

## Characterization of the Low Level Waste Reference Glass (LRM)

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DOE Contract No. **DE-AC09-96SR18500**

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March 30, 1999

## **CHARACTERIZATION OF THE LOW LEVEL WASTE REFERENCE GLASS (LRM)**

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## ABSTRACT

The Savannah River Technology Center (SRTC) has participated in a round robin testing program which was conducted under the auspices of the Department of Energy's (DOE) Tanks Focus Area (TFA) for Immobilization. The round robin, lead by Argonne National Laboratory (ANL), focused on data regarding the chemical composition and durability (as defined by ASTM C 1285, Product Consistency Test (PCT)) of the TFA reference glass (referred to as "LRM"). The LRM glass is a borosilicate glass that contains about 20 mass percent  $\text{Na}_2\text{O}$  and small amounts of other components that are either present in DOE waste streams or may be added to the low activity waste (LAW) to facilitate immobilization.

ANL provided SRTC with two vials of LRM glass. Each vial contained approximately 17 grams of glass which was sufficient to measure both chemical composition and durability (at 40° and 90°C) on each. The two LRM samples are referred to as LRM-a and LRM-b.

The SRTC-Mobile Laboratory (STRC-ML) and the SRTC Analytical Development Section (ADS) provided support for the chemical composition and durability analysis. Chemical composition of the LRM glasses (LRM-a and LRM-b) were measured by both the SRTC-ML and ADS using a combination of dissolution/fusion preparations ( $\text{LiBO}_2/\text{HNO}_3$ ,  $\text{Na}_2\text{O}_2/\text{NaOH}/\text{HCl}$ ,  $\text{CsOH}$ , and microwave) and analytical techniques (ICP-AES, AA, IC, and ISE). The oxide values from each laboratory, based on the recommended analytical technique, are reported in oxide mass percents for both LRM-a and LRM-b.

Durability was evaluated on the LRM-a and LRM-b glasses using ASTM C 1285. Tests at 40°C and 90°C were conducted in triplicate for each glass. Blanks, reference glasses (when applicable), and solution standards were used for control purposes. The SRTC-ML provided support for the solution analysis. The results of the leachate analyses were corrected for the dilution/acidification of the leachate. The corrected values were averaged for the triplicate samples and reported in ppm.

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## INTRODUCTION

The Savannah River Technology Center (SRTC) has participated in a round robin testing program which was conducted under the auspices of the Department of Energy's (DOE) Tanks Focus Area (TFA) for Immobilization. The round robin is being lead by Argonne National Laboratory (ANL) and is focused on data regarding the chemical composition and durability (as defined by ASTM C 1285, Product Consistency Test (PCT)) of the TFA reference glass (referred to as "LRM"). The LRM glass is a borosilicate glass that contains about 20 mass percent Na<sub>2</sub>O and small amounts of other components that are either present in DOE waste streams or may be added to the immobilized low activity waste (ILAW). The LRM glass also contains small amounts of the RCRA metals Ba, Cd, Cr, and Pb. Table I shows the targeted composition of the LRM glass. The glass has been shown to be non-hazardous per the Toxicity Characteristic Leach Procedure (TCLP) [EBERT 1998].

Ferro Corporation produced a 1000 lb. supply of the LRM glass for ANL from which the round robin was conducted. ANL mixed, crushed, sieved, and washed the glass to supply a single source of glass for testing. Sufficient glass was supplied to SRTC to complete both phases of the round robin; chemical analysis of the glass and inter-laboratory precision of a test method when conducted with a "standardized" test material (in an effort to document the response of the LRM glass to the PCT). The PCT was performed at 40°C and 90°C. This report describes the experimental procedures, analytical techniques utilized, and the results obtained for the LRM-a and LRM-b samples.

