLs) for these analyses. For the analytes not detected, these values have been given in Table V.7. For tetrahydrofuran, acetone, and chlorodifluoromethane, the EQLs were 100, 10, and 10 µg/kg respectively. For these analyses, samples were not diluted. Therefore, the instrument quantitation limits were the same as the quantitation limits for the samples.

b. Organic Compounds Measured Using USEPA SW-846 Method 9056 (IC)

Results from Method 9056 analyses for organic compounds have been given in Table V.8. As expected, none of these analytes were detected. Table V.8 includes results for the two volatile compounds acetate and formate as well as the semivolatile compound oxalate. These results have been included together because they were determined by the same method using the same samples.

Detection and quantitation limits for the Method 9056 organic compound analyses have been given in Table V.9. Detection limits were determined according to SW-846 Chapter 1. Quantitation limits were EQLs determined as 10 times the detection limits. The dilution factors were from the extraction of the waste form in which 50 mL of hot water was used to extract 2 grams of glass. The resulting extract was analyzed without further dilution.

2. Semi-Volatile Organic Compounds

As shown in Table V.10, no target semi-volatile analyte was detected. One non-target compound (1,1,2,2-tetrachloroethane) was seen as a tentatively-identified compound (TIC) in a sample and the field blank. Approximate concentrations were 3.1×10^2 and 2.7×10^2 µg/kg in the sample and blank respectively. Application of the USEPA Contract Laboratory Program (CLP)⁶ 5x rule would dictate the result be considered a “nondetect”. Matrix spike and duplicate recoveries met the specified limit^{2,3} (20 to 130 % with relative deviations below 20 %) except the relative deviation (21 %) for 1,2,4-trichlorobenzene.

Table V.7. Volatile Organic Compound Results (µg/kg) Determined by Method 8260B

Analyte	Concentration		Analyte	Concentration	
Ethyl Benzene	<10	<10	cis-1,2-Dichloroethene	<10	<10
Styrene	<10	<10	Cyclopentane	<10	<10
cis-1,3-Dichloropropene	<10	<10	2-Butenaldehyde	<10	<10
trans-1,3-Dichloropropene	<10	<10	Carbon Tetrachloride	<10	<10
3-Heptanone	<10	<10	*3-Methyl-2-Butanone	TIC	TIC
m and p-Xylene	<10	<10	2-Hexanone	<10	<10
Ethylene Dibromide	<10	<10	*Ethyl Alcohol	TIC	TIC
*Butane	TIC	TIC	*2-Propyl Alcohol	TIC	TIC
*1,3-Butadiene	TIC	TIC	^{a,b} 2-Propanone (Acetone)	(5.6)	(10)
Acrolein	<100	<100	Chloroform	<10	<10
3-Chloropropene	<10	<10	*Hexafluoroacetone	TIC	TIC
1,2-Dichloroethane	<10	<10	*n-Propyl Alcohol	TIC	TIC
*Propionitrile	TIC	TIC	n-Butyl Alcohol	<100	<100
Acrylonitrile	<100	<100	Benzene	<10	<10
*2-Pentanone	TIC	TIC	1,1,1-Trichloroethane	<10	<10
4-Methyl-2-Pentanone	<10	<10	Bromomethane	<10	<10
m and p-Xylene	<10	<10	Chloromethane	<10	<10
Methylcyclohexane	<10	<10	Chloroethane	<10	<10
Toluene	<10	<10	1-Chloroethene	<10	<10
Chlorobenzene	<10	<10	Acetonitrile	<100	<100
Cyclohexanone	<10	<10	Dichloromethane	<10	<10
n-Pentane	<10	<10	Carbon Disulfide	<10	<10
^{a,b} Tetrahydrofuran	^B (19)	^B (18)	Oxirane	<10	<10
5-Methyl-2-Hexanone	<10	<10	1,1-Dichloroethane	<10	<10
2-Heptanone	<10	<10	1,1-Dichloroethene	<10	<10
n-Hexane	<10	<10	Dichlorofluoromethane	<10	<10
Cyclohexane	<10	<10	^a Chlorodifluoromethane	(3.7)	(3.7)
Cyclohexene	<10	<10	2-Methyl-2-Propanol	<100	(8.1)
n-Octane	<10	<10	Trichlorofluoromethane	<10	<10
n-Nonane	<10	<10	Dichlorodifluoromethane	<10	<10
4-Heptanone	<10	<10	1,2,2-Trichloro-1,1,2-Trifluoroethane	<10	<10
n-Propionaldehyde	<10	<10	1,2,-Dichloro-1,1,2,2-Tetrafluoroethane	<10	<10
Acetic Acid n-Butyl Ester	<100	<100	1,2-Dichloropropane	<10	<10
1,4-Dioxane	<100	<100	*1-Methylpropyl Alcohol	TIC	TIC
2-Methyl-2-Propenenitrile	<10	<10	2-Butanone	<10	<10
Tetrachloroethene	<10	<10	1,1,2-Trichloroethane	<10	<10
Acetic Acid Ethyl Ester	<100	<100	1,1,2-Trichloroethylene	<10	<10
n-Heptane	<10	<10	1,1,2,2-Tetrachloroethane	<10	<10
*Cyclopentane	TIC	TIC	o-Xylene	<10	<10
trans-1,2-Dichloroethene	<10	<10			

