Analysis of the Sludge Batch 7b
(Macrobatch 9) DWPF Pour Stream Glass Sample

F.C. Johnson, C.L. Crawford and J.M. Pareizs

November 2013

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F.C. Johnson
C.L. Crawford
J.M. Pareizs

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Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.
### REVIEWS AND APPROVALS

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EXECUTIVE SUMMARY

The Defense Waste Processing Facility (DWPF) began processing Sludge Batch 7b (SB7b), also referred to as Macrobatch 9 (MB9), in January 2012. SB7b is a blend of the heel of Tank 40 from Sludge Batch 7a (SB7a) and the SB7b material that was transferred to Tank 40 from Tank 51. SB7b was processed using Frit 418.

During processing of each sludge batch, the DWPF is required to take at least one glass sample to meet the objectives of the Glass Product Control Program (GPCP), which is governed by the DWPF Waste Form Compliance Plan, and to complete the necessary Production Records so that the final glass product may be disposed of at a Federal Repository. Two pour stream glass samples were collected while processing SB7b. The samples were transferred to the Savannah River National Laboratory (SRNL) where one was analyzed and the other was archived. The following conclusions were drawn from the analytical results provided in this report:

- The sum of oxides for the official SB7b pour stream glass is within the Product Composition Control System (PCCS) limits (95-105 wt%).
- The average calculated Waste Dilution Factor (WDF) for SB7b is 2.3. In general, the measured radionuclide content of the official SB7b pour stream glass is in good agreement with the calculated values from the Tank 40 dried sludge results from the SB7b Waste Acceptance Program Specification (WAPS) sample.
- As in previous pour stream samples, ruthenium and rhodium inclusions were detected by Scanning Electron Microscopy-Electron Dispersive Spectroscopy (SEM-EDS) in the SB7b pour stream sample.
- The Product Consistency Test (PCT) results indicate that the official SB7b pour stream glass meets the waste acceptance criteria for durability with a normalized boron release of 0.8 g/L, which is an order of magnitude less than the Environmental Assessment (EA) glass.
- The measured density of the SB7b pour stream glass was 2.70 g/cm³.
- The Fe²⁺/∑Fe ratio of the SB7b pour stream samples was 0.07.
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<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>AD</td>
<td>Analytical Development</td>
</tr>
<tr>
<td>ARG-1</td>
<td>Analytical Reference Glass 1</td>
</tr>
<tr>
<td>AR</td>
<td>Aqua Regia</td>
</tr>
<tr>
<td>ARM</td>
<td>Approved Reference Material</td>
</tr>
<tr>
<td>ASP</td>
<td>Analytical Study Plan</td>
</tr>
<tr>
<td>CPC</td>
<td>Chemical Process Cell</td>
</tr>
<tr>
<td>DWPF</td>
<td>Defense Waste Processing Facility</td>
</tr>
<tr>
<td>EA</td>
<td>Environmental Assessment</td>
</tr>
<tr>
<td>EDS</td>
<td>Electron Dispersive Spectroscopy</td>
</tr>
<tr>
<td>GPCP</td>
<td>Glass Product Control Program</td>
</tr>
<tr>
<td>ICP-AES</td>
<td>Inductively Coupled Plasma – Atomic Emission Spectroscopy</td>
</tr>
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<td>ICP-MS</td>
<td>Inductively Coupled Plasma – Mass Spectrometry</td>
</tr>
<tr>
<td>MB</td>
<td>Macrobatch</td>
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<tr>
<td>MFT</td>
<td>Melter Feed Tank</td>
</tr>
<tr>
<td>PCCS</td>
<td>Product Composition Control System</td>
</tr>
<tr>
<td>PCT</td>
<td>Product Consistency Test</td>
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<tr>
<td>PF</td>
<td>Peroxide Fusion</td>
</tr>
<tr>
<td>PS</td>
<td>Pour Stream</td>
</tr>
<tr>
<td>REDOX</td>
<td>REduction/OXidation</td>
</tr>
<tr>
<td>RSD</td>
<td>Relative Standard Deviation</td>
</tr>
<tr>
<td>SB</td>
<td>Sludge Batch</td>
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<td>SEM</td>
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<tr>
<td>SME</td>
<td>Slurry Mix Evaporator</td>
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<tr>
<td>SRAT</td>
<td>Sludge Receipt and Adjustment Tank</td>
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<tr>
<td>SRNL</td>
<td>Savannah River National Laboratory</td>
</tr>
<tr>
<td>THERMO</td>
<td>Thermodynamic Hydration Energy Reaction MOdel</td>
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<tr>
<td>TTQAP</td>
<td>Task Technical and Quality Assurance Plan</td>
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<tr>
<td>UV-Vis</td>
<td>Ultraviolet-Visible</td>
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<td>Waste Acceptance Product Specifications</td>
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<td>WCP</td>
<td>Waste Form Compliance Plan</td>
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<tr>
<td>WDF</td>
<td>Waste Dilution Factor</td>
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1.0 Introduction
The Defense Waste Processing Facility (DWPF) began processing Sludge Batch 7b (SB7b), also referred to as Macrobatch 9 (MB9), in January 2012. SB7b was a blend of transfers of material from Tank 7 and the Sludge Batch 7a (SB7a) heel in Tank 40. Frit 418 was used during processing of SB7b.2

