Glass Results For the Can In-Canister Demonstration Canister S00144 (U)

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INTRODUCTION AND SUMMARY

During the Defense Waste Processing Facility (DWPF) Proficiency Runs, two test canisters containing small cans of surrogate plutonium glass were filled with glass. One of the test canisters contained a rack with eight small cans and one contained a rack with twenty small cans. The canister with eight small cans was discussed in another report.[1] The canister with twenty small cans was sectioned by the Savannah River Technology Center (SRTC) and samples of the glass were removed for analysis. This report is a summary of the results of the glass testing of the DWPF canister (S00144) which contained a rack with twenty small cans of surrogate plutonium glass. Significant results of the demonstration with the DWPF canister containing the twenty cans of surrogate plutonium glass are:

- While molten, the DWPF glass filled all of the areas around the small cans and the rack. No significant voiding, other than the typical shrinkage cavities near the glass surface, was found.
- There was no significant difference in the chemical composition of the glass removed from the canister centerline compared to the glass taken near the rack or small cans.
- The Product Consistency Test (PCT) results for all of the glass samples were at least two standard deviations below the mean PCT results of the Environmental Assessment (EA) glass.
- The DWPF glass contained less than 4 vol% crystals. No crystals were detected in the surrogate plutonium glass.

GLASS SAMPLING

Canister S00144 was sectioned using a bandsaw. Three cross sectional cuts were made, dividing the canister into four sections. The first cut was made 21 inches from the bottom of the shim plate. The other two cuts were made at 37 inches and 90 inches from the bottom. Samples of the DWPF glass were removed from each of the three levels. In addition, at both the 21 inch and the 37 inch levels, samples of the surrogate plutonium glass were removed from one of the other small cans. These samples were taken and analyzed as directed in SRT-CIC-96-0014, dated April 10, 1996. The analytical results are provided below.

During sampling, it was noted that brown streaks, similar to those found during the DWPF Waste Qualification Campaigns (WSRC-TR-95-0239, Rev. 0, May, 1995), were present around the rack and the small cans. No other discolorations were found in the glass. It was also noted that the DWPF glass filled all the space around the rack and the small cans.

Table 1 provides the names, heights, and location of the samples. Four additional samples (not listed in Table 1) were removed and analyzed for crystalline content. The additional samples include: a sample from another small can in the first row, a sample from another small can in the second row, a sample from the 21 inch level adjacent to the rack, and a sample from the 37 inch level adjacent to a small can.

Table 1
Sample Locations

Sample Name	Sample Height	Sample Location
420	20.8"	Near the wall of the canister
421	21.0"	Adjacent to a small can
422	21.0"	Inside small can in 1st row
423	21.0"	Centerline of canister
424	36.5"	Centerline of canister
425	37.0"	Near the wall of the canister
426	37.0"	Adjacent to the rack for the cans
427	37.0"	Inside small can in 2nd row
428	85.8"	Centerline of canister
429	89.5"	Near the wall of the canister

CHEMICAL COMPOSITION

Ten glass samples were analyzed to determine the chemical composition. Eight of the samples were the DWPF glass from the large canister and two of the samples were the surrogate plutonium glass from the small canisters. In addition, a DWPF glass standard and a surrogate plutonium glass standard were analyzed concurrently with the samples. Each sample was dissolved by two separate dissolution methods according to approved procedures.

The dissolutions were analyzed by Atomic Absorption (AA) spectrometry and Inductively Coupled Plasma Emission Spectroscopy (ICP-ES). The results for the DWPF glass samples are shown in Table 2, along with the measured Fe^{+2}/Fe^{+3} ratio. The results for the surrogate plutonium glasses are provided in Table 3. Results of the DWPF glass standard and the surrogate plutonium glass standard submitted with the samples indicated that the dissolutions were complete and the analyses were performed and calibrated correctly.

Table 2
Chemical Composition (wt% oxides)
of DWPF Glass from Canister S00144

Oxide	420	421	423	424	425	426	428	429
Al2O3	4.95	4.91	4.92	4.87	4.85	4.50	4.61	4.58
B2O3	7.30	7.27	7.26	7.23	7.28	6.81	7.38	7.51
CaO	0.91	0.92	0.94	0.93	0.92	0.87	0.90	0.91
Cr2O3	0.37	0.32	0.35	0.31	0.33	0.29	0.37	0.32
CuO	0.46	0.46	0.47	0.46	0.47	0.43	0.45	0.44
Fe2O3	10.85	10.76	10.89	10.74	10.85	10.09	10.89	10.88
K2O	3.31	2.74	2.75	2.75	2.81	2.87	2.82	2.73
Li2O	3.97	4.01	4.00	4.01	3.99	3.74	3.95	3.99
MgO	1.49	1.50	1.51	1.52	1.50	1.40	1.49	1.52
MnO	2.30	2.31	2.32	2.32	2.30	2.15	2.25	2.25
Na2O	10.01	10.09	10.19	10.11	10.15	9.56	9.92	10.04
NiO	0.93	0.81	0.72	0.74	0.85	0.79	0.72	0.83
P2O5	0.12	0.10	0.13	0.10	0.14	0.14	0.17	0.17
PbO	0.11	0.12	0.14	0.11	0.13	0.15	0.15	0.12
SiO2	52.25	51.22	52.22	52.26	52.05	48.88	50.65	52.47
TiO2	0.35	0.32	0.32	0.32	0.32	0.29	0.30	0.30
ZrO2	1.04	1.01	1.01	0.99	1.00	0.93	0.96	0.94
Total	100.72	98.87	100.12	99.79	99.93	93.89	97.97	100.01
Fe2/Fe3	0.029	0.028	0.113	0.081	0.047	0.047	0.007	0.002

