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ANALYSIS OF A HANFORD TANK 241-AZ-102 GLASS

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LIST OF ACRONYMS

AAS	Atomic Absorption Spectroscopy
ICP-AES	Inductively-Coupled Plasma-Atomic Emission Spectroscopy
ASTM	American Society for Testing and Materials
BNI	Bechtel National, Inc
BWXS	BWXT Services, Inc., a division of BWX Technology, Inc.
CAS	Chemical Abstract Service
CLP	Contract Laboratory Program
EQL	Estimated Quantitation Limit
LCS	Laboratory Control Sample
LRM	Low-Activity Reference Material
MDL	Minimum Detection Limit
MRQ	Minimum Reportable Quantity
MS	Matrix Spike
MSD	Matrix Spike Duplicate
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
RPD	Relative Percent Difference
RPP	River Protection Project
SRS	Savannah River Site
SRTC	Savannah River Technology Center
TCLP	Toxicity Characteristic Leaching Procedure
USEPA	United States Environmental Protection Agency
UTS	Universal Treatment Standard
WTP	Hanford Tank Waste Treatment and Immobilization Plant

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ABSTRACT

A proof-of-technology demonstration for the River Protection Project (RPP) Hanford Tank Waste Treatment and Immobilization Plant (WTP) was performed by SRS. As part of this demonstration, samples from a low-activity AZ-102 glass waste form were characterized. The sample handling, preparation, and analyses were performed according to standard United States of America Environmental Protection Agency (USEPA) protocol to facilitate use of these results for regulatory applications.

Toxicity Characteristic Leaching Procedure (TCLP) tests and cyanide analyses were performed on samples of the AZ-102 glass waste form. Glass samples were also dissolved using an acid dissolution method and using a peroxide fusion dissolution method with an acid strike. The resulting solutions were analyzed to determine the concentration of metals in the glass. Samples were also dissolved using a peroxide fusion dissolution without the acid strike. Samples from these peroxide fusion dissolutions were used to determine the anion concentrations in the glass.

At least one set of results for each metal analyte met all quality assurance (QA) specifications, except for silicon. Silicon results met all QA specifications except two recoveries in the acid dissolved samples that were caused by use of a low spike concentration. For all metal analytes detected above estimated quantitation limits (EQLs), results from acid dissolutions were within three standard deviations (as indicated in the test specification) of the results from the peroxide fusions, except for the arsenic results, which were only slightly above the EQL.

The sum of oxides indicated all major constituents (those at or above 0.5 weight percent) were determined and reported. Results were within ten percent of the target for all but four analytes present in the glass at concentrations above the EQL. Iron and zirconium values were high for one of four results, chromium results were consistently higher than the target, and sodium was low by ten percent. Because chromium was similarly high in a simulant that was prepared prior to the AZ-102 glass, the source of the high-chromium concentration appears to have been in the vitrification process. The sodium concentrations were low because of uncertainty associated with the measured feed stream sodium concentrations that were provided to Catholic University to develop the glass formulation.

Resource Conservation and Recovery Act (RCRA) metal concentrations in the TCLP leachates were below characteristically hazardous limits. Concentrations were also shown to be below the Universal Treatment Standard (UTS) limits except for thallium. Although no thallium was detected in the leachate, Method Detection Limits (MDLs) were higher than the UTS limit. Thallium concentrations measured in one set of glass samples were low enough to show the waste form met UTS limits; however, recoveries were outside acceptable ranges.

Anion concentrations in the dissolved AZ-102 waste form were consistent with the target glass composition. Total cyanide was determined to be below the regulatory limits for both amenable and total cyanide. Sulfate was the only other anion detected in the dissolved glass. Once converted to a glass oxide (SO_3), the sulfur concentration was within three standard deviations of the target composition and within three standard deviations of the sulfur concentrations determined in the samples dissolved for metals analysis.

1.0 SUMMARY OF TESTING

The task addressed in this report was part of a proof-of-technology demonstration performed by the Savannah River Technology Center (SRTC) for Bechtel National, Inc. (BNI). In this demonstration, a sample of AZ-102 high-level-radioactive waste was treated to remove suspended solids and most radionuclides. The resulting low-activity-waste supernate was concentrated, mixed with glass-forming minerals, and vitrified.[1]

The initial glass product contained crystals on one of the surfaces and was not analyzed. The presence of crystals indicated that the glass waste form needed to be reformulated to meet homogeneity requirements of the Waste Treatment and Immobilization Plant (WTP). The AZ-102 glass was reformulated, and a second batch of AZ-102 LAW glass was produced and rapidly cooled. The scope of the task described in the following report was to provide the reformulated, rapidly cooled glass waste form characterization data that would be used for regulatory applications.

1.1 OBJECTIVES

The goals of this task were to measure the concentration of hazardous components in the glass and TCLP leachates to determine whether the AZ-102 glass waste form would be considered hazardous. In addition, this task was to measure the concentration of all primary constituents (those present at concentrations greater than 0.5 weight percent) in the glass. This task was successful at meeting these objectives, except for silicon and thallium.

1.2 CONDUCT OF TESTING

Testing associated with the task described in this report included the preparation, and analysis of the AZ-102 glass waste form samples. Vitrification of the waste form has been addressed in a separate task.[1] During the vitrification task, an AZ-102 low-activity glass waste form and a nonradioactive AZ-102 simulant glass waste form were produced. The scope associated with the following report included sampling, crushing, and dissolving of these glasses at SRTC and analysis at BWXT Services, Inc. (BWXS), a division of BWX Technology, Inc. (BWXT). A list of the sample preparation and analysis methods has been given in Table 1.

Sample preparation at SRTC generated two sample sets. The first was solid samples that had been crushed to particles with diameters of less than 0.9 centimeter. BWXS used these samples to measure for cyanide in the glass using USEPA SW-846[2] Methods 9010B and 9014. The samples were also used to perform TCLP tests (Method 1311), followed by Method 3015 digestions and analysis by Methods 6010B, 7060A, 7740, and 7470.

Table 1. Methods Used to Dissolve, Prepare, and Analyze AZ-102 Glass Samples

Analyte	Dissolution or Leach	Preparation	Analysis
Miscellaneous Metals	Acid Dissolution	None	ICP-AES (6010B)
Mercury	Acid Dissolution	AAS (7470A)	AAS (7470A)
Silver	Acid Dissolution	None	AAS (7761)
Selenium	Acid Dissolution	None	AAS (7740)
Miscellaneous Metals	Peroxide Fusion	Acid Digestion (3015)	ICP-AES (6010B)
Arsenic	Peroxide Fusion	Acid Digestion (3015)	AAS (7060A)
Lead	Peroxide Fusion	Acid Digestion (3015)	AAS (7421)
Mercury	Peroxide Fusion	AAS (7470A)	AAS (7470A)
Silver	Peroxide Fusion	Acid Digestion (3015)	AAS (7761)
Selenium	Peroxide Fusion	Acid Digestion (3015)	AAS (7740)
Thallium	Peroxide Fusion	Acid Digestion (3015)	AAS (7841)
Anions	Peroxide Fusion	None	IC (9056)
Miscellaneous Metals	TCLP (1311)	Acid Digestion (3015)	ICP-AES (6010B)
Arsenic	TCLP (1311)	Acid Digestion (3015)	AAS (7060A)
Selenium	TCLP (1311)	Acid Digestion (3015)	AAS (7740)
Mercury	TCLP (1311)	AAS (7470A)	AAS (7470A)
Cyanide	None	Distillation (9010B)	Photometry (9014)

() Values in parentheses indicate USEPA SW-846 method numbers.

The second set of samples was generated by dissolving the glass samples. Three American Society for Testing and Materials (ASTM) C1463-00 dissolution methods were used.[3] The first was the acid dissolution method. The second was the peroxide fusion method. These samples were analyzed by BWXS using Methods 6010B, 7060A, 7421, 7740, 7761, and 7841. Prior to analyses, peroxide fusion samples were digested by Method 3015. The third set of samples was from a peroxide fusion without the final acid addition. These samples were analyzed using Method 9056 for anions.

1.3 RESULTS AND PERFORMANCE AGAINST OBJECTIVES

Results from these analyses met the task objectives by demonstrating the following:

- Measured concentrations were within 10 percent of expected values for most analytes.
- All primary oxides (present at 0.5 weight percent or more) were accounted for in the glass.
- Glass passed TCLP characteristic and UTS limits for metals with sufficiently low MDLs.
- No cyanide was detected in the glass; concentrations were well below the regulatory limits.
- Anion concentrations were similar to expected values and consistent with metal analyses.

Results from the metal analyses of the acid-dissolved sample were within three standard deviations of results from the peroxide fusion dissolved waste form for all analytes present above the EQL, except arsenic. Although arsenic values were significantly different for the two sets of samples, the measured concentrations were low (124 and 270 mg/kg for the acid and peroxide fusion samples, respectively).

Oxide concentrations were added first using only results for species present at concentrations high enough to be determined quantitatively, and then using values for all species present at concentrations high enough to be detected. For the acid-dissolved samples, the totals were 102.3 and 103.0 weight percent. For the fusion-dissolved samples the totals were 101.0 and 101.8 weight percent. These results suggest that all primary components in the glass were determined.

TCLP leachates were shown to be below characteristic limits for all metals. TCLP leachates were also shown to be below UTS limits for all RCRA metals except thallium. Thallium was shown to be less than 0.5 μg/mL in the leachate; however, the UTS limit is 0.2 μg/mL. Although the result from the peroxide-fusion-sample analysis was low enough to show the maximum TCLP leachate concentration would be 0.15 μg/mL, the matrix spike, duplicate, and post spike recoveries (63.2, 74.5, and 71.7 percent respectively) were below 75 percent.

As expected, the only anion detected in the samples was sulfate. Cyanide was not detected, and the MDL was 0.005 mg/kg. This was well below the limit for total and for amenable cyanide (30 mg/kg). Cyanide analyses met all QA specifications. Anion results, except cyanide, have been given a data flag indicating that samples were analyzed outside of allowable hold times.

1.4 QUALITY REQUIREMENTS

This work was conducted in accordance with the RPP-WTP QA requirements specified for work conducted by SRTC as identified in DOE IWO M0SRLE60. SRTC has provided matrices to WTP demonstrating compliance of the SRTC QA program with the requirements specified by WTP. Specific information regarding the compliance of the SRTC QA program with NQA-1 1989, Part 1, Basic and Supplementary Requirements and NQA-2a 1990, Part 2.7 is contained in these matrices. Because results from this task may be used for regulatory applications, the work was also performed to specifications given in the QAPjP (PL-24590-QA00001) and USEPA SW-846.

1.5 ISSUES

Two issues were encountered that could be applicable to operation of the RPP-WTP. First, although thallium can be determined to UTS limits, QC failures may be encountered at these low levels. Thallium determination was challenging in these samples because of limitations in sensitivity of the ICP-AES method to thallium, high dilutions (500 mL/g) associated with dissolving glass samples, and the complex matrix of the resulting dissolved glass samples. In addition, an argument could be made that unless analyses are performed with less than a 0.5 percent error, we cannot prove all oxides at more than 0.5 weight percent have been determined. Weight percent oxides summed to greater than 101 percent; however, uncertainties could just as easily have caused the total oxide to be one or two percent less than 100 percent.

2.0 DISCUSSION

The purpose of this task was to perform regulatory analysis on samples from a Hanford tank waste Envelope B (AZ-102) demonstration being performed at SRTC. Standard methods were used to prepare and analyze samples as specified in the test specification[4] and task plan[5] for this task. This task was performed to the QA requirements outlined in the quality assurance project plan (QAPjP).[6] Results were validated according to USEPA guidance.[7] To facilitate result discussions, each section has been organized with sample and QA results subsections followed by a narrative subsection.

2.1 AZ-102 GLASS SAMPLE COLLECTION, PREPARATION, AND ANALYSIS METHODS

Vitrification of the pretreated AZ-102 supernate was performed as a separate task[1] and results will be reported upon completion of this task. Once vitrified, the resulting glass waste form was sampled, size reduced, prepared, and analyzed. During this process, samples were collected for regulatory and for process control analyses. The purpose of this report is to present results from the regulatory analyses. Process control results are being reported along with the vitrification details.[1]

2.1.1 Sample Collection and Initial Size Reduction

Samples were taken of the AZ-102 glass, an AZ-102 glass simulant, and a standard glass (Low-Activity Reference Material (LRM) glass). The AZ-102 glass simulant was prepared from an AZ-102 simulated supernate using the same glass forming minerals, the same heating profile, and the same cooling profile as the AZ-102 glass. The purpose of the simulant was to act as a control for identification of any unexpected events or observations. This material has been referred to as a process blank in previous studies, although in most aspects it was more of a standard than a blank.