Sludge is received into the DWPF Chemical Process Cell (CPC) and is processed through the Sludge Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) tank where the frit is added. The treated sludge slurry and frit is then transferred to the Melter Feed Tank (MFT) and fed to the melter. During processing of each sludge batch, the DWPF is required to take at least one glass sample to meet the objectives of the Glass Product Control Program (GPCP), which is governed by the DWPF Waste Form Compliance Plan, and to complete the necessary Production Records so that the final glass product may be disposed of at a Federal Repository.

The DWPF requested various analyses of a radioactive glass sample obtained from the melter pour stream during processing of SB7b.4 Sample analysis followed the Task Technical and Quality Assurance Plan (TTQAP)5 and an Analytical Study Plan (ASP).6 Two pour stream (PS) glass samples were delivered to the Savannah River National Laboratory (SRNL) from the DWPF (Table 1-1). The sample collected while filling canister S04023 was selected as the official pour stream sample for SB7b and full analysis was requested. This report details the visual observations of the as-received SB7b PS glass sample as well as results for the chemical composition, Product Consistency Test (PCT), radionuclide content, noble metals, and glass density. The sample collected while filling canister S04025 was archived in Cell 16 of the SRNL Shielded Cells according to procedure.7

<table>
<thead>
<tr>
<th>Glass Canister</th>
<th>Sample Date</th>
<th>MFT Batch</th>
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<th>Notes</th>
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<td>S04023</td>
<td>April 2013</td>
<td>661</td>
<td>PC0122</td>
<td>Analysis</td>
</tr>
<tr>
<td>S04025</td>
<td>April 2013</td>
<td>661</td>
<td>PC0123</td>
<td>Archive</td>
</tr>
</tbody>
</table>

2.0 Experimental Procedure

2.1 Visual Examination, Extraction and Washing
Upon arrival at SRNL, the SB7b PS glass sample was inspected, removed from the Pt/Au collection boat and washed according to procedure prior to analysis.8

2.2 Chemical Composition
A subsample of SB7b PS glass was ground and then sieved to -200 mesh. Quadruplicate samples of the pour stream glass were digested by two separate methods: aqua regia (AR) and sodium peroxide fusion (PF). Three Analytical Reference Glass (ARG) standards were also digested by each method and submitted along with the samples. All of the prepared samples were submitted to Analytical Development (AD) and analyzed by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES). A multi-element standard and blank were also included in the analyses in order to assess the performance of the instrument over the course of the analyses.

