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Initial Results from the Canistered Waste Forms Produced during the First Campaign of the DWPF Startup Test Program

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ABSTRACT

As part of the Defense Waste Processing Facility (DWPF) Startup Test Program, approximately 90 canisters will be filled with glass containing simulated radioactive waste during five separate campaigns. The first campaign is a facility acceptance test to demonstrate the operability of the facility and to collect intitial data on the glass and the canistered waste forms. During the next four campaigns (the waste qualification campaigns) data will be obtained which will be used to demonstrate that the DWPF product meets DOE's Waste Acceptance Product Specifications (WAPS).

Currently 12 of the 16 canisters have been filled with glass during the first campaign (FA-13). This paper describes the tests that have been carried out on these 12 glass-filled canisters and presents the data with reference to the acceptance criteria of the WAPS. These tests include measurement of canister dimensions prior to and after glass filling, dew point, composition, and pressure of the gas within the free volume of the canister, fill height, free volume, weight, leak rates of welds and temporary seals, and weld parameters.

INTRODUCTION

The Defense Waste Processing Facility (DWPF) will vitrifiy high-level nuclear waste currently stored in underground tanks at Savannah River Site. After processing, the nuclear waste and glass frit will be mixed and then fed into the DWPF melter. The molten radioactive waste glass will be captured by stainless steel canisters as it pours from the melter pour spout. After glass filling, the canisters will be sealed by insertion of a temporary plug in the nozzle. The temperature of the nozzle will be at least 167°C higher than the temperature of the plug at time of insertion. The nozzle (with inserted sleeve) will then contract during cooling to form a water-tight seal with the plug. The leak rate of the temporary seal of the canistered waste form will then be measured in the Melt Cell. When the leak rate is acceptable, the canistered waste form will be moved to the Canister Decontamination Cell where it is decontaminated using an aqueous slurry of glass frit. Smears will then be taken to ensure that the amount of smearable radioactive materials present on the outside surface of the canistered waste form is within specification.

After this is confirmed, the canistered waste form will be transferred to the Weld Cell where the plug and sleeve are pushed down into the nozzle. A final weld plug will be inserted into the mouth of the nozzle, and a final weld will be made by simultaneous application of force and electric current for a short time duration (upset resistance welding). Smear tests will again be taken at this time. The canistered waste form will then be transfered to the Glass Waste Storage Building.

This report summarizes the results of testing of the canistered waste forms produced during the first campaign of the DWPF Startup Test. Durability results of the waste glass from this campaign will be reported separately.

CANISTER DIMENSIONS BEFORE AND AFTER GLASS FILLING

The vendor procurement specifications for length and diameter are:

Height:	117.94 to 118.06 inches or 299.57 to 299.87 cm
Diameter:	23.88 to 24.12 inches or 60.66 to 61.265 cm

The diameter (measured at 36 different locations) and the length of canister S00004 were measured prior to glass filling and the results demonstrated that the length was within the specification, but several diameter values were lower than the specification by a maximum of 0.02 cm. Perpendicularities for this canister were also measured

The ranges and the maximum changes in dimensions for canister after glass filling (canistered waste form) were:

Canistered waste form length:	117.994 to 118.001 inches or 299.705 to 299.723 cm
Maximum change over empty canister:	**0.007 inch or 0.018 cm**
Canistered waste form diameter:	23.896 to 23.957 inches or 60.696 to 60.851 cm
Maximum change over empty canister:	**0.039 inch or 0.099 cm**
Canistered waste form perpendicularity:	-0.114 to +0.093 inch or -0.290 to +0.236 cm
Maximum change over empty canister:	**0.039 inch or 0.099 cm**

It is interesting to note that after glass filling, all of the measured diameters were within specification. This implies that the canister became more round after filling, which is consistent with previous findings by SRTC. The canister became slightly less perpendicular with filling.

The Waste Acceptance Product Specifications (WAPS)[1] requires that the unfilled canister shall have an outer diameter of 61.0 cm (+1.5 cm, - 1.0 cm) and an overall length, after accounting for the closure method, of 3.000 m (+ 0.005 m, - 0.020 m). The maximum plug height using upset resistance welding for final closure is 0.092 inch or 0.234 cm. The unfilled canister S00004 readily met these requirements.

