COMPENDIUM OF HLW SOLIDIFICATION PROCESS

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ABSTRACT

This is an overview compendium of High-Level Waste (HLW) solidification process. The U. S. Waste Acceptance Process includes vitrification or ceramic processing of the Liquid High-Level Waste (LHLW) stored in underground tanks at four different U. S. sites (i.e., West Valley, Savannah River, Hanford, and Idaho). The compendium consists of highlights of issues, which include waste characterizations, key pre-treatment variables, key glass- and ceramic-making processes, errors involved, and elements of Waste Acceptance Preliminary Specifications (WAPS). The highlighted issues are selected mainly for the future compliance assessment of the integrity and performance of HLW glass and ceramics in a geologic repository with regulatory rules. Even though the inventory and the characteristics of LHLW are not the same at the four sites, the glass-making processes are derived from West Valley or Savannah River; the ceramic process is unique at the Idaho site. Much has yet to be determined about details of the ceramic processes. It needs to be confirmed that errors involved in both pre-treatments and glass- or ceramic-makings do not affect the integrity nor the performance of glasses or ceramics significantly. WAPS elements are nearly the same at the four sites. Nevertheless, it is necessary to relate these elements to the glass integrity and performance, more quantitatively, in compliance with the governing rule.

Legacy/main - xcd

INTRODUCTION

Currently, the United State has four sites storing liquid high-level waste (LHLW) -- West Valley, storing commercial LHLW, Savannah River, Hanford, and Idaho all storing defense LHLW. The LHLW at all these sites is planned to be vitrified or ceramic processed for a geologic disposal. It is intended that this overview compendium summarize (a) key process variables related to glass- or ceramic-makings and (b) elements of Waste Acceptance Preliminary Specifications (WAPS). The highlighted issues are selected mainly for (a) the future compliance assessment of the integrity and performance of HLW glass and ceramics with 10 CFR Part 60 (particularly 10 CFR 60.135) and (b) Hanford Issues. The compilation of WAPS elements is from site-specific literature. The Department of Energy (DOE) is attempting, currently, to unify four site-specific WAPS into one criterion applicable to all four sites, while still allowing the producers flexibility.

	West Valley Demonstration Project (WVDP)	Defense Waste Process Facilities (DWPF)	Hanford Waste Vitrification Plant (HWVP)	Idaho Chemical Processing Plant (ICPP)
waste form	borosilicate glass	borosilicate glass	borosilicate glass	glass ceramics. ceramic base: fluorite, calcium stabilized zirconia, zircon, aluminoborosilicate glass. glass base: 33 wt. % WVDP, DWPF and HWVP glass processes.
inventory (year 1,990) M: Million l: Liter Ci: Curie	1.23 Ml	132 Ml	254 Ml	12 Ml
	27.3 MCi	562 MCi	393 MCi	62 MCi
(year 2,020)	0.21 Ml	78 Ml	244 Ml	14 Ml
	13.8 MCi	694 MCi	214 MCi	217 MCi
cumulative number of canisters (year 2,020)	275	5,282	1,960	7,800

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waste type	·PUREX: 100,000 Kg washed sludge. 1.6 ML washing solution. ·THOREX: 0.03 ML acidic solution. ·Zeolite: 35,000 Kg Cs loaded Zeolite from supernatant decontamination processing.	·sludge: 10 vol. %. ·salt cake: 0.63 ML. ·salt solution (supernatant). ·HM HAW: high aluminum high heat waste. ·HM LAW: high aluminum low heat waste. ·PUREX HAW: PUREX (high iron) high heat waste. ·PUREX LAW: PUREX low heat waste. ·Zeolite resin. ·coal and sand. ·silver salts. ·supernatant salt solutions (primarily sodium salts).	(Double-Shell Tanks): ·NCAW: neutralization of current acid waste. ·NCRW: neutralized cladding removal waste. ·PFP: plutonium finishing plant waste. ·CC: complexant concentration waste. ·Cs and Sr products as halide salt. (Single-Shell Tanks): ·sludges, salt-cakes, interstitial liquid, and miscellaneous. (Overall): ·single-shell tanks: 17 v/o (volume percent) and 38 a/o (activity percent). ·double-shell tanks: 9 v/o and 19 a/o. ·Cs and Sr capsules: 0 v/o and 42 a/o. ·mixed waste: 1 v/o. ·solid TRU w aste: 12 v/o and 0 a/o.	·fluorinel waste from the dissolution of zirconium cladding fuel. ·aluminum nitrate liquid waste (dissolution of aluminum-cladding fuel). ·sodium bearing waste (2nd and 3rd cycle extraction solvent cleanup and decontamination).
basic fuel solvent	Nitric Acid		Nitric Acid	Hydrofluoric-nitric acid
neutron poison	None		None	Cd and B

