FINAL REPORT FOR CRUCIBLE-SCALE RADIOACTIVE VITRIFICATION AND PRODUCT TESTING OF WASTE ENVELOPE B (AZ-102) LOW-ACTIVITY WASTE GLASS (U)

APRIL 2004

SAVANNAH RIVER TECHNOLOGY CENTER



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LIST OF ACRONYMS

AAS	Atomic Absorption Spectroscopy
ADS	Analytical Development Section
ARM	Analytical Reference Material
ASTM	American Society for Testing and Materials
BNI	Bechtel National Incorporated
BSE	Backscattered electron
CCC	Container Centerline Cooling
CUA	Catholic University of America
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
LAW	Low Activity Waste
LRM	Low Activity Reference Material
РСТ	Product Consistency Test
ppm	parts per million
QA	Quality Assurance
RPP	River Protection Project
SE	Secondary electron
SEM	Scanning Electron Microscopy
SRS	Savannah River Site
SRTC	Savannah River Technology Center
TCLP	Toxicity Characteristic Leaching Procedure
TRU	Transuranics – defined per WTP Contract as alpha-emitting radionuclides with an atomic number greater than 92 with half-life greater than 10 years
VSL	Vitreous State Laboratory
WTP	Hanford Tank Waste Treatment and Immobilization Plant
XRD	X-ray Diffraction

ABSTRACT

A proof-of-technology demonstration for the Hanford River Protection Project (RPP) Waste Treatment and Immobilization Plant (WTP) was performed by the Savannah River Technology Center (SRTC). As part of this demonstration, treated AZ-102 Low-Activity Waste supernate was vitrified using a crucible-scale furnace. Initial glass samples were quench-cooled and characterized for metals and radionuclides. The glass was also durabilitytested using the American Society for Testing and Materials (ASTM) Product Consistency Test (PCT) protocol. These tests used the AZ-102 glass formulation Low Activity Waste (LAW) B88 that targeted AZ-102 waste loading at 5 wt% Na₂O. After these initial results were obtained with the quench-cooled LAWB88 glass, a prototypical container centerline cooling (CCC) program was supplied to SRTC by WTP. A portion of the quench-cooled LAWB88 glass was remelted and centerline cooled. Samples from the CCC low-activity AZ-102 glass waste form were durability tested using the PCT and characterized for crystalline phase identification.

This final report documents the characterization and durability of this AZ-102 glass. Previous crucible-scale vitrification testing with pretreated AZ-102 at the SRTC had resulted in a product LAW glass with crystalline pyroxene surface material. That previous testing used a glass formulation LAWB53. The presence of crystals using the LAWB53 formulation was classified as a new discovery work scope that led to the present carryover work performed by SRTC for WTP. Thus, another significant goal of this present work was to investigate the influence of reformulating the glass recipe on crystalline formation in the glass. The original LAWB53 was reformulated to preclude crystalline formulation. The reformulation consisted of changing the relative amounts of the various glass forming minerals added to the pretreated AZ-102 supernatant.

Quench-cooled glass samples were 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 and radionuclides in the glass. The sum of metal oxides from both acid dissolution and peroxide fusion dissolution of the AZ-102 LAW glasses indicated all major constituents (those at or above 0.5 weight percent) were determined. Results were typically within ten percent of the target for the AZ-102 glasses and for a Low-Activity Reference Material (LRM) glass. Measured densities for the AZ-102 glass was approximately 2.6 g/cc and the measured density of the LRM glass was approximately 2.5 g/cc compared to a reference value of 2.51 g/cc.

Radionuclides measured in the quench-cooled AZ-102 glass were close to target values calculated from measured radionuclide specific activities in the treated feed. The activities for Cs-137, Sr-90, and Tc-99 were all below the specified BNI-WTP contract upper limits of 3.0 Ci/m³, 20 Ci/m³, and 0.1 Ci/m³, respectively, for LAW glass. The transuranics (TRU) measured in the AZ-102 glass were also well below the upper limit of 100 nCi/g for LAW glass.

PCT results for the AZ-102 quenched glass and container centerline cooled glass indicate that normalized releases for B, Si, and Na are all below the BNI-WTP contract upper limits for normalized release of 2 g/m². The PCT results for the LRM glass also matched previous data for leach testing of that glass. The average normalized releases from the three different AZ-102 glasses (surrogate quenched, rad quenched, and rad CCC) were shown to be statistically different, but these differences are relatively small. Thus the normalized releases from the three different AZ-102 glasses are consistent in that they are similar to within a factor of \pm 20% for B, Si, and Na.

No crystalline phase was observed in the product AZ-102 LAW quenched or container centerline cooled glasses made from the LAWB88 formulation in this study.

1.0 TESTING SUMMARY

The task addressed in this report is part of continuation work on a proof-of-technology demonstration performed by the Savannah River Technology Center (SRTC) for Bechtel National, Inc. (BNI). In the initial demonstration performed by SRTC for the WTP Contractor prior to BNI, 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 per an original glass formulation LAWB53, and vitrified and cooled by a heat treatment cooling curve supplied by the WTP Contractor prior to BNI. The initial glass product contained crystals on the bottom glass surface. Since glass crystallization can influence glass durability, it was decided to stop all product testing on the glass with crystallized surface. Reference #1 (issued to WTP Contractor BNI in March of 2002) describes in detail the efforts with surrogate AZ-102 supernatants and crucible-scale vitrification studies that led to the reformulated glass recipe.

The AZ-102 glass was reformulated to prevent formation of surface crystals (LAWB88) by altering the relative amounts of glass forming minerals used in the glass recipe. A second batch of AZ-102 LAW glass was produced and rapidly cooled, or quench-cooled, in late 2002.^{2,3,4} SRTC was directed to perform the crucible vitrification using quench cooling at that time by WTP, since an approved WTP cooling curve (as prescribed by the Test Specification³ and Task Plan⁴ for these crucible vitrification studies) was not available at that time. The reformulated, rapidly cooled glass was characterized for metals and radionuclides and also leach-tested per the PCT. After this testing on the quench-cooled LAWB88 glass was completed in early 2003, a centerline container cooling curve was supplied to SRTC by WTP in the fall of 2003. A portion of the quenched LAWB88 glass was remelted and cooled per the container centerline cooling curve. This glass was also durability tested per the PCT and the powdered glass was analyzed for crystalline phases. Regulatory analyses results from the initial testing on the LAWB88, rapidly cooled AZ-102 glass have also been specified⁵, planned,⁶ and reported.⁷

The scope of the task described in the following report was to produce a LAWB88 glass with pretreated AZ-102, characterize the glass product for limited metals and radionuclides, and to test and report the glass waste form durability testing (waste form leachability) per the Product Consistency Test (PCT). Another goal of this testing was to investigate the influence of container centerline cooling on both the PCT and potential crystalline phase formation. A separate WSRC technical document reports the rheology and physical properties of the AZ-102 melter feed slurries⁸, that were originally specified in the AZ-102 Scope-62 Vitrification and Product Testing Test Specification issued by WTP.³

1.1 OBJECTIVES

The goals of this task were to produce a reformulated AZ-102 LAW glass that was cooled per a prototypical container centerline cooling. The product centerline cooled glass was leach tested using the PCT and analyzed for crystalline phases that could have formed due to the centerline cooling. Original planning for the AZ-102 LAW crucible glass studies involved testing on container-cooled glasses.^{3,4} However the prototypical container cooling curves were not available when the AZ-102 reformulation LAWB88 glass recipe was completed in 2002. Therefore the AZ-102 reformulation recipe was used to initially produce quenched crucible-scale glass in August/September of 2002 for characterization and durability testing. A WTP-approved schedule and budget change request was later issued to perform the container centerline cooled glass PCT and crystalline analysis in fall 2003.⁹

The test objectives of the current task involving crucible-scale vitrification of pretreated, radioactive AZ-102 supernatant as described in Reference #3, Section 3, 'Objectives' and in Reference 4, Section I-A, 'Task Definition' are listed below. Each of the six objectives was met. Note that the sixth objective pertains to rheology and physical properties testing that has been reported in a separate SRTC report in Reference 8.

Test Objective	Objective Met (Y/N)	Discussion
1. Generate a glass product for subsequent product testing	Yes	Reformulated LAWB88 AZ-102 glass that was originally made with quench cooling was remelted and subjected to a prototypical centerline cooling. (Sections 2.1.1 and 2.1.2)
2. Chemical and radionuclide reporting	Yes	Dissolution and characterization (Section 2.1.3) was performed to target limited metals present at >0.5 wt% (Section 2.2) and to target specified radionuclides (Section 2.4).
3. Waste loading	Yes	The analyzed sodium waste loading in the glass of 4.5 wt% Na ₂ O was within 10% of the target waste loading of 5 wt% Na ₂ O (Section 2.2).
4. Identification and quantification of crystalline and non-crystalline phases	Yes	No crystalline phases determined in the centerline cooled glasses (Section 2.7).

Test Objective	Objective Met (Y/N)	Discussion				
5. Waste form leachability	Yes	Normalized release rates for all analytes measured for PCT on quenched and CCC glasses were below the WTP Contract Specification of 2 g/m ² (Section 2.5).				
6. Perform rheology testing on AZ-102 evaporator melter feed slurry	Yes	Rheology and physical properties testing has been previously reported in Reference 8.				

1.2 TEST EXCEPTIONS

List Test Exceptions	Describe Test Exceptions				
Test Exception Number: 24590-WTP-TEF-RT-03-005	Test Exception pertained to rheological and physical properties measurements as described in Reference #8 and did not apply to tests described in this report.				

1.3 RESULTS AND PERFORMANCE AGAINST SUCCESS CRITERIA

If required by the test specification, this section must compare test results to expected plant conditions.

List Success Criteria	Explain How the Tests Did or Did Not Meet the Success Criteria
1. Identification and quantification of those	Chemical constituents present at
chemical constituents present at	concentrations greater than 0.5 wt% were
concentrations greater than 0.5 wt%	identified and quantified as shown in Table 7
(elemental basis)	through Table 10 of Section 2.2.

List Success Criteria	Explain How the Tests Did or Did Not Meet the Success Criteria					
2. The concentrations of ¹³⁷ Cs, ⁹⁰ Sr, ⁹⁹ Tc, and transuranic (TRU) radionuclides shall be less than 3.0 Ci/m ³ , 20 Ci/m ³ , 0.1 Ci/m ³ , and 100 nCi/g, respectively, and the sum of fractions of radionuclide concentrations shall be less than 1.0 when compared to class C limits of 10 CFR 61.55.	From Section 2.4, the concentrations of ¹³⁷ Cs, ⁹⁰ Sr, ⁹⁹ Tc, and transuranic (TRU) radionuclides were measured at less than 3.0 Ci/m ³ for Cs-137 (measured = 0.98 Ci/m ³); less than 20 Ci/m ³ for Sr-90 (measured = 1.2 Ci/m ³); less than 0.1 Ci/m ³ for Tc-99 (measured = $< 2.7E-3$ Ci/m ³); and less than 100 nCi/g, for transuranics (measured total alpha =2.3 nCi/g) and the sum of fractions of radionuclide concentrations was less than 1.0 (measured = $4.8E-4$)when compared to class C limits of 10 CFR 61.55.					
3. The concentration of waste sodium oxide for Envelope B in the LAW glass shall be greater than 5 wt% as Na ₂ O.	The measured sodium in the AZ-102 waste glass was 4.5 wt% as Na ₂ O vs. the target of 5 wt% as detailed in Section 2.2. This measured waste loading is within 10% of the target. However, this criteria of demonstrating waste loading in the LAWB88 glass of greater than 5 wt% Na ₂ O was not possible since the glass formulation recipe targeted only 5 wt% Na ₂ O, i.e., the recipe did not target sodium waste loading at greater than 5 wt% as Na ₂ O.					
4. Identification and quantification of crystalline and non-crystalline phases.	Analyses for crystalline phases using X-ray diffraction and Scanning Electron Microscopy indicated no presence of crystalline phases (Section 2.7). Thus the entire crucible-scale glass is an amorphous phase with the chemical composition as reported in Section 2.2.					
5. Rheograms, yield stress measurements, and viscosity for the AZ-102 evaporator melter feed slurry.	The rheograms, yield stress measurements, and viscosity for the AZ-102 evaporator melter feed slurry have been previously reported in Reference 8.					

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 WTP⁴. Specific information regarding the compliance of the SRTC QA program with NQA-1 1989, Part 1, Basic and Supplementary Requirements and NQA-2a 1990, Subpart 2.7 is contained in these matrices. This scope of work involves only immobilized low activity waste (ILAW) and thus the RW-0333P QA associated with high level waste does not apply.

1.5 R&T TEST CONDITIONS

List R&T Test Conditions	Were Test Conditions Followed?
1. Glass Fabrication	Heat Treatment: Initial crucible vitrification did not use prescribed heat treatment as requested in the Test Specification because the WTP-prescribed heat treatment was not available when the LAWB88 formulation was originally provided. Thus all initial crucible vitrification was performed with rapid cooling per direction from WTP (Section 2.1.2). The quenched crucible scale glass was then used for metals and radionuclide characterization, and PCT. After the WTP prescribed heat treatment was made available to SRTC, a later remelt of the original glass was performed with heat treatment (Section 2.1.2). This glass was then used for additional PCT (Section 2.5) and analyzed for crystalline phases (Section 2.7).
	Glass Formulation: SRTC provided to WTP/VSL the characterization data for pretreated radioactive AZ-102 sample. The glass former mixture composition and glass former minerals to be used were then provided by WTP/VSL to SRTC with concurrence (Section 2.1.1).

The Test Specification established conditions to ensure that results are valid for project needs. The conditions were followed in this work

List R&T Test Conditions	Were Test Conditions Followed?					
2. Chemical Composition	Yes – Quench-cooled crucible scale glasses were dissolved by both acid dissolution and peroxide fusion per ASTM procedure with noted exceptions pertaining to concentrations of actual powdered glass dissolved in solution (Section 2.1.3). The necessary replicate samples were analyzed using ICP-AES in order to meet the purposes prescribed in the Test Specification pertaining to identification and quantification of chemical constituents > 0.5 wt%, confirmation of waste loading in the glass (sodium content of 5 wt% as Na ₂ O) (Section 2.2), and confirmation of the main radionuclide concentrations in the glass (Cs-137, Sr-90, Tc-99 and transuranics) (Section 2.4).					
3. Radiochemical Composition	Yes – All prescribed radiochemical analyses were performed (Section 2.4) with necessary instrument detection limits to meet the Success Criteria described in Section 1.3.					
4. Product Consistency Test (PCT)	Yes – PCT was performed to ASTM 1285-97 protocol as prescribed in Test Specification. Boron, silicon, and sodium concentrations were determined (Section 2.5) to levels that allowed determination of compliance with contract specification 2.2.2.17.3.					
5. Crystalline and Non- Crystalline Phase Determination	Yes – Crystalline and non-crystalline phases were measured using x-ray diffraction (XRD) and scanning electron microscopy (SEM) on representative powdered-glass samples that were subjected to the prototypical centerline cooling (Section 2.7).					
6. Rheology Testing	Yes – Rheology and physical properties testing has been previously reported in Reference #8. These measurements were performed to the conditions specified in the Test Specification ³ and the subsequent Test Exception 24590-WTP-TEF-RT- 03-005 pertaining only to rheology and physical properties testing.					

1.6 SIMULANT USE

A simulant was produced as part of the original crucible-scale studies for pretreated AZ-102 supernatant.¹ The simulant AZ-102 pretreated supernatant was used to make a simulant glass and was analyzed in parallel with the actual radioactive AZ-102 glass in the quench-cooling studies that used the reformulation LAWB88. The simulant glass was also durability tested in parallel with the radioactive AZ-102 glass. All of these results were produced from the quench-cooled glasses. No simulant glass was tested in the later study on durability testing and crystalline phase measurements on the container-cooled radioactive AZ-102 glass. The following report discusses the comparison of quench-cooled simulant glass analysis vs. the quench-cooled radioactive AZ-102 glass. Also the quench-cooled durability test results for the simulant and radioactive AZ-102 glass are compared to similar durability test results for the centerline-cooled radioactive AZ-102 glass.

1.7 DISCREPANCIES AND FOLLOW-ON TESTS

No discrepancies were identified, and no follow-on work is planned from this study.

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2.0 DISCUSSION

The purpose of this task was to vitrify a treated AZ-102 supernatant using a reformulated recipe developed by the Vitreous State Laboratory (VSL) at the Catholic University of America (CUA) for WTP. Initial attempts at vitrifying the pretreated AZ-102 had resulted in a glass product with a crystalline surface phase.¹ The WTP R&T Scoping Statement S-62 indicated "Demonstrate contract ILAW small crucible scale vitrification of Envelope B, Tank AZ-102."² This scope of work was designated as carryover work from previous testing that involved a prior Contractor before BNI and WGI were involved in the WTP Contract. WTP personnel issued a Test Specification for this task describing the expected glass fabrication, characterization and performance testing of the vitrified product in early 2002.³ SRTC subsequently issued a Task Plan for this task in April of 2002.⁴

The task plan indicated that all testing would be performed per the Test Specification using a container centerline cooling curve to be supplied by WTP. However by fall of 2002 a container cooling curve was not available from WTP, and SRTC was directed by WTP to proceed with crucible glass production, characterization, and product testing with quenched cooling. The quench-cooled glass was analyzed for metals and radionuclides at SRTC (the results of which are described in this report) and regulatory analyses including the Toxicity Characteristic Leaching Procedure (TCLP).⁷ In the fall of 2003 a container cooling curve was made available from WTP, and SRTC was directed to remelt the AZ-102 glass and perform the container centerline cooling, followed by PCT and crystalline analyses using x-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). The tasks described in this report were performed to the QA requirements outlined in the Task Plan⁴, and all analytical measurements were performed to SRTC Analytical Development Section (ADS) routine QA/QC.

2.1 AZ-102 GLASS FORMULATION, VITRIFICATION, AND ANALYSIS METHODS

2.1.1 AZ-102 Glass Formulation

Vitrification of the pretreated AZ-102 supernatant used a reformulation of the original formulation that produced crystalline surface material.¹ The AZ-102 simulant described in Ref. 1 was used by VSL to develop the reformulation recipe for the glass. Information pertaining to the VSL investigations using both the original AZ-102 formulation (LAWB53) and the latter AZ-102 'reformulation' (LAWB88) is presented in Appendix A. Both AZ-102 formulations are presented in Table 1 (LAWB53) and Table 2 (LAWB88), with comparison of the two different formulations (LAWB53 versus LAWB88) shown in Table 3. The elements indicated with an asterisk in Table 3 are glass former species. Comparison of the two glass formulations indicates an increase in B, Ca, Si, and Zn in going from the original to the reformulation relative to the original. Also, the glass former rutile (TiO₂) was not used in the reformulation.

Glass was produced from the melter feed slurry obtained by mixing the prescribed amount of glass forming minerals with the treated AZ-102 supernatant. A listing of the WTP-approved glass forming chemicals is shown in Table 4. A small aliquot of the dry powder glass former mixture (before mixing with actual radioactive supernatant to form the melter feed slurry) was analyzed to confirm that the blend was close to calculated target. These data are shown in Table 5. Calculated values of each individual glass former mineral species divided by the analyzed values (see third row from bottom of Table 5) were all close to unity for the nine major elements added from the glass formers indicating good agreement between the dissolved and analyzed values versus target. There was no rutile mineral (TiO₂) added as glass former in this reformulated recipe for AZ-102. However, trace amounts (1.2 wt%) of rutile are contained in the Kyanite mineral as shown in Table 5. Analysis of the dissolved glass former blend showed that less elemental titania was present (0.03 wt% of the batch) vs. the predicted amount from batching of 0.07 wt%.

