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Low-Level Waste Phase 1 Melter Testing Offgas and Mass Balance Evaluation

C. N. Wilson

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Abstract: Commercially available melter technologies were tested during 1994-95 as part of a multiphase program to test candidate technologies for vitrification of the low-level waste (LLW) stream to be derived from retrieval and pretreatment of Hanford Site tank wastes. Seven vendors were selected for Phase 1 testing to demonstrate vitrification of a high sodium content liquid LLW simulant. The tested melter technologies included four Joule-heated melters, a carbon electrode melter, a combustion melter, and a plasma melter. Various dry and slurry melter feed preparation processes were also tested. Various feed material samples, product glass samples, and process offgas streams were characterized to provide data for evaluation of process decontamination factors and material mass balances for each vitrification technology. This report describes the melter mass balance evaluations and results for six of the Phase 1 LLW melter vendor demonstration tests.

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**LOW-LEVEL WASTE PHASE 1 MELTER TESTING
OFFGAS AND MASS BALANCE EVALUATION**

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June 1996

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LOW-LEVEL WASTE PHASE 1 MELTER TESTING OFFGAS AND MASS BALANCE EVALUATION

1.0 SUMMARY

A multiphase program was initiated in 1994 to test commercially available melter technologies for vitrification of the low-level waste (LLW) stream to be derived from retrieval and pretreatment of defense wastes stored in underground tanks at the Hanford Site. Testing was conducted at vendor facilities using a nonradioactive chemical simulant representing the composition of double-shell slurry feed tank waste. The double-shell slurry feed tank waste is expected to be a major early feed material to an LLW vitrification facility. Seven vendors were contracted for testing and six vendors were successful in obtaining data. The six melter configurations from which offgas and mass balance data were obtained included a gas-fired cyclone combustion melter, a plasma torch-fired cupola furnace, a carbon electrode melter, two high-temperature molybdenum electrode Joule-heated melters, and a low-temperature Inconel^{*} electrode Joule-heated melter. In addition, each vendor was responsible for the approach to feed preparation before melting. The focus of this report is to review the data and test reports from each run and summarize calculated results for each melter system. The results summarized in this report include the following areas:

- Nonselective loss of feed (assumed to be gross entrainment)
- Selective loss of feed components (assumed to be volatilization)
- NO_x generation from feed nitrates and nitrites.

Gross entrainment was severe (8.7%) and significant (2.7%) for the combustion melter and the plasma torch furnace, respectively. One Joule-heated melter had very low entrainment (0.05%) using a dried feed that was rewet before feeding to reduce dusting. Entrainment in other systems ranged from 0.6% and 1.2%.

Volatile losses of halogens, alkali metals, boron, molybdenum (⁹⁹Tc surrogate), phosphate, and sulfate were severe for the non-Joule-heated systems. Volatile losses from Joule-heated systems were less severe but still significant. One Joule-heated system obtained low volatile losses using a full coverage dry feed batch blanket. This melter also demonstrated the significant result of only 10% volatile loss of I. The second best melter system for I retention lost 82% of the I to volatility.

The molar yield of NO_x to the offgas system (including feed preparation and melter offgas) ranged from 2.6% to 68% of the molar NO₃⁻/NO₂⁻ content in the feed. The best performance was obtained using sucrose addition to the feed before slurry feeding of a Joule-heated melter. Other methods demonstrated for NO_x reduction include the addition of powdered sugar and activated carbon to the feed followed by drying and pre-reacting the melter

*Inconel is a trademark of INCO Alloys International, Inc.

feeding (7.4% NO_x yield during feed preparation) and urea addition to the feed before slurry feeding (13% NO_x yield during melting). Considering the melter unit independently from feed preparation processes, the destruction of residual feed NO_x/NO_2 within the carbon electrode furnace resulted in a NO_x yield of only 0.03%. Ammonia emissions were also summarized where data were available.

2.0 BACKGROUND

Following negotiation of the fourth amendment to the Tri-Party Agreement (Ecology et. al. 1994), vitrification became the planned treatment method for the LLW streams derived from retrieval and pretreatment of double-shell tank and single-shell tank wastes at the Hanford Site. A multiphase program was initiated in 1994 to test and evaluate commercially available promising melter technologies using nonradioactive LLW simulants. Seven vendors were selected to participate in Phase 1 testing. A detailed discussion of Phase 1 testing activities and summary results is provided in WHC-SD-WM-ER-498 (Wilson 1996a). The seven Phase 1 vendors and their respective vitrification technologies are as follows.

- Babcock & Wilcox (B&W), Alliance Research Center, Alliance, Ohio--Gas-fired cyclone combustion melter, slurry feed
- Envitco, Inc. (Envitco), Toledo, Ohio--High-temperature Joule-heated melter, molybdenum electrodes, wetted pelletized feed
- GTS Duratek Inc. (Duratek), Columbia, Maryland--Low-temperature Joule-heated melter, Inconel electrodes, slurry feed
- Penberthy Electromelt International Inc., Seattle, Washington--High-temperature Joule-heated melter, sidewall molybdenum electrodes, moist mix-in-the-charger feed
- U.S. Bureau of Mines (USBM), Albany Research Center, Albany Oregon--Carbon electrode melter, prereacted dry feed
- Vectra Technologies Inc. (Vectra), Richland, Washington--High-temperature Joule-heated melter, top-entry molybdenum electrodes, calcined, dried and slurry feed
- Westinghouse Science and Technology Center (WSTC), Pittsburgh, Pennsylvania--Plasma torch-fired cupola furnace, slurry feed.

Phase 1 LLW melter demonstration tests were completed by six of these vendors (testing at Penberthy Electromelt International, Inc. was terminated before completion) from September 1994 through May 1995. A variety of samples were taken during testing by each vendor to address issues defined in WHC-SD-WM-RD-044 (Wilson 1994). Sample types included feed materials, batched melter feed, glass samples, offgas scrub and blowdown solutions, and miscellaneous deposits and residues. Analyses on these samples were performed by contracted independent and government laboratories as follows:

- Quanterra Laboratories, St. Louis, Missouri--Liquid samples including LLW simulant, offgas scrub solutions; and blowdown solutions, chemical analyses, specific gravity, and settled solids

- Corning Laboratory Services, Corning, New York--Solid samples, feed materials, glass, miscellaneous deposits and residues; chemical analyses, microstructural analyses, phase characterization, and glass redox
- U.S. Geological Survey, Denver, Colorado--Solid samples, feed materials, glass, miscellaneous deposits and residues; chemical analyses, microstructural analyses, phase characterization, and glass redox
- Westinghouse Hanford Company (WHC) 222-S Laboratory--Liquid/slurry samples; specific gravity, and settled and centrifuged solids content
- Pacific Northwest National Laboratory--Radioactive samples (from Duratek DuraMelter-100^{*} [DM-100] test), duplicate analyses of selected samples analyzed at other laboratories, and special and/or expedited analyses.

Results from analyses were sent to WHC for review, distribution, and integration into an LLW melter testing electronic database (Mast 1995). In addition to these test sample analyses, each vendor contracted with a qualified air quality laboratory to perform specific offgas emissions measurements using standard U.S. Environmental Protection Agency (EPA) methods. These measurements included isokinetic sampling by Methods 5 and/or 29 for multiple metals, Method 26A for determination of hydrogen halides and halogens, and continuous emissions monitoring for NO_x, CO_x, and SO₂. The air quality laboratory emissions measurement data for each test were reported in each vendor's Phase 1 test reports. These data were reviewed and used as appropriate to perform the presently reported offgas evaluation for each of the six completed Phase 1 vendor LLW melter demonstration tests.

The purpose of this evaluation is to estimate the partitioning of feed components to the offgas system and to estimate the extent to which NO₃⁻ and NO₂ in the feed are converted to NO_x in the offgas. The partitioning information will provide data needed to design offgas treatment systems. The data will also be used to assess the practicality of offgas recycle and to predict the characteristics of potential recycle streams and secondary waste streams resulting from offgas treatment.

^{*}DuraMelter is a trademark of GTS Duratek, Inc.

3.0 OFFGAS AND MASS BALANCE EVALUATION

A review was conducted of the vendor test reports (Hendrickson 1995, Eaton 1995a and 1995b, Wilson 1996b, Higley 1995, and Stegen 1996) from each Phase 1 vendor test. The information generated, along with data from the WHC test sample analyses database (Mast 1995), was evaluated to estimate the losses due to selective and non-selective mechanisms, assumed to be volatility and entrainment, respectively. These values are combined to provide the total partitioning of elements to the offgas system. In the case of the Vectra test, where some data from dry feeding are reported, primarily data from the slurry feeding is evaluated because this is Vectra's recommended approach. In the case of Envitco, only the dry feed approach is evaluated due to the absence of offgas data and sample analyses results for the slurry feed test segment. The Duratek evaluation is based on the larger DuraMelter-1000 (DM-1000) melter test.

3.1 MASS BALANCE EVALUATION METHODOLOGY

The total loss from the melter is determined from estimates of the entrainment (non-selective loss) and volatility (selective loss). The conceptual model of the process is that the gross entrainment occurs first and equally entrains all components. The volatility then occurs from the remainder. The percent volatilized in this conceptual model can be determined based only on glass and feed composition with no knowledge of the degree of gross entrainment. The equation used to recombine the volatility and entrainment values to determine the fraction of each component partitioning to the offgas is shown below.

$$L_T = L_e + L_v \left[1 - \frac{L_e}{100} \right]$$

where:

- L_T = Percent total loss of component to offgas
- L_v = Percent volatile loss of component
- L_e = Percent non-selective entrainment of feed material.

Entrainment loss values are based primarily on aerosol sampling using EPA-approved Methods 5 or 29 for components assumed to be non-volatile, but may also include data for material that collects in downstream offgas equipment. Calculational methods used for determining volatility include a 'tie component' calculation based on glass and feed composition to determine selective losses, and/or determination of the mass of volatilized components based on offgas measurements. When selective losses are much greater than the probable errors in the glass and feed analyses, the tie component method is generally more reliable than estimates based on offgas measurements. When the

selective loss is low relative to probable errors in the glass and feed analyses, volatility estimated based on offgas measurements is generally more accurate.

In the tie component method, a component that is known to have little or no volatility at the melter conditions is selected as an internal standard. In addition to being non-volatile, the component should be present in sufficient quantity to allow accurate analytical determination and should not be subject to contamination from non-feed sources such as refractory or electrode corrosion or other sources. Typical tie components selected for these tests include SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , and SrO with the selection depending on the details of the run. The concentration of the volatile component is compared to the tie component in the feed and glass. The equations are shown below.

$$\text{fraction of component volatilized} = \frac{v_f \frac{t_g}{t_f} - v_g}{v_f \frac{t_g}{t_f}}$$

where:

v_f = Mass fraction volatile component in feed
 v_g = Mass fraction volatile component in glass
 t_f = Mass fraction tie component in feed
 t_g = Mass fraction tie component in glass.

Advantages of the tie component method are that it relies only on compositional analysis and does not require accurate measurement of masses of feed and glass materials. Only the concentration ratios of the components of interest to the concentration of the tie component are important, and the accuracy of the results is not affected by loss of volatile components such as water, nitrates, or carbonates from the feed. In addition, non-selective loss mechanisms such as accumulation, feed spillage, and gross entrainment of the feed, do not affect the results. However, if the melter is not at compositional steady state due to changes in feed composition, errors can be introduced. Also, if there is a source of a component other than from the feed, such as refractory or electrode corrosion, then the selective loss results for the component will smaller than expected or even a negative value.

3.2 ESTIMATES OF GROSS ENTRAINMENT OF FEED

Entrainment refers to material that is physically entrained in the offgas. Entrainment may result from dusting of a dry feed, boiling of wet feed in a cold cap, or a failure of feed to completely segregate from gases for melters such as those at B&W or WSTC. It is assumed that the loss by entrainment is non-selective and equally entrains all components. Selective

entrainment is possible, and if present it is included in this evaluation as volatility. Table 3-1 provides the best estimates of the rate of entrainment. These values are based primarily on aerosol sampling using EPA-approved Methods 5 or 29, but may also examine material that collects in downstream offgas equipment. Table 3-1 is organized roughly in the order of decreasing entrainment and volatilization results as observed during the Phase 1 melter vendor testing. In general, the Joule-heated melters show significantly less entrainment than other types of melters.

3.2.1 B&W

The entrainment for the B&W run was the most severe of any melter system. The entrainment value for B&W was determined based on Method 5 sample analyses along with feed sample data and using the average of results obtained using Al_2O_3 and CaO. There is significant uncertainty in this value due to uncertainty in the feed oxide flow rate and glass production. The value for oxide content of the feed was based on the loss on ignition and loss on drying analytical results (46.3%). This value differs significantly from the value used by B&W (56.47%). Also, the value may be biased low due to losses in the convective pass section before the Method 5 sample point. Additional detail is provided in Appendix A.

3.2.2 WSTC

The WSTC entrainment value of 2.7% is arrived at by taking the difference between the mass of glass poured and the mass of glass expected. It is assumed that the presence of CaO or Al_2O_3 in the offgas samples is due to entrainment only and the mass fraction of CaO and Al_2O_3 in the Method 5 aerosol samples is representative of the composition of the total carryover. This approach results in estimates of 2.1% and 3.3% based on Al_2O_3 and CaO content, respectively, which averages to 2.7%. The WSTC test report (Hendrickson 1995) contained a variety of conflicting values for entrainment including 0.2% (p. 2-10), 0.5% (p. 8-31), 0.7% (p. 8-28), 2.0% (p. 7-18), and

Table 3-1. Percent Gross Entrainment Estimates.

B&W	WSTC	USBM WHC1	USBM WHC2	USBM WHC3	Vectra	Duratek	Envitco
8.7	2.7	1.2	N/A	N/A	0.6	0.6	0.06

B&W = Babcock & Wilcox
 Duratek = GTS Duratek, Inc.
 Envitco = Envitco, Inc.
 USBM = U.S. Bureau of Mines
 Vectra = Vectra Technologies, Inc.
 WHC = Westinghouse Hanford Company
 WSTC = Westinghouse Science and Technology Center

2.2% (p. 7-14). It is believed that the 2.7% entrainment value is the best available estimate of gross entrainment. Additional detail is provided in Appendix B.

3.2.3 USBM

The USBM conducted three test runs designated WHC1, WHC2, and WHC3. Modifications were made to the melter and operating procedures for runs WHC2 and WHC3 aimed at reducing volatile component losses. The 1.2% entrainment estimate for the USBM WHC1 test is based on the mass of SiO_2 collected in downstream offgas processing units. An overall mass balance approach suggests a slightly higher value of 1.7%, but the 1.2% figure is believed to be more accurate. Method 5 samples conducted at the furnace outlet generally indicated lower entrainment values of 0.11% to 0.47% based on Al and Ca results. However, these results are discounted in favor of the evaluation of silica located in the offgas system due to the difficulties experienced in the Method 5 samples such as plugged sampling lines and nozzles, constriction, and partial plugging at the melter exit which disturbed sampling conditions. Insufficient data were collected during the USBM WHC2 and USBM WHC3 tests to allow estimation of entrainment losses for these tests. In the absence of other data, it is suggested that the USBM WHC1-based entrainment value be used. However, modifications made to reduce volatile component losses in runs USBM WHC2 and USBM WHC3 also may have resulted in reduced entrainment losses. Additional data are provided in Appendixes C and D.

3.2.4 Vectra

The value of 0.6% entrainment for the Vectra MT-2 slurry feed test segment is determined based on Method 5 samples and on the glass and feed compositions to estimate total volatile losses. Total mass loss is estimated at 2.4% with 0.6 wt% being by gross entrainment and 1.8% by volatility. The Method 5 data were sufficiently variable that the 0.6 wt% entrainment estimate should be considered a fairly rough estimate. Entrainment data also were available from aerosol samples during the MT-3 run segment when the melter was fed dry batched material formulated to simulate a calcined feed composition, and during the MT-4 run segment when fluid bed calcined feed was run. The entrainment during MT-3 was similar to MT-2 giving an entrainment rate of 0.4% while the entrainment rate during MT-4 was only 0.07%. Additional detail is provided in Appendix E.

3.2.5 Duratek

The Duratek entrainment value of 0.6% was obtained based on an analysis of Method 5 samples for Sr. The Method 5 sampling data provided by the Vitreous State Laboratory of Catholic University of America (VSL-CUA) were used rather than data from Parsons Engineering Science, Inc. (PES), which was contracted to perform offgas measurements. The PES data were systematically lower which makes the VSL-CUA data more conservative. The more conservative value was selected because the sampling conditions were far from optimum in

terms of flow conditions and flow quantification. The entrainment value estimate based on the PES data was 0.1%. Additional detail is provided in Appendix F.

3.2.6 Envitco

Entrainment calculated for the Envitco dry feed test was very low. The 0.05% value was calculated based on the amount of Al detected in the Method 29 samples. A comparison also was made to the total amount of material collected on the Method 29 sample. Additional information is provided in Appendix G.

3.3 ESTIMATES OF VOLATILE LOSSES OF FEED COMPONENTS

Selective loss of components through volatilization is a somewhat greater concern than gross entrainment for the LLW vitrification systems. Volatility estimates were prepared primarily using the previously described tie component method. The tie components typically used were Al_2O_3 , SiO_2 , SrO , CaO , and Fe_2O_3 . In some cases, one or more tie components could not be used because of analytical problems, or because of contamination of the tie component by other sources such as refractory corrosion. In some cases, Method 5 (or 29) offgas sampling results have been used in place of the tie component analysis because the results are believed to be more accurate.

The volatility estimate results are shown in Table 3-2. Shaded values indicate the components of concern for volatility and for which total loss estimates are later provided in Table 3-3. Negative values imply that more material was present in the glass than in the feed material. This may occur because of analytical errors, or from contamination of the glass from refractories or other sources. Component selective loss calculations for each vendor test are contained in Appendixes A through G. All I loss calculations are contained in Appendix H.2.

3.3.1 B&W

In general, the B&W test showed the greatest volatile component selective losses of the melter systems tested. The volatilities were estimated using the tie component method with SrO , SiO_2 , and CaO as the tie components. The values provided in Table 3-2 are the average of the three tie calculation results. The tie component calculations used analytical data from mass balance test sample sets* 1, 2, 3, and 5.

All measurements of SO_2 in B&W glass samples were by USGS and were below detection limits. Corning did not analyze for SO_2 in samples from the initial melter tests. Therefore, the estimate provided for SO_2 volatility is a

*Mass balance sample sets included simultaneous samples from the melter feed, glass, and offgas streams.

Table 3-2. Weight Percent Selective Loss of Feed Components. (2 sheets)

Component	B&W	WSTC	USBM WHC1	USBM WHC2	USBM WHC3	Vectra	Duratek	Envitco
Al_2O_3	-36 ^a	-2.5 ^a	-5.4 ^a	-3 ^a	0.49	~0 ^b	-0.1	0 ^b
B_2O_3	67	22	51	23	18	14	~0 ^b	0.14 ^b
BaO	-51 ^c	5.8	12 ^c	-214 ^c	-243 ^c	N/A	14.9	-61
CaO	-6.5	2.2	-1.5	-5.7	-1.6	2.0	4.9	14
Cl	87	88	82	N/A	97	64	47.8	1 to 13 ^b
Cr_2O_3	-76 ^a	12	-32 ^a	-80 ^a	-64 ^a	-683 ^a	0.6 ^b	0 ^b
Cs_2O	83	84	63	N/A	39	41	13.2	0.6 ^b
F ^d	~92	~91	~91	N/A	~99.7	~15 ^b	~53	<0.85 ^b
Fe_2O_3	-86 ^a	-32	-4.5	2.9	-1.6	0.3	-6.1	0 ^b
I	94	>98	95	N/A	N/A	83	82	10
K_2O	51	48	35	-143	25	15	0 ^b	0 ^b
LiO_2	N/A	2.8	N/A	N/A	N/A	N/A	N/A	N/A
MgO	-29 ^a	-13	-25	-46	-74	-2.3	-12.7	2.9
MnO_2	-18 ^c	22 ^c	13 ^c	-686 ^c	-613 ^c	-563 ^c	22.9 ^c	0.13 ^b
MoO_3	60	24	47	50	45	-49	0.2 ^b	0 ^b
Na_2O	35	15	21	20	6.5	13	~0 ^b	0 ^b
NiO	-453 ^c	70	73	N/A	N/A	N/A	-630	N/A
P	-41 ^a	43	41	-42	54	1.1 ^b	N/A ^f	0 ^b
S	>51	34	88	-303 ^e	94	85	N/A ^f	53
SiO_2	-0.3	11	0	13	-0.5	~0 ^b	0	0.8
SrO	10	-0.1	4.0	-4.2	N/A	~0 ^b	0 ^b	0.10 ^b
TiO_2	-36 ^a	200 ^c	-4.7	5.8	-104 ^c	N/A	6.6	-7 ^c
ZrO_2	14	-0.6	-50 ^c	-88 ^c	-621 ^c	N/A	7.5	-422 ^b

Table 3-2. Weight Percent Selective Loss of Feed Components. (2 sheets)

NOTE: Shaded values indicate components likely to be affected by volatility.

^aNegative values likely due to refractory corrosion increasing component content in the glass.

^bValues based on offgas aerosol data, all other values based on tie component method.

^cLow feed concentration such that differences in detection limits between feed and glass, analytical errors, and/or minor contamination results in large errors.

^dDifficulty was experienced in analyzing melter feeds for fluoride and feed targets, fluoride concentrations were used to determine all fluoride volatility results. Uncertainty also may exist with respect to fluoride glass analysis. Therefore, all fluoride results are considered approximate. See Appendix I.

Additional detail is provided in text.

B&W = Babcock & Wilcox
Duratek = Duratek Inc.
Envitco = Envitco, Inc.
N/A = Not available
USBM = U.S. Bureau of Mines
Vectra = Vectra Technologies, Inc.
WHC = Westinghouse Hanford Company
WSTC = Westinghouse Science and Technology Center

minimum value based on the USGS detection limit in glass. In sample analysis for subsequent melter tests, Corning analyzed for SO_3 and achieved much better detection limits.

The P_2O_5 volatility determination was complicated by the feed analysis being consistently below the target values and a source of P_2O_5 from refractory corrosion. The result provided is based on assuming the feed contained the target P concentration and on the highest obtained loss result corresponding to a period of lower refractory corrosion.

The F volatility estimate contains significant uncertainty. Analysis for F in feed samples was problematic. Therefore, feed target values for fluoride were used in place of feed analysis data. Corning glass results were systematically lower than USGS results. The USGS results were used to arrive at the given F volatility value of 92%. Volatile losses calculated for F would be higher if the Corning data were used in place of the USGS data. Additional discussion of the fluoride analytical problems is provided in Appendix I.

The I volatility was taken as the average of four data points where USGS neutron activation analysis results were available for corresponding feed and glass samples. The individual results ranged from an 88% loss to a 99% loss of I.

Alumina, Cr_2O_3 , Fe_2O_3 , MgO and TiO_2 have negative calculated volatilities most likely because the glass content of these components was increased as a result of refractory and metal corrosion. Additional information is provided in Appendix A.

3.3.2 WSTC

The WSTC data indicate volatility that is lower than the B&W system for several key components including B_2O_3 and Na_2O . Results were determined based on mass balance sample sets 1, 2, 3, and 4 using tie components Ca and Al. For SO_3 , the analysis from sample set 3 was discarded due to suspected analytical error to arrive at the value provided. The measured glass Fe content is well within detection limits and indicates an unidentified source of Fe contamination exists increasing the glass content relative to the feed. The reason for the indicated 11% SiO_2 volatility is uncertain. It is not expected that SiO_2 has any significant volatility. This may be the result of the unsteady mix ratio between waste simulant and glass formers. However, analytical or sampling errors and contamination of the glass with additional Al and Ca as a result of refractory corrosion also may have contributed to the calculated SiO_2 volatility. Additional information is provided in Appendix B.

Iodine volatility was determined based on neutron activation analysis by USGS of one feed sample and one glass sample. The I in the glass sample was below detection limits and the value provided was calculated using the analytical 'less than' result.

3.3.3 USBM

Data from the initial USBM WHC1 test indicate very significant volatility of key components such as B_2O_3 (51%), Na_2O (21%) and MoO_3 (47%). However, the volatility evaluation was complicated from contamination from 'Tree Hugger' feed material used at the beginning of the USBM WHC1 run. Results were based on an SiO_2 tie component using average feed and glass concentrations after correcting SiO_2 , CaO, Al_2O_3 , and TiO_2 for the apparent contamination. The identically zero result for the SiO_2 is a direct result of the selection of SiO_2 as a tie component. Negative volatilities calculated for some components may be related to the addition of Tree Hugger feed components to the glass. Additional information for the USBM WHC1 run is provided in Appendix C.

Calculation of selective component losses by the tie component method for the USBM tests also was complicated by the occasional addition of lime to the melter as a fluxing agent when the glass became too viscous to drain, and by contamination from steel punchings used for starting the furnace in later runs. The volatilities were obtained by first adjusting the glass composition to remove excess Ca and Fe based on the silica and alumina concentrations. The results were taken as the mid-range value of the calculated results using feed analysis and feed targets with SiO_2 and Al_2O_3 tie components.

*Feed material previously melted by USBM for the Tree Hugger Corporation is defined in WHC-SD-WM-VI-030, p. 3-4 (Eaton 1995a).

The 95% I volatility result was determined using USGS neutron activation analysis results for six samples of dried and reacted feed and seven glass samples. The feed samples were used to estimate an average feed composition and loss estimates were made based on each glass sample. Loss estimates ranged from 91% to 99%.

As a result of the high component volatilities observed in the USBM WHC1 run, modifications were made to the melter and in the operating conditions intended to reduce volatility. The USBM reduced the voltage and power input for run USBM WHC2, and based on favorable indications, further decreased the power density on the electrodes for USBM WHC3 by increasing the electrode diameter. Significantly lower volatilities for Na_2O , Cs_2O , and B_2O_3 were achieved during these later runs. Selective loss calculations for USBM runs WHC2 and WHC3 are provided in Appendix D.

The fluoride loss is based on feed target values rather than analytical feed values. However, because of the large loss, the impact of this assumption is only to increase the fluoride loss to 99.7% compared to 99.5% if feed analysis values are used.

Interestingly, while the losses of alkali metals and B were significantly reduced in the USBM WHC3 run, the volatile losses of halogens are consistently greater in the revised operating conditions. The trend is believed to be real although the mechanistic explanation is unknown. Activation analysis data were unavailable to estimate I losses for the USBM WHC2 and USBM WHC3 runs.

3.3.4 Vectra

The Vectra results indicated volatility somewhat similar to the USBM-3 except that halogen losses were much lower. All results in Table 3-2 are evaluated for the slurry feeding method (run MT-2) which was the recommended feed method. Values for Cs_2O , K_2O , F, Cl, I, SO_3 , and P_2O_5 were obtained based on a SrO tie component. This was done because these minor components (plus SrO) were not in the startup glass and may not have reached steady-state concentrations by the time the slurry feed MT-2 run mass balance period was started. The remaining results are the average of results obtained using CaO , Al_2O_3 , Fe_2O_3 , and SiO_2 as the tie components.

The results obtained based on Method 5 offgas analyses are adjusted assuming that gross entrainment is 0.6%. In cases where the Method 5 initial result was <0.6%, the result is expressed as ~0. Aerosol sampling results are believed to be biased low due to line losses between the melter and the sample point. However, for some analytes, the aerosol samples provide improved data over the tie component result. The fluoride result was taken from aerosol sample data due to analytical difficulties experienced in the glass for fluoride. The P_2O_5 data were taken from aerosol samples due to uncertainty about the actual feed P concentrations. The tie component calculation indicates an excess of P_2O_5 in the glass compared to feed target values. The

*Based on glass sample analyses performed by USBM for runs in WHC2 and WHC3.

single feed sample that was analyzed for P indicated an excess of P_2O_5 in the feed. The aerosol-based result is less sensitive to the feed concentration uncertainty. There was a significant discrepancy between the aerosol result and the tie component result for Na_2O . The aerosol result was only 4% Na_2O loss compared to the 13% loss by the tie component result. It was believed that for >10% loss of a major component the tie component calculation was more reliable. However, there is a possibility that the volatility was lower and that the result was related to errors in the feed composition analyses.

The tie component Cr_2O_3 result was -683% which reflects corrosion of refractory and the stainless steel electrode sheaths. Similarly, the tie component analysis result for MoO_3 is -49% which reflects the loss of electrode material. The Method 5 results for Cr_2O_3 and MoO_3 indicate that 5.6% and 115% of the Cr and Mo contained in the feed, respectively, were volatilized to the offgas (corrected for 0.6 wt% entrainment).

The 83% volatility result for I is based on neutron activation analysis of four glass samples by USGS using a SrO tie component calculation. Suitable feed samples were unavailable so the feed target values were used for feed I content.

In addition to the slurry feeding data, Vectra generated data from feeding of dry batched materials with a composition intended to match a calcined feed (Run MT-3) and from running actual calcined feed (Run MT-4). Because of the short durations, tie component calculations were not possible for these testing periods. However, aerosol sample data were taken and can be used to estimate volatile losses. During Run MT-3, it appeared that the volatility was lower than for slurry feeding. During Run MT-4, it appeared that the volatility was as high or higher than slurry feeding and it appeared that significant unidentified sources of chloride and S were present. Additional information is provided in Appendix E.

3.3.5 Duratek

The Duratek DM-1000 melter experienced only moderate volatility losses. Results provided in Table 3-2 are, with a few exceptions discussed below, obtained using USGS analyses results for feed and glass and using SiO_2 as a tie component. Corning analytical results for feed were not used because no analysis was available for mixed feed and the simulant analysis indicated a significant deficiency in Al_2O_3 which may have been due to either sampling or analytical error. In the case of MgO , the Corning analytical results were selected because USGS failed to obtain a result greater than detection limits. For the fluoride result, the target feed concentration was used in place of the analytical value due to the analytical problems that were generally experienced for this analyte. Additional information is provided in Appendix F.

The selective loss values obtained using aerosol results used the VSL-CUA Method 5 data given in Table 5.7e of WHC-SD-WM-VI-027 (Eaton 1995b) and USGS feed analyses data, and were obtained by adjusting the data to remove the contribution from gross entrainment calculated assuming the Sr content in the Method 5 samples was due entirely to entrainment. Where the loss of a

component was less than or equal to the Sr loss, a value of ~0 is given. The VSL-CUA Method 5 results were chosen over the PES data because they were systematically higher values and provided more conservative results. This conservatism is considered warranted due to inadequate offgas flow measurement, the difficult sampling configuration, and the lack of agreement between the VSL-CUA and PES data.

A selective loss value of ~27% for P_2O_5 was obtained based on tie component calculations. The P_2O_5 content for the Duratek target glass was 0.19 wt%. When Duratek performed laboratory-scale test melts before operation, the P_2O_5 content of the glasses was 0.32 wt%. This result implies a ~68% volatility result for the laboratory melt when compared to feed analysis. This would tend to indicate that the feed contained too much P_2O_5 . By examining the data for the P_2O_5 in the glass, it is apparent that the glass concentration, and to a lesser extent the feed concentration, is dropping over time. The ratio of P_2O_5 to SiO_2 calculated for sequential glass sampling times is 7.1, 7.0, 6.6, 6.7, and 5.8 (all $\times 10^{-3}$); the sequential P_2O_5 to SiO_2 ratios in the feed were 4.93, 4.82, and 3.81 (all $\times 10^{-3}$). It is possible that there was phosphorus contamination in the system, and because the glass P_2O_5 to SiO_2 ratio was lagging the feed concentration, the data indicate a volatility gain across the melter with time. Because there are no Method 5 data on which to base an alternate P_2O_5 volatility estimate, it does not appear that there is sufficient information on which to base a defensible P_2O_5 volatility loss estimate.

