

Estimates of Acetonitrile Generation from Scale Melter Testing of LAW Simulants, VSL-19S4573-1, Rev A

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Office of River Protection

**P.O. Box 450
Richland, Washington 99352**

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Summary Report

Estimates of Acetonitrile Generation from Scale Melter Testing of LAW Simulants

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Rev. A

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1.0 Background

With high nitrate feeds, such as Hanford low activity waste (LAW) feeds, the addition of reductants is necessary in order to control foaming of the molten glass pool in the melter because such foaming can lead to extensive downtime. Sugar, which was used for this purpose at the West Valley Demonstration Project vitrification facility, has been selected as the baseline reductant for the Hanford Waste Treatment and Immobilization Plant (WTP). The amount of sugar required increases with the amount of nitrates present in the feed and decreases with the amount of waste organics present in the feed, which themselves act as reductants. Excessive additions of reductants can be deleterious, leading to over-reduction of the melt and formation of sulfides and molten metals. Consequently, the oxidants and reductants in the feed must be suitably balanced. The basis for achieving this balance was developed by VSL and Atkins for the vitrification of high-sodium-nitrate feeds at Savannah River's M-Area and has been successfully applied to the processing of a wide variety of simulated WTP feeds over many years and was ultimately incorporated into the WTP process control models. The reaction of sugar with nitrates and nitrites in the cold cap controls the redox state of the underlying melt, which prevents foaming, and also significantly decreases the amount of NO_x produced. In addition, however, small amount of organics reaction products are formed. Extensive testing at VSL over the past more than 20 years has shown that acetonitrile is the most prevalent such organic species. That testing has also shown that a significant amount of acetonitrile is captured in the liquid scrubbers in the WTP off-gas treatment system and will therefore be present in the secondary liquid effluents. In particular, acetonitrile is prevalent in the submerged bed scrubber (SBS) and wet electrostatic precipitator (WESP) liquid effluents from WTP LAW off-gas treatment. When these liquids are concentrated in the WTP Effluent Management Facility (EMF) evaporator in the direct feed LAW (DFLAW) flow-sheet, VSL testing has shown that the majority of the acetonitrile partitions to the evaporator condensate. Since the evaporator condensate is directed to the Hanford Effluent Treatment Facility (ETF), this creates a potential issue with the ETF waste acceptance criteria. Consequently, there is a need to assess the available test data in order to develop projections of the likely concentrations of acetonitrile in the streams to ETF. The objective of the present report is to provide such an assessment in order to provide input to flow-sheet model projections.

Sections 2 – 6 of this report provide a brief summary of the source documents and test data sets that were used. Section 7 provides an analysis of, and comparisons between, these data sets with respect to acetonitrile generation and capture in the liquid effluents. Finally, Section 8 provides recommendations for further work.

2.0 “Improving Technetium Retention in Hanford LAW Glass – Phase 2,” K.S. Matlack, I.S. Muller, R. Callow, N. D’Angelo, T. Bardakci, I. Joseph, and I.L. Pegg, Final Report, VSL-11R2260-1, Rev. 0, Vitreous State Laboratory, The Catholic University of America, Washington, DC, 7/20/11.

Melter tests were conducted on the DM1200 with the LAW AN-105 waste simulant (Table 2.1), LAWE4H glass composition, and additives including iron (II) oxalate (which was included to increase the retention of technetium in the glass). Sugar was added to the feed as a reductant to achieve a molar ratio of 0.75 total organic carbon (TOC):NO_x, excluding the organic carbon from iron (II) oxalate. A summary of the test including blow-down volumes of the SBS and WESP are provided in Table 2.2. Samples were collected from the SBS, WESP, and packed bed scrubber (PBS) sumps to measure a variety of constituents specified in the criteria for acceptance into the Hanford site secondary waste treatment facility. Three separate samples from each of the sumps were taken over the course of the test and were sent to TestAmerica, Knoxville (TA) for analyses. Analyses were performed for specified volatiles, semi-volatiles, PCBs, total cyanide, and total organic carbon. Volatile organic concentrations for the SBS, WESP, and PBS solutions are summarized in Table 2.3. Acetonitrile was measured at high concentrations, ranging from 52 to 83 mg/l in SBS and WESP solutions as a byproduct of vitrifying high nitrate LAW simulants with sugar. To calculate the amount of acetonitrile captured in primary off-gas system fluids the amount of feed processed and blow-down volumes given in Table 2.2 were used as well as the measured acetonitrile concentrations at the end of the tests, given in Table 2.3.

3.0 “Tracking the Key Constituents of Concern of the WTP LAW Stream,” K.S. Matlack, H. Abramowitz, I.S. Muller, I. Joseph and I.L. Pegg, Final Report, VSL-16R3840-1, Rev. 0, Vitreous State Laboratory, The Catholic University of America, Washington, DC, 2/16/17.

Melter tests were conducted to determine the retention of technetium and other volatiles in glass while processing three different simulated LAW streams through a DM10 melter equipped with a prototypical off-gas system that concentrates and recycles fluid effluents from the SBS and WESP back to the melter feed. Tests were conducted with the LAW AN-105, AN-104, and AZ-102 waste simulants (Tables 2.1, 3.1, 3.2), LAWE4H, LAWE6H, and LAWE10H glass compositions, and additives including iron (II) oxalate (Table 3.3). Sugar was added to the feed as a reductant to achieve a molar ratio of 0.75 TOC:NO_x, excluding organic carbon from the iron (II) oxalate. A summary of the tests is provided in Table 3.4. Evaporator concentrate and condensate samples were collected to measure a variety of constituents specified in the criteria for acceptance into the Hanford site secondary waste treatment facility. Samples from each side of the evaporator (condensate and concentrate) were taken at the end of each test and were sent to TestAmerica, Knoxville (TA) for analysis. Analyses were performed for specified volatiles, semi-volatiles, PCBs, total cyanide, and total organic carbon (TOC). Volatile organic concentrations for the evaporator solutions are summarized in Table 3.5. Most volatile constituents were present at low concentrations below the reporting limits. Acetonitrile was present at well above the reporting limits in most samples. The amount of measured acetonitrile

increases with increasing nitrogen oxide and sugar feed content, as expected. Acetonitrile concentrations are 25 times higher in the evaporator condensate than in the concentrate, indicating that most of the acetonitrile is volatilized in the evaporator. For the purposes of calculating acetonitrile generation and capture, the total run time, average recycle feed rate (concentrate accumulation rate), and average evaporator feed rate in Table 3.4 were used.

4.0 “DFAW Glass and Feed Qualifications to Support WTP Start-Up and Flow-Sheet Development,” K.S. Matlack, H. Abramowitz, I.S. Muller, M. Brandys, and I.L. Pegg, Final Report, VSL-17R4330-1, Rev. 0, Vitreous State Laboratory, The Catholic University of America, Washington, DC, 11/22/17.

Melter tests were conducted to determine the retention of technetium and other volatiles in glass while processing three different simulated LAW streams through a DM10 melter equipped with a prototypical off-gas system that concentrates and recycles fluid effluents back to the melter feed. Tests were conducted with the LAW AN-105, AP-105SPRN, and AP-105 Full Heel waste simulants (Tables 2.1, 4.1, 4.2), LAWE4H, WDFL1, and WDFL2 glass compositions, and additives (Table 4.3). Sugar was added to the feed as a reductant to achieve a molar ratio of 0.75 TOC:NOx. A summary of the tests is provided in Table 4.4. DM10 evaporator concentrate and condensate samples were collected to measure a variety of constituents specified in the criteria for acceptance into the Hanford site secondary waste treatment facility. Samples from each side of the evaporator (condensate and concentrate) were taken at the end of each test and were sent to TestAmerica, Knoxville (TA) for analysis. Analyses were performed for specified volatiles, semi-volatiles, PCBs, total cyanide, and total organic carbon (TOC). Volatile organic concentrations for the evaporator solutions are summarized in Table 4.5. The only constituent detected at concentrations above the reporting limit or at concentrations higher than the blank was acetonitrile. The amount of measured acetonitrile increases with increasing nitrogen oxide and sugar feed content, as expected. Acetonitrile concentrations are 25 times higher in the evaporator condensate than in the concentrate, indicating that most of the acetonitrile is volatilized in the evaporator. For the purposes of calculating acetonitrile generation and capture, the total run time, average recycle feed rate (concentrate accumulation rate), and average evaporator feed rate in Table 4.4 were used.