									Recipe for glass LAWB53 using AZ	102 H	igh S	O3 active	e waste	21-Jun-00	
Envelope		Evapor. feed	GLASS	AZ102	AZ102 wt%	Glass	LAWB53	Additives		Assay	Ratio	Target			
Constituents	calculated	AZ-102	Oxides	wt%	in glass @	Former	this target	this	Source in			Weight	other oxides		
	M	mg/L		As glass	5% Na2O	Mix	for AZ102	sample	Additives			(g)	present		
		5/15/00 email	Loading	1 .000	6.31%	100%	0.0631	93.69%		<u> </u>		Additives	% Fe2O3	%SiO2	Vendor Inormation
Ag			Ag2O	0.000	0.0000		0.0000						-		
AI	0.01	402	AI2O3	0.878	0.055	6.45	6.0987	6.043	Kyanite (Al2SiO5) 325 Mesh	0.990	0.540	70.04	1.16%	43.70%	Kyanite Mining
В		1.1	B2O3	0.004	0.0003	10.70	10.0256	10.025	H3BO3 (Technical Granular)	0.986	0.563	111.90			US Borax
Ba		1	BaO	0.002	0.0001		0.0001								
Ca		1	CaO	0.001	0.0001	7.20	6.7461	6.746	Wollanstonite NYAD 325 Mesh	0.993	0.475	88.62	0.40%	51.00%	NYCO Minerals
Cd		0.1	CdO	0.000	0.0000		0.0000								
Co		0.1	CoO	0.000	0.0000		0.0000								
Cr		532.7	Cr2O3	1.800	0.1135		0.1135								
Cu		0.0	CuO	0.000	0.0000		0.0000								
Fe		0.0	Fe2O3	0.000	0.0000	5.70	5.3406	5.341	Fe2O3 (Iron III oxide, -325 Mesh)	0.998	1.000	28.96	1		Alfa Aesar-Johnson Matthey
K	0.07	2584	K20	3.598	0.227		0.2269		· · · · · · · · · · · · · · · · · · ·				1		
La		0	La203	0.000	0.0000		0.0000								
Li		0	Li2O	0.000	0.0000	6.25	5.8559	5.856	Li2CO3 (Chemetall Foote Co. Tech. gr.)	0.99	0.404	90.72			Cyprus Foote Mineral Co.
Mg		0	MgO	0.000	0.0000	3.20	2.9982	2.998	Olivine (Mg2SiO4) 325 Mesh (#180)	0.990	0.480	39.09	7.68%	42.52%	UNIMIN Corp.
Mn		0	MnO2	0.000	0.0000		0.0000								
Mo		43.2	MoO3	0.075	0.0047		0.0047								
Na	2.21	50888	Na2O	79 298	5.00		5 0000								
Ni		0	NiO	0 000	0 0000		0 0000								
Ph		12	PhO	0.001	0.0001		0.0001								
Sn		0	SnO2	0.000	0.0000		0.0000								
Si		100	ISIO2	0.246	0.0155	52.20	48,9242	48,909	SiQ2 (Sil-co-Sil 75)	0.997	1 000	202.07	-		
Sr		0	SrO	0.000	0.0000		0.0000		(
Ti		0	TiO2	0.000	0.0000	1 50	1 4054	1 405	TiO2 (Rutile Airfloated)	0.954	1 000	9.13	0.71%	0.91%	Chemallov
W		0	WO3	0.000	0.0000		0.0000						1		
Zn		04	ZnO	0.001	0.0000	3.40	3 1857	3 186	ZnO (Kadox-920)	0.997	1 000	19.80			Zinc Corp. of America
Zr		0	7rO2	0.000	0.0000	3.40	3 1856	3 186	Zircon ZrSiO4 (Flour) Mesh 325	0.990	0.651	30.63		33.00%	American Mineral
Br		0	Br	0.000	0.0000	0.10	0,0000	0.100		0.000	0.001			00.0010	
0		67		0.077	0.0049		0.0049								
F		869	E	1 004	0.0633		0.0633								
P04		245	P205	0.212	0.0000		0.0000		Total Sodium Moles			1 00	moles		
504	138 3E-3	13287	SO3	12 802	0.8072		0.8072		Expected Glass vield			620	a		
NO2	0.57	26147	NO2	12.002	0.0012		0.0012		Sum of Additives (a)			691	9		
NO3	0.37	13300	NO3						Sugar as added reductant (decreased for TOC)			001	9		
OH	0.22	10000	OH						ougar as added reductant (decreased for 100)				8		
CO3	0.14	8616	CO3												
oxalate		2400	C												
formate			С												
SUM			SUM	100.000	6.31	100.00	100.00	93.695							
NO2+NO3	0.78	M/I	VSL use	s 12 moles	Carbon (1 m	ole sucro	ose/342.3g)	per 16 Mol	es NOx in order to mitigate foaming.						
TOC		8344.3	mg/L	This feed	requires	[7.059	g/L Carbo	n. This san	nple already includes sufficient TOC						

Table 1. Original AZ-102 Glass Formulation LAWB53

1

												Recipe using AZ102 SRTC 2001 Waste for g	glass:		LAWB88			
Envelope SR	TC AZ-102	Simulant			Envelope	GLASS	Conversion	LAWB AZ'	AZ102 in g	Glass	LAWB88	Additives	Assay	Ratio	Target			
Constituentmc	/L	Molarity			A	Oxides	to wt%	wt.%	glass @	Former	this target	this Source in			Weight	other oxides		
cor	ncentrate		molwt/at r	mol wt.	Ox.Wt		" Oxides"	As glass	5% Na2O	Mix	for AZ102	sample Additives			(g)	present		
em	iail 5/24/02	М				Loading	1	100%	6.28%	100%	6.28%	93.72%			Additives	% Fe2O3	%Si02	Vendor Inormation
AI	798.0	0.0296	50.98	101.96	1.51	AI203	0.56	0.94	0.0589	6.85	6.4788	6.420 Kyanite (Al2SiO5) 325 Mesh	0.990	0.570	58.25	0.78%	40.67%	Kyanite Mining
в	4.6		34.81	69.62	0.01	B2O3	0.01	0.01	0.0006	13.85	12.9810	12.980 H3BO3 (Technical Granular)	0.988	0.565	119.32			US Borax
Ca	67.0	0.0017	56.08	56.08	0.09	CaO	0.04	0.06	0.0037	8.50	7.9700	7.966 Wollanstonite NYAD 325 Mesh	0.993	0.475	86.52	0.40%	51.00%	NYCO Minerals
Cr	1018.1	0.0196	76.00	152.00	1.49	Cr203	0.56	0.92	0.0581		0.0581	0.000						
Cs	0.0	0.0000	140.90	281.80	0.00	Cs2O	0.00	0.00	0.0000		0.0000	0.000						
Fe	5.5	0.0001	79.85	159.70	0.01	Fe203	0.00	0.00	0.0003	2.35	2.2028	2.202 Fe2O3 (Iron III oxide, -325 Mesh)	0.970	1.000	9.63			Prince Mfg. Co.
К	4555.0	0.1165	47.10	94.20	5.49	K20	2.06	3.41	0.21		0.2142	0.000						
Li	2.0		14.94	29.88	0.00	Li20	0.00	0.00	0.0002	5.00	4.6862	4.686 Li2CO3 (Chemetall Foote Co. Tech. gr.)	0.99	0.401	60.20			Chemettal Foote Mineral Co.
Mg	3.0		40.30	40.30	0.00	MgO	0.00	0.00	0.0002	1.50	1.4060	1.406 Olivine (Mg2SiO4) 325 Mesh (#180)	0.990	0.480	15.15	7.68%	42.52%	UNIMIN Corp.
Mn	0.9	0.0000	86.94	86.94	0.00	MnO2	0.00	0.00	0.0001		0.0001	0.000						
Na	95038.5	4.1339	30.99	61.98	128.11	Na2O	47.99	79.63	5.00		5.0000	0.000						
Ni	6.0	0.0001	74.69	74.69	0.01	NiO	0.00	0.00	0.0003		0.0003	0.000						
Si	56.0	0.0020	60.09	60.09	0.12	SiO2	0.04	0.07	0.0047	53.35	50.0049	50.000 SiO2 (Sil-co-Sil 75)	0.997	1.000	175.04			US SILICA
Ti	2.0		79.90	79.90	0.00	TiO2	0.00	0.00	0.0001	0.00	0.0001	0.000 TiO2 (Rutile Airfloated)	0.932	1.000	0.00	0.71%	2.20%	Chemalloy
Zn	2.7		81.39	81.39	0.00	l ZnO	0.00	0.00	0.0001	5.20	4.8736	4.874 ZnO (Kadox-920)	0.999	1.000	24.99			Zinc Corp. of America
Zr	4.0	0.00004	123.22	123.22	0.01	ZrO2	0.00	0.00	0.0002	3.40	3.1867	3.187 Zircon ZrSiO4 (Flour) Mesh 325	0.990	0.660	24.98		32.25%	American Mineral
CI	200.0	0.0056	35.45	35.45	0.20	I CI	0.07	0.12	0.0078		0.0078							
F	1370.0	0.0721	19.00	19.00	1.37	'F	0.51	0.85	0.0535		0.0535							
PO4	704.0	0.0074	70.97	70.97	0.53	P205	0.20	0.33	0.0205		0.0205	Total Sodium Moles			0.83	moles		
SO4 total	26300.0	0.2738	80.06	80.06	21.92	S03	8.21	13.63	0.8555		0.8555	Expected Glass yield			512	g		
N02	49440.0	1.0748	46.00	46.00	49.44	NO2	18.52					Sum of Additives (g)			574	g		
N03	27305.0	0.4404	62.00	62.00	27.31	NO3	10.23					Sugar as added reductant (decreased for TOC)			None - See	note below		
C03	9396.0	0.1566	60.01	60.01	9.40	003	3.52					Volume of Simulant AZ-102 SRTC Envelope	e B Recip	e:	200	ml		
NH3	0.0	0.0000	17.03	17.03	0.00	I NH3	0.00											
ОН	0.0	0.0000	17.00	17.00	0.00	ОН	0.00					Weight of AZ102 sample used			232.2	g		
Org.Carbon	19914.0	1.6595	12.00	12.00	19.91	C	7.46					density			1.161			
SUM			1497.70	1973.27	266.93	SUM	100.00	100.00	6.28	100.00	100.00	93.721 Sodium Molarity			4.13			
						VSL uses	12 moles Ca	arbon (1 mo This feed r	ple sucrose/ equires	342.3g) pe 13.637	er 16 Moles NOx i g/L. Carbon. This	n order to mitigate toaming. sample already includes sufficient TOC						

Table 2. Reformulation of AZ-102 Glass LAWB88

1 of mulation			
Envelope Constituents	LAWB53 this target for AZ102	LAWB88 this target for AZ102	
	Wit% Oxides	Wt% Oxides	Change
A I*	6.10	6.48	0.38
B *	10.03	12.98	2.96
C a *	6.75	7.97	1.22
C r	0.11	0.06	-0.06
Fe*	5.34	2.20	-3.14
К	0.23	0.21	-0.01
Li*	5.86	4.69	-1.17
M g *	3.00	1.41	-1.59
Na	5.00	5.00	0.00
S i*	48.92	50.00	1.08
T i*	1.41	0.00	-1.41
Z n *	3.19	4.87	1.69
Z r *	3.19	3.19	0.00
CI	0.00	0.01	0.00
F	0.06	0.05	-0.01
P O 4	0.01	0.02	0.01
S O 4	0.81	0.86	0.05
SUM	100.00	100.00	

Table 3. Comparison of Original (LAWB53) and Revised (LAWB88) AZ-102 Glass Formulation

* = elements added as glass-forming minerals

No.	Oxide Added:	Mineral	Grade	Company
1	Al ₂ O ₃	Kyanite Al ₂ O ₃ -SiO ₂	Raw –325	Kyanite Mining Corp Dillwyn, VA, 23936
	Alternate	Alumina Al ₂ O ₃	A-2 <325M	Alcoa Alumina Bauxite, AK 72011 www.alunina.alcoa.com
2	B_2O_3	Boric Acid H ₃ BO ₃	Technical Grade-Granular	U.S. Borax Valencia, CA 91355-1847 www.borax.com
3	Na ₂ O/B ₂ O ₃	10M Borax Na ₂ B ₄ O ₇ -10H ₂ O	Technical 10Mole Borax	U.S. Borax Valencia, CA 91355-1847 www.borax.com
4	Na ₂ O	Na ₂ CO ₃ Anhydrous	Dense Soda Ash	Solvay Minerals Houston, TX www.solvayminerals.com
5	CaO	Wollastonite CaSiO ₃	NYADM325 NWest Mexico	NYCO Wilsboro, NY www.nycominerals.com
6	Fe ₂ O ₃	Fe ₂ O ₃	Fe ₂ O ₃ 5001	Prince Mfg. Co. Quincy, IL 62306 www.princemfg.com
7	Li ₂ O	Li ₂ CO ₃	Technical Grade	Chemettal-Foote Kings Mt., NC www.chemetalllithium.com
8	MgO	Olivine	#180 Hamilton, WA	Unimin Corp
9	SiO ₂	SiO ₂	SCS-75 Mill Creek OK	U.S. Silica Berkeley Springs WV www.u-s-silica.com
10	TiO ₂	Rutile (Air floated) TiO ₂ /Fe ₂ O ₃	Air Float Rutile 94 Phil. PA	Chemalloy Co. Bryn Mawr, PA www.chemalloy.com
11	ZnO	ZnO	Kadox 920 Camden, NJ	Zinc Corp Amer. Monaca, PA horseheadinc.com
12	ZrO ₂	ZrSiO ₄	Zircon Flour	Amer. Miner.Inc. Monaca, PA 19406 www.americanminerals.net
13	С	Sugar	Granular Portland OR	Amalgamated Sugar Co. Ogdin, UT www.gfhandle/industry

 Table 4.
 WTP-Approved Glass Forming Minerals

AZ102 SRTC 2001 LAWB88	Batch														
	Weight	SiO2	AI2O3	Fe2O3	B2O3	Cr2O3	CaO	MgO	ZnO	NiO	Li2O	Na2O	K2O	TiO2	ZrO2
Glass Former	(grams)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
Kyanite - Raw -325	116.50	47.18	66.41	0.80										1.22	
H3BO3 - Tech. Gran	238.64				134.88										
Wollastonite - NYAD325	173.04	88.25	0.35	0.69			82.19	0.17						0.03	
Fe2O3 - 5001	19.26	0.26	0.29	18.68			0.01	0.02							
Li2CO3 - Tech. Gran	120.40										48.40				
Olivine - #180	30.30	12.88	0.06	2.33		0.04	0.01	14.55		0.11		0.01	0.00		
SilCoSil -75	350.08	349.03	0.47	0.06			0.03	0.03				0.01	0.06	0.03	
Rutile 94 - Air floated	0.00	0.00	0.00	0.00		0.00								0.00	0.00
ZnO - Kadox 920	49.98			0.00					49.93						
Zircon Flour - Am Min.	49.96	16.11	0.12	0.04										0.05	32.97
Totals:	1,148.16	513.72	67.70	22.60	134.88	0.04	82.24	14.77	49.93	0.11	48.40	0.01	0.06	1.34	32.97
		(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)
Weight % Oxide of Batch		44.74%	5.90%	1.97%	11.75%	0.00%	7.16%	1.29%	4.35%	0.01%	4.22%	0.00%	0.01%	0.12%	2.87%
Weight % Element of Batch		20.93%	3.12%	1.38%	3.65%	0.00%	5.12%	0.78%	3.49%	0.01%	1.96%	0.00%	0.00%	0.07%	2.13%
Weight % Element Analyzed		21.23%	2.99%	1.40%	3.84%		5.32%	0.76%	3.11%		1.97%			0.03%	2.06%
Delta(Calc-Analyzed)		-0.30%	0.13%	-0.02%	-0.19%		-0.20%	0.02%	0.39%		-0.01%			0.04%	0.07%
Calc/Analyzed		0.99	1.04	0.98	0.95		0.96	1.02	1.12		0.99			2.25	1.03
Weight % Oxide		53.02%	6.99%	2.33%	13.92%	0.00%	8.49%	1.52%	5.15%	0.01%	5.00%	0.00%	0.01%	0.14%	3.40%
Weight % Element		24.80%	3.70%	1.63%	4.33%	0.00%	6.07%	0.92%	4.14%	0.01%	2.32%	0.00%	0.01%	0.08%	2.52%

Table 5. Analytical Data for Glass Former Blend LAWB88

2.1.2 Crucible-Scale Vitrification

Crucible-scale vitrifications were performed using 95% Pt/5% Au crucibles that were located inside of the quartz glass offgas system inside of the Deltech furnace. The offgas system was employed to trap offgas from the vitrification process in order to capture the products for return to Hanford as residue. Vitrification tests involved loading the 600-mL crucibles with the prescribed amount of treated AZ-102 supernate and glass former blend. This mixture (representing the 'melter feed slurry') was briefly mixed by hand with a stir rod before placing into the furnace.

Table 6 shows the data for the two replicate AZ-102 surrogate melts and the two replicate AZ-102 radioactive melts. Replicate vitrification tests were performed to produce the approximately 220 gram product glass required for planned analyses. Due to expansion of the static melts during the vitrification process (evaporation, calcining, and melting), it was not possible to produce the entire 220 g batch in a single vitrification task. The heating sequence involved first slow heating to evaporate off the liquid in the slurry, followed by calcining, and then melting at 1150°C. Melted glass samples were allowed to rapidly cool by turning off the furnace power supply. A typical heating sequence plot of temperature versus time and the rapid cooling is shown in Figure 1.

After the quench-cooled glasses were produced in 2002, a container centerline cooling curve was provided to SRTC from WTP personnel in fall of 2003. Table 6 shows data for remelting of all remaining quench-cooled glass that was container cooled. Only the radioactive AZ-102 glass was remelted and container cooled. This glass was the AZ-102 glass left over from previous testing (characterization, PCT, TCLP). Figure 2 shows the remelt and container cooling performed on the original AZ-102 quenched glass. Appendix B contains the WTP communication CCN 074181 that officially transmitted the cooling curve information to SRTC. The cooling curve portion of Figure 2 is an exact replication of the data provided by WTP as Figure 1 and Table 1, LAW CCC Profile for Crucible Testing in Appendix B.

Photographs of both the quench-cooled AZ-102 glass and the centerline cooled AZ-102 glass are shown in Figure 3 and Figure 4 (quenched) and Figure 5 and Figure 6 (CCC) for comparison. Figure 3 and Figure 4 show the top and bottom surface of one of the crucible glass products after quench cooling. Figure 5 and Figure 6 show the top and bottom surface of the glass that was container cooled. No visible differences in the two different AZ-102 glasses (quenched glass in Figure 3 and Figure 4 vs. centerline cooled in Figure 5 and Figure 5 and Figure 6) are present in these comparative photos.

AZ-102 Vitrifi- cation Tests	Date	Furnace	Mass Glass Formers (g)	Volume Liquid (mL)	Target Glass Mass (g)	Notes
#1	8/26/02	Deltech	123.32	40.5	110	First of two replicate
Surrogate						runs using <u>Surrogate</u> <u>AZ-102</u> , rapid cooling
#2	9/3/02	Deltech	123.32	40.5	110	Second of two replicate
Surrogate						runs using <u>Surrogate</u> <u>AZ-102</u> , rapid cooling
#3	9/9/02	Deltech	123.32	43	110	First of two replicate
Radioactive						runs using <u>Radioactive</u> <u>AZ-102</u> , rapid cooling
#4 Radioactive	9/11/02	Deltech	123.32	43	110	Second of two replicate
						<u>AZ-102</u> , rapid cooling
#5 Radioactive	10/21/03	Deltech	NA	NA	110	Radioactive AZ-102, container centerline cooling

Table 6. Details of Melter Feed Slurry and Glass Targets for Crucible-Scale Vitrification Experiments

NA: This crucible vitrification started with 110g solid glass from the quenched cooling study



Figure 1. Heating and Cooling Sequence for Crucible Vitrification with Quenched Cooling



Figure 2. Temperature vs. Time Profile of Remelt and Container Centerline Cooling for AZ-102 Crucible-Scale Glass



Figure 3. Top surface AZ-102 LAW glass (~110 g in 600-mL Pt-crucible) after reformulation and <u>quench-cooling</u>



Figure 4. Bottom surface AZ-102 LAW glass (~ 110 g in 600-mL Pt-crucible) after reformulation and <u>quench-cooling</u>

Note glass is green in color and transparent, i.e., not dark/opaque. The small 'craters' visible on the bottom surface of glass in Figure 4 are similar to other RPP LAW glass melts performed in static Pt-crucible testing. No observable crystalline phases are present.



Figure 5. Top surface AZ-102 LAW glass (~ 110 g in 600-mL Pt-crucible) after remelting and <u>container centerline cooling</u>.



Figure 6. Bottom surface AZ-102 LAW glass (~ 110 g in 600-mL Pt-crucible) after remelting and <u>container centerline cooling</u>.

Note glass is green in color and transparent, i.e., not dark/opaque. The small 'craters' visible on the bottom surface of glass in Figure 6 are similar to other RPP LAW glass melts performed in static Pt-crucible testing. No observable crystalline phases are present.