Initial size reduction was performed using an agate mortar and pestle to avoid trace metal contaminants that would have been introduced using a steel grinder. The mortar and pestle was used to break the glass monolith into particles small enough for use in the appropriate USEPA SW-846 methods.[2] The glass was crushed into particles with diameters of less than 0.9 centimeter as required by TCLP, Method 1311.

Upon completion of the initial size reduction, four sample sets had been generated.

- AZ-102 glass, nonradioactive AZ-102 simulant glass, and LRM glass for TCLP analyses
- AZ-102 glass, nonradioactive AZ-102 simulant glass, and LRM glass for cyanide analyses
- AZ-102 glass, nonradioactive AZ-102 simulant glass, and LRM standard to be dissolved
- AZ-102 glass archived for possible future analyses (biological toxicity, organic analytes)

2.1.2 Solid Sample Preparation and Analysis (TCLP and Cyanide Determination)

Samples of the crushed glass were collected and shipped to BWXS for TCLP and cyanide determinations. Samples were collected in I-Chem 250-milliliter amber glass bottles. Bottles were double-bagged and packed in two coolers with ice. Each cooler was shipped in a ten-gallon drum according to 49 CFR 171.11 and 49 CFR 173.421. Upon receipt, the temperature of the TCLP samples was determined to be 1.0 ^{o}C, and the temperature of the cyanide samples was determined to be 1.5 ^{o}C.

Four 20-gram samples were shipped for TCLP. These were an AZ-102 glass sample, an AZ-102 simulant glass sample, an LRM glass sample, and a TCLP soil standard. Five grams of each was used to determine the appropriate extraction fluid (Method 1311, Section 7.1.4).[2] Section 7.2 was then performed on two 5-gram aliquots of each. The resulting leachates were digested by Method 3015A and analyzed by Methods 6010B, 7060A, and 7740.[2] An aliquot of the extraction fluid was also prepared and analyzed by Method 7470 for mercury.

Five 10-gram samples were shipped for cyanide analyses. These were two AZ-102 glass samples, two AZ-102 simulant glass samples, and an LRM glass sample. The LRM glass was used as a field blank for the cyanide analyses. Five grams of each sample were pretreated using Method 9010B.[2] Cyanide in the resulting distillate was then determined colorimetrically using Method 9014.[2]

2.1.3 Dissolved-Glass Sample Preparation and Analysis

Three subsets of dissolved glass samples were generated using two of the three methods given in ASTM C1463-00.[3] These three subsets of samples were:

- acid-dissolved glass samples, used for metal analyte determinations
- fusion-dissolved glass samples with an acid uptake, used for metal analyte determinations
- fusion-dissolved glass samples without the acid, used for anion analyte determinations

Each of these sets consisted of seven samples. These were duplicate AZ-102 glass samples, duplicate nonradioactive AZ-102 simulant glass samples, duplicate LRM glass standards, and a preparation blank. The LRM glass was not a true field blank since this material was known to contain several of the target analytes; however, a suitable field blank was not available. Water would not be a representative field blank, and a qualified analysis was not available for any reagent-grade sand that could have been used as a field blank.

In an attempt to avoid diluting the samples, 0.5 gram of glass was used instead of 0.25 grams as specified in ASTM C1463-00.[3] Acid-dissolved glass samples were generated by treating samples to a series of acid additions and heating steps including the addition of nitric acid, hydrofluoric acid, boric acid, and hydrochloric acid. The final solution was then brought to 100 milliliters with water instead of 250 milliliters as specified in ASTM C1463-00.[3]

Two sets of peroxide fusion samples were generated. In each set, 0.5 gram of glass was used instead of 0.25 grams. For the metal analyses, samples were generated by adding sodium peroxide and sodium hydroxide to the sample in a nickel crucible and heating the mixture to 700 ^{o}C. Concentrated nitric acid was then added to the samples. All samples were then brought to 100 milliliters with water instead of 250 milliliters as indicated in ASTM C1463-00.[3] For anion analyses, the same procedure was used, except no nitric acid was added.

Dissolved glass samples were collected and shipped to BWX Technology Services (BWXS) for metal and anion analyses. The seven acid-dissolved samples and seven fusion-dissolved samples with acid uptake were digested using Method 3015A. The resulting solutions were analyzed by Methods 6010B, 7060A, 7421, 7740, 7761, and 7841.[2] Aliquots of the original samples were also prepared and analyzed by Method 7470 for mercury.[2] The other seven samples were analyzed using Method 9056.

2.2 CONCENTRATION OF METAL ANALYTES IN AZ-102 GLASS SAMPLES

Results from the regulatory analysis of the metal analytes have been presented in this section. These results are the data provided by BWXS for the dissolved glass samples multiplied by the applicable dissolution factor (approximately 500 mL/g). The data package associated with these results will be issued as a separate package and will include the CLP-type data packages issued by BWXS, the spreadsheet used for data reduction, and the data validation report.[8] Data qualifiers used in this report were consistent with data qualifiers defined in the Tank Waste Information System.

2.2.1 Results from Metal Analyte Determinations

For all target analytes, at least one data set met all QA specifications, except silicon results, which did not meet recovery specifications for either acid or fusion-dissolved samples. Except where noted in this section, samples met all QA specifications of the task plan,[5] the QAPjP,[6] and USEPA SW-846.[2] Results have been presented on an elemental basis and on an oxide basis. Oxide percent results have been presented for the AZ-102 simulant and the LRM glasses.

2.2.1.1 Elemental Concentration of Analytes in AZ-102 Glass Samples

The concentrations of elemental analytes have been reported in the following subsections. Results from the acid-dissolved glass and the fusion-dissolved glass were within three standard deviations for all analytes detected at concentrations greater than the EQL except for arsenic results. Three standard deviations were identified in the test specification for this task. Arsenic concentrations were close to the EQL.

Elemental Results from Acid-Dissolved AZ-102 Glass Samples
With the following exceptions, acid-dissolved glass results (Table 2) met QA requirements:

- Arsenic Atomic Absorption Spectroscopy (AAS) matrix spike (MS) recovery (127 percent) was outside the specified range.
- Mercury results were flagged as estimated because sample-holding time limits were exceeded.
- Lead AAS MS and silicon matrix spike duplicate (MS) recoveries (44 and 71 %, respectively) were outside the specified range (75 to 125 %).
- Thallium AAS MS and MSD recoveries were extremely low (1.8 and 10 %, respectively).
- Thallium and lead AAS MS and MSD relative percent differences (RPDs) (140 and 64 %, respectively) were above the specified limit (25 %).
- Thallium ICP-ES results met QA specifications, but the MDL was above UTS limits.

Elemental Results from Fusion-Dissolved AZ-102 Glass Samples
With the following exceptions, fusion-dissolved glass results (Table 3) met QA requirements:

- MS and MSD recoveries for silver determined by AAS (9.4 and 0.2 %) and Inductively-Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) (73 and 70 %) were outside the specified range (75 to 125 %)..
- Arsenic AAS MS and MSD recoveries (135 and 152 %, respectively) were outside the specified range (75 to 125 %).
- Silicon MS and MSD recoveries (44 and <0 %) were outside the specified range (75 to 125 %).
- Thallium AAS MS and MSD recoveries (63 and 74.5 %, respectively) were outside the specified range of 75 to 125 percent.
- Thallium ICP-AES results met QA specifications, but the MDL was above UTS limits.

2.2.1.2 Oxide Composition of AZ-102 Glass Samples and Standards

Measured oxide compositions for AZ-102, nonradioactive simulant, and LRM glasses (Table 3) were within ten percent of target compositions with the following exceptions:

- Silica concentrations for the fusion-dissolved glasses suffered from very poor precision.
- Chromium concentrations were higher than expected in the AZ-102 glass and simulant.
- One iron and one zirconium result were high for the acid-dissolved glass samples.

2.2.1.3 Summary for Results from Metal Analyte Determinations

For most analytes, results given in Table 2 and Table 3 showed good agreement and were in line with expected values. Of analytes detected above the EQL, only arsenic differed by more than three standard deviations between the acid and fusion-dissolved glasses. Silver results were significantly different when measured by the two different methods. Although arsenic was not detected by AAS, it was reported by the ICP-AES at concentrations two orders of magnitude higher. Although interference corrections were made according to USEPA SW-846 protocol, the elevated ICP-AES arsenic were most likely due to incomplete correction for the high aluminum concentration in the glass. Although the ICP-AES results are usually less reliable for arsenic, the conservative approach would be to use this higher result since arsenic is a RCRA metal.

Table 2. Metal Concentrations Determined from Acid-Dissolved Samples

CAS No.	[1]Analyte	Sample Results (mg/kg)			Duplicate Results (mg/kg)			[5]Average (mg/kg)
		Result	[2]MDL	[3]EQL	Result	[2]MDL	[3]EQL	
7440-22-4	Ag(AA)	19.0	1	11	17.0	1	10	18±1.4
7440-22-4	Ag	1.246E2	6	59	1.235E2	6	58	1.241±0.0082E2
7429-90-5	Al	3.347E4	9E2	9E3	3.330E4	9E2	9E3	3.34±0.012E4
7440-38-2	[4]As	1.368E2	13	1E2	1.385E2	13	1E2	1.38±0.012E2
7440-39-3	Ba	[J]1.23E2	30	3E2	[J]1.05 E2	30	3E2	[J]1.1±0.013E2
7440-41-7	Be	[U]4	4	38	[U]4	4	38	[U]4
7440-70-2	Ca	5.463E4	1E3	1E4	5.460E4	1E3	1E4	5.462±0.0015E4
7440-43-9	Cd	[J]2.2	2	17	[U]2	2	17	[J]2.2
7440-47-3	Cr	5.039E2	8	81	5.061E2	8	80	5.05±0.015E2
7439-89-6	Fe	1.96E4	3E2	3E3	1.61E4	3E2	3E3	1.8±0.24E4
7439-97-6	Hg(AA)	[UY]0.4	0.4	4	[UY]0.4	0.4	4	[UY]0.4
7440-09-7	K	[J]4.5E3	3E3	3E4	[U]3E3	3E3	3E4	[J]4.5E3
7439-93-2	Li	2.135E4	2E2	2E3	2.055E4	2E2	2E3	2.09±0.057E4
7439-95-4	Mg	9.002E3	6E2	6E3	8.657E3	6E2	6E3	8.83±0.024E3
7439-98-7	Mo	[U]2E2	2E2	2E3	[U]2E2	2E2	2E3	[U]2E2
7440-23-5	Na	3.385E4	1E3	1E4	3.349E4	1E3	1E4	3.37±0.026E4
7440-02-0	Ni	[J]6.8E2	1E2	1E3	[U]1E2	1E2	1E3	[J]6.8E2
7723-14-0	P	[U]3E2	3E2	3E3	[U]3E2	3E2	3E3	[U]3E2
7439-92-1	[4]Pb	[U]36	36	4E2	[U]35	35	4E2	[U]35
63705-05-5	S	3.424E3	2E2	2E3	3.387E3	2E2	2E3	3.41±0.026E3
7440-36-0	Sb	[U]15	15	2E2	[U]15	15	1E2	[U]15
7782-49-2	Se(AA)	[U]5	5	53	[U]5	5	52	[U]5
7782-49-2	Se	[U]29	29	3E2	[U]29	29	3E2	[U]29
7440-21-3	Si	[b]2.520E5	6E2	6E3	[b]2.454E5	6E2	6E3	[b]2.49±0.046E5
7440-32-6	Ti	[J]1.166E3	2E2	2E3	[J]1.122E3	2E2	2E3	[J]1.14±0.030E3
7440-28-0	Tl	[U]48	48	5E2	[U]48	48	5E2	[U]48
7440-62-2	V	33.08	3	25	32.34	2	25	32.7±0.52
7440-66-6	Zn	4.020E4	83	8E2	4.051E4	82	8E2	4.04±0.023E4
7440-67-7	Zr	2.7E4	8E2	8E3	2.4E4	8E2	8E3	2.6±0.22E4

[1] Analyzed by Method 6010B except where "(AA)" indicates results from atomic absorption spectroscopy.

[2] MDLs were from USEPA SW-846 MDL studies using standards in a water matrix. Dilution factors applied.

[3] EQLs were estimated as ten times the MDL. Calibrations used at least one concentration less than the EQL.

[4] ICP-AES results for these elements should be used exclusively. AAS results are included in the data package.

[5] Uncertainties are given as one standard deviation between the sample and the duplicate.

[J] Estimated value. Result was less than the EQL and greater than the MDL.

[U] Undetected. The analyte was not detected. The concentration was less than the MDL.

[Y] Result is an estimate because mercury pretreatment performed outside allowable hold times by 14 days.