^aThese analytes were detected in the field blanks at similar concentrations.

^bThese analytes were detected in the trip blanks at similar concentrations.

<These analyte concentrations were less than the MDL (commonly designated with a "U" data qualifier).

() These analytes were detected but at less than the EQL. This is commonly designated with a "J" qualifier.

^BThis analyte was detected in the laboratory method blank at a concentration of 18 µg/kg.

* These analytes could only be monitored for as TICs.

Table V.8. Results ($\mu\text{g/kg}$) of Volatile Organic Compounds Determined by IC

Analyte	Samples		Field Blank	
Formate	$<6 \times 10^3$	$<6 \times 10^3$	$<6 \times 10^3$	$<6 \times 10^3$
Acetate	$<9 \times 10^2$	$<9 \times 10^2$	$<9 \times 10^2$	$<9 \times 10^2$
Oxalate	$<7 \times 10^2$	$<7 \times 10^2$	$<7 \times 10^2$	$<7 \times 10^2$

<These analyte concentrations were less than the MDL (commonly designated with a “U” data qualifier).

Table V.9. Detection and Quantitation Limits for Volatile Organic Compound IC Analyses

Analyte	Dilution (L/kg)	Instrument Limits ($\mu\text{g/L}$)		Method Limits ($\mu\text{g/L}$)	
		Detection	Quantitation	Detection	Quantitation
Formate	25	2.5×10^2	2.5×10^3	6.25×10^3	6.25×10^4
Acetate	25	37.5	37.5×10^2	9.4×10^2	9.4×10^3
Oxalate	25	28	2.8×10^2	7.0×10^2	7.0×10^3

Detection limits for the samples have been given in Table V.10. With the exception of 2-propenoic acid and oxalic acid, EQLs for these analyses were $4 \times 10^3 \mu\text{g/kg}$ for one sample, and $1 \times 10^3 \mu\text{g/kg}$ for the other sample and the field blank. Oxalic acid limits have already been given. The 2-propenoic acid EQL was $1.2 \times 10^4 \mu\text{g/kg}$. EQLs were calculated from minimum ICLs and overall dilution factors. Dilution factors were the product of the Method 5035 dilution factors and the ratio of the injection size (1 μL) to the final extraction volume (1 mL).

3. Pesticides

As expected, no pesticides were detected in the samples. Results have been given in Table V.11. Matrix spike and duplicate recoveries were between 101 and 131 %, and relative deviations were between 1 and 6 %. These recoveries were easily within the specifications given in the QAPjP² and task plan³ (between 20 and 130 % with relative deviations of less than 20 %).

The minimum method calibration levels (MCLs) were used as the EQLs for the pesticide analyses. The sample EQLs have been given in Table V.11. These values were calculated from the instrument calibration levels and overall dilution factors. Dilution factors were the product of the Method 5035 dilution factors and the ratio of the injection size (1 μL) to the final extraction volume (10 mL).