2.3 Radionuclide Composition
The SB7b PS glass sample was prepared in quadruplicate using a PF digestion and was analyzed by AD using Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS) to determine actinide
and fission product content. Aliquots of the PF and AR digestions were analyzed by counting methods and alpha spectroscopy to calculate the radionuclide concentration. An aliquot was collected in quadruplicate using the AR digestion and was analyzed by beta counting to determine Tc-99 content. The reportable radionuclides for the GPCP and WCP not measured in this study were calculated from the SB7b total dried solids results using a calculated Waste Dilution Factor (WDF).

2.4 Noble Metals
Noble metal concentrations were analyzed in the SB7b PS glass sample using ICP-MS from the PF dissolution. The total silver concentration is calculated using the measured concentration of $^{109}$Ag and the calculated concentration of $^{107}$Ag. Due to interference from Cd, the palladium concentration is calculated using the sum of the measured concentration of $^{105}$Pd and the calculated concentrations of $^{106}$Pd, $^{107}$Pd, $^{108}$Pd, and $^{110}$Pd using their fission yields. The total concentration of ruthenium is calculated from the sum of the measured concentrations of three isotopes: $^{101}$Ru, $^{102}$Ru, and $^{104}$Ru. The reported concentration of rhodium is from the measured concentration of a single isotope, $^{103}$Rh.

In addition, a sample of SB7b PS glass sample (-200 mesh) was analyzed using Scanning Electron Microscopy (SEM) along with Energy Dispersive Spectroscopy (EDS) to image and analyze any inhomogeneities in the glass.

2.5 PCT
The PCT was performed on quadruplicate samples of SB7b PS glass sample to assess chemical durability using Method A of the procedure. Also included was the Environmental Assessment (EA) glass, the Approved Reference Material (ARM) glass and blanks from the sample cleaning batch. Samples were ground, washed and prepared according to standard procedure. ARM and EA were only prepared in triplicate. The resulting solutions were sampled (filtered and acidified) and analyzed by AD. Samples of a multi-element standard were also included with the glass samples as a check of the accuracy of the ICP-AES. Normalized release rates were calculated based on the average measured composition using the average of the leachate concentrations.

2.6 Density
The density of SB7b PS glass sample was measured in triplicate with a Hubbard specific gravity bottle. By using the masses of the empty bottle ($m_0$), bottle and sample ($m_1$), bottle, sample and water ($m_2$) and bottle and water ($m_3$), the density of the sample ($\rho_s$) is calculated by

$$\rho_s = \frac{\rho_{H2O} (m_1 - m_0)}{(m_3 - m_0) - (m_2 - m_1)}$$

where $\rho_{H2O}$ is the density of water at the measurement temperature. A reference glass was measured prior to its placement into the shielded cells via the Archimedes method. This same

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$^a$ Zr-93 content was determined using the AR dissolution.

$^b$ ASTM C1285 states that the sample mass must be $\geq 1$ g; however, the sample mass of EA-2 was 0.998 g. The difference of 0.002 g will not have any impact on the technical conclusions in this report.

$^c$ The measured concentration of uranium in the SB7b-3 leachate was below the detection limit. It was excluded from the normalized release rate calculation.

$^d$ The average measured compositions for ARM and EA were taken from Table B.1, page B.8 of Reference 16.

$^e$ The density of H$_2$O was assumed to be 1 g/cm$^3$ for all measurements.

$^f$ The density of a sample of NIST 1830 glass was determined to be 2.49 g/cm$^3$ using the Archimedes method.
sample was remeasured in the shielded cells in order to verify the measurement technique.

2.7 REDOX
A sample of SB7b PS glass was ground and then sieved to -200 mesh. The ground SB7b glass was then prepared for REDOX measurement and analyzed via UV-Vis spectroscopy according to procedure.\(^\text{18}\) In addition to the pour stream sample, the EA glass was included in each set of measurements as an internal check of the measured REDOX value.