2.1.3 Dissolved-Glass Sample Preparation and Analysis

Samples were taken of the AZ-102 quench-cooled glass, the AZ-102 surrogate quenchcooled glass, and the LRM glass.¹⁰ The AZ-102 glass surrogate was prepared from an AZ-102 simulated supernate (See Reference 1) using the same glass forming minerals, the same heating profile, and the same cooling profile as the AZ-102 radioactive glass. The purpose of the AZ-102 surrogate glass 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 of less than 0.9 centimeter as required by Toxicity Characteristic Leaching Procedure (TCLP), Method 1311 of USEPA Manual SW-846. The TCLP testing of the glasses was performed and the resulting data has been reported in a separate SRTC report on the regulatory analyses of the AZ-102 glass.⁷

Upon completion of the initial size reduction, two sample sets were generated:

- AZ-102 glass, nonradioactive AZ-102 surrogate glass, and LRM glass for PCT analyses
- AZ-102 glass, nonradioactive AZ-102 surrogate glass, and LRM glass to be dissolved and analyzed for metals and radionuclides

Samples of the crushed glass were subjected to grinding in a tungsten blade grinder with stainless steel chamber for preparation for the PCT. Approximately 5 grams of glass powder in the > 100 mesh to < 200 mesh size was prepared for each glass.

Samples of the crushed glass were also subjected to pulverizing in a ball and cup 'wigglebug' grinder using agate balls, cups and caps. These pulverized glass powders were screened through a 200 mesh sieve to produce < 200 mesh glass powders for dissolution. Two subsets of dissolved glass samples were generated using methods given in ASTM C1463-00 Procedure for Dissolving Radioactive Glass.¹¹ The two subsets of samples were:

- acid-dissolved glass samples, used for metal analyte determinations and for radionuclide analyte determinations
- fusion-dissolved glass samples with an acid uptake, used for metal analyte determinations and for radionuclide analyte determinations

Each of these sets consisted of seven samples. These were duplicate AZ-102 glass samples, duplicate nonradioactive AZ-102 surrogate glass samples, duplicate LRM glass, and a preparation blank (no glass added).

A 0.5 gram portion of pulverized < 200 mesh glass was used in the acid dissolution. Aciddissolved 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. This process dissolved 0.5 g of glass into a total of 100 mL of solution to give a ratio of 0.5g/100mL, or 0.005 g glass per mL of solution. This ratio of glass to solution is higher than the ratio suggested in ASTM C1463-00 that uses 0.25 g glass in 250mL of solution, for a ratio of 0.001 g glass per mL of solution. The higher ratio of glass to solution (0.005 g glass/mL) was used in order to maximize the concentration of dissolved glass analyte in solution.

Peroxide fusion samples were generated also using 0.5 gram portions of < 200 mesh pulverized glass. 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 °C. Concentrated nitric acid was then added to the samples. All samples were then brought to 100 milliliters with water.

2.2 CONCENTRATION OF METAL ANALYTES IN AZ-102 GLASS SAMPLES AND THE LRM STANDARD

Results from the dissolution and analysis of the crucible scale glasses for the metal analytes are presented in this section. These results were determined from dissolving nominally 0.5 gram of pulverized glass (< 200 mesh) in 0.1 L of total solution by either the acid dissolution method or the peroxide fusion method. The metal analyses were performed with Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES). Potassium values were measured by Atomic Absorption Spectroscopy (AAS). Table 7 and Table 8 present the elemental analyses results for the various metals (Al through Zr) in the glasses for the acid dissolution and the peroxide fusion, respectively. Boron values from peroxide fusion are shown in the acid dissolution data since boron is used in the acid dissolution process. Nickel and sodium values from acid dissolution are shown in the peroxide fusion process performed in nickel crucibles. Measured values shown in Table 9 and Table 10 for Cl, F, and SO₃ are taken from Ferrara.⁷

Table 9 and Table 10 show the oxide components of the glasses for the acid dissolution and the peroxide fusion, respectively. For most of the major analytes measured above 0.5 wt%, results given in Table 9 and Table 10 were within three standard deviations of the targets. The VSL provided the AZ-102 target composition along with the quantities of AZ-102 supernate and glass-forming minerals used to make the glass. To determine the simulant target, the composition of the AZ-102 supernate simulant¹ was used. Results from Table 9 indicate that measured Al₂O₃ from acid dissolution were consistently low vs. target, whereas Al₂O₃ values from Table 10 for peroxide fusion are in better agreement with target for all major glasses. The B₂O₃ values from Table 9 and Table 10 also indicate lower analyzed values vs. target for the glasses. Similar low analyzed values for CaO vs. target are also found in Table 9 and Table 10 for the AZ-102 glasses.

The Na₂O values for the radioactive AZ-102 glasses measured from acid dissolution were about 10% lower than the target 5 wt% Na₂O for the radioactive AZ-102 glasses. Much better agreement was found for analyzed Na₂O for surrogate AZ-102 and LRM glasses vs. their target Na₂O values. The SiO₂ values measured from acid dissolution of the radioactive AZ-102 glasses were 10% higher than target shown in Table 9, but the measured SiO₂ values from peroxide fusion were in excellent agreement with target shown in Table 10. Except for the totals given for the acid dissolution of the radioactive AZ-102 glass constituents (those present above 0.5 wt percent) in Table 9, most total oxide sums are close to 100 wt%. The totals for the acid dissolution of the radioactive AZ-102 glass are higher than the target of 100 wt% mainly due to the high bias in measured SiO₂ discussed above.

		AZ-102		AZ-102		
	AZ-102	Rad	AZ-102	Surrogate	LRM	LRM
	Rad	Duplicate	Surrogate	Dup		Duplicate
	Elemental	Elemental	Elemental	Elemental	Elemental	Elemental
	wt%	wt%	wt%	wt%	wt%	wt%
AI	2.94	2.65	3.18	3.17	4.94	4.83
В	3.87	3.87	3.87	3.91	2.23	2.30
Ba	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Ca	4.75	4.40	4.96	4.90	0.36	0.35
Cd	< 0.01	< 0.01	< 0.01	< 0.01	0.14	0.14
Cr	0.05	0.05	0.05	0.05	0.13	0.13
Cu	0.05	0.06	0.04	0.04	0.02	0.02
Fe	1.68	1.63	1.54	1.53	0.99	0.98
K(AA)	0.15	0.12	0.11	0.13	1.12	1.08
La	0.01	0.01	0.01	0.01	0.01	0.01
Li	2.20	2.14	2.29	2.27	0.04	0.04
Mg	1.14	1.05	0.92	0.92	0.06	0.06
Mn	0.01	0.01	0.01	0.01	0.06	0.06
Мо	< 0.04	< 0.04	0.03	0.02	0.08	0.08
Na	3.27	3.41	3.79	3.74	15.39	14.98
Ni	< 0.01	< 0.01	< 0.15	< 0.16	0.14	0.14
Р	< 0.09	< 0.10	< 0.18	< 0.18	0.22	0.21
Pb	< 0.05	< 0.05	< 0.10	< 0.09	0.07	0.07
Si	26.66	26.10	24.00	23.00	27.54	27.08
Sn	0.31	0.33	0.06	0.06	0.06	0.06
Sr	1.04	0.96	0.08	0.08	0.08	0.08
Ti	0.10	0.10	0.10	0.10	0.06	0.06
U	0.29	0.27	0.15	0.15	< 0.01	< 0.01
Zn	4.17	4.10	3.84	3.81	< 0.01	< 0.01
Zr	2.45	2.36	2.30	2.28	0.70	0.69

 Table 7.
 Elemental Analyses Results for Acid Dissolved Glasses

				AZ-102		
	AZ-102	AZ-102	AZ-102	Surrogate	LRM	LRM
	Rad	Rad-Dup	Surrogate	Dup		Dup
	Elemental	Elemental	Elemental	Elemental	Elemental	Elemental
	wt%	wt%	wt%	wt%	wt%	wt%
AI	3.41	3.39	3.38	3.34	4.98	5.10
В	3.86	3.86	3.90	3.86	2.22	2.29
Ba	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Ca	4.93	4.95	4.87	4.86	0.35	0.35
Cd	< 0.00	< 0.00	< 0.00	< 0.04	0.14	0.14
Cr	0.05	0.05	0.04	0.04	0.13	0.13
Cu	0.04	0.04	0.04	0.04	0.02	0.02
Fe	1.56	1.56	1.56	1.53	0.96	0.99
K (AA)	0.19	0.19	0.18	0.18	1.09	1.10
La	0.01	0.01	0.01	0.01	0.01	0.01
Li	2.27	2.26	2.26	2.26	0.04	0.05
Mg	0.94	0.95	0.97	0.96	0.06	0.06
Mn	0.01	0.01	0.01	0.01	0.05	0.05
Мо	0.01	0.01	0.01	0.00	0.06	0.07
Na	3.27	3.41	3.79	3.74	15.39	14.98
Ni	0.01	0.01	< 0.01	< 0.01	0.14	0.14
Р	< 0.08	< 0.08	< 0.07	< 0.08	0.14	0.15
Pb	< 0.04	< 0.04	< 0.04	< 0.04	0.09	0.09
Si	23.17	23.97	23.97	22.97	27.54	27.08
Sn	0.06	0.06	0.06	0.06	0.01	0.01
Sr	1.06	1.05	1.05	10.50	0.11	0.11
Ti	0.10	0.10	0.10	0.09	0.06	0.06
U	0.12	0.12	0.12	0.12	< 0.01	< 0.01
Zn	4.30	4.31	4.24	4.16	0.01	0.01
Zr	2.39	2.38	2.40	2.37	0.65	0.67

Table 8. Elemental Results for Analyses of Peroxide Fusion Dissolved Glasses

		AZ-102		AZ-102					AZ-102	AZ-102				LRM	LRM
		Rad		Rad-Dup	AZ-102		AZ-102		Surrogate	Surrogate		LRM		Dup	Target
		oxide		oxide	Rad Target		Surrogate		Duplicate	Target		oxide		oxide	oxide
	-	wt%		wt%	oxide wt%	-	oxide wt%		oxide wt%	oxide wt%		wt%		wt%	wt%
Al2O3		5.55		5.01	6.5		6.01		5.99	6.43		9.33		9.12	9.5
B2O3		12.46		12.46	13		12.46		12.59	13		7.18		7.41	7.9
BaO	<	0.01	<	0.01	-	<	0.01	<	0.01	-	<	0.01	<	0.01	0.01
CaO		6.65		6.15	8		6.93		6.85	7.97		0.51		0.49	0.54
CdO	<	0.01	<	0.01	-	<	0.01	<	0.01	-		0.16		0.15	0.16
Cr2O3		0.08		0.08	0.06		0.07		0.07	0.06		0.19		0.19	0.19
CuO		0.06		0.07	-		0.05		0.05	-		0.03		0.03	-
Fe2O3		2.39		2.32	2.2		2.20		2.19	2.2		1.41		1.40	1.42
K2O		0.19		0.15	0.21		0.14		0.16	0.2		1.35		1.31	1.48
La2O3		0.01		0.01	-		0.01		0.01	-		0.01		0.01	0.02
Li2O		4.74		4.61	4.69		4.93		4.89	4.69		0.09		0.09	0.11
MgO		1.88		1.75	1.41		1.53		1.52	1.41		0.10		0.10	0.1
MnO2		0.01		0.01	-		0.01		0.01	-		0.09		0.09	0.08
MoO3	<	0.06	<	0.06	-		0.04		0.04	-		0.12		0.12	0.1
Na2O		4.40		4.60	5		5.11		5.04	5.08		20.74		20.19	20.03
NiO	<	0.02	<	0.02	-	<	0.19	<	0.20	-		0.17		0.17	0.19
P2O5	<	0.21	<	0.22	0.02	<	0.41	<	0.41	0.02		0.50		0.48	0.53
PbO	<	0.05	<	0.05	-	<	0.11	<	0.10	-		0.08		0.08	0.1
SiO2		57.04		55.84	50		51.34		49.20	50		58.92		57.93	54.3
SnO2		0.40		0.42	-		0.08		0.08	-		0.07		0.07	0.03
SrO		1.23		1.14	-		0.09		0.09	-		0.09		0.09	-
TiO2		0.17		0.17	-		0.16		0.16	-		0.10		0.10	0.11
UO2		0.33		0.31	-		0.17		0.17	-	<	0.01	<	0.01	-
ZnO		5.19		5.11	4.87		4.78		4.74	4.88	<	0.01	<	0.01	-
ZrO2		3.31		3.18	3.19		3.10		3.07	3.19		0.95		0.93	0.93
CI	<	0.20	<	0.20	0.01	<	0.20	<	0.20	-	<	0.20	<	0.20	0.07
F	<	0.20	<	0.20	0.05	<	0.20	<	0.20	0.1		0.74		0.74	0.86
SO3		0.85		0.85	0.86		0.94		0.94	0.77		0.45		0.45	0.3
TOTALS		107.73		105.00	100.07		101.29		99.00	100		103.61		101.97	99.06

Table 9. Weight Percent Oxide Results for Analyses of Acid Dissolved Glasses

		AZ-102		AZ-102			AZ-102		AZ-102	AZ-102				LRM	LRM
		Rad		Rad-Dup	AZ-102		Surrogate		Surrogate	Surrogate		LRM		Dup	Target
		oxide		oxide	Rad Target		oxide		Duplicate	Target		oxide		oxide	oxide
		Wt%		wt%	Oxide wt%		Wt%		oxide wt%	oxide wt%		Wt%		Wt%	Wt%
AI2O3		6.44		6.40	6.50		6.39		6.31	6.43		9.41		9.64	9.54
B2O3		12.44		12.44	13.00		12.57		12.44	13.00		7.16		7.39	7.90
BaO	<	0.01	<	0.01	-	<	0.01	<	0.01	-	<	0.01	<	0.01	0.01
CaO		6.90		6.92	8.00		6.81		6.80	7.97		0.49		0.50	0.54
CdO	<	0.01	<	0.01	-	<	0.00	<	0.04	-		0.15		0.16	0.16
Cr2O3		0.07		0.07	0.06		0.06		0.06	0.06		0.19		0.20	0.19
CuO		0.05		0.05	-		0.05		0.05	-		0.03		0.03	-
Fe2O3		2.23		2.23	2.20		2.24		2.19	2.20		1.37		1.42	1.42
K2O		0.22		0.23	0.21		0.22		0.22	0.20		1.31		1.33	1.48
La2O3		0.01		0.01	-		0.01		0.01	-		0.01		0.01	0.02
Li2O		4.89		4.87	4.69		4.87		4.87	4.69		0.10		0.10	0.11
MgO		1.57		1.58	1.41		1.61		1.59	1.41		0.10		0.11	0.10
MnO2		0.02		0.02	-		0.02		0.02	-		0.07		0.08	0.08
MoO3		0.02		0.02	-		0.01		0.00	-		0.09		0.10	0.10
Na2O		4.40		4.60	5.00		5.11		5.04	5.08		20.74		20.19	20.03
NiO		0.02		0.02	-	<	0.01	۷	0.01	-		0.17		0.17	0.19
P2O5	<	0.18	<	0.19	0.02	<	0.17	<	0.18	0.02		0.32		0.34	0.53
PbO	<	0.04	۷	0.05	-	۷	0.04	۷	0.04	-		0.10		0.10	0.10
SiO2		49.57		51.28	50.00		51.29		49.14	50.00		58.92		57.93	54.26
SnO2		0.08		0.08	-		0.07		0.07	-		0.01		0.02	0.03
SrO		1.25		1.24	-		1.24		1.24	-		0.13		0.13	-
TiO2		0.16		0.16	-		0.16		0.16	-		0.10		0.10	0.11
UO2		0.14		0.14	-		0.14		0.14	-	<	0.01	<	0.01	-
ZnO		5.35		5.37	4.87		5.28		5.18	4.88		0.01		0.01	-
ZrO2		3.23		3.22	3.19		3.25		3.21	3.19		0.88		0.90	0.93
CI	<	0.20		0.20	0.01		<0.2		<0.2	-		<0.2		<0.2	0.07
F	<	0.20		0.20	0.05		<0.2		<0.2	0.10		0.74		0.74	0.86
SO3		0.85		0.85	0.86		0.92		0.92	0.77		0.45		0.45	0.30
TOTALS		100.56		102.43	100.07		102.55		99.94	100.00		103.07		102.14	99.06

Table 10. Weight Percent Oxide Results for Analyses of Peroxide Fusion Dissolved Glasses
2.3 DENSITY OF THE AZ-102 AND LRM GLASSES

Densities of the AZ-102 glasses and the LRM glass were measured by determining the volume displacement of water from a known mass of glass. These measurements used approximately 2 grams of crushed glass in 5-mL graduated cylinders that were partially filled with deionized water. The volume of water was measured before and after addition of the glass to the cylinders. The data for measured mass and volume displacement are shown in Table 11. The calculated density for the AZ-102 radioactive glass is 2.59 g/cc and for the surrogate AZ-102 glass is 2.54 g/cc. The measured density value for the LRM glass of 2.50 g/cc compares well with the published value of 2.516 +/- 0.009 g/cc.¹⁰

Measured Glass Densities	Measured Glass Densities							
Glass	Mass	Volume	Density	Average	St.Dev.	%RSD		
	(g)	(mL)	g/cm ³	g/cm ³	g/cm ³			
AZ-102 Radioactive	2.112	0.80	2.64					
	2.056	0.80	2.57					
	2.036	0.80	2.55					
				2.59	0.05	1.91		
AZ-102 Surrogate	2.045	0.80	2.56					
	2.099	0.80	2.62					
	2.085	0.85	2.45					
				2.54	0.09	3.38		
LRM	2.010	0.80	2.51					
	2.079	0.85	2.45					
	2.039	0.80	2.55					
				2.50	0.05	2.09		

 Table 11. Density Data for AZ-102 Glasses and LRM Glass

2.4 RADIONUCLIDES MEASURED IN AZ-102 GLASSES

2.4.1 Estimated Radionuclide Concentration in AZ-102 Glass

The radionuclide concentrations in the AZ-102 glass can be estimated from data presented previously in Reference 1 for the evaporator concentrate that was used to produce the glass. Table 12 shows the average radionuclide concentrations for the evaporator concentrate and the ratios of the volume amount of the concentrate used along with the target glass masses. These data can be used to calculate the expected specific activities of the three radionuclides noted in the BNI-WTP contract.¹² The measured AZ-102 glass density from this study was also used in the calculation. These results indicate that the expected concentration for Cs-137 in the AZ-102 glass is 1.21 Ci/m³ to 1.29 Ci/m³. The expected concentration Sr-90 in the AZ-102 glass is 1.1 Ci/m³ to 1.4 Ci/m³. Similar estimates for Tc-99 give much lower expected concentrations in the range of approximately 1E-4 Ci/m³ for Tc-99 from ICP-MS and less than 1E-2 Ci/m³ for Tc-99 from separation and counting.

Radionuclide	Analysis Method	Average Concentration (μCi/mL) in Pretreated Concentrate (from Ref. 1)	Volume Pretreated Concentrate Liquid (mL)	Target Mass Glass (g)	Expected Range of Activity in Glass (μCi/g)	Expected Range of Activity in Glass (Ci/m ³) [#]
Cs-137	Gamma Pulse Height Analysis	1.24±0.04	43	110	0.47 - 0.50	1.21 -1.29
Sr-90	Separation, Counting	1.24±0.16	43	110	0.42 - 0.55	1.1 – 1.4
Тс-99	ICP-Mass Spec	1E-04±1E-05	43	110	3.7E-05 – 4.4E-05	9.5E-05 – 1.1E-04
Тс-99	Separation, Counting	<9.4E-03±9E-04	43	110	<3.3E-03 – 4.0E-03	<8.6E-03 – 1.0E-2

Table 12. Expected Average Radionuclide Activities in AZ-102 Glass

Notes:

[#] Specification limits (BNI-WTP Contract): $< 3 \text{ Ci/m}^3$ for Cs-137, $< 20 \text{ Ci/m}^3$ for Sr-90 and $< 0.1 \text{ Ci/m}^3$ for Tc-99; Measured density for Env. B glass $\sim 2.59 \text{ g/cm}^3$.

2.4.2 Measured Radionuclides in AZ-102 Glass

Radionuclides were measured in the product AZ-102 glasses using the methods described in the task plan for this work. These methods included gamma scan, total alpha, and total beta analyses performed on the dissolved glasses without any separation techniques. Individual radionuclides were determined by separation and counting methods. Average radionuclide analyses results are shown in Table 13 for the primary LAW radionuclides specified in the WTP Contract (Cs-137, Sr-90 and Tc-99) as well as the overall total alpha and total beta activities in the glass. Data for the actual radioactive glass, the surrogate glass, and the blank solutions are shown in Appendix C.