[b] Matrix spike, matrix spike duplicate, or post spike recovery outside the acceptance range.

Table 3. Metal Concentrations Determined from Fusion-Dissolved Samples

CAS No.	[1]Analyte	Sample Results (mg/kg)			Duplicate Results (mg/kg)			[4]Average (mg/kg)
		Result	[2]MDL	[3]EQL	Result	[2]MDL	[3]EQL	
7440-22-4	Ag(AA)	[b]54.8	1	11	[b]62.4	1	11	[b]59±5.4
7440-22-4	Ag	[b]1.216E2	12	1E2	[b]1.235E2	12	1E2	[b]1.23±0.019E2
7429-90-5	Al	3.197E4	9E2	9E3	3.287E4	9E2	9E3	3.24±0.064E4
7440-38-2	As(AA)	[Ub]1	1	14	[Ub]1	1	13	[Ub]1
7440-38-2	As	2.81E2	26	3E2	2.63E2	13	3E2	2.7±0.10E2
7440-42-8	B	4.05E4	3E2	3E3	3.77E4	3E2	3E3	3.9±0.20E4
7440-39-3	Ba	[J]1.014E2	31	3E2	[J]1.011E2	31	3E2	[J]1.01±0.0015E2
7440-41-7	Be	[U]4	4	39	[U]4	4	39	[U]4
7440-70-2	Ca	5.34E4	1E3	1E4	5.52E4	1E3	1E4	5.4±0.13E4
7440-43-9	Cd	[U]4	4	35	[J]6.4	4	35	[J]6.4
7440-47-3	Cr	5.07E2	17	2E2	5.40E2	16	2E2	5.2±0.23E2
7439-89-6	Fe	1.511E4	3E2	3E3	1.556E4	3E2	3E3	1.53±0.032E4
7440-09-7	K	[J]4.04E3	4E3	4E4	[J]3.52E3	4E3	4E4	[J]3.8±0.36E3
7439-93-2	Li	2.086E4	2E2	2E3	2.023E4	2E2	3E3	2.05±0.044E4
7439-95-4	Mg	8.713E3	6E2	6E3	8.773E3	6E2	6E3	8.74±0.042E3
7439-98-7	Mo	[U]2E2	2E2	2E3	[U]2E2	2E2	2E3	[U]2E2
7723-14-0	P	[U]3E2	3E2	3E3	[U]3E2	3E2	3E3	[U]3E2
7439-92-1	Pb(AA)	[J]12.1	2	19	[J]6.2	2	18	[J]9±4.1
7439-92-1	Pb	[J]1.055E2	72	7E2	[J]1.117E2	72	7E2	[J]1.09±0.044E2
63705-05-5	S	3.57E3	2E2	2E3	3.27E3	2E2	2E3	3.4±0.21E3
7440-36-0	Sb	[U]30	30	3E2	[U]30	30	3E2	[U]30
7782-49-2	Se(AA)	[J]6.9	5	53	[U]5	5	53	[J]6.9
7782-49-2	Se	[U]60	60	6E2	[U]60	60	6E2	[U]60
7440-21-3	Si	[b]3.4E5	6E3	6E4	[b]1.7E5	6E3	6E4	[b]2.5±1.1E5
7440-32-6	Ti	[J]1.333E3	2E2	2E3	[J]1.350E3	2E2	2E3	[J]1.34±0.012E3
7440-28-0	Tl(AA)	[Ub]3	3	27	[Ub]3	3	27	[Ub]3
7440-28-0	Tl	[U]97	97	1E2	[U]97	97	1E2	[U]97
7440-62-2	V	[J]36.65	5	51	[J]36.96	5	51	[J]36.8±0.22
7440-66-6	Zn	3.899E4	84	8E2	3.988E4	84	8E2	3.94±0.063E4
7440-67-7	Zr	2.242E4	8E2	8E3	2.179E4	8E2	8E3	2.21±0.045E4

[1] Analyzed by Method 6010B except where "(AA)" indicates results from atomic absorption spectroscopy.
[2] MDLs were from USEPA SW-846 MDL studies using standards in a water matrix. Dilution factors applied.
[3] EQLs were estimated as ten times the MDL. Calibrations used at least one concentration less than the EQL.
[4] Uncertainties are given as one standard deviation between the sample and the duplicate.
[J] Estimated value. Result was less than the EQL and greater than the MDL.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL.
[b] Matrix spike, matrix spike duplicate, or post spike recovery outside the acceptance range.

For most analytes, results given in Table 4 were within three standard deviations of the targets. Catholic University provided the AZ-102 target composition along with the quantities of AZ-102 supernate and glass-forming minerals used to make the glass. This will be reported upon completion of the vitrification task.[1] To determine the simulant target, the composition of the AZ-102 supernate simulant was used. Totals given in Table 4 suggested analyses were successful in determining all major glass constituents (those present above 0.5 wt percent). The purpose of the simulant was to act as a nonqualified "standard" for the vitrification process and to verify any issues prior to vitrification with the AZ-102 material.

2.2.2 Preparation Blank Results for Metal Analyte Determinations

With the following exceptions, preparation blank results (Table 5) were below the minimum reportable quantities (MRQs):

- Thallium, silver, and lead MDLs were above the MRQs for the acid-dissolved SRTC blanks. Neither thallium nor lead was detected in the blanks.
- Silver and sodium were detected above the MRQs in the acid-dissolved SRTC blanks.
- Thallium, lead, and antimony MDLs were above MRQs in fusion-dissolved SRTC blanks.
- Six metals (silver, arsenic, calcium, cadmium, chromium, and silicon) were detected above the MRQs in the fusion-dissolved SRTC blanks.

2.2.3 Recoveries Associated with Metal Analyte Determinations

QA specifications established acceptable values for recoveries. MS, MSD, and post spike (PS) recoveries were acceptable from 75 to 125 percent. Laboratory control samples (LCS) acceptable recoveries were from 80 to 120 percent. Serial dilution acceptable recoveries were 90 to 110 percent. LCS recoveries have been included in the following subsections. MS, MSD, PS, and serial dilution recoveries have been included in Appendix A.

2.2.3.1 Laboratory Control Sample Recoveries for Metal Analytes

All LCS recoveries were between 90 and 110 percent. These results have been given in Table 6. Because the BWXS standard LCS mix did not contain all target analytes, a blank spike was prepared and analyzed as the LCS. Because samples were often analyzed at two dilutions, the data package for these analyses often contains two LCS results. Table 6 contains LCS recoveries for the dilutions associated with the reported results. For the dilution that was not reported, the boron LCS recovery was high (130 percent).

2.2.3.2 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Recoveries

MS and MSD results have been given in Appendix A. With the following exceptions, acid-dissolved samples met MS, MSD, and Relative Percent Difference (RPD) specifications. The AAS MS recovery for arsenic was slightly high (127 percent). The MSD recovery for silicon was low (70.8 percent). For lead, the AAS MS recovery was low (43.6 percent), and RPD was high (63.7 percent). For thallium, AAS MS and MSD recoveries were extremely low (1.8 and 10.3 percent) and the RPD was high (140 percent).

With the following exceptions, fusion-dissolved glass results met MS recovery and RPD specifications. For silver, MS and MSD recoveries were low for AAS (22.2 and 25.4 percent) and ICP-AES analyses (72.9 and 70.0 percent). For the thallium AAS analysis, MS and MSD recoveries were low (63.2 and 74.5 percent). The RPD for the thallium AAS analyses (16 percent) met the QA specification (< 20 percent,) but was higher than most other RPDs. MS and MSD recoveries were high for arsenic AAS analysis (135 and 152 percent). MS and MSD recoveries were very low (43.8 and 0 percent), and RPD was high (84.9 percent) for silicon.

2.2.3.3 Post Spike (PS) Recoveries for Metal Analytes

PS recovery results have been given in Appendix A. With the following exceptions, all analyses met the PS recovery specification. The acid and fusion PS recoveries were low for the thallium AAS analysis (16.7 and 71.7 percent) and for the silicon recoveries (58.6 and 35.4 percent). The silver PS recovery was very low for the fusion samples (15 percent). For arsenic, the AAS analysis acid and fusion PS recoveries were high (131 and 161 percent).

2.2.3.4 Serial Dilution Results for Metal Analytes

Serial dilution results have been given in Appendix A. Analyses met the serial dilution specification for all analytes in all samples. Serial dilutions apply only to analytes that have been detected in the samples at concentrations that will be more than ten times the analyte MDL after dilution. In the data package for these analyses, some of the silver results carry a flag indicating a serial dilution failure. Because the silver concentration was below ten times the MDL, the serial dilution qualifiers should not have been applied.

Table 4. Metal Oxide Concentrations

Oxide	**[1]Target (weight %)**			**[4]Measured Composition (weight %)**				
	Spl	**Sim**	**LRM**	**AZ-102 Sample**		**Simulant**		**LRM**
				Acid	Fusion	Acid	Fusion	
Ag_2O	-	-	-	1.33±0.009E-2	[b]1.32±0.020E-2	1.29±0.020E-2	[b]1.30±0.093E-2	[J]3.7±0.29E-3
Al_2O_3	6.5	6.43	9.5	6.31±0.023	6.13±0.12	6.01±0.080	5.92±0.016	9.3±0.17
As_2O_3	-	-	-	1.82±0.016E-2	3.6±0.13E-2	1.76±0.030E-2	[J]2.87±0.066E-2	1.9±1.8E-2
B_2O_3	13.0	13.0	7.9	[N]12.6±0.64	12.6±0.64	[N]13.1±0.66	13.1±0.66	7.9±0.16
BaO	-	-	-	[J]1.3±0.15E-2	[J]1.13±0.017E-2	[J]1.3±0.28E-2	[J]1.21±0.074E-2	[U]3±E-3
BeO	-	-	-	[U]1E-3	[U]1E-3	[J]2.1E-3	[U]1E-3	[U]1±E-3
CaO	8.0	7.97	0.54	7.642±0.0021	7.6±0.18	7.4±0.11	7.54±0.058	[J]0.70±0.036
CdO	-	-	0.16	[J]2.5E-4	[J]7.3E-4	[U]2E-4	[J]5.59±0.015E-4	0.18±0.029
Cr_2O_3	0.06	0.06	0.19	7.38±0.022E-2	7.6±0.33E-2	7.20±0.035E-2	7.66±0.081E-2	0.20±0.014
Fe_2O_3	2.20	2.20	1.42	2.6±0.35	2.19±0.046	2.30±0.013	2.19±0.095	1.5±0.10
K_2O	0.21	0.20	1.48	[J]0.54	[J]0.49	[U]0.4	[U]0.4	[J]0.15±0.13
Li_2O	4.69	4.69	0.11	4.5±0.12	4.42±0.096	4.4±0.10	4.53±0.22	[J]0.115±0.0022
MgO	1.41	1.41	0.10	1.46±0.040	1.450±0.0069	1.49±0.012	1.450±0.0069	[J]0.14
MoO_3	-	-	0.10	[U]3E-2	[U]3E-2	[U]3E-2	[U]3E-2	[J]9.5±0.90E-2
Na_2O	5.00	5.08	20.03	4.54±0.035	[N]4.54±0.035	5.07±0.043	[N]5.07±0.043	20.3±0.33
NiO	-	-	0.19	[J]8.7E-2	[JN]8.7E-2	[J]2.0±0.18E-2	[JN]2.0±0.18E-2	0.196±0.0033
P_2O_5	0.02	0.02	0.53	[U]7E-2	[U]7E-2	[U]6E-2	[U]7E-2	[J]0.39±0.096
PbO(AA)	-	-	0.10	[Jbc]7.8±1.5E-4	[J]9.9±4.5E-4	[Jbc]7.2±4.2E-4	[J]4.43±0.14E-4	2.50±0.064E-2
PbO	-	-	0.10	[U]4E-3	[J]1.17±E-2	[J]3.8±0.28E-3	[J]9.18±E-3	0.11±0.030
SO_3	0.86	0.77	0.30	0.850±0.0066	0.85±0.053	0.94±0.021	0.92±0.040	[J]0.45±0.037
SeO_2	-	-	-	[U]7E-4	[J]9.7E-4	[U]7E-4	[J]9.2E-4	[J]8.0±0.76E-4
SiO_2	50.0	50.0	54.3	[b]53.2±0.99	[3b]53.2±0.99	[b]49.7±0.35	[b]49.7±0.35	56±1.3
TiO_2	-	-	0.11	[J]0.191±0.0051	[J]0.224±0.0020	[J]0.17±0.031	[J]0.188±0.0095	[J]0.108±0.0055
V_2O_3	-	-	-	4.81±0.077E-3	[J]5.41±0.032E-3	4.8±0.14E-3	[J]5.1±0.39E-3	[J]1.23±0.57E-3
ZnO	4.87	4.88	-	5.02±0.029	4.91±0.078	4.87±0.087	4.83±0.022	[J]2.26±0.80E-2
ZrO_2	3.19	3.19	0.93	3.5±0.30	2.99±0.060	3.44±0.088	3.0±0.14	0.95±0.15
Cl	0.01		0.07	[UY]0.2	[UY]0.2	[UY]0.2	[UY]0.2	[UY]0.2
F	0.05	0.10	0.86	[UY]0.2	[UY]0.2	[UY]0.2	[UY]0.2	[J]0.74±0.015
[2]Total	100	100	100	102.3-103.0	101.0-101.8	98.8-99.0	98.4-98.6	96-100

[1] Spl-AZ102 target. Sim-AZ102 target adjusted for simulant composition. LRM standard glass composition.