5.0 “DFAW Glass and Feed Qualifications for AP-107 to Support WTP Start-Up and Flow-Sheet Development,” K.S. Matlack, H. Abramowitz, I.S. Muller, I. Joseph, and I.L. Pegg, Final Report, VSL-18R4500-1, Rev. 0, Vitreous State Laboratory, The Catholic University of America, Washington, DC, 9/27/18.

A melter test was conducted to determine the retention of technetium and other volatiles in glass while processing the simulated LAW AP-107 stream through a DM10 melter equipped with a prototypical off-gas system that concentrates and recycles fluid effluents back to the melter feed. Tests were conducted with the LAW AP-107 waste simulant (Table 5.1), AP107WDFL glass composition, and additives (Table 5.2). Sugar was added to the feed as a

reductant to achieve a molar ratio of 0.75 TOC:NOx. A summary of the extended duration 200-hour test is provided in Table 5.3. The recycle rate set point was increased from 0.4 kg/hr used in previous tests to 1.2 kg/hr because of the larger evaporator sump volume in order to more rapidly reach steady state. As a result, the recycle liquid was collected in batches that were subsequently mixed with the feed batches instead of being directly and continuously fed into the melter. The average liquid flow rate from the containment tank (into which the SBS and WESP fluids are combined) was 11.5 kg/hr. DM10 evaporator concentrate and condensate samples were collected to measure a variety of constituents specified in the criteria for acceptance into the Hanford site secondary waste treatment facility. Samples from each side of the evaporator (condensate and concentrate) were taken at the end of the test and were sent to TestAmerica, Knoxville (TA) for analysis. Analyses were performed for volatiles, semi-volatiles, PCBs, total cyanide, dissolved and total organic carbon (DOC and TOC), diethylene glycol, and ethanol. Volatile organic concentrations for the evaporator solutions are summarized in Table 5.4. The only constituent detected at concentrations above the reporting limit or at concentrations higher than the blank was acetonitrile. The amount of measured acetonitrile in the condensate is about a factor of two lower than in the preceding tests (4.5 mg/l vs. 7.2 – 10.5 mg/l) and no acetonitrile was detected in the evaporator concentrate. The only detected, unqualified constituent unique to the expanded list of analytes from the present tests (Table 5.5) was acrylonitrile in the condensate. For the purposes of calculating acetonitrile generation and capture, the total run time, average recycle feed rate (concentrate accumulation rate), and average evaporator feed rate in Table 5.3 were used.

6.0 “Iodine Speciation Effects in LAW Feeds,” K.S. Matlack, H. Abramowitz, I.S. Muller, M. Brandys, and I.L. Pegg, Test Plan, VSL-19T4740-1, Rev. 0, Vitreous State Laboratory, The Catholic University of America, Washington, DC, 8/7/19. Also: “Iodine Speciation Effects in LAW Feeds,” K.S. Matlack, H. Abramowitz, and I.L. Pegg, Summary Report, VSL-19S4740-1, Rev. 0, Vitreous State Laboratory, The Catholic University of America, Washington, DC, 10/8/19.

A series of melter test were conducted to determine the retention of iodine in glass and its distribution in off-gas solution effluents while processing the simulated LAW AP-107 stream with various forms of iodine through a DM10 melter equipped with a prototypical off-gas system that concentrates and recycles fluid effluents back to the melter feed. Tests were conducted with the LAW AP-107 waste simulant (Table 5.1), AP107WDFL glass composition, and additives (Table 5.2). Sugar was added to the feed as a reductant to achieve a molar ratio of 0.75 TOC:NOx. Methyl iodide was used as an iodine source for one of the tests and therefore off-gas effluent solutions from that test were sent to TestAmerica, Knoxville (TA) for SW-846-8260B volatile analysis, which includes acetonitrile. A summary of the test with methyl iodide including the total amount of solution blow-down from the WESP and SBS, which includes the amount of solution in the SBS at the end of the test, as well as the measured acetonitrile concentration in that solution is provided in Table 6.1. For the purpose of calculating acetonitrile generation and capture, the total solution mass from the SBS and WESP blow-downs and test-end sums was used.

7.0 Calculated Acetonitrile Generation and Capture

7.1 Results from Tests with Evaporation and Recycle

Acetonitrile generation in the melter and capture in the evaporator condensate and concentrate solutions generated during seven DM10 tests is provided in Table 7.1. In all of these tests the SBS and WESP fluids were directed to the evaporator, which generated the evaporator condensate and concentrate solutions. Consequently, acetonitrile generations and capture is based on only those solutions and there is no need to consider the intermediate SBS and WESP solutions. The accumulated amounts of acetonitrile are the product of the analyzed acetonitrile concentration from samples taken at the end of each test and the total amount of evaporator concentrate and condensate solutions generated during each test. The total volume of concentrate is the product of the test average measured concentrate generation rate and the test duration. The total volume of condensate is the product of the test average condensate generation rate from the evaporator (the measured flow into the evaporator minus the measured concentrate generation rate) and the test duration. This method of calculation provides a conservative result, particularly for longer tests since the highest acetonitrile concentrations would occur in solutions taken at the end of tests. The calculation takes into account the amount of acetonitrile in the SBS sump at the end of each test by projecting how much additional evaporator concentrate and concentrate it would have produced, and assuming the same measured test-end acetonitrile concentrations in those fluids. This contribution is small, however, because the approximately 40 liters in the SBS is twenty times less than the 800 liters that accumulate in the condensate tank during each test.

In order to provide a common basis for comparison of the acetonitrile generation and capture across the various tests, the total amount of acetonitrile was divided by either the total amount of sugar feed to the melter or the total amount of organic carbon fed to the melter. These normalizations are motivated by the fact that acetonitrile is generated from these organics in the melter. The mass of acetonitrile generated and captured per unit mass of sugar in the feed ranged from about 0.45 g/kg sugar for the AP107WDFL composition to about 1.1 for the WLDF1 composition. Feeds with more than 60 g sugar/kg glass resulted in the highest measured concentrations of acetonitrile in process solutions as well as the highest amounts normalized to sugar, indicating that higher sugar concentrations, and therefore higher nitrate and nitrite concentrations, in the feed contribute to higher levels of acetonitrile concentrations. The AP107WDFL composition has a relatively high sugar content normalized to glass, at slightly less than 60 g sugar /kg; the waste simulant is diluted to 5.6 M Na, which increases the water content of the feed which may suppress acetonitrile generation. Normalization of acetonitrile capture to feed carbon shows the same trends, with acetonitrile capture ranging from about 0.83 to about 2.3 g/kg feed carbon. It is also evident that the carbon from the ferric oxalate reduces the amount of acetonitrile normalized to carbon, suggesting that oxalate is less effectively converted to acetonitrile than is sugar. This is consistent with comparisons of tests processing LAWE4H with and without ferric oxalate, which show that ferric oxalate has no effect on the amount of acetonitrile in process solutions, despite the additional organic as oxalate.

7.2 Results from Tests without Recycle

Acetonitrile capture calculated from SBS and WESP solutions generated during DM1200 and DM10 tests without recycle are provided in Table 7.2. The accumulated amounts of acetonitrile are the product of the analyzed acetonitrile concentrations in SBS and WESP samples taken at the end of each test and the total amounts of those solutions generated during each test. In the case of the DM10 test, all of the WESP and SBS blow-downs and the contents of the SBS sump were placed into a single container and the contents were totalized and sampled for analysis. The amount of acetonitrile generated and captured while processing the LAWE4H composition with ferric oxalate on the DM1200 was only about fifteen percent higher than that calculated from the results for the DM10 tests with the same feed when normalized to the amount of sugar or carbon in the feed (1.127 vs. 0.972 g per kg feed sugar). This close agreement is notable since not only were there differences in scale (a factor of 60 in melter scale) and operation (with or without recycle) but also in the manner in which the accumulations were calculated (direct measurement of SBS and WESP solutions vs. measurement of evaporator concentrate and condensate solutions).