The surrogate glass was analyzed for radionuclides to investigate any significant contamination of the glasses during the vitrification process. Measured density values for the radioactive AZ-102 glass and the surrogate glass were used to calculate the final column data for Table C-1 in Appendix C in units of curies per cubic meter of glass (Ci/m³). This concentration of Cs-137 in the AZ-102 glass of 0.98 ± 0.03 Ci/m³ is less than the WTP Contract specification of <3 Ci/m³.

The measured value for Sr-90 in the AZ-102 glass of 1.2 Ci/m³ is well below the WTP Contract specification of <20 Ci/m³. Measured values for Tc-99 of <2.7E-3 Ci/m³ are also well below the WTP Contract specification of < 0.1 Ci/m³. It should also be noted that the gross beta measured in the radioactive AZ-102 dissolved glass of approximately 3.3 + - 0.1 Ci/m³ is in good agreement with the sum of measured beta-emitters Cs-137 = 0.98 Ci/m³, Sr-90 (and Y-90) = 2 x 1.2 uCi/m³ (sum = 3.4 uCi/m³). Finally, Table 13 data and Appendix C data for measured total alpha activity and Pu-241 in the glass indicates that the sum of alphas is 2.3 nCi/g in the radioactive AZ-102 glass. This value is well below the WTP Contract specified value for < 100 nCi/g transuranics in LAW glass.

Table 14 shows additional radionuclide analyses data (determined for separation and alpha and beta counting) for the radioactive AZ-102 glass and the surrogate. Note these tables indicate that predominately less than detectable quantities of the various radionuclides were present in the radioactive AZ-102 glass. Peroxide fusion samples were not analyzed for tritium, I-129, C-14 due to the expected loss of these volatile radionuclides in this high-temperature fusion process. Data for the actual radioactive glass, the surrogate glass, and the blank solutions are shown in Appendix C. Measured values for Am-241 indicate that three of the quadruplicate values were less than detection limits of ~ 2E-3 Ci/m³, with one measured value reported slightly below this limit at 1.0E-3 Ci/m³. Three of the quadruplicate values for Pm-147/Sm-151 were below the detection limit of 2.4E-3 Ci/m³. One value of the quadruplicate data set for Pm-141/Sm-151 was measured as 1.9E-1 Ci/m³, which is ~ 45 times the detection limit. This value is considered high bias and inaccurate.

No direct separation and counting measurements were performed for Se-79. However comparison of the total gross beta value $(3.3 + - 0.1 \text{ Ci/m}^3)$ with the sum of all other measured beta emitters (major Cs-137, Sr-90(Y-90), minor Ni-63, Tc-99, I-129, Sm-151 and Pu-241) the sum of which is 3.51 Ci/m^3 , indicates that Se-79 levels in the AZ-102 glass are likely in the same range as the other non-detectable beta emitters (Ni-63, Tc-99, I-129, Sm-151 and Pu-241), i.e., in the range of 10^{-5} to 10^{-2} Ci/m^3 .

Table 15 shows the data obtained from ICP-MS analyses of the acid-dissolved glasses and the reagent blank. These data show that the nominal instrument detection limit for the ICP-MS for these glass dissolutions (0.5 gram glass dissolved in 100 mL solution) was about 0.043 μ g/L (ppb). The only significant actinides present in these glasses are the mass-232 and mass-238 species associated with Th-232 and U-238. These isotopes are also present as naturally occurring impurities in the glass forming mineral zircon¹ used in the vitrification process.

Table 16 shows all of the minimum detectable activities (MDA) associated with the gamma scan measurement. These values represent the minimum detectable gamma activity present in the AZ-102 glass using approximately 0.5 g glass dissolution in 100 mL volumes for this study. None of these the radionuclides shown in Table 16 were detected in the radioactive AZ-102 glass at or above the MDA values shown, which are in the range of 10^{-2} to 10^{-3} Ci/m³.

Table 13.	Radionuclide	Analyses for	Cs-137, Sr-90,	, Tc-99, To	tal Alpha and	Total Beta
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	Average	St. Dev.	
	Ci/m ³	Ci/m ³	%RSD
Cs-137	9.8E-01	2.9E-02	3.0
Sr-90	1.2E+00	4.4E-01	36.2
Alpha	5.9E-03	2.5E-03	42.7
Beta	3.3E+00	1.3E-01	3.8
Тс-99	< 1.3E-03	NA	NA

NA = not applicable; all values for separation and counting for Tc-99 reported as less than detection limit of 10^{-3} Ci/m³.

	Average	St. Dev.	
	<u>Ci/m³</u>	Ci/m ³	%RSD
Pu-239/240	<1.9E-04	(1)	(1)
Cm-242	<3.7E-05	(1)	(1)
Pu-238	1.1E-03	3.8E-04	34
Pu-241	<4.8E-03	(1)	(1)
Ni-63	<3.9E-03	(1)	(1)
Am-241	1.0E-03	(2)	(2)
Pm-147/Sm-151	<2.4E-03	(3)	(3)
Cm-244	<2.9E-04	(1)	(1)
Tritium	<3.4E-03	(1)	(1)
I-129	<1.5E-05	(1)	(1)
C-14	<1.2E-04	(1)	(1)

 Table 14.
 Radionuclide Analyses by Separation and Counting Methods

Notes:

- (1) All values less than detection.
- (2) One value reported as detectable.
- (3) Three of four values reported as less than detection. One of four values detected at $\sim 45 X$ the detection limit. See text.

	AZ-102 Rad		AZ-102 Rad D	Dup	AZ-102 Surrog	ate	AZ-102 Surrog	gate Dup
	(ug/L)	wt%	(ug/L)	wt%	(ug/L)	wt%	(ug/L)	wt%
mass 230	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07
mass 231	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07
mass 232 (Th)	63.357	1.27E-03	54.201	1.08E-03	48.579	9.72E-04	54.715	1.09E-03
mass 233	0.052	1.04E-06	0.054	1.08E-06	< 0.043	< 8.56E-07	0.045	8.93E-07
mass 234 (U)	0.093	1.86E-06	0.079	1.58E-06	0.063	1.26E-06	0.052	1.03E-06
mass 235 (U)	0.715	1.43E-05	0.637	1.27E-05	0.501	1.00E-05	0.565	1.13E-05
mass 236 (U)	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07
mass 237 (Np)	0.589	1.18E-05	0.537	1.07E-05	0.056	1.12E-06	0.084	1.67E-06
mass 238 (Pu & l	J 91.364	1.83E-03	85.658	1.71E-03	66.986	1.34E-03	75.619	1.51E-03
mass 239 (Pu)	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07
mass 240 (Pu)	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07	< 0.043	< 8.56E-07

Table 15. ICP-MS Analyses for Actinides in AZ-102 Glasses

Table 16. Minimum Detectable Activities from Gamma Scan Analyses

Radionuclide	uCi/mL	Ci/g	Ci/m³
K-40	1.12E-04	2.14E-08	5.52E-02
Co-60	7.55E-06	1.44E-09	3.72E-03
Zn-65	1.42E-05	2.71E-09	7.00E-03
Rb-84	8.40E-06	1.60E-09	4.14E-03
Sr-85	7.87E-06	1.50E-09	3.88E-03
Nb-94	6.40E-06	1.22E-09	3.15E-03
Zr-95	1.13E-05	2.15E-09	5.57E-03
Ru-106	5.25E-05	1.00E-08	2.59E-02
Sb-125	1.36E-05	2.59E-09	6.70E-03
Sb-126	3.86E-06	7.36E-10	1.90E-03
Sn-126	8.44E-06	1.61E-09	4.16E-03
Cs-134	4.22E-06	8.05E-10	2.08E-03
Ce-144	1.87E-05	3.57E-09	9.22E-03
Eu-152	3.04E-05	5.80E-09	1.50E-02
Eu-154	5.36E-06	1.02E-09	2.64E-03
Eu-155	1.08E-05	2.06E-09	5.32E-03
TI-208	7.96E-06	1.52E-09	3.92E-03
Bi-212	8.16E-05	1.56E-08	4.02E-02
Pb-212	6.12E-06	1.17E-09	3.02E-03
Bi-214	1.13E-05	2.15E-09	5.57E-03
Pb-214	1.13E-05	2.15E-09	5.57E-03
Ra-226	9.60E-05	1.83E-08	4.73E-02
Ac-228	1.17E-04	2.23E-08	5.77E-02
Pa-231	1.33E-04	2.54E-08	6.56E-02
Pa-233	7.31E-06	1.39E-09	3.60E-03
Pa-234	1.24E-05	2.36E-09	6.11E-03
Th-234	1.06E-04	2.02E-08	5.23E-02
U-235	7.11E-06	1.36E-09	3.50E-03
Np-237	7.30E-06	1.39E-09	3.60E-03
Np-239	1.10E-05	2.10E-09	5.42E-03
Am-241	1.87E-05	3.57E-09	9.22E-03
Am-243	8.13E-06	1.55E-09	4.01E-03
Cm-243	2.87E-05	5.47E-09	1.41E-02
Cm-245	8.77E-06	1.67E-09	4.32E-03

Notes: μ Ci/mL values for ~ 0.5g glass dissolved in 0.1 L Ci/m³ values calculated using measured glass density of 2.59 g/cc

2.4.3 Comparison of Measured versus Expected Radionuclide Concentrations in AZ-102 Glass

The calculated radionuclides in the AZ-102 glass from Table 12 can be compared to the measured values from Table 13 and Appendix C. Table 17 shows the calculated range of expected Cs-137, Sr-90 and Tc-99 radionuclides in the AZ-102 glass. These data represent the average and range of expected radionuclides using analytical data from the pretreated AZ-102 supernatant.¹ The measured average values and ranges for these radionuclides in the AZ-102 glass are also shown in Table 17.

The measured average Cs-137 in the AZ-102 glass of 0.98 Ci/m³ is 78% of the average expected Cs-137 of 1.25 Ci/m³. Measured Sr-90 agrees well with the expected amount, as the measured average Sr-90 of 1.22 Ci/m³ is 97% of the expected 1.26 Ci/m³. Both radiochemical separation and counting measurements on the pretreated AZ-102 supernatant and the dissolved AZ-102 glasses gave Tc-99 values that were reported less than the detection limit. Using the value from measured Tc-99 in the supernatant, an expected upper limit value of <9.5E-3 Ci/m³ is obtained vs. the measured upper limit value of <2.7E-3Ci/m³. The ICP-MS measurements from the AZ-102 supernatant were more sensitive for Tc-99, giving an expected concentration in the glass of 1E-4 Ci/m³. However, measured values for Tc-99 in the dissolved glasses were complicated by presence of interference from certain ICP-MS standards measured on the ICP-MS instrument before the dissolved glass samples were measured. This is evident from the fact that similar integrated areas for the mass-99 peak were found for all three glasses analyzed (radioactive AZ-102, surrogate AZ-102 and the reference LRM glass) as well as the dissolution blank with no glass added (See Appendix C, lower data set of Table C-1). Thus the reported average values for Tc-99 in the dissolved glasses of 0.041 Ci/m³ are biased high.

	Average						Average				
	Calc.		Range				Meas.		Range		
Radionuclide	AZ-102		Low		High		AZ-102		Low		High
	Ci/m ³		Ci/m ³		Ci/m ³		Ci/m ³		Ci/m ³		Ci/m ³
Cs-137	1.25		1.21		1.29		0.98		0.95		1.00
Sr-90	1.26		1.10		1.41		1.22		0.78		1.66
Tc-99 (ICP-MS)	0.00010		0.00009		0.00011		0.041		0.0097		0.073
Tc-99 (Sep./Count)	< 0.0095	<	0.0086	<	0.0104	<	0.0027	<	0.0006	<	0.0048

 Table 17.
 Comparison of Measured versus Expected Radionuclide Concentrations in AZ-102 Glass

2.4.4 Comparison to Class C Limits

The measured radionuclides in the AZ-102 glass can be used to determine the low activity waste classification of the glass per the Code of Federal Regulations 10 CFR 61.55¹³. The WTP ILAW product specification 2.2.2.8 'Radionuclide Concentration Limits' in the WTP Contract¹² specifies that the radionuclide concentration of the ILAW form shall be less than Class C limits as defined in 10CFR61.55. The 10 CFR 61.55 Class C limits apply to long-lived radionuclides as a group separate from short-lived radionuclides. Guidance is also given for determining the waste classification of a waste form containing both long-lived and short-lived radionuclides.

Table 18 shows the various radionuclide levels specified by 10 CFR 61.55. The column labeled '10 CFR 61.55 Table 1' applies to classification as determined by long-lived radionuclides. The column labeled '10 CFR 61.55 Table 2, Column 3 Class C limits' applies to classification as determined by short-lived radionuclides. The column of measured radionuclides from the AZ-102 LAW glass shows data determined in this study. (See Table 13 through Table 16 and Appendix C.) The TRU amounts measured in the AZ-102 glass of 2.3 nCi/g are higher than 1% of the 100 nCi/g Class C limit shown. However, this value of 2.3 nCi/g is well below the Class C limit of 100 nCi/g. Since the long-lived radionuclides were less than the Class C limits, long-lived radionuclides met the contract requirements. Considering the short-lived radionuclides, it is necessary to determine the sum of fractions by dividing each nuclide's concentration by the appropriate Class C limit and add the resulting values. The sum of the fractions for the Class C column must be less than 1.0. The final column of Table 18 clearly shows that sum of fractions as determined vs. the Class C limits is only 4.8E-4 which is orders of magnitude lower than 1.0.

Radionuclide	10 CFR 61.55 Table 1 (Ci/m ³)	10 CFR 61.55 Table 2, Column 3 Class, C limits (Ci/m ³)	Measured (Ci/m ³)	Fractions and Sum of Fractions
Long-lived:				-
C-14	8	-	< 1.2 E-4	-
Tc-99	3	-	< 2.7 E-3	-
I-129	0.08	-	< 2.0 E-5	-
Transuranics	100 (nCi/g)	-	2.3 (nCi/g)	-
(TRU)*				
Pu-241	3,500 (nCi/g)	-	<5.2 (nCi/g)	-
Cm-242	20,000 (nCi/g)	-	< 0.054 (nCi/g)	-
Short-lived:	· · · · · ·		· · · · ·	
Н-3	-	(no limit)**	< 3.9 E-3	-
Co-60	-	(no limit)**	< 3.7 E-3	-
Ni-63	-	700	< 6.8 E-2	9.7E-5 (=6.8E-2/700)
Sr-90	-	7000	1.2	1.7E-4 (=1.2/7000)
Cs-137	-	4600	0.98	2.1E-4 (=0.98/4600)
				Sum = 4.8E-4

Table 18.	Code of Federal	Regulation	Class C I	Radionuclide Limits
1 abic 10.	Couc of Feueral	Regulation		

* The transuranics are estimated from the measured total alpha and Pu-241. Total alpha could contain counts from uranium alpha-emitting species (U-233,234,235,236) that are not transuranic species.

** No limits are established for these radionuclides in Class C wastes. Practical considerations such as the effects of external radiation and internal heat generation on transportation, handling, and disposal will limit the concentrations for these wastes. (See 10 CFR 61.55.)

2.5 RESULTS OF PCT ON AZ-102 GLASS

The data table in this section shows the results of the standard ASTM C 1285 –97 PCT¹⁴ on the radioactive AZ-102 quenched glass, the surrogate AZ-102 quenched glass, and the AZ-102 container cooled glass. The quenched glass PCT results were obtained after the initial crucible vitrifications using quenched cooled glasses. After a portion of the quench-cooled glasses were remelted and container centerline cooled, a separate PCT was performed. This durability test is commonly called the Product Consistency Test (PCT) and is performed at 90°C. The procedure for PCT-A of the ASTM C 1285-97 was strictly followed for this test. Triplicate samples of the AZ-102 glass and, as prescribed by the procedure, triplicate blanks, were used. A Low Activity Reference Material (LRM)¹⁰ glass and an Analytical Reference Material (ARM)¹⁵ glass were also leached in each of the tests with the AZ-102 glasses.

In the WTP contract¹², it is required to subject the AZ-102 glass to the PCT and report the results for B, Si, and Na for the AZ-102 glass. Section 2.2.2.17.2 of Mod. No. A029 of the contract specifies that in the PCT, the glass shall have a normalized mass loss less than 2 g/m^2 (2 grams of glass per square meter of exposed surface area of glass tested in a 90°C PCT) based on each of the elements B, Si, and Na. The LRM glass and the standard (ARM) glass were also tested with the AZ-102 glass in both the quenched AZ-102 glass PCT and the container cooled AZ-102 glass PCT to confirm that the test conditions for the PCT were properly controlled.

Table 19 gives the average concentrations in parts per million (ppm) of B, Si, and Na, in the final leachates after the tests. The range of the final pH values of the leachates is also presented. The concentrations have been corrected for the acidification dilutions of the leachates as required by the ASTM procedure. The raw data that are the bases of these averages are presented for the quenched glasses and the container-cooled glasses in Appendix D. The last row of the table presents the consensus average results of the PCT of a round robin on the LRM glass involving six different laboratories.¹⁰ As can be seen, the concentrations measured in both the quenched glass PCT (LRM(a)) and the centerline cooled glass PCT (LRM(b)) for LRM glass were similar to the consensus concentrations.

The results for the blanks indicate that contamination of the leachates from possible impurities in the water or on the stainless steel vessels was negligible. The results for the standard ARM-1 glass were compared to a control chart based on results for previous Product Consistency Tests on this standard glass.¹⁶ This comparison is part of the ASTM procedure. The results were between the lower and upper control limits (See Appendix D for PCT data sheet on ARM glass for quench-cooled PCT glass testing and container-cooled PCT glass testing) indicating that all the test conditions were properly controlled. Standard solutions containing B, Si, and Na were submitted for analysis with the leachates. The measured results agreed within 10% of the known values (see Appendix D) indicating that the analyses were sufficiently accurate. Thus the results of the PCT are acceptable.

Sample ID	Concentration (ppm), or Normalized release (g glass / m ²)	В	Si	Na	pH Range
Blanks(a)	(ppm)	< 0.031	< 0.018	0.225	7.0-7.0
Blanks(b)	(ppm)	< 0.061	< 0.046	0.058	5.4-6.8
ARM(a)	(ppm)	19.8	67.1	40.1	9.5-9.5
ARM(b)	(ppm)	17.0	62.1	36.1	9.4-9.4
AZ-102(a) Surrogate	(ppm)	17.7	45.3	15.29	8.9-9.0
AZ-102(a) Surrogate	g glass / m ²	0.219 ± 0.004	0.097 ± 0.001	0.203 ± 0.005	-
AZ-102(a) Radioactive	(ppm)	16.1	42.4	11.8	8.9-9.0
AZ-102(a) Radioactive	g glass / m ²	0.199 ± 0.002	0.091 ± 0.001	0.159 ± 0.002	-
AZ-102(b) Radioactive	(ppm)	12.8	37.1	10.1	8.9-9.0
AZ-102(b) Radioactive	g glass / m ²	0.159 ± 0.006	0.079 ± 0.002	0.137 ± 0.006	-
LRM(a)	(ppm)	23.7	78.5	149.5	10.1-10.2
LRM(a)	g glass / m ²	0.506 ± 0.015	0.173 ± 0.006	0.482 ± 0.016	-
LRM(b)	(ppm)	19.6	72.5	132.4	10.1-10.1
LRM(b)	g glass / m ²	$\begin{array}{c} 0.420 \pm \\ 0.040 \end{array}$	0.160 ± 0.030	0.427 ± 0.020	-
LRM(c)	(ppm)	26.7	82.0	159.7	11.7
LRM(c)	g glass / m ²	0.570	0.170	0.450	-

Table 19. Results from 90°C PCT for Quenched and Centerline Cooled AZ-102 Glass

(a) Based on triplicate tests on quenched AZ-102 glass PCT.
(b) Based on triplicate tests on centerline cooled AZ-102 glass PCT.
(c) Published consensus values for LRM glass.¹⁰

The final pH is an approximate indication of the durability of the glass in a PCT. The higher the final pH, the lower the durability. The measured concentrations are a much more accurate indication. Normalized mass losses are the best indication of the durability of a glass in a PCT. Normalization accounts for the concentration of an element in the glass. The normalized release is a measure of the total mass of glass leached in a PCT based on a specific element in the glass. The specification for ILAW glass is that the normalized mass losses based on B, Si, and Na, shall each be <2 grams of glass per square meter of exposed surface area of glass tested in a 90°C PCT for 7 days.¹² In the PCT, the glass is carefully sieved through standard mesh size sieves so that the surface area of the glass is reproducible from test to test. The sieved glass is also washed with both water and alcohol to remove any glass fines. This also helps to ensure a reproducible surface area of the powdered glass. The exposed surface area of the glass in a PCT has been estimated by assuming that the particles are spherical and that the distribution of particle sizes is Gaussian.¹⁷ The size of the holes in the 100 and 200 mesh sieves are 0.149 mm and 0.074 mm, respectively. Thus the diameter of the spheres range between these two values with an average value of 1.12×10^{-4} m. Based on these assumptions the exposed surface area has been calculated to be 0.02 m² per gram of sieved glass.