2.8 Quality Assurance
Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.\(^\text{19}\)

3.0 Results and Discussion

3.1 Visual Examination
Upon receipt inspection, the SB7b glass sample appeared black and shiny with no visible signs of any surface films. Surface films have been observed on the surfaces of previous pour stream samples\(^\text{20,21}\) and have been attributed to salt deposits.

3.2 Chemical Composition
Table 5-1 and Table 5-2 in Appendix A provide the measured elemental data from glasses prepared using aqua regia and peroxide fusion, respectively. Detectable values were measured for the blank sample for Ba, Ca, Na and Zn (AR) and Fe, Li and Mg (PF). All of the blank measured values were less than 10% of the lowest measured sample value for either ARG-1 or the SB7b PS sample except for Zn. The concentration of Zn is so low that there is no practical significance.

3.2.1 ARG-1
Table 3-1 shows a comparison of the published\(^\text{11}\) and measured composition of the ARG-1 glass. The measured value is the average of three replicates from either the aqua regia or peroxide fusion data as noted in the table. In general, the measured values are consistent with the published values and relative standard deviation (%RSD) values for the major glass components (> 0.5 wt%) are less than 10%, indicating good precision in the results. The sum of oxides is within the Product Composition Control System (PCCS) acceptance limits (the interval of 95 to 105 wt%).

3.2.2 SB7b PS glass sample
Table 3-2 lists the oxide composition of the SB7b PS glass sample glass. The measured value is the average of four replicates from either the aqua regia or peroxide fusion data as noted in the table. Some of the analytes were below the detection limit of the instrument and are noted by a “<.” The %RSD values for the major glass components (> 0.5 wt%) are less than 10%, indicating good precision in the results. The sum of oxides is within the Product Composition Control System (PCCS) acceptance limits (the interval of 95 to 105 wt%).
### Table 3-1. Published\(^{11}\) and Measured Values of ARG-1

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Published (wt%)</th>
<th>Measured (wt%)</th>
<th>% RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al(_2)O(_3)</td>
<td>4.73</td>
<td>4.72</td>
<td>1.6</td>
</tr>
<tr>
<td>B(_2)O(_3)</td>
<td>8.67</td>
<td>8.19</td>
<td>1.8</td>
</tr>
<tr>
<td>BaO</td>
<td>0.09</td>
<td>0.09</td>
<td>5.2</td>
</tr>
<tr>
<td>CaO</td>
<td>1.43</td>
<td>1.43</td>
<td>5.1</td>
</tr>
<tr>
<td>Cr(_2)O(_3)</td>
<td>0.09</td>
<td>0.09</td>
<td>3.4</td>
</tr>
<tr>
<td>Fe(_2)O(_3)</td>
<td>14.0</td>
<td>13.69</td>
<td>1.4</td>
</tr>
<tr>
<td>K(_2)O</td>
<td>2.71</td>
<td>2.47</td>
<td>6.4</td>
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<tr>
<td>Li(_2)O</td>
<td>3.21</td>
<td>3.07</td>
<td>1.5</td>
</tr>
<tr>
<td>MgO</td>
<td>0.86</td>
<td>0.84</td>
<td>1.7</td>
</tr>
</tbody>
</table>

Peroxide Fusion: Al, B, Fe, Li, Mg, Mn, Ni, Si and Ti
Aqua Regia: Ba, Ca, Cr, K, Na, P, Zn and Zr