Table V.10. Semivolatile Organic Compounds that Were below the Method Detection Limits

Analyte	CAS Number	Samples (µg/kg)		Field Blank (µg/kg)
p-Nitrochlorobenzene	100-00-5	<2x10 ³	<5x10 ²	<5x10 ²
1,4-Dinitrobenzene	100-25-4	<2x10 ³	<5x10 ²	<5x10 ²
Benzyl Alcohol	100-51-6	<2x10 ³	<5x10 ²	<5x10 ²
4-Methylphenol	106-44-5	<2x10 ³	<5x10 ²	<5x10 ²
1,4-Dichlorobenzene	106-46-7	<2x10 ³	<5x10 ²	<5x10 ²
Phenol	108-95-2	<2x10 ³	<5x10 ²	<5x10 ²
Pyridine	110-86-1	<2x10 ³	<5x10 ²	<5x10 ²
Di-sec-Octyl Phthalate	117-81-7	<2x10 ³	<5x10 ²	<5x10 ²
n-Dioctyl Phthalate	117-84-0	<2x10 ³	<5x10 ²	<5x10 ²
1,2,4-Trichlorobenzene	120-82-1	<2x10 ³	<5x10 ²	<5x10 ²
2,4-Dinitrotoluene	121-14-2	<2x10 ³	<5x10 ²	<5x10 ²
Triethylamine	121-44-8	<2x10 ³	<5x10 ²	<5x10 ²
N,N-Diphenylamine	122-39-4	<2x10 ³	<5x10 ²	<5x10 ²
Tributyl Phosphate	126-73-8	<2x10 ³	<5x10 ²	<5x10 ²
2,6-Bis(tert-butyl)-4-Methylphenol	128-37-0	<2x10 ³	<5x10 ²	<5x10 ²
Pentachloronaphthalene*	1321-64-8	-	-	-
Hexachloronaphthalene*	1335-87-1	-	-	-
Tetrachloronaphthalene	1335-88-2	<2x10 ³	<5x10 ²	<5x10 ²
Oxalic Acid	144-62-7	<7x10 ²	<7x10 ²	<7x10 ²
Benzo(a)pyrene	50-32-8	<2x10 ³	<5x10 ²	<5x10 ²
Dibenz[a,h]anthracene	53-70-3	<2x10 ³	<5x10 ²	<5x10 ²
1,3-Dichlorobenzene	541-73-1	<2x10 ³	<5x10 ²	<5x10 ²
1,1-Dimethylhydrazine*	57-14-7	-	-	-
Methylhydrazine*	60-34-4	-	-	-
N-Nitroso-N,N-Dimethylamine	62-75-9	<2x10 ³	<5x10 ²	<5x10 ²
Nitric Acid, Propyl Ester	627-13-4	<2x10 ³	<5x10 ²	<5x10 ²
Hexachloroethane	67-72-1	<2x10 ³	<5x10 ²	<5x10 ²
2-Propenoic Acid	79-10-7	<6x10 ⁴	<2x10 ⁴	<2x10 ⁴
Pentachloronitrobenzene (PCNB)	82-68-8	<2x10 ³	<5x10 ²	<5x10 ²
Hexachlorobutadiene	87-68-3	<2x10 ³	<5x10 ²	<5x10 ²
Pentachlorophenol	87-86-5	<2x10 ³	<5x10 ²	<5x10 ²
2-sec-Butyl-4,6-Dinitrophenol	88-85-7	<2x10 ³	<5x10 ²	<5x10 ²
Naphthalene	91-20-3	<2x10 ³	<5x10 ²	<5x10 ²
1,1-Biphenyl	92-52-4	<2x10 ³	<5x10 ²	<5x10 ²
2-Methylphenol	95-48-7	<2x10 ³	<5x10 ²	<5x10 ²
1,2-Dichlorobenzene	95-50-1	<2x10 ³	<5x10 ²	<5x10 ²
Acetophenone	98-86-2	<2x10 ³	<5x10 ²	<5x10 ²
Nitrobenzene	98-95-3	<2x10 ³	<5x10 ²	<5x10 ²

<These analyte concentrations were less than the MDL (commonly designated with a "U" data qualifier).

*This analytes was monitored for as a TIC. No detection limit should be associated with this result.