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salt solution	·PUREX supernatant: sodium salts of 80% original volume decontaminated. (40% nitrates and carbonates, and 50% activity sulfates. mainly Cs salt with minor Sr and Pu.). ·sludge: mixed salts (nitrates, carbonates, hydroxides, 10 vol % of fission products and heavy meats.). ·THOREX: Nitric acid solution of waste nitrate salts.	·95% as liquid and salt cake (Cs-137): sodium nitrate, sodium sulfate, sodium nitrite, sodium aluminate, sodium carbonate and sodium hydroxide. ·5% as sludge (Sr-90 and Pu-239): hydroxides and hydrous oxides of Fe, Mn, Al, and Mg.	·NCAW: sodium, aluminum, nitrate, nitrite, sulfate, hydroxide (Cs-137). ·PFP: sodium, aluminum, potassium, nitrate, nitrite, hydroxide. ·cc: sodium, aluminum, iron, nitrate, carbonate, hydroxide (Cs-137, Sr-90, activities).	·Aluminum Nitrate Zirconium Fluoride Sodium Bearing Waste. ·acidic: 5 mole nitric acid.
method of radionuclides concentration	·Zeolite (IE-96) ion-exchange media for Cs adsorption during the PUREX supernatant decontamination operation. ·Titanium treated IE-96 for Cs, Pu and Sr adsorption during the PUREX sludge washing campaigns.	·For salt formation: NaTPB (sodium tetraphenyl borate) precipitation.	(1) ion-exchange or precipitations for Cs. (2) TRUEX for transuranics.	·calcination for granular solid: particle size reduction and control Fluorinel-Na calcine. ·neutralize acidic LHLW up to pH = 13.5. ·remove supernate, including Cs, Al, F, B and possibly Sr. ·extract Cs, and possibly other radioactive elements from supernate and add to HLW. ·redissolve calcine. ·remove actinides from LHLW and/or dissolved calcine. ·separate Zr, Cs, Sr, Tc and Cd from actinide-depleted stream and add to HLW.
special additives	·sodium nitrite added to the PUREX waste tanks to obtain a nitrite/nitrate concentration ratio needed for corrosion passivation. ·NaOH added to maintain high pH for corrosion passivation and U/Pu solubility during the sludge washing campaigns.	·sodium titanate for capturing a trace quantity of Sr-90, Pu and other actinides. ·agitation.	·diatomaceous earth for filtrating. ·nitric acid for ion-exchange regeneration. ·nitrite and caustic for corrosion control.	·aluminum to complex the fluoride to prevent corrosion. ·calcium to form a stable CaF_2 prior to calcination. ·titania.

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sludge treatment	The sludge is washed with water adjusted with NaOH to maintain the pH at approximately 12.5 to remove the nitrate salts and sodium sulfate. Cesium, plutonium, and strontium will be recovered from the waste solution with titanium treated Zeolite.	·leach high-aluminum sludge with excess caustic to dissolve about 75% of the hydrated alumina. ·wash with water to reduce the soluble salt content of the sludge slurry.	·NCAW: sludge washing to remove solubles. ·NCRW, PFP, CC: TRUEX solvent extraction process.	·no real sludge because wastes have never been neutralized. ·minor moves around.
waste transfer and blending method	·The washed PUREX sludge, THOREX and radionuclide loaded Zeolite will be combined in the waste tank farm. The mixture will pass through a recycle grinding stage to improve homogenization of the wastes. The combined waste slurry will then be pumped in a series of batches to the vitrification facility for solidification. ·The improvement of slurry analysis is by a factor of 3 to 5.	slurry pumping	·Resuspension and pumping of slurry. ·Blending with mechanical propellers.	calcine - additive blend analysis and control
sludge and precipitates dilution		formic acid in PRBT (precipitate reactor bottom tank) with Cu catalysts	(not applicable)	no precipitates
glass-making additives	The individual chemicals needed to achieve the target glass composition will be prepared in the Cold Chemical Addition System and then blended with the wastes after ends patch transfer to the Vitrification Facility.	frit	frit (75%)	frit
waste loading	37.5 wt.%: Purex + Thorex: 26%. Zeolite: 11.5%.	·99.9999% activities goes to glass. ·sludge oxides: 28%. ·precipitate oxides: 8%. ·frit: 64%.	25% (wt.) minimal	glass: 33%. ceramics: (50 - 70) %.