A special estimate for SO_2 volatility was prepared based on an average of the USGS sample analysis data providing information on SO_2 (three feed, two glass) and using SiO_2 as a tie component. The result indicates SO_2 volatility of 7.2% with sample standard deviation of $\pm 17.3\%$ based on analysis of the USGS sample variability. Analytical data consistently indicate that feed contained more S than the feed target composition. The offgas SO_2 data at the high-efficiency particular air filter exit are inconsistent with feed data and indicate substantially more SO_2 in the offgas than was fed to the melter.

The 82% volatility result for I was determined based on neutron activation analysis of three feed samples and two glass samples using an SiO_2 tie component calculation.

3.3.6 Envitco

The Envitco dry feed test had the lowest selective component volatility losses of any test. Results were determined based on an average of the results obtained using SiO_2 , SrO, and Al_2O_3 tie components. With the exception of S and I, losses were too low to accurately determine the volatility using the tie component calculation. However, the results of the tie component calculation confirm that the volatile losses were low. Results obtained based on Method 29 sample data assume that the gross entrainment was 0.05% and have been corrected to account for entrainment. The volatile loss calculations for Cl and F were hindered by poor detection limits on the samples and are given as ranges.

The SO_2 selective loss value of 53% was based on only one glass sample for which detectable SO_2 was reported. Based on a second glass sample for which a less than value was reported, the SO_2 volatility loss would be >34%. Offgas SO_2 monitoring, which indicated SO_2 emissions accounted for 37% of the feed S content, also indicated significant S volatility.

The I result of 10% volatility was determined using USGS neutron activation analysis data for one feed sample and three glass samples. The feed sample appeared to be slightly deficient in I and if the target feed composition is used, the volatility is calculated at 16% with a maximum individual result of 24%. However, regardless of the treatment of the data the qualitative conclusion remains that the melter retained a significant fraction of the I within the glass.

3.4 TOTAL PARTITIONING TO OFFGAS

Total partitioning of volatile components to the offgas system obtained by combining the non-selective losses (assumed to be entrainment) in Table 3-1 with the selective losses (assumed to be volatility) in Table 3-2, using the methodology described in Section 3.1, are provided in Table 3-3. Supporting calculations are provided in Appendix H.3.

3.5 NO_x EMISSIONS

The generation of NO_x was examined for each of the vendor technologies. An NO_x yield was calculated by comparing the molar feed rate of NO_3^- and NO_2^- to the molar emission rate of NO_x in the offgas. No attempt was made to separate NO_x sources between thermal NO_x resulting from N_2 in air and NO_x resulting from feed N. A 100% yield was assumed to result in 1 mol of NO_x for each mole of NO_3^- and NO_2^- in the feed to the melter. Where appropriate, NO_x yields were calculated separately for the feed preparation processes and for the actual melter unit itself. A summary of the NO_x yields considering the melter unit only is provided in Table 3-4 followed by a brief description of the calculation used to arrive at the value for each vendor. Supporting calculations for NO_x yields during feed preparation and melter operations are provided in Appendix H.1.

The NO_x yields during preparation of the USBM Type B feed and Vectra fluid bed calcined feed are given in Table 3-5. The USBM data are from a sample of damp USBM Type B feed taken from the ribbon blender during feed production and was tested by PNNL to measure the gases released during the drying and reaction process. The Vectra calcination offgas data were measured during feed calcination by Procedyne and reported in WHC-SD-WM-VI-031, Part 1 (Stegen 1996).

3.5.1 B&W

The NO_x yield of 68% for B&W applies to operation without feed additives to reduce $\text{NO}_3^-/\text{NO}_2^-$ in the feed and with neither reburn or selective non-catalytic reduction operating. The relatively high yield may be due in part

Table 3-3. Percent of Feed Components Lost to Offgas for Evaluated Melter Systems.

Component	B&W	WSTC	USBM WHC1	USBM WHC2	USBM WHC3	Vectra	Duratek	Envitco
B ₂ O ₃	70	24	52	24	19	15	0.6	0.2
Cl	88	88	82	N/A	97	64	48	1 to 13
Cs ₂ O	85	84	63	N/A	40	41	14	0.6
F*	93	91	91	N/A	99.7	16	53	0.90
I	95	98	95	N/A	N/A	83	82	10
K ₂ O	55	49	36	SE	26	16	0.6	0.05
Li ₂ O	N/A	5.4	N/A	N/A	N/A	N/A	N/A	N/A
MoO ₃	64	26	48	51	46	SE	0.8	0.05
Na ₂ O	41	17	22	21	7.6	13.5	0.6	0.05
P	46	45	42	SE	55	1.7	SE	0.05
S	55	36	88	SE	94	85	SE	53

*Analytical difficulties experienced with fluoride analysis make all values approximate. See Appendix I.

B&W = Babcock & Wilcox

Duratek = Duratek Inc.

Envitco = Envitco, Inc.

N/A = Not available; no analytical information available on which to base estimates.

SE = Source error; no result can be determined because of a significant non-waste simulant source of the component.

USBM = U.S. Bureau of Mines

Vectra = Vectra Technologies, Inc.

WHC = Westinghouse Hanford Company

WSTC = Westinghouse Science and Technology Center

to a relatively high offgas flow rate and generation of thermal NO_x. A substantial fraction of the NO_x measured may be thermal NO_x and not originate from feed N.

3.5.2 WSTC

Additives were not used to reduce NO₃⁻/NO₂⁻ in the WSTC feed. The NO_x yield for the WSTC plasma torch was calculated as 8% based on online monitor data. The NO_x is attributed to thermal sources and no change in the online reading was observed between feeding and non-feeding operating conditions. Bomb samples indicated NO_x levels 5 to 20 times higher than the online

Table 3-4. Percent Yield NO_x Based on Melter Feed Nitrate and Nitrite Concentrations.

B&W	WSTC	USBM WHC1	USBM WHC2	USBM WHC3	Vectra	Duratek	Envitco
68	~8	0.03*	N/A	N/A	2.6	13	~33 ^a

*Prereacted feed. Does not include NO_x generation in feed preparation. Values including the feed preparation process emissions are USBM WHC1 = 7.4%, Envitco = 25% to 50%.

B&W = Babcock & Wilcox
 Duratek = GTS Duratek, Inc.
 Envitco = Envitco, Inc.
 USBM = U.S. Bureau of Mines
 Vectra = Vectra Technologies, Inc.
 WHC = Westinghouse Hanford Company
 WSTC = Westinghouse Science and Technology Center

Table 3-5. Offgas for U.S. Bureau of Mines Type B Feed Reaction and Vectra Feed Calcination.

Gaseous species	U.S. Bureau of Mines Type B feed laboratory data		Vectra Technologies, Inc. fluid bed calcination
	mmol release	% of gaseous N released as	% of gaseous N released as
NO_x	23	9.6	3.7
N_2O	18.8	15.6	4.7
NH_3	23.5	9.8	0.4
N_2	78.2	65	91

instruments. The average yield would be approximately 62% if the results of bomb samples were used. The results for Ar from the bomb samples were uncertain and WSTC believed that the bomb samples were more likely the incorrect analysis for NO_x . However, there was significant potential to lose some NO_x due to condensation in sample lines and subsequent scrubbing of NO_x . Therefore, a value higher than ~8% yield may be justified depending on the use of the data.

3.5.3 USBM

In the USBM feed preparation process, powdered sugar and activated carbon were added to the feed and the mixture was heated to dry the feed and react

the nitrates before feeding the melter. The feed preparation process removed 77.5% of the $\text{NO}_3^-/\text{NO}_2^-$ from the feed. Offgas data were unavailable from the feed preparation process. However, a sample of the unreacted feed was reacted in the laboratory (at PNNL) and the gases evolved were measured and are shown in Table 3-5. It is estimated that of the nitrogen released as gas in the feed preparation reaction, 9.6% of it is released as NO_x .

The calculated efficiency based on the $\text{NO}_3^-/\text{NO}_2^-$ destruction within the melter itself is extremely high (99.97%). This value is based on the remaining residue of $\text{NO}_3^-/\text{NO}_2^-$ within the feed and the NO_x data resulting from monitoring the offgas. Thus, the majority of the NO_x was evolved during the feed preparation process while the amount of NO_x generated within the melter was negligible. Looked at as an integrated process, the overall NO_x yield is $(9.6\%)(0.775) = 7.4\%$.

Because the majority of the NO_x would be expected to be generated during the feed preparation process, it would be expected that USBM WHC2 and USBM WHC3 would have roughly the same NO_x yield as USBM WHC1. However, offgas data were not collected during the WHC2 and WHC3 runs to allow independent NO_x yield estimates to be made for these runs.

3.5.4 Vectra

The Vectra slurry feed test (MT-2) used sucrose as a feed additive. The 2.6% NO_x yield was the lowest yield obtained from any of the combined feed preparation/melter systems. The estimate was based on two offgas sampling periods during slurry feeding operation that were designated Run 1 and Run 2. The feed characterization data were lacking and the results have been adjusted upward to account for a potential $\text{NO}_3^-/\text{NO}_2^-$ shortage in the feed corresponding to the apparent shortage of Na.

Feed for the Vectra calcined feed test (MT-4) was prepared from LLW simulant with sucrose added which was calcined in a fluid bed calciner at $\sim 500^\circ\text{C}$ with glass formers. The estimated fate of the N contained in the simulant $\text{NO}_3^-/\text{NO}_2^-$ is provided in Table 3-5 for the calcination run. The conversion of 3.7% of the feed nitrogen in $\text{NO}_3^-/\text{NO}_2^-$ to NO_x is fairly consistent with the data observed with slurry feeding of the melter. However, the data appear to have been taken from a sample point downstream from a caustic scrubber which introduces uncertainty that NO_2 may have been scrubbed before measurement. In any case, the reduction in the NO_x emission during calcination was clearly significant. The data indicate more efficient conversion to N_2 than was experienced in the USBM-1 test with much lower ammonia emissions.

3.5.5 Duratek

The Duratek test added urea to the feed and used a slurry feeding approach. The result of the 13% NO_x yield was based on feed analysis and an offgas flow of 90 stdft³/min, the glass production rate of 1,800 kg/day, and

glass and feed analysis results. The result is reasonably consistent with results obtained by Duratek (Eaton 1995b). The NO_x yield was calculated based only on the feed $\text{NO}_3^-/\text{NO}_2^-$ and does not include N contained in the urea.

3.5.6 Envitco

The Envitco test approach added activated carbon to the feed slurry before a spray dry operation. The spray dry operation removed 20% to 30% (assumed 25% for calculations) of the $\text{NO}_3^-/\text{NO}_2^-$ from the feed. However, information is unavailable on what fraction of the $\text{NO}_3^-/\text{NO}_2^-$ destroyed during feed preparation was evolved as NO_x . The melter itself evolved 33% of the $\text{NO}_3^-/\text{NO}_2^-$ remaining in the spray-dried feed as NO_x . When considering the feed preparation and melter as an integrated system, the melter accounts for a yield of 25% based on $\text{NO}_3^-/\text{NO}_2^-$ in the feed to the spray dry process, which represents the minimum system NO_x yield. An upper bound of 50% NO_x yield is estimated by assuming that all missing $\text{NO}_3^-/\text{NO}_2^-$ in the spray-dried feed was evolved as NO_x .

3.6 AMMONIA EMISSIONS

Ammonia emissions were not monitored in all offgas runs. The data that are available are summarized below. Supporting calculations are provided in Appendix H.1.

3.6.1 USBM

As shown in Table 3-5, the reactions during feed preparation generated 9.8 mol of NH_3 per 100 mol of N released as gas. The feed preparation process destroys 77.5% of the total feed $\text{NO}_3^-/\text{NO}_2^-$. Therefore, the NH_3 yield is $(9.8)(0.775) = 7.6\%$ based on the total molar feed rate of $\text{NO}_3^-/\text{NO}_2^-$ fed to the process. The melter itself produces an NH_3 yield of 0.9% based on the $\text{NO}_3^-/\text{NO}_2^-$ remaining in the melter feed after the feed preparation process. The overall NH_3 yield based on the feed preparation and melter system is $7.6 + (0.9)(0.225) = 7.8\%$.

3.6.2 Vectra

Vectra did not collect data on NH_3 emissions for slurry feeding. However, data were available for NH_3 emissions for the fluid bed calciner feed preparation run. As shown in Table 3-5, 0.4% of the feed N contained in $\text{NO}_3^-/\text{NO}_2^-$ was emitted as NH_3 during calcination of the feed slurry.

3.6.3 Duratek

The Duratek NH_3 yield is calculated neglecting the addition of urea to the feed and is based only on the $\text{NO}_3^-/\text{NO}_2^-$ content of the feed. On this basis, the melter emitted ~8.8% of the molar feed $\text{NO}_3^-/\text{NO}_2^-$ as NH_3 .

3.6.4 Envitco

Data are unavailable from the spray dry process. However, looking only at the melter and using the NO_3/NO_2 content of the dried feed, the NH_3 yield is 4.1%.

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5.0 GLOSSARY

ABBREVIATIONS AND ACRONYMS

B&W	Babcock & Wilcox
Duratek	GTS Duratek, Inc.
Envitco	Envitco, Inc.
EPA	U.S. Environmental Protection Agency
LLW	low-level waste
USBM	U.S. Bureau of Mines
Vectra	Vectra Technologies, Inc.
WHC	Westinghouse Hanford Company
WSTC	Westinghouse Science and Technology Center

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APPENDIX A

REVIEW OF BABCOCK AND WILCOX TEST REPORT

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DON'T SAY IT -- *Write It!*

Date: September 29, 1995

To: Greg Whyatt

From: Charles Freeman

Subject: Review of Babcock & Wilcox Off-Gas Data

(Revised 3/13/96 by Greg Whyatt)

The following review is of the Babcock & Wilcox (B&W) LLW Phase-1 testing off-gas results. The data used for this review came primarily from B&W's final test report along with Westinghouse Hanford's analytical database. Although the B&W report gave results from their scoring tests, only the 24-hour test data was reviewed for the present effort.

Sample Selection:

Table 1, attached, shows the samples collected during the 24 hour B&W test along with the sampling time. Samples were originally collected to provide information for a total of five mass-balance points. However, the method-5 sample for the fourth mass-balance set was collected over one hour prior to its corresponding glass sample. Additionally, the nearest feed sample was collected three hours later. As a result, the fourth mass-balance point was not calculated for this effort. The samples used for calculating the other four mass-balance points are identified in the sixth column of Table 1. Unfortunately, a good chronology of events during the run was not given in the B&W report. A shutdown was discussed but the times at which it occurred were not clear. Therefore, Table 1 also shows some of the events which were described at run times as best as they could be determined.

Condensible Off-gas Constituents:

The non-condensable off-gas data was given but only as averaged points for a two hour time frame. The raw data for these values were also not included making it impossible to verify the calculations. As a result the non-condensable off-gas value calculations could not be verified beyond what was reported in Table B.2-4 of the B&W report.

Combined Entrainment & Volatility (Carryover):

There is considerable uncertainty in the carryover estimates made from the B&W mass-balance run data, but it is expected that total carryover is on the order of 13% or greater. The analyses used to derive this estimate are described below.

The only feed, off-gas and glass stream mass data given in the B&W report are totalized values reported in mass balance Figure 5-2, page 5-13. Unfortunately, two problems are apparent in B&W's total mass balance analysis. First, B&W shows the percent theoretical oxides in their feed slurry to be 56.47%. There are no references as to how this value was obtained. By using the average loss on drying and loss on ignition values reported by Corning and USGS for the feed samples the percent theoretical oxides in the slurry becomes 46.3%. This is a substantial difference from the value used by B&W in their mass balance calculations. Second, B&W reports a cumulative glass mass, however, test observations recall that the quenched glass frit was weighed wet. A similar fritting process used by Vortec produced cullet which held up as much as 23% water by mass after draining (PNL laboratory record book number 55332, pg. 41). It is assumed and concurred by Bruce Higley, that B&W did not account for water hold-up when reporting their cumulative glass mass.

The uncertainty in the actual mass of glass produced (which results from the unknown amount of water hold-up) and the uncertainty in the slurry feed oxide loading prevent an accurate estimation of total entrainment and carryover. For the purpose of the analyses described herein, it is assumed that the B&W glass exhibited a 23% water hold-up and the slurry contained 46.3 wt% glass-forming and waste simulant oxides. These calculations should be revisited if either of these estimates are subsequently improved.

Table 6 shows the steps taken to correct the reported B&W feed and glass totals, as described above. The revised feed and glass masses were used to estimate the total carryover during the 24-hour test. The total oxide carryover estimates are given in Tables 2 through 5. The total carryover values are reported as a percentage of the total expected glass production rate when 100 lbs/h of 46.3 wt% oxide feed is processed. The overall carryover estimates range from 9.8% to 15.9% of the total theoretical glass production. The average carryover rate is about 13.2%. If the 56.47% oxide feed loading given by B&W is used instead of 46.3%, then these overall carryover estimates are about 82% as large as those shown in Tables 2 through 5.

The data in Table 6 indicate that the overall feed oxide carryover was approximately 28%. This is considerably higher than the 13.2% carryover estimate obtained from the Method 5 aerosol sampling data. The Table 6 carryover estimate, however, is highly-dependent on the assumed fraction of water hold-up in the product glass. If it is assumed that water hold-up was insignificant, then a calculation like that shown in Table 6 leads to an estimated carryover (combined entrainment and volatility) of only 6.5%. A water hold-up fraction of about 7.2%

yields a overall carryover estimate of 13.2% (which is consistent with the carryover estimate given by the Method 5 data). It is clear that an accurate measure of water hold-up is needed to refine the carryover estimate given in Table 6.

Another estimate of total carryover was attempted by using B&W's reported cumulative dry scrubber and baghouse masses (also reported in Figure 5-2 of the B&W report). This value came out to be approximately 57% of the theoretical glass mass. However, since the caustic scrubber utilized a liquid slurry of calcium carbonate, I am suspicious of the equivalent oxide values shown entering and exiting that unit. Additionally, if the baghouse solids were somewhat wet it could have resulted in this higher carryover estimate. Therefore, I would only recommend using the feed and glass masses and the Method 5 samples to estimate carryover.

Fractional Volatilities:

The feed analytical data provided by USGS was reported on a dry basis. Apparently, no data indicating water content was analyzed. Therefore, the subsequent calculations were made using only the Corning feed analytical data which did provide water content. Water content was critical for the total carryover calculations since the feed rate reported by B&W was in pounds per hour of slurry. This same approach was used by the authors of the B&W report. Unlike the feed data, the glass analyses from Corning were averaged with the USGS values for the calculations. The average of these two samples is denoted in the attached tables by an "X" in place of a "U" or a "C" in the sample number. Where only one analysis provided data for an analyte, the single value was used. In the case of fluoride, the feed target was selected as being more accurate than the analytical results. For feed iodine, the result from neutron activation analysis of USGS samples were used and ratioed back to the corning sample based on sodium content. The glass and feed values were used along with "tie" components to calculate constituent losses to the off-gas due to volatility.

Tables 2 through 5 show the results of the volatility and carryover calculations for mass balance sets 1, 2, 3, and 5, respectively. Each table shows volatility calculations for each constituent using five different tie components; Al, Fe, Si, Ca and Sr. These constituents were chosen since the volatility estimation equation assumes the tie component to have zero volatility. However, by looking through each mass balance set it is apparent that the values using the Al and Fe tie components are biased higher than each of the other components. It is believed that corrosion of the refractories is the cause of this bias. As a result, the values generated from the other tie components (Si, Ca and Sr) were assumed to be more comparable for the present effort and, correspondingly, averaged for each mass balance set to produce the

average volatilities shown (third to last column of Tables 2 through 5).

Two constituent showed consistently irregular volatility values. Barium and nickel were found to have highly negative estimated volatilities for all the mass balance/tie component sets. This would suggest more of this constituent in the glass than in feed. NiO is below detection limits in all feed samples and barium is at low levels in the feed. Although contamination sources have not been identified for these constituents, very little contamination could account for the result due to the small feed inventory. It is also possible that systematic analytical errors could contribute to this result.

Mass balance sets 1 and 3 showed highly negative volatility values for Al, Cr, Fe, and Ti, indicating much more of this material in the glass than in the feed. Indeed, the report describes the use of Plibrico 85-S and Shamrock 881-Plastic refractories. The Shamrock contains 9.8 wt% Cr_2O_3 and both refractories contain aluminum as a major component with iron and titanium as minor components. One peculiarity is that the mass balance set in between these two sets (number 2) shows reasonable (around zero volatility) values for the Al, Cr, Fe, and Ti constituents. One possible explanation is that the first mass balance set was taken while the refractory was still new(?) and sloughing material into the glass. Next, the second mass balance set was taken after a significant amount of material had been processed and a three hour feed stoppage (see Table 1) which cleaned the system of accumulated material. However, the third mass balance set was taken after cyclone gas inlet locations were modified to heat the front end more. This change could have produced more refractory corrosion on these "newly" heated areas and consequently generated the elevated levels of refractory constituents seen in the glass. After a number of hours the fifth mass balance set showed no apparent elevated levels of the refractory constituents. This may have been a result of the large amount of feed material processed through the system since the third set.

Results provided for fluoride are based on USGS glass results. The USGS glass analysis consistently gave substantially higher fluoride concentrations while Corning analysis found the fluoride to be below detection. If the Corning results are correct then volatile loss of fluoride was higher than indicated.

All analytical results for SO_3 in the glass were below detection limits. All calculations were performed using the detection limit. Therefore, the results from the tie component calculation provide the minimum loss while the maximum may range up to 100%.

Feed analysis for P_2O_5 content consistently provided results that were substantially below the target composition. The results calculated in the attached sheet utilize the analyzed result for the feed and result in an average of a 70% increase in P_2O_5 content over the feed. If

recalculated using the feed targets values, the volatile losses for the four mass balance periods are 26, 41, -53, and 26% respectively. Because the Plibrico Special 85-S and the Shamrock 881 Plastic refractories contain P_2O_5 (2.7 wt% and 2.5 wt% respectively) a source of P_2O_5 other than the feed is known to be present. Based on the results for Al and Fe, the refractory corrosion appears to be lowest during mass balance periods 2 and 5. Of these two periods the second mass balance period provides the highest volatility of 41%. It is assumed that the loss result is highest during this period because the source from the refractory is minimum. As a result, it is suggested that an approximate estimate of 41% volatile loss for P_2O_5 be considered.

The average results for volatile components of interest are provided below:

Oxide Component	Average % volatility	Notes
B_2O_3	67	
CaO	83	
K_2O	51	
MoO_3	60	
Na_2O	35	
P_2O_5	~41 %	Maximum loss based on feed target values. Due to refractory source, result is approximate.
SO_3	> 51	All glass SO_3 results are below detection limit
Cl	87	
F	92	based on feed target and USGS glass data, higher losses predicted using Corning data
I	94	Neutron activation data

Entrainment:

The Method 5 sample elemental analyses were used, along with the feed sample data, to generate the fractional carryover values for each mass balance set. These values are shown in the last column of Tables 2 through 5 under the column title of "% Carryover Based on Feed and Method 5 Samples." In order to estimate entrainment for the entire off-gas stream the carryover of a constituent with a low volatility, such as alumina, calcia, or silica, can typically be used. However, the silica carryover values for each mass balance set were low (about 1% to 3%) compared to the alumina and calcia values (5% to 11%). This may be a result of a silica-based filter having been used for the Method 5 sampling. If this were the case the analytical error for silica in the Method 5 sample would be high. Mass balance set 5 showed low/reasonable volatility data for alumina and calcia. The corresponding carryover values were 9.4% and 7.4%, respectively.

The average uniform entrainment is estimated to have been 8.7%. This value was obtained by averaging the % carryover estimates (right-most column) for alumina and calcia given in Tables 2 through 5. It must be stressed that the accuracy of this value depends to a sizeable extent on the actual amount of feed oxides processed by the melter during the mass balance test.

Table 1. Samples Taken for 24hr B&W Test

Sample Number	Sampling Medium	24hr Test Date	Sampling Start Time	Sampling End Time	Mass Balance Calculation #	Comments
B1F7-002C1	Feed	10/13/94	13:30		1	Assumed Start Time of Run
B1F7-002U1	Feed	10/13/94	13:30		1	
B1G9-002C1	Glass	10/13/94	13:30		1	
B1G9-002U1	Glass	10/13/94	13:30		1	
B1G9-005U2	Glass	10/13/94	13:30			
B1G9-006U	Glass	10/13/94	13:30			
B1G9-008U3	Glass	10/13/94	13:30			
B&W-M5-R1	Method 5 Filter	10/13/94	13:44	14:14	1	
B1W10-002C1	Baghouse	10/13/94	14:00			
B1D12-002Q1	Dry Scrubber	10/13/94	14:15		1	Feed Stopped at ~15:40 for 3 hours, Accumulated Material Melted Away
B&W-IMP-R1	Impactor (particle)	10/13/94	14:43	14:49	1	
B1W10-003C3	Baghouse	10/13/94	19:28			
B1G9-005C2	Glass	10/13/94	19:50		2	
B1G9-005U2	Glass	10/13/94	19:50		2	
B&W-IMP-R2	Impactor (particle)	10/13/94	20:09	20:14	2	
B1F7-005C2	Feed	10/13/94	20:10		2	
B1F7-005U2	Feed	10/13/94	20:10		2	
B&W-M5-R2	Method 5 Filter	10/13/94	20:30	21:30	2	Off-gas Damper Plugging Noted, Gas Inlets Modified to Redistribute Heat
B1G9-006C	Glass	10/13/94	21:30			
B1G9-006G	Glass	10/13/94	21:30			
B1F7-008C3	Feed	10/14/94	1:20		3	
B1F7-008U3	Feed	10/14/94	1:20		3	
B1G9-008C3	Glass	10/14/94	1:25		3	
B1G9-008U3	Glass	10/14/94	1:25		3	
B1W10-004C3	Baghouse	10/14/94	1:40			
B&W-IMP-R3	Impactor (particle)	10/14/94	1:44	1:49	3	
B&W-M5-R3	Method 5 Filter	10/14/94	2:00	3:00	3	
BHE-MR-R1	Baghouse	10/14/94	2:00	3:00	3	
B1D12-003Q3	Dry Scrubber	10/14/94	2:04		3	
B&W-IMP-R4	Impactor (particle)	10/14/94	6:40	6:45		
B&W-M5-R4	Method 5 Filter	10/14/94	6:55	7:55		
B1G9-011C4	Glass	10/14/94	8:13			
B1G9-011G4	Glass	10/14/94	8:13			
B&W-IMP-R5	Impactor (particle)	10/14/94	10:18	10:23	5	
B&W-M5-R5	Method 5 Filter	10/14/94	10:27	11:27	5	
B1F7-013C	Feed	10/14/94	11:30		5	
B1F7-013U	Feed	10/14/94	11:30		5	
B1M14-001C	Method 5 Probe	10/14/94	11:30		5	
B1D12-004Q4	Dry Scrubber	10/14/94	11:45		5	
B1W10-006C5	Baghouse	10/14/94	11:45			
B1G9-013C5	Glass	10/14/94	11:55		5	
B1G9-013G5	Glass	10/14/94	11:55			
B1G9-013U5	Glass	10/14/94	11:55		5	
B1G9-014C	Glass	10/14/94	14:30			
B1W11-001C	Baghouse	10/18/94	16:10			

Table 2. Off-gas Calculations Based on Mass Balance Set #1 (in weight % unless otherwise specified)

Sample No. Date Time	B1F7-002C1 10/13/94 1:30 PM		B1G9-002X1 10/13/94 1:30 PM		% Volat (Al tie) Based On Feed & Glass		% Volat (Fe tie) Based On Feed & Glass		% Volat (Si tie) Based On Feed & Glass		% Volat (Ca tie) Based On Feed & Glass		% Volat (Sr tie) Based On Feed & Glass		% Volatility (Ave, Sr,Si,Ca) Based O Feed & Glass		B&W-M5-R1 Method 5 (Elem Compositions)		% Carry-Over Based on Feed & Method 5 Samp.
	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	ug	g/hr	Ave.
Al2O3	5.4E+0	6E-2	1.6E+1	4E+0	0.00	39.00	20.41	30.95	-56.57	44.92	-46.70	40.51	-62.42	46.03	-55.23	25.34	62086	146.5	11.33
B2O3	2.9E+0	1E-2	2.4E+0	7E-1	72.90	10.68	78.43	8.48	57.57	12.42	60.24	11.22	55.98	12.73	57.93	7.01	81623	192.7	47.21
BaO	3.6E-3	3E-4	1.3E-2	9E-3	-21.81	90.04	3.05	71.60	-90.73	131.67	-78.70	122.58	-97.85	136.30	-89.10	75.23	ND	ND	
CaO	2.6E+0	2E-2	5.3E+0	6E-2	31.83	18.82	45.75	14.89	-6.73	8.59	0.00	2.05	-10.72	7.41	-5.81	3.84	33678	79.5	9.44
Cr2O3	2.4E-2	9E-4	6.7E-2	9E-3	8.36	28.55	27.07	22.62	-43.48	23.71	-34.43	19.60	-48.84	23.68	-42.25	12.94	1190	2.8	37.49
Cs2O	9.2E-2	0E+0	4.3E-2	1E-2	84.68	6.74	87.81	5.35	76.02	8.45	77.53	7.72	75.12	8.69	76.22	4.79	<22000	51.9	<131.49
Fe2O3	1.6E-1	6E-3	5.9E-1	2E-1	-25.65	48.85	0.00	38.76	-96.73	56.13	-84.33	50.59	-104.08	57.50	-95.05	31.65	1145	2.7	5.45
K2O	1.1E+0	9E-3	1.2E+0	2E-1	65.16	11.10	72.27	8.79	45.45	9.71	48.89	8.18	43.41	9.74	45.92	5.33	59210	139.8	33.51
Li2O		0E+0		0E+0	N/A	NA	N/A	NA	N/A	NA	N/A	NA	N/A	NA	NA	NA			
MgO	7.8E-2	3E-3	1.8E-1	4E-2	23.33	27.93	38.98	22.15	-20.04	30.11	-12.47	26.82	-24.52	30.74	-19.01	16.90	856	2.0	9.46
MnO2	9.2E-3	3E-4	2.3E-2	3E-3	17.11	26.13	34.03	20.70	-29.79	22.31	-21.60	18.64	-34.63	22.35	-28.67	12.22	247	0.6	22.04
MoO3	9.2E-2	0E+0	8.7E-2	3E-2	68.83	13.22	75.19	10.50	51.19	16.20	54.27	14.75	49.37	16.65	51.61	9.17	4495	10.6	38.02
Na2O	1.1E+1	3E-2	1.5E+1	1E+0	54.82	13.02	64.04	10.30	29.26	8.13	33.72	5.61	26.62	7.77	29.87	4.19	406873	960.3	26.10
NiO	7.1E-4	0E+0	1.1E-2	6E-3	-391.94	311.93	-291.53	247.97	-670.25	444.00	-621.69	412.19	-699.02	459.19	-663.65	253.40	ND		
P2O5	1.1E-1	3E-3	1.6E-1	5E-2	52.26	20.51	62.00	16.28	25.25	25.33	29.96	23.10	22.46	26.04	25.89	14.35	1270	3.0	14.02
SO3	1.4E-1	0E+0	1.3E-1	4E-2	70.72	11.57	76.69	9.18	54.15	13.47	57.04	12.17	52.44	13.81	54.54	7.60	5902	13.9	54.00
SiO2	2.9E+1	4E-1	5.6E+1	4E+0	36.13	18.33	49.17	14.50	0.00	11.20	6.30	7.54	-3.74	10.65	0.86	5.73	45378	107.1	1.72
SrO	5.5E-2	6E-4	1.0E-1	7E-3	38.43	17.45	51.00	13.81	3.60	9.89	9.68	6.04	0.00	9.24	4.43	4.94	1929	4.6	21.43
TiO2	8.0E-2	3E-3	2.4E-1	5E-2	1.94	33.51	21.96	26.56	-53.53	33.29	-43.85	29.11	-59.27	33.79	-52.22	18.55	ND		
ZrO2	3.0E-2	3E-3	6.3E-2	6E-3	29.72	21.54	44.07	17.06	-10.04	17.10	-3.10	13.87	-14.15	16.99	-9.09	9.27	ND		
Cl-	2.3E-1	0E+0	1.1E-1	7E-3	84.09	4.51	87.34	3.57	75.09	2.54	76.66	1.54	74.16	2.37	75.30	1.27	27100	64.0	61.32
F-	1.7E-1	5E-10	4.0E-2	6E-2	92.08	11.16	93.70	8.88	87.60	17.16	88.38	16.06	87.13	17.80	87.70	9.83	2250	5.3	6.97
I-	7.5E-2	0E+0	1.6E-2	N/A	93.12	N/A	94.53	N/A	89.23	NA	89.91	NA	88.83	NA	89.33	NA	<222	0.5	1.53