### Table 3-2. Average Measured Composition of SB7b PS glass sample

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Measured (wt%)</th>
<th>%RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al(_2)O(_3)</td>
<td>7.58</td>
<td>2.4</td>
</tr>
<tr>
<td>B(_2)O(_3)</td>
<td>4.72</td>
<td>2.0</td>
</tr>
<tr>
<td>BaO</td>
<td>0.05</td>
<td>2.5</td>
</tr>
<tr>
<td>BeO</td>
<td>&lt;0.001</td>
<td>2.2</td>
</tr>
<tr>
<td>CaO</td>
<td>0.43</td>
<td>2.5</td>
</tr>
<tr>
<td>CdO</td>
<td>0.02</td>
<td>2.3</td>
</tr>
<tr>
<td>Ce(_2)O(_3)</td>
<td>0.07</td>
<td>3.0</td>
</tr>
<tr>
<td>CoO</td>
<td>0.01</td>
<td>4.6</td>
</tr>
<tr>
<td>Cr(_2)O(_3)</td>
<td>0.08</td>
<td>2.2</td>
</tr>
<tr>
<td>CuO</td>
<td>0.19</td>
<td>1.3</td>
</tr>
<tr>
<td>Fe(_2)O(_3)</td>
<td>8.67</td>
<td>1.8</td>
</tr>
<tr>
<td>Gd(_2)O(_3)</td>
<td>0.04</td>
<td>4.1</td>
</tr>
<tr>
<td>K(_2)O</td>
<td>&lt;0.34</td>
<td>2.1</td>
</tr>
<tr>
<td>La(_2)O</td>
<td>0.04</td>
<td>4.4</td>
</tr>
<tr>
<td>Li(_2)O</td>
<td>4.83</td>
<td>3.1</td>
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<tr>
<td>MgO</td>
<td>0.22</td>
<td>2.2</td>
</tr>
<tr>
<td>MnO</td>
<td>1.66</td>
<td>1.7</td>
</tr>
</tbody>
</table>

Peroxide Fusion: Al, B, Cu, Fe, Li, Mg, Mn, Ni, Si, Th, Ti and U
Aqua Regia: Ba, Be, Ca, Cd, Ce, Co, Cr, Gd, K, La, Mo, Na, P, Pb, Sb, Sn, Sr, V, Zn and Zr
3.2.3 WDF

The WDF for a specific sludge batch is given by

\[
WDF(i) = \frac{CS(i)}{CG(i)}
\]

where \(CS(i)\) is the concentration of component \(i\) in the dried Tank 40 sludge \(^{22}\) (as measured) and \(CG(i)\) is the concentration of component \(i\) in the corresponding pour stream glass sample. Table 3-3 contains the calculated WDF values for Al, Ca, Fe and Mn for SB7b. The average WDF value will be used in Section 3.3 to calculate the concentration of radionuclides that were not directly measured in the glass.

**Table 3-3. Waste Dilution Factor for the SB7b PS glass sample Glass**

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (wt%)</th>
<th>WDF</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>SB7b WAPS</td>
<td>Glass</td>
</tr>
<tr>
<td>Al</td>
<td>9.18</td>
<td>4.01</td>
</tr>
<tr>
<td>Ca</td>
<td>0.699</td>
<td>0.311</td>
</tr>
<tr>
<td>Fe</td>
<td>13.9</td>
<td>6.07</td>
</tr>
<tr>
<td>Mn</td>
<td>3.09</td>
<td>1.28</td>
</tr>
<tr>
<td>Average</td>
<td>---</td>
<td>---</td>
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</table>

3.3 Radionuclide Composition

Based on measurements and analytical detection limits, twenty-seven radionuclides were identified as reportable for DWPF SB7b (MB9) as specified by the Waste Acceptance Product Specification (WAPS).\(^{13,23,g,h}\) Selected radionuclides were directly measured in quadruplicate either by gamma counting, beta counting, alpha spectroscopy or ICP-MS. Table 5-3 through Table 5-5 in Appendix A provide the actual measured radiological chemical data and ICP-MS data, respectively.