The comparison of calculated acetonitrile generation and capture for the two DM10 tests with the LAWAP107 composition is not as favorable: The more recent test without recycle, where the calculation is based on the measured acetonitrile concentrations in the SBS and WESP solutions, showed only about half the acetonitrile generation and capture (0.243 vs. 0.454 g per kg feed sugar) compared to the longer test with recycle, where the calculation is based on the analysis of the evaporator solutions. It is possible that some of this difference may be attributable to the difference in durations and the approach to steady state. Analysis of solutions collected in liquid impingers simulating the caustic scrubber downstream of the WESP indicates that acetonitrile was emitted from the off-gas system at a rate of about 1.0 mg/min during the test with methyl iodide (Table 6.1), demonstrating that not all of the acetonitrile generated in the melter is captured in primary off-gas system fluids. In fact, the total amount of acetonitrile collected in the simulated caustic scrubber solutions was about 2.25 g (Table 6.1), which is about 2.5 times the amount collected in the evaporator condensate plus concentrate (0.902 g; Table 7.2). Therefore, in that test, the majority (about 70%) of the acetonitrile was *not* captured in the SBS and WESP solutions. It is noted that acetonitrile in the exhaust from the WESP ultimately passes through the thermal catalytic oxidizer (TCO) in the WTP LAW off-gas treatment system. Consequently, the destruction efficiency of acetonitrile in the TCO will determine the amount of acetonitrile reporting to the liquid effluents from the caustic scrubber (which are also directed to the ETF). The concentration of methyl iodide in the feed was low (0.14 wt% iodine in the glass assuming total retention) and no methyl iodide was detected in any of the process fluids and therefore the methyl iodide is not likely to have an effect on acetonitrile generation or capture in primary off-gas system solutions.

8.0 Recommendations

Based on the results from the tests described above, a conservative overall level of acetonitrile generation and capture in the primary off-gas system fluids is one gram of

acetonitrile per kilogram of sugar fed to the melter. This estimate is based on analysis of solutions generated while processing LAW simulants with a WTP prototypical melter and off-gas system components. The corresponding value based on total carbon would be two grams of acetonitrile per kilogram of carbon fed to the melter. However, the contribution to acetonitrile generation of carbon in forms other than sugar is lower and is not well known; therefore acetonitrile generation and capture normalized to sugar is a more reliable convention. A predictive model for the amount of acetonitrile generation and capture as a function of feed composition would require further testing with a broader range of LAW feeds with sampling and analysis for acetonitrile in primary off-gas solutions as well as the process exhaust stream.

The amount of acetonitrile in primary off-gas system solutions is a product of two distinctly different processes: one is the generation of acetonitrile in the melter plenum as a byproduct of reactions between nitrates/nitrites and sugar; the other is efficiency of acetonitrile capture from the exhaust stream into the SBS and WESP (and caustic scrubber) liquids. The former certainly depends on the amount of nitrates/nitrites and sugar in the melter feed. However, while oxalate appears to have little effect on acetonitrile generation, the same cannot be asserted for all other organic compounds in the waste or for ammonia which is present in the recycle stream. Testing to assess the generation of acetonitrile with the range of organics expected to be present in LAW streams would therefore be useful. The effect of feed water content on the generation of acetonitrile is also an unknown. To fully quantify acetonitrile generation as a function of feed composition, tests would need to be performed with systematically varying melter feed compositions with monitoring of the SBS and WESP fluids as well as the exhaust stream for acetonitrile. The extent to which the SBS and WESP remove acetonitrile from the melter exhaust stream is also not well characterized. Some of the differences between the acetonitrile accumulations normalized to feed sugar content in the test data may be attributable to differences in SBS solution chemistry, which evolves over the course of each test. Profound differences in decontamination factors for scrubbers are often observed as a function of solution pH. Measurements of the scrubbing efficiency of the SBS over a range of solution chemistries would be useful in order to better define estimates of overall acetonitrile capture in the primary off-gas system effluents. These uncertainties in acetonitrile generation and removal in the primary off-gas system fluids could be readily addressed with scaled melter and off-gas system testing using a range of LAW compositions with liquid effluent and exhaust sampling for acetonitrile.

Table 2.1. LAWE4H Waste Simulant Recipe at 8 Molar Sodium Based on AN-105 Waste Composition.

Envelope Constituents	Simulant AN-105 Modified for E4H	Glass Oxides	LAWE4H Simulant as Oxides (wt%)	Waste Contribution to Glass	Source in Simulant	Order for Addition	Formula Weight	Assay*	Target Weight (g)	
-	mg/L	M	Loading	-	27.72%	In 270.42 ml water add following compounds in the order listed below				
Al	30554	1.132	Al ₂ O ₃	17.87	4.95	Al(NO ₃) ₃ .9H ₂ O, 60% sol. Al(OH) ₃	1 8	375.14 78.00	0.61 1.00	422.01 35.22
B	79	0.007	B ₂ O ₃	0.08	0.02	H ₃ BO ₃	2	61.83	0.99	0.45
Cr	624	0.012	Cr ₂ O ₃	0.28	0.08	Na ₂ CrO ₄ *4H ₂ O	9	234.04	0.99	2.85
K	5223	0.134	K ₂ O	1.95	0.54	KOH	7	56.10	0.91	8.25
Na	183920	8.000	Na ₂ O	76.74	21.27	NaOH, 50% sol. d=1.53	6	40.00	0.50	458.04
Ni	70	0.001	NiO	0.03	0.01	NiO	3	74.69	1.00	0.09
Pb	83	0.0004	PbO	0.03	0.01	PbO	4	223.20	1.00	0.09
Si	157	0.006	SiO ₂	0.10	0.03	SiO ₂	5	60.09	0.99	0.34
Cl	2304	0.065	Cl	0.71	0.20	NaCl	11	58.45	0.99	3.84
F	912	0.048	F	0.28	0.08	NaF	12	42.00	0.99	2.04
PO ₄	1899	0.020	P ₂ O ₅	0.44	0.12	Na ₃ PO ₄ .12H ₂ O	10	380.12	0.99	7.68
SO ₄	5764	0.060	SO ₃	1.49	0.41	Na ₂ SO ₄	13	142.06	0.99	8.61
NO ₂	85428	1.857	-	-	-	NaNO ₂	17	69.00	0.97	128.79
NO ₃	126988	2.048	-	-	-	NaNO ₃	-	84.99	0.99	0.00
TOC	2093	0.174	-	-	-	Na ₂ CO ₃	-	105.99	1.00	0.00
Acetate	2250	0.038	-	-	-	Sodium Acetate (C2)	14	136.08	0.99	5.24
Formate	2135	0.047	-	-	-	Sodium Formate (C1)	15	68.01	0.99	3.26
Glycolate	1936	0.025	-	-	-	Glycolic Acid (C2)	16	76.05	0.71	2.73
-	-	-	SUM	100.00	27.72	Target Glass			1165.59	
-	-	-				Estimated Total simulant wt.			1359.94	

- Empty data field.

* Assay refers to the purity of the raw material as specified by the vendor.

Table 2.2 Summary of DM1200 Test Conditions and Results.

Glass and Feed Composition		LAWE4H + Fe(II) Oxalate
Time	Feed Start	8/17/10 16:37
	Feed End	8/20/10 2:31
	Water Feeding (hr)	1.0
	Feeding Interruptions (min)	4
	Interval (hr)	57.9
Feed Processed (kg)		12031
Steady State Production Rate (kg/m ² /day)		2150
Average Production Rate (kg/m ² /day)*		2140
Average Production Rate (kg/m ² /day)\$		2020
Glass Discharged (kg)		5847
SBS blowdown Volume (gal)		830
WESP blowdown Volume (gal)		140.5

* - Rates calculated from feed data.

\$ - Rates calculated from glass poured.