[2] Total range is: Lower value only from species above the EQL. Higher value from all species above the MDL.

[3] Peroxide fusion result showed poor precision. Therefore, the silicon value was taken from the acid results.

[4] Uncertainties are given as one standard deviation between the sample and the duplicate.

[J] Estimated value. Result was less than the EQL and greater than the MDL given in Table 3 or Table 4.

[U] Undetected. The analyte was not detected. The concentration was less than the MDL given in Table 3 or Table 4.

[b] Matrix spike, matrix spike duplicate, or post spike recovery outside the acceptance range.

[c] The relative percent difference between the MS and MSD was outside the acceptance range.

[N] Boron values taken from the fusion results. Sodium and nickel values taken from the acid results.

[Y] Result is an estimate because mercury pretreatment performed outside allowable holdtimes by 14 days.

- Indicates an analyte that was only present at trace levels and for which no analytical value was available.

Table 5. Preparation Blank Results for AZ-102 Glass Sample Metal Analyses

CAS No.	[1]Analyte	MRQ (mg/kg)	[2]Laboratory Blank (mg/kg)		[3]SRTC Blank (mg/kg)	
			Acid	Fusion	Acid	Fusion
7440-22-4	Ag(AA)	2.8	^{U}4E-2	^{U}4E-2	17	^{b}58
7440-22-4	Ag	2.8	^{U}1	^{U}1	^{U}6	^{Jb}25
7429-90-5	Al	5E3	^{U}9	^{U}9	8.6E2	^{U}9E2
7440-38-2	As(AA)	1E2	^{U}5E-2	^{U}5E-2	^{Ub}1	^{Ub}1
7440-38-2	As	1E2	^{U}3	^{U}3	^{U}13	^{J}2.5E2
7440-42-8	B	5E3	^{U}9	^{U}3	N	^{J}6.8E2
7440-39-3	Ba	4.2E2	^{J}0.60	^{J}0.61	^{J}87	^{J}1.2E2
7440-41-7	Be	24.4	^{U}4E-2	^{U}4E-2	^{U}4	^{J}16
7440-70-2	Ca	5E3	^{U}13	^{U}14	^{U}1E3	^{J}4.2E3
7440-43-9	Cd	2.2	^{U}0.3	^{U}0.4	^{U}2	^{J}9.6
7440-47-3	Cr	12	^{U}2	^{U}2	^{U}8	^{J}35
7439-89-6	Fe	5E3	^{J}6.7	^{J}6.8	^{J}3.3E2	^{J}2.0E3
7439-97-6	Hg	0.5	^{U}4E-2	^{U}4E-2	^{UY}0.4	^{UY}0.4
7440-09-7	K	5E3	^{U}34	^{U}35	^{U}3E3	^{U}4E3
7439-93-2	Li	5E3	^{U}2	^{U}2	^{U}2E2	^{U}2E2
7439-95-4	Mg	5E3	^{U}6	^{U}6	^{U}6E2	^{U}6E2
7439-98-7	Mo	5E3	^{U}2	^{U}2	^{U}2E2	^{U}2E2
7440-23-5	Na	5E3	^{U}10	^{U}11	1.2E4	N
7440-02-0	Ni	2.2E2	^{U}1	^{U}1	^{U}1E2	N
7723-14-0	P	5E3	^{U}3	^{U}3	^{U}3E2	^{U}3E2
7439-92-1	Pb(AA)	15	^{U}7E-2	^{U}7E-2	^{Jbc}5.2	^{Jbc}5.2
7439-92-1	Pb	15	^{U}7	^{U}7	^{U}36	^{U}36
63705-05-5	S	5E3	^{J}6.4	^{J}6.5	^{J}7.0E2	^{J}7.9E2
7440-36-0	Sb	23	^{U}3	^{U}3	^{U}15	^{U}31
7782-49-2	Se(AA)	1.14E2	^{U}0.2	^{U}0.2	^{U}5	^{U}5
7782-49-2	Se	1.14E2	^{U}6	^{U}6	^{U}30	^{U}60
7440-21-3	Si	5E3	^{U}6	^{U}6	^{Jb}1.0E3	^{Jb}6.2E3
7440-32-6	Ti	5E3	^{J}2.0	^{J}2.1	^{U}2E2	^{U}2E2
7440-28-0	Tl(AA)	4	^{U}0.1	^{U}0.1	^{URc}3	^{Ub}3
7440-28-0	Tl	4	^{U}10	^{U}9.8	^{U}48	^{U}98
7440-62-2	V	32	^{U}0.5	^{U}0.5	^{U}3	^{U}5
7440-66-6	Zn	86	^{U}0.8	^{U}0.8	^{U}83	^{J}6.2E2
7440-67-7	Zr	5E3	^{U}8	^{U}8	^{U}8E2	^{U}8E2

[1] Analyzed by Method 6010B except where "(AA)" indicates results from atomic absorption spectroscopy.
[2] Preparation blank generated by BWXS during the final acid digestion.
[3] Digestion blank generated by SRTC during acid dissolution or peroxide dissolution of the glass waste form.
[J] Estimated value. Result was less than the EQL and greater than the MDL given in Table 3 or Table 4.
[R] Atomic absorption thallium results for acid dissolved samples unusable because of extremely low recoveries.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL given in Table 3 or Table 4.
[Y] Result is an estimate because mercury pretreatment performed outside allowable hold times.
[b] Matrix spike or matrix spike duplicate recovery outside the acceptance range.
[c] The relative percent difference between the MS and MSD was outside the acceptance range.
N Boron results only included for fusion samples. Sodium and nickel results only included for acid samples.

Table 6. Laboratory Control Sample Results for Metal Analyses

CAS No.	[1]Analyte	[2]Concentrations (μg/L)				[3]Recoveries (%)	
		Acid		Fusion		Acid	Fusion
		True	Measured	True	Measured		
7440-22-4	Ag(AA)	5.00	5.04	2.14E2	2.01E2	101	93.9
7440-22-4	Ag	2.14E2	2.14E2	2.14E2	2.09E2	100	97.7
7429-90-5	Al	8.67E2	8.53E2	8.67E2	8.53E2	98.4	98.4
7440-38-2	As(AA)	15.0	15.4	15.0	15.4	103	103
7440-38-2	As	3.66E2	3.63E2	3.66E2	3.59E2	99.2	98.1
7440-42-8	B	[N]1.25E2	[N]136E2	1.00E3	1.06E3	[N]109	[Y]106
7440-39-3	Ba	1.00E3	1.00E3	1.00E3	1.00E3	100	100
7440-41-7	Be	1.11E2	1.12E2	1.00E3	1.00E3	101	100
7440-70-2	Ca	1000E3	1.08E2	1.00E3	1.08E3	108	108
7440-43-9	Cd	73.1	68.5	73.1	67.2	93.7	91.9
7440-47-3	Cr	3.70E2	3.66E2	3.70E2	3.58E2	98.9	96.8
7439-89-6	Fe	3.58E2	3.99E2	3.58E2	3.99E2	111	111
7439-97-6	Hg	17.5	16.6	17.5	16.6	94.9	94.9
7440-09-7	K	1.00E4	9.77E3	1.00E4	9.77E3	97.7	97.7
7439-93-2	Li	1.00E3	9.75E2	1.00E3	9.75E2	97.5	97.5
7439-95-4	Mg	1.00E3	9.65E2	1.00E3	9.65E2	96.5	96.5
7439-98-7	Mo	1.53E2	1.52E2	1.53E2	1.52E2	99.3	99.3
7440-23-5	Na	1.00E3	1.04E3	[N]1.00E3	[N]1.06E3	104	[N]106
7440-02-0	Ni	1.00E3	1.01E3	[N]1.00E3	[N]1.07E3	101	[N]107
7723-14-0	P	2.00E3	2.00E3	2.00E3	2.00E3	100	100
7439-92-1	Pb(AA)	10.0	10.0	10.0	10.0	100	100
7439-92-1	Pb	2.76E2	2.62E2	2.76E2	2.57E2	94.9	93.1
63705-05-5	S	2.00E3	2.10E3	2.00E3	2.10E3	105	105
7440-36-0	Sb	1.73E2	1.90E2	1.73E2	1.64E2	110	94.8
7782-49-2	Se(AA)	15.0	15.8	15.0	15.8	105	105
7782-49-2	Se	2.64E2	2.54E2	264.0	2.53E2	96.2	95.8
7440-21-3	Si	5.00E2	5.09E2	5.00E2	5.09E2	102	102
7440-32-6	Ti	1.00E3	1.03E3	1.00E3	1.03E3	103	103
7440-28-0	Tl(AA)	15.0	14.0	15.0	14.0	93.3	93.3
7440-28-0	Tl	7.31E2	7.15E2	7.31E2	7.05E2	97.8	96.4
7440-62-2	V	8.24E2	8.04E2	8.24E2	7.98E2	97.6	96.8
7440-66-6	Zn	1.74E2	1.80E2	1.74E2	1.80E2	103	103
7440-67-7	Zr	2.00E3	2.16E3	2.00E3	2.16E3	108	108

[1] Analyzed by Method 6010B except where “(AA)” indicates results from atomic absorption spectroscopy.

[2] Acceptable values for the LCS recoveries were from 75 percent to 125 percent.

[3] Several LCS results have been included in the data package for sample dilutions that were not reported.

[Y] LCS recovery failed (130 percent) for a dilution that was not used in determining this analyte.

[N] Although this was the reported LCS, the corresponding analyte result was not determined in these samples.

2.2.4 Narrative for Metal Analyte Results

Results from the metal analytes determinations demonstrated the following:

- At least one determination met all QA specifications for each analyte, except for silicon.
- Silicon results from acid-dissolutions failed two recoveries due to low spike concentrations.
- Except arsenic, results were within three standard deviations for acid and fusion samples.
- Results for most analytes were within three standard deviations of the target composition.
- Total oxide content indicates analyses were successful in analyzing major glass constituents.

At least one determination met all QA specifications for each analyte, except for silicon. Mercury measurements in the dissolved glass were completed after the allowable holding time; however, TCLP results indicated the mercury concentration was below the RCRA limits. Although all other metal analyses were performed within the specified holding times, mercury holding times are more restrictive. The 28-day mercury holding time limit was exceeded due to delays in initiation of the sample preparation at BWXS. For arsenic and thallium, only ICP-AES results met all QA specifications in both sets of samples. Only the acid-dissolved glass sample analyses met all QA specifications for silver, and results for silver were much higher for the ICP-AES results than for the AAS results.

Silicon results from acid dissolution met all QA specifications, except two recovery failures caused by use of a low spike concentration rather than by the sample matrix. The acid-dissolved glass sample contained 1.310 x 10^3 μg/L in the sample. The sample was spiked with 500 μg/L to create the MS, MSD, and PS. The MS recovery and RPD met specifications; however, the MSD and PS did not.

For all metal analytes detected above the EQLs, results from the metal dissolutions were within three standard deviations of the results from the peroxide fusions except for the arsenic results. In reality, all results were within two standard deviations; however, the success criterion was specified as three standard deviations in the test specification for this task. Although the difference between the arsenic results was large, these values were close to the EQL.

Results were shown to be within ten percent of the target for all analytes, except chromium. Chromium oxide was determined to be in the glass at 0.0738 and 0.076 weight percent for the acid and fusion samples respectively, compared to the target concentration of 0.06 weight percent. The measured chromium was similarly high in the nonradioactive simulant, 0.076 and 0.0720 weight percent for the acid and fusion samples respectively, compared to the target value of 0.06 weight percent.

The total measured oxides (102.3 to 103.0 weight percent) indicated all major constituents (those present at 0.5 weight percent or higher) had been reported. This was a success criterion for the task. The lower value was the sum of oxides present at above the EQL. This was the sum of oxides that could be determined quantitatively. The second was the total of oxides present at concentrations above the MDL. This was the sum of oxides present at concentrations high enough to be detected, even if they could not be determined quantitatively.