Total mass of oxide analytes carried over (lbs) = 6.39

Total carryover of theoretical glass mass (Method 5 Sample) = 13.8%

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3/13/96
MT
3/27/96

Table 3. Off-gas Calculations Based on Mass Balance Set #2 (in weight % unless otherwise specified)

Sample No. Date Time	B1F7-005C2 10/13/94 8:10 PM		B1G9-005X2 10/13/94 7:50 PM		% Volatl (Al tie) Based On Feed & Glass		% Volatl (Fe tie) Based On Feed & Glass		% Volatl (Si tie) Based On Feed & Glass		% Volatl (Ca tie) Based On Feed & Glass		% Volatl (Sr tie) Based On Feed & Glass		% Volatility (Ave, Si,Ca, Sr) Based O Feed & Glass		B&W-M5-R2 Method 5 (Elem Compositions)		% Carry-Over Based on Feed & Method 5 Samp.
	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	ug E	g E/hr	Ave.
Al2O3	5.6E+0	1E-1	1.2E+1	7E-1	0.00	9.52	24.56	17.93	2.53	6.88	-6.39	8.17	-23.92	9.99	-9.26	4.88	51786	81.7	6.13
B2O3	2.8E+0	6E-3	2.3E+0		61.94	2.56	71.29	6.54	62.91	0.80	59.51	1.50	52.84	2.09	58.42	0.90	101983	160.9	40.26
BaO	3.6E-3	3E-4	9.5E-3	3E-3	-27.37	43.54	3.91	38.94	-24.15	41.69	-35.51	45.69	-57.85	53.36	-39.17	27.23	ND	ND	
CaO	2.7E+0	2E-2	5.3E+0	2E-1	6.00	7.22	29.09	16.37	8.38	3.92	0.00	5.24	-16.48	6.73	-2.70	3.13	25958	40.9	4.70
Cr2O3	2.3E-2	4E-4	4.2E-2	1E-2	14.68	20.67	35.63	20.97	16.84	19.44	9.23	21.39	-5.73	25.05	6.78	12.75	1187	1.9	25.71
Cs2O	8.5E-2	0E+0	3.5E-2	8E-3	80.45	4.79	85.25	4.83	80.94	4.50	79.20	4.96	75.77	5.80	78.64	2.95	<22000	34.7	<95.07
Fe2O3	1.6E-1	3E-3	4.4E-1	1E-1	-32.55	31.50	0.00	32.23	-29.20	29.57	-41.02	32.56	-64.27	38.14	-44.83	19.41	521	0.8	1.66
K2O	1.0E+0	7E-3	1.1E+0	8E-2	48.91	5.11	61.46	9.23	50.21	3.83	45.65	4.49	36.69	5.46	44.18	2.68	76100	120.0	30.92
Li2O		0E+0		0E+0	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	NA	NA	NA	N/A			
MgO	7.8E-2	0E+0	1.7E-1	9E-3	-0.56	8.83	24.14	17.81	1.99	5.91	-6.98	7.22	-24.62	8.94	-9.87	4.31	686	1.1	5.06
MnO2	9.2E-3	1E-3	2.1E-2	6E-3	-8.29	33.82	18.30	31.10	-5.55	32.27	-15.21	35.40	-34.20	41.36	-18.32	21.09	157	0.2	9.35
MoO3	9.1E-2	3E-3	8.2E-2	6E-3	57.05	4.60	67.60	7.86	58.14	3.60	54.31	4.16	46.78	5.02	53.08	2.48	5110	8.1	29.30
Na2O	1.1E+1	3E-2	1.3E+1	7E-1	40.11	5.13	54.82	10.57	41.63	3.33	36.29	4.11	25.78	5.12	34.57	2.45	434873	686.0	19.11
NiO	7.1E-4	0E+0	3.5E-3	4E-3	-134.64	251.65	-77.01	193.72	-128.70	244.84	-149.62	267.35	-190.77	311.50	-156.36	159.32	ND		
P2O5	1.1E-1	4E-3	1.2E-1	4E-2	45.68	17.29	59.02	15.80	47.06	16.51	42.21	18.11	32.69	21.15	40.65	10.79	1550	2.4	11.73
SO3	1.2E-1	0E+0	1.3E-1	4E-2	52.33	13.86	64.04	13.06	53.54	13.18	49.29	14.46	40.93	16.91	47.92	8.62	8287	13.0	57.43
SiO2	2.9E+1	5E-1	6.3E+1	7E-1	-2.60	7.24	22.60	17.72	0.00	3.02	-9.15	4.66	-27.14	6.26	-12.10	2.79	34349	54.2	0.87
SiO	5.8E-2	1E-3	9.8E-2	4E-3	19.31	6.50	39.12	14.13	21.35	3.87	14.15	4.96	0.00	6.27	11.83	2.96	1673	2.6	11.95
TiO2	8.7E-2	4E-3	2.0E-1	2E-2	-7.10	15.80	19.20	21.25	-4.39	13.88	-13.94	15.54	-32.72	18.39	-17.02	9.26	ND		
ZrO2	3.0E-2	1E-3	7.0E-2	5E-3	-11.73	12.56	15.71	20.65	-8.90	10.07	-18.87	11.57	-38.46	13.89	-22.08	6.90	ND		
Cl-	2.3E-1	0E+0	7.0E-2	3E-3	85.49	1.14	89.06	2.53	85.86	0.65	84.57	0.85	82.02	1.08	84.15	0.51	39901	62.9	60.33
F-	1.6E-1	0E+0	2.0E-2	3E-2	94.18	7.86	95.61	6.01	94.33	7.65	93.81	8.35	92.79	9.73	93.64	4.98	2900	4.6	6.15
I-	8.4E-2	0E+0	1.1E-2	N/A	94.01	N/A	95.48	N/A	94.17	N/A	93.63	N/A	92.58	NA	93.46	NA	<22	0.4	0.92

Total mass of oxide analytes carried over (lbs) = 4.58

Total carryover of theoretical glass mass (Method 5 Sample) = 9.9%

Bray Wyatt
3/13/96
M. Paul
3/27/96

Table 4. Off-gas Calculations Based on Mass Balance Set #3 (in weight % unless otherwise specified)

Sample No. Date Time	B1F7-008C3 10/14/94 1:20 AM		B1G9-008X3 10/14/94 1:25 AM		% Volatl (Al tie) Based On Feed & Glass		% Volatl (Fe tie) Based On Feed & Glass		% Volatl (Si tie) Based On Feed & Glass		% Volatl (Ca tie) Based On Feed & Glass		% Volatl (Sr tie) Based On Feed & Glass		% Volatility (Ave, Si,Ca,Sr) Based O Feed & Glass		B&W-M5-R3 Method 5 (Elem Compositions)		% Carry-Over Based on Feed & Method 5 Samp.
	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	ppm E	g E/hr	Ave.
Al2O3	5.6E+0	1E-1	1.9E+1	6E+0	0.00	43.77	41.29	18.57	-87.41	69.66	-48.51	50.41	-90.69	72.54	-75.54	37.50	93500	146.6	10.93
B2O3	2.9E+0	2E-2	7.5E-1		92.49	2.33	95.59	0.29	85.92	2.90	88.85	1.56	85.68	3.17	86.82	1.52	132983	208.5	51.16
BaO	3.6E-3	3E-4	1.1E-2	3E-3	14.46	35.05	49.78	13.88	-60.30	54.25	-27.02	38.44	-63.11	56.75	-50.14	29.14	ND	ND	
CaO	2.7E+0	6E-2	6.2E+0	8E-1	32.66	22.86	60.47	6.09	-26.20	31.38	0.00	19.72	-28.41	33.57	-18.20	16.67	56858	89.1	10.32
Cr2O3	2.4E-2	2E-3	1.7E-1	1E-1	-110.44	155.97	-23.55	83.59	-294.40	277.73	-212.52	214.92	-301.29	284.44	-269.40	150.64	1507	2.4	32.22
Cs2O	8.6E-2	0E+0	1.5E-2	3E-3	95.08	1.75	97.11	0.54	90.78	2.49	92.69	1.64	90.61	2.65	91.36	1.33	<22000	34.5	<93.82
Fe2O3	1.6E-1	3E-3	9.2E-1	6E-2	-70.33	53.88	0.00	9.24	-219.22	68.95	-152.95	38.95	-224.80	74.90	-198.99	36.33	776	1.2	2.43
K2O	1.0E+0	9E-4	7.8E-1	9E-2	78.09	7.25	87.14	1.73	58.94	9.73	67.46	5.93	58.22	10.46	61.54	5.16	93200	146.1	37.37
Li2O		0E+0		0E+0	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	NA	NA	N/A	N/A			
MgO	7.9E-2	0E+0	2.9E-1	7E-2	-5.53	41.79	38.04	15.83	-97.78	63.60	-56.72	44.46	-101.24	66.72	-85.24	34.11	1250	2.0	9.10
MnO2	1.1E-2	3E-4	2.5E-2	1E-3	33.47	20.99	60.94	3.50	-24.68	26.78	1.20	15.05	-26.86	29.11	-16.78	14.11	214	0.3	10.90
MoO3	8.4E-2	3E-3	3.2E-2	1E-2	88.95	5.01	93.52	2.19	79.30	8.08	83.60	5.90	78.94	8.40	80.61	4.36	6280	9.8	38.54
Na2O	1.1E+1	3E-2	1.2E+1	3E+0	66.10	12.59	80.10	4.29	36.47	18.47	49.66	12.49	35.36	19.50	40.50	9.88	675873	1059.6	29.51
NiO	7.2E-4	0E+0	1.3E-2	7E-3	-409.14	323.56	-198.91	167.05	-854.18	564.86	-656.10	432.70	-870.87	580.06	-793.72	306.01	ND		
P2O5	1.1E-1	2E-3	3.2E-1	1E-1	12.76	38.77	48.78	16.67	-63.50	62.05	-29.56	45.08	-66.36	64.55	-53.14	33.42	2240	3.5	16.85
SO3	1.3E-1	0E+0	1.3E-1	4E-2	70.84	12.23	82.88	4.97	45.35	19.12	56.70	13.65	44.39	19.96	48.81	10.28	2993	4.7	20.66
SiO2	3.1E+1	3E-1	5.6E+1	1E+1	46.64	19.83	68.67	6.77	0.00	29.11	20.76	19.70	-1.75	30.74	6.34	15.57	133791	209.7	3.22
SrO	5.7E-2	1E-3	1.0E-1	2E-2	47.56	19.95	69.21	7.10	1.72	29.69	22.12	20.36	0.00	31.27	7.95	15.90	2993	4.7	21.36
TiO2	9.9E-2	9E-4	3.3E-1	2E-2	4.08	30.36	43.68	5.23	-79.77	38.86	-42.45	21.97	-82.92	42.21	-68.38	20.48	ND		
ZrO2	3.6E-1	2E-3	6.7E-2	2E-2	94.58	2.43	96.82	1.06	89.85	3.91	91.96	2.85	89.67	4.07	90.49	2.11	ND		
Cl-	2.7E-1	0E+0	1.5E-2	1E-2	98.38	1.18	99.05	0.63	96.96	2.10	97.59	1.62	96.91	2.15	97.16	1.14	41908	65.7	53.64
F-	1.6E-1	0E+0	2.0E-2	3E-2	96.44	4.93	97.91	2.82	93.33	9.10	94.71	7.17	93.21	9.28	93.75	4.95	53000	83.1	111.77
I-	7.3E-2	0E+0	2.5E-3	0E+0	99.00	N/A	99.41	N/A	98.12	N/A	98.51	N/A	98.09	NA	98.24	NA	<22	0.3	1.05

Total mass of oxide analytes carried over (lbs) =

7.41

Total carry-over of theoretical glass mass (Method 5 Sample) =

16.0%

200g 1/2 gact
3/13/96
mi Pan
3/27/96

Table 5. Off-gas Calculations Based on Mass Balance Set #5 (in weight % unless otherwise specified)

Sample No. Date Time	B1F7-013C 10/14/94 11:30 AM		B1G9-013X5 10/14/94 11:55 AM		% Volatl (Al tie) Based On Feed & Glass		% Volatl (Fe tie) Based On Feed & Glass		% Volatl (Si tie) Based On Feed & Glass		% Volatl (Ca tie) Based On Feed & Glass		% Volatl (Sr tie) Based On Feed & Glass		% Volatility (Ave, Al,Si,Ca) Based O Feed & Glass		B&W-M5-R5 Method 5 (Elem Compositions)		% Carry-Over Based on Feed & Method 5 Samp.
	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	ppm E	g E/hr	Ave.
Al2O3	5.7E+0	5E-2	1.3E+1	1E+0	0.00	11.92	0.93	16.54	-8.58	10.64	-5.50	9.09	-25.65	15.50	-4.69	6.96	78286	127.3	9.37
B2O3	2.7E+0	1E-2	2.1E+0		65.19	2.94	65.51	4.97	62.20	1.90	63.27	0.69	56.26	3.95	63.55	1.48	119983	195.1	50.45
BaO	3.6E-3	4E-4	9.5E-3	1E-3	-20.38	22.97	-19.27	26.68	-30.71	23.31	-27.00	21.86	-51.26	29.26	-26.03	14.44	ND	ND	
CaO	2.7E+0	2E-2	5.7E+0	9E-2	5.21	8.17	6.09	13.64	-2.92	5.48	0.00	2.55	-19.10	10.95	0.76	4.17	39758	64.7	7.38
Cr2O3	2.3E-2	7E-4	4.9E-2	1E-2	2.98	29.94	3.88	31.72	-5.34	31.72	-2.35	30.45	-21.91	37.83	-1.57	19.33	1457.1	2.4	33.37
Cs2O	8.6E-2	0E+0	2.8E-2	2E-3	85.48	1.58	85.61	2.29	84.23	1.34	84.68	1.08	81.76	2.06	84.80	0.90	<22000	35.8	<97.47
Fe2O3	3.1E-1	9E-3	6.9E-1	1E-1	-0.93	16.85	0.00	20.37	-9.59	16.71	-6.48	15.46	-26.82	21.55	-5.67	10.45	7675.1	12.5	12.80
K2O	1.0E+0	3E-3	1.0E+0	5E-2	54.90	4.38	55.32	6.79	51.03	3.41	52.42	2.46	43.33	5.80	52.78	2.39	90100	146.5	37.79
Li2O		0E+0		0E+0	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	NA	NA	N/A	N/A			
MgO	7.9E-2	0E+0	1.7E-1	2E-2	2.08	12.97	2.99	17.14	-6.32	12.10	-3.30	10.72	-23.04	16.77	-2.51	7.76	1060	1.7	8.01
MnO2	9.3E-3	1E-3	2.1E-2	6E-3	-2.35	34.46	-1.40	36.14	-11.13	36.65	-7.98	35.25	-28.60	43.49	-7.15	22.30	208	0.3	12.70
MoO3	8.4E-2	3E-3	8.1E-2	2E-2	56.51	9.53	56.91	10.70	52.78	9.84	54.12	9.32	45.35	12.10	54.47	6.06	5950	9.7	37.94
Na2O	1.0E+1	0E+0	1.4E+1	1E+0	38.81	7.01	39.38	9.92	33.56	6.14	35.45	5.15	23.11	9.15	35.94	4.06	590873	960.9	27.36
NiO	7.2E-4	0E+0	4.5E-3	4E-3	-185.12	278.31	-182.48	277.71	-209.58	301.46	-200.80	292.57	-258.25	349.89	-198.50	182.24	ND		
P2O5	1.0E-1	5E-3	1.6E-1	5E-2	29.70	22.74	30.35	23.95	23.66	24.14	25.83	23.20	11.66	28.71	26.40	14.70	2040	3.3	16.26
SO3	1.2E-1	0E+0	1.3E-1	4E-2	54.69	13.37	55.11	14.25	50.80	14.13	52.20	13.55	43.07	16.90	52.56	8.62	8207	13.3	58.78
SiO2	3.0E+1	4E-1	6.1E+1	3E+0	7.90	9.03	8.75	13.92	0.00	7.08	2.84	5.17	-15.72	11.93	3.58	4.93	47079	76.6	1.20
SrO	5.9E-2	1E-3	1.0E-1	9E-3	20.41	9.82	21.15	13.40	13.59	8.91	16.04	7.72	0.00	12.75	16.68	5.79	2742.6	4.5	19.83
TiO2	7.2E-2	0E+0	1.7E-1	5E-2	-4.54	31.82	-3.58	33.77	-13.51	33.69	-10.29	32.32	-31.36	40.21	-9.45	20.54	ND		
ZrO2	2.6E-2	8E-4	5.6E-2	8E-3	2.32	16.51	3.22	19.88	-6.06	16.42	-3.05	15.21	-22.73	21.11	-2.26	10.25	ND		
Cl-	3.1E-1	0E+0	4.5E-2	6E-3	93.42	1.08	93.49	1.32	92.86	1.07	93.06	0.99	91.74	1.39	93.12	0.67	40901	66.5	47.30
F-	1.6E-1	5E-10	2.0E-2	3E-2	94.35	7.64	94.40	7.60	93.87	8.28	94.04	8.04	92.90	9.60	94.09	5.01	3400	5.5	7.60
I-	8.2E-2	0E+0	7.5E-3	0E+0	95.88	N/A	95.92	N/A	95.52	N/A	95.65	N/A	94.82	NA	95.69	NA	<222	0.4	0.97

Total mass of oxide analytes carried over (lbs) =	6.15
Total carryover of theoretical glass mass (Method 5 Sample) =	13.3%

See Report
 3/13/96
M. Powell
 2/27/96

Table 6. Total Carryover Estimates from Cumulative Glass and Feed Values

Slurry feed rate reported =	100 lb slurry/hr
Total amount of feed reported in figure 5-2 (oxide basis) =	753 kg oxides
Conversion of reported total feed oxides to slurry using B&W's conversion (56.47% of slurry = oxides) =	1333 kg slurry
Calculated total feeding time using B&W's feed rate and total =	29.4 hrs
Combined "loss on drying" and "loss on ignition" from feed analytical values =	53.7 %
Revised %oxides-in-slurry value using loss on drying & ignition value =	46.3 %
Revised calculation of total oxides fed using new slurry-to-oxides conversion =	617 kg oxides
Total amount of glass generated, reported in Figure 5-2 (note that this material was measured wet, after fritting, and B&W did not correct for water in their report) =	577 kg
Estimated amount of residual water in glass frit (from Vortec glass cullet measurements) =	23 %
Revised total amount of dry glass generated (correcting for residual water) =	444 kg
Revised total volatility/entrainment estimate using loss drying & ignition data and correcting for residual water in glass cullet =	28.0 %

Reviewed by MR Powell
by T Powell 9/23/85

11/27/96
Randy

Summary Tie Component Results

Sample No. Date Time	Balance #1		Balance #2		Balance #3		Balance #5		Overall Average
	% Volatility (Ave, Sr,Si,Ca) Based On		% Volatility (Ave, Si,Ca, Sr) Based On		% Volatility (Ave, Si,Ca,Sr) Based On		% Volatility (Ave, Al,Si,Ca) Based On		
	Feed & Glass		Feed & Glass		Feed & Glass		Feed & Glass		
	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	Ave.	Stdv	
Al2O3	-55.23184	25.338177	-9.260139	4.8753003	-75.53531	37.500089	-4.692535	6.961635	-36.17995
B2O3	57.93127	7.0082752	58.420222	0.8988644	86.815453	1.5227093	63.551294	1.4793541	66.67956
BaO	-89.09538	75.231292	-39.16834	27.228643	-50.14456	29.139483	-26.03306	14.443024	-51.11033
CaO	-5.81472	3.8423025	-2.700114	3.1278143	-18.20136	16.667274	0.7634419	4.1680208	-6.488189
Cr2O3	-42.24935	12.93854	6.777186	12.748735	-269.4033	150.64078	-1.572697	19.333547	-76.61204
Cs2O	76.223215	4.7899457	78.636438	2.953796	91.36073	1.3288177	84.798644	0.8955057	82.754757
Fe2O3	-95.04593	31.652346	-44.8283	19.405396	-198.9892	36.332091	-5.670314	10.450208	-86.13343
K2O	45.919469	5.3332395	44.181118	2.6793105	61.539325	5.1571238	52.783667	2.3868114	51.105895
Li2O	NA	NA	NA	N/A	N/A	N/A	N/A	N/A	NA
MgO	-19.01108	16.901022	-9.869745	4.3082859	-85.24329	34.112609	-2.51493	7.7632384	-29.15976
MnO2	-28.67437	12.222702	-18.32126	21.094939	-16.7791	14.108605	-7.153615	22.304434	-17.73209
MoO3	51.607249	9.1709523	53.076463	2.4836488	80.610872	4.3552673	54.4662	6.0556406	59.940196
Na2O	29.868474	4.1904643	34.566463	2.4543263	40.4988	9.8750688	35.938495	4.0551422	35.218058
NiO	-663.6544	253.39761	-156.3627	159.32385	-793.7176	306.00693	-198.4994	182.23868	-453.0585
P2O5	25.890506	14.350288	40.651556	10.790597	-53.1389	33.415096	26.396538	14.700844	9.9499262
SiO3	54.544381	7.6004166	47.919158	8.6206262	48.813424	10.276358	52.564938	8.6199359	50.960475
SiO2	0.856122	5.731869	-12.09744	2.7894269	6.3367274	15.566048	3.5799702	4.9342398	-0.331154
SrO	4.4266472	4.9408672	11.832921	2.9614706	7.9470858	15.896127	16.679042	5.7865908	10.221424
TiO2	-52.21598	18.550739	-17.01574	9.264255	-68.38158	20.477714	-9.449764	20.537913	-36.76577
ZrO2	-9.093486	9.2682836	-22.07749	6.8992571	90.490845	2.108387	-2.263668	10.254927	14.264049
Cl-	75.300435	1.2662675	84.150681	0.5050964	97.15603	1.1384423	93.11579	0.6704205	87.430734
F-	87.704313	9.827274	93.644556	4.9780117	93.752957	4.9484233	94.085315	5.0050353	92.296785
I-	89.326696	NA	93.460134	NA	98.240713	NA	95.685178	NA	94.17818

Long 11/16/16
 3/13/16

W. B. Burt
 3/27/16

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CALCULATION WORKSHEET

Calc. No. _____

Page 1 of 3Special Estimates B+WMass Balance 1 - Feed

- No SO_2 data in selected sample so use slurry and 29 wt% loss on drying
 $.17 \text{ wt\% } \text{SO}_4 \left(\frac{32.06 + 3(16)}{32.06 + 4(16)} \right) \left(\frac{1}{1-.29} \right) = 0.200 \text{ wt\% } \text{SO}_2 \text{ in dry sample}$ $\leftarrow \text{SO}_2$
- No Cl data, use slurry data .23 wt% and loss on dry with CaO check
 check: $.23 \text{ wt\%} \left(\frac{3.66}{2.60} \right) = .3165 \text{ wt\% Cl in dry sample}$
 use loss on dry basis $.23 \left(\frac{1}{1-.29} \right) = .3239 \text{ Cl in dry sample}$ $\leftarrow \text{Cl}$
- No F data, use feed target $.1406 \text{ kg/hr}$ vs Sodium = 6.79 kg/hr
 $\left(15.4 \text{ N}_2\text{O in feed} \right) \left(\frac{.1406 \text{ F}}{6.79 \text{ kg Na} \left(\frac{46+16}{46} \right)} \right) = .2366 \text{ wt\% F in dry sample}$ $\leftarrow \text{F}$
- No I⁻ data, use activation analysis BIF 7-0024 = $0.0904 \text{ wt\%}$
 Sodium = $13.1 \text{ wt\% Na}_2\text{O}$
 $15.4 \left(\frac{.0904}{13.1} \right) = .1063 \text{ wt\% I in dried sample}$ $\leftarrow \text{I}$

Glass P_2O_5 only 1 data point so use that

SO_2 neither sample has data except detection limit, use that

I^- 1 data point use that

Mass Balance 2 - Feed

- NO SO_2 data, use slurry
 $.15 \text{ SO}_4 \left(\frac{.8334 \frac{\text{SO}_2}{\text{SO}_4}}{.609} \right) \left(\frac{1}{1-.289} \right) = .1758 \text{ wt\% } \text{SO}_2$
- No Cl data, use slurry $.23 \left(\frac{1}{1-.289} \right) = .3235 \text{ wt\% Cl}$
- No F data use target $15.0 \left(\frac{1}{1.536 \times 10^2} \right) = .2305 \text{ wt\% F}$
- No I data use activation $.00542 = .1082$, Ratio Sodium $13.8 \text{ wt\%}$
 $15.0 \left(\frac{.1082}{13.8} \right) = .1176 \text{ wt\%}$

Glass same as mass balance #1

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Title Special Estimates used in Spreadsheets		Project LLW Vitrification	
Prepared by: Greg Whyatt	Date 3/13/96	Reviewed by: [Signature]	Date 3/27/96

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CALCULATION WORKSHEET

Calc. No. _____

Page 2 of 3

Mass Balance #3 Feed

$$SO_3 \text{ from } .15 SO_4^- \left(.8334 \right) \left(\frac{1}{1-.284} \right) = .1746$$

$$Cl \quad .027 Cl \left(\frac{1}{1-.284} \right) = .3771$$

$$F \quad 14.9 \left(1.536 \times 10^{-2} \right) = .2289$$

$$I \quad .0941 \left(\frac{14.9}{13.8} \right) = .1016$$

Glass Same as Mass Balance #1

Mass Balance #5 Feed

$$SO_3 \quad .15 SO_4^- \left(.8334 \right) \left(\frac{1}{1-.285} \right) = .1748$$

$$Cl \quad .31 \left(\frac{1}{1-.285} \right) = .4336$$

$$F \quad 14.6 \left(1.536 \times 10^{-2} \right) = .2243$$

$$I \quad .1082 \left(\frac{14.6}{13.7} \right) = .1153$$

Glass Same as Mass Balance #1

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Title

Project

Prepared by:

Date

Reviewed by:

Date

Battelle

Pacific Northwest Laboratories

CALCULATION WORKSHEET

Calc. No. _____

Page 3 of 3 P_2O_5 Target Feed ValuesTarget = .1209 P_2O_4 , 6.77 N_2

$$.1209 \left(\frac{2(50.97) + 5(16)}{(30.97 + 4(16))2} \right) = .09035 P_2O_5$$

$$6.77 N_2 \left(\frac{46 + 16}{46} \right) = 9.1517 N_2O$$

Value From Spreadsheet
for P_2O_5 Loss

$$\text{Feed \#1} \quad 10.934 \left(\frac{.09035}{9.1517} \right) = .1080 \rightarrow 25.9$$

$$\#2 \quad 10.665 \left(\frac{.09035}{9.1517} \right) = .1053 \quad 40.7$$

$$\#3 \quad 10.668 \left(\frac{.09035}{9.1517} \right) = .1053 \quad -53.14$$

$$\#5 \quad 10.439 \left(\frac{.09035}{9.1517} \right) = .1031 \quad 26.4$$

Avg = 10.0

Re calculate Spreadsheet for P_2O_5 with target feed $\uparrow$

BEST AVAILABLE COPY

Title _____

Project _____

Prepared by: _____

Date _____

Reviewed by: *Mr. [Signature]*Date 3/27/96

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APPENDIX B

REVIEW OF WESTINGHOUSE SCIENCE AND TECHNOLOGY CENTER TEST REPORT

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Westinghouse Science and Technology Center Melter (WSTC) Entrainment and Volatility Estimates

The WSTC analytical data were examined and estimates made of the relative volatility and entrainment for each oxide analyte. These analyses and the results are described below. The volatility estimates were made by comparing the melter feed and glass product compositions. Assuming uniform entrainment of feed and zero volatility for a known low volatility component, the fractional volatility of each glass oxide species can be estimated. The entrainment of melter feed was estimated based on the observed average aerosol composition and the total mass of oxides known to have escaped to the off-gas system.

Entrainment

Feed entrainment during the 24-hour demonstration run test is estimated to have been between 2% and 3% of the feed oxides. Lower entrainment estimates based on aerosol collection rates are given in the vendor-prepared test report, but these estimates are suspect because of problems with the aerosol sampling procedures and test conditions. These problems include large uncertainties in the off-gas flow rate and the accumulation of solids upstream of the aerosol collection point. Further, the reported aerosol flow rates are much too low to account for the more than 1000 kg of glass mass that was missing from the glass product.

The 2% to 3% entrainment estimate was developed as follows. Based on the feed analyses and the known total mass of feed delivered to the melter, a total of 8383 kg of glass should have been produced during the 24-h demonstration test assuming no entrainment or volatility losses (p. 6-9 of McLaughlin et al.). Only 7272 kg of glass were recovered during the test (p. 6-6, *ibid.*). Therefore, a total of 1011 kg of glass must have been directed to the melter off-gas system. Assuming that the aerosol samples collected during the test accurately reflect the composition of the aerosols (but not necessarily the aerosol flow rate), the rate of feed entrainment can be estimated using the aerosol sample concentrations of non-volatile oxides like SiO_2 , CaO , and Al_2O_3 . Using this approach (see attached calculation worksheet), the feed entrainment is estimated by SiO_2 concentrations to be 1.2%, CaO gives 3.3%, and Al_2O_3 gives 2.1%. Because SiO_2 analyses have proven to be somewhat unreliable in the past, the entrainment estimates given by Al_2O_3 and CaO are probably more accurate.

Volatility

Fractional volatility estimates were generated for each of the glass oxide analytes. To make these calculations, it is necessary to select a tie-component, which is assumed to have zero volatility. Experience has shown that SiO_2 , CaO , and Al_2O_3 are generally good low volatility tie-components. The volatility calculations were made independently for each tie-component for each of the four snapshot samples taken during the 24-h demonstration test. The results are given in the attached spreadsheets.