Note: Rates do not take into account the time for water feeding and cold cap burn-off.

Table 2.3. Volatile Constituents from the SBS, WESP, and PBS Sumps During the DM1200 Test.

Constituent	Reporting limit µg/L	SBS Sump			WESP Sump			PBS Sump		
		Sample Z-1765-R1 µg/L	Sample Z-1783-R2 µg/L	Sample Z-1801-R3 µg/L	Sample Z-1771-R1 µg/L	Sample 1789-R2 µg/L	Sample Z-1807-R3 µg/L	Sample Z-1777-R1 µg/L	Sample Z-1795-R2 µg/L	Sample Z-1813-R3 µg/L
Acetone	10	1500	1600	1900	1300	1500	1300	11 J	ND	11 J
Acetonitrile	20	80000	80000	83000 D	52000	70000	67000 D	210	100	120
Benzene	10	4.5 J	4.5 J	3.5 J	4.3 J	4.3 J	2.7 J	0.21 J	0.22 J	0.22 J
Bromodichloromethane	1	ND	ND	ND	ND	ND	ND	ND	ND	0.68 J
2-Butanone (MEK)	5	ND	ND	100	ND	ND	76	ND	ND	ND
Carbon Disulfide	1	ND	ND	ND	ND	ND	0.93 J	ND	0.21 J	0.21 J
Carbon Tetrachloride	1	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chlorodibromomethane	1	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	1	ND	ND	1.5 J	ND	ND	1.8 J	0.31 J	1.4 J	2.2 J
Methylene chloride	2	29 J,B	41 J,B	4.2 J,B	31 J,B	30 J,B	4.6 J,B	1.6 J,B	1.3 J,B	1.4 J,B
4-Methyl-2-pentanone (MIBK)	5	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene	1	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrahydrofuran	4	ND	ND	ND	75 J	ND	21 J	4.1 J	ND	7.6 J
1,1,1-Trichloroethane	1	ND	ND	ND	ND	ND	ND	ND	ND	ND
1-butyl alcohol (tentatively identified)	TBD	ND	ND	ND	ND	ND	ND	ND	ND	ND

B - Method blank contamination. The associated method blank contains the target analyte at a reportable level.

J – Estimated result. Result is less than RL (reporting limit).

D - Result was obtained from the analysis of a dilution.

ND – Not Detectable

TBD – To Be Determined

Table 3.1. LAWE6H Simulant Recipe at Nominal 7 Molar Sodium Based on AN-104 Waste Composition.

Envelope Constituents	AN-104 Waste from 11.6M to 7 M Na + recycles	Simulant AN-104 Modified for E6H		Recycled Additions	Glass Oxides	LAWE6H Simulant As Oxides (wt%)	Waste Contribution To Glass (wt%)	Source in Simulant	Order for Addition	Formula Weight	Assay*	Target Weight (g)		
-	mg/L	mg/L	Molarity	M	Loading	-	20.88%	In 380.21 ml water add following compounds in the order listed below						
Al	25069	25069	0.929	-	Al ₂ O ₃	16.55	3.46	Al(NO ₃) ₃ .9H ₂ O, 60% sol.	1	375.14	0.61	412.76		
-	-	-	-	-	-	-	-	Al(OH) ₃	4	78.00	1.00	20.47		
Cr	734	734	0.014	-	Cr ₂ O ₃	0.37	0.08	Na ₂ CrO ₄ .4H ₂ O	5	234.04	0.99	3.35		
K	6140	6140	0.157	-	K ₂ O	2.58	0.54	KOH	3	56.10	0.91	9.70		
Na	160930	159183&	6.924&	0.076\$	Na ₂ O	75.81	15.66+0.17\$	NaOH, 50% sol. d=1.53	2	40.00	0.50	294.59		
Ni	84	84	0.001	-	NiO	0.04	0.01	NiO	6	74.69	1.00	0.11		
Pb	99	99	0.001	-	PbO	0.04	0.01	PbO	7	223.20	1.00	0.11		
Si	228	228	0.008	-	SiO ₂	0.17	0.04	SiO ₂	8	60.09	0.99	0.49		
Cl	2688	1274&	0.036&	0.040\$	Cl	0.94	0.09+0.11\$	NaCl	10	58.45	0.99	2.12		
F	1064	894&	0.047&	0.009\$	F	0.37	0.07+0.01\$	NaF	11	42.00	0.99	2.00		
PO ₄	2238	2238	0.024	-	P ₂ O ₅	0.58	0.12	Na ₃ PO ₄ .12H ₂ O	9	380.12	0.99	9.05		
SO ₄	8708	7401&	0.077&	0.014\$	SO ₃	2.54	0.45+0.08\$	Na ₂ SO ₄	12	142.06	0.99	11.06		
NO ₂	78634	78634	1.709	-	NO ₂	-	-	NaNO ₂	15	69.00	1.00	118.54		
NO ₃	124203	124203	2.003	-	NO ₃	-	-	NaNO ₃		84.99	0.99	0.00		
CO ₃	31659	31659	0.528	-	CO ₃	-	-	Na ₂ CO ₃	16	105.99	1.00	55.92		
Org.Carbon	2044	2044	0.170	-	-	-	-	-	-	-	-	-		
Acetate	2284	2284	0.039	-	-	-	-	Sodium Acetate (C2)	13	136.08	0.99	5.31		
Formate	4176	4176	0.093	-	-	-	-	Sodium Formate (C1)	14	68.01	0.99	6.37		
								Target Glass Weight (with recycled off-gas solution to 7M Na) #					1370.37	
					SUM	100.00	20.51+0.37\$	Total Simulant Weight					1332.16	

- Empty data field.

* Assay refers to the purity of the raw material as specified by the vendor.

& Does not include the contribution from recycled off-gas streams.

\$ Addition due to off-gas recycle estimated to be 111% Cl⁻, 19% F⁻ and 17.65% SO₄²⁻ as sodium salts.

Amount of glass produced from the "Total Simulant Weight" once glass formers are added and the mixture is vitrified.

Table 3.2. LAWE10H Simulant Recipe at 2 Molar Sodium Based on AZ-102 Waste Composition.

Envelope Constituents	AZ-101 Waste from 3.5M to 2 M Na + recycles	Simulant AZ-102 Modified for E10H	Recycled Additions	Glass Oxides	LAWE10H Simulant as Oxides (wt%)	Waste Contribution to Glass (wt%)	Source in Simulant	Order for Addition	Formula Weight	Assay*	Target Weight (g)							
-	mg/L	mg/L	Molarity	M	Loading	-	6.20%	In 957.12 ml water add following compounds in the order listed.										
Al	123	123	0.005	-	Al ₂ O ₃	0.26	0.02	Al(NO ₃) ₃ .9H ₂ O, 60% sol.	2	375.14	0.61	2.82						
Cr	760	760	0.015	-	Cr ₂ O ₃	1.25	0.08	Na ₂ CrO ₄ .4H ₂ O	5	234.04	0.99	3.47						
K	6412	6412	0.164	-	K ₂ O	8.70	0.54	KOH	4	56.098	0.91	10.13						
Na	45980	42830&	1.863&	0.137	Na ₂ O	69.84	4.03+0.30 ^s	NaOH, 50% sol. d=1.53	3	40.00	0.50	6.12 &						
Ni	88	88	0.002	-	NiO	0.13	0.01	NiO	6	74.69	1.00	0.11						
Pb	103	103	0.001	-	PbO	0.13	0.01	PbO	7	223.20	1.00	0.11						
Si	171	171	0.006	-	SiO ₂	0.41	0.03	SiO ₂	8	60.09	0.99	0.37						
Cl	2809	1331&	0.038&	0.042 ^s	Cl	3.17	0.09+0.11 ^s	NaCl	10	58.45	0.99	2.22&						
F	1111	933&	0.049&	0.009 ^s	F	1.25	0.07+0.01 ^s	NaF	11	42.00	0.99	2.08&						
PO ₄	2323	2323	0.024	-	P ₂ O ₅	1.96	0.12	Na ₃ PO ₄ .12H ₂ O	9	380.12	0.99	9.39						
SO ₄	13744	9621&	0.100&	0.043 ^s	SO ₃	12.91	0.56+0.24 ^s	Na ₂ SO ₄	12	142.06	0.99	14.37&						
NO ₂	23680	23680	0.515	-	NO ₂	-	-	NaNO ₂	14	69.00	1.00	35.70						
NO ₃	7837	7837	0.126	-	NO ₃	-	-	NaNO ₃	15	84.99	0.99	9.67						
CO ₃	23080	23080	0.385	-	CO ₃	-	-	Na ₂ CO ₃	1	105.99	1.00	40.76						
Org.Carbon	608	608	0.051	-	-	-	-	-	-	-	-							
Oxalate	2242	2242	0.025	-	-	-	-	Oxalic Acid (C2)	13	126.00	1.00	3.21						
								Target Glass (with recycled off-gas solution to 2M Na) [#]				1431.41						
				SUM	100.00	5.55+0.65	Total Simulant Weight				1097.66							

- Empty data field.