	West Valley Demonstration Project (WVDP)	Defense Waste Process Facilities (DWPF)	Hanford Waste Vitrification Plant (HWVP)	Idaho Chemical Processing Plant (ICPP)
base components in melt-feed	Glass forming chemicals plus blended washed PUREX sludge, THOREX, and radionuclides loaded Zeolites	·PHA (Precipitates Hydrolysis Aqueous): 8%. ·frit: 64%. ·sludge: 28%.	·major glass-forming components: 75% frit critical components that must be maintained below specific low concentrations to avoid precipitation in the melter. ·fission products present in minor concentration (less than a few percent) that have solubility in the glass. ·volatile anions that could interact with formic acid used to adjust glass redox state in the melts.	·glass: silica-alkalide-borate :similar to frit, but differs in Na and B. ·ceramics: (1) Zirconia, Alumina, and Calcium Fluoride. (2) Sintering at high pressures.
toxic chemical recovery		mercury from SRAT (Sludge Receipt and Adjustment Tank) by steam stripping.	None (Toxic Chemical-Waste: nitrates, nitrites, chromium, cobolt, heavy metals, cyanides, carbon tetrachloride, selenium, and trichlorethylene)	No. (Maybe in the future.)
organic removal	·sources of organics in closed canisters: lubricants and fluids from the processing and handling equipment. ·Destroyed during the melting process. The canisters are covered during handling and storage to prevent organic contaminations.	·formic acid hydrolysis in SRAT: phenyl groups to benzene groups and subsequent steam distillation. ·sources of organics in closed canisters: hydraulic fluid from can-ister welding process only when there is a leak.	·Formic acid hydrolysis in SRAT. ·Residual organic destruction in melter. ·Qualification of canister closure process to control lubricants and hydraulic fluids.	Not appreciable amount of organics
mix concentration	evaporation	evaporation	evaporation	some evaporation of liquid tank (not the same as DWPF)

	West Valley Demonstration Project (WVDP)	Defense Waste Process Facilities (DWPF)	Hanford Waste Vitrification Plant (HWVP)	Idaho Chemical Processing Plant (ICPP)
melting:				
·heating	1150°C (1050°C ~ 1200°C)	1150°C	1150°C: Conversion process proceeds at a measurable rate.	hot isostatic pressing (HIP): 950° - 1050°, 1200° C at 13.8 MPa (2000 psi)
·conductivity	0.2 - 5.0 ohm-cm at 1150°C.	2 ~ 4 ohm-cm at 1150°C	18-50 s/m at 1150°C	TBD (to be determined)
·redox control	sucrose addition to the waste slurry feed prior to the melter.	formic acid (direct measurement of redox).	formic acid adjustment.	TBD
·redox (Fe^{+3}/Fe^{+2})	0.01 - 0.50	0.0 ~ 0.5 (normally 0.1)	0.005 - 0.3	TBD
·residence time	Melter: (50 - 70) hours (h.). Mean Turntable: (130 - 200) h. batch preparation: 100 h.	65 h. (11 h.: research melt time)	65 h.	20 h.: complete cycle. 1.5 h.: product heat-up. 1-4 h.: product formation.
·cooling rate	600° C/less than 10 h.	From the end of pour to Tg: ·Centerline: 20 h. ·7.6 cm (3") from Center: 16 h. ·15.2 cm (6") from Center: 12 h. ·22.9 cm (9") from Center: 8 h. ·surface: < 1 h.		TBD