Table I. Targeted Composition of LRM Glass (in oxide mass %). [EBERT 1998]

Oxide	LRM	Oxide	LRM
Al <sub>2</sub> O <sub>3</sub>	10	MgO	0.1
B <sub>2</sub> O <sub>3</sub>	8	MnO	0.1
BaO	0.005	MoO <sub>3</sub>	0.1
CaO	0.5	Na <sub>2</sub> O	20
CdO	0.2	NiO	0.1
Cl	0.8	P <sub>2</sub> O <sub>5</sub>	0.5
Cr <sub>2</sub> O <sub>3</sub>	0.2	PbO <sub>2</sub>	0.1
F	1	SO <sub>3</sub>	0.2
Fe <sub>2</sub> O <sub>3</sub>	1	SiO <sub>2</sub>	54.37
HgO	0.002	SnO <sub>2</sub>	0.1
I	0.002	TiO <sub>2</sub>	0.1
K <sub>2</sub> O	1.5	ZrO <sub>2</sub>	1
La <sub>2</sub> O <sub>3</sub>	0.009		
Li <sub>2</sub> O	0.1	Total	100

## EXPERIMENTAL

### *Receipt of Glass*

SRTC received two small vials (labeled GS713) each containing approximately 17 grams of the LRM glass from ANL. For internal tracking purposes at SRTC, the two vials were labeled upon receipt as LRM-a and LRM-b. Information accompanying these samples indicated that each vial contained -100, +200 mesh size fractions which had been washed to removed the fines. The quantity of LRM glass in each vial allowed for both chemical composition and PCT (in triplicate) to be performed on both LRM-a and LRM-b. Therefore, it was decided to provide chemical composition and durability (as defined by the PCT) results on both LRM-a and LRM-b as individual glasses (i.e., two sets of data will be reported in this document).

### *Chemical Composition Analysis*

Compositional analysis was performed by both the SRTC Mobile Laboratory (SRTC-ML) and the SRTC's Analytical Development Services (ADS) Division for both LRM-a and LRM-b. Dissolution/digestion techniques as well as analytical techniques utilized are reported.

#### *SRTC-ML Glass Preparation Methods*

A ~4 g sample of LRM-a and LRM-b was obtained from each vial, placed in an appropriately labeled glass vials, and submitted to the SRTC Mobile Laboratory (SRTC-ML) for elemental analysis. The LRM glasses were prepared for compositional analysis using two fusion techniques: (1)  $\text{Na}_2\text{O}_2/\text{NaOH}/\text{HCl}$  and (2)  $\text{LiBO}_2/\text{HNO}_3$ . Table II identifies the elements analyzed, method of analysis, and the estimated detection limit.<sup>a</sup> For tracking purposes, sample submission forms and chain-of-custody forms are a part of the records package.

#### *$\text{Na}_2\text{O}_2/\text{NaOH}/\text{HCl}$ Fusion Preparation*

Approximately  $0.25 \pm 0.01$  g of crushed LRM glass was fused with 1.5 g  $\text{Na}_2\text{O}_2$  and 1.0 g NaOH at  $600^\circ\text{C}$  for 15 minutes in a Pt crucible. The fused sample was dissolved in water and 25ml of HCl acid. The fused sample was diluted to 250 ml with deionized water.

Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) was used to measure for B, Si, Al, and Li in the  $\text{Na}_2\text{O}_2/\text{NaOH}/\text{HCl}$  fusion preparation. Prior to running the solution through the ICP-AES, a 1/10 dilution on the final 250ml solution was performed. Calibration standards were matrix matched.

#### *$\text{LiBO}_2/\text{HNO}_3$ Fusion Preparation*

Approximately  $0.1 \pm 0.01$  grams of crushed LRM glass was fused with 0.3 g of  $\text{LiBO}_2$  at  $900^\circ\text{C}$  for 15 minutes in a Pt crucible. The fused sample was dissolved in a 4%  $\text{HNO}_3$  solution. The dissolved sample was diluted to a final volume of 250 ml. No further dilutions were made prior to running this solution through the ICP-AES.

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<sup>a</sup> No restrictions were placed on the method(s) used to dissolve the glass for chemical analysis, except the method(s) be reported.