The normalized mass loss in terms of grams of glass leached is calculated using the following equation

$$NR_i = (C_i/C_{ig})/0.02X10^3$$

Where:

NR_i = normalized release based on element i, in grams of glass leached per square meter of glass exposed in the PCT

 C_i = the concentration of element i in ppm in the leachate

 C_{ig} = the weight percent of element i in the glass.

The PCT procedure prescribes that for every gram of glass, there is exactly 10 mL of leachate; thus there is 0.02 m^2 of glass surface area per 10 mL of leachate. The factor of 1000 in the denominator results from C_i being in ppm, C_{ig} in weight percent, and the test condition of 10 mL per 0.02 m^2 of glass.

Table 19 presents the normalized releases calculated from the PCT data and the measured composition of the AZ-102 glass (see Table 7 and Table 8 for the AZ-102 glass composition). Table 19 presents the averages and standard deviations based on triplicate tests. The normalized releases for all three elements are less than the upper limit of 2 g glass/m² specified in the WTP ILAW product specification 2.2.2.17.2 Product Consistency Test.¹² Thus the glass meets this specification. If one takes the average normalized release for all three AZ-102 glasses (surrogate quenched, rad quenched and rad CCC) for each element (B, Si, Na) and divides each of these averages by the specification limit of 2 g glass/m₂, then these ratios show that the AZ-102 glasses have normalized release for B for AZ-102 glasses is 0.192 g glass/m². This value divided by 2 gives 0.1, or the normalized release for B for the AZ-102 glasses is only 10% of the upper limit of 2 g glass/m².

Table 19 also shows similar normalized results for the LRM glass calculated from the PCT data and the measured composition of the LRM glass (see Table 7 and Table 8 for the LRM glass composition). The average normalized releases for the LRM glass (B, Si, Na) in the later PCT performed on CCC AZ-102 glass (LRM(b)) are lower than the initial LRM normalized release from the PCT performed on quenched glasses (LRM(a)). However, if one normalizes the LRM normalized releases on the basis of the ARM leach data, the normalized releases from LRM from the two different PCTs are the same within a 95% confidence level. For example using B, the ARM(b) test showed release of 17 ppm and the ARM(a) test showed slightly higher B release of 19.8 ppm. Taking the ratio of ARM(b)/ARM(a) and multiplying by the normalized release of B for LRM(a), one gets (17ppm/19.8ppm)*0.506 g glass/m² = 0.434 g glass/m². This value is similar to the normalized B release from LRM(b) = (0.420 +/- 0.040 g glass/m²) to within the 95% confidence level. Similar results are obtained for Si and Na.

Comparison of the normalized releases for the various AZ-102 glasses (surrogate quenched, rad quenched and rad CCC) is important to show that the durability of these glasses are consistent. For instance, the WTP Contract Section C, Standard 2, Research, Technology, and Modeling, (a), (3), (v) Immobilized Low-Activity Waste Qualification Testing (C) indicates that non-radioactive glasses may be used for the leachability index ANSI/ANS-16.1 procedure and the Vapor Hydration Test, "provided that the results from 2.2.2.17.2 (PCT) are consistent for the non-radioactive glass and the radioactive glass".¹²

If one performs a statistical analysis on the normalized release for the AZ-102 glasses for each element B. Si and Na, the analysis shows that the average normalized release for these three data sets are different for each B, Si and Na element to within the 95% confidence level. This was done with the professional statistical software package JPM[®]: Statistics and Graphics, Version 5.¹⁸ Scaling the data sets to the slightly different ARM releases also renders average normalized release values for the AZ-102 glasses that are statistically different (except for normalized sodium release). However, even though the average normalized releases from the three different AZ-102 glasses are thus shown to be statistically different, these differences are relatively small. This can be shown by taking the average and standard deviation of the average normalized releases for the three AZ-102 glasses for each element B, Si and Na. Table 20 shows these data. It can be seen that the percent relative standard deviation (%RSD) of the averages (= (St.Dev./Average)*100) for each element is in the range of 10% to 20%. Thus while the average normalized releases from the three different AZ-102 glasses can be shown to be statistically different, this difference is very small and one can conclude that the normalized B, Si and Na releases are consistent in that the three glasses leach to within a factor of +/-20%.

	Average B Normalized Release, (g glass/m ²)	Average Si Normalized Release, (g glass/m ²)	Average Na Normalized Release, (g glass/m ²)
AZ-102 Surrogate Quench	0.219	0.097	0.203
AZ-102 Rad Quench	0.199	0.091	0.159
AZ-102 Rad CCC	0.159	0.079	0.137
Average	0.192	0.089	0.166
St.Dev.	0.031	0.009	0.034
%RSD	15.9	10.3	20.2

Table 20.Averages of the PCT

2.6 MEASURED DOSE RATES OF THE RADIOACTIVE AZ-102 CONTAINER-COOLED LAW GLASS

The dose rate was measured for a portion of the container-cooled radioactive AZ-102 glass. A 40.2 g section of the container-cooled 110 g glass monolith was packaged in a doublelayered plastic wrap and removed from the radiochemical hood. This sealed glass piece was transferred to a relatively low background area in close proximity of the SRTC radiochemical laboratories. The glass piece was then put in contact with an Eberline RO-20 dose rate instrument in an open-window configuration. The RO-20 dose rate instrument used for these measurements was calibrated and maintained by SRTC radiological control personnel.

The dose rate measured for the 40.2 g piece of radioactive AZ-102 glass was 160 mrem/hr. Although the sealed glass piece was put in contact with the open window of the RO-20 dose rate instrument, the glass piece was actually about five centimeters from the counting surface that is located inside of the RO-20 instrument window. Similar measurements performed with the AZ-102 glass in contact with the closed-window configuration of the RO-20 instrument registered no detectable dose above the background, which was measured to be approximately 0.3 mrem/hr.

These data for the open-window and closed-window dose rates indicate that the radiation dose is primarily derived from the beta-emitters contained in the radioactive AZ-102 glass matrix, i.e., Cs-137, Sr-90 and Y-90. These measurements were repeated (open and closed-window) for the same glass piece with the same RO-20 instrument located approximately 30 cm away from the glass piece. No detectable dose was indicated above the 0.3 mrem/hr background reading for either the open-window or the closed-window at the 30 cm distance.

The dose rate of 160 mRem/hr measured on this unshielded small piece of AZ-102 glass is well below the WTP Contract ILAW product specification 2.2.2.9 Dose Rate Limitations¹² limit of <500 mRem/hr on the outside of a full LAW container made of 304-L stainless steel. Computational methods involving the measured radionuclides in the AZ-102 glass, the glass mass and geometry, and the container shielding could be applied to estimate the actual dose rate on the outside of a LAW container if it were filled with this particular AZ-102 glass made to the LAWB88 formulation.

2.7 CRYSTALLINE AND NON-CRYSTALLINE PHASE DETERMINATION

X-ray diffraction analysis of a portion of the glass powder used for the PCT was performed on the container cooled glass. Figure 7 shows the x-ray diffraction pattern. No observable peaks attributed to any crystalline structure are present in this scan indicating that the glass is amorphous.

Figure 8 through Figure 17 show SEM photographs of the glass powders obtained from grinding the AZ-102 glass in preparation for the PCT. These glass powders were obtained during preparation of the glass for PCT durability tests using a Techmar tungsten blade grinder with stainless steel grinding compartment. Images obtained from backscattered electron (BSE) and secondary electron (SE) were obtained for increasing levels of magnification from 50X to 250X to 500X to 2000X. Generally, the SEM technique uses backscattered electrons or incident electrons to indicate potential density differences in the image particles. Use of secondary electron imaging that involves electrons from the matrix material provides topography images of the matrix.

Figure 8 and Figure 13 show the 50X magnification of the glass powders. These photos show that the glass particles are all very similar in size due to the sieving of the glass powders in preparation for the PCT. These glass particles were passed through a 100 mesh sieve and retained by the 200 mesh sieve. Figure 9 through Figure 12 and Figure 14 through Figure 17 show higher magnification of the glass powders. Very fine particles can be seen on each individual glass particle. These finer particles are present because these glass powders were sampled from the PCT glass powder to be imaged before they were washed free of the glass fines. Figure 9, Figure 10, and Figure 12 have certain very fine particles indicated as spots A, B, C and D. The energy dispersive x-ray analyses of these particular spots are shown in Figure 18 through Figure 21.

Figure 18 shows the elements present in spot A (See Figure 10 and Figure 12) mainly as iron and chromium possibly from steel contamination from the grinder compartment. Figure 19 shows the elements present from the area indicated as spot B in Figure 10 and Figure 12. The elements present are indicative of the major elements associated with the glass matrix such as Si, Ca, Fe and Zn. The gold and palladium indicated in this spectrum derive from the Au/Pd coating used to mount the samples. Figure 20 shows the elements present from the area indicated as spot C in Figure 9. This spot is similar to spot A and the elements iron and chromium are also indicative of steel contamination from the grinder. Figure 21 shows the elements present from the area indicated as spot D in Figure 10 and Figure 12. This spot is likely a glass fine particle since the elements of Si, Ca and Fe from the glass matrix are present. This EDAX spectrum is similar to the Figure 19 spectrum of the larger glass particle matrix.



Figure 7. XRD Scan of the AZ-102 Glass Powder



Figure 8. SEM Image of the AZ-102 Glass Powders with BSE at 50X.



Figure 9. SEM Image of the AZ-102 Glass Powders with BSE at 250X Spot C in this figure is discussed in the text.



Figure 10. SEM Image of the AZ-102 Glass Powders with BSE at 500X Spots A, B, and D in this figure are discussed in the text.



Figure 11. Image of the AZ-102 Glass Powders with BSE at 500X



Figure 12. SEM Image of the AZ-102 Glass Powders with BSE at 2000X Spots A, B, and D in this figure are discussed in the text.



Figure 13. SEM Image of the AZ-102 Glass Powders with SE at 50X



Figure 14. SEM Image of the AZ-102 Glass Powders with SE at 250X



Figure 15. SEM Image of the AZ-102 Glass Powders with SE at 500X



Figure 16. SEM Image of the AZ-102 Glass Powders with SE at 500X



Figure 17. SEM Image of the AZ-102 Glass Powders with SE at 2000X



Figure 18. EDAX Spectrum of Spot A from Figure 10 and Figure 12



Figure 19. EDAX Spectrum of Spot B from Figure 10 and Figure 12



Figure 20. EDAX Spectrum of Spot C from Figure 9



Figure 21. EDAX Spectrum of Spot D from Figure 10 and Figure 12

3.0 CONCLUSIONS

This proof-of-technology demonstration for the Hanford River Protection Project (RPP) Waste Treatment and Immobilization Plant (WTP) has successfully produced crucible-scale glass using a reformulated recipe for the AZ-102 LAW sample. This reformulation (LAWB88) involved decreasing Fe, Li, Mg and Ti, and increasing B, Ca, Si and Zn from the original AZ-102 formulation (LAWB53). The ~ 110g glass monoliths produced in this crucible study with rapid cooling contained no visible crystalline phase on any surface of the glass. Dissolution of these reformulated, rapidly cooled AZ-102 LAW glasses and analyses of the resulting dissolved glasses provided measured glass species at close to target values for both the radioactive and the surrogate AZ-102 glass. The target waste loading in these reformulated AZ-102 glasses was nominally 5 wt% Na₂O with no sodium added as glass forming minerals. Measured densities for the AZ-102 glass were approximately 2.6 g/cc and the measured density of the LRM glass was approximately 2.5 g/cc compared to a reference value of 2.51 g/cc.

Radionuclide measurements of the dissolved glasses also show that Cs-137, Sr-90 and Tc-99 were all at predictable levels in the glass and all radionuclides were below the BNI-WTP contract upper limits. The Cs-137 levels in this AZ-102 LAW glass are below the contract specification of 3 Ci/m³, but the measured levels of 1 Ci/m³ are above the current engineering target of 0.3 Ci/m³. Transuranic levels in the AZ-102 glass were measured to be 2.3 nCi/g, which is well below the contract specification of 100 nCi/g for LAW glasses.

The product durability of these AZ-102 reformulated glasses have been tested by the PCT and the leach results indicate that all species are released at normalized levels below the BNI-WTP contract upper limit specification of 2 g/m². The reference LRM glass and the PCT reference ARM glass leach results have been compared to previous PCT results. These data show that the PCT was performed with proper control of all test conditions.

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APPENDIX A. NOTES ON COMPARISON OF RESULTS ON LAWB53 AND LAWB88 FOR AZ-102 SRTC ACTUAL WASTE SAMPLE, THE CATHOLIC UNIVERSITY OF AMERICA, VITREOUS STATE LABORATORY, REV. 0, 7/31/02

Glass formulation LAWB53 was developed previously for the vitrification of an actual LAW sample from AZ-102 at SRTC; this LAW stream is very high in sulfate. Although no crystallization was found in the simulant glass on container cooling heat treatment, crystals were found at the bottom of the SRTC actual waste melt. A sample of that glass was shipped to VSL, where it was determined that the material was a platinum-nucleated crystalline pyroxene phase that formed as a result of interaction with the platinum crucible. SRTC subsequently performed additional analysis of their waste sample, particularly with respect to organic speciation. When glasses were prepared at VSL using a simulant based on the revised waste characterization data, the same platinum-nucleated crystallization was reproduced upon container-cooling heat treatment. Thus, the simulant and actual waste data were ultimately in agreement. Although this artifact has no bearing on actual WTP production glasses, it does present a practical issue for fabricating representative samples from AZ-102 using crucible melts for subsequent testing and characterization. In order to address this issue, VSL has investigated revised glass formulations.

The previously tested formulation, LAWB53, and a new proposed formulation, LAWB88, have been prepared and tested in parallel from AZ-102 simulant formulations in order to compare the properties of the two glasses, particularly upon container-cooling heat treatment. Note that for consistency, and since the Project has not supplied a revised LAW container cooling profile, the same profile that was used for the original actual waste melt was used in the present work.

Two simulant recipes were tested:

- A simulant developed by Russ Eibling of SRTC and described in WSRC-TR-2001-00395 DRAFT, Appendix D-3 (page 95), referred to herein as "SIM1"
- A revised and WTP-approved simulant recipe, also developed by Russ Eibling, sent in late November 2001, which differs slightly from the above simulant. This simulant at 2.77 Molar Na was slightly lower in organics and used mostly sodium salts of organic acids. This simulant was approved by Reid Peterson of the WTP for use in pretreatment testing (referred to herein as "SIM2"), as confirmed by D. Swanberg (WTP).

Simulant SIM1, which was used at SRTC and later at VSL in 2001, again confirmed the formation of a pyroxene phase at the platinum interface after melting the LAWB53 formulation followed by a container-cooling heat treatment. Since platinum seeds were found in the middle of the pyroxene crystals, it was concluded that chemical reactions of organic compounds and sulfate species resulting in attack of the platinum favored the growth of crystals that would otherwise be expected only at lower temperatures and longer heat treatment times. Heat treatments at 700°C for 20 hours also revealed these pyroxene crystals, but at much lower concentration.

The testing was repeated in July 2002 at VSL for various new formulations and also included several repetitions of LAWB53. The platinum-nucleated pyroxene crystallization in LAWB53 was again confirmed (see recent results with LAWB53 in Figure A- 1 and Figure A- 2).

The absence of crystals with the new proposed batching recipe, LAWB88, was verified by SEM after three successive testings and multiple heat treatments, as low as 700°C (see Figure A- 3 and Figure A- 4).

Simulant SIM2 was also tested for both batching recipes and revealed a much lower amount of the pyroxene with LAWB53 and none in LAWB88. Again, as was found in the work performed in 2001, the simulant formulation, and particularly the amount and composition of the organics, appear to significantly affect the formation of these crystals. As a result, it must be noted that conclusions drawn from the present work as to the behavior of the actual waste sample are only as reliable as the actual waste sample characterization data and the accuracy of the simulant formulation. With this caveat, the present recommendation for the SRTC AZ-102 actual waste melt is to use the LAWB88 formulation presented in Table 2 of this report.



Figure A-1. SEM Backscattered Electron Image of LB53SRCC1 Using SIM1

This photo shows LAWB53 glass after container cooling. The crystalline bottom layer shows the platinum-nucleated pyroxene phase reaching a few volume%.





This photo shows LAWB53 after container cooling. The crystalline bottom layer shows fewer pyroxene crystals.



Figure A- 3. SEM Backscattered Electron Image of LB88SRCC2 Using SIM1

This photo shows LAWB88 after container cooling. The bottom layer shows no pyroxene present.



Figure A-4. SEM Backscattered Electron Image of LAWB88

This photo shows LAWB88 glass heat-treated at 700°C for 20 hours after 1 hour premelt at 1200°C. The bottom layer shows no crystallization.

APPENDIX B. WTP CONTAINER COOLING



Memorandum

To:	Chris Musick	Date:	October 16, 2003
From:	Lawrence Petkus	CCN:	074181
Ext:	371-8436		
Fax:	371-8346		

Subject: LAW Container Centerline Cooling Data

- References: 1) 24590-LAW-TSP-RT-03-001 Test Specification "Filling Prototypic LAW Containers with Glass" Rev. 0. June 12, 2003.
 - 2) TP-PLT-038 "RPP-WTP Pilot Melter Prototypic LAW Container and HLW Container Glass Fill Test Plan," Rev. 0, Duratek Inc. August 8, 2003.
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Testing has been completed where prototypic LAW containers were filled with glass to collect container centerline cooling (CCC) data. Container centerline cooling data was taken in accordance with references 1 and 2. The container-cooling portion of the test was done in accordance with a supplier Quality Assurance program compliant with NQA-1 (1989). In the test, three instrumented containers were filled with glass to greater than 95% fill height. The first two containers were filled at a production rate of 15 metric tons of glass per day and the last container; LT-003, was filled at 20 metric tons of glass per day. There was no discernable difference in the cooling profiles between the two fill rates.

To recommend a CCC profile for glass testing, the worst case, *i.e.* slowest, cooling curve was selected from the available data. Cooling times to 400 C for the container, ranged from 3459 minutes to 3710 minutes. The cooling data, shown in Figure 1, is taken from container LT-002, collected at one-minute intervals. The thermocouple was positioned at the centerline 54 inches from the bottom of the container. The peak temperature of 1114 °C was taken from the peak among the centerline data presented, which occurs in container LT-001. The cooling profile has been plotted over the data, with black diamonds, showing seven straight-line cooling segments. The segments were selected to reproduce the cooling profile at that point in the container. Table 1 includes the CCC profile for crucible testing, which details each cooling segment.

Although the centerline cooling data was contained in the test summary report, reference 3, the final and complete test results will be issued in February, 2004. A complete description of the filling and cooling will be available at that time.



Figure 1. Time-temperature profile of LAW Container centerline at 54" along with line segments defined in Table 1.