	West Valley Demonstration Project (WVDP)	Defense Waste Process Facilities (DWPF)	Hanford Waste Vitrification Plant (HWVP)	Idaho Chemical Processing Plant (ICPP)
•others		formic acid to minimize foaming and to improve the rheology	•formic acid to minimize foaming and to reduce ruthenium volatilization. •density: expansion in volume by accumulating gases. •component accumulation. •conversion rate: the terminal process is affected by the chemical form of raw materials, grain size of raw materials and temperature. •conversion heat: heating the blanket from above in the form of pre-heated batch or upper heating, or producing exothermic reactions within the blanket, enhancing the melting rate. However, exothermic reactions may produce undesirable effects leading to the melting rate reduction.	TBD

	West Valley Demonstration Project (WVDP)	Defense Waste Process Facilities (DWPF)	Hanford Waste Vitrification Plant (HWVP)	Idaho Chemical Processing Plant (ICPP)
·off-gas treatment	The process off-gases are routed through a series of treatment devices: ·cold cap digestion. ·submerged bed scrubber (SBS) - condenses steam from the melter and collects particles > 3 μm diameter. ·high efficiency mist eliminator (HEME) - removes small particles and mist existing the SBS ·high efficiency particulate air (HEPA) filters - a series of these filters are used for final particulate removal. ·selective catalytic reduction (SCR) reactor - used to catalytically remove nitrogen oxides from the process off-gas stream. volatile radionuclides: Tc-99 and Cs-137.	venting for ~30 minutes (min.) after completion of melt fill to collect volatile radionuclides such as Cs-137	·cold cap digestion. ·steam plus nitrogen oxide scrubbing. SBS: submerged bed scrubber. HEME: high efficiency mister eliminator. HEPA: high efficiency particulate air. volatile radionuclides: Tc-99, I-129 and Cs-137.	·calcining off-gas system: - solidify acid waste by heating up. - calcining process as to oxides and fluorides. ·New Waste Calcining Facility (NWCF): wet scrubbing → silicon gel bed → HEPA → stainless steel bean in (3.66-18.29 m) concrete silo. ·volatile radionuclides: Tc-99 and I-129.
·additives/bubbles		agitation		ceramics: frit
·volatility control		small volatility		
·cold trap		(may not be important)		
·removal methods of flammable hydrogen/air mixture		(may not be important)		
·(Balance) -enthalphy and free energy balance. -mass and energy balance. -heat transfer activity balance.		(inappropriate problems)	conversion heat	TBD
·batching or continuous process	semi-continuous or batch	continuous or semi-continuous	batch	

	West Valley Demonstration Project (WVDP)	Defense Waste Process Facilities (DWPF)	Hanford Waste Vitrification Plant (HWVP)	Idaho Chemical Processing Plant (ICPP)
hot operation	1996	12/1993	12/1999	2014
Error:			TBD	TBD
·composition error				
- homogeneity of vessels with mixing.				
- sampling points.				
- sampling frequency.				
- sampling variability	< 5 %	< 5 %		
- accuracy of sample analysis.	> 1 - 5 %			
- knowledge of vessel volume.				
- potential buildup of materials in tank.				
·process troubles				
- non-steady state.		overall process < 10 %.		
- process upset.				
- failing sensor.		no steady state.		
·batching error				
- temperature fluctuation.		~ 25 % (macro batch error < 10 %).		
- melter bubbler pressure fluctuation.				
maximum standard deviation				
overall process simplicity		no problem		·ceramic process: TBD. ·glass process: similar to DWPF.
overall process flexibility		no problem		
overall productivity		no problem		
overall operational variation in glass optimization		difficult to address. Want a full range.		
determination of acceptability		95 % confidence		
chemical composition	> 0.5% by wt. (except o) will be reported	> 0.5% by wt. will be reported.	> 0.5%. All oxide components reported.	> 0.5% 1. zirconia and fluorite. 2. zircon, perovskite and sphere.