The fractional volatilities calculated using CaO and Al_2O_3 as the tie-component are judged most reliable. As noted earlier, SiO_2 analyses are often in error because of analytical difficulties. There is no compelling reason to select CaO as the tie component rather than Al_2O_3 or vice versa, so reported in Table 1 for each glass oxide is the average of the CaO-tie volatility and the Al_2O_3 -tie volatility.

The volatility data for certain glass oxides require some further explanation. The fractional volatilities for Na_2O , K_2O , and F show a significant increase over the course of the test. This is consistent with the hypothesis that the glassformer addition rate decreased during the test, thereby increasing the waste loading. As the waste loading is increased, it is reasonable to expect a concomitant increase in the fractional volatilities for the waste-borne volatile components. The effects of the changing glassformer addition rate are explored in detail in the report prepared by McLaughlin et al. (1995).

The SO_3 volatilities vary between -0.4 and 0.43. This is believed to be the result of analytical errors primarily in the analyses of the third feed sample. The SO_4^{2-} concentrations for the first, second, and forth snapshot analyses are 0.17 wt%, 0.18 wt%, and 0.14 wt%. The third snapshot, however, was reported to contain only 0.091 wt%. In view of this, it is expected that the best estimate for SO_3 fractional volatility is approximately 0.3 to 0.4.

It is not clear why MgO , Fe_2O_3 , and TiO_2 are found to have negative volatilities (which imply a net gain of the species as the feed passes through the melter). The concentrations of MgO and TiO_2 are both quite low in the feed and the glass samples, so it may be appropriate to attribute these data to analytical uncertainties. The ferric oxide concentrations, however, are well within the measurable range, so it is unlikely that the negative Fe_2O_3 volatilities are due to random analytical uncertainties alone.

Reference:

McLaughlin, D. F., W. C. Yang, W. R. Gass, S. V. Dighe, N. D'Amico, R. L. Swensrud, and M. F. Darr. 1995. *Evaluation of Plasma Melter Technology for Vitrification of High-Sodium Content Low-Level Radioactive Liquid Wastes -- Demonstration Test #4 Final Test Report*. 95-9TD2-VITNA-R2. Distributed by Westinghouse Hanford as document WHC-SD-WM-VI-016, Rev. A. April 28, 1995.

Table 1. Fractional Volatility Calculations with CaO and Al₂O₃ Tie Components

Estimate of Feed Oxide Volatilities (avg. of CaO and Al ₂ O ₃ tie values)					
Oxide	Snap #1	Snap #2	Snap #3	Snap #4	Avg.
Al ₂ O ₃	-0.051	0.010	-0.054	-0.0020	-0.025
B ₂ O ₃	0.31	0.14	0.22	0.19	0.22
BaO	0.30	0.0074	-0.12	0.039	0.058
CaO	0.047	-0.010	0.049	0.0019	0.022
Cr ₂ O ₃	0.13	0.24	-0.011	0.10	0.12
Cs ₂ O	0.83	0.83	0.81	0.88	0.84
Fe ₂ O ₃	-0.28	-0.13	-0.20	-0.66	-0.32
K ₂ O	0.43	0.48	0.43	0.59	0.48
Li ₂ O	0.017	0.045	0.072	-0.024	0.028
MgO	-0.14	-0.039	-0.10	-0.23	-0.13
MnO ₂	0.30	0.42	0.25	-0.072	0.22
MoO ₃	0.15	0.22	0.18	0.39	0.24
Na ₂ O	0.026	0.17	0.098	0.31	0.15
NiO	0.69	0.69	0.69	0.73	0.70
P ₂ O ₅	0.38	0.56	0.41	0.36	0.43
SO ₃	0.37	0.43	-0.40	0.23	0.34 ^(a)
SiO ₂	0.14	0.092	0.12	0.092	0.11
SrO	-0.12	0.042	-0.088	0.16	-0.0005
TiO ₂	-1.2	-1.5	-3.4	-2.0	-2.0
ZrO ₂	0.0047	-0.0082	0.017	-0.038	-0.0061
Cl ⁻	0.88	0.87	0.85	0.90	0.88
F ⁻	0.42	0.44	0.58	0.65	0.52

(a) The snapshot #3 feed result for sulfate is believed to be in error so the average sulfur loss is calculated based on snapshots 1, 2 and 4 only.

Traceability Aid For Analytical Samples

Sample analysis results are located in WHC-SD-WM-DP-131, Rev.0 "Low-Level Waste Vitrification Phase 1 Test Sample Analyses Data" under the following sample numbers.

Snapshot #1 was based on the following samples:

glass W1G07-404C

feed W1F12-401C1(d)

Snapshot #2 was based on the following samples:

glass W1G07-404C2

feed W1F12-401C2(d)

Snapshot # was based on the following samples:

glass W1G07-404C3

feed W1F12-401C3(d)

Snapshot # was based on the following samples:

glass W1G07-404C4

feed W1F12-401C4(d)

Estimate WSTC entrainment during 24-h run

Data: from Rev. A of "Evaluation of Plasma Melter Technology for Vitrification of High-Sodium Content Low-Level Radioactive Liquid Wastes -- Demonstration Test #4 final Test Report." 4/26/95

p. 6-6: Total glass mass produced = 7372 kg

p. 6-9: Total theoretical glass (no losses) = 8383 kg (assuming linearly decreasing frit rate)

p. 7-16: Average species concentrations in aerosol Samples (from Table 7.6):

SiO₂ = 4.04% ✓
CaO = 1.11% ✓
Al₂O₃ = 2.92% ✓

p. 5-8: Glassformer analysis (Table 5.3)

SiO₂ = 55.43 ± 1.35% ✓
CaO = 5.61 ± 0.27% ✓
Al₂O₃ = 17.97 ± 1.02% ✓

p. 5-21: Simulant analysis (Table 5.11)

SiO₂ = 0 ✓
CaO = 0.46 g/L ✓
Al₂O₃ = 56.98 g/L ✓

p. 5-19: Average simulant flow rate = 3.755 L/min ✓

p. 6-9: Average glassformer feed rate ≈ 3.95 kg/min ✓

Calculations:

Total missing oxide mass = 8383 - 7372 = 1011 kg ✓

Estimated amount of CaO in missing mass = (1011 kg) (0.011) = 11.22 kg CaO ✓

Amt of CaO fed to melter = (0.0561) (3.95 $\frac{\text{kg}}{\text{min}}$) (1540 min) + (0.00046 $\frac{\text{kg}}{\text{min}}$) (3.755 $\frac{\text{L}}{\text{min}}$) (1540 min)
= 343.9 kg CaO fed ✓

∴ % CaO entrained = $\frac{11.22 \text{ kg}}{343.9 \text{ kg}} \times 100\% = 3.26\%$ ✓

(assuming zero CaO volatility)

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Title WSTC entrainment estimate		Project LLW vit.	
Prepared by M.R. Powell A.H. Towell	Date 8/21/95	Reviewed by Gregory Whiggatt	Date 9/23/95

(Cont.):

Similarly for SiO_2 and Al_2O_3 ,

$$\text{SiO}_2 \text{ entrained} = (1011 \text{ kg})(.0404) = 40.84 \text{ kg}$$

$$\text{SiO}_2 \text{ fed} = (.5543)(3.95)(1540) = 3372 \text{ kg}$$

$$\% \text{ SiO}_2 \text{ Entrained} = \frac{40.84}{3372} \times 100 = \underline{\underline{1.21 \%}}$$

$$\text{Al}_2\text{O}_3 \text{ entrained} = (1011 \text{ kg})(.0292) = 29.5 \text{ kg}$$

$$\text{Al}_2\text{O}_3 \text{ fed} = (.1797)(3.95)(1540) + (.05698)(3.755)(1540) = 1422.6 \text{ kg}$$

$$\% \text{ Al}_2\text{O}_3 \text{ Entrained} = \frac{29.5}{1422.6} = \underline{\underline{2.08 \%}} \quad \checkmark$$

Note that some significant problems have been noted with some of the SiO_2 analyses. For this reason it is judged more likely that the CaO and Al_2O_3 entrainment estimates are accurate.

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Title WSTC entrainment estimate		Project LLW V.I.T.	
Prepared by: M. R. Powell <i>[Signature]</i>	Date 8/21/95	Reviewed by: <i>[Signature]</i>	Date 9/23/95

WSTC Averaging of Volatile Losses from Spreadsheets (non-linked, copy-paste special)						
oxide	snap 1	snap 2	snap 3	snap 4	avg	
Al ₂ O ₃	-0.05142	0.009849	-0.05445	-0.00195	-0.02449	
B ₂ O ₃	0.308476	0.143535	0.219128	0.189858	0.215249	
BaO	0.301708	0.0074	-0.11608	0.038783	0.057953	
CaO	0.046629	-0.01005	0.049103	0.00194	0.021906	
Cr ₂ O ₃	0.130415	0.242449	-0.01051	0.096615	0.114742	
Cs ₂ O	0.833688	0.829904	0.811631	0.877869	0.838273	
Fe ₂ O ₃	-0.27871	-0.13047	-0.20136	-0.66357	-0.31853	
K ₂ O	0.433001	0.477308	0.429249	0.590225	0.482445	
Li ₂ O	0.01725	0.045006	0.07178	-0.02358	0.027613	
MgO	-0.14202	-0.03947	-0.10176	-0.23173	-0.12874	
MnO ₂	0.298794	0.416997	0.246525	-0.0716	0.222679	
MoO ₃	0.154464	0.215962	0.184925	0.387656	0.235752	
Na ₂ O	0.025903	0.167959	0.097619	0.313914	0.151349	
NiO	0.68552	0.686385	0.689746	0.7321	0.698437	
P ₂ O ₅	0.380867	0.561919	0.408577	0.363737	0.428775	
SO ₃	0.371946	0.428462	-0.40086	0.230171	0.157429	
SiO ₂	0.140758	0.091752	0.115251	0.092388	0.110037	
SrO	-0.11815	0.041731	-0.08827	0.162811	-0.00047	
TiO ₂	-1.20136	-1.50892	-3.42113	-2.01388	-2.03632	
ZrO ₂	0.00468	-0.00822	0.017357	-0.03837	-0.00614	
Cl*	0.883326	0.868023	0.849433	0.900524	0.875327	
F	0.892696	0.883118	0.922849	0.941508	0.910043	

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Reviewed
 George H. Hight 3/27/12

WHC-SD-WM-ER-517
 Revision 0

MR Powell 8/19/95								
WSTC Data: Estimated Oxide Volatilities based on glass and feed compositions and tie components								
Snapshot #1 taken 12/07/94 at 17:00								
	Average	Average		Tie =	Tie =	Tie =	Tie =	Avg of Ca
	Glass	Feed		SiO2	Fe2O3	CaO	Al2O3	and Al2O3
				Vi	Vi	Vi	Vi	
Al2O3	19.425	14.525		-0.22367	0.177743	-0.10285	0	-0.05142
B2O3	6.665	7.5775		0.195192	0.4592	0.274654	0.342298	0.308476
BaO	1.0525	1.185		0.187316	0.453907	0.267555	0.335861	0.301708
CaO	4.32	3.5625		-0.10955	0.254424	0	0.093257	0.046629
Cr2O3	0.0365	0.033		-0.01204	0.319949	0.087884	0.172946	0.130415
Cs2O	0.0275	0.13		0.806444	0.869937	0.825554	0.841823	0.833688
Fe2O3	0.7075	0.435		-0.48818	0	-0.34125	-0.21616	-0.27871
K2O	0.97	1.345		0.340116	0.556583	0.405269	0.460732	0.433001
Li2O	0.8375	0.67		-0.14374	0.231449	-0.03082	0.065315	0.01725
MgO	0.08425	0.058		-0.32911	0.10689	-0.19788	-0.08617	-0.14202
MnO2	0.00825	0.00925		0.183925	0.451628	0.264499	0.33309	0.298794
MoO3	0.1425	0.1325		0.015951	0.338756	0.113109	0.195818	0.154464
Na2O	19.7	15.9		-0.13367	0.238216	-0.02174	0.073545	0.025903
NiO	0.004	0.01		0.634003	0.754064	0.670139	0.700901	0.68552
P2O5	0.1575	0.2		0.279443	0.515813	0.350586	0.411149	0.380867
SO3	0.1525	0.1909		0.26906	0.508836	0.341228	0.402664	0.371946
SiO2	37.35	34.175		0	0.328038	0.098734	0.182783	0.140758
SrO	0.16	0.1125		-0.30132	0.125559	-0.17284	-0.06346	-0.11815
TiO2	0.028	0.01		-1.56198	-0.72155	-1.30903	-1.09369	-1.20136
ZrO2	2.1775	1.72		-0.15837	0.221618	-0.044	0.05336	0.00468
Cl	0.052	0.3504		0.864213	0.908756	0.87762	0.889033	0.883326
F*	0.04	0.293075		0.875118	0.916084	0.887448	0.897944	0.892696
Fluoride feed value is based on a feed target F:Cl ratio of 0.8364 due to analytical difficulties.								

Revised
by J. H. J. 3/27/96

WHC-SD-WM-ER-517
Revision 0

MR Powell 8/19/95								
WSTC Data: Estimated Oxide Volatilities based on glass and feed compositions and tie components								
Snapshot #2 taken on 12/08/94 at 01:00								
	Average	Average	Tie =	Tie =	Tie =	Tie =		Avg. of Ca
	Glass	Feed	SiO ₂	Fe ₂ O ₃	CaO	Al ₂ O ₃		and Al ₂ O ₃
			Vi	Vi	Vi	Vi		
Al ₂ O ₃	18.375	14.55	-0.09018	0.124127	0.019697	0		0.009849
B ₂ O ₃	7.9525	7.28	0.057015	0.242384	0.152054	0.135016		0.143535
BaO	1.285	1.015	-0.09287	0.121961	0.017273	-0.00247		0.0074
CaO	4.335	3.365	-0.11208	0.106528	0	-0.02009		-0.01005
Cr ₂ O ₃	0.03575	0.037	0.165921	0.329882	0.249984	0.234915		0.242449
Cs ₂ O	0.032	0.1475	0.81272	0.849535	0.831595	0.828212		0.829904
Fe ₂ O ₃	0.62	0.43	-0.24467	0	-0.11923	-0.14172		-0.13047
K ₂ O	0.97	1.455	0.424505	0.537634	0.482507	0.472109		0.477308
Li ₂ O	0.81	0.665	-0.05147	0.155227	0.054505	0.035507		0.045006
MgO	0.07325	0.05525	-0.14448	0.080499	-0.02913	-0.04981		-0.03947
MnO ₂	0.00725	0.00975	0.358102	0.484285	0.422796	0.411198		0.416997
MoO ₃	0.1325	0.1325	0.136758	0.306452	0.22376	0.208163		0.215962
Na ₂ O	18.2	17.15	0.083906	0.263989	0.176235	0.159683		0.167959
NiO	0.004	0.01	0.654703	0.722581	0.689504	0.683265		0.686385
P ₂ O ₅	0.11175	0.2	0.517663	0.61248	0.566276	0.557561		0.561919
SO ₃	0.1525	0.2092	0.370725	0.494426	0.434146	0.422777		0.428462
SiO ₂	37.475	32.35	0	0.196577	0.100785	0.082718		0.091752
SrO	0.1375	0.1125	-0.05507	0.15233	0.051262	0.0322		0.041731
TiO ₂	0.032	0.01	-1.76237	-1.21935	-1.48397	-1.53388		-1.50892
ZrO ₂	2.125	1.6525	-0.11007	0.108145	0.001809	-0.01825		-0.00822
Cl	0.054	0.3208	0.854691	0.883256	0.869336	0.866711		0.868023
F*	0.04	0.268317	0.87131	0.896608	0.88428	0.881955		0.883118
Fluoride feed value is based on a feed target F:Cl ratio of 0.8364 due to analytical difficulties.								

MHC-SD-WM-ER-517
Revision 0

MR Powell 8/19/95								
WSTC Data: Estimated Oxide Volatilities based on glass and feed compositions and tie components								
Snapshot #3 taken on 12/08/94 at 09:05								
	Average	Average	Tie =	Tie =	Tie =	Tie =		Avg. of Ca
	Glass	Feed	SiO2	Fe2O3	CaO	Al2O3		and Al2O3
			Vi	Vi	Vi	Vi		
Al2O3	17.775	13.075	-0.19181	0.122287	-0.1089	0		-0.05445
B2O3	7.4575	7.4075	0.117409	0.350011	0.178805	0.259451		0.219128
BaO	2.5325	1.76	-0.26146	0.070988	-0.17371	-0.05845		-0.11608
CaO	3.92	3.1975	-0.07476	0.208484	0	0.098205		0.049103
Cr2O3	0.04625	0.0355	-0.14215	0.158861	-0.06269	0.041669		-0.01051
Cs2O	0.034	0.14	0.787094	0.843204	0.801904	0.821358		0.811631
Fe2O3	0.515	0.3325	-0.35786	0	-0.2634	-0.13933		-0.20136
K2O	1.0725	1.4575	0.3549	0.524913	0.399776	0.458721		0.429249
Li2O	0.73	0.61	-0.04913	0.22736	0.023848	0.119711		0.07178
MgO	0.0625	0.044	-0.24528	0.08291	-0.15865	-0.04486		-0.10176
MnO2	0.0085	0.00875	0.148374	0.372816	0.207617	0.285433		0.246525
MoO3	0.155	0.1475	0.07875	0.32154	0.142836	0.227014		0.184925
Na2O	20.825	17.9	-0.01993	0.248868	0.051021	0.144216		0.097619
NiO	0.004	0.01	0.649331	0.741748	0.673724	0.705767		0.689746
P2O5	0.1525	0.2	0.331536	0.507706	0.378037	0.439117		0.408577
SO3	0.19	0.1052	-0.58335	-0.16606	-0.4732	-0.32853		-0.40086
SiO2	35.475	31.1	0	0.263545	0.069564	0.160938		0.115251
SrO	0.1375	0.098	-0.23003	0.09414	-0.14446	-0.03207		-0.08827
TiO2	0.057	0.01	-3.99704	-2.6801	-3.64943	-3.19283		-3.42113
ZrO2	1.9225	1.5175	-0.11065	0.182059	-0.03338	0.068099		0.017357
Cl	0.07	0.3606	0.829819	0.87467	0.841658	0.857208		0.849433
F*	0.03	0.301606	0.912799	0.935781	0.918865	0.926833		0.922849
Fluoride feed value is based on a feed target F:Cl ratio of 0.8364 due to analytical difficulties								

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Reviewed by NG 3/27/96

WMC-SD-MM-ER-517
 Revision 0

WSTC#4

MR Powell 8/19/95							
WSTC Data: Estimated Oxide Volatilities based on glass and feed compositions and tie components							
Snapshot #4 taken on 12/08/94 at 15:00							
	Average Glass	Average Feed	Tie = SiO ₂ Vi	Tie = Fe ₂ O ₃ Vi	Tie = CaO Vi	Tie = Al ₂ O ₃ Vi	Avg. of Ca and Al ₂ O ₃
Al ₂ O ₃	18.7	12.5	-0.10394	0.397714	-0.00389	0	-0.00195
B ₂ O ₃	7.17	5.9275	0.107393	0.513012	0.188284	0.191433	0.189858
BaO	1.55	1.08	-0.05906	0.422198	0.036915	0.040652	0.038783
CaO	3.99	2.6775	-0.09965	0.400051	0	0.00388	0.00194
Cr ₂ O ₃	0.058	0.043	0.004658	0.456962	0.094859	0.098371	0.096615
Cs ₂ O	0.031	0.17	0.865437	0.926585	0.877632	0.878106	0.877869
Fe ₂ O ₃	0.77	0.31	-0.83291	0	-0.66681	-0.66034	-0.66357
K ₂ O	1.06	1.7325	0.548513	0.753678	0.589428	0.591021	0.590225
Li ₂ O	0.81	0.53	-0.12777	0.38471	-0.02557	-0.02159	-0.02358
MgO	0.08	0.0435	-0.35711	0.259591	-0.23412	-0.22933	-0.23173
MnO ₂	0.014	0.00875	-0.18068	0.355844	-0.07368	-0.06952	-0.0716
MoO ₃	0.16	0.175	0.325325	0.631911	0.386466	0.388846	0.387656
Na ₂ O	21	20.5	0.244076	0.587583	0.31258	0.315247	0.313914
NiO	0.004	0.01	0.70483	0.838961	0.731579	0.73262	0.7321
P ₂ O ₅	0.19	0.2	0.29897	0.617532	0.3625	0.364973	0.363737
SO ₃	0.19	0.1653	0.151809	0.537244	0.228675	0.231668	0.230171
SiO ₂	35.2	25.975	0	0.45442	0.090624	0.094152	0.090388
SrO	0.15	0.12	0.077592	0.496753	0.161184	0.164439	0.162811
TiO ₂	0.045	0.01	-2.32067	-0.81169	-2.01974	-2.00802	-2.01388
ZrO ₂	2	1.29	-0.14407	0.375818	-0.04039	-0.03636	-0.03837
Cl	0.061	0.4107	0.890398	0.940203	0.900331	0.900717	0.900524
F*	0.03	0.343509	0.935554	0.96484	0.941394	0.941622	0.941508
Fluoride feed value is based on feed target F:Cl ratio of 0.8364 due to analytical difficulties							

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WMC-SD-WM-ER-517
 Revision 0

Reviewed: *Eng. W. H. H. H.*
 5/27/96

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APPENDIX C

REVIEW OF UNITED STATES BUREAU OF MINES TEST REPORT, RUN WHC-1

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DON'T SAY IT -- Write It!

Date: April 28, 1995

To: Will Eaton

From: Mike Powell

Subject: Review of USBM test data

I examined the USBM preliminary report along with the recently-obtained sample analytical data. My analyses focused on the data gathered during the 24-hour demonstration test. The following conclusions can be drawn:

Total Entrainment/Volatility:

The combined entrainment and volatility amounts to $8.7 \pm 2.5\%$ of the theoretical glass mass. An estimate of $4.0 \pm 2.5\%$ is obtained if the data are not corrected for the unintended addition of Treehugger feed (estimated to be 540 lbs). Individual component mass balances are given in Tables 1a and 1b. Table 1a has been corrected for the Treehugger feed addition (and gives 8.7% loss), while Table 1b has not (giving 4% loss). Based on the mass of silica collected in the off-gas system (see Table 3), the entrainment amounts to 1.2% of the theoretical glass mass. The overall mass balance approach (Table 1a) indicates a $1.7 \pm 3.5\%$ loss of silica. The 1.2% entrainment estimate is believed to be more accurate (replicate silica analyses for the off-gas solids are more consistent than for the combined feed and glass analyses). However, if off-gas solids accumulations were not fully recovered and weighed, then the 1.2% entrainment estimate will be too low.

Apparent Addition of Treehugger Feed:

The glass composition data indicate a net gain of CaO during the 24 hour test. The mass of CaO gained by the glass is estimated to be 242 ± 95 lbs (see Table 1b). It is inferred from the concomitant increase in TiO_2 concentration that approximately 540 lbs of "Treehugger" feed became mixed with the WHC glass during the 24 hour test. The Treehugger feed had apparently accumulated on the melter refractory above the glass surface level during an earlier test run. The peak in CaO, Al_2O_3 , and TiO_2 concentrations observed during the 24 hour run (see plots 1 and 2) can be adequately accounted for by contamination of the WHC glass with Treehugger glass. (see handwritten calculations attached). A significant fraction of the elevation in alumina concentration may have been due to refractory degradation, but this is not certain. The concentrations of CaO and TiO_2 both imply an addition of about 540 lbs of Treehugger feed. If this number is accurate, then 180 lbs of additional alumina would be present in the WHC glass. The feed and glass composition data (Table 1b) show that the additional alumina was 221 ± 87 lbs. Because the predicted 180 lbs alumina is well within the 134-308 lb range given by this ± 87 lb confidence interval, it is not known whether refractory degradation made a significant contribution to the elevated alumina concentrations. It may be possible to determine the refractory contribution (roughly) if we can get Cr_2O_3 composition data for both the Treehugger feed and the refractory.

Calculation of Fractional Volatilities:

The fractional volatilities of the glass components were estimated based on the ratios of glass and feed oxide concentrations and the assumption of zero SiO_2 volatility. These data are presented in Tables 2a and 2b. The data in Table 2a have been corrected for the effects of the Treehugger feed on the SiO_2 , CaO , Al_2O_3 , and TiO_2 concentrations (see attached handwritten calculations for correction procedure). The Table 2b data have not been corrected. The data in Table 2a are more accurate. Table 2b has been included to show the effect of not correcting for the Treehugger feed addition. Upon applying the correction for the Treehugger feed effects, Na_2O volatility was estimated to be $20.5 \pm 5.2\%$, B_2O_3 volatility was $50.7 \pm 4.4\%$, and K_2O volatility was $35.3 \pm 4.5\%$. Negative volatilities were calculated for Al_2O_3 , CaO , Cr_2O_3 , Fe_2O_3 , MgO , TiO_2 , and ZrO_2 . It is believed that most of these negative volatilities result from the addition of the Treehugger feed to the WHC glass. Al_2O_3 , CaO , Cr_2O_3 , MgO , and TiO_2 glass concentrations all peak at near 1150 minutes into the 24-hour test (see attached plots 1 and 2). The negative ZrO_2 volatility probably results from analytical errors because of the low ZrO_2 concentration in the USBM glass. Regardless, all of the computed negative volatilities for these oxides are within their respective 95% confidence intervals of zero (with the exception of ZrO_2 and MgO). It may be possible to correct the ZrO_2 and MgO volatilities if we can get a more complete characterization of the Treehugger feed. The 2 wt% "other" in the given Treehugger composition could easily account for the observed inconsistencies in ZrO_2 and MgO concentrations.

The cause of the negative Fe_2O_3 volatility is not known. It is worth noting, however, that the average Fe_2O_3 concentration in the feed was within 1% of the target concentration, but the average Fe_2O_3 concentration in the glass was about 12% higher than the target glass concentration. This implies that there may have been an iron source within the melter. Analytical errors may also be contributing to this discrepancy. The Fe_2O_3 concentration for the glass does not show a "peak" at about 1150 minutes (see plot 1), so it is unlikely that this additional ferric oxide can be attributed to the Treehugger feed.

Loss Estimates Based on Collected Off-Gas Solids:

Using the reported masses of off-gas solids collected during the test and the reported compositions of those solids, % loss estimates were generated (Table 3). A weighted-average of all the % loss estimates gives an overall entrainment/volatility loss average of 5.3% for the 24 hour run. I haven't had time to complete an error analysis on this 5.3% estimate. I suspect that the confidence interval of the 5.3% estimate will overlap with the $8.7 \pm 2.5\%$ estimate given in Table 1a. For now, I recommend using the 8.7% estimate. It should also be pointed out that the values used for the total Baghouse, Cyclone, and Thermal Oxidizer solids masses are only estimated values. The solids were measured at the end of the test and then it was assumed that since the 24-hour run constituted 59% of the total melter run time then 59% of the solids from each of the collection points must have been collected during the 24-hour run. The extent to which this assumption is valid can have a dramatic effect on the 5.3% loss

estimate. If all the collected solids were produced solely during the 24-hour run, then the % loss estimate increases to about 9.0% (this is an unlikely scenario, though).

The lowest % loss values calculated in Table 3 were for SiO_2 (1.19%) and Fe_2O_3 (1.23%). All others were in excess of 2%. This supports the use of SiO_2 as the tie component for the volatility estimates described above. If any of the components had a lower volatility than silica, it would be expected that the % loss data for those components would be lower than that calculated for silica. Further, the composition of the thermal oxidizer solids (also shown in Table 3) implies that the SiO_2 entrainment/volatility was less than that of Al_2O_3 and CaO. The thermal oxidizer solids concentrations of alumina and lime are higher (relative to silica and ferric oxide) than would be expected from entrainment alone. It is likely that the elevated level of CaO is due to entrainment of Treehugger feed or lime. However, it is expected that most of the enrichment in Al_2O_3 is the result of corrosion of the refractory in the thermal oxidizer which consisted of 80 wt% Al_2O_3 .

Review of Feed Pellet Preparation Procedures:

The Operating Procedure for Feed Preparation was examined in an effort to determine why the prepared feed pellets did not meet target specifications in some cases. Table 4 (attached) shows a comparison of the average feed composition (with 95% confidence interval in the adjacent column) with the target feed composition. The sodium and potassium concentrations in the feed (and in the resulting glass) were significantly lower than the targets. I reviewed the data given in Tables 4 and 5 of the Operating Procedure and did not find any errors in the procedure used to formulate the simulant recipe. It is possible that the procedure was not followed accurately or maybe there was a calibration problem with their scale. It may be useful to verify the stated glass-former and LLW simulant compositions given in Table 4 of the Operating Procedure. I believe that the specified simulant recipes should have given feed compositions near the targets, so the problem is likely elsewhere. Could something have happened to the sodium and/or potassium in the dryer during denitrification? I can't think of any reasonable possibilities, but that doesn't mean there aren't any.

Data Sources for USBM Calculations

This page is intended to aid in the traceability of the data used within this appendix.

Table 2a, 2b Data Sources

Glass average composition and standard deviation are derived from the following glass samples:
M1G1-002C, 002U, 004C, 004U, 005C, 005U, 008C, 008U, 010C, 010U, 011U, 011C

Feed average composition and standard deviation are derived from the following feed samples:

M1APR-001C, -001U, -002U

M1BPR-022C, -022U, -026U, -043U, -050U

Table 3 data sources

samples analyzed include (average of samples shown)

Baghouse M1B5-001C, -001U

Cyclone M1C4 -001C, -001U

Thermal Oxidizer M1T3-001C, -001U

Masses Collected in Baghouse, cyclone and thermal oxidizer were initially calculated from the mass of material recovered differ slightly from values given in the report. This is most likely due to differences in the conversion from the sample quantity to the quantity associated with the 24 hour run period. The differences are not deemed significant enough to warrant revision to the calculations.

	Values Used	Page 3-22 of Final Report
Baghouse	631	645
Cyclone	45	40
Thermal Oxidizer	94	97
Feed	12954	12954

Table 4

Feed Targets are taken from Table 1.1 of test plan.