* Assay refers to the purity of the raw material as specified by the vendor.

& Does not include the contribution from recycled off-gas streams.

^s Addition due to off-gas recycle estimated to be 111% Cl⁻, 19% F⁻ and 42.86% SO₄²⁻ as sodium salts.

[#] Amount of glass produced from the "Total Simulant Weight" once glass formers are added and the mixture is vitrified.

Table 3.3. Glass Former Additives for 1 Liter of LAWE Waste Simulants and Corresponding Feed Properties.

Additive Source	Feed LAWE4H	Feed LAWE6H	Feed LAWE10H
Additives in Glass (wt%)	72.28%	79.12%	93.80%
Kyanite (Al_2SiO_5) 325 Mesh (Kyanite Mining) (g)	18.15	56.86	147.13
H_3BO_3 (US Borax – Technical Granular) (g)	202.23	239.75	253.08
Wollastonite NYAD 325 Mesh (NYCO Minerals) (g)	63.09	164.63	219.20
Fe_2O_3 (97% Alfa) (g)	-	-	71.22
Fe(II) Oxalate dihydrate (g)	137.67	162.69	-
Li_2CO_3 (Chemetall Foote Co. Technical grade) (g)	-	81.55	152.46
Olivine (Mg_2SiO_4) 325 Mesh (#180 Unimin) (g)	34.48	39.26	84.78
Na_2CO_3 (Technical grade) (g)	-	-	34.18
SiO_2 (Sil-co-Sil 75 US Silica) (g)	446.62	459.37	465.92
TiO_2 (Rutile Airfloated Chemaloy) (g)	16.92	20.04	21.08
ZnO (KADOX – 920 Zinc Corp. of America) (g)	39.98	47.28	49.81
Zircon ZrSiO_4 (Flour) Mesh 325 (AM. Mineral) (g)	51.69	60.97	64.55
Sucrose as Reductant (added only to actual melter feed) (g)	78.50	74.50	12.26
Estimated Simulant Weight for 1 liter (g) &	1356	1332	1098
Sum of Additives (g)	933	1241	1563
Sum of Complete Batch (g)	2289	2573	2661
Final Volume (l)	1.36	1.49	1.61
Measured Density on crucible batch (g/ml)	1.69	1.73	1.65
Target Glass Produced (g/l simulant)	1166	1370	1431
Measured Weight % Water in Slurry Feed on crucible batch	40%	35%	40%
Target Weight % Additives in Slurry	48%	48%	59%
Target Glass Yield for crucible batch (g/kg of Feed)	508	532	539
Measured Glass Yield on crucible batch (g/kg of Feed)	480	510	536
Target Glass Yield (g/l of Feed)	859	920	989
Target Total Solids (g/l of Feed)	1012	1127	993
Target Additives (g/l of Feed)	688	833	969

- Empty data field

& These do not include the contribution from recycled off-gas streams. All measured property values are for feed including estimated recycled off-gas.

Table 3.4. Summary of DM10 Test Conditions and Results.

Test		1	2	3
Time	Feed Start	4/26/16 12:00	5/10/16 12:00	5/23/16 11:45
	Feed End	4/29/16 11:20	5/13/16 12:00	5/26/16 12:02
	Interval	71.3 hr	72.0 hr	72.3 hr
Glass	Target Glass	LAWE10H	LAWE6H	LAWE4H
	Mass Poured	153.5 kg	136.2 kg	142.8 kg
	Average Glass Production Rate	2437 kg/m ² /day	2152 kg/m ² /day	2257 kg/m ² /day
Feed	Mass Fed (excluding recycle)	262 kg	280 kg	272.5 kg
	Target glass Conversion	0.55 kg/kg	0.50 kg/kg	0.53 kg/kg
	Iron Additive Source	Fe ₂ O ₃	Fe(II) oxalate	Fe(II) oxalate
	Average Slurry Feed Rate	3.7 kg/hr	3.9 kg/hr	3.8 kg/hr
	Average Recycle Feed Rate	0.41 l/h	0.40 l/h	0.40 l/h
	Average Solution Flow into the Evaporator	11.8 kg/hr	12.2 kg/hr	12.6 kg/hr

Table 3.5. Measured Volatile Constituents in Evaporator Solutions (µg/L).

Constituent	LAWE10H			LAWE6H				LAWE4H			
	Reporting limit	Concentrate	Condensate	Reporting limit	Concentrate	Reporting Limit	Condensate	Reporting Limit	Concentrate	Reporting Limit	Condensate
Acetone	50	ND	12 J	50	13J	10	86	50	20 J	200	150 J
Acetonitrile	100	ND	740	100	220	20	5800 E	100	400	400	10000
Benzene	5.0	ND	ND	5.0	0.13 J	1.0	0.12 J	5.0	ND	20	0.59 J
Bromodichloromethane	5.0	0.59 J	ND	5.0	0.75 J	1.0	0.083 J	5.0	0.74 J	20	ND
2-Butanone (MEK)	25	ND	ND	25	ND	5.0	5.0	25	ND	100	ND
Carbon Disulfide	5.0	ND	ND	5.0	ND	1.0	ND	5.0	ND	20	ND
Carbon Tetrachloride	5.0	ND	ND	5.0	0.41 J	1.0	ND	5.0	0.29 J	20	ND
Chlorodibromomethane	5.0	ND	ND	5.0	ND	1.0	ND	5.0	ND	20	ND
Chloroform	5.0	1.5 J	0.34 J	5.0	3.3 J	1.0	0.69 J	5.0	3.4 J	20	0.93 J
Methylene chloride	10	1.1 J, B	1.1 J, B	10	0.87 J, B	2.0	0.082 J, B	10	0.75 J, B	40	2.8 J, B
4-Methyl-2-pentanone (MIBK)	25	ND	ND	25	ND	5.0	ND	25	ND	100	ND
Tetrachloroethene	5.0	ND	ND	5.0	ND	1.0	ND	5.0	ND	20	ND
Tetrahydrofuran	20	ND	3.7 J	20	ND	4.0	3.1 J	20	ND	80	ND
1,1,1-Trichloroethane	5.0	ND	ND	5.0	ND	1.0	ND	5.0	ND	20	ND

E – Estimated result. Result concentration exceeds calibration range.

B - Method blank contamination. The associated method blank contains the target analyte at a reportable level.

J – Estimated result. Result is less than RL (reporting limit).

D - Result was obtained from the analysis of a dilution.

ND – Not Detectable

TBD – To Be Determined

Table 4.1. LAW AP-105 Waste Simulant Recipe at 5.6 Molar Sodium for “AP-105 SPRN”.