Again, calibration standards were matrix matched. Anions (F, Cl, and SO<sub>3</sub>) and Hg were not analyzed by the SRTC-ML as indicated (“-”) in Table II.

Table II. Elemental Analysis Based on Dissolution Procedure and Analytical Technique for the SRTC-ML.

Element	Dissolution Technique	Analytical Technique	Detection Limit (µg/ml)
Al	Na <sub>2</sub> O <sub>2</sub> /NaOH/HCl	ICP-AES	< 0.200
B	Na <sub>2</sub> O <sub>2</sub> /NaOH/HCl	ICP-AES	< 0.180
Ba	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.01
Ca	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.010
Cd	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.020
Cl	-	-	-
Cr	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.030
F	-	-	-
Fe	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.020
Hg	-	-	-
K	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.600
La	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.030
Li	Na <sub>2</sub> O <sub>2</sub> /NaOH/HCl	ICP-AES	< 0.020
Mg	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.001
Mn	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.001
Mo	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.080
Na	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.530
Ni	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.060
P	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.640
Pb	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.220
Si	Na <sub>2</sub> O <sub>2</sub> /NaOH/HCl	ICP-AES	< 0.180
SO <sub>3</sub>	-	-	-
Sn	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.260
Ti	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.001
Zr	LiBO <sub>2</sub> /HNO <sub>3</sub>	ICP-AES	< 0.080

### *ADS Glass Preparation Methods*

#### *CsOH Fusion Dissolution*

Approximately 0.25 g of powdered LRM glass was combined with 2.5 g of CsOH·H<sub>2</sub>O in either a Zr or Ni crucible (with lid) and placed in a muffle furnace pre-heated to 500°C. The mixture was heated for 5 minutes and then removed from the furnace. After cooling, de-ionized water containing 3 – 4 drops of 30% H<sub>2</sub>O<sub>2</sub> was added to dissolve the flux pellet and this slurry transferred to a 250 ml plastic volumetric flask. Twenty-five (25) ml of concentrated HNO<sub>3</sub> was then added to dissolve the slurry.

De-ionized water was added to bring the volume to 250 ml and the solution mixed by capping the flask and inverting several times.

#### *Na<sub>2</sub>O<sub>2</sub>/NaOH Fusion Dissolution*

Approximately 0.25 g of powdered glass was combined with 1.5 g of Na<sub>2</sub>O<sub>2</sub> and 1.0 g of NaOH in either a Zr or Ni crucible (with lid) and placed in a muffle furnace pre-heated to 675°C. The mixture was heated for 5 minutes and then removed from the furnace. After cooling, de-ionized water containing 3 – 4 drops of 30% H<sub>2</sub>O<sub>2</sub> was added to dissolve the flux pellet and this slurry transferred to a 250 ml plastic volumetric flask. Twenty-five (25) ml of concentrated HNO<sub>3</sub> was then added to dissolve the slurry. The de-ionized water was added to bring the volume to 250 ml and the solution mixed by capping the flask and inverting several times.

#### *Microwave Dissolution*

Approximately 0.25 grams of powdered glass was combined with 5 ml of concentrated HF and 5 ml of concentrated HNO<sub>3</sub> in a Teflon pressure vessel. The vessel was capped and heated at 60% power for 12 minutes. After cooling, 40 ml of 0.6M H<sub>3</sub>BO<sub>3</sub> and 5 ml concentrated HCl were added and the mixture heated again at 60% power for 12 minutes. After cooling, the solution was transferred to a plastic volumetric flask and diluted with de-ionized water to bring the volume to 250 ml. The solution was mixed by capping the flask and inverting several times.

Table III identifies the dissolution procedure and analytical technique used for each elemental analysis. Estimated detection limits for each technique is also provided. Analytical techniques utilized by ADS include ICP-ES, Atomic Absorption (AA), and Ion Chromatography (IC), and Ion Selective Electrode (ISE).