2.3 TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

Results from the TCLP tests on the AZ-102 glass samples are presented in this section. Results were taken from data provided by BWXS on the crushed glass samples. The data will be issued as a separate data package. It will include the BWXS CLP-type data package, the spreadsheet used for data reduction, and the validation report for the TCLP results and for the dissolved glass results.[8] Data qualifiers used in this report are consistent with qualifiers defined in the Tank Waste Information System.

2.3.1 Results from TCLP Tests

For all target analytes, TCLP results met all QA specifications except that the PS recoveries for nickel and lead were slightly low, 74.7 and 74.9 percent respectively. Extraction blanks contained low concentrations of zinc (0.094 mg/L) and barium (0.064 mg/L). Except where noted in this section, samples met all QA specifications of the task plan,[5] the QAPjP,[6] and USEPA SW-846.[2] Results have been presented for the AZ-102 radioactive glass waste form samples, the nonradioactive simulant glass waste form samples, and a TCLP standard.

2.3.1.1 TCLP Results for AZ-102 Glass Waste Form Samples

Results for the AZ-102 radioactive glass waste form TCLP tests have been given in Table 7. These results showed:

- All metals were well below the limits for characteristically hazardous material.
- All metals were well below the UTS limits except thallium.
- Thallium detection limit was higher than the UTS limit for thallium.
- All QA specifications were met, except for PS recoveries for nickel and lead.

TCLP results given in Table 7 were sufficient to show the TCLP leachate RCRA metals were below the RCRA metal characteristic and UTS limits for all RCRA metals except thallium. Thallium was not detected in any of the samples. The AAS result for thallium in the fusion-dissolved glass were low enough to show the waste form did not contain enough to fail a TCLP test; however, the matrix spike and matrix spike duplicate recoveries were outside specified limits for these samples (63.2 and 74.5 percent respectively).

Table 7. TCLP Results from AZ-102 Glass Analyses

CAS No.	Analyte	Lab Limits (mg/L)		Results (mg/L)			Federal Limits (mg/L)	
		[1]MDL	[2]EQL	Sample	Duplicate	Average	Characteristic	UTS
7440-22-4	Ag	6E-2	0.6	[U]6E-2	[U]6E-2	[U]6E-2	5	0.14
7440-38-2	As	3E-2	0.3	[U]3E-2	[U]3E-2	[U]3E-2	5	5
7440-39-3	Ba	2E-2	0.2	[B]0.246	[B]0.214	[B]0.23±0.023	1E2	21
7440-41-7	Be	2E-3	2E-2	[U]2E-3	[U]2E-3	[U]2E-3	-	1.22
7440-43-9	Cd	2E-2	0.2	[U]2E-2	[U]2E-2	[U]2E-2	1	0.11
7440-47-3	Cr	8E-2	0.8	[U]8E-2	[U]8E-2	[U]8E-2	5	0.6
7439-97-6	Hg	2E-3	2E-2	[U]2E-3	[U]2E-3	[U]2E-3	0.2	2.5E-2
7440-02-0	Ni	6E-2	0.6	[UY]6E-2	[UY]6E-2	[UY]6E-2	-	11
7439-92-1	Pb	0.4	4	[UY]0.4	[UY]0.4	[UY]0.4	5	0.75
7440-36-0	Sb	0.2	2	[J]0.25	[U]0.2	[J]0.25	-	1.15
7782-49-2	Se	0.1	1	[U]0.1	[U]0.1	[U]0.1	1	5.7
7440-28-0	Tl	0.5	5	[U]0.5	[U]0.5	[U]0.5	-	0.2
7440-62-2	V	3E-2	0.3	[J]2.8E-2	[J]4.3E-2	[J]3.6±2.3E-2	-	1.6
7440-66-6	Zn	4E-2	0.4	1.47	2.15	1.8±0.48	-	4.3

[1] MDLs were from USEPA SW-846 MDL studies using standards in a water matrix. Dilution factors applied.
[2] EQLs were estimated as ten times the MDL. Calibrations used at least one concentration less than the EQL.
[B] Extraction blank concentration was 0.0635 mg/L. By the method blank 5 times rule, this result is a nondetect.
[J] Estimated value. Result was less than the EQL and greater than the MDL.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL.
[Y] Post spike recoveries for nickel and lead were slightly low, 74.7 and 74.9 percent respectively.
- Indicates an analyte that is not used for determining whether a waste form is characteristically hazardous.

2.3.1.2 TCLP Results for Simulant Glass Waste Form Samples and Standard

The simulant glass TCLP results have been given in Table 8 and the results for the TCLP standards have been given in Table 9. The results show:

- TCLP results were very similar for the simulant and radioactive AZ-102 glasses.
- For the simulant, all metals were well below the UTS limits, except thallium.
- Thallium detection limit was higher than the UTS limit for thallium.
- All QA specifications were met, except for PS recoveries for nickel and lead.
- Some results for the TCLP standard were outside the standard acceptance range. The high barium concentration in one of the standard results was most likely due to contamination from the filtration medium used on the TCLP leachate.

Table 8. TCLP Results from Nonradioactive Simulant Glass

CAS No.	Analyte	[1]Blank (mg/L)	Simulant (mg/L)			Lab Limits (mg/L)		UTS (mg/L)
			Sample	Duplicate	Average	[2]MDL	[3]EQL	
7440-22-4	Ag	[U]6E-3	[U]6E-2	[U]6E-2	[U]6E-2	6E-2	0.6	0.14
7440-38-2	As	[U]3E-4	[U]3E-2	[U]3E-2	[U]3E-2	3E-2	0.3	5
7440-39-3	Ba	6.35E-2	0.88	[B]0.29	0.88	2E-2	0.2	21
7440-41-7	Be	[J]2.0E-4	[U]2E-3	[U]2E-3	[U]2E-3	2E-3	2E-2	1.22
7440-43-9	Cd	[U]2E-3	[U]2E-2	[U]2E-2	[U]2E-2	2E-2	0.2	0.11
7440-47-3	Cr	[U]8E-3	[U]8E-2	[U]8E-2	[U]8E-2	8E-2	0.8	0.6
7439-97-6	Hg	[U]2E-3	[U]2E-3	[U]2E-3	[U]2E-3	2E-3	2E-2	2.5E-2
7440-02-0	Ni	[JY]7.5E-3	[UY]6E-2	[UY]6E-2	[UY]6E-2	6E-2	0.6	11
7439-92-1	Pb	[UY]4E-2	[UY]0.4	[UY]0.4	[UY]0.4	0.4	4	0.75
7440-36-0	Sb	[U]2E-2	[J]0.23	[U]0.2	[J]0.23	0.2	2	1.15
7782-49-2	Se	[U]1E-3	[U]0.1	[U]0.1	[U]0.1	0.1	1	5.7
7440-28-0	Tl	[U]5E-2	[U]0.5	[U]0.5	[U]0.5	0.5	5	0.2
7440-62-2	V	[U]3E-3	[J]4.3E-2	[U]3E-2	[J]4.3E-2	3E-2	0.3	1.6
7440-66-6	Zn	9.37E-2	2.68	1.83	2.3±0.60	4E-2	0.4	4.3

[1] The data package includes two preparation blank results. The values in this table were for the extraction blank.
[2] MDLs were from USEPA SW-846 MDL studies using standards in a water matrix. Dilution factors applied.
[3] EQLs were estimated as ten times the MDL. Calibrations used at least one concentration less than the EQL.
[B] Extraction blank concentration was 0.0635 mg/L. By the method blank 5 times rule, this is a nondetect.
[J] Estimated value. Result was less than the EQL and greater than the MDL.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL.
[Y] Post spike recoveries for nickel and lead were slightly low, 74.7 and 74.9 percent respectively.

Table 9. TCLP Results from Standard Analyses

CAS No.	Analyte	[1]TCLP Standard (mg/L)		
		Standard	**Duplicate**	**Range**
7440-22-4	Ag	[ZJ]0.119	[ZJ]0.127	0.514-1.12
7440-38-2	As	[Z]0.96	1.54	1.27-2.59
7440-39-3	Ba	14.3	16.9	8.47-15.1
7440-41-7	Be	0.74	0.56	0.369-1.18
7440-43-9	Cd	1.36	1.47	0.991-1.98
7440-47-3	Cr	[J]0.73	[ZJ]0.60	0.645-1.68
7439-97-6	Hg	0.047	0.091	0.0138-0.052
7440-02-0	Ni	[Y]8.6	[Y]9.6	6.34-11.4
7439-92-1	Pb	[JY]2.27	[JY]1.80	0.43-2.83
7440-36-0	Sb	[ZJ]1.25	[J]1.42	1.31-2.81
7782-49-2	Se	[ZJ]0.437	[ZJ]0.428	0.46-0.87
7440-28-0	Tl	[U]0.5	[U]0.5	-
7440-62-2	V	[J]0.027	[J]0.030	-
7440-66-6	Zn	5.16	6.12	4.30-6.73

[1] Certified TCLP metals in soil standard from Environmental Resource Associates.
[J] Estimated value. Result was less than the EQL and greater than the MDL.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL.
[Y] Post spike recoveries for nickel and lead were slightly low, 74.7 and 74.9 percent respectively.
\- Certified ranges for thallium and vanadium were not given as part of this standard.
[Z] The results were outside of the acceptance range provided by Environmental Resource Associates.

2.3.2 Preparation Blank Results for TCLP Determinations

TCLP preparation blank results (Table 8) were below the MRQs for all target analytes. The following analytes were detected in the preparation blanks below the MRQs:

- Barium was detected in the preparation blank at 0.0635 mg/L.
- Zinc was detected in the preparation blank at 0.0937 mg/L.
- Beryllium and nickel were detected in the preparation blank below the EQLs.

Table 10. Laboratory Control Sample Results

CAS No.	Analyte	MRQ (mg/L)	[2]Blank (mg/L)	Laboratory Control Sample		
				Concentrations (mg/L)		[1]Recovery
				True	Measured	(%)
7440-22-4	Ag	0.14	[U]6E-3	80.7	79.5	98.5
7440-38-2	As	5	[U]3E-4	2.99E2	3.24E2	108
7440-39-3	Ba	21	6.35E-2	2.26E2	2.34E2	103
7440-41-7	Be	1.22	[J]2.0E-4	3.61E2	3.55E2	98.3
7440-43-9	Cd	0.11	[U]2E-3	5.22E2	4.86E2	93.1
7440-47-3	Cr	0.6	[U]8E-3	1.94E2	1.95E2	101
7439-97-6	Hg	2.5E-2	[U]2E-3	3.28	2.98	90.9
7440-02-0	Ni	11	[JY]7.5E-3	5.98E2	6.02E2	101
7439-92-1	Pb	0.75	[UY]4E-2	3.21E2	3.05E2	95.0
7440-36-0	Sb	1.15	[U]2E-2	3.98E2	3.71E2	93.2
7782-49-2	Se	5.7	[U]1E-3	4.91E2	5.03E2	102
7440-28-0	Tl	0.2	[U]5E-2	4.05E2	3.68E2	90.9
7440-62-2	V	1.6	[U]3E-3	5.88E2	5.92E2	101
7440-66-6	Zn	4.3	9.37E-2	6.86E2	6.78E2	98.8

[1] The specified range for LCS recoveries was between 90 and 110percent.
[J] Estimated value. Result was less than the EQL and greater than the MDL.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL.
[Y] Post spike recoveries for nickel and lead were slightly low, 74.7 and 74.9 percent respectively.

2.3.3 Recoveries Associated with TCLP Determinations

QA specifications were the same for the TCLP determinations as for the metal analytes described in the previous section. MS, MSD, and PS recoveries were acceptable from 75 to 125 percent. LCS acceptable recoveries were from 80 to 120 percent. Serial dilution acceptable recoveries were 90 to 110 percent. LCS recoveries have been included in the following subsections. MS, MSD, PS, and serial dilution recoveries have been included in Appendix B.

2.3.3.1 Laboratory Control Sample, Matrix Spike, and Post Spike Recoveries

All LCS recoveries were between 90 and 110 percent. These results have been included in Table 10. In addition, the TCLP results met the MS, MSD, PS, and RPD specifications for all analytes except the PS for nickel and lead. Nickel and lead recoveries were slightly low, 74.7 and 74.9 percent, respectively. The MS, MSD, PS, and RPD results have been given in Appendix B.

2.3.3.2 Serial Dilution Results for TCLP Analyses

Serial dilution results have also been given in Appendix B. All analyses met the serial dilution specification. As noted in the previous section, serial dilutions apply only to analytes detected at more than ten times the MDL. One of the arsenic recoveries reported in the data package appears to have failed the serial dilution requirement. Although the recovery was calculated and reported, arsenic results were not flagged because the diluted sample was not 10 times the MDL. This is consistent with guidance from SW-846.