Envelope Constituents [#]	AP-105 SPRN	Diluted to 5.6 M Na with 725.18 ml /L of 0.1 M NaOH	Glass Components	Simulant as Glass Components (wt%)	Source in Simulant	Order for Addition	Formula Weight	Assay*	Target Weight (g)		
-	mg/L	mg/L	Molarity	Loading	100%	In 544.3 ml water add following compounds in the order listed below					
Al	20620	11962	0.4433	Al ₂ O ₃	10.87	Al(NO ₃) ₃ .9H ₂ O, 60% sol.	1	375.14	0.607	274.04	
Ca	71	41	0.0010	CaO	0.03	Ca(NO ₃) ₂ .4H ₂ O	2	236.16	0.998	0.24	
Cr	500	290	0.0056	Cr ₂ O ₃	0.20	Na ₂ CrO ₄ .4H ₂ O	8	234.04	0.995	1.31	
K	4814	2792	0.0714	K ₂ O	1.62	KOH	7	56.10	0.908	4.41	
Na	221939	128744	5.6000	Na ₂ O	83.48	NaOH, 50% sol. d=1.53	6	40.00	0.501	274.94	
Ni	46	27	0.0005	NiO	0.02	Ni(OH) ₂	3	92.72	1.000	0.04	
Pb	25	14	0.0001	PbO	0.01	PbO	4	223.20	1.000	0.02	
Si	111	64	0.0023	SiO ₂	0.07	SiO ₂	5	60.09	0.990	0.14	
Cl	6411	3719	0.1049	Cl	1.79	NaCl	10	58.45	0.994	6.17	
F	111	64	0.0034	F	0.03	NaF	11	42.00	1.005&	0.14	
PO ₄	3263	1893	0.0199	P ₂ O ₅	0.68	Na ₃ PO ₄ .12H ₂ O	9	380.12	1.006&	7.53	
SO ₄	5184	3007	0.0313	SO ₃	1.21	Na ₂ SO ₄	12	142.06	0.998	4.46	
NO ₂	91183	52894	1.1499	-	-	NaNO ₂	16	69.00	0.995	79.74	
NO ₃	182828	106056	1.7106	-	-	NaNO ₃	17	84.99	0.990	32.49	
CO ₃	11317	6565	0.1094	-	-	Na ₂ CO ₃	18	105.99	1.000	11.60	
Org. Carbon	2718	2792	0.2327	-	-	-	-	-	-	-	
Acetate ^s	6857	3978	0.0673	-	-	Sodium Acetate (C2)	13	136.08	1.001&	9.15	
Formate ^s	6857	3978	0.0884	-	-	Sodium Formate (C1)	14	68.01	1.013&	5.93	
Oxalate	734	426	0.0048	-	-	Sodium Oxalate (C2)	15	134.00	0.990	0.65	
-	-	-	-	SUM	100.0	Total simulant Weight (Estimated)					1257.3

- Empty data field.

^s Equal amounts (mg/L) of acetate and formate are added to meet the TOC content.

[#] Constituents at concentrations less than 25 mg/L are not included. This eliminated Bi, Fe, Hg, I, La, Mn, Sr, Tc, U and Zr.

* Assay refers to the purity of the raw material as specified by the vendor.

& Assay value greater than one for any raw material containing sodium is based on the sodium content of that raw material.

Table 4.2. LAW AP-105 Waste Simulant Recipe at 5.6 Molar Sodium for “Full-Heel”.

Envelope Constituents [#]	AP-105 Full Heel		Glass Components	Simulant as Glass Components (wt%)	Source in Simulant	Order for Addition	Formula Weight	Assay*	Target Weight (g)
-	mg/L	Molarity	Loading	100%	In 732.2 ml water add following compounds in the order listed below				
Al	3742	0.1387	Al ₂ O ₃	3.59	Al(NO ₃) ₃ .9H ₂ O, 60% sol.	1	375.14	0.607	85.73
Ca	-	-	CaO	-	-	-	-	0.998	-
Cr	303	0.0058	Cr ₂ O ₃	0.22	Na ₂ CrO ₄ .4H ₂ O	6	234.04	0.995	1.37
K	3651	0.0934	K ₂ O	2.23	KOH	5	56.10	0.908	5.77
Na	128160	5.575	Na ₂ O	87.60	NaOH, 50% sol. d=1.53	4	40.00	0.501	157.80
Ni	-	-	NiO	-	-	-	-	1.000	-
Pb	20	0.0001	PbO	0.01	PbO	2	223.20	1.000	0.02
Si	2	0.0001	SiO ₂	0.00	SiO ₂	3	60.09	0.990	0.00
Cl	3987	0.1125	Cl	2.02	NaCl	8	58.45	0.994	6.61
F	720	0.0379	F	0.37	NaF	9	42.00	1.005 ^{&}	1.58
PO ₄	3107	0.0327	P ₂ O ₅	1.18	Na ₃ PO ₄ .12H ₂ O	7	380.12	1.006 ^{&}	12.36
SO ₄	6587	0.0686	SO ₃	2.78	Na ₂ SO ₄	10	142.06	0.998	9.76
NO ₂	56873	1.2364	-	-	NaNO ₂	14	69.00	0.995	85.74
NO ₃	115328	1.8601	-	-	NaNO ₃	15	84.99	0.990	123.97
CO ₃	10274	0.1712	-	-	Na ₂ CO ₃	16	105.99	1.000	18.15
Org. Carbon	3025	0.2521	-	-	-	-	-	-	-
Acetate ^{\$}	4448	0.0753	-	-	Sodium Acetate (C2)	11	136.08	1.001 ^{&}	10.23
Formate ^{\$}	4448	0.0988	-	-	Sodium Formate (C1)	12	68.01	1.013 ^{&}	6.63
Oxalate	120	0.0014	-	-	Sodium Oxalate (C2)	13	134.00	0.990	0.18
-	-	-	SUM	100.0	Total simulant Weight (Estimated)				1258.1

- Empty data field.

^{\$} Equal amounts (mg/L) of acetate and formate are added to meet the TOC content.

[#] Constituents at concentrations less than 25 mg/L are not included. This eliminated Bi, Fe, Hg, I, La, Mn, Sr, Tc, U and Zr.

* Assay refers to the purity of the raw material as specified by the vendor.

& Assay value greater than one for any raw material containing sodium is based on the sodium content of that raw material.

Table 4.3. Glass Former Additives for 1 Liter of LAWE4H and DFLAW “AP105-SPRN” (WDFL1) and DFLAW “Full Heel” (WDFL2) Waste Simulants and Corresponding Feed Properties.

Additive Source	Feed LAWE4H	Feed WDFL1	Feed WDFL2
Additives in Glass (wt%)	72.28%	74.84%	82.58%
Kyanite (Al ₂ SiO ₅) 325 Mesh (Kyanite Mining) (g)	18.15	48.28	106.91
H ₃ BO ₃ (US Borax – Technical Granular) (g)	202.23	147.52	202.05
Wollastonite NYAD 325 Mesh (NYCO Minerals) (g)	63.09	37.73	130.10
Fe ₂ O ₃ (99.6% Alfa) (g)	60.14	43.30	59.08
Li ₂ CO ₃ (Chemetall Foote Co. Technical grade)	-	-	60.67
Olivine (Mg ₂ SiO ₄) 325 Mesh (#180 Unimin) (g)	34.48	25.09	65.34
SiO ₂ (Sil-co-Sil 75 US Silica)) (g)	446.62	305.20	364.39
TiO ₂ (Rutile Airfloated Chemaloy) (g)	16.92	10.70	14.65
ZnO (KADOX – 920 Zinc Corp. of America) (g)	39.98	28.92	39.62
Zircon ZrSiO ₄ (Flour) Mesh 325 (AM. Mineral) (g)	51.69	37.39	51.22
Sucrose as Reductant (nominal) (g)	78.50	54.51	59.00
Simulant Weight for 1 liter (g)	1360	1257	1258
Sum of Additives (g)	933	684	1062
Sum of Complete Batch (g)	2293	1941	2320
Final Volume (l)	1.324	1.30	1.45
Measured Density (g/ml)	1.69	1.49	1.60
Expected Glass Produced (g) ; i.e., Glass Yield (g/l of simulant)	1166	826	1132
Measured Weight % Water in Slurry Feed	40%	49%	42%
Weight % Additives in Slurry	48%	35%	47%
Glass Yield calculated (g/kg of Feed)	508	426	488
Glass Yield (g/l of Feed)	859	634	781
Estimated Total Solids (g/l of Feed)	1012	745	960
Estimated Additives (g/l of Feed)	688	525	732
Measured glass Yield (g/kg of Feed)	480	413	482

Table 4.4. Summary of DM10 Test Conditions and Results.