### ***Glass Durability (PCT) Analysis***

The round robin instructions stated that “the procedure for PCT Method A is to be followed for tests at both 40° and 90°C”.<sup>b</sup> The glass “as-received” required no additional sample preparation prior to initiating the PCTs (i.e., grinding, sieving, and washing performed by ANL). For tests at 40°C and 90°C, 1.5 grams of -100, +200 mesh glass was placed into an unsensitized Type 304L stainless steel vessel (~22 ml capacity). Fifteen (15) ml of ASTM Type I water (exactly 10 times the mass of glass used) was added to each LRM glass. PCTs for both the LRM-a and LRM-b glasses were performed in triplicate at both temperatures. Duplicate blank tests with ASTM Type I water were also run within each test. Although a standard reference material was not required, the Environmental Assessment (EA) [JANTZEN 1992] and Approved Reference Material (ARM-1) glasses were included with the 90°C test. PCT data sheets were utilized to record the vessel identification number, sample ID, weight of vessel (empty), weight of vessel with glass, and the weight of vessel with glass and water. The data sheets are also a part of the record package.

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<sup>b</sup> ASTM 1285 Method A prescribes a strict set of test conditions, one being that the test is performed at 90 ± 2°C. ASTM 1285 Method B allows more flexibility in the test parameters including test temperature. Therefore, SRTC used Method B for the 40°C tests with all other tests parameters (such as glass mass, water mass, vessel type, etc) “equivalent” that that used for Method A.

After the 7-day test, vessels were allowed to cool to room temperature. The final weight of each vessel and the solution pH were recorded on the data sheet. Leachate solutions were then filtered through a 0.45  $\mu\text{m}$  pore size filter. Six (6) ml of each leachate solution was then acidified with 4 ml of 0.4M  $\text{HNO}_3$  to ensure that the cations remain in solution. Leachate solutions and blanks were then analyzed for Si, B, Na, and Li concentration by the SRTC-ML using ICP-AES. A solution standard was also submitted with the PCT leachates for control purposes.

Table III. Elemental Analysis Based on Dissolution Procedure and Analytical Technique for ADS.

Element	Dissolution Technique	Analytical Technique	Detection Limit ( $\mu\text{g/ml}$ )
Al	CsOH in Zr	ICP-ES	0.025
B	CsOH in Zr	ICP-ES	0.010
Ba	CsOH in Zr	ICP-ES	0.005
Ca	Microwave	ICP-ES	0.010
Cd	CsOH in Zr	ICP-ES	0.005
Cl	$\text{Na}_2\text{O}_2/\text{NaOH}$	ISE	1.000
Cr	Microwave	ICP-ES	0.005
F	$\text{Na}_2\text{O}_2/\text{NaOH}$	ISE	1.000
Fe	CsOH in Zr	ICP-ES	0.015
Hg	Microwave	Cold Vapor AA	0.001
K	Microwave	Flame AA	0.100
La	CsOH in Zr	ICP-ES	0.010
Li	Microwave	ICP-ES	0.020
Mg	CsOH in Zr	ICP-ES	0.010
Mn	CsOH in Zr	ICP-ES	0.010
Mo	CsOH in Zr	ICP-ES	0.010
Na	Microwave	ICP-ES	0.050
Ni	CsOH in Zr	ICP-ES	0.040
P	CsOH in Zr	ICP-ES	0.100
Pb	CsOH in Zr	ICP-ES	0.250
Si	CsOH in Zr	ICP-ES	0.025
$\text{SO}_3$	$\text{Na}_2\text{O}_2/\text{H}_2\text{O}$	IC	1.000 ( $\text{SO}_4$ )
Sn	CsOH in Zr	ICP-ES	0.250
Ti	Microwave	ICP-ES	0.015
Zr	CsOH in Ni	ICP-ES	0.040

## RESULTS

### *Glass Composition Analysis*

#### *SRTC-ML Analysis*

Table IV summarizes the SRTC-ML analytical results for the LRM-a and LRM-b glasses (in oxide mass percent). Also shown in Table IV is the "target" composition of the LRM glass as defined by Ebert et. al. [EBERT 1998]. The oxide values reported by the SRTC-ML are not averaged values but individual reads.