Table 3-4 lists the average concentrations of these radionuclides.\(^{i}\) Some of the analytes were below the detection limit of the instrument and are noted by a result preceded with a “<.” The content of each radionuclide was also calculated from measured values of the Tank 40 dried SB7b sludge and the average WDF value shown in Table 3-3.\(^{22}\) It should be noted that the measured SB7b glass concentrations include the Cs-137 additions from the Cs-laden strip effluent stream that was processed at DWPF, whereas the SB7b WAPS concentrations and the calculated concentrations for the SB7b glass are based on the sludge stream only.

3.4 Noble Metals

The average measured concentrations of the noble metals based on quadruplicate measurements of SB7b PS glass sample are list in Table 3-5. Table 5-4 in Appendix A provides the actual measured ICP-MS data. The calculated noble metal concentration in the glass is determined from

\(^{a}\) Th-229 was identified as reportable for SB7b; however, there is no direct method for measuring its concentration, so its value will not be presented in this report. Based on the calculated values in SRNL-STI-2012-00294, Th-229 becomes reportable in the year 3115, which is of no practical significance to this study. Total ThO\(_2\) is reported in Table 3-2.

\(^{b}\) In addition to the twenty-seven radionuclides identified above, U-235 and U-236 are also reportable per the requirements of the WAPS.

\(^{i}\) Th-232 was also added to the list as it was measured at greater than 0.2 wt% by ICP-MS.
the concentration in the Tank 40 dried sludge\textsuperscript{22} and the average WDF value (Table 3-3).

In addition to ICP-MS, the SB7b PS glass sample was also analyzed with SEM-EDS for noble metal inclusions. Examination of the glass with EDS indicated the presence of both Ru and Rh, which corresponds to the results of the ICP-MS noble metals analysis in Table 3-5.\textsuperscript{1} Noble metal inclusions have been observed in previous pour stream samples from SB4 through SB7a.\textsuperscript{20,24,25}

**Table 3-4. Reportable Radionuclide Concentration in the SB7b PS glass sample Glass**

<table>
<thead>
<tr>
<th>Radionuclide</th>
<th>SB7b WAPS</th>
<th>SB7b Glass Calculated</th>
<th>SB7b Glass Measured</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(Ci/kg)</td>
<td>wt%</td>
<td>(Ci/kg)</td>
</tr>
<tr>
<td>Cl-36</td>
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<td>&lt;1.2E-05</td>
<td>&lt;5.2E-06</td>
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<td>Zr-93</td>
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<td>8.0E-03</td>
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<td>Cd-113m</td>
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<td>Sn-121m</td>
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<td>&lt;1.1E-03</td>
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<td>Sm-151</td>
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<td>&lt;1.0E-04</td>
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<td>2.2E-06</td>
<td>9.3E-07</td>
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### Alpha Spectroscopy:
- Pu-238

### Beta Counting:
- Sr-90, Tc-99 and Pu-241

### Gamma Counting:
- Cs-137 and Am-241
- Zr-93, Th-232, U-233, U-234, U-235,
- U-236, U-238, Np-237, Pu-239, Pu-240 and Pu-242

\textsuperscript{1} More details can be found in SRNL electronic laboratory notebook L3293-00022-10.
Table 3-5. Noble Metal Concentration in the SB7b PS glass sample Glass

<table>
<thead>
<tr>
<th>Noble Metal</th>
<th>SB7b WAPS</th>
<th>SB7b Glass Calculated</th>
<th>SB7b Glass Measured</th>
<th>%RSD</th>
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</thead>
<tbody>
<tr>
<td>Ag</td>
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<td>0.005</td>
<td>0.01</td>
<td>15.1</td>
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<tr>
<td>Pd</td>
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<td>0.001</td>
<td>0.0007</td>
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<tr>
<td>Rh</td>
<td>0.02</td>
<td>0.009</td>
<td>0.007</td>
<td>6.6</td>
</tr>
<tr>
<td>Ru</td>
<td>0.1</td>
<td>0.04</td>
<td>0.02</td>
<td>1.7</td>
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</table>