SEGMENT	TIME min	START TEMP. °C	RATE °C/min				
1	0-16	1114	-7.125				
2	16-73	1000	-1.754				
3	73-195	900	-0.615				
4	195-355	825	-0.312				
5	355-640	775	-0.175				
6	640-1600	725	-0.130				
7	1600-3710	600	-0.095				

Table 1. LAW CCC Profile for Crucible Testing

Lawrence Petkus Research Engineer Research and Technology

R&T/llp

PDC	MS11-B	Р
Barnes, SM	MS1-B	Р
Carl, DE	MS7-ESW	V
Larson, DE	MS1-B	V
Lee, ED	MS1-A	Т
Perez, JM	MS1-B	

Peters, RD	MS7-ESW
Prindiville, K	MS1-B
Viena, JD	MS1-B
Wesick, JH	MS1-B
Tamosaitis, WL	MS1-B

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APPENDIX C. MEASURED RADIONUCLIDE CONCENTRATIONS RADIOACTIVE, SURROGATE, AND BLANK SOLUTIONS

			Cs-137							Sr-90						
			uCi/mL	Ci/g		Ci/m ³				dpm/mL		uCi/mL		Ci/g		Ci/m ³
Peroxide	Rad		1.96E-03	3.74E-07		9.66E-01		-		5.03E+03		2.27E-03		4.32E-07		1.12E+00
Fusions	Rad		1.96E-03	3.86E-07		9.99E-01		-		8.15E+03		3.67E-03		7.24E-07		1.87E+00
	Surrogate	<	8.10E-06	< 1.46E-09	<	3.77E-03		-	<	9.77E+02	<	4.40E-04	<	7.91E-08	<	2.05E-01
	Surrogate	<	7.30E-06	< 1.42E-09	<	3.66E-03		-	<	9.58E+02	<	4.32E-04	<	8.38E-08	<	2.17E-01
	Reagblnk	<	7.72E-06	-		-		-	<	1.12E+03	<	5.04E-04	-	-	-	-
Acid	Rad		2.00E-03	3.86E-07		9.99E-01		-		4.23E+03		1.91E-03		3.68E-07		9.52E-01
Dissolutions	Rad		1.85E-03	3.63E-07		9.39E-01		-		4.10E+03		1.85E-03		3.62E-07		9.37E-01
	Surrogate	<	7.30E-06	< 1.44E-09	<	3.73E-03		-	<	1.13E+03	<	5.09E-04	<	1.01E-07	<	2.60E-01
	Surrogate	<	6.38E-06	< 1.25E-09	۷	3.24E-03		-	۷	9.98E+02	۷	4.50E-04	<	8.82E-08	<	2.28E-01
	Reagblnk	<	8.82E-06	-		-		-	۷	1.09E+03	۷	4.91E-04	-	-	-	-
			Alpha							Beta						
			dpm/mL	uCi/mL		Ci/g		Ci/m ³		dpm/mL		uCi/mL		Ci/g		Ci/m ³
Peroxide	Rad		17.2	7.75E-06		1.48E-09		3.82E-03		1.42E+04		6.40E-03		1.22E-06		3.15E+00
Fusions	Rad		16.8	7.57E-06		1.49E-09		3.86E-03		1.42E+04		6.40E-03		1.26E-06		3.26E+00
	Surrogate	<	8.16	< 3.68E-06	<	6.61E-10	<	1.71E-03	<	18.4	<	8.29E-06	<	1.49E-09	<	3.85E-03
	Surrogate	<	8.38	< 3.77E-06	<	7.33E-10	<	1.89E-03	<	29.2	<	1.32E-05	<	2.55E-09	<	6.60E-03
	Reagblnk	<	10.2	< 4.59E-06		-		-	<	24.6	<	1.11E-05		-		-
Acid	Rad		39.8	1.79E-05		3.46E-09		8.95E-03		1.53E+04		6.89E-03		1.33E-06		3.44E+00
Dissolutions	Rad		31	1.40E-05		2.74E-09		7.09E-03		1.47E+04		6.62E-03		1.30E-06		3.36E+00
	Surrogate	<	8.58	< 3.86E-06	<	7.64E-10	<	1.98E-03	<	29.6	<	1.33E-05	<	2.64E-09	<	6.82E-03
	Surrogate	<	7.17	< 3.23E-06	<	6.34E-10	<	1.64E-03	<	19.2	<	8.65E-06	<	1.70E-09	<	4.39E-03
	Reagblnk	<	8.19	< 3.69E-06		-		-	<	20.8	۷	9.37E-06		-		-
			Tc-99													
			dpm/mL	uCi/mL		Ci/g		Ci/m ³								
Peroxide	Rad	<	8.76E+00	< 3.95E-06	<	7.52E-10	<	1.95E-03								
Fusions	Rad	<	2.56E+01	< 1.15E-05	<	2.27E-09	<	5.88E-03								
	Surrogate	<	7.17E+00	< 3.23E-06	<	5.81E-10	<	1.50E-03								
	Surrogate	<	8.39E+00	< 3.78E-06	۷	7.34E-10	<	1.90E-03								
	Reagblnk		9.36E+00	4.22E-06		-		-								
Acid	Rad	<	5.68E+00	< 2.56E-06	۷	4.94E-10	۷	1.28E-03								
Dissolutions	Rad	<	7.61E+00	< 3.43E-06	۷	6.73E-10	<	1.74E-03								
	Surrogate		4.18E+01	1.88E-05		3.72E-09		9.63E-03								
	Surrogate		6.25E+01	2.82E-05		5.52E-09		1.43E-02								
	Reagblnk	<	5.04E+00	< 2.27E-06		-		-								
			ug/L	uCi/mL		Ci/g		Ci/m ³								
Acid	Rad		7.64E+00	1.27E-04		2.46E-08		6.36E-02								
Dissolutions	Rad		2.24E+00	3.73E-05		7.33E-09		1.89E-02								
ICP-MS	Surrogate		5.09E+00	8.48E-05		1.68E-08		4.34E-02								
	Surrogate		5.00E+00	8.33E-05		1.63E-08		4.23E-02								
	Reagblnk		8.90E-01	1.48E-05		-		-								
	Reagbink		8.90E-01	1.48E-05		-		-								

Table C-1. Radionuclide Analyses for Cs-137, Sr-90, Tc-99, Total Alpha and Total Beta

Pu-239/240										
		dpm/mL	uCi/mL	Ci/q	Ci/m ³					
Peroxide	Rad	< 2.82E+00	< 1.27E-06	< 2.42E-10) < 6.26E-04					
Fusions	Rad	< 171F+01	< 7.70F-06	< 1.52E-09	3 < 3.92 E - 0.3					
1 dolorio	Surrogate	< 3.30E+00	< 1.49E-06	< 2.67E-10	(-2.00) < -0.01 = 0.02 = 0.00 = 0.0					
	Surrogate	< 1.95E+00	< 8.78F-07	< 1 70F-10	(-4.41) < 4.41 = -0.4					
	Readblok	< 7.59E+00	< 3.42F-06	-	-					
Acid	Rad	< 5.89E+00	< 2.65E-06	< 5.13E-10) < 1.33E-03					
Dissolutions	Rad	< 8.38E-01	< 3.77E-07	< 7.41E-11	1 < 1.92E-04					
	Surrogate	< 1.17E+00	< 5.27E-07	< 1.04E-10) < 2.69E-04					
	Surrogate	< 2.26E+00	< 1.02E-06	< 2.00E-10) < 5.16E-04					
	Reagblnk	< 2.69E-01	< 1.21E-07	-	-					
	Ŭ	Cm-242								
		dpm/mL	uCi/mL	Ci/a	Ci/m ³					
Peroxide	Rad	< 1.67E-01	< 7.52E-08	< 1.43E-11	1 < 3.71E-05					
Fusions	Rad	< 9.39E-01	< 4.23E-07	< 8.34E-11	1 < 2.16E-04					
	Surrogate	< 1.93E-01	< 8.69E-08	< 1.56E-11	1 < 4.04E-05					
	Surrogate	< 1.10E+00	< 4.95E-07	< 9.62E-11	1 < 2.49E-04					
	Reagblnk	< 6.70E-01	< 3.02E-07	-	-					
Acid	Rad	< 4.59E-01	< 2.07E-07	< 3.99E-11	1 < 1.03E-04					
Dissolutions	Rad	< 8.65E-01	< 3.90E-07	< 7.65E-11	1 < 1.98E-04					
	Surrogate	< 7.51E-01	< 3.38E-07	< 6.69E-11	1 < 1.73E-04					
	Surrogate	< 5.30E-01	< 2.39E-07	< 4.68E-11	1 < 1.21E-04					
	ReagbInk	< 4.57E-01	< 2.06E-07	-	-					
		Pu-238	•		•					
		dpm/mL	uCi/mL	Ci/q	Ci/m ³					
Peroxide	Rad	3.45E+00	1.55E-06	2.96E-10	7.66E-04					
Fusions	Rad	5.73E+00	2.58E-06	5.09E-10) 1.32E-03					
	Surrogate	< 3.02E+00	< 1.36E-06	< 2.45E-10) < 6.32E-04					
	Surrogate	3.29E+00	1.48E-06	2.88E-10) 7.44E-04					
	ReagbInk	3.96E+00	1.78E-06	-	-					
Acid	Rad	6.74E+00	3.04E-06	5.87E-10) 1.52E-03					
Dissolutions	Rad	3.45E+00	1.55E-06	3.05E-10	7.89E-04					
	Surrogate	5.31E+00	2.39E-06	4.73E-10) 1.22E-03					
	Surrogate	5.38E+00	2.42E-06	4.75E-10) 1.23E-03					
	Reagblnk	4.26E+00	1.92E-06	-	-					
		Ni-63								
		dpm/mL	uCi/mL	Ci/g	Ci/m ³					
Peroxide	Rad	< 2.89E+02	< 1.30E-04	< 2.48E-08	3 < 6.42E-02					
Fusions	Rad	< 1.68E+01	< 7.57E-06	< 1.49E-09) < 3.86E-03					
	Surrogate	< 1.11E+01	< 5.00E-06	< 8.99E-10						
	Surrogate	< 5.22E+01	< 2.35E-05	< 4.56E-09	<u>} < 1.18E-02</u>					
	Reagblnk	< 1.41E+01	< 6.35E-06	-	-					
Acid	Rad	< 7.21E+02	< 3.25E-04	< 6.27E-08	$\frac{3}{1.62 \pm 01}$					
Dissolutions	Rad	< 1.75E+02	< 7.88E-05	< 1.55E-08	$\frac{3}{3} < 4.00 \pm 0.02$					
	Surrogate	< 4.07E+00	< 1.83E-06	< 3.63E-10	$\frac{1}{2}$ < 9.37E-04					
	Surrogate	< 8.95E+01	< 4.03E-05	< 7.91E-08	j < 2.04E-02					
	Reagbink	< 1.10E+01	< 4.95E-06	-	-					
		Pu-24 I			Ci/m ³					
Porovido	Pad									
Fusions	Rad	< 1.20E+U2	< 3.03E-03		$2 < 2.70 \pm -02$					
1 0310115	Surrogate	< 1.58E+02	< 7.12F-05	< 1.28F-08	3 < 3.31F-02					
	Surrogate	< 1.00E+02	< 545F-05	< 1.06F-08	2 74F-02					
	Readblok	< 1.46F+01	< 6.58F-06	-	-					
Acid	Rad	< 2.12E+01	< 9,55E-06	< 1.84E-09	e 4.77E-03					
Dissolutions	Rad	< 2.19E+01	< 9.86E-06	< 1.94E-09	e 5.01E-03					
	Surrogate	< 3.52E+01	< 1.59E-05	< 3.14E-09) < 8.11E-03					
	Surrogate	< 5.47E+01	< 2.46E-05	< 4.83E-09) < 1.25E-02					
	ReagbInk	< 2.01E+01	< 9.05E-06		-					

Table C- 2. Radionuclide Analyses by Separation and Counting Methods
		Am-241						
		dpm/mL		uCi/mL		Ci/g		Ci/m ³
Peroxide	Rad	4.64E+00		2.09E-06		3.98E-10		1.03E-03
Fusions	Rad	< 1.07E+01	<	4.82E-06	<	9.50E-10	<	2.46E-03
	Surrogate	< 1.21E+01	<	5.45E-06	<	9.80E-10	<	2.53E-03
	Surrogate	< 7.71E+01	<	3.47E-05	<	6.74E-09	<	1.74E-02
	ReagbInk	< 8.61E+00	<	3.88E-06	<	-	<	-
Acid	Rad	< 1.97E+01	<	8.87E-06	<	1.71E-09	<	4.43E-03
Dissolutions	Rad	< 5.59E+00	<	2.52E-06	<	4.94E-10	<	1.28E-03
	Surrogate	< 1.91E+01	<	8.60E-06	<	1.70E-09	<	4.40E-03
	Surrogate	1.66E+00		7.48E-07		1.47E-10		3.79E-04
	Reagblnk	< 1.12E+01	<	5.04E-06	<	-	<	-
		Pm-147/Sm-15	51					
		dpm/mL		uCi/mL		Ci/g		Ci/m ³
Peroxide	Rad	< 3.25E+01	<	1.46E-05	<	2.79E-09	<	7.22E-03
Fusions	Rad	8.25E+02		3.72E-04		7.32E-08		1.89E-01
	Surrogate	< 2.34E+02	<	1.05E-04	<	1.90E-08	<	4.90E-02
	Surrogate	< 1.09E+01	<	4.91E-06	<	9.53E-10	<	2.46E-03
	Reagblnk	< 1.32E+00	<	5.95E-07	<	-	<	-
Acid	Rad	< 1.22E+01	<	5.50E-06	<	1.06E-09	<	2.74E-03
Dissolutions	Rad	< 1.06E+01	<	4.77E-06	<	9.37E-10	<	2.42E-03
	Surrogate	< 1.50E+01	<	6.76E-06	<	1.34E-09	<	3.45E-03
	Surrogate	< 9.77E+00	<	4.40E-06	<	8.63E-10	<	2.23E-03
	Reagblnk	< 1.32E+01	<	5.95E-06	<	-	<	-
		Cm-244						2
		dpm/mL		uCi/mL		Ci/g		Ci/m³
Peroxide	Rad	< 1.60E+00	<	7.21E-07	<	1.37E-10	<	3.55E-04
Fusions	Rad	< 1.28E+00	<	5.77E-07	<	1.14E-10	<	2.94E-04
	Surrogate	< 5.00E-01	<	2.25E-07	<	4.05E-11	<	1.05E-04
	Surrogate	< 3.66E+00	<	1.65E-06	<	3.20E-10	<	8.27E-04
	Reagblnk	< 7.29E-01	<	3.28E-07	<	-	<	-
Acid	Rad	< 4.91E+00	<	2.21E-06	<	4.27E-10	<	1.10E-03
Dissolutions	Rad	< 3.63E+00	<	1.64E-06	<	3.21E-10	<	8.30E-04
	Surrogate	< 5.10E-01	<	2.30E-07	<	4.54E-11	<	1.17E-04
	Surrogate	< 8.87E-01	<	4.00E-07	<	7.84E-11	<	2.03E-04
	Reagblnk	< 2.19E+00	<	9.86E-07	<	-	<	-
		Tritium				0		
		uCi/mL		Ci/g		Ci/m³		
Acid	Rad	< 7.79E-06	<	1.51E-09	<	3.89E-03	I	
Dissolutions	Rad	< 7.79E-06	<	1.53E-09	<	3.95E-03		
	Surrogate	< 6.68E-06	<	1.32E-09	<	3.42E-03		
	Surrogate	< 7.79E-06	<	1.53E-09	<	3.95E-03		
	Reagblnk	< 6.68E-06	<	-	<	-		

Table C-3. Additional Radionuclide Analyses by Separation and Counting Methods

Note: Tritium values determined only for Acid Dissolution as explained in text.

	I-129				C-14			
	uCi/g	Ci/g	Ci/m ³		dpm/g	Ci/g		Ci/m ³
Rad	< 5.71E-06	< 5.71E-12	< 1.48E-05	<	1.05E+02	< 4.72E-11	<	1.22E-04
Rad	< 9.53E-06	< 9.53E-12	< 2.46E-05	۷	1.05E+02	< 4.72E-11	۷	1.22E-04
Surrogate	< 1.16E-05	< 1.16E-11	< 3.00E-05	<	5.48E+01	< 2.46E-11	۷	6.37E-05
Surrogate	< 1.88E-05	< 1.88E-11	< 4.86E-05	<	5.48E+01	< 2.46E-11	<	6.37E-05

Table C- 4. Radionuclide Analyses for Iodine-129 and Carbon-14

APPENDIX D. PCT DATA SHEETS FOR QUENCH-COOLED AND CENTERLINE-COOLED AZ-102 GLASSES

- Table D-1. Quench-cooled PCT Data ARM Glass
- Table D-2. Quench-cooled PCT Data LRM Glass
- Table D-3. Quench-cooled PCT Data AZ-102 Simulant Glass
- Table D-4. Quench-cooled PCT Data AZ-102 Radioactive Glass
- Table D-5. Container Centerline-Cooled PCT Data ARM Glass
- Table D-6. Container Centerline-Cooled PCT Data LRM Glass
- Table D-7. Container Centerline-Cooled PCT Data AZ-102 Radioactive Glass

Table D- 1. Quench-cooled PCT Data ARM Glass

PROCEDURE AST	M-1285-97											т			
DATA AND RESUL	LTS FOR 7 D	AY PCT TEST	r	TEST NAME	ARM STAN	90C PCT WI	TH RPP ENVI	ELOPE LAW B	B GLASS						
PCT A. SA/V = 200	00M-1									LEACHATE D	ILUTION FACT	ORS:			
SPREADSHEET R	EDUCED 70%	6 FOR PRINT	OUT							BLANKS: 101	ML SPL AND 0.	1ML CONC HI	103	DF =10.1/10	= 1.01
										RPP ARM I	RM GLASSES	STDS 1 2 4	8.5		
				/EN	10/10/2002			=N	1215	6MI SPI /0.10	ML CONC HNC	03 4 MI ASTI	M H2O	DE=10 1/6=1	683
			DATE OUT O	DF OVEN	10/17/2002		TIME OUT C		1015	STDS. 3 & 6:	6ML STANDAR	RD	SUBMIT UNI	DILUTED	
						INITIAL pH =	6.81								
RAW EXPERIMEN	TAL DATA:				INITIAL	FINAL				SRTC					
SAMPLE		WEIGHTS	-	WEIGHT	WEIGHT	WEIGHT	INIT.	WATER	FINAL	ADS	RESULTS (pr	om) FOR ACIE	IFIED LEACH	ATES.	
NAME	EMPTY	W/GLASS	GLASS	W/H2O	IN PCT	IN PCT	VOL (ML)	1.055	nН	NUMBER	B	Si	Na	11	Δ 1
B-185 BLANK 1	121 230	N A	N A	137 236	338 088	338.066	16.006	0.022	6.95	187600	<0.031	<0.018	0 222	<0.047	<0.063
B-187 BLANK 2	121 584	N A	ΝA	137 597	338 919	338 927	16 013	-0.008	6.95	187606	<0.031	<0.018	0.285	<0.047	<0.063
B 100 BLANK 2	121.004	N.A.	NLA.	107.001	227.690	227 604	16.004	0.005	7.00	197610	-0.031	-0.010	0.167	-0.047	-0.063
B-190, BLAINK 3	121.141	N.A.	N.A.	137.145	337.009	337.094	16.004	-0.005	7.00	10/012	<0.031	<0.018	0.167	<0.047	<0.063
BLANK AVERAGE									6.97		<0.031	<0.018	0.2247	<0.047	<0.063
SAMPLES											RESULTS (pr			ATES	
B-182, ARM-1	122.034	123.646	1.612	139,770	342.645	342.652	16.124	-0.007	9.46	187602	11.8	40.3	23.8	9.08	2.68
B-183 ARM-2	121 222	122 823	1 601	138 836	340 318	340 318	16.013	0.000	9.53	187608	12.5	40.7	24.9	9.51	2.64
B-184, ARM-3	121.000	122.606	1.606	138.640	340.400	340.406	16.034	-0.006	9.50	187614	11.0	38.6	22.9	8.84	2.74
									CONTROL C					SPCT	
										HIGH	22.7	73.4	43.6	16.3	
CALCULATED RE	SULTS: pH	I VALUES AN	D LEACHATE		ATIONS CORF	ECTED FOR	BLANKS			LOW	12.9	49.0	28.9	10.8	
SAMPLE				GLASS	INIT.	FINAL	%	pH VALU	ES	ADS		CORRECTED	CONCENTR	ATIONS (PPM	I)
NAME				WEIGHT	VOL.(ML)	VOL.(ML)	LOSS	INITIAL	FINAL	NUMBER	В	Si	Na	Li	AI
B-182, ARM-1				1.612	16.124	16.131	-0.04	6.88	9.79	187602	19.9	67.8	40.1	15.28	4.51
B-183, ARM-2				1.601	16.013	16.013	0.00	6.88	9.72	187608	21.0	68.5	41.9	16.01	4.44
B-184, ARM-3				1.606	16.034	16.040	-0.04	6.88	9.81	187614	18.5	65.0	38.5	14.88	4.61
AVERAGE								AVERAGE			19.8	67 1	40.2	15 39	4 52
STANDARD DEVIA	ATION							STANDARD	DEVIATION		13	19	17	0.57	0.08
REL STD DEVIAT	FION (%)							REL STD D	EVIATION (%)	1	6.38	2.80	4 20	3 71	1.87
	NORMALIZE		TIONS:		ELEMENTAL	WEIGHT PE	RCENT IN GL	ASS	(/)	,	0.00	2.00	1120		
					NORMALIZE	D MASS LOS	S (GRAMS G	LASS/LITER)				N	OT APPLICAE	BLE	1
					NORMALIZE	D MASS LOS	STANDAR	DEVIATION				N	OT APPLICAE	BLE	
					NORMALIZE	D MASS LOS	S PLUS 2 SIG	MA				N	OT APPLICAE	BLE	
											В	Si	Na	Li	AI
QUALITY ASSURA	ANCE INFOR	MATION:								LIMS NO.		ICP RESULT	S (PPM)		
							STANDARD	RESULTS:	S-1	187599	12.0	31.7	51.7	5.91	2.35
LABS= B-111						EXPIRATION	I DATE	3/31/2003	S-2	187605	12.0	31.7	51.8	5.97	2.35
PH METER ACCUM	MET AB 15		BUFFERED	TO PH 7AND	10.	LOT NO.	205009		S-3	187611	20.2	53.6	87.7	10.10	3.91
BALANCE SER.#=	GT1478								S-4	187617	11.9	31.3	51.4	5.92	2.33
OVEN SER.B-111									S-5	187620	12.0	31.6	51.2	5.92	2.36
TEMPERATURE R	EADOUT: GT	-1248 FLUKE	RTD						S-6	187621	20.1	52.9	86.1	9.94	3.93
FILTER SIZE:.45 N	IICRON												ACTOR		
									S-1	187500	20.2	53.4	87.0	0.05	3.96
									5-1	107.059	20.2	53.4	97.0	5.55 10.0F	3.50
RESEARCHER: NE	D BIBLER								5-2	18/005	20.2	53.4	87.Z	10.05	3.90
									5-3	18/611	20.2	53.6 53.7	8/./ 96 E	10.10	3.91
									5-4 6 E	18/01/	20.0	52.7	00.0 00.0	9.90	3.92
									0-C	187621	20.2	50.Z	00.2 86 1	9.90	3.31
						STANDARD	COMPOSITIC		0.0	10/021	20.0+/-0.1	50.0+/.0.2	91 01/04	10.00+/-0.05	4 00 1/0 02