Based on the composition shown in Table I, the concentrations of Sn, Ba, La, and Mo in the  $\text{LiBO}_2/\text{HNO}_3$  preparations were below the detection limits of the ICP-AES. Therefore, these elements were not analyzed ("NA") and their respective oxides are reported as such. As previously mentioned, anions (F, Cl, and  $\text{SO}_3$ ) and Hg were not analyzed by the SRTC-ML as indicated ("NA").

#### *ADS Analysis*

Triplicate values were reported by ADS for each element. These values were converted to oxides and the averaged values for LRM-a and LRM-b (in oxide mass percentage) are reported in Table V. It should be noted that the elemental values reported for the initial replicate of LRM-b (i.e., LRM-b-1) based on the CsOH dissolution in Zr crucibles appeared to be inconsistent with the reported replicate (LRM-b-2 and LRM-b-3) data. Therefore, the values reported in Table V for Al, B, Ba, Cd, Fe, La, Mg, Mn, Mo, Ni, P, Pb, and Sn (i.e., those elements determined by the CsOH dissolution in Zr crucibles as shown in Table III) are averages of the second and third replicates only. All other oxide values for the LRM-b glass are averages of triplicate analyses. Also shown in Table V is the "target" composition of the LRM glass as defined by Ebert et. al. [EBERT 1998].

Table IV. SRTC-ML Comparison of Target and "As-Measured" LRM Glass Compositions (in oxide mass %).

Oxide	Target Composition	LRM-a (measured)	LRM-b (measured)
Al <sub>2</sub> O <sub>3</sub>	10	10.1	10.0
B <sub>2</sub> O <sub>3</sub>	8	8.07	8.39
BaO	0.005	NA	NA
CaO	0.5	0.490	0.486
CdO	0.2	0.156	0.152
Cl	0.8	-	-
Cr <sub>2</sub> O <sub>3</sub>	0.2	0.177	0.169
F	1	-	-
Fe <sub>2</sub> O <sub>3</sub>	1	1.39	1.39
HgO	0.002	-	-
K <sub>2</sub> O	1.5	1.31	1.32
La <sub>2</sub> O <sub>3</sub>	0.009	NA	NA
Li <sub>2</sub> O	0.1	0.168	0.167
MgO	0.1	0.096	0.090
MnO	0.1	0.081	0.076
MoO <sub>3</sub>	0.1	NA	NA
Na <sub>2</sub> O	20	20.6	20.2
NiO	0.1	0.182	0.164
P <sub>2</sub> O <sub>5</sub>	0.5	0.559	0.540
PbO	0.1	0.123	0.090
SO <sub>3</sub>	0.2	-	-
SiO <sub>2</sub>	54.37	54.5	53.9
SnO <sub>2</sub>	0.1	NA	NA
TiO <sub>2</sub>	0.1	0.087	0.090
ZrO <sub>2</sub>	1	0.864	0.867
Total	100.00	99.0	98.0

Table V. ADS Comparison of Target and "As-Measured" LRM Glass Compositions (in oxide mass %).

Oxide	Target Composition	LRM-a (measured)	LRM-b (measured)
Al <sub>2</sub> O <sub>3</sub>	10	9.85	9.53
B <sub>2</sub> O <sub>3</sub>	8	7.99	7.73
BaO	0.005	BDL	BDL
CaO	0.5	0.52	0.52
CdO	0.2	0.17	0.17
Cl	0.8	0.17	0.13
Cr <sub>2</sub> O <sub>3</sub>	0.2	0.21	0.20
F	1	0.67	0.72
Fe <sub>2</sub> O <sub>3</sub>	1	1.54	1.48
HgO	0.002	BDL	BDL
K <sub>2</sub> O	1.5	1.28	1.26
La <sub>2</sub> O <sub>3</sub>	0.009	BDL	BDL
Li <sub>2</sub> O	0.1	0.08	0.07
MgO	0.1	0.11	0.11
MnO	0.1	0.08	0.07
MoO <sub>3</sub>	0.1	0.11	0.11
Na <sub>2</sub> O	20	20.49	19.91
NiO	0.1	0.19	0.19
P <sub>2</sub> O <sub>5</sub>	0.5	0.55	0.56
PbO	0.1	0.08	0.08
SO <sub>3</sub>	0.2	0.40	0.47
SiO <sub>2</sub>	54.37	56.29	54.25
SnO <sub>2</sub>	0.1	BDL	BDL
TiO <sub>2</sub>	0.1	0.10	0.09
ZrO <sub>2</sub>	1	1.00	1.01
Total	100.00	101.87	98.65