Table V.11. Results (µg/kg) of Pesticide Analyses

Samples	<16 µg/kg	Samples	<8 µg/kg	Samples	<8x10 ² µg/kg
Field Blank	<5 µg/kg	Field Blank	<3 µg/kg	Field Blank	<3x10 ² µg/kg
Hexachlorobenzene		Aldrin		Toxaphene	
Octachloronaphthalene		alpha-BHC			
Isodrin		beta-BHC			
Dichlorodiphenyl Trichloroethane		gamma-BHC (Lindane)			
Dieldrin		Heptachlor			
Endrin					
1,1-Bis(4-Chlorophenyl)-2,2 Dichloroethane					

<These analyte concentrations were less than the MDL (commonly designated with a “U” data qualifier).

4. Polychlorinated Biphenyls (PCBs)

As expected, no PCBs were detected in the samples. Table V.12 is a list of the analytes that were shown by Method 8082 not to be present above the method detection limits. Matrix spike and matrix spike duplicate recoveries were between 55 and 84 %, and relative deviations were both 5 %. These recoveries were easily within the specifications given in the QAPjP² and task plan³ (between 20 and 130 % with relative deviations of less than 20 %).

Table V.12. Polychlorinated Biphenyls (PCBs) which Were below the Method Detection Limits

Analyte	CAS No.	Samples (µg/kg)	Field Blank (µg/kg)
Aroclor-1016	12674-11-2	<2x10 ²	<50
Aroclor-1221	11104-28-2	<3x10 ²	<1x10 ²
Aroclor-1232	11141-16-5	<2x10 ²	<50
Aroclor-1242	53469-21-9	<2x10 ²	<50
Aroclor-1248	12672-29-6	<2x10 ²	<50
Aroclor-1254	11097-69-5	<2x10 ²	<50
Aroclor-1260	11096-82-5	<2x10 ²	<50
Aroclor-1262	37327-23-5	<2x10 ²	<50
Aroclor-1268	11100-14-4	<2x10 ²	<50

<These analyte concentrations were less than the MDL (commonly designated with a “U” data qualifier).

The minimum method calibration levels (MCLs) were used as the EQLs for the PCB analyses. The sample EQLs have been given in Table V.8. These values were calculated from the instrument calibration levels and overall dilution factors. Dilution factors were the product of the Method 5035 dilution factors and the ratio of the injection size (1 µL) to the final extraction volume (10 mL).

D. Radionuclides

Radionuclide concentrations have been presented in Table V.13. Results are similar to those determined at SRTC.⁴ Although carbon-14 was reported in the sample, other beta-emitting radionuclides can interfere with this measurement. The method used to determine carbon-14 was one in which the sample was oxidized and the resulting gas trapped and counted. Some radionuclides (e.g. cesium isotopes) can interfere with this process either because of volatility or as a result of entrainment. The carbon-14 results given in Table V.9 are believed to be due to such interferences.

In addition, uranium-234 was reported at similar concentrations for the samples and one of the field blanks. In addition, the uranium-234 results were all within a factor of 2 from the minimum detection limits. Detection of uranium-234 in the field blank at concentrations similar to the sample would suggest the source to be from contamination during sampling, transportation, preparation or analysis or from interference during analysis. For these reasons, the uranium-234 results should be considered as “nondetects”.

Cobalt-60, cesium-134, cesium-137, plutonium-239/240, and strontium-90 were reported at slightly above the minimum detection limits in one or both field blanks. Cobalt-60, cesium-134, cesium-137, and plutonium-239/240 results in the field blank analyses were within a factor of two of the minimum detection limits. Strontium-90 results were within a factor of eight. With the exception of cesium-134, these analytes were reported at concentrations of at least two orders of magnitude higher in the samples. Cesium-134 was not detected in the samples.

E. Density

Results from density measurements were 3.15 and 2.59 g/cc. The density results were close to the expected values (~2.7 g/cc) although better precision was expected. For the density measurements, the “field blank” acted more like a standard than a blank. The density of the “field blank” was found to be 2.55 g/cc. The density of this was known to be 2.52 g/cc.⁹

6.0 Discussion

This chapter is a discussion of the results that have been presented in the previous chapter. The purpose of this chapter is to explain the basis for the conclusions, which are given in the following chapter. Except where noted, results met the requirements of the QAPjP² as they were implemented by the project task plan³ and were in compliance with USEPA SW-846 protocol.