Test		1	2	3
Time	Feed Start	2/14/17 11:05	2/28/17 12:00	3/21/17 11:15
	Feed End	2/17/17 11:05	3/3/17 12:00	3/24/17 11:15
	Interval	72.0 hr	72.0 hr	72.0 hr
Glass	Target Glass	LAWE4H	WLDF1	WLDF2
	Mass Poured	135.1 kg	119.0 kg	128.5 kg
	Average Glass Production Rate	2144 kg/m ² /day	1889 kg/m ² /day	2040 kg/m ² /day
Feed	Mass Fed (excluding recycle)	256.6 kg	287.0 kg	266.6 kg
	Target glass Conversion	0.53 kg/kg	0.414 kg/kg	0.476 kg/kg
	Average Slurry Feed Rate	3.56 kg/hr	4.0 kg/hr	3.7 kg/hr
	Average Recycle Feed Rate	0.40 l/h	0.41 l/h	0.42 l/h
	Average Glass Production Rate	2159 kg/m ² /day	1886 kg/m ² /day	2014 kg/m ² /day
Average Bubbling Rate		3.0 lpm	1.3 lpm	0.2 lpm
Average Solution Flow into the Evaporator		11.3 kg/hr	12.3 kg/hr	10.4 kg/hr

Table 4.5. Measured Volatile Constituents (SW-846-8260B) in DM10 Evaporator Solutions (µg/L).

Constituent	AN-105 / LAWE4H				AP-105 / WDFL1				AP-105 / WDFL2			
	Reporting limit	Concentrate	Reporting limit	Condensate	Reporting limit	Concentrate	Reporting Limit	Condensate	Reporting Limit	Concentrate	Reporting Limit	Condensate
Acetone	11.7	6.99 J B	50	272 B	10	ND	250	ND	13.5	5.8 J	200	172 J
Acetonitrile	23.4	397	100	10500 E	20	396	500	9090	27	273	400	7180
Benzene	1.2	0.17	5.0	ND	1.0	ND	25	ND	1.4	ND	20	ND
Bromodichloromethane	1.2	ND	5.0	ND	1.0	ND	25	ND	1.4	ND	20	ND
2-Butanone (MEK)	5.9	ND	25	10.6 J	5.0	ND	125	ND	6.8	ND	100	ND
Carbon Disulfide	1.2	ND	5.0	ND	1.0	ND	25	ND	1.4	0.1 J	20	ND
Carbon Tetrachloride	1.2	0.12 J	5.0	ND	1.0	0.21 J	25	ND	1.4	ND	20	ND
Chlorodibromomethane	1.2	ND	5.0	ND	1.0	ND	25	ND	1.4	ND	20	ND
Chloroform	1.2	0.10 J	5.0	ND	1.0	ND	25	ND	1.4	ND	20	ND
Methylene chloride	2.3	0.21 J, B	10	2.75 J, B	2.0	0.07 J, B	50	11.2 J, B	2.7	0.71 J, B	40	7.91 J
4-Methyl-2-pentanone (MIBK)	5.9	ND	25	ND	5.0	ND	125	ND	6.8	ND	100	ND
Tetrachloroethene	1.2	ND	5.0	ND	1.0	ND	25	ND	1.4	ND	20	ND
Tetrahydrofuran	4.7	ND	20	7.44 J	4.0	ND	100	ND	5.4	ND	80	ND
1,1,1-Trichloroethane	1.2	ND	5.0	ND	1.0	ND	25	ND	1.4	ND	20	ND

E – Estimated result. Result concentration exceeds calibration range.

B - Method blank contamination. The associated method blank contains the target analyte at a reportable level.

J – Estimated result. Result is less than RL (reporting limit).

ND – Not Detectable

TBD – To Be Determined

Table 5.1. LAW AP-107 Waste Simulant Recipe with Recycle at 5.6 Molar Sodium for Small-Scale Tests (per liter).

Envelope Constituents	LAW AP-107 [60]	Diluted to 5.6 M Na and with Recycle [6]		Glass Oxides	Simulant as Oxides (wt%)	Source in Simulant	Order for Addition	Formula Weight	Assay*	Target Weight (g)
-	mg/L	mg/L	Molarity	Loading	100%	In 623 ml water add following compounds in the order listed below				
Al	15100	9818	0.364	Al ₂ O ₃	8.92	Al(NO ₃) ₃ .9H ₂ O, 60% sol.	1	375.14	0.607	224.94
Ca	81	53	0.001	CaO	0.04	Ca(NO ₃) ₂ .4H ₂ O	2	61.83	0.998	0.31
Cr	800	520	0.010	Cr ₂ O ₃	0.37	Na ₂ CrO ₄ .4H ₂ O	8	234.04	0.995	2.35
Fe	37	24	0.0004	Fe ₂ O ₃	0.02	Fe(NO ₃) ₃ .9H ₂ O	9	404.01	0.999	0.18
K	4960	3225	0.082	K ₂ O	1.87	KOH	7	56.10	0.908	5.10
Na	198000	129259	5.622	Na ₂ O	83.76	NaOH, 50% sol. d=1.53	6	40.00	0.501	171.91
Ni	36	23	0.0004	NiO	0.01	Ni(OH) ₂	3	92.72	1.000	0.04
Pb	31	20	0.0001	PbO	0.01	PbO	4	223.20	1.000	0.02
Si	49	32	0.001	SiO ₂	0.03	SiO ₂	5	60.09	0.990	0.07
Cl	4050	4206	0.119	Cl	2.02	NaCl	11	58.45	0.994	6.98
F	629	412	0.022	F	0.20	NaF	12	42.00	0.999	0.91
PO ₄	2660	1730	0.018	P ₂ O ₅	0.62	Na ₃ PO ₄ .12H ₂ O	10	380.12	0.999	6.93
SO ₄	8010	5338	0.056	SO ₃	2.14	Na ₂ SO ₄	13	142.06	0.998	7.91
NO ₂	78600	5107	1.111	-	-	NaNO ₂	17	69.00	0.995	77.05
NO ₃	184000	119641	1.930	-	-	NaNO ₃	18	84.99	0.990	71.60
CO ₃	50100	32576	0.543	-	-	Na ₂ CO ₃	19	105.99	1.000	57.55
Org. Carbon	2820	1834	0.153	-	-	-	-	-	-	-
Acetate ^s	3793	2466	0.042	-	-	Sodium Acetate (C2)	14	136.08	0.999	5.69
Formate ^s	3793	2466	0.055	-	-	Sodium Formate (C1)	15	68.01	0.999	3.68
Oxalate	984	640	0.007	-	-	Sodium Oxalate (C2)	16	134.00	0.990	0.98
-	-	-	-	SUM	100.0	Total simulant Weight				1267.2

- Empty data field.

^s Equal amounts (mg/L) of acetate and formate are added to meet the TOC content.

* Assay refers to the purity of the raw material as specified by the vendor.

Table 5.2. Glass Former Additives for 1 Liter of LAW AP-107 Waste Simulant at 5.6 M Sodium and Corresponding Feed Properties.