**GLASS DURABILITY (PCT) ANALYSIS**

Tables VI and VII summarize the initial weight (vessel, glass, and water), final weight, % loss, and final pH for the 40° and 90°C tests respectively. The % loss was calculated based on water loss alone (i.e., vessel weight was not considered). All tests are considered valid based on the loss percentage (i.e., < 5%) with the exception of the initial replicate of LRM-a (i.e., LRM-a-1) at 40°C which had a total loss of 5.79%.

Table VI. Vessel Weights, % Loss and Final pH for the 40°C tests.

Vessel #	Sample ID	Initial Weight (g)	Final Weight (g)	% Loss	PH
P185	Blank-1	14.992	14.668	2.16%	6.78
P186	Blank-2	14.993	14.774	1.46%	6.76
P191	LRM-a-1	16.496	15.541	5.79%	9.91
P192	LRM-a-2	16.494	16.086	2.47%	9.98
P193	LRM-a-3	16.504	16.082	2.56%	9.96
P194	LRM-b-1	16.493	15.889	3.66%	9.95
P195	LRM-b-2	16.493	16.157	2.04%	9.99
P196	LRM-b-3	16.498	16.02	2.90%	10.00

Table VII. Vessel Weights, % Loss and Final pH for the 90°C tests.

Vessel #	Sample ID	Initial Weight	Final Weight	% Loss	PH
P1	Blank-1	14.996	14.698	1.99%	6.72
P2	Blank-2	14.995	14.829	1.11%	6.82
P3	LRM-a-1	16.494	15.993	3.04%	11.02
P4	LRM-a-2	16.497	16.314	1.11%	11.02
P5	LRM-a-3	16.496	16.307	1.15%	11.04
P6	LRM-b-1	16.496	16.289	1.25%	11.04
P7	LRM-b-2	16.499	16.101	2.41%	11.06
P8	LRM-b-3	16.505	16.245	1.58%	11.06
P24	EA-1	16.51	15.928	3.53%	11.83
P40	EA-2	16.503	16.255	1.50%	11.84
P46	EA-3	16.495	16.18	1.91%	11.86

The results of the leachate analyses were corrected for the dilution/acidification of the leachate performed prior to submittal to the SRTC-ML for analysis. The corrected values were averaged for the triplicate samples and reported in ppm. These values are presented in Table VIII for the samples tested at 40°C and Table IX for the samples tested at 90°C.

Table VIII. Averaged Concentrations of B, Si, Na, and Li (in ppm) from 40°C PCT Test Corrected for Dilution Factor.

Sample ID	B (ppm)	Si (ppm)	Na (ppm)	Li (ppm)	pH (average)
LRM-a	2.59	13.47	22.28	0.00	9.95
LRM-b	2.47	13.06	21.62	0.00	9.98
Blanks	< 0.180	< 0.180	< 0.530	< 0.100	6.78

Table IX. Averaged Concentrations of B, Si, Na, and Li (in ppm) from 90°C PCT Test Corrected for Dilution Factor.