2.3.4 Narrative for TCLP Results

Results from the TCLP determinations demonstrated the following:

- All metals were well below the limits for characteristically hazardous material.
- All metals were well below the UTS limits, except thallium, which could not be determined.
- All QA specifications were met, except for PS recoveries for nickel and lead.
- Results from the nonradioactive AZ-102 simulant glass were similar to the AZ-102 glass.
- TCLP standard results indicated a potential low bias to the silver leachate concentrations.

Of the eight metals that are used to determine whether a sample exhibits the characteristics of hazardous (or in this case, mixed) waste, all leachate metal concentrations were well below the characteristically hazardous limits. Only one of these metals, barium, was detected in the leachate at concentrations above the EQLs, and this was well below the characteristic (0.2 percent of the limit).

Similarly, for the metals used to determine whether a sample meets UTS limits, all leachate metal concentrations were shown to be well below the UTS limits, except thallium. Zinc was shown to be present in the leachate at almost half the UTS limit. Zinc was added to the AZ-102 supernate as a major component in one of the glass-formers. As a result, zinc was one of the primary components in the glass and would be expected to be seen in the leachate.

PS recoveries were slightly low for nickel and lead, 74.7 and 74.9 percent respectively. The results have been flagged accordingly in Table 7 through Table 10. Nickel and lead results met all other QA specifications. Nickel and lead concentrations were below the MDLs. For nickel, the MDL was less than 1 percent of the UTS limit. The lead MDL was less than 10 percent of the characteristic limit and about half of the UTS limit for lead. Therefore, although PS recoveries were slightly low, these detection limits were not close to the mixed waste limits.

Results were similar for the AZ-102 radioactive glass and for the nonradioactive simulant glass, except for one of the simulant barium results, which was higher than the other results. For the TCLP standard, silver concentrations in the leachate were significantly lower than the standard acceptance limit. The silver EQL was high enough to be within the standard acceptance range; however, the silver results (~0.1 mg/L) were well below the acceptance range (0.514 to 1.12 mg/L). SW-846 Method 1311 does not require use of a standard.

2.4 CONCENTRATION OF ANION ANALYTES IN AZ-102 GLASS SAMPLES

Results from target anion analytes are presented in this section. For most of the anion analytes, results have been presented for analyses performed on glass samples dissolved by peroxide fusion without the final acid addition that was used for the metals analyses dissolutions. For cyanide analyses, the SW-846 preparation and analysis methods were performed on crushed glass samples.

2.4.1 Results from Anion Analyses

Results have been given in Table 11 and Table 12 for the AZ-102 glass and the simulant glass. These results showed:

- Anion results were consistent with the target composition.
- Only sulfate was detected in the samples at concentrations above the EQLs.
- Sulfate and phosphate results were consistent with metals results for sulfur and phosphorus.
- Cyanide concentrations were well below the regulatory limit.

Table 11. AZ-102 Glass Waste Form Anion Concentrations

CAS No.	Analyte	Sample Results (mg/kg)			Duplicate Results (mg/kg)			[3]Average (mg/kg)
		Result	[1]MDL	[2]EQL	Result	[1]MDL	[2]EQL	
24959-67-9	Br^-	[UY]2E3	7E2	2E3	[UY]2E3	7E2	2E3	[UY]2E3
16887-00-6	Cl^-	[UY]2E3	2E2	2E3	[UY]2E3	2E2	2E3	[UY]2E3
16984-48-8	F^-	[UY]2E3	2E2	2E3	[UY]2E3	2E2	2E3	[UY]2E3
14797-65-0	NO_2^-	[UY]2E3	8E2	2E3	[UY]2E3	9E2	2E3	[UY]2E3
14797-55-8	NO_3^-	[BY]2.97E3	6E2	2E3	[BY]3.92E3	6E2	2E3	[Y]3.4±0.67E3
14265-44-2	PO_4^{3-}	[UY]2E3	8E2	2E3	[UY]2E3	9E2	2E3	[UY]2E3
14808-79-8	SO_4^{2-}	[Y]8.71E3	6E2	2E3	[Y]8.66E3	6E2	2E3	[Y]8.68±0.039E3
57-12-5	CN^-	[U]5E-3	5E-3	5E-2	[U]5E-3	5E-3	5E-2	[U]5E-3

[1] MDLs were from USEPA SW-846 MDL studies using standards in a water matrix. Dilution factors applied.
[2] EQLs were based on lowest concentration standard used for calibration. Dilution factors applied.
[3] Uncertainties are given as one standard deviation between the sample and the duplicate.
[B] Extraction blank concentration: 2.4 x 10^3 mg/L. By the method blank 5 times rule, this result is a nondetect.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL.
[Y] Result are estimates because analyses were performed outside allowable hold times.

Table 12. Simulant and LRM Standard Anion Concentrations

CAS No.	Anion	Simulant Results (mg/kg)				LRM Results (mg/kg)		
		Sample	Duplicate	Average	[1]Target	Sample	Duplicate	[2]Target
24959-67-9	Br^-	[UY]2E3	[UY]2E3	[UY]2E3	-	[UY]2E3	[UY]2E3	-
16887-00-6	Cl^-	[UY]2E3	[UY]2E3	[UY]2E3	-	[UY]2E3	[UY]2E3	7E2
16984-48-8	F^-	[UY]2E3	[UY]2E3	[UY]2E3	1.0E2	[Y]7.32E3	[Y]7.53E3	8.6E3
14797-65-0	NO_2^-	[UY]2E3	[UY]2E3	[UY]2E3	-	[UY]2E3	[UY]2E3	-
14797-55-8	NO_3^-	[Y]5.5E3	[UY]2E3	[Y]5.5E3	-	[UY]2E3	[UY]2E3	-
14265-44-2	PO_4^{3-}	[UY]2E3	[UY]2E3	[UY]2E3	2.6E2	[Y]8.0E3	[Y]1.04E4	7.2E3
14808-79-8	SO_4^{2-}	[Y]9.6E3	[Y]9.9E3	[Y]9.8±0.21E3	9.2E3	[Y]4.50E3	[Y]4.72E3	3.6E3
57-12-5	CN^-	[U]5E-3	[U]5E-3	[U]5E-3	-	[U]5E-3	[U]5E-3	-

[1] Target composition from AZ-102 supernate composition and composition of glass-forming components.
[2] Target composition from multiple-laboratory, round-robin analyses of the standard LRM glass.
[J] Estimated value. Result was less than the EQL and greater than the MDL.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL.
[Y] Results are estimates because analyses were performed outside allowable hold times.
- Indicates an analyte that was only present at trace levels and for which no analytical value was available.

2.4.2 Preparation Blank Results for Anion Determinations

Anion preparation blank results were all nondetects except nitrate which was detected in the SRTC preparation blank at slightly below the EQL. These results have been given in Table 13. For the dissolved glass samples, these results include both SRTC preparation results and BWXS preparation results. The SRTC results were from the reagent blank generated during dissolution of the glass.

Table 13. Blanks and Laboratory Control Sample Recoveries for Anion Analyses

CAS No.	Analyte	MRQ (mg/kg)	Blanks (mg/kg)		Laboratory Control Sample		
			BWXS	SRTC	Concentrations (mg/L)		[1]Recovery
					Added	Measured	(%)
24959-67-9	Br^-	5E3	^{U}48	^{UY}2E3	2.50	2.51	100
16887-00-6	Cl^-	5E3	^{U}48	^{UY}2E3	2.50	2.44	98
16984-48-8	F^-	5E3	^{U}48	^{UY}2E3	2.50	2.52	101
14797-65-0	NO_2^-	5E3	^{U}48	^{UY}2E3	2.50	2.45	98
14797-55-8	NO_3^-	5E3	^{U}48	^{JY}2.4E3	2.50	2.48	99
14265-44-2	PO_4^{3-}	5E3	^{U}48	^{UY}2E3	2.50	2.54	102
14808-79-8	SO_4^{2-}	5E3	^{U}48	^{UY}2E3	2.50	2.42	97
57-12-5	CN^-	30	^{U}5E-3	-	0.204	0.214	105

[1] Acceptable values for the LCS recoveries were from 75 percent to 125 percent.
^{J}Estimated value. Result was less than the EQL and greater than the MDL.
^{U}Undetected. The analyte was not detected. The concentration was less than the MDL.
^{Y}Result are estimates because analyses were performed outside allowable hold times.
- No SRTC dissolution was performed for the samples used in the cyanide determinations.

2.4.3 Recoveries Associated with Anion Determinations

All LCS recoveries were between 90 and 110 percent. These results have been included in Table 13. In addition, results met the MS and RPD specifications for all analytes. These results have been given in Appendix C. Serial dilutions are not called for by the methods used to determine anions. RPDs were determined by performing duplicate analyses on one of the samples. Ion chromatography analyses were determined on one-to-fifty dilutions of the dissolved glass samples.

2.4.4 Narrative for Anion Results

Anion results met all QA and QC specifications except that the holding time limits were not met for the ion chromatography analyses. Cyanide determinations were performed within the specified holding times. Total cyanide concentrations were well below the hazardous limit for amenable cyanide (30 mg/kg) and the limit for total cyanide (590 mg/kg). All anion results for the AZ-102 glass waste form should be considered nondetects except sulfate. Although nitrate was also detected in one of the samples, application of the method blank five times rule would dictate that this result be considered a nondetect at 10 mg/kg.

2.5 CONCLUSIONS

With few exceptions, these results met protocol outlined for regulatory application of characterization data. The AZ-102 glass waste form was shown to meet regulatory limits for a characteristically nonhazardous waste form. Total cyanide concentrations were well below limits on total and amenable cyanide. In addition, TCLP leachate concentrations were below limits for characteristically hazardous waste forms. With the exception of thallium, the AZ-102 glass also met UTS limits. Although one set of thallium results did show that thallium was not present at concentrations high enough to exceed the UTS limits, MS recoveries were outside of specifications for this set of analyses.

As indicated by the total measured oxide content, all primary components (those present at 0.5 weight percent or higher) were reported. In addition, measured concentrations were close to target values, except for chromium, which was slightly high in the AZ-102 glass and the nonradioactive simulant, and for sodium, which was slightly low in the AZ-102 glass. For sodium, the difference between target and measured concentrations appears to have been due to uncertainty in the concentrations measured in the treated supernate used to make the glass. The high chromium value appears to have been due to contamination during the vitrification process.

For all target metal analytes, except silicon, at least one set of measurements met all QA and QC specifications. The quality control (QC) failure associated with the silicon value in the acid-dissolved glass was caused by use of low spike concentrations and was not indicative of a sample-matrix effect. Metal analyte determinations were also performed within all QC requirements except the mercury determinations which were made outside the allowable holding time for the dissolved glass. TCLP results showed no mercury present in the leachate.

For analytes determined in the two different sample sets, results were very similar. For acid and peroxide fusion dissolutions, only arsenic varied by more than the three standard deviations guideline given in the test specification. The difference between the ICP-AES and AA results for arsenic appear to have been due to interference from aluminum in the ICP-AES results. Sulfur as determined by ICP-AES was also consistent with sulfate results as determined by ion chromatography. As expected, sulfate was the only anion detected. A single nitrate result was classified as a nondetect as a result of the method blank 5 times rule.

The QAPjP for this task states, “Completeness will be reported as the percent of all measurements judged to be valid.” The QAPjP also states that acceptance criteria must be established for data quality indictors (including completeness). The test specification for this task set a minimum completeness criteria at 90%. This value was based on standard practice for groundwater samples. Because of the complex matrix associated with the dissolved glass samples, meeting a 90% completeness criteria proved to be difficult.

Deviations of anion holding times are not expected to have compromised date quality, and low nickel and lead post spike recoveries are so close to the allowable values that they are not expected to have been indicative of a QA issue; however under a strict interpretation of USEPA SW-846 protocol, these results could be judged as invalid. Anion determinations were performed outside of holding time specifications. As noted in the validation report,[9] this was not expected to have compromised data quality. Including these results as invalid, this task was performed at a completeness of 79 percent. Nickel and lead post spike recoveries were slightly low (74.7 and 74.9 percent respectively). These measurements met all other QA and QC specifications. Considering these results to be invalid, this task was performed at a completeness of 77 percent.

Future regulatory analysis samples will benefit from three improvements currently being implemented by SRTC and BWXS. First, BWXS is evaluating options for lowering the thallium detection limit without compromising the method's ability to meet recovery specifications. Efforts are currently focusing on pretreatment options for these samples. These improvements are expected to be sufficient to allow for thallium to be determined in future glasses.