The WAPS also requires that the canister after glass filling and final closure shall be such that it will stand upright without support on a flat horizontal surface and will fit completely without forcing when lowered vertically into a right-circular, cylindrical cavity, 64.0 cm in diameter and 3.01 m in length. This specification was also met. In fact, the changes in these canister dimensions after being filled with glass are insignificant and provide assurance that canisters procured to the current DWPF specifications will readily meet the WAPS.

ICC LEAK RATES BEFORE AND AFTER DECONTAMINATION

After filling a canister with glass, a temporary heat shrink seal is made between an Inner Canister Closure (ICC) plug and the sleeve in the nozzle. This temporary seal is intended to prevent water from entering the canister during the decontamination process. This ICC seal must be leak tight to less than 2×10^{-4} atm·cc/sec for helium, a limit demonstrated to preclude inleakage of water into the canister. Water exclusion from the canister is important in order to prevent internal corrosion of the stainless steel canister. Decontamination is then carried out by frit blasting the canister surface with an aqueous slurry of glass frit.

The helium leak rate of the normal ICC seal on canister S00145 was measured three times before and after decontamination. The results were:

Before:	8×10^{-6} atm·cc/sec	After:	9.3×10^{-5} atm·cc/sec
	6×10^{-7} atm·cc/sec		9.0×10^{-5} atm·cc/sec
	-3×10^{-7} atm·cc/sec		8.6×10^{-5} atm·cc/sec

The before decontamination leak rates were essentially at the limit of sensitivity of the equipment.

The measured leak rate of the ICC plug of canister S00114 was 7×10^{-5} atm·cc/sec. As planned, the nozzle was then heated to drop the ICC plug and sleeve into the canister and a new seal was made using a repair plug. The leak rate of this repair ICC seal was measured both before and after decontamination.

Before: 1.3×10^{-4} atm·cc/sec After: 1.2×10^{-4} atm·cc/sec

The results for canisters S00114 and S00145 demonstrated that seals with leakrates less than 2×10^{-4} atm·cc/sec for helium can be made. In fact all 12 canisters filled during this campaign met this requirment (Table I). Furthermore, it was demonstrated that aqueous frit blasting did not significantly change the leak rate of the seal.

TABLE I
Leak Rates of ICC Plug and Repair Plug Seals on Canisters Produced during FA-13

CANISTER #	LEAK RATE ICC PLUG	LEAK RATE ICC REPAIR PLUG
S00111	7×10^{-5}	N/A
S00122	2×10^{-4}	N/A
S00116	2×10^{-5}	N/A
S00010	2×10^{-5}	N/A
S00121	5×10^{-5}	N/A
S00145	8×10^{-6}	N/A
S00123	6×10^{-5}	3.0×10^{-5}
S00112	1×10^{-5}	N/A
S00146	2×10^{-5}	N/A
S00114	7×10^{-5}	1.3×10^{-4}
S00004	8×10^{-6}	N/A
X00101	5×10^{-6}	N/A

CANISTER WELD PARAMETERS

The final weld is made by forcing the weld cap into the nozzle opening under a simultaneous application of DC current. The parameters of force, current, and time of application of current used to make the final upset resistance weld of nine of the canisters produced during FA-13 are presented in Table II.

TABLE II
Force, Current and Time Values for the Final Welds Made during FA-13

CANISTER #	FORCE (pounds)	CURRENT (amps)	TIME (seconds)
S00111	81,182	244,000	1.58
S00122	77,961	249,000	1.58
S00116	75,179	257,000	1.58
S00010	77,375	253,000	1.58
S00121	75,619	247,000	1.58
S00145	-	-	-
S00123	-	-	-
S00112	75,765	251,000	1.58
S00146	77,082	253,125	1.58
S00114	76,497	253,000	1.58
S00004	75,619	254,000	1.58
X00101	-	-	-

The nominal values to be used for making final welds are a force of 80,000 pounds, a current of 248,000 amps, and a time of 1.58 seconds.