Sample ID	B (ppm)	Si (ppm)	Na (ppm)	Li (ppm)	pH (average)
LRM-a	29.228	84.128	169.478	0.196	11.03
LRM-b	29.673	84.295	170.256	0.199	11.05
EA	627.348	983.296	1822.587	195.595	11.84
Blanks	< 0.180	< 0.180	< 0.530	< 0.100	6.77

Jantzen, et al. [Jantzen 1995], control charted the leachate concentrations for B, Si, Na, and Li obtained by the PCT procedure on ARM-1 glasses over a six year span. From these data, an upper control limit (UCL) and lower control limit (LCL) were identified for each of the elements and the pH of the leachate. The leachate concentrations for B, Si, Na, and Li and the pH are compared the LCL and UCL in Table X to validate the PCT test conditions.

Table X. Comparison of ARM-1 Glass from LRM Work to Control Charted ARM-1 Results.

	This Study	Mean	LCL	UCL
B	19.78	19.03	11.58	26.48
Si	67.85	65.22	41.84	88.60
Na	41.62	40.69	20.08	61.30
Li	15.75	15.12	7.66	22.59
PH	10.46	10.17	9.29	11.05

Elemental values (ppm) for the solution standard are compared to averaged measured values (see Appendix A for individual measurements) reported by the SRTC-ML in Table XI. The three solution standards were intermittently run with the PCT solutions during the ICP-AES analysis for control purposes.

Table XI. Comparison of the Solution Standard and Measured Elemental Concentrations (in ppm).

Element	Solution Standard (ppm)	Average Value (40°C) (ppm)	Average Value (90°C) (ppm)
B	20	21.4	21.2
Si	50	50.3	49.6
Na	81	79.4	81.1
Li	10	9.8	9.91

## REFERENCES

### **ASTM C 1285**

Standardized Test Methods for determining Chemical Durability of Nuclear Waste Glasses: The Product consistency Test (PCT), Standard C 1285-97, annual Book of ASTM Standards, Vol. 12.01, American Society for Testing and Materials, West Conshohocken, PA.

### **EBERT 1998**

W.L. Ebert, S.F. Wolf, and D.M. Strachan, "Evaluation of A Standard Test Method and Material for Low-Activity Waste Product Acceptance," Proceedings of XVIII International Glass Conference, July 5-10, 1998, San Francisco, CA. Eds. M.K. Choudary, N.T. Huff, and C.H. Drummond III (1998).

### **JANTZEN 1992**

C.M. Jantzen, N.E. Bibler, D.C. Beam, C.L. Crawford, and M.A. Pickett, Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Reference Material (U), Westinghouse Savannah River Company, WSRC-TR-92-346, Rev. 1 (1992).

### **JANTZEN 1995**

C.M. Jantzen, M.A. Pickett, K.G. Brown, T.B. Edwards, and D.C. Beam, Process/Product Models for the Defense Waste Processing Facility (DWPF): Part 1. Predicting the Glass Durability from Composition Using a Thermodynamic *E*nergy *H*ydration *M*odel (THERMO) (U), Westinghouse Savannah River Company, WSRC-TR-93-672, Rev. 1 (1995).

### **WOLF 1998**

A Data Base and a Standard Reference Material for Use in Acceptance Testing of Low Activity Waste Products, S.F. Wolf, W.L. Ebert, J.S. Luo, and D.M. Strachan, Argonne National Laboratory report ANL-98/9 (1998).

## **APPENDIX A**

PCT Raw Data

**Raw Elemental Data (ug/ml) From ICP-AES (40°C PCT)**

	<u>STD1</u>	<u>BLK1</u>	<u>BLK2</u>
B	20.9	0.181	<0.180
Si	51.1	<0.180	<0.180
Na	78.3	0.698	<0.530
Li	10.0	<0.100	<0.100

	<u>STD2</u>	<u>LRM-a-1</u>	<u>LRM-a-2</u>	<u>LRM-a-3</u>
B	21.4	1.66	1.52	1.49
Si	51.1	8.30	7.71	8.23
Na	78.7	13.9	12.8	13.4
Li	9.59	<0.100	<0.100	<0.100

	<u>LRM-b-1</u>	<u>LRM-b-2</u>	<u>LRM-b-3</u>	<u>STD3</u>
B	1.54	1.42	1.48	22.1
Si	8.22	7.05	7.28	48.7
Na	12.8	12.7	13.4	81.1
Li	<0.100	<0.100	<0.100	9.82