Note: .8839 from LOI sample M1BPR-022P

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Table 1a

Reviewed Greg Whyatt 10/3/95

USBM Data: Estimated Oxide Volatilities based on glass and feed compositions and tie components

Average Glass Composition Data corrected for Treehugger feed addition

	Average				Tie =				Tie =				Tie =							
	Glass		95% CI		Average Feed		95% CI		SiO2		95% CI		Fe2O3		95% CI		CaO		95% CI	
									Vi	95% CI	Vi	95% CI	Vi	95% CI	Vi	95% CI	Vi	95% CI	Vi	95% CI
Al2O3	11.39	0.717474	8.84125	0.290562					-0.0538	0.08341	-0.00858	0.132247	-0.03828	0.183318						
B2O3	2.712057	0.220574	4.5025	0.054876					0.507286	0.044015	0.528432	0.064844	0.514547	0.088038						
BaO	0.006885	0.000809	0.006388	0.00048					0.118242	0.126785	0.156083	0.150025	0.131235	0.185483						
CaO	5.354	0.852913	4.315	0.117996					-0.01496	0.167828	0.028602	0.190033	0	0.228585						
Cr2O3	0.048084	0.013526	0.029875	0.012333					-0.31657	0.659288	-0.26007	0.644597	-0.29717	0.681072						
Cs2O	0.050248	0.005326	0.11	0.12706					0.626342	0.433618	0.642378	0.41669	0.631848	0.431162						
Fe2O3	1.16875	0.071766	0.915	0.083735					-0.04484	0.120778	0	0.155853	-0.02944	0.201389						
K2O	0.971429	0.04538	1.22875	0.047603					0.353309	0.045268	0.381062	0.077869	0.362838	0.110007						
Li2O	0	0	0	0										#DIV/0!						
MgO	0.329107	0.022038	0.21625	0.016076					-0.24489	0.131907	-0.19146	0.177346	-0.22655	0.233159						
MnO2	0.01133	0.001559	0.010625	0.002232					0.127703	0.221147	0.165139	0.228944	0.140557	0.256659						
MoO3	0.084357	0.005569	0.130875	0.008462					0.472752	0.052075	0.49538	0.072573	0.480521	0.096717						
Na2O	16.10893	0.577606	16.575	0.707738					0.205008	0.052287	0.239126	0.093973	0.216723	0.133925						
NiO	0.002294	0.000745	0.007025	0.002049					0.732919	0.116969	0.744381	0.115094	0.736855	0.1225						
P2O5	0.087196	0.01002	0.12025	0.026312					0.406852	0.148048	0.432308	0.15362	0.415592	0.172581						
SO3	0.21136	0.091888	1.41875	0.094223					0.878139	0.053762	0.883368	0.052879	0.879934	0.056257						
SiO2	60.407	1.063608	49.4125	1.488016					0	0.049333	0.042916	0.110633	0.014735	0.162919						
SrO	0.096929	0.0027	0.082625	0.003221					0.040401	0.056871	0.081583	0.110365	0.054541	0.159391						
TiO2	0.1536	0.103885	0.12	0.009994					-0.04703	0.714426	-0.0021	0.691744	-0.0316	0.722483						
ZrO2	0.008227	0.001122	0.0045	0.000632					-0.49553	0.297359	-0.43135	0.321536	-0.47349	0.374055						
Carbon	0.027143	0.013699	2.765	0.404897					0.99197	0.004229	0.992315	0.004127	0.992088	0.00435						
Cl*	0.065821	0.01064	0.30125	0.04372					0.821273	0.039324	0.828943	0.041669	0.823907	0.047682						
F	0.029643	0.003483	0.04	0.008939					0.393809	0.154504	0.419824	0.159828	0.402741	0.179049						

Reviewed: Greg Whyatt
10/3/95

MR Powell	4/27/95										
USBM Data: Estimated Oxide Volatilities based on glass and feed compositions and tie components											
	Average		Average			Tie =		Tie =		Tie =	
	Glass	95% CI	Feed	95% CI		SiO2		Fe2O3		CaO	
						Vi	95% CI	Vi	95% CI	Vi	95% CI
Al2O3	12.47321	0.717474	8.84125	0.290562		-0.19834	0.089914	-0.1045	0.142021	0.167122	0.114057
B2O3	2.712057	0.220574	4.5025	0.054876		0.488367	0.045774	0.528432	0.064844	0.6444	0.051688
BaO	0.006885	0.000809	0.006388	0.00048		0.084383	0.131731	0.156083	0.150025	0.36362	0.117036
CaO	7.309107	0.852913	4.315	0.117996		-0.43879	0.179739	-0.32612	0.215917	0	0.169498
Cr2O3	0.048084	0.013526	0.029875	0.012333		-0.36713	0.684637	-0.26007	0.644597	0.049807	0.488134
Cs2O	0.050248	0.005326	0.11	0.12706		0.611994	0.450273	0.642378	0.41669	0.730325	0.314474
Fe2O3	1.16875	0.071766	0.915	0.083735		-0.08496	0.125529	0	0.155853	0.245921	0.122778
K2O	0.971429	0.04538	1.22875	0.047603		0.328477	0.047123	0.381062	0.077869	0.533272	0.062701
Li2O	0	0	0	0							#DIV/0!
MgO	0.329107	0.022038	0.21625	0.016076		-0.29269	0.13712	-0.19146	0.177346	0.101542	0.140272
MnO2	0.01133	0.001559	0.010625	0.002232		0.094208	0.229682	0.165139	0.228944	0.370449	0.175171
MoO3	0.084357	0.005569	0.130875	0.008462		0.452507	0.054142	0.49538	0.072573	0.619476	0.057587
Na2O	16.10893	0.577606	16.575	0.707738		0.174481	0.054447	0.239126	0.093973	0.426241	0.075844
NiO	0.002294	0.000745	0.007025	0.002049		0.722664	0.121468	0.744381	0.115094	0.807243	0.087264
P2O5	0.087196	0.01002	0.12025	0.026312		0.384076	0.153763	0.432308	0.153362	0.571915	0.117584
SO3	0.21136	0.091888	1.41875	0.094223		0.873459	0.055829	0.883368	0.052879	0.91205	0.04009
SiO2	58.17321	1.063608	49.4125	1.488016		0	0.049823	0.078308	0.106639	0.304971	0.086826
SrO	0.096929	0.0027	0.082625	0.003221		0.003553	0.059258	0.081583	0.110365	0.307441	0.089392
TiO2	0.3925	0.103885	0.12	0.009994		-1.77826	0.777068	-1.5607	0.764504	-0.93097	0.58363
ZrO2	0.008227	0.001122	0.0045	0.000632		-0.55296	0.308872	-0.43135	0.321536	-0.07935	0.247738
Carbon	0.027143	0.013699	2.765	0.404897		0.991662	0.004392	0.992315	0.004127	0.994205	0.003124
Cl*	0.065821	0.01064	0.30125	0.04372		0.81441	0.040844	0.828943	0.041669	0.871101	0.032004
F	0.029643	0.003483	0.04	0.008939		0.370532	0.160467	0.419824	0.159828	0.562502	0.122273

Reviewed: Greg Wilgatt
10/3/95

Table 3

% Loss Values Determined from Collected Off-Gas System Solids USBM Data										
Some analytes may have escaped as gases (e.g., CO2) thus invalidating the % loss estimate for that analyte.										
Note: Masses collected differ slightly from values arrived at in USBM final report. Difference is small and is likely due to differences in conversion from mass collected to mass attributable to 24-hr test period. Masses in report are 645 lb baghouse, 42 lb cyclone, 27 lb Oxidizer.										
Collected Masses during test:			631 lbs Baghouse							
			45 lbs Cyclone							
			94 lbs Thermal Oxidizer							
			12954 lbs feed during test							
Wt% Oxides			Masses Collected							
Oxide	Baghouse	Cyclone	Th. Oxidr	Baghouse	Cyclone	Th. Oxidr	Average Feed wt%	% Loss		
Al2O3	0.36	0.68	37.6	2.2716	0.306	35.344	8.84125	3.311076	0.29274	
B2O3	22.6	24.1	15.95	142.606	10.845	14.993	4.5025	28.88005	1.300324	
BaO	0.0017	0.00185	0.0055	0.010727	0.000833	0.00517	0.006388	2.021846	0.000129	
CaO	0.505	2.25	7.89	3.18655	1.0125	7.4166	4.315	2.078064	0.089668	
Cr2O3	0.027	0.0295	0.0215	0.17037	0.013275	0.02021	0.029875	5.267561	0.001574	
Cs2O	0.99	0.83	0.0055	6.2469	0.3735	0.00517	0.11	46.49719	0.051147	
Fe2O3	0.11	0.265	0.685	0.6941	0.11925	0.6439	0.915	1.229445	0.011249	
K2O	5.18	4.42	0.555	32.6858	1.989	0.5217	1.22875	22.11221	0.271704	
Li2O	0	0	0	0	0	0	0	0	0	
MgO	0.065	0.105	0.13	0.41015	0.04725	0.1222	0.21625	2.069038	0.004474	
MnO2	0.0075	0.008	0.0075	0.047325	0.0036	0.00705	0.010625	4.21219	0.000448	
MoO3	0.4765	0.4325	0.097	3.006715	0.194625	0.09118	0.130875	19.42083	0.025417	
Na2O	32.1	31.7	21.4	202.551	14.265	20.116	16.575	11.03485	1.829026	
NiO	0.00575	0.00655	0.00215	0.036283	0.002948	0.002021	0.007025	4.532985	0.000318	
P2O5	0.509	0.4975	0.3575	3.21179	0.223875	0.33605	0.12025	24.21307	0.029116	
SO3	13.05	10.35	0.062	82.3455	4.6575	0.05828	1.41875	47.3713	0.67208	
SiO2	9.62	9.425	12.1	60.7022	4.24125	11.374	49.4125	1.192293	0.589142	
SrO	0.0025	0.006	0.0305	0.015775	0.0027	0.02867	0.082625	0.440474	0.000364	
TiO2	0.0125	0.0175	1.2025	0.078875	0.007875	1.13035	6.12	7.829628	0.009396	
ZrO2	0.0025	0.0025	0.1105	0.015775	0.001125	0.10387	0.0045	20.71775	0.000932	
Carbon	1.56	1.73	0.05	9.8436	0.7785	0.047	2.765	2.978714	0.082361	
Cl*	1.99	1.4975	0.01	12.5569	0.673875	0.0094	0.30125	33.92835	0.102209	
F	0.1925	0.18	0.015	1.214675	0.081	0.0141	0.04	25.27742	0.010111	
Total =	89.36745	88.5344	98.28715	563.9086	39.84048	92.38992	91.15341		Wtd avg=	5.373931 % lost
* USGS and Corning Chloride data disagree for baghouse and cyclone samples by about a factor of 10										
Carbon = 5.29%										

Reviewed: *Greg Whyatt*
10/3/95

WIC-SD-MW-ER-517
Revision 0

MR Powell 4/27/95				
Comparison of Target and Actual average Feed Compositions				
USBM Data				
	Average		Feed	Target
	Feed	95% CI	Target	Met?
Al ₂ O ₃	8.84125	0.290562	8.968047	Y
B ₂ O ₃	4.5025	0.054876	4.508773	Y
BaO	0.006388	0.00048	0	
CaO	4.315	0.117996	4.512308	L
Cr ₂ O ₃	0.029875	0.012333	0.035356	Y
Cs ₂ O	0.11	0.12706	0.127282	Y
Fe ₂ O ₃	0.915	0.083735	0.921907	Y
K ₂ O	1.22875	0.047603	1.447828	L
Li ₂ O	0	0	0	
MgO	0.21625	0.016076	0.279312	L
MnO ₂	0.010625	0.002232	0.001768	H
MoO ₃	0.130875	0.008462	0.129049	Y
Na ₂ O	16.575	0.707738	18.06603	L
NiO	0.007025	0.002049	0	
P ₂ O ₅	0.12025	0.026312	0.188271	L
SO ₃	1.41875	0.094223	0.219207	H
SiO ₂	49.4125	1.488016	47.33018	H
SiO	0.082625	0.003221	0.091926	L
TiO ₂	0.12	0.009994	0	
ZrO ₂	0.0045	0.000632	0	
Carbon	2.765	0.404897	0	
Cl	0.30125	0.04372	0.304062	Y
F	0.04	0.008939	0.254563	L
Feed target estimated using target glass composition data given in Table 1.1 of test plan. Target glass concentrations were multiplied by 0.8839 to account for mass loss upon melting.				

Note: feed targets from Table 1.1 are for 4 pellets while .8839 is LOI for 8 pellets. Discrepancy is small however. *SW*

Table 4

USBM Data

Normalized using Target Concentrations

USBM Data

Normalized using Target Concentrations

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CALCULATION WORKSHEET

Calc. No. _____

Page _____ of _____

During CaO spike,

TiO_2 increased from target $\approx .214 \text{ wt\%}$ to $.763 \text{ wt\%}$

CaO increased from target = $5.105 \text{ wt\%}$ to $10.35 \text{ wt\%}$

Al_2O_3 increased from target = $10.146 \text{ wt\%}$ to $15.05 \text{ wt\%}$

SiO_2 decreased from initial $\approx 59.7 \text{ wt\%} \pm 2$ to $55.3 \text{ wt\%}$

Treehugger composition was approximately

15 wt% SiO_2

45 % CaO

33 % Al_2O_3

5 % TiO_2

98%

Let x = mass of treehugger added to glass to get the "peak" concentrations.

Using CaO as basis gives:

$$\frac{5.105 + .45x}{100 + x} = .1035 \Rightarrow x = 15.14 \frac{\text{g treehugger}}{100 \text{ g wt\% glass}}$$

Using $x = 15.14$ to estimate expected TiO_2 concentration,

$$\text{wt\% TiO}_2 = \frac{.214 + (.05)(15.14)}{115.14} \times 100\% = 0.84 \text{ wt\%}$$

Using TiO_2 as basis gives:

$$\frac{.214 + (.05)x}{100 + x} = .00763 \Rightarrow x = 13.0 \frac{\text{g treehugger}}{100 \text{ g wt\% glass}}$$

Using $x = 13.0$ to estimate CaO conc gives:

$$\text{wt\% CaO} = \frac{5.105 + (.45)(13)}{113} \times 100\% = 9.7 \text{ wt\%}$$

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Title

Treehugger Correction

Project

LLW V-1

Prepared by:

MR Powell M. Farn

Date

10/3/95

Reviewed by:

Greg Whiggett

Date

10/3/95

Assuming TiO_2 estimate for x is best, should be able to estimate mass of any additional CaO .

First estimate $[Al_2O_3]$ and $[SiO_2]$ using $x=13.0$

$$[Al_2O_3] = \frac{10.146 + (13)(1.33)}{113} \times 100\% = 12.8 \text{ wt\%}$$

$$[SiO_2] = \frac{59.7 + (13)(6.15)}{113} \times 100\% = 54.6 \text{ wt\%}$$

SiO_2 estimate is within exp error of measured wt% SiO_2 .

Al_2O_3 estimate is significantly low. Therefore expect that some additional Al_2O_3 was added to the glass as a result of refractory wear.

Also a fraction of the added CaO (prob 10-20%) may have come from original CaO addition -- tough to judge these things though -- errors in analytical data and/or trechugger formulation can be significant.

Overall conclusion: Most of the CaO spike is due to trechugger material.

How much Trechugger?

Based on Table 1a,

$$242 \text{ lbs } CaO \text{ gained} \Rightarrow \frac{242}{.45} = 538 \text{ lbs trechugger} \leftarrow$$

$$\text{or } 27.4 \text{ lbs } TiO_2 \text{ gained} \Rightarrow \frac{27.4}{.05} = 548 \text{ lbs trechugger} \leftarrow$$

This would predict that Al_2O_3 gained = $(545)(.33) = 179 \text{ lbs}$

This is within the CI of the lbs of Al_2O_3 gained $\Rightarrow$ 221 ± 87 lbs Al_2O_3 gained.

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Title Trechugger correction		Project LLW V.I.R	
Prepared by: MR Powell	Date 10/3/95	Reviewed by: Greg Whigatt	Date 10/3/95

Correct volatility estimates for the unintended addition of treeshugger feed:

Need to correct the measured "Average Glass" composition data in Table 2b to remove the effects of the Treeshugger feed addition. Corrected values will be placed into Table 2a.

Al_2O_3 : Average wt% = 12.473

10954 lbs glass $\Rightarrow$ 1266.3 lbs Al_2O_3

Subtract $(540)(.32) = 172.8$ lbs Al_2O_3 from Treeshugger

$\Rightarrow$ 1138.1 lbs Al_2O_3 in WHC glass

Divide by total WHC glass = $10954 - 540$ lbs = 10414

$\Rightarrow$ Al_2O_3 wt% (corrected) = $\frac{1138.1}{10414} = 11.4$ wt% Table 2a Value

CaO :

Avg wt% = 7.309 %

$\Rightarrow$ 800.6 lbs CaO

$-(540)(.45) = 557.6$ lbs CaO

$\Rightarrow$ 5.35 wt% CaO Table 2a Value

SiO_2 :

58.173 wt% = 6372.2 lbs SiO_2

$-(540)(.15)$

6291.3 lbs SiO_2 Table 2a Value

$\Rightarrow$ 60.41 wt% SiO_2

TiO_2 :

0.3925 wt% = 42.99 lbs TiO_2

$-(.05)(540)$

16 lbs TiO_2

$\Rightarrow$ 0.1536 wt% TiO_2 Table 2a Value

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Title Treeshugger Correction

Project LLW Wt

Prepared by MR Powell me

Date 10/3/95

Reviewed by: Greg Whelan 10/3/95

Date

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APPENDIX D

REVIEW OF UNITED STATES BUREAU OF MINES TEST REPORT, RUNS WHC-2, 3

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DON'T SAY IT -- Write It!

Date: September 5, 1995

To: Will Eaton

From: Greg Whyatt

Subject: Review of WHC2 and WHC3 Run Report Data

I have reviewed the sections of the USBM final report (EDT 611877) dealing with the additional work performed by the USBM after initial phase I testing was completed. The tests are designated as WHC2 and WHC3.

WHC2

The primary change made prior to WHC2 was a reduction in the voltage and power input to the furnace. The furnace was initially full of glass and was heated from 07:35 to 11:35 after which type B feed was added and at 12:30 the furnace was tapped. The offgas exit port was plugged or impaired for much of the operation time and the test was terminated at 17:24.

The report cites increasing concentrations of sodium and boron in the product glass as evidence of significant improvements (reductions in volatility) obtained due to the lower power operation. This claim appears to be substantiated. My estimate of the volatility (discussed below) is 20-25% loss of boron and 16-23% loss of sodium for the WHC2 test, based on an assumption that the process was at steady state when the final glass sample was taken. Mike Powell had earlier estimated that the losses during the WHC1 test were 50.7 +/- 4.4% for Boron and 20.5 +/- 5.2% for sodium.

The report claims that the vaporization losses were reduced to 0.57%. Based on analysis of sample WHC2-SP3, I conclude that this claim is overly optimistic. Based only on sodium and boron loss estimates the loss in the glass mass is about 5.0 wt%. A statement is made that the off gases accounted for 11.1% of the furnace feed but no data is provided to support this. The mass balance provided does not explain how the mass of glass initially in the melter and left in the melter at the end of the run is determined. Thus, I am unable to support conclusions about losses

of volatile or semi-volatile materials based on the mass balance.

Offgas Calculations for WHC2

The glass compositions still contain evidence of the contamination by Tree-Hugger feed introduced to the furnace to start the WHC1 test. The final glass sample (WHC2-SP3P(2)) was selected as the most useful sample information to characterize the operating conditions because:

- 1) Initial heating and prior operation at higher temperatures in WHC1 likely resulted in lower concentrations of volatile components in the glass at earlier times. Thus, the earlier glass samples are believed to be less representative of the eventual steady state condition.
- 2) The analytical information is more complete than for other samples.

Based on increases in the TiO_2 , CaO and Al_2O_3 content over the target values, the glass is apparently still being contaminated with Treehugger feed material. Using the TiO_2 content, the glass composition was adjusted to remove the influence of the Treehugger contamination. This results in tie -component results that are more consistent. FeO , SiO_2 , CaO , and Al_2O_3 were used as tie components to estimate volatility. However, the most reliable results are believed to be obtained from the FeO and Al_2O_3 due to the difficulty in getting good analytical data on SiO_2 and the potential errors in CaO content resulting from the use of CaO as a fluxing agent and the fact that it is present as 45 wt% of the Treehugger feed. Calculations were performed using FeO and Al_2O_3 as tie components and for both a feed composition matching the analytical results from WHC1 method B feed and also based on the feed target values from the testplan. This results in four different volatile loss values. The range of values obtained is provided in Table 1. Note that the ranges provide the range of values obtained for different tie component and feed composition assumptions and do not represent a confidence interval. All results are based on only a single glass analysis result.

Table 1. Volatility loss estimates for WHC2 sample WHC2-SP3

Oxide Component	Minimum Percent Loss ^a	Maximum Percent Loss ^a
Al ₂ O ₃	-6	0
B ₂ O ₃	20	25
BaO	-219	-209
CaO	-8.8	-2.5
Cr ₂ O ₃	-91	-68
FeO	0	5.8
K ₂ O	-156	-130
MgO	-42	-50
MnO	-856	-517
MoO ₃	47	53
Na ₂ O	16	23
P ₂ O ₅	-50	-34
SO ₃	-318	-288
SiO ₂	10	16
SrO	-10	1.6
TiO ₂ ^b	4.3	7.3
ZrO ₂ ^b	-91	-85

^a Negative Values indicate a gain (i.e. presence in excess of expected based on feed analysis composition and test plan target values.)

^b These components do not have test plan target compositions so the range of values is based only on the feed analysis values.

Volatility of boron was reduced substantially from the WHC1 test. The effect on sodium volatility was less obvious. A number of the components in Table 1 show negative values which indicate increases above the amount present in the feed. Some such as Cr_2O_3 may be due to refractory corrosion and contamination from the Treehugger feed. Others such as Ba, Mn, and ZrO_2 are present in such small quantities in the glass that a small amount of contamination relates to a large percentage change. However, MgO and SO_3 are roughly 0.25% of oxides in the feed and show significant increases from unknown sources. The most unusual component was the K_2O which has a feed target of 1.71 wt% and increased to 4.25wt% in the glass. No reason for the elevated K_2O levels is known.

WHC3

During the WHC3 test, the electrodes were made larger to reduce the energy density of the electrodes to 1/4 of the previous configuration. A lance for injection of air or oxygen was installed and failed prior to introduction of method B feed. Graphite inserts intended to control the flow in the drain were tested but failed to provide continuous tapping. The furnace was started on "about 1000 lb" of crushed INEL glass and 98 lb of carbon steel punchings. An additional 200 lb carbon steel punchings were later added to the melter. After 13 hours, the bottom drain was opened and the furnace was emptied. The remainder of the glass and metal in the melter was sufficient to plug the drain hole. Method B pellets were then added and the melter operated. As has been experienced in previous tests, the drain froze and an addition of 303 lb of CaO was needed fluidize the melt. Four glass samples were taken as well as a cyclone and baghouse sample.

ANALYSIS OF DATA

Determination of split factors through comparison of the feed and glass composition is complicated by the fact that potential tie components may have significant contamination present due to addition of CaO, iron punchings, possible INEL glass residue of unknown composition, and remaining residue from the Treehugger feed utilized in test WHC1. The analytical results from the four glass samples were adjusted to remove the excess iron and calcium content based on the SiO_2 and Al_2O_3 content. The glass composition was then normalized to estimate the glass composition without the extra calcium and iron. The results obtained using SiO_2 compare very well with the results obtained using Al_2O_3 and the two results were averaged to arrive at the glass composition without the excess CaO and FeO. No adjustments were made to the TiO_2 content although it is noted that it is high by about a factor of 2 which is likely due to contamination by the Treehugger feed material which was 5 wt% TiO_2 . After adjustment of CaO and FeO and normalization, the compositions for the four samples were evaluated using SiO_2 and Al_2O_3 as tie components and the analysis of feed data from Table 3.5 of the WHC1 report. The volatility

estimates for each component obtained using each of the two tie components were in good agreement and were averaged for each component. The results are provided in Table 1. The results are also plotted in the attached figure, which provides a convenient visualization of the volatility results.

Table 1. Estimated Loss of Oxide Components. Data is provided for each of four glass samples. Negative values indicate a gain in composition above the feed analysis.

Oxide	Mass Fraction Loss of Oxide Component				Average Percent
Component	19:55	20:42	21:19	00:42	Loss
Al ₂ O ₃	0	0.0175	0.001	0.001	0.49
B ₂ O ₃	0.19268	0.14748	0.21917	0.14804	17.68
BaO	-2.3538	-2.3499	-2.4959	-2.5392	-243.47
Cl	0.96558	0.97355	0.9862	0.97206	97.44
CaO	-0.016	-0.016	-0.016	-0.016	-1.61
Cr ₂ O ₃	-0.8294	-0.3704	-0.6685	-0.6892	-63.94
Cs ₂ O	0.36555	0.3855	0.45223	0.344	38.68
F	0.9968	0.9968	0.99332	0.99324	99.5
FeO	-0.016	-0.016	-0.016	-0.016	-1.57
K ₂ O	0.26452	0.25362	0.27015	0.22385	25.3
MgO	-0.6903	-0.5678	-0.9717	-0.7413	-74.28
MnO ₂	-5.7076	-5.6998	-7.3902	-5.7245	-613.06
MoO ₃	0.51275	0.507	0.4987	0.29215	45.27
Na ₂ O	0.0817	0.0555	0.16318	-0.041	6.48
P ₂ O ₅	0.45368	0.54526	0.57289	0.5676	53.49
SO ₃	0.9165	0.9166	0.95648	0.95594	93.64
SiO ₂	0	-0.018	0	0	-0.51
TiO ₂	-0.9913	-0.989	-1.2942	-0.8802	-103.87
ZrO ₂	-7.0491	-5.0298	-7.3902	-5.3706	-621

The glass analysis indicates quantities of BaO, Cr₂O₃, MgO, MnO₂, TiO₂ that range from 64% to 613% in excess of that in the feed. These components likely come from steel punchings, refractory corrosion, Treehugger feed and possibly INEL glass. However, the precise sources are impossible to determine. Comparison of volatility losses to the results of the WHC1 test creates some confusing observations. The volatility of B₂O₃, Na₂O, Cs₂O and K₂O was reduced significantly. The volatility of the major oxides Na₂O and B₂O₃ was reduced to about 1/3 of the volatility observed in WHC1. The volatility of Cs₂O was roughly 64% of the previous quantity and K₂O volatility was 77% of its previous rate. However, the estimates for volatility of Cl, F, SO₃ and P₂O₅ were much worse for WHC3 than in WHC1. No reason is known why the volatility of these components would be worse under the WHC3 operating conditions. Volatility of MoO₃ was roughly unchanged.

The USBM report makes several claims for the WHC3 run, among them:

1) Sodium present in glass in excess of feed

While my calculations do indicate that the final sample contained a 4% excess of sodium, I suspect that this is an analytical problem as sodium clearly deviates from other analytes which can be seen in the attached figure. I would conclude that the average of the four samples which is 6.5% loss is a better value for sodium volatility.

2) Silica present in excess of feed.

My silica values average to 0.5 wt% gain in silica which is essentially no volatility which is what would be expected.

3) Over 88% Boria reports to Glass.

I get a slightly less favorable result for Boria of 18% loss average and 15% loss if based on the last sample only. This is only a small discrepancy.

4) Over 77% Cesia reports to Glass.

I calculate a 39% loss of cesium which is somewhat greater than the <23% loss provided by USBM.

5) Over ½ of Chloride was lost.

I have a major discrepancy on this item as I calculate 97% of the chloride as being lost from the glass. How USBM reached the lower value is not known.

6) Over 90% of Fluoride was lost.

I calculate 99.5% of the fluoride lost which may be somewhat different than greater than 90% loss. Analytical difficulties have been experienced in the past on fluoride so the accuracy of this value is suspect in the absence of additional information.

The material collected from the offgas system was not used to estimate the volatile losses because the collected material in most cases represents only a small fraction of the material that was not present in the glass. For example the mass balance for B_2O_3 , indicates that only 18% of the material not reporting to the glass was recovered in the cyclone or the baghouse. The discrepancy is worse for species which may escape as a gas such as SO_3 .