Checks are being put into place to ensure that PS concentrations are appropriately adjusted when an MS or MSD failure is caused by a spike concentration that is low compared to the sample concentration. Such an adjustment will allow results such as the acid-dissolved glass silicon value to meet SW-846 protocol and not be designated as invalid.

Finally, a mechanism is being implemented by SRTC and BWXS to improve visibility of samples that need to be given higher priority to avoid exceeding analyte holding times. Such a system would have identified the mercury and anion analyses in sufficient time to have the analyses performed in compliance with all QA specifications.

3.0 REFERENCES

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7. United States of America Environmental Protection Agency, National Contract Laboratory (CLP) Data Review Standard Operating Procedures, Rev. 1.1, Environmental Services Division, Region IV, Athens, GA, June 19, 1992.

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APPENDIX A. METAL ANALYTE QA RESULTS

Table 14. Matrix Spike Results for AZ-102 Acid-Dissolved Glass Metal Analyses

CAS No.	[1]Analyte	Concentrations (μg/L)				Recoveries (%)		
		Added	Sample	Spike	Duplicate	[2]Spike	[2]Duplicate	[3]RPD
7440-22-4	Ag(AA)	5.00	3.87	9.41	9.17	110.8	106.0	2.6
7440-22-4	Ag	1.00E3	1.300E2	9.53E2	9.68E2	82.3	83.8	1.6
7429-90-5	Al	1.00E3	1.750E2	2.654E3	2.683E3	90.4	93.3	1.1
7440-38-2	As(AA)	15.0	[U]0.3	19.08	17.99	[b]127.2	119.9	5.9
7440-38-2	As	1.00E3	1.460E2	1.128E3	1.131E3	98.2	98.5	0.3
7440-42-8	B	N	N	N	N	N	N	N
7440-39-3	Ba	1.00E3	[J]5.50	9.92E2	1.001E3	98.7	99.6	0.9
7440-41-7	Be	1.00E3	[U]0.2	9.74E2	9.86E2	97.4	98.6	1.2
7440-70-2	Ca	1.00E3	2.870E3	3.768E3	3.796E3	89.8	92.6	0.7
7440-43-9	Cd	1.00E3	[U]2	9.13E2	9.16E2	91.3	91.6	0.3
7440-47-3	Cr	1.00E3	5.310E2	1.462E3	1.464E3	93.1	93.3	0.1
7439-89-6	Fe	1.00E3	8.50E2	1.783E3	1.798E3	93.3	94.8	0.8
7439-97-6	Hg	5.00	[UY]0.2	5.12	5.06	102.4	101.2	1.2
7440-09-7	K	1.00E4	[U]2E2	9.76E3	9.85E3	97.6	98.5	0.9
7439-93-2	Li	1.00E3	[U]10	1.004E3	9.81E2	100.4	98.1	2.3
7439-95-4	Mg	1.00E3	4.550E2	1.400E3	1.414E3	94.5	95.9	1.0
7439-98-7	Mo	1.00E3	[U]11	1.068E3	1.043E3	106.8	104.3	2.4
7440-23-5	Na	1.00E3	1.760E3	2.762E3	2.758E3	100.2	99.8	0.1
7440-02-0	Ni	1.00E3	[U]6	9.96E2	1.010E3	99.6	101.0	1.4
7723-14-0	P	2.00E3	[U]15	2.256E3	1.911E3	112.8	95.6	2.1
7439-92-1	Pb(AA)	10.0	[J]0.85	5.21	10.07	[b]43.6	92.2	[c]63.7
7439-92-1	Pb	1.00E3	[U]37	9.20E2	9.18E2	92.0	91.8	0.1
63705-05-5	S	2.00E3	[J]37	2.176E3	2.097E3	107.0	103.0	3.7
7440-36-0	Sb	1.00E3	[U]16	9.51E2	9.46E2	95.1	94.6	0.5
7782-49-2	Se(AA)	15.0	[U]1	15.5	15.9	103.3	105.9	2.5
7782-49-2	Se	1.00E3	[U]31	9.61E2	9.67E2	96.1	96.7	0.6
7440-21-3	Si	5.00E2	1.310E3	1.886E3	1.664E3	115.2	[b]70.8	12.5
7440-32-6	Ti	1.00E3	[J]59.00	1.058E3	1.068E3	99.9	100.9	0.9
7440-28-0	Tl(AA)	15.0	[UR]0.6	0.27	1.54	[b]1.8	[b]10.3	[c]140
7440-28-0	Tl	1.00E3	[U]50	9.74E2	9.50E2	97.4	95.0	2.6
7440-62-2	V	1.00E3	34.00	9.975E2	9.974E2	96.4	96.3	1x10^{-4}
7440-66-6	Zn	1.00E3	2.130E3	3.044E3	3.074E3	91.4	94.4	1.0
7440-67-7	Zr	2.00E3	[U]40	2.256E3	2.162E3	112.8	108.1	4.3

[1] Analyzed by Method 6010B except where "(AA)" indicates results from atomic absorption spectroscopy.

[2] Acceptable values for the matrix spike and matrix spike duplicate recoveries were from 75 to 125 percent.

[3] Acceptable relative percent difference between matrix spike and duplicate recoveries was < 20 percent.

[J] Estimated value. Result was less than the EQL and greater than the MDL.

[R] Atomic absorption thallium results unusable because of extremely low recoveries.

[U] Undetected. The analyte was not detected. The concentration was less than the MDL.

[Y] Result is an estimate because mercury pretreatment performed outside allowable hold times.

[b] Matrix spike or matrix spike duplicate, recovery outside the acceptance range.

[c] The relative percent difference between the MS and MSD was outside the acceptance range.

N Boron values only included for peroxide fusion samples. Boron was introduced during acid dissolution.

Table 15. Matrix Spike Results for AZ-102 Fusion-Dissolved Glass Metal Analyses

CAS No.	[1]Analyte	Concentrations (μg/L)				Recoveries (%)		
		Added	Sample	Spike	Duplicate	[2]Spike	[2]Duplicate	[3]RPD
7440-22-4	Ag(AA)	5.00	12.9	14.01	14.17	[b]22.2	[b]25.4	1.1
7440-22-4	Ag	5.00E2	62.40	4.27E2	4.13E2	[b]72.9	[b]70.0	3.4
7429-90-5	Al	1.00E3	1.640E3	2.60E3	2.59E3	96.0	94.7	0.5
7440-38-2	As(AA)	15.0	[U]0.3	20.2	22.7	[b]134.6	[b]151.7	11.9
7440-38-2	As	5.00E2	1.44E2	6.689E2	6.652E2	105	104.2	0.6
7440-42-8	B	1.00E3	34.6	1.023E3	1.037E3	98.8	100.2	1.4
7440-39-3	Ba	1.00E3	[J]5.2	9.79E2	9.69E2	97.4	96.3	1.1
7440-41-7	Be	1.00E3	[U]0.2	9.910E2	9.823E2	99.1	98.2	0.9
7440-70-2	Ca	1.00E3	2.74E3	3.686E3	3.699E3	94.6	95.9	0.4
7440-43-9	Cd	5.00E2	[U]2	4.781E2	4.848E2	95.6	97.0	1.4
7440-47-3	Cr	5.00E2	2.60E2	7.296E2	7.197E2	93.9	91.9	1.4
7439-89-6	Fe	1.00E3	7.75E2	1.737E3	1.734E3	92.6	95.9	0.2
7439-97-6	Hg	5	[U]0.2	5.12	5.06	102.4	101.2	1.2
7440-09-7	K	1.00E4	[J]2.07E2	9.642E3	9.652E3	94.4	94.5	0.1
7439-93-2	Li	1.00E3	10.9	1.035E3	1.047E3	103.5	104.7	1.2
7439-95-4	Mg	1.00E3	4.47E2	1.373E3	1.375E3	92.6	92.8	0.1
7439-98-7	Mo	1.00E3	[U]11	9.96E2	1.028E3	99.6	102.8	3.1
7440-23-5	Na	N	N	N	N	N	N	N
7440-02-0	Ni	N	N	N	N	N	N	N
7723-14-0	P	2.00E3	[U]15	2.024E3	1.989E3	101.2	99.5	1.7
7439-92-1	Pb(AA)	10	[J]1.28	10.23	10.55	89.5	92.7	3.1
7439-92-1	Pb	5.00E2	[J]54.1	5.28E2	5.40E2	94.7	97.2	2.4
63705-05-5	S	2.00E3	40.3	2.084E3	2.122E3	102.2	104.1	1.8
7440-36-0	Sb	5.00E2	[U]16	3.94E2	4.48E2	78.8	89.6	12.9
7782-49-2	Se(AA)	15	[J]1.1	12.3	13.9	82	92.9	12.5
7782-49-2	Se	5.00E2	[U]31	5.12E2	4.90E2	102.3	98.0	4.3
7440-21-3	Si	5.00E2	1.7E3	1.959E3	7.916E2	[b]43.8	Z	[c]84.9
7440-32-6	Ti	1.00E3	[J]68.4	1.101E3	1.111E3	103.3	104.3	0.9
7440-28-0	Tl(AA)	15	[U]0.6	9.5	11.2	[b]63.2	[b]74.5	16.5
7440-28-0	Tl	5.00E2	[U]50	4.68E2	4.85E2	93.6	96.9	3.5
7440-62-2	V	5.00E2	[J]18.8	4.861E2	4.796E2	93.5	92.2	1.3
7440-66-6	Zn	1.00E3	2.00E3	2.965E3	2.963E3	96.5	96.3	0.1
7440-67-7	Zr	2.00E3	42.1	2.119E3	2.225E3	106	111.3	4.9

[1] Analyzed by Method 6010B except where "(AA)" indicates results from atomic absorption spectroscopy.

[2] Acceptable values for the matrix spike and matrix spike duplicate recoveries were from 75 to 125 percent.

[3] Acceptable relative percent difference between matrix spike and duplicate recoveries was < 20 percent.

[J] Estimated value. Result was less than the EQL and greater than the MDL.

[R] Atomic absorption thallium results unusable because of very low recoveries for acid dissolved samples.

[U] Undetected. The analyte was not detected. The concentration was less than the MDL.

[Y] Result is an estimate because mercury pretreatment performed outside allowable hold times.

[b] Matrix spike or matrix spike duplicate, recovery outside the acceptance range.

[c] The relative percent difference between the MS and MSD was outside the acceptance range.

N Only acid values reported for sodium and nickel. These metals are introduced during fusion dissolution.

Z This recovery was included because the spiked sample result was so low, the recovery would be < 0.

Table 16. Post Spike Results for AZ-102 Glass Sample Metal Analyses

CAS No.	[1]Analyte	Concentrations (μg/L)					[2]Recoveries (%)	
		Added	Acid		Fusion		Acid	Fusion
			Sample	Spike	Sample	Spike		
7440-22-4	Ag(AA)	5.0	3.87	9.00	12.9	14	96.2	[b]15
7440-22-4	Ag	50.0	1.300E2	1.86E2	62.40	1.10E2	112.2	90.8
7429-90-5	Al	1.00E3	1.750E2	2.76E3	1.640E3	2.648E3	101.4	100.8
7440-38-2	As(AA)	15	[U]0.3	20	[U]0.3	24	[b]130.7	[b]161
7440-38-2	As	2.00E3	1.460E2	2.39E3	1.44E2	2.555E3	112.3	122.3
7440-42-8	B	1.00E3	N	[3]N	34.6	9.31E2	N	89.7
7440-39-3	Ba	1.00E3	[J]5.50	1.02E3	[J]5.2	1.009E3	101.3	100.4
7440-41-7	Be	1.00E3	[U]0.2	1.01E3	[U]0.2	1.010E3	101.4	101
7440-70-2	Ca	1.00E3	2.870E3	3.88E3	2.74E3	3.742E3	100.9	100.2
7440-43-9	Cd	50	[U]2	50	[U]2	63	100.6	121.8
7440-47-3	Cr	2.00E2	5.310E2	7.55E2	2.60E2	4.89E2	108.9	109.8
7439-89-6	Fe	1.00E3	8.50E2	1.83E3	7.75E2	1.755E3	97.9	98
7439-97-6	Hg	5	[UY]0.2	-	[UY]0.2	5.67	-	113.4
7440-09-7	K	1.00E4	[U]2E2	1.00E4	[J]2.07E2	9.993E3	100.2	97.9
7439-93-2	Li	1.00E3	[U]10	8.62E2	10.9	9.37E2	86.2	93.7
7439-95-4	Mg	1.00E3	4.550E2	1.45E3	4.47E2	1.401E3	99.2	95.4
7439-98-7	Mo	1.00E3	[U]11	9.04E2	[U]11	9.40E2	90.4	94
7440-23-5	Na	1.00E3	1.760E3	2.76E3	N	2.186E4	99.8	N
7440-02-0	Ni	1.00E3	[U]6	1.04E3	N	2.103E3	104.2	N
7723-14-0	P	1.00E3	[U]15	89.9E2	[U]15	8.91E2	89.9	89.1
7439-92-1	Pb(AA)	10.0	[J]0.85	10.0	[J]1.28	11	92	100.3
7439-92-1	Pb	5.00E2	[U]37	5.05E2	[J]54.1	6.13E2	100.9	112.6
63705-05-5	S	1.00E3	[J]37	9.66E2	40.3	1.008E3	91.9	96.8
7440-36-0	Sb	5.00E2	[U]16	5.29E2	[U]16	5.65E2	105.9	113
7782-49-2	Se(AA)	15	[U]1	16	[J]1.1	13	109.1	88.5
7782-49-2	Se	2.00E3	[U]31	2.19E3	[U]31	2.343E3	109.7	117.2
7440-21-3	Si	5.00E2	1.310E3	1.60E3	1.7E3	1.917E3	[b]58.6	[b]35.4
7440-32-6	Ti	1.00E3	[J]59.00	1.11E3	[J]68.4	1.122E3	105.4	105.4
7440-28-0	Tl(AA)	15	[UR]0.6	3.00	[U]0.6	11	[b]16.7	[b]71.7
7440-28-0	Tl	2.00E3	[U]50	2.13E3	[U]50	2.285E3	106.7	114.3
7440-62-2	V	5.00E2	34.00	5.88E2	[J]18.8	5.46E2	110.3	105.9
7440-66-6	Zn	1.00E3	2.130E3	3.10E3	2.00E3	2.999E3	97.2	99.9
7440-67-7	Zr	5.00E2	[U]40	5.07E2	42.1	4.53E2	101.4	90.7

[1] Analyzed by Method 6010B except where "(AA)" indicates results from atomic absorption spectroscopy.