The parametric study[2] for weld performance determined acceptable windows for force and current of 90,000 ± 15,000 lbs and 248,000 ± 22,000 amps, respectively at 1.58 seconds. Hence all welds were made within the parametric window for acceptable welds.

WEIGHTS OF CANISTERED WASTE FORMS

The weights of the empty canisters were measured in DWPF prior to glass filling. After being filled with glass, the canister weight as measured on the pour turntable (PTT) with the bellows raised was also recorded. After arrival at TNX, all canistered waste forms were weighed using a calibrated overhead crane scale. The weights of the canisters were measured at TNX to determine if it would be appropriate to use the DWPF weight system to approximate the fill height. The net glass weight for each canister sent to TNX is given in Table III, along with the corresponding weight measured at DWPF.

TABLE III
Weights of the Glass within the Canistered Waste Forms Produced during FA-13

CANISTER #	DWPF WEIGHT (pounds)	TNX WEIGHT (pounds)	DELTA
S00111	3845	3749	96
S00122	3801	N/A	N/A
S00116	3755	N/A	N/A
S00010	3761	N/A	N/A
S00121	3694	N/A	N/A
S00145	3661	3618	43
S00123	3436	3390	54
S00112	3714	3633	81
S00146	3691	3606	85
S00114	3739	3707	32
S00004	3778	3732	46
X00101	3658	N/A	N/A

At DWPF the weight of the glass was obtained by subtracting the weight of the empty canister from the weight of the glass filled canister as measured on the pour turntable with the bellows raised. At TNX, the weight was obtained using the crane scale measured weight minus the combined weight of the empty canister plus the weights of the canister closure plugs. The weights of the closure plugs are as follows:

Weld plug	4 pounds
ICC plug	10 pounds
ICC repair plug (if used)	12 pounds

In all cases the weights measured at DWPF were greater than those obtained at TNX. These differences, listed as deltas in Table III, range from 32 to 96 pounds. These results indicate that the pour turntable provided a fairly accurate estimate of the canistered waste form weight with the bellows raised.

The differences between the weights of the filled canisters with the bellows lowered and raised are provided in Table IV. The delta increased with filling such that the average additional weight of the bellows was 278 pounds after filling, an increase of 60 pounds over the average difference of the empty canisters. Hence, the force and weight observed were not constant as the canister was filled with glass. The difference is manageable since 60 pounds corresponds to only 1.5 inches of glass fill height. However, significant scatter existed in the delta from canister to canister. The maximum differential was 120 pounds which corresponded to 3 inches of glass fill height. These results suggest that weight alone as an indicator of fill height has an uncertainty on the order of 2 inches.

TABLE IV
Weights on the Pour Turntable of Glass-Filled Canisters with and without the Bellows in Place

CANISTER #	PTT WEIGHT BELLOWS (pounds)	PTT WEIGHT W/O BELLOWS (pounds)	DELTA (pounds)
S00111	5073	4872	201
S00122	5184	4891	293
S00116	5030	4788	242
S00010	5118	4786	332
S00121	5066	4788	278
S00145	5060	4788	272
S00123	4757	4529	228
S00112	5098	4757	341
S00146	5040	4806	234
S00114	5058	4761	297
S00004	5085	4812	273
X00101	5074	4732	342

FREE VOLUME AND FILL HEIGHTS

The WAPS requires that the canister must be filled with glass to a height equivalent to at least 80% by volume of the empty canister. For a DWPF canister, 80% corresponds to a fill height of 86 inches.

The glass fill height was measured on S00146 and determined to be 90.2 inches. This corresponds to a canister that has been filled to approximately 84%.

The free volume of canister S00146 was determined to be 119.6 liters as described previously[3]. The total volume of an empty canister has been estimated to be ~735 liters. Using this value, the glass occupies 83.7% by volume of this canister.

Both free volume and fill height measurements indicate that the acceptance requirement was met.

For a Westinghouse built canister which uses stainless steel plate for the cylinder, the typical wall thickness is 0.43 inches and the diameter averages ~ 23.95 inches. Canister S00146 was representative of these dimensions. Using these dimensions, the volume occupied by a 1 inch thick section of the canister was calculated to be 6.86 liters. The measured density of the glass from canister S00146 was found to be 2.695 g/cc.[4] Therefore, each one inch section of the canister, with no voids, should contain 40.7 pounds of glass.