3.5 PCT

The average normalized release values for ARM\textsuperscript{16}, EA (published\textsuperscript{26} and measured) and SB7b PS glass sample are shown in Table 3-6. No water loss issues were observed over the course of the test. Table 5-6 through Table 5-8\textsuperscript{8} in Appendix A provides the as-received elemental leachate concentrations for the solutions samples generated by the PCTs. The normalized release values of the pour stream glass for B, Li, Na and Si are very acceptable with respect to the EA glass benchmark values provided in Table 3-6 and meet the acceptance criteria as defined in the WAPS.\textsuperscript{1,27}

3.6 Density

The average density of the SB7b PS glass sample glass was determined to be 2.70 g/cm\textsuperscript{3}. Data from the density measurements are shown in Table 5-9 in Appendix A.

3.7 REDOX

The average value of Fe\textsuperscript{2+}/\Sigma Fe and Fe\textsuperscript{2+}/Fe\textsuperscript{3+} for the SB7b pour stream sample were 0.07 and 0.08, respectively. The EA glass results are consistent with the target values (0.22-0.23±0.01 for Fe\textsuperscript{2+}/Fe\textsuperscript{3+}), indicating that the measurements were in control.\textsuperscript{26} Complete sets of data for each of the replicates and EA samples included with the individual sets are shown in Table 5-10 in Appendix A.

Table 3-6. Normalized PCT Results for the SB7b PS glass sample Glass (g/L)

<table>
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<tr>
<th>Glass ID</th>
<th>NL B</th>
<th>NL Li</th>
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<th>NL U</th>
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<td>0.5</td>
<td>0.3</td>
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<tr>
<td>St. Dev.</td>
<td>0.006</td>
<td>0.003</td>
<td>0.004</td>
<td>0.001</td>
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<tr>
<td>%RSD</td>
<td>1.5</td>
<td>0.6</td>
<td>0.9</td>
<td>0.6</td>
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<tr>
<td>EA - Measured</td>
<td>16.6</td>
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<td>4.0</td>
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<td>St. Dev.</td>
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<td>0.1</td>
<td>0.1</td>
<td>0.02</td>
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<tr>
<td>%RSD</td>
<td>0.7</td>
<td>0.7</td>
<td>0.7</td>
<td>0.4</td>
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<tr>
<td>EA - Published</td>
<td>16.7</td>
<td>9.6</td>
<td>13.3</td>
<td>3.9</td>
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<tr>
<td>St. Dev.</td>
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<td>0.7</td>
<td>0.9</td>
<td>0.4</td>
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<tr>
<td>%RSD</td>
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<td>7</td>
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<td>SB7b PS</td>
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<td>St. Dev.</td>
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<td>0.005</td>
<td>0.007</td>
<td>0.003</td>
<td>0.004</td>
</tr>
<tr>
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<td>0.6</td>
<td>0.7</td>
<td>0.6</td>
<td>0.9</td>
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</table>

\textsuperscript{k} As-measured radionuclide data are shown in Tables 5-8 and 5-9.

\textsuperscript{l} The normalized release of boron, sodium, and lithium from the waste glass must be at least two standard deviations better than the EA glass.
4.0 Conclusions

- The sum of oxides for the SB7b pour stream glass is within the PCCS limits (95-105 wt%).

- The average calculated WDF for SB7b is 2.3. In general, the measured radionuclide content of the official SB7b pour stream glass is in good agreement with the calculated values from the Tank 40 dried sludge results from the SB7b WAPS sample.

- As in previous pour stream samples, ruthenium and rhodium inclusions were detected by SEM-EDS in the official SB7b pour stream sample.

- The PCT results indicate that the official SB7b pour stream glass meets the waste acceptance criteria for durability with a normalized boron release of 0.8 g/L, which is an order of magnitude less than the EA glass.

- The measured density of the SB7b pour stream glass was 2.70 g/cm³.

- The Fe²⁺/∑Fe ratio of the SB7b pour stream sample was 0.07.