Table V.13. Results (pCi/g) of Radionuclide Analyses

	Samples				Field Blanks	
	Acid		Fusion		Acid	Fusion
Am-241	1.1x10 ⁴	8.9x10 ³	1.1x10 ⁴	1.0x10 ⁴	<30	<70
Cs-134	<2x10 ²	<1x10 ²	<2x10 ²	<1x10 ²	<31	^U 1.1x10 ²
Cs-137	3.2x10 ⁴	3.2x10 ⁴	3.0x10 ⁴	3.4x10 ⁴	<32	67
Co-60	2.7x10 ⁴	2.7x10 ⁴	2.6x10 ⁴	2.9x10 ⁴	<32	^U 80
Nb-94	<2x10 ²	<2x10 ²	<2x10 ²	<1x10 ²	<27	<27
Eu-152	^U 4.9x10 ²	4.5x10 ²	<7x10 ²	5.0x10 ²	<91	<2x10 ²
Eu-154	2.8x10 ⁴	2.7x10 ⁴	2.7x10 ⁴	2.8x10 ⁴	<94	<2x10 ²
Eu-155	1.6x10 ⁴	1.6x10 ⁴	1.5x10 ⁴	1.8x10 ⁴	<87	<2x10 ²
Ru-103	<2x10 ²	<1x10 ²	<3x10 ²	<1x10 ²	<29	<62
Ru-106 (Rh-106)	<2x10 ³	<1x10 ³	<2x10 ³	<1x10 ³	<3x10 ²	<5x10 ²
Ra-226	<3x10 ²	<2x10 ²	^U 6.2x10 ²	<3x10 ²	^U 95	^U 1.7x10 ²
Total Alpha	2.1x10 ⁴	1.8x10 ⁴	2.9x10 ⁴	3.2x10 ⁴	<1x10 ²	<2x10 ²
Total Beta	2.0x10 ⁶	1.9x10 ⁶	1.8x10 ⁶	2.0x10 ⁶	<1x10 ²	1.2x10 ³
Sr-90 (Y-90)	1.1x10 ⁶	1.0x10 ⁶	8.2x10 ⁵	1.1x10 ⁶	<3x10 ²	4.7x10 ³
Tc-99	1.6x10 ⁴	1.6x10 ⁴	1.7x10 ⁴	1.7x10 ⁴	<2x10 ²	<4x10 ²
Np-237	<12	<19	<7	<10	<15	<14
Pu-238	5.1x10 ²	4.7x10 ²	5.0x10 ²	5.6x10 ²	<51	<45
Pu-239/240	6.2x10 ²	6.5x10 ²	6.0x10 ²	6.5x10 ²	<22	29
Pu-241	2.4x10 ³	<2x10 ³	<2x10 ³	<2x10 ³	<3x10 ³	<3x10 ³
Cm-244	8.0x10 ³	7.3x10 ³	9.0x10 ³	8.0x10 ³	<27	<27
Cm-242	62	<42	59	35	<29	<28
Nb-94	<2x10 ²	<1x10 ²	<2x10 ²	<2x10 ²	<27	<57
*Se-79	^J <1x10 ⁴	^J <9x10 ³	^J <2x10 ⁴	^J <1x10 ⁴	^J <7x10 ³	^J <2x10 ⁴
H-3	<2x10 ³	<2x10 ³	<2x10 ³	<2x10 ³	<2x10 ³	<5x10 ³
I-129	<8x10 ²	<4x10 ²	<2x10 ²	<8x10 ²	<2x10 ²	<6x10 ²
U-234	17	^U 14	15	18	^U 12	<27
U-235	<7	<10	<8	<7	<7	<19
U-236	<7	<11	<8	<7	<7	<22
U-238	11	<6	<8	<6	<4	<21
**C-14	^U 8.8		^U 9.8		<8	

*Carrier recovery for this analytes was not measured. SRTC and BWXT personnel have indicated that recoveries associated with this separation method can be less than 10 % which is well below the acceptable recovery range of 70 to 125% given in Reference 2.

**Carbon-14 was measured in the solid glass waste form to avoid potential losses during dissolution processes.

<These analyte concentrations were less than the minimum detectable activity (MDA). This is commonly designated with a "U" data qualifier.

^JThese analytes were regarded as not detected, but the reported detection limit was uncertain.

^UThese analytes were regarded as not detected. The detection limit should be considered the value given.