The DWPF canister has a reverse dished bottom which reduces the volume of glass a canister can hold. In addition, the bottom head has a thickness of 0.5 inches. Since the fill height is measured from the bottom of the canister, the amount of volume excluded by the dished bottom and wall thickness can be estimated to accurately determine the glass volume. The excluded volume for the bottom skim cut was calculated to be approximately 4.1 liters, while the half inch wall thickness causes an additional 3.43 liters to be excluded. Therefore, the total volume excluded is 7.53 liters. This corresponds to a 1.1 inch slice of the canister.

For canister S00146, the fill height was measured at 90.2 inches. Therefore, the amount of glass present should be:

$$(90.2 - 1.1) \bullet 40.7 \text{ pounds/inch} = 3626 \text{ pounds.}$$

The measured weight of the glass at TNX was 3606 pounds. This corresponds well with the calculated value, but it does not take into account two factors. First, a void (shrinkage cavity) was detected in this canister several inches below the top surface of the glass. This void was not spherical, but was approximated to be a sphere of 7 inches in diameter. Therefore, the approximate calculated volume would be 3 liters. This corresponds to ~17 pounds of glass which must be subtracted from TNX measured weight (3606 pounds) and brings the calculated and measured values even closer together. The other factor was the fill height itself, since the top surface was concave. As the canister wall was approached from the center axis, the glass fill height increased slightly. It may have decreased again at the wall, but it was difficult to determine since numerous pieces of glass had broken away from the top surface. This latter effect, although small, has not been accounted for in this analysis.

The fill heights of a total of five canisters were measured at TNX. Measurements were performed using standard measuring devices after the nozzles had been removed from the canisters. The distance to the glass surface was measured four times at 90 degree intervals around the base of the canister nozzle, therefore all measurements were towards the center of the canister (in the concave region). These four measurements were then averaged and compared to the fill heights calculated from the canister weights using the 40.7 pounds/inch factor, as above. The measured and calculated fill heights are shown in Table V.

TABLE V
Measured Fill Heights

CANISTER #	MEASURED FILL HEIGHT (inches)	CALCULATED FILL HEIGHT (inches)	DIFFERENCE (meas.-calc.)
S00112	91.5	90.4	1.1
S00114	91.8	92.2	-0.4
S00123	85.1	84.4	0.7
S00145	90.9	90.0	0.9
S00146	90.2	89.7	0.5

The maximum difference between the measured and the calculated fill heights was 1.1 inches which along with the data on free volume, indicates that no significant glass porosity or voids existed in the canisters.

DEW POINT OF THE GAS WITHIN THE FREE VOLUME

The dew point of the gas within the free volume space of canister S00146 was measured as previously described[3]. The dew points obtained with the two hygrometers (calibrated the hygrometers within ± 2°C) were:

Hygrometer #1: 5.1°C

Hygrometer #2: 2.6°C

Average dew point: 3.9°C

The dew point was also monitored as a function of internal gas pressure. This was accomplished by slowly pumping the system and monitoring the dew point as a function of gas pressure. The dew point (hygrometer # 1) fell from 4.9°C at 700 Torr to 3.0°C at 600 Torr to 0.9°C at 500 Torr. The partial pressure (vapor pressure) of water in gas is a linear function of the overall gas pressure. Hence the dew point should fall as the pressure is reduced. The measured dew points as a function of overall pressure followed this expected dependence.

The observed dew point within the canister is evidently dependent on the dew point of the air within the melt cell. The temperature and relative humidity for the melt cell when each canister was sealed was recorded and is shown in Table VI.