**Raw Elemental Data (ug/ml) From ICP-AES (90°C PCT)**

	<u>STD1</u>	<u>BLK1</u>	<u>BLK2</u>
B	20.9	<0.180	<0.180
Si	50.1	<0.180	<0.180
Na	78.4	0.842	<0.530
Li	9.81	<0.100	<0.100

	<u>EA-1</u>	<u>EA-2</u>	<u>EA-3</u>
B	376	362	391
Si	591	572	607
Na	1090	1050	1030
Li	120	115	117

	<u>LRM-a-1</u>	<u>LRM-a-2</u>	<u>LRM-a-3</u>	<u>STD-2</u>
B	17.5	17.1	18.0	21.6
Si	49.6	51.4	50.4	49.9
Na	101	102	102	82.5
Li	0.111	0.115	0.126	9.93

	<u>LRM-b-1</u>	<u>LRM-b-2</u>	<u>LRM-b-3</u>	<u>STD-3</u>
B	18.1	17.8	17.5	21.0
Si	49.6	50.8	51.3	48.7
Na	97.4	104	105	82.3
Li	0.120	0.116	0.123	9.99

# Westinghouse Savannah River Company Document Approval Sheet

Document No.  
WSRC-TR-99-00095, Rev. 0

Title  
**Characterization of the Low Level Waste Reference Glass (LRM)**

Primary Author/Contact (Must be WSRC) <b>David Peeler</b>	Location <b>773-43A</b>	Phone No. <b>5-0623</b>	Position <b>Senior Research Sci.</b>	User ID <b>L6814</b>
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Organization Code <b>L3100</b>	Organization (No Abbreviations) <b>Immobilization Technology Section</b>
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Other Authors  
**A.D. Cozzi, D.R. Best, C.J. Coleman, and I.A. Reamer**

Deadline Date for Approval  
~~4/30/99~~ **5/28/99**

Has an invention disclosure been submitted related to this information?  Yes  No

Disclosure No. (If Known) \_\_\_\_\_ Title \_\_\_\_\_ Date Submitted \_\_\_\_\_

Do you intend to submit an invention disclosure?  Yes  No If yes, projected date \_\_\_\_\_

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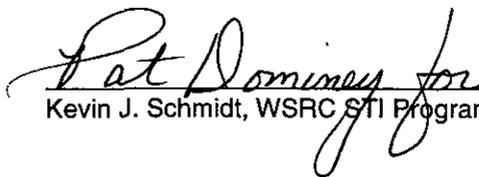
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MSD-STI-97-4218

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I. DETAILS OF REQUEST FOR RELEASE

Document Number: WSRC-TR-99-00095, Rev. 0

Author's Name: D. Peeler

Location: 773-43A

Phone 5-0623

Department: Immobilization Technology Section

Document Title: Characterization of the Low Level Waste Reference Glass (LRM)

Presentation/Publication:

Meeting/Journal:

Location:

Meeting Date:

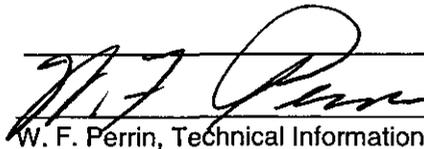
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c. Patent Assignee .....

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B. STI PRODUCT TITLE Characterization of the Low Level Waste Reference Glass (LRM).....

C. AUTHOR(s) D. Peeler.....

E-mail Address(es):

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\_\_\_\_ 2. DOE Contract Number(s) DE-AC09-96SR18500.....

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Keywords PCT, Chemical Composition, TFA.....

K. DESCRIPTION/ABSTRACT

The Savannah River Technology Center (SRTC) has participated in a round robin testing program which was conducted under the auspices of the Department of Energy's (DOE) Tanks Focus Area (TFA) for Immobilization.

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 E-mail \_\_\_\_\_ Phone (803) 725-2321  
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