Table D- 2. Quench-cooled PCT Data LRM Glass

PROCEDURE AS	TM-1285-97														
DATA AND RESU	LTS FOR 7	DAY PCT TES	ST	TEST NAME		90C PCT W	ITH RPP ENV	ELOPE LAW B	GLASS			1			
				GLASS. I	RM Refere	nce GLAS	s								
PCT A, SA/V = 20	00M-1						•			LEACHATE	DILUTION FA	CTORS:			
SPREADSHEET R	REDUCED 7	% FOR PRIN	т оџт							BLANKS: 10	ML SPL AND	0.1ML CONC	HNO3	DF =10.1/10 =	1.01
										RPP ARM	I RM GLASSE	S STDS 1 2	4 & 5:		
					10/10/2002			EN	1215	6MI SPI /0 1				DE-10 1/6-1	692
					10/17/2002				1015	STDS 3&6	6ML STAND	ARD	SUBMIT UNI		000
			DATE OUT		10/11/2002			I OVEN	1010	0120.040			000000000000	5120125	
						INITIAL pH =	= 6.81								
RAW EXPERIMEN		:	_		INITIAL	FINAL				SRTC					
SAMPLE		WEIGHTS		WEIGHT	WEIGHT	WEIGHT	INIT.	WATER	FINAL	ADS	RESULTS (p	pm) FOR ACI	DIFIED LEAC	HATES.	
NAME	EMPTY	W/GLASS	GLASS	W/H2O	IN PCT	IN PCT	VOL.(ML)	LOSS	pН	NUMBER	в	Si	Na	Li	AI
B-185, BLANK 1	121.230	N.A.	N.A.	137.236	338.088	338.066	16.006	0.022	6.95	187600	<0.031	<0.018	0.222	<0.047	< 0.063
B-187, BLANK 2	121.584	N.A.	N.A.	137.597	338.919	338.927	16.013	-0.008	6.95	187606	<0.031	<0.018	0.285	<0.047	< 0.063
B-190, BLANK 3	121.141	N.A.	N.A.	137.145	337.689	337.694	16.004	-0.005	7.00	187612	<0.031	<0.018	0.167	<0.047	< 0.063
BLANK AVERAGE	E								6.97		<0.031	<0.018	0.2247	<0.047	<0.063
SAMPLES															
B-179,LRM-1	122.247	123.853	1.606	139.923	340.874	340.874	16.070	0.000	10.12	187604	14.0	46.5	88.6	0.065	8.21
B-180,LRM-2	121.096	122.700	1.604	138.745	341.431	341.439	16.045	-0.008	10.17	187610	14.5	48.4	91.9	0.071	8.60
B-181,LRM-3	122.784	124.398	1.614	140.546	341.512	341.517	16.148	-0.005	10.12	187616	13.7	45.0	85.9	0.052	7.93
CALCULATED RE	ESULTS:	H VALUES A	ND FILTERE	D LEACHATE	CONCENTRA	TIONS CORF	RECTED FOR	BLANKS							
SAMPLE				GLASS	INIT.	FINAL	%	pH VALUE	S			CONC	ENTRATIONS	S (PPM)	
NAME				WEIGHT	VOL.(ML)	VOL.(ML)	LOSS	INITIAL	FINAL		в	Si	Na	Li	AI
B-179.LRM-1				1.606	16.070	16.070	0.00	6.88	10.12	187604	23.6	78.3	149.1	0.11	13.82
B-180.LRM-2				1.604	16.045	16.053	-0.05	6.88	10.17	187610	24.4	81.5	154.7	0.12	14.47
B-181 RM-3				1 614	16 148	16 153	-0.03	6.88	10.12	187616	23.1	75.7	144.6	0.09	13.35
AVERAGE					10.110	10.100	0.00		10.12	101010	23.7	78.5	149.5	0.00	13.88
								STANDARD			0.7	2.0	5.1	0.02	0.57
										3	2.97	2.5	2 29	15 50	4.09
KEL. STD. DEVIA			TIONE					ACC		•)	2.07	3.03	3.30	0.05	4.08 5.10
<u>-</u>	NORMALIZE	DCALCULA	HUN3.	-				ASS			2.34	0.35	15.5	0.05	3.10
					NORMALIZE	D MASS LOS		LASS/LITER)			1.01	0.35	0.90	0.21	
					NORMALIZE	D MASS LOS			.)		0.506	0.173	0.402	0.105	
					NORMALIZE	D MASS LOS		DEVIATION			0.015	0.008	0.010	0.016	
					NORMALIZE	D WA35 LUG	53 FLU3 2 310				0.029	0.013	0.035	0.033	
												e i	Na		A1
		DMATION.									D			-	Ai
QUALITY ASSOR		NMATION.					STANDARD	DESIII TS.	S-1	187500	12.0	31 7	51 7	5.91	2 35
LABS- B-111						EXPIRATION		3/31/2002	5.2	187605	12.0	31.7	51.7	5.97	2.35
PH METER ACOU			BUFFEDED		10		205000	0/01/2003	G-2	187611	20.2	52.6	87.7	10 10	2.00
BALANCE SED #	-CT1470		DOLLEKED	I O FILLAND	10.	LOT NU.	200009		0-0 C 4	107011	20.2	24.2	54 4	5.00	0.91
OVEN SED D 411	-011470								0-4	10/01/	10.0	31.3	51.4	0.92	2.00
UVEN SEK.B-111		T 4040 EL							5-5	18/620	12.0	31.0	51.2	5.92	2.30
IEMPERATURE F	KEADOUT: (91-1248 FLUK	EKID						5-6	187621	20.1	52.9	86.1	9.94	3.93
FILTER SIZE:.45 M	MICRON									RESULTS C	ORRECTED F	OR DILUTION	FACTOR		
									S-1	187599	20.2	53.4	87.0	9.95	3.96
RESEARCHER: N	ED BIBLER								S-2	187605	20.2	53.4	87.2	10.05	3.96
									S-3	187611	20.2	53.6	87.7	10.10	3.91
									S-4	187617	20.0	52.7	86.5	9.96	3.92
									S-5	187620	20.2	53.2	86.2	9.96	3.97
									S-6	187621	20.1	52.9	86.1	9.94	3.93
						STANDARD	COMPOSITIC	N (PPM)			20.0+/-0.1	50.0+/-0.3	81.0+/-0.4	10.00+/-0.05	4.00+/-0.02

Table D- 3. Quench-cooled PCT Data AZ-102 Simulant Glass

PROCEDURE ASTN	1-1285-97											_			
DATA AND RESULT	S FOR 7 DAY	PCT TEST		TEST NAME		90C PCT WI	TH RPP ENVE	ELOPE LAW B	GLASS						
				GLASS.	INVELOPE	B BLANK	NONRAD G	GLASS							
PCT A, SA/V = 2000	M-1										DILUTION FA	CTORS:		BE 10.140	.
SPREADSHEET RE	DUCED 70%	FOR PRINT O	UT							BLANKS: 1	0 ML SPL AND	0.1ML CONC	HNO3	DF =10.1/10 =	1.01
				(5)	10/10/2002				1015	RPP, ARM,	LRM GLASSE	S, STDS 1, 2,	4, & 5: ETM H2O	DE 10 1/6 1 6	200
					10/17/2002				1015	STDS 3&F		ARD	SUBMIT UNI		000
			DATE OUT		10/11/2002			I OTEN	1010	0100.000		,, u (B	00000000		
						INITIAL pH =	6.81								
RAW EXPERIMENT	AL DATA:		_		INITIAL	FINAL				SRTC					
SAMPLE		WEIGHTS		WEIGHT	WEIGHT	WEIGHT	INIT.	WATER	FINAL	ADS	RESULTS (p	pm) FOR ACI	DIFIED LEAC	HATES.	
NAME	EMPTY	W/GLASS	GLASS	W/H2O	IN PCT	IN PCT	VOL.(ML)	LOSS	pН	NUMBER	в	Si	Na	Li	AI
B-185, BLANK 1	121.230	N.A.	N.A.	137.236	338.088	338.066	16.006	0.022	6.95	187600	<0.031	<0.018	0.222	<0.047	<0.063
B-187, BLANK 2	121.584	N.A.	N.A.	137.597	338.919	338.927	16.013	-0.008	6.95	187606	<0.031	<0.018	0.285	<0.047	<0.063
B-190, BLANK 3	121.141	N.A.	N.A.	137.145	337.689	337.694	16.004	-0.005	7.00	187612	<0.031	<0.018	0.167	<0.047	<0.063
BLANK AVERAGE									6.97		<0.031	<0.018	0.2247	<0.047	<0.063
	EMPTY														
SAMPLES	W LUG														
B-167, BBLK-1	322.985	NA	1.689	NA	341.298	341.301	16.816	-0.003	8.91	187601	10.6	27.1	9.14	6.96	0.448
B-168, BBLK-2	322.052	NA	1.658	NA	340.217	340.197	16.651	0.020	8.95	187607	10.7	27.2	9.31	7.07	0.440
B-169, BBLK-3	324.298	NA	1.689	NA	342.752	342.757	16.849	-0.005	9.01	187613	10.5	26.9	9.09	7.05	0.447
B-176, BBLK-4	322.045	NA	1.701	NA	340.413	340.418	16.785	-0.005	8.88	187618	10.3	26.4	8.80	6.88	0.476
CALCULATED RES	ULTS: pH \	ALUES AND	FILTERED L	EACHATE CO	NCENTRATIO	NS CORRECT	ED FOR BLA	NKS			_				
SAMPLE		FINAL	CALC	GLASS	INIT.	FINAL	%	pH VALUE	S			CONC	ENTRATION	S (PPM)	
NAME		GL+WA	GL+WA	WEIGHT	VOL.(ML)	VOL.(ML)	LOSS	INITIAL	FINAL		В	Si	Na	Li	Al
B-167, BBLK-1		18.316	18.505	1.689	16.816	16.819	-0.02	6.88	8.91		17.8	45.6	15.38	11.71	0.754
B-168, BBLK-2		18.145	18.309	1.658	16.651	16.631	0.12	6.88	8.95		18.0	45.8	15.67	11.90	0.741
B-169, BBLK-3		18.459	18.538	1.689	16.849	16.854	-0.03	6.88	9.01		17.7	45.3	15.30	11.87	0.752
B-176, BBLK-4		18.373	18.486	1.701	16.785	16.790	-0.03	6.88	8.88		17.3	44.4	14.81	11.58	0.801
AVERAGE											17.7	45.3	15.29	11.76	0.762
STANDARD DEVIAT											0.3	0.6	0.36	0.15	0.03
REL. STD. DEVIATIO											1.62	1.32	2.33	1.25	3.51
	NORMALIZE	DCALCULA	HUNS:					ASS/LITED			4.04	0 10	0.41	2.18	0.02
						D MASS LOS	S (GRAMS GI	ASS/METER2			0.77	0.10	0.41	0.27	0.01
						DMASSIOS	S STANDARD		,		0.00	0.00	0.00	0.00	0.00
					NORMALIZE	D MASS LOS	S PLUS 2 SIG	MA			0.23	0.10	0.21	0.28	0.01
											В	Si	Na	Li	Al
QUALITY ASSURAN	CE INFORM	ATION:								LIMS NO.		ICP RESULT	S (PPM)		
							STANDARD	RESULTS:	S-1	187599	12.0	31.7	51.7	5.91	2.35
LABS= B-111						EXPIRATION	DATE	3/31/2003	S-2	187605	12.0	31.7	51.8	5.97	2.35
PH METER ACCUM	ET AB 15		BUFFERED	TO PH 7AND	10.	LOT NO.	205009		S-3	187611	20.2	53.6	87.7	10.10	3.91
BALANCE SER.#=G	T1478								S-4	187617	11.9	31.3	51.4	5.92	2.33
OVEN SER.B-111									S-5	187620	12.0	31.6	51.2	5.92	2.36
TEMPERATURE RE	ADOUT: GT-1	248 FLUKE R	TD						S-6	187621	20.1	52.9	86.1	9.94	3.93
FILTER SIZE:.45 MIC	CRON														
									S-1	187500	20.2	53.4	87.0	0.05	3.96
									0-1	10/099 19760F	20.2	52 A	07.0 87.0	5.90 10.05	3.90
NEGEARGHER: NEL	DIDLER								3-2 5-3	187611	20.2	53.6	07.2 87.7	10.05	3.90
									S-4	187617	20.2	52.7	86.5	9.96	3.92
									S-5	187620	20.0	53.2	86.2	9.96	3.97
									S-6	187621	20.2	52.9	86.1	9.94	3.93
						STANDARD	COMPOSITIO	N (PPM)			20.0+/-0.1	50.0+/-0.3	81.0+/-0.4	10.00+/-0.05	4.00+/-0.02

Table D- 4. Quench-cooled PCT Data AZ-102 Radioactive Glass

PROCEDURE AST	M-1285-97														
DATA AND RESUL	LTS FOR 7 D	AY PCT TEST		TEST NAME		90C PCT W	ITH RPP ENVE	LOPE LAW B	GLASS			1			
				GLASS.	ENVELOPE	B RAD GL	ASS								
PCT A, SA/V = 200	00M-1									LEACHATE	DILUTION FA	CTORS:			_
SPREADSHEET R	EDUCED 70%	6 FOR PRINT	OUT							BLANKS: 1	0 ML SPL AND	0.1ML CONC	HNO3	DF =10.1/10 =	1.01
										RPP, ARM,	LRM GLASSE	S, STDS 1, 2,	4, & 5:		
			DATE IN OV	/EN	10/10/2002		TIME IN OVE	N	1215	6ML SPL/0.1	10 ML CONC H	INO3, 4 ML AS	STM H2O	DF=10.1/6=1.6	383
			DATE OUT O	OF OVEN	10/17/2002		TIME OUT OF	F OVEN	1015	STDS. 3 & 6	6ML STANE	ARD	SUBMIT UND	DILUTED	
						INITIAL pH =	= 6.81								
RAW EXPERIMEN	TAL DATA:		_		INITIAL	FINAL				SRTC					
SAMPLE		WEIGHTS		WEIGHT	WEIGHT	WEIGHT	INIT.	WATER	FINAL	ADS	RESULTS (p	pm) FOR ACI	DIFIED LEAC	HATES.	
NAME	EMPTY	W/GLASS	GLASS	W/H2O	IN PCT	IN PCT	VOL.(ML)	LOSS	рН	NUMBER	В	Si	Na	Li	AI
B-185, BLANK 1	121.230	N.A.	N.A.	137.236	338.088	338.066	16.006	0.022	6.95	187600	<0.031	<0.018	0.222	<0.047	<0.063
B-187, BLANK 2	121.584	N.A.	N.A.	137.597	338.919	338.927	16.013	-0.008	6.95	187606	<0.031	<0.018	0.285	<0.047	<0.063
B-190, BLANK 3	121.141	N.A.	N.A.	137.145	337.689	337.694	16.004	-0.005	7.00	187612	<0.031	<0.018	0.167	<0.047	<0.063
BLANK AVERAGE									6.97		<0.031	<0.018	0.2247	<0.047	<0.063
	EMPTY														
SAMPLES	W LUG														
B-126,BRAD-1	324.296	NA	1.697	NA	342.688	342.693	16.792	-0.005	8.86	187603	9.690	25.200	7.080	6.380	0.570
B-130,BRAD-2	322.356	NA	1.649	NA	340.391	340.365	16.571	0.026	8.94	187609	9.550	25.200	7.040	6.400	0.472
B-150,BRAD-3	322.652	NA	1.702	NA	341.290	341.298	17.019	-0.008	8.85	187615	9.480	25.200	6.980	6.460	0.486
B-158,BRAD-4	323.535	NA	1.676	NA	341.976	341.977	16.890	-0.001	9.02	187679	9.460	25.100	6.890	6.450	0.489
	CUIL TO:														
CALCULATED RE	50L15: pr	I VALUES AN				UNS CURRE	CIED FOR BL		<u></u>		-	0010			
SAMPLE		FINAL		GLASS		FINAL	%		5				ENTRATIONS	(PPW)	
		GL+WA	GL+WA	WEIGHT	VOL.(ML)	VOL.(ML)	LOSS	INITIAL	FINAL		B 40.04	51	Na 44.00	LI 40.74	AI
B-126,BRAD-1		18.397	18.489	1.697	16.792	16.797	-0.03	6.88	8.86		16.31	42.41	11.92	10.74	0.96
B-130,BRAD-2		18.009	18.220	1.649	16.5/1	16.545	0.16	6.88	8.94		16.07	42.41	11.85	10.77	0.79
B-150,BRAD-3		18.646	18.721	1.702	17.019	17.027	-0.05	6.88	8.85		15.95	42.41	11.75	10.87	0.82
B-158,BRAD-4		18.442	18.566	1.676	16.890	16.891	-0.01	6.88	9.02		15.92	42.24	11.60	10.86	0.82
AVERAGE											16.06	42.37	11.78	10.81	0.85
STANDARD DEVIA	ATION										0.18	0.08	0.14	0.07	0.07
REL. STD. DEVIAT	IION (%)										1.09	0.20	1.18	0.60	8.82
	NORMALIZE	D CALCULA	TIONS:		ELEMENTAL	WEIGHT PE	RCENT IN B R	AD GLASS			4.04	23.37	3.71	2.18	3.44
					NORMALIZE	D MASS LUS	SS (GRAMS GL	ASS/LITER)			0.40	0.18	0.32	0.50	0.02
					NORMALIZE	D MASS LOS	SS (GRAMS GL	ASS/METER2			0.20	0.09	0.16	0.25	0.01
					NORMALIZE	D MASS LOS	SS STANDARD	DEVIATION			0.00	0.00	0.00	0.00	0.00
					NORMALIZE	D MASS LOS	S PLUS 2 SIG	MA			0.20	0.09	0.16	0.25	0.01
											в		Na C (DDM)	Li	AI
QUALITY ASSURA	ANCE INFORI	WATION:					CTANDADD			LIMS NO.	40.0	ICP RESULT	<u>S (PPM)</u>	5.04	0.05
1 A D O D 444							STANDARD	RESULTS:	5-1	187599	12.0	31.7	51.7	5.91	2.35
LABS= B-111					10	LOT NO	DATE	3/31/2003	5-2	18/605	12.0	31.7	51.8	5.97	2.35
PH METER ACCU	MET AB 15		BUFFERED	TO PH 7AND	10.	LUT NU.	205009		5-3	18/611	20.2	53.6	87.7	10.10	3.91
BALANCE SER.#=0	G11478								S-4	187617	11.9	31.3	51.4	5.92	2.33
OVEN SER.B-111									S-5	187620	12.0	31.6	51.2	5.92	2.36
TEMPERATURE R	EADOUT: GT	-1248 FLUKE	RID						S-6	187621	20.1	52.9	86.1	9.94	3.93
FILTER SIZE:.45 M	IICRON									RESULTS C	ORRECTED F		FACTOR		
									S-1	187599	20.2	53.4	87.0	9.95	3.96
RESEARCHER: NF	ED BIBLER								S-2	187605	20.2	53.4	87.2	10.05	3.96
									S-3	187611	20.2	53.6	87.7	10.10	3.91
									S-4	187617	20.0	52.7	86.5	9.96	3.92
									S-5	187620	20.0	53.2	86.2	9.96	3.97
									S-6	187621	20.1	52.9	86.1	9.94	3.93
						STANDARD	COMPOSITIO		50	.5/021	20.0+/-0.1	50.0+/.0.2	91.0+/.0.4	10.00+/-0.05	4 00+/ 0 02