TABLE VI
Temperatures and Relative Humidities (RH) in the Melt Cell at the Time of Sealing for Each Canister

CANISTER #	TEMPERATURE(°C)	RH (%)	DEW POINT (°C)
S00111	27	65	20
S00122	27	60	19
S00116	27	60	19
S00010	-	-	-
S00121	24	62	16
S00145	29	55	19
S00123	27	58	18
S00112	24	60	16
S00146	24	51	13
S00114	22	51	11
S00004	24	57	15
X00101	27	62	19

Canister S00146 was sealed at a dew point of 13°C in the melt cell. If the dew point in the melt cell at the time of sealing was higher, then the dew point in the canister free volume space is expected to be higher. The dew point in the melt cell at the time of sealing was higher than 13°C for all but one of the other canisters. This implies that the other canisters have dew points within the free volume greater than 0°C. It is important to control the dew point of the gas within the canister free volume. If the temperature of the canistered waste forms within the Glass Waste Storage Facility at SRS reaches 0°C, then dew will form on the inside walls of the canister for those canisters having dew points greater than 0°C. The concern here is that liquid water can lead to corrosion of the canisters.

The leak rate from the ICC seal of S00146 was 2×10^{-5} atm·cc/sec for helium, a rate which is an order of magnitude less than required for this seal. (See section on ICC Leak Rates before and after Decontamination). Therefore, it has also been demonstrated that no significant inleakage of water occurred during the decontamination process.

INTERNAL PRESSURE OF THE GAS WITHIN THE FREE VOLUME

The internal gas pressure of S00146 was measured as 741 Torr at 31°C. Since the waste acceptance criterion limits the internal gas pressure at a temperature of 25°C, the measured gas pressure of 741 Torr was converted to a pressure at 25°C. This pressure became 726 Torr (0.96 atm or 14.0 psi) at 25°C, which readily meets the acceptance requirement of a pressure less than 1.5 atm or 22 psi at 25°C immediately after filling and sealing.

COMPOSITION OF THE GAS WITHIN THE FREE VOLUME

Mass spectrometric data obtained on the air within the free volume of canister S00146 did not reveal any compounds, other than air, within the sensitivity of the equipment. The acceptance criterion of the WAPS is: No detectable foreign materials present in the free gas. Therefore this acceptance criterion was met.

Scans of the canister free volume gas were obtained using a mass spectrometer from 1 to 200, 1 to 50, 40 to 95, and 90 to 200 mass units. These data were compared to equivalent scans taken from room air, which was considered the standard since the gas within the free volume space of the canistered waste form should contain only air from the melt cell. Consequently, the mass spectrometer was looking for changes between the composition of the gas within the free volume and within the room air. The only difference noted was in the amount of carbon dioxide present. Relative to Argon 40, approximately 25% less carbon dioxide was present in the canister air than in room air.

Scans using a multiplier to increase the sensitivity were also performed from 90 to 200 and 45 to 90 mass units. These regions were where detection of foreign materials would be expected. These data were again compared to the scans obtained with air, and no differences were noted.

Sensitivity of the Extrel Questor II process mass spectrometer was estimated in two ways. The first method used isotopes of argon as a sensitivity indicator with Ar-38 present at ~ 6 ppm. For organics, benzene concentrations of 1, 10, and 100 ppm in air were used. Both techniques demonstrated sensitivities less than 10 ppm, with an estimate of 5 ppm. Hence, detection of foreign materials was bounded by this sensitivity.

CONCLUSIONS

The results of the testing on canistered waste forms produced during the first campaign of Startup Testing at DWPF have demonstrated that the DWPF is ready to continue on with the next four Waste Qualification campaigns.

REFERENCES

1. Office of Environmental Restoration and Waste Management, "Waste Acceptance Product Specifications for Vitrified High-Level Waste Forms", USDOE Document EM-343, Rev. 0, February, 1993.

2. B. J. EBERHARD, J. R. HARBOUR and M. J. PLODINEC, "DWPF Welder Parametric Study (U)", WSRC-TR-94-024, Rev. 0, (January1994).

3. J. R. HARBOUR, T. J. MILLER, and M. J. WHITAKER, "The Determination of Pressure, Dew point, and Composition of the Gas within the Free Volume of Canistered Waste Forms (U)", WSRC-RP-90-1167, (1990).

4. M. K ANDREWS and R. M. HOOKS, "Density of Glass from Canister S00146 (U)", SRT-GTC-94-0047, (September 1994).