Chart1

Pacific Northwest Laboratories

CALCULATION WORKSHEET

Calc. No. WHC 2

Page 1 of 5

WHC 2 spreadsheet C:\LLWGlass\Vendors\USBM\WHC2 DF.XLS

- ✓ Col. A - labels for oxides
- ✓ Col B - Target Composition based on Table 3.5 of report for WHC 1 (centered values)
- Col C - Column intentionally blank

- ✓ Col D - Target Composition based on feed composition target in test plan Table 1.3 Jan 12, 95 Test Plan

- ✓ Col E - Glass Composition of Sample WHC2-SP3P(2) From Table 4.2 of report (7/31/95)

Col F - Loss estimate based on SiO_2 tie calculation and B feed Target for Sodium (Row 20):

$$F20 = (B20 * E23 / B\$23 - E20) / (B20 * E\$23 / B\$23)$$

where B20 = Table 3.5 % Na_2O in target by feed analysis
B23 = " " % SiO_2 " "
E20 = Test Glass % Na_2O " "
E23 = Test Glass % SiO_2 " "

Col G - Same as column F except test plan rather than analysis of feed is used to set feed target values (Col D)

$$G20 = (D20 * E\$23 / D\$23 - E20) / (D20 * E\$23 / D\$23)$$

Col H Loss estimate based on FeO , otherwise same as Col F

$$H20 = (\$B20 * E\$14 / B\$14 - E20) / (\$B20 * E\$14 / B\$14)$$

where FeO = Row 14

Col I Loss estimate based on FeO (Row 14) and Test plan (Col D)

$$I20 = (\$D20 * E\$14 / D\$14 - E20) / (\$D20 * E\$14 / D\$14)$$

Col J Loss estimate based on CaO (Row 10) and feed Analysis (Col B)
* Note for convenience \$'s will be eliminated from here on *

$$J20 = (B20 * E10 / B10 - E20) / (B20 * E10 / B10)$$

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Title USBM WHC2 spreadsheet Documentation		Project LLWGLASS	
Prepared by: Gray Whynatt Gray Whynatt	Date 9/27/95	Reviewed by: Mike Pavell M Pavell	Date 9/29/95

without Technology - Adjustment

Pacific Northwest Laboratories

CALCULATION WORKSHEET

Calc. No. WHC 2

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Col K: Loss estimate based on CaO (Row 10) and Testplan feed (Col D)

$$K_{20} = (D_{20} * E_{10}/D_{10} - E_{20}) / (D_{20} * E_{10}/D_{10})$$

Col L Loss estimate based on Al_2O_3 (Row 6) and feed Analysis (Col B)

$$L_{20} = (B_{20} * E_6/B_6 - E_{20}) / (B_{20} * E_6/B_6)$$

Col M Loss estimate based on Al_2O_3 (Row 6) and Testplan (Col D)

$$M_{20} = (D_{20} * E_6/D_6 - E_{20}) / (D_{20} * E_6/D_6)$$

Col N Sample analysis adjusted for Trechugger Composition

Assume 1 kg glass product, trechugger Content is:

$$1000 \text{ g} \left(\frac{.19 - .096}{100} \text{ excess } \text{T}_2\text{O}_2 \right) \left(\frac{1 \text{ g trechugger}}{.05 \text{ g } \text{T}_2\text{O}_2} \right) = 18.8 \text{ g trechugger / kg glass}$$

where .19 % Sample analysis - $\text{H}_2\text{T}_2\text{O}_2$ Content (Col E)
.096 % feed analysis % (Col B)

Trechugger Composition is ~

- .33 Al_2O_3
- .45 CaO
- .13 SiO_2
- .05 T_2O_2

Correct masses of affected components

- $\text{Al}_2\text{O}_3 = 117.1 \text{ g} - 18.8 (.33)$
- $\text{CaO} = 65.3 - 18.8 (.45)$
- $\text{SiO}_2 = 512.2 - 18.8 (.13)$
- $\text{T}_2\text{O}_2 = 1.9 - 18.8 (.05)$

all others = 10 * Column E or if no analysis use feed target

Renormalize to 1 kg product by multiplying by $1000 / (1000 - 18.8)$

Sodium $\text{N}_{20} = E_{20} * 10 * 1000 / (1000 - 18.8)$ general formula

where 10 - convert col E to 1 kg basis (100% = 1 kg)

$$\left. \begin{aligned} \text{Al}_2\text{O}_3 &= (E_6 * 10 - 18.8 * .33) / (1000 - 18.8) \\ \text{CaO} &= (E_{10} * 10 - 18.8 * .45) / (1000 - 18.8) \\ \text{SiO}_2 &= (E_{23} * 10 - 18.8 * .13) / (1000 - 18.8) \\ \text{T}_2\text{O}_2 &= (E_{25} * 10 - 18.8 * .05) / (1000 - 18.8) \end{aligned} \right\} \text{specula}$$

Note: Composition was not normalized. If minor targets are added for non-analyzed species total $\approx 99\%$

Col O Convert from g/kg to percent 98.4?

$$O_{20} = N_{20} / 10$$

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Title USBM-WHC2		Project LLW Vitr.	
Prepared by: <i>Greg Whysatt</i>	Date 7/27/95	Reviewed by: <i>Paul</i>	Date 7/28/95

Battelle

Pacific Northwest Laboratories

CALCULATION WORKSHEET

Calc. No. WHC 2

Page 3 of 5

Col P loss estimate based on SiO_2 (Row 23) and B feed target (Column B)

$$= (B20 * 023 / B23 - 020) / (B20 * 023 / B23)$$

Col Q loss estimate based on SiO_2 (Row 23) and testplan target (Column D)

$$= (D20 * 023 / D23 - 020) / (D20 * 023 / D23)$$

Col R loss estimate based on FeO (Row 14) and feed analysis (Col B)

$$= (B20 * 014 / B14 - 020) / (B20 * 014 / B14)$$

Col S loss estimate based on FeO Row 14 and testplan (Col D)

$$= (D20 * 014 / D14 - 020) / (D20 * 014 / D14)$$

Col T loss estimate based on CaO (Row 10) and feed analysis (Col B)

$$= (B20 * 010 / B10 - 020) / (B20 * 010 / B10)$$

Col U loss estimate based on CaO (Row 10) and testplan (Col D)

$$= (D20 * 010 / D10 - 020) / (D20 * 010 / D10)$$

Col V loss estimate based on Al_2O_3 (Row 6) and feed analysis (Col B)

$$= (B20 * 006 / B06 - 020) / (B20 * 006 / B06)$$

Col W loss estimate based on Al_2O_3 (Row 6) and testplan (Col D)

$$= (D20 * 006 / D06 - 020) / (D20 * 006 / D06)$$

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Title

USBN - WHC 2

Project

Prepared by:

Greg Whigart

Date

9/27/95

Reviewed by:

Mr. Tamm

Date

9/28/95

whc2

8/3/95, pg 1/2

A	B	C	D	E	F	G	H	I	J	K	L
Oxide	B Feed	column	Testplan	WHC2	Tie=SiO2		Tie=FeO		Tie=CaO		Tie=Al2O3
Compone	Target	intentionally	B feed		Table 3.5	Theoretical	Table 3.5	Theoretical	Table 3.5	Theoretical	Table 3.5
	Table 3.5	blank	Target		B feed	Testplan	B feed	Testplan	B feed	Testplan	B feed
					Target	Target	Target	Target	Target	Target	Target
Al2O3	✓ 10.28		✓ 10	✓ 11.71	-22.09%	-25.08%	-8.94%	-11.99%	12.78%	10.30%	0.00%
B2O3	✓ 5.01		✓ 5.003	✓ 4.16	11.00%	11.18%	20.59%	20.48%	36.42%	36.31%	27.11%
BaO	✓ 0.003		✓ 0	✓ 0.01	-257.28%		-218.79%		-155.23%		-192.63%
Cl	✓ 0.38		✓ 0.344								
CaO	✓ 5		✓ 5.002	✓ 6.53	-39.98%	-39.45%	-24.90%	-24.85%	0.00%	0.00%	-14.65%
Cr2O3	✓ 0.044		✓ 0.04	✓ 0.08	-94.88%	-113.64%	-73.89%	-91.27%	-39.22%	-53.20%	-59.61%
Cs2O	✓ 0.157		✓ 0.144								
F	✓ 0.314		✓ 0.288								
FeO	✓ 0.811		✓ 0.811	✓ 0.848	-12.07%	-11.69%	0.00%	0.00%	19.94%	19.90%	8.21%
Fe2O3											
K2O	✓ 1.71		✓ 1.585	4.25	-166.39%	-186.42%	-137.69%	-156.44%	-90.30%	-105.40%	-118.19%
MgO	✓ 0.25		✓ 0.248	0.39	-67.21%	-67.98%	-49.19%	-50.40%	-19.45%	-20.46%	-36.95%
MnO2	✓ 0.003		✓ 0.002	0.02	-614.56%	-968.18%	-537.58%	-856.37%	-410.46%	-666.00%	-485.25%
MoO3	✓ 0.159		✓ 0.146	0.08	46.07%	41.47%	51.88%	47.60%	61.47%	58.03%	55.83%
Na2O	✓ 19.94		✓ 20.483	17.4	✓ 6.47%	9.26%	16.55%	18.76%	33.18%	34.93%	23.39%
P2O5	✓ 0.221		✓ 0.204	0.32	-55.20%	-67.56%	-38.48%	-50.02%	-10.87%	-20.16%	-27.11%
SO3	✓ 0.241		✓ 0.231	1.01	-349.20%	-367.04%	-300.80%	-318.15%	-220.89%	-234.92%	-267.91%
SiO2	✓ 54.9		✓ 54.712	51.22	0.00%	0.00%	10.77%	10.47%	28.56%	28.29%	18.10%
SrO	✓ 0.113		✓ 0.104	0.12	-13.82%	-23.25%	-1.56%	-10.35%	18.69%	11.62%	6.77%
TiO2	✓ 0.096		✓ 0	0.19	-112.14%		-89.28%		-51.54%		-73.75%
ZrO2	✓ 0.005		✓ 0	0.01	-114.37%		-91.27%		-53.14%		-75.58%
I	✓ 0.139		✓ 0.128								
unknown?			✓ 0.436								
Total	99.776		99.911	98.348							

D-15

WHC-SD-WM-ER-517
Revision 0

USBM WHC-2
Prepared: 9/27/95
by: [signature]
Reviewed by: [signature]
The [signature] 9/29/95

4/5

8/31/95 pg 2/2

whc2

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M

N

O

P

Q

R

S

T

U

V

W

		g/Kg glass adj for THu	TiO2 Tree-hggr	Tie=SiO2		Tie=FeO		Tie=CaO		Tie=Al2O3	
Compone	Theoretical	add minors	Adjusted	Table 3.5	Theoretical	Table 3.5	Theoretical	Table 3.5	Theoretical	Table 3.5	Theoretical
	Testplan	by target	Product	B feed	Testplan	B feed	Testplan	B feed	Testplan	B feed	Testplan
	Target	feed analy	wt%	Target	Target	Target	Target	Target	Target	Target	Target
Al2O3	0.00%	✓ 113.02	11.302	-16.18%	-19.02%	-3.23%	-6.12%	5.11%	2.41%	0.00%	0.00%
B2O3	28.99%	42.37	4.237	10.63%	10.81%	20.59%	20.48%	27.00%	26.87%	23.08%	25.07%
BaO		0.10	0.010	-258.78%		-218.79%		-193.04%		-208.81%	
Cl											
CaO	-11.48%	57.93	5.793	-22.43%	-21.96%	-8.79%	-8.74%	0.00%	0.00%	-5.38%	-2.47%
Cr2O3	-70.79%	0.81	0.081	-95.70%	-114.53%	-73.89%	-91.27%	-59.84%	-75.90%	-68.44%	-80.24%
Cs2O											
F											
FeO	10.71%	8.64	0.864	-12.54%	-12.16%	0.00%	0.00%	8.08%	8.04%	3.13%	5.77%
Fe2O3											
K2O	-128.98%	43.29	4.329	-167.51%	-187.62%	-137.69%	-156.44%	-118.50%	-135.82%	-130.25%	-141.65%
MgO	-34.29%	3.97	0.397	-67.91%	-68.68%	-49.19%	-50.40%	-37.14%	-38.30%	-44.52%	-41.72%
MnO2	-753.97%	0.20	0.020	-617.55%	-972.64%	-537.58%	-856.37%	-486.08%	-779.48%	-517.62%	-801.19%
MoO3	53.21%	0.81	0.081	45.85%	41.23%	51.88%	47.60%	55.77%	51.81%	53.39%	50.62%
Na2O	27.46%	177.23	17.723	6.08%	8.88%	16.55%	18.76%	23.29%	25.29%	19.16%	23.44%
P2O5	-33.96%	3.26	0.326	-55.85%	-68.26%	-38.48%	-50.02%	-27.29%	-37.96%	-34.14%	-41.36%
SO3	-273.38%	10.29	1.029	-351.08%	-368.99%	-300.80%	-318.15%	-268.43%	-284.53%	-288.25%	-294.03%
SiO2	20.05%	✓ 519.52	51.952	0.00%	0.00%	11.15%	10.84%	18.32%	18.01%	13.93%	15.98%
SrO	1.46%	1.22	0.122	-14.30%	-23.77%	-1.56%	-10.35%	6.64%	-1.48%	1.62%	-3.98%
TiO2		0.98	0.098	-7.70%		4.30%		12.03%		7.30%	
ZrO2		0.10	0.010	-115.27%		-91.27%		-75.82%		-85.29%	
I											
unknown?											
Total		983.75	98.38								

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WHC-SD-MM-ER-517
Revision 0

Revised: 9/6/95
by WJG
Reviewed by M. J. J. 9/26/95

US&M WHC-2

Pacific Northwest Laboratories

CALCULATION WORKSHEET

Calc. No. WHC 3

Page 1 of 8

Documentation of WHC 3 spreadsheet:

File C:\LLWGlass\vendors\USBM\WHC3 DF-B.xls

The cells are documented only where they contribute to reported values. Other calculations are information only.

✓ Col. B Table 3.5 of USBM report on WHC1, analysis of B feed (entered)

✓ Col. D Target glass composition based on test plan (entered)

✓ Col. E Analysis of WHC3-SP1BM(1) (entered)

✓ Col. F excess CaO and FeO subtracted, missing analytes Sr+I set @ test plan for normalization purposes only

$$\text{CaO} = \text{E24} \times 5.002 / 54.712$$

$$\text{FeO} = \text{E24} \times .811 / 54.712$$

Total calculated in F30

all other cells copied from col E

} values from test plan targets.
E24 = SiO₂ analysis

✓ Col. G Normalize col. F $\text{G21} = \text{F21} / \text{F30} \times 100$

✓ Col. H similar to col. F except Al₂O₃ (E7) is used instead of SiO₂ (E24)

$$\text{CaO} = \text{E7} \times 5.002 / 10$$

$$\text{FeO} = \text{E7} \times .811 / 10$$

total H30

other cells copied from col E

Sr+I set at test plan target for normalization

✓ Col. I normalize col. H $\text{I20} = \text{H20} / \text{H30} \times 100$

✓ Col. J Analysis of WHC3-SP1DM(1)

col. K similar to column F, SiO₂ basis to adjust col. J

$$\text{CaO} = \text{J24} \times 5.002 / 54.712$$

$$\text{FeO} = \text{J24} \times .811 / 54.712$$

total in K30

other cells from J

Sr+I at test plan target

✓ Col. L Normalize col. K result $\text{L20} = \text{K20} / \text{K30} \times 100$

✓ Col. M similar to col. K except Al₂O₃ basis (Row 7)

$$\text{CaO} = \text{J7} \times 5.002 / 54.712$$

$$\text{FeO} = \text{J7} \times .811 / 10$$

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Title USBM Spreadsheet Documentation		# WHC 3	Project LLW vit.	
Prepared by: Greg Whyatt	Date 9/27/95	Reviewed by: Mike Powell	Date 9/28/95

Pacific Northwest Laboratories

CALCULATION WORKSHEET

Calc. No. WHC 3

Page 2 of 8

- ✓ Col N normalize Col M $N_{20} = M_{20}/M_{30} \times 100$
- ✓ Col O Analysis of WHC3-SPI BM(1) -----
- ✓ Col P as in F, SiO_2 basis, col O
- $$CaO = 0.24 \times 5.002 / 54.712$$
- $$FeO = 0.24 \times .811 / 54.712$$
- ✓ Col Q normalize Col Z1 $Z1 = P_{21}/P_{30} \times 100$
- ✓ Col R as in H: Al_2O_3 basis, col O
- $$CaO = 0.7 \times 5.002 / 10$$
- $$FeO = 0.7 \times .811 / 10$$
- ✓ Col S normalize R : $S_{20} = R_{20}/R_{30} \times 100$ -----
- ✓ Col T analysis of WHC3-SPI BM(1) -----
- ✓ Col U as in F: SiO_2 , Col T
- $$CaO = 7.24 \times 5.002 / 54.712$$
- $$FeO = 7.24 \times .811 / 54.712$$
- ✓ Col V normalize col U : $V_{20} = U_{20}/U_{30} \times 100$
- ✓ Col W as in H: Al_2O_3 Col T
- $$CaO = 7.7 \times 5.002 / 10$$
- $$FeO = 7.7 \times .811 / 10$$
- ✓ Col X normalize col W -----
- ✓ Col AW Calculate losses for Sample WHC3-SPI BM(1) by calculating using the average of SiO_2 and Al_2O_3 normalized compositions and averaging the $SiO_2 + Al_2O_3$ & component results. For Sodium (Row 21): $AW_{21} =$
- $$\left(\left((B_{21} \times (G_{24} + I_{24})/2) / B_{24} - (G_{21} + I_{21})/2 \right) / (B_{21} \times (G_{24} + I_{24})/2 / B_{24}) \right) + \left((B_{21} \times (G_7 + I_7)/2) / B_7 - (G_{21} + I_{21})/2 \right) / (B_{21} \times (G_7 + I_7)/2 / B_7) \right) / 2$$
- BEST AVAILABLE COPY**
- where $G = Al_2O_3$, $Z1 = Na_2O$, $24 = SiO_2$, $G =$ normalized composition adj.
for CaO, FeO using SiO_2 , $I =$ norm. comp adj using Al_2O_3

Title <u>USBM Spreadsheet Documentation #WHC3</u>		Project <u>LLW vit.</u>	
Prepared by: <u>Greg Whysatt</u>	Date <u>9/27/95</u>	Reviewed by: <u>M. Paul</u>	Date <u>9/28/95</u>

Pacific Northwest Laboratories

CALCULATION WORKSHEET

Calc. No. WHC-3

Page 3 of 8

✓ Col AY: Same as AW except SiO_2 adjusted = L Al_2O_3 adjusted = N

$$= ((B21 * (L24 + N24) / 2 / B24 - (L21 + N21) / 2) / (B21 * (L24 + N24) / 2 / B24)) + ((B21 * (L7 + N7) / 2 / B7 - (L21 + N21) / 2) / (B21 * (L7 + N7) / 2 / B7)) / 2$$

✓ Col BA: Same as AW except SiO_2 adjusted = Q Al_2O_3 adjusted = S

BA21 =

$$(((B21 * (Q24 + S24) / 2 / B24 - (Q21 + S21) / 2) / (B21 * (Q24 + S24) / 2 / B24)) + ((B21 * (Q7 + S7) / 2 / B7 - (Q21 + S21) / 2) / (B21 * (Q7 + S7) / 2 / B7)) / 2$$

✓ Col BC: Same as AW except SiO_2 adj = V Al_2O_3 adjusted = X

$$BC21 = (((B21 * (V24 + X24) / 2 / B24 - (V21 + X21) / 2) / (B21 * (V24 + X24) / 2 / B24)) + ((B21 * (V7 + X7) / 2 / B7 - (V21 + X21) / 2) / (B21 * (V7 + X7) / 2 / B7)) / 2$$

- ✓ Sheet 2 Col B = Sheet 1 Col AW
- ✓ Sheet 2 Col C = Sheet 1 Col AY
- ✓ Sheet 2 Col D = Sheet 1 Col BA
- ✓ Sheet 2 Col E = Sheet 1 Col BC

✓ Sheet 2 Col F average loss results

F21 = average (B21:E21)

(Sr, I value deleted)

Reviewer's comments:

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* Check marks identify cells, columns, and formulas verified by the reviewer

* Some of the input glass concentration values shown on the attached spreadsheet do not match the values given in the USERN report. The difference is due solely to the cell formats used to display the values in Excel. The values are correct.

Title USRM Spreadsheet #WHC3

Project LLW vit.

Prepared by: Greg Whigatt

Date 9/27/95

Reviewed by: Tom

Date 9/28/95

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
2	Target Feed Values—			Test Glass		SiO2		Al2O3		Test Glass	SiO2		Al2O3	
3	Oxide	B Feed	?	Testplan	WHC3-SP1BM(1)	Ratio on CaO		Ratio on CaO		WHC3-SP1DM(1)	Ratio on CaO		Ratio on CaO	
4	Component	Target	normalized	B feed	19.55	and FeO		and FeO		20.42	and FeO		and FeO	
5		Table 3.5	Target	Target	analysis	NA entries		NA entries		analysis	NA entries		NA entries	
6					on TP target	Normalized		on TP target	Normalized		on TP target	Normalized	on TP target	Normalized
7	Al2O3	✓ 10.28	10.3030789	10	✓ 10.22	10.22	10.72	10.22	10.70	✓ 10.05	10.05	10.35	10.05	10.36
8	B2O3	✓ 5.01	5.021247595	5.003	✓ 4.02	4.02	4.22	4.02	4.21	✓ 4.25	4.25	4.38	4.25	4.38
9	BaO	✓ 0.003	0.003006735	0	✓ 0.01	0.01	0.01	0.01	0.01	✓ 0.01	0.01	0.01	0.01	0.01
10	Cl	✓ 0.38	0.380853111	0.344	✓ 0.013	0.013	0.01	0.013	0.01	✓ 0.01	0.01	0.01	0.01	0.01
11	CaO	✓ 5	5.011225144	5.002	✓ 7.25	4.987189282	5.23	✓ 5.112044	✓ 5.35	✓ 7.81	5.08501316	5.24	5.02701	5.18
12	Cr2O3	✓ 0.044	0.044098781	0.04	✓ 0.08	0.08	0.08	0.08	0.08	✓ 0.06	0.06	0.06	0.06	0.06
13	Cs2O	✓ 0.157	0.15735247	0.144	✓ 0.099	0.099	0.10	0.099	0.10	✓ 0.10	0.10	0.10	0.10	0.10
14	F	✓ 0.314	0.314704939	0.288	✓ 0.001	0.001	0.00	0.001	0.00	✓ 0.00	0.00	0.00	0.00	0.00
15	FeO	✓ 0.811	0.812820718	0.811	✓ 1.4	0.808598662	0.85	✓ 0.828842	0.87	✓ 1.38	0.824459351	0.85	0.815055	0.84
16	Fe2O3				✓ 0	0	0.00	✓ 0	0.00	✓ 0.00	0.00	0.00	0.00	0.00
17	K2O	✓ 1.71	1.713838999	1.585	✓ 1.25	1.25	1.31	✓ 1.25	1.31	✓ 1.27	1.27	1.31	✓ 1.27	1.31
18	MgO	✓ 0.25	0.250561257	0.248	✓ 0.42	0.42	0.44	✓ 0.42	0.44	✓ 0.39	0.39	0.40	✓ 0.39	0.40
19	MnO	✓ 0.003	0.003006735	0.002	✓ 0.02	0.02	0.02	✓ 0.02	0.02	✓ 0.02	0.02	0.02	✓ 0.02	0.02
20	MoO3	✓ 0.159	0.15935696	0.146	✓ 0.077	0.077	0.08	✓ 0.077	0.08	✓ 0.08	0.08	0.08	✓ 0.08	0.08
21	Na2O	✓ 19.94	19.98476588	20.483	✓ 18.2	18.2	19.09	✓ 18.2	19.06	✓ 18.74	18.74	19.30	✓ 18.74	19.32
22	P2O5	✓ 0.221	0.221496151	0.204	✓ 0.12	0.12	0.13	✓ 0.12	0.13	✓ 0.10	0.10	0.10	✓ 0.10	0.10
23	SO3	✓ 0.241	0.241541052	0.231	✓ 0.02	0.02	0.02	✓ 0.02	0.02	✓ 0.02	0.02	0.02	✓ 0.02	0.02
24	SiO2	✓ 54.9	55.02325208	54.712	✓ 54.55	54.55	57.21	✓ 54.55	57.12	✓ 55.62	55.62	57.30	✓ 55.62	57.33
25	SnO	✓ 0.113	0.113253688	0.104	✓ 0.104	0.104	0.11	✓ 0.104	0.11	✓ 0.104	0.104	0.11	✓ 0.104	0.11
26	TiO2	✓ 0.096	0.096215523	0	✓ 0.19	0.19	0.20	✓ 0.19	0.20	✓ 0.19	0.19	0.20	✓ 0.19	0.20
27	ZrO2	✓ 0.005	0.005011225	0	✓ 0.04	0.04	0.04	✓ 0.04	0.04	✓ 0.03	0.03	0.03	✓ 0.03	0.03
28	I	✓ 0.139	0.139312059	0.128	✓ 0.128	0.128	0.13	✓ 0.128	0.13	✓ 0.128	0.128	0.13	✓ 0.128	0.13
29	Unknown?			0.436										
30	Total	99.776	100	99.911	97.98	95.35778794	100	95.502886	100	100.125	97.07647251	100	97.009065	100

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Prepared by WPC 3/4/8
 Reviewed by WPC 7/29/95

WHC-SD-WM-ER-517
 Revision 0

WHC 3/4/8

	A	O	P	Q	R	S	T	U	V	W	X	Y	Z
2		Test Glass	SiO2		Al2O3		Test Glass	SiO2		Al2O3		Test Glass	
3	Oxide	WHC3-SP1EM(1)	Ratio on CaO		Ratio on CaO		WHC3-SP1AM(1)	Ratio on CaO		Ratio on CaO		WHC3-SP1BM(1)	
4	Component	21:19	and FeO		and FeO		20:42	and FeO		and FeO		19:55	
5	analysis	NA entries			NA entries		analysis	NA entries		NA entries			
6		on TP target	Normalized		on TP target	Normalized		on TP target	Normalized	on TP target	Normalized	SiO2	Al2O3
7	Al2O3	9.79	9.79	10.89	9.79	10.87	9.67	9.67	10.40	9.67	10.38	10.72	10.70
8	B2O3	3.73	3.73	4.15	3.73	4.14	4.02	4.02	4.32	4.02	4.32	4.22	4.21
9	BaO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
10	Cl	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
11	CaO	13.43	4.791541527	5.33	4.896958	5.44	12.34	4.733030048	5.09	4.836934	5.19	5.23	5.35
12	Cr2O3	0.07	0.07	0.08	0.07	0.08	0.07	0.07	0.08	0.07	0.08	0.08	0.08
13	Cs2O	0.08	0.08	0.09	0.08	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.10
14	F	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
15	FeO	1.42	0.776877285	0.86	0.793969	0.88	1.13	0.767390518	0.83	0.784237	0.84	0.85	0.87
16	Fe2O3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
17	K2O	1.19	1.19	1.32	1.19	1.32	1.25	1.25	1.34	1.25	1.34	1.31	1.31
18	MgO	0.47	0.47	0.52	0.47	0.52	0.41	0.41	0.44	0.41	0.44	0.44	0.44
19	MnO2	0.02	0.02	0.03	0.02	0.03	0.02	0.02	0.02	0.02	0.02	0.02	0.02
20	MoO3	0.08	0.08	0.08	0.08	0.08	0.11	0.11	0.11	0.11	0.11	0.08	0.08
21	Na2O	15.91	15.91	17.69	15.91	17.67	19.55	19.55	21.02	19.55	20.99	19.09	19.06
22	P2O5	0.09	0.09	0.10	0.09	0.10	0.09	0.09	0.10	0.09	0.10	0.13	0.13
23	SO3	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.02
24	SiO2	52.41	52.41	58.29	52.41	58.21	51.77	51.77	55.66	51.77	55.58	57.21	57.12
25	SiO		0.10	0.12	0.10	0.12		0.10	0.11	0.10	0.11	0.11	0.11
26	TiO2	0.21	0.21	0.23	0.21	0.23	0.17	0.17	0.18	0.17	0.18	0.20	0.20
27	ZrO2	0.04	0.04	0.04	0.04	0.04	0.03	0.03	0.03	0.03	0.03	0.04	0.04
28	I		0.13	0.14	0.13	0.14		0.13	0.14	0.13	0.14	0.13	0.13
29	unknown?											0.00	0.00
30	Total	98.969	89.91941881	100	90.041927	100	100.754	93.01642057	100	93.137171	100	100.00	100.00

Total correct

D-21

Prepared by J. W. Wright
9/27/96
Forward by J. W. Wright
9/28/96

WHC-SD-WM-ER-517
Revision 0

5/16

	A	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ	AK	AL	AM	AN
2		Test Glass		Test Glass		Test Glass							Fractional	Volatility	Final Glas
3	Oxide	WHC3-SP1DM(1)		WHC3-SP1EM(1)		WHC3-SP1AM(1)		4 sample	Tie=SiO2		Tie=Al2O3				Test Glas
4	Component	20:42		21:19		20:42		Avg value	Table 3.5	Theoretical	Table 3.5	Theoretical	Avg loss	Avg loss	WHC3-SP
5								both methods	B feed	Testplan	B feed	Testplan	% of oxide	% of glass	20:42
6		SiO2	Al2O3	SiO2	Al2O3	SiO2	Al2O3	Target	Target	Target	Target	Target	Target	Target	SiO2
7	Al2O3	10.35	10.36	10.89	10.87	10.40	10.38	✓10.58	0.99%	-1.44%	0.00%	0.00%	-0.11%	-0.01138	10.40
8	B2O3	4.38	4.38	4.15	4.14	4.32	4.32	✓4.26	18.15%	18.31%	17.33%	19.47%	18.32%	0.916973	4.32
9	BaO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	-241.73%	NA	-245.14%	NA	-243.44%	-0.0073	0.01
10	Cl	0.01	0.01	0.01	0.01	0.01	0.01	0.01	97.45%	97.20%	97.43%	97.24%	97.33%	0.352335	0.01
11	CaO	5.24	5.18	5.33	5.44	5.09	5.19	5.26	-1.10%	-0.72%	-2.11%	0.71%	-0.81%	-0.04032	5.09
12	Cr2O3	0.06	0.06	0.08	0.08	0.08	0.08	0.07	-63.20%	-78.90%	-64.82%	-76.37%	-70.82%	-0.02975	0.08
13	Ca2O	0.10	0.10	0.09	0.09	0.10	0.10	0.10	39.05%	33.77%	38.44%	34.71%	36.49%	0.054924	0.10
14	F	0.00	0.00	0.00	0.00	0.00	0.00	0.00	99.51%	99.46%	99.50%	99.47%	99.49%	0.299451	0.00
15	FeO	0.85	0.84	0.86	0.88	0.83	0.84	0.85	-1.06%	-0.72%	-2.07%	0.71%	-0.79%	-0.00637	0.83
16	Fe2O3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	NA	NA	NA	NA	NA	NA	0.00
17	K2O	1.31	1.31	1.32	1.32	1.34	1.34	1.32	25.71%	20.12%	24.97%	21.25%	23.01%	0.37911	1.34
18	MgO	0.40	0.40	0.52	0.52	0.44	0.44	0.45	-73.57%	-74.37%	-75.30%	-71.90%	-73.79%	-0.18373	0.44
19	MnO2	0.02	0.02	0.03	0.03	0.02	0.02	0.02	-610.48%	-962.08%	-617.57%	-947.04%	-784.29%	-0.01961	0.02
20	MoO3	0.08	0.08	0.08	0.08	0.11	0.11	0.09	45.65%	41.02%	45.11%	41.85%	43.41%	0.066197	0.11
21	Na2O	19.30	19.32	17.69	17.67	21.02	20.99	19.27	7.07%	9.85%	6.15%	11.12%	8.55%	1.727824	21.02
22	P2O5	0.10	0.10	0.10	0.10	0.10	0.10	0.11	53.71%	50.02%	53.25%	50.73%	51.93%	0.110345	0.10
23	SO3	0.02	0.02	0.01	0.01	0.01	0.01	0.02	93.67%	93.42%	93.61%	93.51%	93.55%	0.220789	0.01
24	SiO2	57.30	57.33	58.29	58.21	55.66	55.58	57.09	0.00%	0.00%	-1.00%	1.42%	0.10%	0.057364	55.66
25	SiO	0.11	0.11	0.12	0.12	0.11	0.11	0.11	NA	NA	NA	NA	NA	NA	0.11
26	TiO2	0.20	0.20	0.23	0.23	0.18	0.18	0.20	-103.09%	NA	-105.12%	NA	-104.10%	-0.09994	0.18
27	ZrO2	0.03	0.03	0.04	0.04	0.03	0.03	0.04	-618.95%	NA	-626.12%	NA	-622.53%	-0.03113	0.03
28	Is	0.13	0.13	0.14	0.14	0.14	0.14	0.14	5.59%	-2.17%	4.65%	-0.72%	1.84%	0.002508	0.14
29	unknown?	0.00	0.00	0.00	0.00	0.00	0.00								
30	Total	100.00	100.00	100.00	100.00	100.00	100.00	100						3.758291	100

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	A	AO	AP	AQ	AR	AS	AT	AU	AV	AW	AX	AY	AZ	BA	BB	BC	BD
2	Sample Only										Test Glass	Test Glass	Test Glass	Test Glass			
3	Oxide			Tie=SiO2		Tie=Al2O3				WHC3-SP1BM(1)	WHC3-SP1DM(1)	WHC3-SP1EM(1)	WHC3-SP1AM(1)				
4	Component	AM(1)		Table 3.5	Theoretical	Table 3.5	Theoretical	Avg loss	Avg loss	19:55		20:42		21:19		0:42	
5				B feed	Testplan	B feed	Testplan	% of	% of glass								
6		Al2O3	avg	Target	Target	Target	Target	oxide	tot mass								
7	Al2O3	10.38	10.39	0.25%	-2.20%	0.00%	0.00%	-0.49%	-0.0494	-0.00027		0.017514		0.00121		0.001233	
8	B2O3	4.32	4.32	14.91%	15.08%	14.70%	16.91%	15.40%	0.770952	0.192675		0.14748		0.219171		0.14804	
9	BaO	0.01	0.01	-253.49%	NA	-254.36%	NA	-253.92%	-0.00762	-2.35381		-2.3499		-2.49594		-2.53924	
10	Cl	0.01	0.01	97.21%	96.93%	97.20%	96.99%	97.08%	0.351442	0.965579		0.973553		0.9862		0.972059	
11	CaO	5.19	5.14	-1.48%	-1.10%	-1.74%	1.07%	-0.81%	-0.04054	-0.01612		-0.01623		-0.0161		-0.0161	
12	Cr2O3	0.08	0.08	-68.71%	-84.94%	-69.13%	-80.97%	-75.94%	-0.03189	-0.82935		-0.37041		-0.66851		-0.68918	
13	Cs2O	0.10	0.10	34.48%	28.81%	34.32%	30.34%	31.99%	0.048142	0.365553		0.385496		0.452229		0.344002	
14	F	0.00	0.00	99.32%	99.27%	99.32%	99.28%	99.30%	0.29889	0.996796		0.996799		0.99332		0.993237	
15	FeO	0.84	0.83	-1.44%	-1.10%	-1.70%	1.07%	-0.79%	-0.00641	-0.01571		-0.01582		-0.0157		-0.0157	
16	Fe2O3	0.00	0.00	NA	NA	NA	NA	NA	na								
17	K2O	1.34	1.34	22.48%	16.65%	22.29%	18.44%	19.97%	0.328959	0.264515		0.253619		0.270147		0.223852	
18	MgO	0.44	0.44	-73.92%	-74.72%	-74.35%	-70.96%	-73.49%	-0.18298	-0.69032		-0.56775		-0.97171		-0.7413	
19	MnO2	0.02	0.02	-571.62%	-903.99%	-573.29%	-882.42%	-732.83%	-0.01832	-5.70762		-5.6998		-7.39024		-5.72455	
20	MoO3	0.11	0.11	29.30%	23.27%	29.13%	24.92%	26.66%	0.040649	0.512748		0.506996		0.498696		0.292153	
21	Na2O	20.99	21.00	-3.97%	-0.87%	-4.23%	1.30%	-1.94%	-0.39271	0.081654		0.055509		0.163185		-0.041	
22	P2O5	0.10	0.10	56.81%	53.38%	56.71%	54.38%	55.32%	0.117551	0.453678		0.545262		0.572895		0.567605	
23	SiO3	0.01	0.01	95.60%	95.42%	95.59%	95.52%	95.53%	0.225461	0.916503		0.9166		0.956482		0.955943	
24	SiO2	55.58	55.62	0.00%	0.00%	-0.25%	2.15%	0.48%	0.26045	0.000271		-0.01815		-0.00121		-0.00124	
25	SiO	0.11	0.11	2.40%	-5.68%	2.16%	-3.41%	NA	na	0.073992		0.075072		0.034751		0.022795	
26	TiO2	0.18	0.18	-87.79%	NA	-88.25%	NA	-88.02%	-0.0845	-0.99133		-0.989		-1.29421		-0.88022	
27	ZrO2	-0.03	0.03	-536.28%	NA	-537.85%	na	-537.06%	-0.02685	-7.04915		-5.02982		-7.39024		-5.37062	
28	unknown?	0.14	0.14	2.35%	-5.68%	2.10%	-3.41%	-1.16%	-0.00155	0.073479		0.07456		0.034216		0.022254	
30	Total	100	100					-13.2473	1.599721								

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Prepared by: [Signature]
 9/27/06
 Reviewed by: [Signature]
 9/27/06

Target Feed Values—					
Oxide	Time	Of Glass	Sampling	Average	
Compone	19:55	20:42	21:19	0:42	loss
Al ₂ O ₃	0.000	0.018	0.001	0.001	0.005
B ₂ O ₃	0.193	0.147	0.219	0.148	0.177
BaO	-2.354	-2.350	-2.496	-2.539	-2.435
Cl	0.966	0.974	0.986	0.972	0.974
CaO	-0.016	-0.016	-0.016	-0.016	-0.016
Cr ₂ O ₃	-0.829	-0.370	-0.669	-0.689	-0.639
Cs ₂ O	0.366	0.385	0.452	0.344	0.387
F	0.997	0.997	0.993	0.993	0.995
FeO	-0.016	-0.016	-0.016	-0.016	-0.016
Fe ₂ O ₃	0.000	0.000	0.000	0.000	0.000
K ₂ O	0.265	0.254	0.270	0.224	0.253
MgO	-0.690	-0.568	-0.972	-0.741	-0.743
MnO ₂	-5.708	-5.700	-7.390	-5.725	-6.131
MoO ₃	0.513	0.507	0.499	0.292	0.453
Na ₂ O	0.082	0.056	0.163	-0.041	0.065
P ₂ O ₅	0.454	0.545	0.573	0.568	0.535
SO ₃	0.917	0.917	0.956	0.956	0.936
SiO ₂	0.000	-0.018	-0.001	-0.001	-0.005
SrO					
TiO ₂	-0.991	-0.989	-1.294	-0.880	-1.039
ZrO ₂	-7.049	-5.030	-7.390	-5.371	-6.210

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9/27/85
 prepared by W. J. J. J.
 9/24/85
 Reviewed by [Signature]

MIC-SD-MM-ER-517
 Revision 0

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APPENDIX E
REVIEW OF VECTRA TEST REPORT

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**Vectra VE-SKULL Phase I LLW Melter Demonstration
Entrainment and Volatility Estimates (MR Powell 9/8/95)
(Revised 3/27/96 GA Whyatt)**

The Vectra melter run analytical data were examined and estimates made of the relative volatility and entrainment for each oxide analyte. These analyses and the results are described below. Volatility estimates were made by comparing the melter feed and glass product compositions. Assuming uniform entrainment of feed and zero volatility for a known low volatility component, the fractional volatility of each glass oxide species can be estimated from feed and glass compositions. The entrainment rate of melter feed was estimated based on the observed average collection rate of low volatility oxides on isokinetically-gathered filter samples.