Table D- 5. Container Centerline-Cooled PCT Data ARM Glass

PROCEDURE AST	FM-1285-97				•									•	·
DATA AND RESUL	LTS FOR 7 D	AY PCT TEST		TEST NAME		90C PCT WIT	TH RPP ENVE	LOPE LAW B GL	ASS			1			
				GLASS.	ARM STANE	ARD GLAS	S								
PCT A. SA/V = 200	DOM-1			OLACO.		DARD GLAG	0			LEACHATE D	ILUTION FACT	ORS:			
SPREADSHEET R	EDUCED 70%	FOR PRINT	онт							BLANKS: 10	VI SPLANDO	1ML CONC HN	03	DE =10 1/10 :	- = 1.01
	200020.07									RPP ARM I	RM GLASSES		5	51 -10.1710	
					44/4/0000				0.00		NI CONCLIN	31031, 2, 4, 6		DE 40.4/0.4	coo
			DATE IN OV		11/4/2003		TIME IN OVE		9:26	6ML SPL/0.10	ML CONC HNC	03, 4 ML ASTN	/I H2O	DF=10.1/6=1.	.683
			DATE OUT C	FOVEN	11/11/2003		TIME OUT O	FOVEN	9.45	3103.3 & 0.	OIVIL STANDAR	(D	SUDIVITI UNI	JILUTED	
							0.00								
						INITIAL PH =	6.33								
RAW EXPERIMEN	ITAL DATA:		-		INITIAL	FINAL				SRTC					
SAMPLE		WEIGHTS		WEIGHT	WEIGHT	WEIGHT	INIT.	WATER	FINAL	ADS	RESULTS (p)	om) FOR ACID	IFIED LEACH	IATES.	
NAME	EMPTY	W/GLASS	GLASS	W/H2O	IN PCT	IN PCT	VOL.(ML)	LOSS	рН	NUMBER	В	Si	Na	Li	AI
B-111, BLANK 1	121.432	N.A.	N.A.	137.452	338.158	338.152	16.020	0.006	6.80	203108	<0.061	<0.046	0.047	<0.065	<0.172
B-125, BLANK 2	122.04	N.A.	N.A.	138.099	340.748	340.641	16.059	0.107	5.39	203113	<0.061	<0.046	0.058	<0.065	<0.172
B-122, BLANK 3	120.779	N.A.	N.A.	136.826	338.186	338.142	16.047	0.044	6.81	203118	<0.061	<0.046	0.068	<0.065	<0.172
BLANK AVERAGE									6.33		<0.061	<0.046	0.0577	<0.065	<0.172
SAMPLES											RESULTS (p	om) FOR ACID	IFIED LEACH	IATES.	
B-131, ARM-1	121.117	122.737	1.620	138.988	340.399	340.390	16.251	0.009	9.38	203109	10.60	38.30	22.10	8.33	2.51
B-112, ARM-2	121.525	123.144	1.619	139.344	339.700	339.485	16.200	0.215	9.44	203114	9.96	36.50	21.50	7.84	2.49
B-107, ARM-3	121.737	123.351	1.614	139.510	341.433	341.247	16.159	0.186	9.44	203119	9.76	35.90	20.80	7.66	2.49
									CONTROL	CHART VALUES	S FOR CONCEN	ITRATIONS IN	ARM GLASS	PCT	
										HIGH	22.7	73.4	43.6	16.3	
CALCULATED RE	SULIS: pr	I VALUES AN	DLEACHATE	CONCENTRA	TIONS CORRI	ECTED FOR BL	ANKS			LOW	12.9	49.0	28.9	10.8	
SAMPLE				GLASS			% 1.055		CINIAL	AUS	в	CURRECTEL	No	ATIONS (PPM)	
B-131 ARM-1				1 620	16 251	16 242	0.06	6.88	0.38	203109	17.8	64.5	37.2	14.02	A1 4 22
B-112 ARM-2				1.619	16 200	15 985	1.33	6.88	9.44	203103	16.8	61.4	36.2	13.19	4.19
B-107, ARM-3				1.614	16.159	15.973	1.15	6.88	9.44	203119	16.4	60.4	35.0	12.89	4.19
,															
AVERAGE								AVERAGE	9.42		17.0	62.1	36.1	13.37	4.20
STANDARD DEVIA	ATION							STANDARD DEV	IATION		0.7	2.1	1.1	0.58	0.02
REL. STD. DEVIAT	TION (%)							REL. STD. DEVIA	ATION (%)		4.34	3.38	3.03	4.37	0.46
	NORMALIZE	D CALCULAT	IONS:		ELEMENTAL	WEIGHT PERC	CENT IN GLA	SS							
					NORMALIZE	D MASS LOSS	(GRAMS GLA	ASS/LITER)				N	OT APPLICA	BLE	
					NORMALIZE	D MASS LOSS	STANDARD	DEVIATION				N	OT APPLICA	BLE	
					NORMALIZE	D MASS LOSS	PLUS 2 SIGN	IA				N	OT APPLICA	BLE]
											В	Si	Na	Li	AI
QUALITY ASSURA	ANCE INFOR	MATION:								LIMS NO.		ICP RESULT	S (PPM)		
							STANDARD	RESULTS:	S-1	203107	11.6	30.4	48.2	5.97	2.15
LABS= B-111	NET 40 46				40	EXPIRATION	DATE	10/31/2004	S-2	203112	11.5	30.3	47.8	5.97	2.12
PH METER ACCU	OT1479		BUFFERED	TO PH /AND	10.	LUT NO.	326902		5-3	203117	18.8	49.7	78.4 47.5	9.76	3.45
BALANCE SER.#=	G11478								5-4	203122	11.4	29.9	47.5	5.89	2.14
OVEN SER.B-111			DTD						5-5	203124	11.4	30.1	47.3	5.88	2.13
TEMPERATURE R	(EADOUT: GT	-1248 FLUKE	RID						S-6	203125	18.8	49.2	77.3	9.61	3.46
FILTER SIZE:.45 N	/ICRON														
										RESULTS CO	RRECTED FOR	DILUTION FA	CTOR	10.05	
									S-1	203107	19.5	51.2	81.1	10.05	3.62
RESEARCHER: C.	L.Crawford								S-2	203112	19.4	51.0	80.4	10.05	3.57
									S-3	203117	18.8	49.7	78.4	9.76	3.45
									S-4	203122	19.2	50.3	79.9	9.91	3.60
									S-5	203124	19.2	50.7	79.6	9.90	3.58
							ouncorr		5-0	203125	18.8	49.2	11.3	9.01	3.40
1						SIANDARD	JOMPOSITIO	N (PPM)			20.0+/-0.1	50.0+/-0.3	81.0+/-0.4	10.00+/-0.05	4.00+/-0.02

Table D- 6. Container Centerline-Cooled PCT Data LRM Glass

PROCEDURE ASTN	M-1285-97														
DATA AND RESULT	TS FOR 7 DA	Y PCT TEST		TEST NAME	RM Refere	90C PCT W	ITH RPP ENV	ELOPE LAW B GL	ASS]			
PCT A, SA/V = 2000	DM-1			<u></u>			•			LEACHATE	DILUTION FA	CTORS:			
SPREADSHEET RE	DUCED 70%	FOR PRINT O	JUT							BLANKS: 10	ML SPL AND	0.1ML CONC	HNO3	DF =10.1/10 =	- 1.01
-										RPP. ARM.	LRM GLASSE	S. STDS 1. 2. 4	4. & 5:		
			DATE IN O	/FN	11/4/2003		TIME IN OVE	FN	9.26	6ML SPL/0.1		NO3 4 MI A5	TM H2O	DF=10 1/6=1 (683
			DATE OUT (OF OVEN	11/11/2003		TIME OUT C	DF OVEN	9:45	STDS, 3 & 6	6ML STAND	ARD	SUBMIT UNI	DILUTED	500
						INITIAL pH =	= 6.33	3							
RAW EXPERIMENT	AL DATA:		_		INITIAL	FINAL				SRTC					
SAMPLE		WEIGHTS		WEIGHT	WEIGHT	WEIGHT	INIT.	WATER	FINAL	ADS	RESULTS (p	pm) FOR ACII	DIFIED LEAC	HATES.	
NAME	EMPTY	W/GLASS	GLASS	W/H2O	IN PCT	IN PCT	VOL.(ML)	LOSS	pН	NUMBER	В	Si	Na	Li	AI
B-111, BLANK 1	121.432	N.A.	N.A.	137.452	338.158	338.152	16.020	0.006	6.80	203108	<0.061	<0.046	0.047	<0.065	<0.172
B-125, BLANK 2	122.04	N.A.	N.A.	138.099	340.748	340.641	16.059	0.107	5.39	203113	<0.061	<0.046	0.058	<0.065	<0.172
B-122, BLANK 3	120.779	N.A.	N.A.	136.826	338.186	338.142	16.047	0.044	6.81	203118	<0.061	<0.046	0.068	<0.065	<0.172
BLANK AVERAGE								Average:	6.33		<0.061	<0.046	0.0577	<0.065	<0.172
SAMPLES															
B-118,LRM-1	121.215	122.829	1.614	138.974	340.929	340.769	16.145	0.160	10.12	203111	11.8	43.8	79.1	0.065	7.37
B-129,LRM-2	122.037	123.655	1.618	139.824	340.539	340.536	16.169	0.003	10.11	203116	11.6	42.2	78.5	0.065	7.21
B-113,LRM-3	121.996	123.613	1.617	139.779	341.202	341.184	16.166	0.018	10.15	203121	11.6	43.3	78.4	0.065	7.38
CALCULATED RES	ULTS: pH	VALUES AND	FILTERED L	EACHATE CO	NCENTRATIO	NS CORRECT	ED FOR BLA	NKS		_	2.45	25.38	14.86	0.05	3.44
SAMPLE				GLASS	INIT.	FINAL	%	pH VALUES				CONC	ENTRATION	<u>3 (PPM)</u>	T
NAME				WEIGHT	VOL.(ML)	VOL.(ML)	LOSS	INITIAL	FINAL		В	Si	Na	Li	AI
B-118,LRM-1				1.614	16.145	15.985	0.99	6.33	10.12	203111	19.9	73.7	133.1	0.11	12.40
B-129,LRM-2				1.618	16.169	16.166	0.02	6.33	10.11	203116	19.5	71.0	132.1	0.11	12.13
B-113,LRM-3				1.617	16.166	16.148	0.11	6.33	10.15	203121	19.5	72.9	131.9	0.11	12.42
AVERAGE								AVERAGE	10.13		19.6	72.5	132.4	0.11	12.32
STANDARD DEVIA	TION							STANDARD DE	/IATION		0.2	1.4	0.6	0.00	0.16
REL. STD. DEVIATI	ION (%)							REL. STD. DEVI	ATION (%)		0.99	1.90	0.48	0.00	1.30
	NORMALIZE	D CALCULAT	FIONS:	_	ELEMENTAL	WEIGHT PEF	RCENT IN GLA	ASS			2.34	22.62	15.5	0.05	5.10
				_	NORMALIZE	D MASS LOS	S (GRAMS GL	ASS/LITER)			0.84	0.32	0.85	0.22	
					NORMALIZE	D MASS LOS	S (GRAMS GL	ASS/METER2)			0.420	0.160	0.427	0.109	
					NORMALIZE	D MASS LOS	SSTANDARD	DEVIATION			0.004	0.003	0.002	0.000	
					NORMALIZE	D MASS LOS	S PLUS 2 SIGI	MA			0.008	0.006	0.004	0.000	
											_	<u>.</u>			
											в		Na S (DDM)	L	AI
QUALITY ASSORA		ATION.					STANDARD	DECULTO.	C 1	202107	11.6	20.4	40.0	E 07	2.15
								10/21/2004		203107	11.0	30.4	40.2	5.97	2.13
LABS= B-111	ET AD 45				40	EXPIRATION	N DATE	10/31/2004	5-2	203112	11.5	30.3	47.8	5.97	2.12
PH METER ACCUM	IET AB 15		BOFFERED	TO PH 7AND	10.	LOT NO.	326902		5-3	203117	18.8	49.7	78.4	9.76	3.45
BALANCE SER.#=G	GT1478								S-4	203122	11.4	29.9	47.5	5.89	2.14
OVEN SER.B-111									S-5	203124	11.4	30.1	47.3	5.88	2.13
TEMPERATURE RE	ADOUT: GT-	1248 FLUKE F	₹TD						S-6	203125	18.8	49.2	77.3	9.61	3.46
FILTER SIZE: 45 MI	ICRON									RESULTS C	ORRECTED F	OR DILUTION	FACTOR		
									S-1	203107	19.5	51.2	81.1	10.05	3.62
RESEARCHER: C.L	Crawford								S-2	203112	19.4	51.0	80.4	10.05	3.57
									S-3	203117	18.8	49.7	78.4	9.76	3.45
									S-4	203122	19.2	50.3	79.9	9.91	3.60
									8.5	202124	10.2	50.7	70.6	0.00	2.59
									5-5	203124	19.2	40.2	73.0	9.50	3.30
							COMPOSITIC		3-0	203125	10.0	49.2	11.3	9.01	3.40
						STANDARD	CONFOSITIC	(r [.])		AVG:	20.0+/-0.1	50.0+/-0.3	31.0+/-0.4	10.00+/-0.05	+.00+/-0.02

Table D- 7. Container Centerline-Cooled PCT Data AZ-102 Radioactive Glass

PROCEDURE AST	M-1285-97														
DATA AND RESUL	_TS FOR 7 D/	AY PCT TEST		TEST NAME		90C PCT WI	ITH RPP ENVE	LOPE LAW B	GLASS			1			
				GLASS. E	NVELOPE	B RAD GLA	ASS								
PCT A, SA/V = 200	0M-1									LEACHATE	DILUTION FA	CTORS:			_
SPREADSHEET R	EDUCED 70%	FOR PRINT	OUT							BLANKS: 10	ML SPL AND	0.1ML CONC	HNO3	DF =10.1/10 =	1.01
										RPP, ARM,	LRM GLASSE	S, STDS 1, 2, 4	4, & 5:		
			DATE IN OV	/EN	11/4/2003		TIME IN OVE	EN .	9:26	6ML SPL/0.1	10 ML CONC H	NO3, 4 ML AS	STM H2O	DF=10.1/6=1.6	683
			DATE OUT O	OF OVEN	11/11/2003		TIME OUT O	F OVEN	9:45	STDS. 3 & 6	: 6ML STAND	ARD	SUBMIT UND	ILUTED	
						INITIAL pH =	6.33	1							
RAW EXPERIMEN	TAL DATA:		_		INITIAL	FINAL				SRTC					
SAMPLE		WEIGHTS		WEIGHT	WEIGHT	WEIGHT	INIT.	WATER	FINAL	ADS	RESULTS (p	pm) FOR ACII	DIFIED LEACH	ATES.	
NAME	EMPTY	W/GLASS	GLASS	W/H2O	IN PCT	IN PCT	VOL.(ML)	LOSS	рН	NUMBER	в	Si	Na	Li	AI
B-111, BLANK 1	121.432	N.A.	N.A.	137.452	338.158	338.152	16.020	0.006	6.80	203108	<0.061	<0.046	0.047	<0.065	<0.172
B-125, BLANK 2	122.04	N.A.	N.A.	138.099	340.748	340.641	16.059	0.107	5.39	203113	<0.061	<0.046	0.058	<0.065	<0.172
B-122, BLANK 3	120.779	N.A.	N.A.	136.826	338.186	338.142	16.047	0.044	6.81	203118	<0.061	<0.046	0.068	<0.065	<0.172
BLANK AVERAGE								Average:	6.33		<0.061	<0.046	0.0577	<0.065	<0.172
	EMPTY														
SAMPLES	W LUG														
B-128,BRAD-1	323.822	NA	1.618	NA	341.679	341.675	16.249	0.004	8.96	203110	7.890	22.900	6.340	5.570	0.499
B-106,BRAD-2	323.853	NA	1.608	NA	341.622	341.618	16.175	0.004	8.87	203115	7.620	21.900	5.900	5.250	0.442
B-119,BRAD-3	325.082	NA	1.599	NA	342.788	342.721	16.130	0.067	8.9	203120	7.230	21.500	5.730	5.120	0.469
B-123,BRAD-4	319.874	NA	1.620	NA	337.677	337.67	16.170	0.007	8.87	203123	7.680	22.000	6.140	5.390	0.474
CALCULATED RE	SULTS: pH	VALUES AN	D FILTERED I		NCENTRATIC	INS CORRECT	ED FOR BLA	NKS							
SAMPLE		FINAL	CALC	GLASS	INIT.	FINAL	%	pH VALUE	ES		_	CONC	ENTRATIONS	(PPM)	
NAME		GL+WA	GL+WA	WEIGHT	VOL.(ML)	VOL.(ML)	LOSS	INITIAL	FINAL		В	Si	Na	Li	AL
B-128.BRAD-1		17.853	17.867	1.618	16.249	16.245	0.02	6.33	8.96	203110	13.28	38.54	10.67	9.37	0.84
B-106.BRAD-2		17,765	17,783	1.608	16.175	16.171	0.02	6.33	8.87	203115	12.82	36.86	9.93	8.84	0.74
B-119.BRAD-3		17 639	17 729	1.599	16.130	16.063	0.42	6.33	8.90	203120	12.17	36.18	9.64	8.62	0.79
B-123.BRAD-4		17.796	17,790	1.620	16,170	16.163	0.04	6.33	8.87	203123	12.93	37.03	10.33	9.07	0.80
AVERAGE								Average:	8.90		12.80	37.15	10.14	8.97	0.79
STANDARD DEVIA								STANDARD	DEVIATION		0.46	0.99	0.45	0.32	0.04
REL. STD. DEVIAT	ION (%)							REL. STD. DI	EVIATION (%	.)	3.62	2.68	4.44	3.62	4.96
	NORMALIZE	D CALCULA	TIONS:		ELEMENTAL	WEIGHT PER	CENT IN B RA	AD GLASS		,	4.04	23.37	3.71	2.18	3.44
					NORMALIZE	D MASS LOSS	GRAMS GL	ASS/LITER)			0 317	0 159	0 273	0 412	0.023
					NORMALIZE	D MASS LOSS	GRAMS GL	ASS/METER2)			0.159	0.079	0.137	0.206	0.012
					NORMALIZE		STANDARD	DEVIATION			0.006	0.002	0.006	0.007	0.001
					NORMALIZE	D MASS LOSS	S PLUS 2 SIGN	1A			0.170	0.084	0.149	0.221	0.013
											В	Si	Na	Li	AI
QUALITY ASSURA	ANCE INFORM	MATION:								LIMS NO.		ICP RESULT	S (PPM)		
							STANDARD	RESULTS:	S-1	203107	11.6	30.4	48.2	5.97	2.15
LABS= B-111						EXPIRATION	DATE	10/31/2004	S-2	203112	11.5	30.3	47.8	5.97	2.12
PH METER ACCUM	VIET AB 15		BUFFERED	TO PH 7AND 1	0.	LOT NO.	326902		S-3	203117	18.8	49.7	78.4	9.76	3.45
BALANCE SER.#=0	GT1478								S-4	203122	11.4	29.9	47.5	5.89	2.14
OVEN SER.B-111									S-5	203124	11.4	30.1	47.3	5.88	2.13
TEMPERATURE R	EADOUT: GT	-1248 FLUKE	RTD						S-6	203125	18.8	49.2	77.3	9.61	3.46
FILTER SIZE:.45 M	IICRON														
										RESULTS C	ORRECTED F	OR DILUTION	FACTOR		
									S-1	203107	19.5	51.2	81.1	10.05	3.62
RESEARCHER: C.	L.Crawford								S-2	203112	19.4	51.0	80.4	10.05	3.57
									S-3	203117	18.8	49.7	78.4	9.76	3.45
									S-4	203122	19.2	50.3	79.9	9.91	3.60
									S-5	203124	19.2	50.7	79.6	9.90	3.58
									S-6	203125	18.8	49.2	77.3	9.61	3.46
						STANDARD	COMPOSITIO	N (PPM)			20.0+/-0.1	50.0+/-0.3	81.0+/-0.4	10.00+/-0.05	4.00+/-0.02
										AVG:	19.1	50.3	79.5	9.9	3.5