Samples 423, 424 and 428 were removed from the canister centerline. Comparing the results of these three samples to the results of the samples from the same level shows no significant differences in the chemical composition. Therefore, the composition does not appear to be affected by the rack or the small cans.

Table 3
Chemical Composition (wt% oxides)
of Surrogate Plutonium Glass

Oxide	422	427	Corning
Al2O3	14.66	15.48	17.3
B2O3	2.88	2.97	3.04
BaO	3.50	3.45	3.54
Ce2O3	5.98	5.37	6.79
Cr2O3	0.12	0.12	
Fe2O3	0.57	0.60	
K2O	0.03	0.03	
La2O3	13.89	13.30	14.8
Na2O	0.14	0.17	
Nd2O3	15.34	14.59	15.6
P2O5	0.05	0.11	
PbO	7.47	6.62	9.06
SiO2	29.12	29.09	28.9
ThO2	0.04	0.04	
ZrO2	0.55	1.20	
Total	94.35	93.13	99.03

PRODUCT CONSISTENCY TEST (PCT)

The PCT[2] was performed on the ten glass samples that were analyzed for chemical composition. Each sample was subjected to the PCT in triplicate and the test included the appropriate blanks and standards. The results for the standards and blanks indicated that the test was acceptable. The PCT releases for several elements were measured. These values were used along with the composition of the glass to calculate the average normalized release for boron, sodium, lithium, and silicon. Silicon is not required by the Waste Acceptance Product Specifications[3] but is provided for additional information since it is a major component of the DWPF glass. The leachate pH was measured as part of the PCT protocol and provides a secondary indication of glass durability.

The normalized elemental releases reported in Table 4 indicate that the DWPF glass samples met the acceptance criterion which states that the glass produced must be at least two standard deviations better than the Environmental Assessment (EA) glass.[4]

Table 4
Normalized PCT Results (g/L)

Sample	B	Si	Na	Li	pH
420	0.63	0.38	0.63	0.69	10.4
421	0.62	0.38	0.61	0.68	10.4
423	0.63	0.38	0.63	0.72	10.4
424	0.63	0.38	0.63	0.70	10.4
425	0.65	0.39	0.64	0.71	10.4
426	0.73	0.40	0.68	0.80	10.5
428	0.63	0.39	0.65	0.72	10.4
429	0.63	0.37	0.64	0.71	10.4
EA[5]	16.70	3.92	13.35	9.57	11.9

The normalized elemental releases for the surrogate plutonium glass samples are given in Table 5. Sodium and lithium were not included in Table 5 because they were not in the product specification. Aluminum was included since the surrogate plutonium glasses contained relatively large quantities of aluminum. The results for boron and silicon are lower than the EA glass. However, the EA glass was designed as a benchmark for high-level waste glass and is not an appropriate comparison for the surrogate plutonium glass due to the difference in elemental composition. The durabilities of the surrogate plutonium glasses can be compared to the measured durabilities of radioactive plutonium glasses.[6] The radioactive plutonium glasses are even more durable than the surrogate plutonium glasses.

Table 5
Normalized PCT Results (g/L)

Sample	B	Si	Al	pH
422	0.03	0.02	0.01	8.7
427	0.02	0.02	0.00	8.0

CRYSTALLINE CONTENT

X-Ray Diffraction (XRD) was performed on all the samples to determine the crystalline content. Scanning Electron Microscopy (SEM) was also performed and confirmed the XRD results. No crystals were detected by XRD or SEM in any of the surrogate plutonium glass samples. Some of the DWPF glass samples did contain crystals - up to 3.7 vol% crystals were detected by XRD. The crystals detected were trevorite (nickel iron oxide) and acmite (sodium iron silicate). The brown streaks found in the DWPF glass around the rack and small cans contained less than 3.7 vol% crystals. Brown glass was also found in the DWPF glass during the Waste Qualification Campaigns. (WSRC-TR-95-0239, Rev. 0, May, 1995)

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