The phase I Vectra test included three "mass balance" periods, which are referred to as MT-2, MT-3, and MT-4. Mass balance test MT-2 used slurry feed and MT-3 and MT-4 used dried feed. Relatively little glass was produced by the feeds used for MT-3 and MT-4, so it is unlikely that the melter glass composition reached steady-state levels before the mass balance tests. For this reason, volatility estimates using tie component calculations were made only for MT-2. Also, slurry feeding is of greater interest since that is the recommended feeding approach. Aerosol sampling results are summarized for MT-3 and MT-4 at the end of this writeup.

Entrainment

Feed entrainment during the MT-2 mass balance period is estimated to be between 0.06% and 1.5%. These estimates are derived from the aerosol sampling data included in the final Vectra report (WHC-SD-WM-VI-031, Rev 0.). The 0.06% to 1.5% range is established by the range of non-volatile species entrainment rates. Aerosol collection rates for aluminum, silicon, and iron were used in this calculation (see attached calculation worksheet). The average of the Al, Si, and Fe entrainment rates is 0.61%. Two aerosol flow rate samples were taken during MT-2. The average bulk particulate flow rate as determined by these two samples was about 0.81 kg/h total solids as oxides. If this solids flow rate was due exclusively to entrainment, then the entrainment rate would be $(0.81/33.6)*100\% = 2.4\%$ entrainment. Glass and feed composition data, however, indicate that some waste components were lost by volatilization rather than just entrainment. It is recommended that a 0.6% entrainment rate be taken as representative of the Vectra melter operated with slurry feed.

It should be noted that the aerosol data presented in the Vectra report gives widely varying entrainment estimates depending on which low-volatility analyte is selected for making the estimate. Using the MT-2 data, for example, the entrainment rate given by the second aerosol sample silica data is about 0.015%. For the first aerosol sample alumina data, the entrainment rate is about 0.49%. The calcium data for the first sample yields an entrainment

rate of 14.2%. Clearly, either the aerosol results are erroneous or the assumption of uniform entrainment of feed is incorrect. The description of the aerosol monitoring and analyses given in the Vectra report is not sufficient to determine whether the aerosol data are likely to be correct. Because of the large variability in entrainment estimates, the estimated 0.5% entrainment rate should be considered to be an "order of magnitude" entrainment estimate.

Feed entrainment during the MT-3 (dry simulated calcine feed material) was similar to MT-2 while entrainment during MT-4 (actual calcine feed material) may have been lower than during slurry feeding. The rates calculated for MT-3 based on non-volatile analytes of Al, Fe, Sr were 0.13, 0.97, 0.20 respectively for an average of 0.43%. The rates calculated for MT-4 based on non-volatile analytes of Fe and Sr were 0.056% and 0.084% respectively for an average of 0.07%. Thus, the entrainment during MT-4 appears to have been lower than the slurry feeding or simulated dry calcine feeding periods.

Volatility

Fractional volatility estimates were generated for each of the glass oxide analytes. To make these calculations, it is necessary to select a tie-component, which is assumed to have zero volatility. Experience has shown that SiO_2 , CaO , Fe_2O_3 and Al_2O_3 are generally good low volatility tie-components. The volatility calculations were made using the average MT-2 glass composition data and the estimated feed composition based on the feed makeup data records (see attached spreadsheet). An approach based on estimated feed composition was necessary because sampling and analysis of the feed was not adequate to characterize the melter feed. The variabilities of the glass analyses were used to estimate the 95% confidence intervals for each oxide concentration (uncertainty in the target feed composition was not quantified and was neglected). Systematic analytical errors are not included in these uncertainty estimates. The results are given in the attached spreadsheets and summarized in the tables below.

The attached spreadsheet gives volatility estimates for MT-2 using SiO_2 , Fe_2O_3 , CaO , and Al_2O_3 as tie components. The feed and glass concentration data from mass balance run MT-2 were used to prepare these estimates. Too little feed was supplied to the melter during MT-3 and MT-4 to be sure that the glass composition had reached steady-state conditions. An average % volatility was calculated for each analyte based on the average of the volatilities given by the four tie components. Along with the average % volatility values are given estimated 95% confidence intervals. These confidence intervals were estimated based on the variance amongst the four fractional volatility estimates for each analyte. The fractional volatility data are presented in Table 1. Table 1 does not include the minor components which were not in the original startup glass and may not have reached their full concentration prior to the mass balance period. The minor components not in the startup glass include SrO , Cs_2O , K_2O , F , Cl , I , SO_3 and P_2O_5 . Because these minor components were not at steady state, their volatilities were calculated using SrO as the tie component. These results are provided in Table 2. If it is assumed that the volatility occurs from the cold cap prior to incorporation

into the molten glass, then this approach should provide accurate volatility estimates.

Table 1. Volatilities based on SiO₂, Al₂O₃, CaO, Fe₂O₃ tie components^(a)

Estimate of Waste Oxide Volatilities					
Oxide	SiO ₂ Tie (%)	Al ₂ O ₃ Tie (%)	CaO Tie (%)	Fe ₂ O ₃ (%)	Avg. (%) ^(b)
Al ₂ O ₃	8.59 ± 5.0	0	1.4 ± 5.3	3.1 ± 6.1	3.4 ± 3.0
B ₂ O ₃	19.4 ± 3.4	11.4 ± 5.6	12.6 ± 3.5	14.2 ± 4.4	14.4 ± 2.2
BaO	No Data	No Data	No Data	No Data	No Data
CaO	7.71 ± 2.4	-1.4 ± 5.5	0	1.8 ± 3.9	2.0 ± 1.9
Cr ₂ O ₃	-638 ± 27	-710 ± 49	-699 ± 27	-685 ± 37	-683 ± 18
Fe ₂ O ₃	6.1 ± 3.9	-3.2 ± 6.5	-1.8 ± 4.0	0	0.3 ± 2.5
Li ₂ O	No Data	No Data	No Data	No Data	No Data
MgO	3.6 ± 3.7	-5.9 ± 6.5	-4.5 ± 3.8	-2.6 ± 5.0	-2.3 ± 2.4
MnO ₂	-525 ± 272	-586 ± 301	-577 ± 295	-565 ± 290	-563 ± 145
MoO ₃	-40.5 ± 11.0	-54.3 ± 14.1	-52.2 ± 11.8	-49.5 ± 13	-49 ± 6.2
Na ₂ O	17.7 ± 2.3	-9.6 ± 5.0	10.8 ± 2.3	12.4 ± 3.6	12.6 ± 1.7
NiO	No Data	No Data	No Data	No Data	No Data
SiO ₂	0	-9.8 ± 6.1	-8.4 ± 2.8	-6.4 ± 4.4	-6.2 ± 2.1
TiO ₂	No Data	No Data	No Data	No Data	No Data
ZrO ₂	No Data	No Data	No Data	No Data	No Data

- (a) The minor components SrO, Cs₂O, K₂O, F, Cl, I, SO₃ and P₂O₅ are provided in Table 2.
- (b) The confidence intervals on the average do not account for cross correlation of the different tie component results and may therefore underestimate the magnitude of the potential error.
- (c) Statistics are not presented for SO₃ because only two analytical results above detection limits were obtained.

Table 2. Minor Component Volatile Loss Based on SrO Tie Component

Component	Percent Volatile Loss
Cl	64
Cs ₂ O	41
F	9
K ₂ O	15
I	83
P ₂ O ₅	-45
SO ₃	85

However, this approach could potentially underestimate the volatility if the volatility has a significant dependence on the concentration of the volatile species within the molten glass. In any case, the result should be more accurate than comparing to major glass components that are at steady state concentrations which results in a significant over estimation of volatility. Taking this approach does result in making all tie-component results dependant on the Sr analysis results.

The tie component volatility analysis indicates that during the MT-2 mass balance run significant volatility was observed for B₂O₃, Cs₂O, K₂O, Na₂O, SO₃, Cl, and I.

The tie component results in Table 1 determined negative volatilities for Cr₂O₃, MnO₂, and MoO₃. The negative Cr₂O₃ volatility is probably due to refractory and electrode sheath degradation during the run. The Vectra report notes that the elevated chrome concentrations in the glass samples are consistent with the amount of observed refractory loss (p. 3-9 of final report). The negative MnO₂ volatility is probably just an artifact of the low MnO₂ concentrations in the feed and the glass. The MnO₂ concentrations reported in the glass samples are all at or near the MnO₂ detection limit. Negative MoO₃ volatilities are expected as a consequence of the erosion of the molybdenum electrodes in the melter. During the 28 days of total melter operation, more than 140 kg of electrode mass was lost. This loss rate is more than large enough to account for the elevated molybdenum oxide concentrations observed during the MT-2 mass balance run.

The results in Table 2 determined a significant negative volatility for P₂O₅. No source has been identified and the single feed analysis raises suspicions that the feed may have been too high in P₂O₅ by roughly a factor of 2.

Volatility Comparisons to Aerosol Sampling Data

A second method to determine volatilities is to examine the method 5 data, accounting for some amount of uniform entrainment of feed (0.6 wt% in this case) and compare the aerosol oxide emission rates with the feed rates to the melter. Two 1 hour aerosol samples were obtained during the MT-2 slurry feeding period. A comparison of the volatility results from aerosol samples to those using the tie component method are shown in Table 3. The aerosol samples provide volatility estimates that are systematically lower than the tie component method results by a factor of 1.5 to 3.5 (excluding F which typically has larger analytical uncertainty).

Table 3. Comparison of Aerosol and Tie Component Based Volatilities

Oxide	Aerosol Sample Volatility	Tie Component Volatility	Factor Difference
B ₂ O ₃	6.8	14	2.1
Cs ₂ O	no data	41	NA
Cl	42	64	1.5
F	15	9	0.6
I	32	83	2.6
K ₂ O	8.5	15	1.8
Na ₂ O	3.6	13	3.6
P ₂ O ₅	1.1	-45	
SO ₃	56	85	1.5

It is expected that the reason for the generally lower losses predicted by the aerosol samples is that there was loss of particulate between the melter exit and the sample point. Sampling was performed on a section of the offgas line outside the building some distance from the melter exit. Deposition in the line between the melter and the sample point cannot be ruled out and in fact some loss is expected. Other possible sources of the differences include:

- Unsteady melter operation such that higher volatility occurred during periods when aerosol sampling was not being performed.
- Detection limits for analysis of scrub solutions was too high to achieve detection of gaseous emissions of acid gases. Thus, offgas sampling results only include the aerosol portion of emissions.
- Variations in offgas flow during operation could account for errors in method 5

sampling.

In addition, however, errors in feed makeup such that the feed is deficient in the volatile components could account for some of the differences as well. The tie component calculation based on feed targets would then include deficiencies in the volatility result. There is only one slurry feed sample analysis available during the MT-2 processing (V1M2F35-108PA). The sample number indicates it is a mixed feed sample taken during MT-2 from the feed slurry tank and analyzed at PNL. The analytical results report very low silica content ($\text{SiO}_2\text{:Na}_2\text{O}$ wt ratio of 0.5 vs 3.1 target) which indicates the sample may not be representative. The ratio of Na_2O to CaO for the feed analysis (5.97) and target values (5.89) indicates good agreement between sodium and calcium levels. Similarly, the Na_2O to MgO ratios agree to within 3%. However, the aluminum to sodium ratios are feed analysis 1.49 and target 1.80 indicating either a 17% deficiency in sodium or excess aluminum. The feed analysis does perhaps explain the results for P_2O_5 , suggesting roughly a factor of 2 excess in the feed. While there is insufficient data to reach a firm conclusion on possible feed deficiencies, it is expected that large measurement errors in feed makeup for major components such as sodium or boron are unlikely.

Conclusion: The aerosol samples are believed to be the better source of volatility data for F due to the analytical difficulties for the glass samples for this analyte. The volatile loss for P_2O_5 is also best determined by aerosol measurement. In this case there is uncertainty about the actual feed concentration and the aerosol result is less sensitive to this type of error. For other analytes, the tie component calculation with the appropriate tie component is believed to be the best data.

Overall Mass Balance for MT-2

At first glance, the entrainment and volatility estimates are not consistent with the overall mass balance data. During mass balance test MT-2, a total of 865 kg of slurry feed was delivered to the melter. The theoretical glass mass produced by 865 kg of slurry feed is about 469 kg. Only 406 kg of glass was withdrawn from the melter during MT-2. Thus, there is about 63 kg of missing glass mass. The aerosol flow rate data account for roughly 9 kg of this mass, leaving 54 kg of glass unaccounted for. Despite the discrepancy, the Vectra report does not address where this mass went. It is expected that due to the intermittent pouring from the Vectra melter, there was some difference between starting and ending levels within the melter. Although the melter was on load cells during the run, no data from the load cells is reported in the final report. Assuming a specific gravity of 2.6 for the glass and a inside diameter of the melter of 3.2 ft, the change in level to account for the missing 54 kg is only 2.8 cm. Some mass may also have collected in the offgas system prior to the aerosol sample point.

MT-3 Aerosol Results

During MT-3, a simulated calcine material was fed which consisted of dry materials with an

average composition matching the expected composition of calcined feed material. No feed characterization information was available so any comparisons to the feed rate are made to the target feed compositions. The feed rate was highly variable such that there is significant uncertainty about the appropriate feed rate to use for comparison of aerosol emission rates. The aerosol samples indicate an entrainment rate of 0.43% (average of Al, Fe, Sr entrainment values). The volatile losses of selected components are shown below. A spreadsheet with complete results is attached. Due to poor detection limits, the results shown here include only particulate losses and acid gases would be in addition to the losses described here.

MT-3 Aerosol Sample Based Volatile Losses

Component	Total Aerosol Loss (%)	Volatility (assuming 0.43% entrainment)
B ₂ O	3.3	2.9
MoO ₃	3.81	3.4
Na ₂ O	0.90	0.5
SO ₃	6.84	6.4
Cl	21.6	21.2
F	2.1	1.7

MT-4 Aerosol Results

During MT-4, a calcined feed material was processed through the melter. Due to caking problems in the calciner, boric acid was added after the calcining operation. No feed characterization information was available so any comparisons to the feed rate are made to the target feed compositions. The feed rate was highly variable such that there is significant uncertainty about the appropriate feed rate to use for comparison of aerosol emission rates. The aerosol samples indicate an entrainment rate of 0.07% (average of Fe and Sr entrainment values). The volatile losses of selected components are shown below. A spreadsheet with complete results is attached. Volatile losses for the actual calcine were somewhat higher than the simulated calcine. Significant sources of sulfur and chloride are indicated because the loss of these components exceed the feed by factors of 3.8 and 3.0 based on particulate emissions alone.

Due to poor detection limits, the results shown here include only particulate losses and acid gases would be in addition to the losses described here. Losses of sulfur were high enough to be detected in scrub solutions during this processing but aerosol measurements alone indicate

that the losses were far in excess of the feed amount so the SO_2 emissions were not included in the calculations. The source of excess sulfur is not known.

MT-4 Aerosol Sample Based Volatile Losses

Component	Total Aerosol Loss (%)	Volatility (assuming 0.07% entrainment)
B_2O	21.4	21.3
MoO	19.5	19.4
Na_2O	10.6	10.5
SO_3	381	381
Cl	297	297
F	11.2	11.1

Fluid Bed Calciner/RVR Calciner Results

A fluid bed calciner and rotary dryer were tested for preparation of calcined feed. Discussion of the experiments is provided in the Vectra final report. In general, the rotary dryer suffered from severe caking such that it is not recommended for drying the feed. The fluid bed calciner was most successful operating at about 500°C and with 100% stoichiometric sucrose addition. The mass balance provided indicates about 91% of the NO_x is converted to N_2 with 3.6% converted to NO_x , 4.7% converted to N_2O , and 0.4% converted to ammonia. Insufficient information is provided in the report to verify the numbers. The sample was apparently taken downstream from a caustic scrubber which adds uncertainty to the result. The results indicate much lower ammonia production and much higher conversion to nitrogen than the USBM tests in which material was heated in a laboratory setting to evaluate the gases generated (USBM used sucrose addition and a belt dryer).

Vectra Mt2
3/27/96 5:28 PM

Greg A Whyatt 3/27/96									
Vectra Slurry Feed Method 5 Loss Estimates									
Mass balance period MT2									
Total Glass Mass over 14 hours			406.42 kg						
					average				
					offgas	Feed			
					mass	Oxides	Feed	Percent	Volatility
					oxide	MT-2	Oxides	Partition	(adjust
								oxide	assuming
									0.6 wt%
species	kg/h #1	kg/h #2	avg	oxide	kg/hr	kg	kg/hr	to offgas	entrainment)
Al	0.00857	0.00213	0.00535	Al2O3	0.010109	46.49655	3.321182	0.304382	-0.295618
B	0.08026	0.04728	0.06377	B2O3	0.205337	39.10279	2.793056	7.351693	6.75169259
CaCO3	0.25623	0.16532	0.210775	CaO	0.118108	14.1717	1.012264	11.66772	11.0677152
Cl	0.04776	0.04457	0.046165	Cl	0.046165	1.52798	0.109141	42.29833	41.6983263
Cr	0.00078	0.0003	0.00054	Cr2O3	0.000789	0.177428	0.012673	6.227584	5.62758431
F	0.01505	0.01321	0.01413	F	0.01413	1.279603	0.0914	15.45949	14.8594884
Fe	0.00531	0.0019	0.003605	Fe2O3	0.005154	4.88542	0.348959	1.477033	0.87703282
I	0.01711	0.0095	0.013305	I	0.013305	0.569781	0.040699	32.69148	32.0914848
K	0.04123	0.02689	0.03406	K2O	0.041028	6.344135	0.453152	9.054004	8.45400384
Mn	0.00013	0.00004	0.000085	MnO2	0.000135	0.009757	0.000697	19.29985	18.6998461
Mo	0.00408	0.06714	0.03561	MoO3	0.053426	0.646267	0.046162	115.7364	115.136428
Na	0.22073	0.14928	0.185005	Na2O	0.249383	83.5109	5.965064	4.180719	3.58071866
P	0.00028	0.00058	0.00043	P2O5	0.000985	0.82856	0.059183	1.664854	1.06485352
SO4	0.05368	0.03721	0.045445	SO3	0.037876	0.934643	0.06676	56.7342	56.1341957
Si	0.00892	0.00129	0.005105	SiO2	0.010921	258.0784	18.43417	0.059246	-0.5407545
Sr	0.000062	0.000011	3.65E-05	SrO	4.32E-05	0.465237	0.033231	0.129894	-0.4701065
				other		10.9			
totals	0.760182	0.566651	0.663417	totals	0.806895	469.9292	32.7878		

Reviewed by
Mr. Paul
3/28/96

WMC-SD-MW-ER-517
Revision 0

VSIM (MT-3)

GA Whyatt 3/27/96										
Vectra Dry Feed Data: VSIM material										
Mass Balance run data 4/22/95 approx 19:00 to 22:00										
VSIM Feeding Rate= 43.2 kg/hr LOI est = 0.25										
	VSIM	VSIM	Aerosol	Aerosol	Aerosol	Aerosol				
	Target	oxide	Reporting	Run#3	run4	Run#3	run4			
	Glass	kg/hr	Basis	19:10	21:05	basis	basis	% loss	% loss	Offgas los
	wt%			kg/h	kg/h	kg/hr	kg/hr	in offgas	in offgas	Average
Al2O3	10	3.24	Al	0.00249	0.0021	0.004705	0.003968	0.145215	0.122471	0.133843
B2O3	8	2.592	B	0.02954	0.02345	0.095118	0.075508	3.669663	2.913121	3.291392
Cr2O3	0.04	0.01296	Cr	0.00012	0.00005	0.000175	7.31E-05	1.353309	0.563879	0.958594
Cs2O	0.15	0.0486	Cs	nd	nd			0	0	0
Fe2O3	1	0.324	Fe	0.0034	0.00099	0.004861	0.001415	1.50035	0.436867	0.968608
K2O	1.52	0.49248	K	0.00873	0.00667			0	0	0
MgO	2.1	0.6804	Mg	nd	nd			0	0	0
MnO2	0.0023	0.000745	Mn	0.00006	0.00001	9.49E-05	1.58E-05	12.74134	2.123557	7.43245
MoO3	0.15	0.0486	Mo	0.00097	0.0015	0.001455	0.00225	2.994451	4.630595	3.812523
Na2O	20	6.48	Na	0.04628	0.03989	0.062385	0.053771	0.962733	0.829806	0.896269
P2O5	0.2	0.0648	P	0.00012	nd	0.000275		0.424365		0.424365
SO3	0.22	0.07128	SO4	0.00635	0.00535	0.005292	0.004459	7.424764	6.25551	6.840137
SiO2	52.8	17.1072	Si	0.00219	0.00191	0.004685	0.004086	0.027387	0.023886	0.025636
SrO	0.11	0.03564	Sr	0.000077	0.000042	9.11E-05	4.97E-05	0.255501	0.139364	0.197433
Cl	0.29	0.09396	Cl	0.02265	0.0179	0.02265	0.0179	24.106	19.05066	21.57833
F	0.28	0.09072	F	0.00217	0.00159	0.00217	0.00159	2.391975	1.752646	2.07231

Reviewed by
 Mr. Powell
 3/29/96

Dry Calcine (MT-4)

GA Whyatt 3/22/96											
Vectra Feeding of dry calcined product with boric acid added after calcine											
Mass Balance run data 4/23/95 approx 10:00 to 14:00											
Calcine Feeding Rate=			25.6 kg/hr		LOI est.=		0.95				
	VSIM	VSIM	Aerosol	Aerosol	Aerosol	Aerosol					
	Target	oxide	Reporting	Run #5	Run#6	Run #5	Run#6				
	Glass	kg/hr	Basis	10:41	12:02	basis	basis	% loss	% loss	Offgas los	
	wt%			kg/h	kg/h	kg/hr	kg/hr	in offgas	in offgas	Percent	
Al2O3	10	0.128	Al			0	0	0	0	0	
B2O3	8	0.1024	B	0.00659	0.007	0.02122	0.02254	20.72221	22.01145	21.36683	
Cr2O3	0.04	0.000512	Cr	0.00055	0.00012	0.000804	0.000175	157.005	34.25564	95.63033	
Cs2O	0.15	0.00192	Cs	0.00009	0.00011	9.54E-05	0.000117	4.969667	6.074037	5.521852	
Fe2O3	1	0.0128	Fe	0.00001	0	1.43E-05	0	0.111699	0	0.055849	
K2O	1.52	0.019456	K	0.00263	0.00247	0.003168	0.002975	16.28331	15.29269	15.788	
MgO	2.1	0.02688	Mg					0	0	0	
MnO2	0.0023	2.94E-05	Mn			0	0	0	0	0	
MoO3	0.15	0.00192	Mo	0.00033	0.00017	0.000495	0.000255	25.78662	13.28402	19.53532	
Na2O	20	0.256	Na	0.02314	0.01707	0.031193	0.02301	12.18459	8.988372	10.58648	
P2O5	0.2	0.00256	P			0	0	0	0	0	
SO3	0.22	0.002816	SO4	0.01933	0.00645	0.01611	0.005376	572.1051	190.899	381.5021	
SiO2	52.8	0.67584	Si			0	0	0	0	0	
SrO	0.11	0.001408	Sr	0.000001		1.18E-06	0	0.083992		0.083992	
Cl	0.29	0.003712	Cl	0.01049	0.01159	0.01049	0.01159	282.597	312.2306	297.4138	
F	0.28	0.003584	F	0.00031	0.00049	0.00031	0.00049	8.649554	13.67188	11.16071	

E-13

Revised by
M. T. T. T.
3/27/96

WHC-SD-WM-ER-517
Revision 0

Vectra MT-2
Minor Component
Volatility Estimate
Based on SrO Tie Calculation
3/17/96 3:20 PM

Correction of Fractional Volatility Estimates for Vectra MT-2 Mass Balance Test. (GA Whyatt 3/17/96)												
Target	Feed Compositions (from feed makeup data, p. 3-78)											pg3-78 Na2O target wt% in glass
	SrO	Cl	Cs2O	Cs2O	K2O	K2O	I	F	P2O5	SO3	Na2O	
g/l simulant	1.727	5.672	2.348	2.348	23.55	23.55	2.115	4.75	3.076	3.4695	310	17.17
wt% glass	0.095654	0.314156	0.130049	0.130049	1.304366	1.304366	0.117144	0.263089	0.170371	0.192166	17.17	
Glass Compositions												
Sample #	SrO	Cl	USGS/C Cs2O	PNL Cs2O	USGS/C K2O	PNL K2O	I	F	P2O5	SO3		
1	0.0503	0.05	0.035	0.078	0.58	1.16	0.0093	0.125	0.12	0.006		
2	0.055	0.07	0.048		0.63		0.0118	0.14	0.16			
3	0.055	0.07	0.05		0.67		0.0131	0.14	0.16			
4	0.0597	0.07	0.044	0.112	0.68	2.22	0.0123	0.145	0.126	0.029		
Percent Volatile Loss Calculations												
Sample #	SrO	Cl	USGS/C Cs2O	PNL Cs2O	USGS/C K2O	PNL K	I	F	P2O5	SO3		
1		69.73378	48.8207	-14.0567	15.44069	-69.1186	84.90278	9.647379	-33.9428	94.06243		
2		61.24824	35.8092		16		82.48132	7.452632	-63.329			
3		61.24824	33.13458		10.66667		80.5513	7.452632	-63.329			
4		64.29905	45.79085	-37.9869	16.47125	-172.697	83.17664	11.69356	-18.4956	75.8204		
Avg % volatile loss		64.13233	40.88883	-26.0218	14.64465	-120.908	82.77801	9.061549	-44.7741	84.94142		
sample#1 includes sample analysis of V1M2/G32-011P1, -012U1, -013C1 sample#2 includes sample analysis of V1M2/G32-025U sample#3 includes sample analysis of V1M2/G32-038U sample#4 includes sample analysis of V1M2/G32-040P2, -041C2, -042U2												

*Revised by
MT
3/27/96*

E-15

Revised by
M. J. Smith
3/27/96

Vectra MT2
3/14/96 2:19 PM

	AB	AC	AD	AE	AF	AG	AH	AI	AJ	AK
1	Vectra Data analysis for entrainment and volatility: MR Powell 9/5/95 .Modified 3/14/96 GA Whyatt									
2	Using assumed LLW simulant composition									
3	Pg3-81 data									
4	Simulant Volume =		269.39	liters						
5	Glass product mass=		406.42	kg						
6	Total RVR mix=		429.5	kg	pg 3-77	mass				
7		pg3-76			RVR	oxides in		Missing	Missing	
8		simulant	Total		g oxide	RVR	Frit/GF	Total	Mass	Mass as
9	Oxide	oxide g/l	mass (kg)		per 1362g	Mix	(kg)	oxides kg	kg	% of fed
10					of mix	mass %				
11	Al2O3	51.82967	13.96239		103.17	7.57489	32.53415	46.49655	7.886647	16.96179
12	B2O3	0	0		124	9.104258	39.10279	39.10279	10.34858	26.46506
13	BaO	0	0					0	-0.0042	
14	CaO	0.05888	0.015862		44.89	3.295888	14.15584	14.1717	2.243275	15.82926
15	Cr2O3	0.65863	0.177428					0.177428	-1.01643	-572.868
16	Cs2O	2.34843	0.632644					0.632644	0.406064	64.18534
17	Fe2O3	0.06122	0.016492		15.44	1.133627	4.868928	4.88542	0.699294	14.3139
18	K2O	23.55	6.344135					6.344135	3.756594	59.21365
19	Li2O	0	0					0	0	
20	MgO	0.04165	0.01122		32.51	2.386931	10.25187	10.26309	1.240565	12.08763
21	MnO2	0.03622	0.009757					0.009757	-0.04582	-469.603
22	MoO3	2.399	0.646267					0.646267	-0.18181	-28.133
23	Na2O	310	83.5109					83.5109	20.82062	24.93161
24	NiO	0	0					0	-0.10821	
25	P2O5	3.07569	0.82856					0.82856	0.278538	33.61716
26	SO3	3.46948	0.934643					0.934643	0.86352	92.3903
27	SiO2	0	0		818.4	60.08811	258.0784	258.0784	22.71043	8.799818
28	SrO	1.727	0.465237					0.465237	0.241706	51.95326
29	TiO2	0	0					0	-0.09144	
30	ZrO2	0	0					0	-0.1834	
31	Cl	5.672	1.52798					1.52798	1.270581	83.15427
32	F	4.75	1.279603					1.279603	0.724162	56.59272
33	I	2.11508	0.569781					0.569781	0.523958	91.95764
34										
35				Theoretical Glass =				469.9249		

Revised by
M. Fawell
3/27/96

Revised volatility estimates for Vectra MT-2 mass balance run.