A. Analysis to Support Delisting Petition

The purpose of these analyses was to demonstrate that the waste form did not exhibit the characteristics of Hazardous Waste.⁷ The analyses were successful in doing this by showing

- The samples did not contain concentrations of cyanide above land disposal restriction limits.
- The samples did not exhibit the characteristic of ignitability.
- The samples did not contain detectable amounts of dioxins or furans.
- RCRA metal concentrations in TCLP leachates were below UTS limits.

Cyanide was not detected in the samples or blanks. Although limited by the amount of samples available, detection limits were low enough to show that the cyanide level in these samples were more than two orders of magnitude below the land disposal restriction limits (590 mg/kg).⁷ Matrix spike and duplicate recoveries were between 82 and 106 %. The relative deviation was 26 %. No specification was given for cyanide recoveries in the QAPjP.²

As expected, the samples showed no signs of being flammable. As samples were heated to above 150 °F, the presence of a flame did not ignite the samples. This ignitability test was performed by applying USEPA SW-846 Method 1010 to the samples. Although written for liquids, the method was modified to make it applicable to the solid samples. Method 1030 could not be performed on the samples because sufficient sample was not available. Application of Method 1010 in this manner is similar to performing the preliminary test required by Method 1030.

All dioxin and furan results should be considered “nondetects”. As shown in Table V.1, all concentrations except 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin (OCDD) were shown to be below the method detection limits. Although OCDD was reported in both samples, it was also reported to be present in the field blank, and the analytical method blanks at similar concentrations. Application of the CLP 5x rule would dictate that the OCDD result be considered a “nondetect”.

As shown in Table V.3, RCRA metal concentrations in TCLP leachates were well below the UTS limits. Although barium contamination was apparent in the field blanks (Table IV.3) and extraction blank (1.2×10^3 µg/L), barium results were still less than one-seventh the UTS limit. Matrix spike and duplicate recoveries were within the required range² (75 to 125 %) except for mercury. Acid sample results (Table V.1) showed the mercury concentration in the glass to be between 0.43 and 0.45 mg/kg (or µg/g). Even complete release of mercury would give 22 µg/L ($0.44 \text{ µg/g} \times 0.5 \text{ g} / 0.1 \text{ L}$) in the TCLP leachate, still below the UTS limit.

B. Analysis to Support Characterization of Tank Waste Samples

Results in Table V.5 indicated the waste form composition was within 10 % of the target for all major components except sulfur. Matrix spike and duplicate recoveries were within the specified range² (75 to 125 %) except: Acid sample recoveries were low in lithium, palladium, and zirconium matrix spikes; and palladium and zirconium duplicates. Fusion recoveries were low in

bismuth, palladium, tantalum, and zirconium matrix spike and duplicates and uranium matrix spike. Chloride and fluoride could not be determined because of a matrix interference. Chloride and fluoride could not be determined in the acid samples because hydrochloric acid was used in the dissolution. With pretreatment, Method 9056 can be used to measure chloride and fluoride in the peroxide fusion samples; however, without pretreatment the matrix is known to interfere with determinations of these ions.

C. Analysis to Support Permitting

No organic analytes could be considered to have been detected in the samples. The results can be organized into three groups. The first was target analytes shown to be below the method detection limit. The second was the target analytes monitored for as TICs. The third was analytes detected in the samples, but also detected at similar concentrations in the field blanks. These analytes were determined to be “nondetects” as a result of the CLP 5x rule.

During the pesticide determinations (Method 8081) a continuing calibration check showed the octachloronaphthalene instrument response to have increased by more than the specified 15 %. Since this analyte was not detected in any sample and since the instrument had drifted to a more sensitive condition, the results were valid with no further actions according to SW-846 protocol.

D. Radionuclides

Several radionuclides were detected. Most of the four replicate results were within ten percent. The most notable exceptions were total alpha and curium-244. The beta activity was primarily strontium-90 and yttrium-90. The alpha activity was primarily americium-241 and curium-244. Total alpha results showed the waste form would not need to be handled as transuranic waste.