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radionuclide inventory	•> 0.05% at $t_{1/2}$ > 10 years up to over 1100 years. •$2x10^5$ (104,300 and maximum 114,700) Ci/can. •2.683 x 10^7 Ci/total (fission products) and 1.489 x 10^5 Ci/total (actinides).	•> 0.05% at $t_{1/2}$ > 10 years up to 1100 years. •1.5 ~ 2.34 10^5 (234,400) Ci/can.	•All with $t_{1/2}$ > 10 years •> 0.05% in Ci to 1100 years. •$\geq$ 2.7 x 10^5 (137,000 or maximum 298,000) Ci/can.	> 0.05% at $t_{1/2}$ > 10 years and up to 1100 years. •108,900 Ci/can.
melt viscosity	•20 < η < 100 poise at 1100°C •1013 η at < 500°C (glass)	20 < η < 100 poise at 1150°C	20 < η < 100 poise at 1150°C	TBD (will be similar to existing ones.) (all below)
liquids temperature	< 1050°C (100°C greater for melting)	< 1050°C (pouring temperature: 1000°C)	pouring temperature: 1000°C)	TBD
leak-tightness	< 10^{-4} atm.cc/sec He (< 10^{-7} atm.cc/sec He (ANSI 14.5))	•10^{-4} atm.cc/sec He: - 10^{-7} atm.cc/sec He at 1.6 MPa (225 psi). - neck: 10^{-8} atm.cc/sec He at 34.5 MPa (5,000 psi). •(liquid penetration test before pouring.)	10^{-4} atm.cc/sec	TBD
canister material	•304L-ASTM A240 (plate) (UNS 530403). •304L-ASTM A312 (pipe) (UNS 530403). •308L-ER308L (W30843) (weld filler metal).	304L, F304L, S21800, F308L	•304L (C<0.03 wt. %). •Nitronic 60 sleeve. •308 weld rod.	TBD
welding: •type	gas tungsten arc welding	upset resistance welding	upset resistance welding	TBD
•condition		force: 330,000 newtons or 70,000 ~ 75,000 lb. power: 240,000 A at 10 ~ 30 V or 0.5 kwh. time: 1.5 second.	force: 330,000 newtons or 70,000 ~ 75,000 lb. power: 240,000 A at 10 ~ 30 V or 0.5 kwh. time: 1.5 second.	
•NDE criteria •inspectibility •microstructural control		not determined not determined not determined		

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labeling	weld bend	welding	welding	TBD
gas	Approximately atmospheric pressure when sealed.	·48 (maximum 150) kPa at 25°C: helium pressure from Alpha decay after 1000 years: < 7 kPa. ·water: vaporization < 4 gm in vapor space. ·organics: CO_2 atmosphere. ·volatile materials: semi-volatile salts condensed during glass pouring inside canisters, mostly alkali-boride, a little chlorides and sulfates.	48 kPa at 25° C	TBD
free liquid	The canister will contain no free liquids (aqueous decontamination solution may exist).	·N_2/H_2O: 19.26 - 43.82. ·maximum 4.6 g of water vapor trapped. ·humidity: 5.5 ~ 18.5% < 20% at 20°C. ·CO_2 depletion.	similar to DWPF	TBD
heat generation (canister)	300 - 376 (311, maximum 342 or 1,500) watts	·< 1500 watts ± 15% or 709 watts. ·< 440°C (transition temperature, Tg).	≤ 1500 watts at year 2025 or 389 (maximum 869) watts	800 watts, or 339 watts (maximum 1,500 watts)
dose rate: · maximum gamma: 10^5 rem/hr. · maximum neutron: 10 rem/hr.	·7,500 rem/hr: gamma. ·10 rem/hr: neutron.	·10^5 rem/hr: gamma. ·10^3 rem/hr: neutron (actual amount < 0.5). ·(2,900 rem/hr at 1 m, 5,500 - 6,000 rem/hr at surface).	≤10^5 rem/hr: gamma. ≤10^3 rem/hr: neutron.	10^5 rem/hr: gamma. 10^3 rem/hr: neutron.
subcriticality	K_{eff} < 0.95 (K_{eff} + 2σ < 4.9x10^{-3})	K_{eff} < 0.95 (conservatively calculated: 0.27. actual value: 0.03.)	K_{eff} < 0.95 (calculated K_{eff} ≤ 0.007)	TBD
temperature	maximum 400° C	maximum 400° C (surface <100 °C)	maximum 400° C	
uniformity and stability of amorphous phase	·Tg: (440 - 480)°C ·softening point: ~510°C	Tg: (450 ~ 500)°C. or (440 ~ 460)°C	Tg: (440 ~ 460)°C	TBD