5.0 References


17. “Glass Density Using the Mettler AT400 (or Equivalent) Balance,” Savannah River National Laboratory, Aiken, SC, ITS-0057, Latest Revision.
18. “Determining Fe²⁺/Fe³⁺ and Fe²⁺/Fe(Total) Using UV VIS Spectrometer,” Savannah River National Laboratory, Aiken, SC, ITS-0042, Latest Revision.


Appendix A. Supplemental Data Tables
Table 5-1. Measured Elemental Concentrations (µg/g) for Glasses (AR)

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<tr>
<th>Glass ID</th>
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<td>300305018</td>
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<td>3</td>
</tr>
<tr>
<td>Al</td>
<td>23600</td>
<td>23300</td>
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</tr>
<tr>
<td>B</td>
<td>25700</td>
<td>25500</td>
<td>23500</td>
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<td>Ba</td>
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*Axial
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Table 5-5 cont. As-Measured Concentrations of m/z (µg/g) via ICP-MS (AR)

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Table 5-7 cont. As-Measured Concentrations (µg/L) for the PCT Solutions via ICP-MS

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Table 5-7 cont. As-Measured Concentrations (µg/L) for the PCT Solutions via ICP-MS

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Table 5-8. As-Measured Radionuclide Concentrations (dpm/mL) via Gamma and Beta Counting

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</tr>
<tr>
<td>m3 (g)</td>
<td>58.386</td>
<td>58.46</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>2.50</td>
<td>2.48</td>
</tr>
<tr>
<td>%RSD</td>
<td>0.7</td>
<td></td>
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</tbody>
</table>

Table 5-10. SB7b Pour Stream Glass REDOX Data

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Trial</th>
<th>Fe²⁺</th>
<th>∑Fe</th>
<th>Fe³⁺</th>
<th>Fe²⁺/∑Fe</th>
<th>Fe²⁺/Fe³⁺</th>
</tr>
</thead>
<tbody>
<tr>
<td>EA</td>
<td>---</td>
<td>0.0938</td>
<td>0.5482</td>
<td>0.4544</td>
<td>0.17</td>
<td>0.21</td>
</tr>
<tr>
<td>SB7b PS</td>
<td>1</td>
<td>0.0407</td>
<td>0.5761</td>
<td>0.5354</td>
<td>0.07</td>
<td>0.08</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.0358</td>
<td>0.503</td>
<td>0.4672</td>
<td>0.07</td>
<td>0.08</td>
</tr>
</tbody>
</table>
Distribution:

S. L. Marra, 773-A
T. B. Brown, 773-A
D. R. Click, 999-W
S. D. Fink, 773-A
C. C. Herman, 773-A
E. N. Hoffman, 999-W
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
Records Administration (EDWS)
J. M. Bricker, 704-30S
T. L. Fellinger, 766-H
E. J. Freed, 704-S
J. M. Gillam, 766-H
B. A. Hamm, 766-H
E. W. Holtzscheiter, 766-H
J. F. Iaukea, 704-27S
D. K. Peeler, 999-W
J. W. Ray, 704-S
H. B. Shah, 766-H
D. C. Sherburne, 704-S
M. E. Stone, 999-W
F. C. Johnson, 999-W
J. M. Pareizs, 773-A
C. L. Crawford, 773-42A
J. W. Amoroso, 999-W
C. J. Bannochie, 773-42A
T. B. Edwards, 999-W
H. H. Elder, 704-27S
C. M. Jantzen, 773-A
M. T. Keefer, 704-56H
D. P. Lambert, 999-W
J. D. Newell, 999-W
J. R. Zamecnik, 999-W
P. R. Jackson, DOE-SR, 703-46A
K. H. Subramanian, 241-156H
M. A. Broome, 704-29S
R. N. Hinds, 704-S
R. R. Arrington, 704-71S
W. A. Drown, 773-41A