As indicated previously, results given in Table V.13 were similar to SRTC results. Carbon-14 results were believed to have been due to interference from strontium-90 and yttrium-90 and not to the presence of carbon-14. Uranium-234 results should also be used with caution because this radionuclide was reported in one of the field blanks at levels similar to those of the sample. Selenium-79 recoveries could not be measured because a selenium-79 standard was not available. Studies in which nonradioactive selenium was separated from similar matrices (and analyzed by ICP-ES) showed recoveries to be low (often less than 10 %).

E. Density

As indicated previously, the average measured density was similar to the expected value. Poor precision may have been associated with sample collection. To determine the bulk density of the samples, size reduction was kept to a minimum. If small bubbles or void spaces were present in one of the samples, the density determinations could have been biased low. Size reduction could have caused the densities to be biased high; however, the presence of void spaces and the effect of size reduction together is expected to have been responsible for less than a 10 % variation in the density measurements.

7.0 Conclusions

Analysis of the AN-102 glass waste form samples showed the actual composition to be very close to the target composition. The primary conclusions from these analyses were

- The waste form samples did not exhibit any characteristics of Hazardous Waste.
- Major component concentrations were within 10 % of the target values except for sulfur.
- No organic analytes were detected in the samples.
- Total beta activity in the sample was 1.9×10^6 pCi/g and total alpha activity was 2.1×10^4 pCi/g.
- Duplicate density measurements indicated the density to be 2.9 g/cc.

These analyses showed the samples did not exhibit characteristics of Hazardous Waste. Cyanide concentrations were below the land disposal limits given in 40CFR 268 and did not contain measurable concentrations of dioxins or furans. In addition, the samples were not ignitable. Results also showed the samples met UTS requirements for RCRA metals.

Major component concentrations were shown to be within 10 % of the target glass composition (See target in Table V.5) except for sulfur. Measured sulfur concentrations were approximately 30 % higher than expected. Since the sulfur value used in the target was from measurement of sulfate, this deviation may have been caused by the presence of sulfur in forms other forms (e.g. as sulfide). Fluorine and chlorine could not be determined using the standard USEPA ion chromatography method. A modified method has been developed and can be used in future sample analyses.

No organic compounds were detected in the samples. Results were reported for concentrations of four target volatile compounds, one of the dioxins, and one nontarget semivolatile compound (reported as a tentatively-identified compound); however, these compounds were also reported in the blanks at levels similar to the concentrations reported for the samples. Application of the CLP 5x rule dictated that these result be considered “nondetects”.

Several radionuclides were detected in the samples. These results were similar to the results from SRTC analyses of the samples. The primary beta-emitting radionuclides were strontium-90 and its daughter radionuclide yttrium-90. The primary alpha-emitting radionuclides were americium-241 and curium-244. Gross alpha levels were low enough to show the waste form would not need to be handled as transuranic waste.

The average density of the waste form was 2.87 ± 0.40 g/cc. Precision for these duplicate density determinations was poor. The relative deviation was 10 %. Future density measurements will be performed on at least four samples. This will allow us to identify outlying results.

Most of the sample collection, sample preparation, and sample analyses provided results with sufficient pedigree to support the rigor associated with regulatory application of these results. In future studies, some alternative approaches should be taken to increase the number of target analytes that can be determined to this rigor. Five changes should be made to the approaches that were taken in this study

-Carbon-14 analyses were subject to strong interference from other beta-emitting radionuclides. BWXT is currently implementing an alternative method for carbon-14 determination.

-In both the acid-dissolved glass samples and the fusion-dissolved glass samples, either the matrix spike recovery, the matrix spike duplicate recovery or both were outside of the specified range for silver, palladium, selenium, and zirconium. To determine these metals, modifications may be necessary in the glass-dissolution methods.

-Barium was reported at relatively high concentrations in the TCLP blanks. Although the waste form could still be shown to meet the UTS for barium, the source of the barium contamination needs to be identified, and steps need to be taken to minimize future contaminations

-Thallium detection limits were too high to show the waste form would have met the UTS for thallium. The thallium detection limit for the TCLP leachate can be improved by reducing leachate dilution prior to analysis. This would probably require the TCLP to be performed with larger samples.

-In the dissolved glass samples, chloride and fluoride determinations were not possible because of strong interferences and baseline shifts. In future samples, a pretreatment step will be introduced in which chloride and fluoride are separated from interferences in the peroxide fusion samples. This procedure has been implemented successfully at SRTC.

8.0 References

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