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void and density	2.7 g/cm³ (room temperature)	2.73 g/cm³	2.75 g/cm³	·(3.0 ~ 3.4) g/cm³ ceramics. ·2.6 g/cm³: glass.
cracking, particulate, stress, and strength	·α = 10x10⁻⁶/°C (25-474°C). ·α = 69.7x10⁻⁶/°C (474-510°C). ·particulate crystals ~ 5-200 µm.	·< 10 µm particles from 10 J/cm² impact. ·surface area; 125 m². ·wt. % fines: 0.14 ~ 0.18.	·< 10 µm particles from 10 J/cm² impact. ·surface area; 125 m². ·wt. % fines: 0.14 ~ 0.18.	TBD
spill control		yes or not during transportation.		TBD
weight, height, > diameter, > thickness,	2,150 (maximum 2,500 kg - 3 m (+0.005, - 0.020) - 61 cm (+1.5-1.0) - 0.34 cm (minimum)	2,182 (maximum 2,500) kg - 3 m (+0.005- 0.020) - 0.61 m (+0.015- 0.010) - 0.95 (minimum 0.85) cm	2,150 (maximum 2,500) kg - 3 m (+0.005- 0.020) - 0.61 m (+0.015- 0.010) - 0.95 (minimum 0.85) cm	2,325 (maximum 2,500) kg - 3.0 m - 61 cm - 0.95 cm
weight of glasses or ceramics alone and fill-height	·1,900 kg. ·85 ± 5% fill.	·1,682 kg. ·upper weight limit: 3,500 kg. ·ovality: ±0.13 cm. ·bowing: ±0.79 cm. ·lifting device geometry: ±0.05 cm. ·waste volume: 626 liter. ·85 (minimum 80) % fill or 15% void space.	·1,650 kg. ·upper weight limit: 3,500 kg. ·minimum thickness: 0.85 cm. ·ovality: ±0.13 cm. ·bowing: ±0.79 cm. ·lifting device geometry: ±0.05 cm. ·waste volume: 626 liter. ·85 (minimum 80) % fill or 15% void space.	1,825 kg minimum 80 % fill
surface contamination	The canister will be decontaminated using a Ce⁺⁴/nitric acid solutions: ·maximum 220 dpm/100 cm² alpha. ·maximum 2200 dpm/100 cm² beta and gamma.	·smear test: maximum 220 dpm/100 cm² alpha. maximum 2200 dpm/100 cm² beta and gamma. ·50 g of canister materials. ·1.1 mg/cm². 0.78 mg/cm². 10⁻⁴ Ci/cm². ·(1) wet glass frit blasting. (2) (575 - 775)° C decomposition of organics.	·smear test: maximum 220 dpm/100 cm² alpha. maximum 2200 dpm/100 cm² beta and gamma. ·50 g of canister materials. ·1.1 mg/cm². 0.78 mg/cm². 10⁻⁴ Ci/cm². ·(1) wet glass frit blasting.	·maximum 220 dpm/100 cm² alpha. ·maximum 2200 dpm/100 cm² beta and gamma.

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drop test and handling feature	7 meters, grapple	7 meter, grapple	7 meter, grapple engaged with the flange	7 meter
volatility		41 mg/can	41 mg/can	TBD
fire tolerance	300 min. at 800°C and 0.9 emissivity coefficient			
Hazardous Materials	TBD	TBD	TBD	TBD
chemical compatibility (impurity attack)				
consistency test	PCT of EA glass	PCT of EA glass (Li + Na + B)	PCT of EA glass	
quality assurance	DOE/RW - 0214	RW - 0214 OGR/B - 14 ANSI/ASME NQA-1	RW - 0214	

SUMMARY

(1) The LHLW inventories at each of four sites are significantly different from one another.

(2) The physical and chemical formulations and the important pre-treatment processes for the LHLW solidification at these sites are all derivatives of DWPF or WVDP, except for the calcine process of ICCP.

(3) Glass-making processes are also very similar to each other, at the four sites. There is still much to determine about the details of the ICCP ceramic processes.

(4) It needs to be confirmed that errors involved in both pre-treatment and glass- or ceramic-making do not affect the integrity nor the performance of glasses nor ceramics.

(5) WAPS elements are nearly the same at the four sites. Nevertheless, it is necessary to relate these elements to the integrity and performance of glasses and ceramics, in compliance with the governing rules.

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