Glass Forming Chemical Additive Source	Feed AP107WDFL
Additives in Glass (wt%)	79.46%
Kyanite (Al_2SiO_5) 325 Mesh (Kyanite Mining) (g)	70.42
H_3BO_3 (US Borax – Technical Granular) (g) – <i>Added at VSL</i>	179.89
Wollastonite NYAD 325 Mesh (NYCO Minerals) (g)	87.57
Fe_2O_3 (99.6% Alfa) (g)	53.92
Li_2CO_3 (Chemetall Foote Co. Technical grade) – <i>Added at VSL</i>	22.56
Olivine (Mg_2SiO_4) 325 Mesh (#180 Unimin) (g)	30.03
SiO_2 (Sil-co-Sil 75 US Silica)) (g)	359.63
TiO_2 (Rutile Airfloated Chemaloy) (g)	15.02
ZnO (KADOX – 920 Zinc Corp. of America) (g)	35.45
Zircon ZrSiO_4 (Flour) Mesh 325 (AM. Mineral) (g)	45.62
Sucrose as Reductant (nominal) (g)	60.64
Simulant Weight for 1 liter (g)	1267
Sum of Additives (g)	900
Sum of Complete Batch (g)	2167
Estimated Final Volume (l)	1.5
Estimated Density (g/ml)	1.5
Expected Glass Produced (g) ; i.e., Glass Yield (g/l of simulant)	1013
Estimated Weight % Water in Slurry Feed	50%
Estimated Weight % Additives in Slurry	41%
Estimated Glass Yield (g/kg of Feed)	466
Estimated Glass Yield (g/l of Feed)	694
Estimated Total Solids (g/l of Feed)	745
Additives (g/l of Feed)	617

- Empty data field

Note: All raw material quantities are based on typical assays; adjustments were made based on assays of available materials.

Table 5.3. Summary of DM10 Test Conditions and Results.

Feed Start	4/18/18 11:00
Feed End	4/26/18 18:45
Interval	199.75 hr
Target Glass	AP107WDFL
Mass Poured	345 kg
Average Glass Production Rate	1974 kg/m ² /day
Divalent / total iron	4.8 %
Mass Fed (including recycle)	795.4 kg
Target glass Conversion	0.441 kg/kg
Average Slurry Feed Rate	3.98 kg/hr
Proportion of Recycled Solutions in Melter feed	14.4 kg/ 47.866 kg feed
Average Glass Production Rate	2007 kg/m ² /day
Average Recycle Feed Rate	1.2 kg/hr
Average Solution Flow into the Evaporator	11.5 kg/hr

Table 5.4. Measured Volatile Constituents (SW-846-8260B) in Evaporator Concentrate and Condensate Solutions (µg/L).

Constituent	RL	Concentrate	RL	Condensate
Acetone	2000	ND	100	71.2 J
Acetonitrile	4000	ND	200	4450
Benzene	200	ND	10	ND
Bromodichloromethane	200	ND	10	ND
2-Butanone (MEK)	1000	ND	50	ND
Carbon Disulfide	200	ND	10	ND
Carbon Tetrachloride	200	ND	10	ND
Chlorodibromomethane	200	ND	10	ND
Chloroform	200	ND	10	ND
Methylene chloride	400	19.0 J, B	10	1.86 J, B
4-Methyl-2-pentanone (MIBK)	1000	ND	50	ND
Tetrachloroethene	200	ND	10	ND
Tetrahydrofuran	800	164 J, B	40	ND
1,1,1-Trichloroethane	200	ND	10	ND

RL – Reporting Limit

E – Estimated result. Result concentration exceeds calibration range.

B - Method blank contamination. The associated method blank contains the target analyte at a reportable level.

J – Estimated result. Result is less than RL (reporting limit).

ND – Not Detectable

Table 5.5. Measured Constituents in Evaporator Solutions with Values above Detectable Levels which were not Previously Evaluated.

-	Analytical Method	Reporting limit	Concentrate	Reporting limit	Condensate	Units
Acrylonitrile	SW-846-8260B	4000	ND	200	203	µg/L
Bromomethane	SW-846-8260B	400	ND	20	2.2 J	µg/L
n-Butylbenzene	SW-846-8260B	200	ND	10	0.46 J	µg/L
1,2,3-Trichlorobenzene	SW-846-8260B	200	ND	10	1.6 J	µg/L
1,2,4-Trichlorobenzene	SW-846-8260B	200	ND	10	1.1 J, B	µg/L
No values above detection limit for any of the new analytes	SW-846-8270C	-	-	-	-	-
Diethylene glycol	SW-846-8015B	1000	ND	1000	ND	mg/L
Ethanol	SW-846-8015B	100	ND	100	ND	mg/L

E – Estimated result. Result concentration exceeds calibration range.

B - Method blank contamination. The associated method blank contains the target analyte at a reportable level.

J – Estimated result. Result is less than RL (reporting limit).

ND – Not Detectable

– Empty data field

Table 6.1 Summary of DM10 Iodine Test Results.

	Test	CH ₃ I
Time	Feed Start	8/26/19 9:30
	Feed End	8/27/19 21:00
	Interval (hr)	35.5
Iodine Fed (g)		85.0
Waste Simulant		LAW AP-107
Target Glass		AP107WDFL
Glass Discharged		62.1 kg
SBS and WESP Sump and Blow-Downs	Total Amount	195 kg
	Measured Acetonitrile Concentration	4.63 mg/l
Simulated Caustic Scrubber	Measured Acetonitrile Capture Rate	1.0 mg/min
	Total Acetonitrile Captured over the Test	2.249 g

Table 7.1. Summary of Acetonitrile Generation and Capture in DM10 Tests with Recycle System.

Glass	LAWE10H	LAWE6H + Ferric Oxalate	LAWE4H + Ferric Oxalate	LAWE4H	WLDF1	WLDF2	AP107WDFL
Moles NO _x per liter simulant	0.641	3.712	3.905	3.905	2.8605	3.0965	3.041
Sugar (g) per kg glass	8.57	54.38	67.32	67.32	63.38	44.70	59.86
Carbon (g) per kg glass	4.03	46.28	48.45	30.14	29.94	21.11	27.02
kg glass per test	153.5	136.2	142.8	135.1	119	128.5	345
Test Duration (hr)	71.3	72	72.3	72	72	72	199.75
Flow into the evaporator (kg/hr)	11.8	12.2	12.6	11.3	12.3	10.4	11.5
Concentrate Flow (kg/hr)	0.41	0.4	0.4	0.4	0.41	0.42	1.2
Acetonitrile in Concentrate (mg/l)	0	0.22	0.4	0.397	0.396	0.273	0
Acetonitrile in Condensate (mg/l)	0.74	5.8	10	10.5	9.09	7.18	4.45
Total Sugar (kg)	1.315	7.406	9.614	9.095	7.543	5.744	20.652
Total Carbon (kg)	0.619	6.303	6.919	4.072	3.562	2.713	9.321
Total Concentrate (kg)	31.1	30.5	30.6	30.7	31.3	32.5	245.3
Total Condensate (kg)	863.9	900.9	932.9	837.3	907.2	772.5	2105.9
Total Acetonitrile in Concentrate (g)	0.00	0.01	0.01	0.01	0.01	0.01	0.00
Total Acetonitrile in Condensate (g)	0.64	5.23	9.33	8.79	8.25	5.55	9.37
Acetonitrile in Concentrate (g/kg sugar)	0	0.001	0.001	0.001	0.002	0.002	0
Acetonitrile in Condensate (g/kg sugar)	0.486	0.705	0.970	0.967	1.093	0.966	0.454
Acetonitrile in Concentrate (g/kg carbon)	0	0.001	0.002	0.003	0.003	0.003	0
Acetonitrile in Condensate (g/kg carbon)	1.033	0.829	1.348	2.159	2.315	2.044	1.005
Total Acetonitrile (g/kg sugar)	0.486	0.706	0.972	0.968	1.095	0.967	0.454
Total Acetonitrile (g/kg carbon)	1.033	0.830	1.350	2.162	2.318	2.048	1.005

Note: Calculation above includes the estimated amount acetonitrile in the liquid in the SBS sump at the end of each test, had that been fed to the evaporator.

**Table 7.2. Summary of Acetonitrile Generation and Capture in Tests without Recycle
(Calculated from the Analysis of SBS and WESP Solutions).**

Test	Location	Blow-Down Volume	Acetonitrile			
			mg/L	Total g	g/kg sugar	g/kg carbon
DM1200 LAWE4H + Ferric Oxalate	SBS	1197 gal	83	384.9	1.030	1.431
	WESP	140.5 gal	67	36.5	0.098	0.136
	Total	-	-	421.3	1.127	1.567
DM10 AP107WDFL + Methyl iodide	Total (SBS + WESP)	194.78 kg	4.63	0.902	0.243	0.537