[2] Acceptable values for the post spike recoveries were from 75 percent to 125 percent.

[3] No boron spike was added to the acid-dissolved samples. No boron analyses performed for these samples.

[J] Estimated value. Result was less than the EQL and greater than the MDL.

[R] Atomic absorption thallium results unusable because of very low recoveries for acid-dissolved samples.

[U] Undetected. The analyte was not detected. The concentration was less than the MDL.

[Y] Result is an estimate because mercury pretreatment performed outside allowable hold times.

[b] Matrix spike or matrix spike duplicate, recovery outside the acceptance range.

[c] The relative percent difference between the MS and MSD was outside the acceptance range.

N Boron results only included for fusion samples. Sodium and nickel results only for acid samples.

\- Mercury post spike recovery not performed on acid dissolution.

Table 17. Serial Dilution Results for AZ-102 Glass Sample Metal Analyses

CAS No.	[1]Analyte	Concentrations (µg/L)				[2]Recoveries (%)	
		Acid		Fusion		Acid	Fusion
		Sample	Diluted	Sample	Diluted		
7440-22-4	Ag(AA)	3.9	[J]5.1	12.9	[J]9.5	-	-
7440-22-4	Ag	1.30E2	1.28E2	64.7	65.0	1.5	0.5
7429-90-5	Al	1.75E3	1.62E3	1.64E3	[J]1.59E3	7.4	-
7440-38-2	As(AA)	[U]0.3	[U]1	[U]0.3	[U]1	-	-
7440-38-2	As	1.48E2	[J]1.19E2	[J]1.09E2	[J]80	-	-
7440-42-8	B	[N]5.69E3	[N]5.62E3	[J]35	[J]1.1E2	1.2	-
7440-39-3	Ba	[J]5.5	[U]8	[J]5.2	[U]8	-	-
7440-41-7	Be	[U]0.2	[U]1	[U]0.2	[U]1	-	-
7440-70-2	Ca	2.87E3	[J]2.93E3	2.74E3	[J]2.82E3	-	-
7440-43-9	Cd	[U]2	[U]9	[J]2.5	[U]9	-	-
7440-47-3	Cr	5.37E2	5.45E2	2.69E2	[J]2.65E2	1.5	-
7439-89-6	Fe	8.50E2	8.48E2	7.75E2	7.93E2	0.2	2.3
7440-09-7	K	[U]2E2	[U]9E2	[J]2.1E2	[U]9E2	-	-
7439-93-2	Li	[U]10	[U]49	[U]10	[U]49	-	-
7439-95-4	Mg	4.6E2	[J]5.6E2	4.47E2	[J]4.90E2	-	-
7439-98-7	Mo	[U]11	[U]55	[U]11	[U]55	-	-
7440-23-5	Na	1.76E3	[J]1.82E2	[N]1.38E3	[N]1.35E3	-	2.2
7440-02-0	Ni	[U]6	[U]32	[UN]6	[UN]32	-	-
7723-14-0	P	[U]15	[U]75	[U]15	[U]75	-	-
7439-92-1	Pb(AA)	[J]0.85	[J]13	[J]1.3	[J]11	-	-
7439-92-1	Pb	[U]37	[U]2E2	[J]50	[U]2E2	-	-
63705-05-5	S	[J]37	[J]1.1E2	[J]40	[J]1.6E2	-	-
7440-36-0	Sb	[U]16	[U]78	[U]16	[U]78	-	-
7782-49-2	Se(AA)	[U]1	[U]5	[U]1	[J]8.6	-	-
7782-49-2	Se	[U]31	[U]2E2	[U]31	[U]2E2	-	-
7440-21-3	Si	1.31E3	[J]1.35E3	1.74E3	1.70E3	-	2.3
7440-32-6	Ti	[J]59	[U]50	[J]68	[U]50	-	-
7440-28-0	Tl(AA)	[U]0.6	[U]3	[U]0.6	[U]3	-	-
7440-28-0	Tl	[U]50	[U]2.5E2	[U]50	[U]2.5E2	-	-
7440-62-2	V	36.4	33.0	[J]17	[U]13	9.3	-
7440-66-6	Zn	2.13E3	2.12E3	2.00E3	2.03E3	0.5	1.5
7440-67-7	Zr	[U]42	[U]2E2	[U]42	[U]2E2	-	-

[1] Analyzed by Method 6010B except where "(AA)" indicates results from atomic absorption spectroscopy.

[2] Acceptable values for serial dilution recoveries were less than 10 percent.

[J] Estimated value. Result was less than the EQL and greater than the MDL.

[U] Undetected. The analyte was not detected. The concentration was less than the MDL.

[N] Sample result not given in the report because of incompatability with the sample preparation method.

- Serial dilution not applicable since analyte not present in the diluted sample at above 10 x the MDL.

APPENDIX B. TCLP ANALYTE QA RESULTS

Table 18. Matrix Spike Results for AZ-102 TCLP Analyses

CAS No.	Analyte	Concentrations (μg/L)				Recoveries (%)		
		Added	Sample	Spike	Duplicate	[1]Spike	[1]Duplicate	[2]RPD
7440-22-4	Ag	1.00E3	[J]11.9	9.89E2	9.88E2	97.7	97.6	0.1
7440-38-2	As	15.0	8.68	24.25	24.31	103.8	104.2	0.2
7440-39-3	Ba	1.00E3	1430	2.666E3	2.618E3	123.6	118.8	1.8
7440-41-7	Be	1.00E3	74.1	1.024E3	1.021E3	95	94.7	0.3
7440-43-9	Cd	1.00E3	1.36E2	1.073E3	1.088E3	93.7	95.2	1.4
7440-47-3	Cr	1.00E3	[J]72.7	1.036E3	1.040E3	96.3	96.7	0.4
7439-97-6	Hg	5.00	[U]2	4.29	4.53	85.8	90.6	5.44
7439-97-6	Hg	5.00	[U]2	5.22	5.19	104.4	103.8	0.58
7440-02-0	Ni	1.00E3	8.59E2	1.893E3	1.900E3	103.4	104.1	0.4
7439-92-1	Pb	1.00E3	[J]2.27E2	1.093E3	1.103E3	86.6	87.6	0.9
7440-36-0	Sb	1.00E3	[J]1.25E2	1.125E3	1.133E3	100	100.8	0.7
7782-49-2	Se	15.0	[J]3.93	19.05	18.7	100.8	98.5	1.9
7440-28-0	Tl	1.00E3	[U]50	9.51E2	9.331E2	95.1	93.3	1.9
7440-62-2	V	1.00E3	[J]2.7	1.004E3	1.008E3	100.1	100.5	0.4
7440-66-6	Zn	1.00E3	5.16E2	1.546E3	1.548E3	103	103.2	0.1

[1] Acceptable values for the matrix spike and matrix spike duplicate recoveries were from 75 to 125 percent.
[2] Acceptable relative percent difference between matrix spike and duplicate recoveries was < 20 percent.
[J] Estimated value. Result was less than the EQL and greater than the MDL.
[U] Undetected. The analyte was not detected. The concentration was less than the MDL.

Table 19. Post Spike and Serial Dilution Results for AZ-102 Glass TCLP

CAS No.	Analyte	Concentrations (µg/L)					Recovery (%)	
		Post Spike			Serial Dilution		Post Spike	Dilution
		Added	Sample	Spike	Sample	Diluted		
7440-36-0	Sb	1.00E3	[J]1.25E2	1.02E3	[J]1.25E2	[J]1.84E2	89.1	-
7440-38-2	As	1.50E2	8.7	1.58E2	8.7	[J]5.9	99.4	-
7440-39-3	Ba	1.00E3	1.43E3	2.38E3	1.43E3	1.61E3	94.9	12.6
7440-41-7	Be	1.00E3	143E3	9.93E2	143E3	79.0	94.9	6.6
7440-43-9	Cd	1.00E3	1.36E2	9.61E2	1.36E2	1.28E2	82.5	5.9
7440-47-3	Cr	1.00E3	[J]72.7	9.83E2	[J]72.7	[J]80.0	91	-
7439-92-1	Pb	1.00E3	[J]2.27E2	9.76E2	[J]2.27E2	[J]2.38E2	[b]74.9	-
7440-02-0	Ni	1.00E3	8.59E2	1.6E3	8.59E2	8.91E2	[b]74.7	3.7
7782-49-2	Se	1.50E2	[J]3.9	1.66E2	[J]3.9	[J]8.3	107.9	-
7440-22-4	Ag	1.00E3	[J]11.9	8.67E2	[J]11.9	[J]34.0	85.5	-
7440-28-0	Tl	1.00E3	[U]50	8.35E2	[U]50	[U]2.5E2	83.5	-
7440-62-2	V	1.00E3	[J]2.7	9.62E2	[J]2.7	[J]13	96	-
7440-66-6	Zn	1.00E3	5.16E2	1.306E3	5.16E2	5.35E2	79	3.7

[J] Estimated value. Result was less than the EQL and greater than the MDL.

[U] Undetected. The analyte was not detected. The concentration was less than the MDL.

- Serial dilution not applicable since analyte not present in the diluted sample at above 10 x the MDL.

[b] Post spike recovery outside the acceptance range.

APPENDIX C. ANION ANALYTE QA RESULTS

Table 20. Matrix Spike and Duplicate Analysis Results for AZ-102 Anion Analytes

CAS No.	Analyte	Matrix Spike				Duplicate Analysis	
		Concentrations (μg/L)			[1]Recovery (%)	Concentration (μg/L)	[2]RPD
		Added	Sample	Spike			
24959-67-9	Br^-	1.00E2	[U]13	1.18E2	1.18E2	[U]13	-
16887-00-6	Cl^-	1.00E2	[U]13	1.16E2	1.16E2	[U]13	-
16984-48-8	F^-	1.00E2	[U]13	91	91	[U]13	-
14797-65-0	NO_2^-	1.00E2	[U]13	1.09E2	1.09E2	[U]13	-
14797-55-8	NO_3^-	1.00E2	[J]12.1	1.21E2	1.09E2	[J]12.8	5.6
14265-44-2	PO_4^{3-}	1.00E2	[U]13	96.2	96	[U]13	-
14808-79-8	SO_4^{2-}	1.00E2	[U]13	1.03E2	1.03E2	[U]13	-
57-12-5	CN^-	5.0E-2	[U]5E-3	4.7E-2	94	[Y]5.0E-2	7.2

[1] Acceptable values for the matrix spike recoveries were from 75 percent to 125 percent.

[2] Acceptable relative percent differences between the sample and duplicate were less than 20 percent.

[J] Estimated value. Result was less than the EQL and greater than the MDL.

[U] Undetected. The analyte was not detected. The concentration was less than the MDL.

[Y] This value is for the matrix spike duplicate. The matrix spike duplicate recovery was 101 percent.

-Indicates a value that was not available. RPD values were not available for analytes below the MDL.

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