The glass composition in the melter during the MT-2 mass balance was stable for the major oxide components. However, some of the minor components were not present in the feed delivered to the melter before the MT-2 run.

Because of this, fractional volatilities calculated for certain minor components will be biased high if a major component oxide is used as the tie component (i.e., if Al_2O_3 , CaO , or SiO_2 are used as tie component).

Improved volatility estimates can be obtained by using a zero-volatility minor component oxide as the tie component. The minor component (with $V_i = 0$) selected must have not been present in the start-up feed. SiO_2 meets these criteria.

Calculation of revised fractional volatilities for the minor components not present in the start-up feed (Cl, Cs, and I)

Fractional Volatility is calculated by:

$$V_i = 1 - \frac{C_{gi} \cdot C_{g,tie}}{C_{fi} \cdot C_{f,tie}}$$

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Where

C_{fi} = Concentration of component i in the feed
 C_{gi} = Concentration of component i in the glass
 $C_{f,tie}$ = Concentration of tie component in the feed
 $C_{g,tie}$ = Concentration of tie component in the glass

The relevant concentration data are:

Time/date		Sample #	% SiO_2	% Cl	% Cs_2O	% I	Theoretical Glass based on Feed Bul
4/21		22:30	VIM2/632-01P1	.05	-	.078*	.01
		22:30	-012U1	.05	.05	.03%	.0085
		22:30	-013C1	.05	.05	.034	(.01) < det. limit not used.
		Average Concentrations →		.0503	.05	.035	.0093
4/22		8:00	VIM2/632-02SU	.055	.07	.048	.0118
4/22		11:00	VIM2/632-03BU	.055	.07	.050	.0131
4/22		14:00	VIM2/632-04OP2	.06	-	.112*	.015
		14:00	-041C2	.06	.07	.042	.01
		14:00	-042U2	.059	.07	.046	.0118
		Average Concentrations →		.0597	.07	.044	.0123

(*) Those concentrations judged to be in error and were not used in calculations

Title Correction of MT-2 fractional volatilities		Project LLW - Vectra	
Prepared by MR Powell M. Powell	Date 1/26/96	Reviewed by GA Whitt	Date 2/27/96

Revised MT-2 volatility calculation for minor components. (Cont.)

Sample Calculation:

For the 4/21 @ 22:30 sample,

$$V_{C1} = 1 - \frac{(0.05)(0.0862)}{(0.283)(0.0503)} = 0.70 \quad \checkmark$$

Note: - The Feed concentrations listed on the preceding page were estimated based on feed makeup records (taken from Appendix 3C of the Vectra report, page 3-76). The given feed concentration data are actually estimated "theoretical glass composition" data, but this does not adversely affect the volatility calculation.

- Further, the feed concentrations represent steady-state glass component concentrations assuming no volatility losses. The fact that the feed concentration was changing (for the certain minors) is corrected for by using $SrBV$ as the tie component.

The above volatility calculation is made for the remaining data on the preceding page to give (see attached spreadsheet):

Time/date	V_{C1}	V_{C2O}	V_F
4/21 22:30	.70	0.49	0.85
4/22 8:00	.61	0.36	0.83
4/22 11:00	.61	0.33	0.81
4/22 14:00	.64	0.46	0.83

Average = 0.64 0.41 0.83

95% C.I. = ± 0.06 ± 0.12 ± 0.03

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95% confidence interval is estimated based on the deviation of the 4 calculated values from their mean according to the equation

$$95\% \text{ CI} \approx \frac{S_x \cdot (\text{student's } t)}{\sqrt{N}} = \frac{(S_x)(3.182)}{\sqrt{4}}$$

where S_x = Estimated population standard deviation

Title Correction of MT-2 fractional volatilities		Project LLW - Vectra	
Prepared by: MR Powell	Date 1/26/96	Reviewed by: G. Whyatt	Date 2/27/96

Vectra MT-2
Special Estimate for I, Cl, Cs
Based on SrO Tie Calculation
3/14/96 4:36 PM

Correction of Fractional Volatility Estimates for Vectra MT-2							
Mass Balance Test. (MR Powell 1/26/96)							
Feed Compositions (from feed makeup data, p. 3-76)							
Feed							
Conc =	0.099	0.3252	0.1346	0.1213			
Glass Compositions							
Sample #	SrO	Cl	Cs2O	I	V cl	Vcs2o	V I
1	0.0503	0.05	0.035	0.0093	0.697388	0.488212	0.8491
2	0.055	0.07	0.048	0.0118	0.612546	0.358098	0.824897
3	0.055	0.07	0.05	0.0131	0.612546	0.331352	0.805606
4	0.0597	0.07	0.044	0.0123	0.643049	0.457914	0.831847
Average =				0.641382	0.408894	0.827862	
95% CI =				0.063656	0.120774	0.028624	

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APPENDIX F

REVIEW OF DURATEK TEST REPORT

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DON'T SAY IT -- WRITE IT!

Date: September 25, 1995

To: Greg Whyatt
Subject: Review of Duratek Test Data

From: Eve Tracey

I examined the Phase I Hanford LLW Melter Tests final report by GTS Duratek along with the provided analytical data.

Results of Volatility Calculations

Total percent losses were calculated using the difference between the mass of oxides in the feed and the mass of oxides in the glass and by using the total mass of oxides trapped in the offgas equipment. First, assuming 3838 kg oxides are in the feed and 3688 kg oxides are in the glass, gives an overall loss of $3.9 \pm 1.6\%$. (The error was calculated using the USGS data.) Using the offgas data, 5.2 kg oxides were accumulated in the offgas which was divided by the oxides in the feed, giving an overall loss of $0.14 \pm \%$. (The error was not calculated for the offgas data.) Because both the initial volume of feed and the final glass mass were approximations and were used to calculate the total losses, any error associated with those numbers needs to be considered. Once this is done, the two total losses may be in agreement.

Table 1 lists the component masses for the feed (using recipe, USGS, and Corning data), the masses of the components in the glass, the scrubber, the baghouse, and the filtercake. Note that the recipe and USGS data are close while the Corning data is substantially different. Therefore, in most cases the USGS data was used for comparison except for Mg. In this case the Corning data should be used because no Mg was found in the feed by USGS, but it was found in the glass.

The fractional volatilities of the glass components were estimated four ways: comparison of the oxides in the feed and the glass, comparison of the oxides in the feed and the glass assuming SiO₂ is not volatile, total mass of oxides found in the offgas equipment, and using the emissions data directly from the melter. Because there are three different feed values, Table 2a uses the recipe feed values, Table 2b uses the USGS feed data, and Table 2c uses the Corning feed data. The emissions data was pulled directly from the report and is shown on all three tables even though the values do not change. Table 2a utilizes the recipe feed data to calculate the fractional volatilities. With or without assuming the SiO₂ tie, negative volatilities were calculated for Al₂O₃, B₂O₃, P₂O₅ and SO₃. The sulfur value in glass was much higher than the recipe value. This may be due to error in the analytical results or due to the separation of sulfate salts in the vitrification process. With or without assuming no SiO₂ volatility, the losses calculated using the glass data are higher than the losses using the offgas data.

Table 2b utilizes the USGS feed data to calculate the fractional volatilities. Negative volatilities were calculated for NiO. Assuming no silica volatility, negative volatilities were calculated for B₂O₃, Fe₂O₃, K₂O, Na₂O, and NiO. The glass value of NiO is three times higher than the USGS and Corning analytical data. Again, the losses using the glass data are higher than the losses using the offgas data.

Table 2c utilized the Corning feed data to calculate the fractional volatilities. The Corning data appeared to be inaccurate for several of the components. This created negative volatilities for many of the components and larger errors. This data should not be used except to estimate the MgO loss.

For all three tables, in general, the emissions calculated using ES and VSL data were similar to the percent losses calculated using the offgas masses.

Calculations

1) **Feed:** The feed values were calculated using the recipe values, the USGS data, and the Corning data. All three values were normalized to 57.5 wt% oxides (or 3838 kg oxides). All three feed data were used to calculate losses. Although the Corning analytical feed data were used to calculate volatilities, in general the Corning data should not be used because several values, such as Al, were incorrect. No magnesium was found in the USGS feed analysis but it was found in the glass therefore the Corning magnesium value should be used to report percent losses.

The total mass of oxides (3838 kg) was calculated using Table 5.3b. Knowing the approximate slurry volume used (4500 L), the density, and the wt % oxides, the total mass of oxides was calculated (see attached calculation sheet). Because the numbers 57.5 wt%, 4500 L, and 1.75 g/cm³ were used to calculate the total oxides, they play an important factor in the mass balance calculations and should be accurate. In Table 5.3b, the feed density (with urea and water) is higher than the feed density of the feed without urea and water. This is not likely since urea and water have lower densities than the feed.

3) **Glass:** The glass values were calculated by averaging the five Corning samples, normalizing to 100%, and multiplying by 3700 kg. Again, because the value of 3700 kg is very important to the percent loss calculations, an accurate value should be provided rather than an approximation. Because chromium is a refractory component, the target glass value was used rather than the analytical glass value. The sulfur content of the glass is much higher than expected. Sulfur salts separate from the glass during vitrification, therefore the glass sample analyzed may have had a higher sulfur content than the overall glass.

4) **Baghouse:** The baghouse values were calculated by assuming that no silica was accumulated in the baghouse, averaging the three used baghouse samples, subtracting the difference between the blank and the used baghouse and multiplying by 13 kg. Because the mass accumulated in the baghouse was not directly reported, the 13 kg was determined by fitting my calculations to the values for the masses reported in Table 5.8.f. The baghouse was precoated with diatomaceous earth.

5) **Filter Cake:** The filter cake values were calculated from one sample which was normalized to 100% and multiplied by 1.09 kg. Again, the mass accumulated in the filter cake was not directly reported so the same method as described above was used.

6) **Scrubber:** The scrubber values were calculated by taking the difference between the first and the last samples and multiplying by the scrubber volume (1000 L). The blowdown volume was not used in the calculation. It was assumed that precipitated materials would be captured on the filter rather than being lost in the blowdown volume. (Sodium hydroxide was used to control the pH in the scrubber.) The authors of the Duratek report took the difference between the first and the second

samples, then extrapolated. I did not feel comfortable with this method because many of the elements did not increase steadily throughout the melter run.

7) The fractional volatilities of the glass components were calculated by adding the masses found in the scrubber, baghouse, and filter cake then dividing by the mass in the feed (see "Offgas Masses" in Tables 2a-c). The percent losses determined directly from the offgas emissions were reported directly from table 5.8h (see "Emissions" in tables 2a-c).

8) The fractional volatilities of the glass components were also estimated based on the ratios of the glass and feed oxide concentrations (see "Glass" in Tables 2a-c).

9) The fractional volatilities of the glass components were also estimated based on the ratios of the glass and feed oxide concentrations and the assumption of zero SiO₂ volatility (see "Glass, SiO₂ tie" in Tables 2a-c).

NOTE: This DSI was revised by G. Whyatt in March 1996 to reflect the use of Corning data rather than Pacific Northwest National Laboratory sample data in calculations.

**Addendum to "Review of Duratek Test Data" by Eve Tracey, 9/25/95
Prepared by Greg Whyatt 3/4/96**

Eve Tracey has left PNNL and is not available to address WHC review comments. A few minor comments were addressed within Eve's writeup using redline text while items requiring more lengthy discussion are included here.

Calculation item #1)

To perform the calculations a feed density was needed. A value of 1.75 g/cm³ for the feed prior to urea addition was selected. This density was provided in Table 5.3b of the final report and was used along with other data in that table including an 18% volume increase for the addition of urea and water, and a total oxide content of 57.5 wt% prior to water and urea addition to conclude a feed:glass ratio of 1.17 liters/kg of glass. The final feed density after water and urea addition was measured by WHC at 1.82 to 1.89 g/cm³ which is greater than the value measured by Duratek of 1.75 g/cm³ prior to addition of water and urea. The WHC measured density is believed to be in error based on two observations. First, the actual ratio of feed to glass produced, was reported as 1.22 (pg 5-2 of final report) which is closer to the theoretical 1.17 value than the value predicted by the WHC density of 0.94 liters/kg. Secondly, the feed density is unlikely to increase with the addition of water and urea due to the lower densities of these materials.

Calculation item #2)

The glass analysis values were arbitrarily normalized to a 100% oxide basis. Duratek opted not to perform this normalization. Before normalization, adjustments were made to account for chrome contamination (0.1 wt%), SO₃ was added because USGS did not report a result (0.56%), and Cl was added because there was no USGS data (0.19%). The average percent of total mass that was accounted for prior to normalization was 95.7%.

Additional Comments Pertinent to chloride Losses

(comments refer to pages 5-5 and 5-6 of the Duratek final report)

Sulfuric acid scrubbers were used to remove NH₃ from the sample gas at the melter exit prior to analysis for NO_x in the chemiluminescent analyzer. This gas was drawn through a heated filter, scrubbed in the sulfuric acid solution and then transported to the gas analysis trailer and analyzed. During the melter test it was requested that the filter and scrubber solution be analyzed to estimate the melter chloride emissions. Particulates should be deposited on the filter and HCl would show up in the scrub solution while any Cl₂ would not be detected. Capture of HCl in a sulfuric acid scrub solution would be expected. In fact, capture of HCl in a 0.1N sulfuric acid scrubbing solution is the basis of 40CFR60 app A, Method 26 "Determination of Hydrogen Chloride from Stationary Sources". The Duratek final report contains fundamental errors with respect to this data.

Duratek Final Report, pg 5-5 par 3 states:

"Lastly, analysis of these solution permits an estimate of particulate fluorides and chlorides to be made. Acidic gases such as HCl and HF would not have been trapped in this strongly acidic

scrubbing solution, however particulates such as NaCl might have been retained."

Duratek Final Report, Pg 5-6 par 3 states:

"Particulate halide salts were only detected at the melter exhaust location, as would be expected. VSL measurements using electrochemical sensors showed the presence of HCl in the final emissions but no chloride was detected in the sulfuric acid scrubbing solution. This supported the notion that particulate chlorides, not HCl, were detected in the acid scrubbing solutions."

The errors in this text include:

- 1) Based on the sampling train, there should be no particulates transported to the scrub solution so analysis of the scrub solution will not provide information on particulates.
- 2) HCl should be captured by the sulfuric acid scrub solution.
- 3) Analysis of the filter was not reported so it is not known if any data exists on particulate chloride emissions from the melter.
- 4) Data in Table 5.7h indicates an air concentration at the melter exit of 83 mg chloride/m³ based on the chloride collected in the sulfuric acid scrub solution. Therefore, it appears that the lack of detection statement applies to the sulfuric acid scrub solutions from downstream of the offgas scrubber. The lack of detection directly conflicts with the reading on the electrochemical HCl analyzer and does not support the chloride being detected at the melter exit being particulate.

Chloride losses are reported in the Duratek Final Report based on an electrochemical (pg 5-6) offgas analyzer. It is my opinion that none of the data from this instrument are of any value. Concentrations from this analyzer averaged 55 ppm HCl (pg 8-40). The concentrations reported passing the scrubber are much higher than would be expected. The reason for the error in the instrument is not clear but may relate to interference, drift or other problems.

Probably of greater value is the analysis of the sulfuric acid scrub solutions from the downstream locations. Both the post demister, and post HEPA measurements resulted in air concentrations <1 mg/m³ Cl (pg 5-41). The 55 ppm on the CEM suggests a concentration of about 90 mg/m³ Cl. Thus, it is concluded that the scrub solution result is more likely correct and the CEM is more likely incorrect.

Very little about chloride losses can be concluded based on the offgas scrubber. Analysis results for chloride in the scrubber solution were very erratic and the solids collected were not analyzed for Cl.

The sulfuric acid scrub solution at the melter exhaust indicates about 7% of the feed chloride exits as HCl. Duratek reports 5% "particulate" (pg 5-6). The reason these values do not agree is not clear although it is noted that there is significant uncertainty due to the poor characterization of the offgas flowrate at the melter exit.

The analysis of the glass and feed indicates that about 1/2 of the chloride is lost and this result is believed to be valid. Approximately 7% of the chloride is lost as HCl (based on analysis of sulfuric acid scrub solution). It is highly likely that during sampling a significant fraction of the chloride withdrawn from the melter exhaust existed as particulate and was deposited on the filter (based on difference between feed/glass analysis calculated losses and losses as HCl in the scrub solution).

Sample Identification Aid

The tables attached provide summary results of the mass balance, tie component and emission measurements. This section is intended to improve traceability of the results to the analytical samples on which results are based.

Table 1.

USGS analytical feed is based on samples D1F3-111U, -112U, and -114U.

Corning analytical feed is based on samples D1F3-111C, 112C, 114C.

Scrubber results are based on D1O6-101P and -148P.

Baghouse results are based on D1O7-101C, -102C, -103C.

Filter Cake results are based on sample D1O10-101C(d).

Glass results are based on Corning glass samples D1G4-108C5, -109C6, -110C7, -112C8, and -115C9. The normalized concentration is then applied to a glass quantity of 3700 kg.

Table 2a.

"Glass SiO₂ Tie" is based on corning glass results and "recipe feed" values in Table 1.

"Offgas Masses" is based on scrubber, baghouse and filtercake from Table 1 compared to the recipe composition with 3838 kg of glass.

"Glass" is based on recipe feed in Table 1, and corning glass analysis in Table 1. The total mass of feed is 6675kg total (3838kg oxide at 57.5wt% oxide) and glass is 3700kg. Loss is then calculated for each component as 1-(kg in glass)/(kg in feed).

"Emissions" are based on aerosol sampling results.

Table 2b.

"SiO₂ Tie" is based on USGS analytical feed in Table 1 and on Corning glass samples in Table 1.

"Offgas Mass" is based on scrubber, baghouse and filter cake samples from Table 1 compared to the USGS feed composition applied to 6675 total mass feed (3838 kg feed oxide).

"Flowsheet" is based on the Corning glass analysis from Table 1 compared to USGS feed samples in Table 1. It is assumed that there are 3700 kg glass and 6675 kg total mass feed with % loss as 1-(glass/feed).

Table 2c.

"SiO₂ Tie" is based on Corning glass samples in Table 1 along with corning feed samples D1F3-111C, -112C and -114C.

"Offgas" is based on scrubber, baghouse and filter cake samples from Table 1 compared to corning feed samples D1F3-111C, -112C and -114C.

"Glass" is based on Corning feed samples D1F3-111C, -112C and -114C, and corning glass

analysis in Table 1. The total mass of feed is 6675kg total (3838kg oxide at 57.5wt% oxide) and glass is 3700kg. Loss is then calculated for each component as 1-(kg in glass)/(kg in feed).

“Emissions” are based on aerosol sampling results.

Table 1

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DURATEC
MASS DISTRIBUTION

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	Recipe Feed (KG)	USGS Analytical Feed (KG)	Corning Analytical Feed (KG)	Scrubber (KG)	Baghouse (KG)	Filtercake (KG)	Glass (KG)
Al ₂ O ₃	240.74	257.00	55.24	0.01	0.07	0.01	241.04
B ₂ O ₃	239.19	245.40	281.83	0.60	0.11	0.01	249.62
BaO	0.00	0.53	0.63	0.00	0.01	0.00	0.43
CaO	304.50	305.86	143.32	0.02	0.04	0.05	272.58
Cr ₂ O ₃ ¹	1.55	2.68	2.64	0.02	0.02	0.00	1.55
Cs ₂ O	5.80	5.23	5.52	0.14	0.16	0.00	4.25
Fe ₂ O ₃	291.36	243.62	278.36	0.01	0.41	0.08	242.12
K ₂ O	143.36	133.22	152.92	0.63	0.56	0.01	132.81
Li ₂ O	NA	NA	NA	0.00	NA	NA	NA
MgO	0.00	0.00	12.01	0.00	0.00	0.00	11.60
MnO ₂	0.00	6.42	5.20	0.00	0.00	0.00	4.64
MoO ₃	5.80	5.40	5.36	0.03	0.01	0.00	5.02
Na ₂ O	707.14	677.72	786.51	NA	0.79	0.10	682.60
NiO	NA	0.13	0.11	0.0000	0.0015	0.0001	0.87
P ₂ O ₅	7.34	9.10	7.66	NA	0.01	0.00	10.82
SO ₃	8.11	NA	NA	0.42	NA	NA	21.52
SiO ₂	1642.28	1697.86	1857.51	0.05	0.00	0.79	1590.93
SrO	4.25	4.41	0.13	0.00	0.00	0.00	4.25
TiO ₂	38.64	37.81	32.29	0.00	0.00	0.00	33.09
ZrO ₂	198.23	205.46	210.57	0.00	0.00	0.00	178.03
Carbon	NA	187.09	178.28	NA	NA	NA	NA
Cl	13.52	14.62	13.08	1.42	NA	NA	7.15
F	11.21	3.75	4.80	NA	0.12	0.00	5.10
SUM OF OXIDES	3838	3838	3838	1.93	2.20	1.09	3688

ss value based on target

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Table 2a

EVE TRACEY
PERCENT LOSS
DURATEC
BASED ON Recipe

	Glass, SiO ₂ Tie (%)	95% Cl (+ %)	Offgas Masses (%)	95% Cl (not calculated)	Glass (%)	95% Cl (+ %)	Emissions	
							VSL (% Loss)	ES (% Loss)
Al ₂ O ₃	-3.4	2.1	0.0		-0.8	0.7		
B ₂ O ₃	-7.7	2.8	0.3		-5.1	1.8	0.5	0.2-0.4
BaO	#DIV/0!	#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!		
CaO	7.6	2.1	0.0		9.9	1.1		
Cr ₂ O ₃	-3.3	36.6	2.4		-0.7	35.6	2.3	1.8-2.1
Cs ₂ O	24.3	1.4	5.4		26.1	0.0	5.5	4.9-6.5
Fe ₂ O ₃	14.2	1.8	0.2		16.3	0.9		
K ₂ O	4.4	2.0	0.9		6.7	0.9	0.6	0-0.2
Li ₂ O	#VALUE!	#VALUE!	NA		#VALUE!	#VALUE!		
MgO	#DIV/0!	#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!		
MnO ₂	#DIV/0!	#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!		
MoO ₃	10.5	1.7	0.6		12.7	0.0	0.8	0.4-0.5
Na ₂ O	0.4	1.9	0.1		2.8	0.4	0.3	0.1-0.15
NiO	#VALUE!	#VALUE!	NA		NA	NA		
P ₂ O ₅	-52.2	10.0	0.2		-48.4	9.3		
SO ₃	-173.7	19.4	NA		-167.0	18.2		
SiO ₂	0.0	2.7	0.1		2.5	1.8		
SrO	-3.3	1.9	0.0		-0.7	0.0	0.6	.08-.1
TiO ₂	11.6	4.1	0.0		13.8	3.7		
ZrO ₂	7.3	3.0	0.0		9.6	2.4		
Carbon	#VALUE!	#VALUE!	NA		NA	NA		
Cl	45.4	17.7	10.6		46.8	17.3		
F	53.0	12.3	1.1		54.2	12.0		

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Table 2b

EVE TRACEY
 PERCENT LOSS
 DURATEC
 BASED ON USGS ANALYTICAL RESULTS

	SiO ₂ Tie (%)	95 % CI (+ %)	Offgas Mass (%)	95 % CI (not calculated)	Flowsheet (%)	95 % CI (+ %)	Emissions	
							VSL (%Loss)	ES (%Loss)
Al ₂ O ₃	-0.1	4.6	0.0		6.2	3.2		
B ₂ O ₃	-8.6	5.2	0.3		-1.7	3.8	0.5	0.2-0.4
BaO	14.9	11.0	1.6		20.3	10.0		
CaO	4.9	4.6	0.0		10.9	3.3		
Cr ₂ O ₃	38.3	23.8	1.4		42.2	22.2	2.3	1.8-2.1
Cs ₂ O	13.2	5.7	5.9		18.6	4.8	5.5	4.9-6.5
Fe ₂ O ₃	-6.1	5.2	0.2		0.6	3.8		
K ₂ O	-6.4	3.9	0.9		0.3	2.0	0.6	0-0.2
Li ₂ O	NA	#VALUE!	NA		NA	NA		
MgO	#DIV/0!	#DIV/0!	#DIV/0!		NA	NA		
MnO ₂	22.9	2.3	0.1		27.8	0.0		
MoO ₃	0.8	3.9	0.6		7.0	2.3	0.8	0.4-0.5
Na ₂ O	-7.5	5.5	0.1		-0.7	4.1	0.3	0.1-0.15
NiO	-629.7	195.6	1.3		-583.8	182.1		
P ₂ O ₅	-27.0	38.2	0.1		-19.0	35.6		
SO ₃	NA	#VALUE!	NA		NA	NA		
SiO ₂	0.0	4.3	0.0		6.3	2.8		
SrO	-3.0	4.8	0.0		3.5	3.4	0.6	.08-.1
TiO ₂	6.6	50.7	0.0		12.5	47.4		
ZrO ₂	7.5	37.4	0.0		13.3	35.0		
Carbon	NA	#VALUE!	0.0		NA	NA		
Cl	47.8	17.9	9.7		51.1	16.7		
F	-45.4	97.2	3.2		-36.2	91.0		

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Table 2c

EVE TRACEY
 PERCENT LOSS
 DURATEC
 BASED ON CORNING ANALYTICAL RESULTS

	Glass, SiO ₂ Tie (%)	95% CI (+ %)	Offgas (%)	95% CI (not calculated)	Glass (%)	95% CI (+ %)	Emissions	
							VSL (%Loss)	ES (%Loss)
Al ₂ O ₃	-409.4	63.6	0.2		-336.3	16.0		
B ₂ O ₃	-3.4	24.2	0.3		11.4	17.9	0.5	0.2-0.4
BaO	21.5	38.5	1.4		32.8	32.0		
CaO	-122.1	35.4	0.1		-90.2	20.1		
Cr ₂ O ₃	31.7	28.9	1.4		41.5	23.8	2.3	1.8-2.1
Cs ₂ O	10.1	19.4	5.6		23.0	13.9	5.5	4.9-6.5
Fe ₂ O ₃	-1.6	21.6	0.2		13.0	15.3		
K ₂ O	-1.4	18.3	0.8		13.2	11.8	0.6	0-0.2
Li ₂ O	NA	#VALUE!	NA		NA	NA		
MgO	-12.7	94.8	0.0		3.4	80.3		
MnO ₂	-4.1	17.2	0.1		10.9	10.2		
MoO ₃	-9.4	16.9	0.6		6.3	9.2	0.8	0.4-0.5
Na ₂ O	-1.3	18.9	0.1		13.2	12.4	0.3	0.1-0.15
NiO	-846.9	1049.2	1.5		-711.0	893.4		
P ₂ O ₅	-65.0	75.8	0.1		-41.3	62.7		
SO ₃	NA	#VALUE!	NA		NA	NA		
SiO ₂	0.0	16.9	0.0		14.4	10.2		
SrO	-3620.1	3232.1	0.7		-3086.2	2742.1	0.6	.08-.1
TiO ₂	-19.6	117.6	0.0		-2.5	99.9		
ZrO ₂	1.3	53.3	0.0		15.5	44.5		
Carbon	NA	#VALUE!	0.0		NA	NA		
Cl	36.2	22.8	10.9		45.3	18.4		
F	-24.0	62.5	2.5		-6.2	52.0		

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Calculations

Feed:

4500 L LLW and urea

$$\frac{4500 \text{ L}}{1.18} = 3814 \text{ L LLW} \quad \left(\begin{array}{l} \text{Simulant prior} \\ \text{to urea addition} \\ \text{24 3/4\%} \end{array} \right)$$

$$(3814 \text{ L})(1.75 \text{ g/cm}^3) = \boxed{6674.5 \text{ kg}}$$

57.5 wt% oxides

$$(6674.5 \text{ kg})(.575) = \boxed{3838 \text{ kg oxides}}$$

Glass:

3700 kg glass

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Duratek SO_2 Estimate - Volatile Loss

Feed SO_2 Results

	SO_2 wt%	SiO_2 wt%
DIF3-1114	0.37	32.0
DIF3-1124	0.52	31.8
DIF3-1144	0.46	31.4

Feed Avg .447 31.7

Glass SO_2 Results

Note: D164-110P7 discarded due to inconsistent SO_2 analysis

D164-11047	.54	42.3
D164-11549	.57	42.5

Glass Avg .555 42.4

$$\text{Tic Calculation} = \frac{.447 \left(\frac{42.4}{31.7} \right) - .555}{.447 \left(\frac{42.4}{31.7} \right)} = 7.17 \times 10^{-2} \text{ or } 7.2\% \text{ loss}$$

Std deviations of input quantities to loss estimate

$$\text{SO}_2 \text{ feed } .37, .52, .45 \quad S = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}} = \left(\frac{5.929 \times 10^{-3} + 5.329 \times 10^{-3} + 9.0 \times 10^{-6}}{2} \right)^{1/2}$$

$$\text{Avg } = 0.447 \quad S = 7.51 \times 10^{-2}$$

$$\text{SiO}_2 \text{ feed } 32, 31.8, 31.4 \quad S = \left(\frac{9.0 \times 10^{-2} + 1.0 \times 10^{-2} + 9.0 \times 10^{-2}}{2} \right)^{1/2} = 0.308$$

$$\text{Avg } 31.7$$

$$\text{SO}_2 \text{ glass } .54, .57 \quad S = \left(\frac{2.25 \times 10^{-4} + 2.25 \times 10^{-4}}{1} \right)^{1/2} = 2.12 \times 10^{-2}$$

$$\text{Avg } .555$$

$$\text{SiO}_2 \text{ glass } 42.3, 42.5 \quad S = \left(\frac{1 \times 10^{-2} + 1 \times 10^{-2}}{1} \right)^{1/2} = 0.141$$

$$\text{Avg } 42.4$$

First propagate uncertainty to $.447 \left(\frac{42.4}{31.7} \right)$ term using linear approximation over $\pm 3\sigma$

See attached spread sheet, Result = .5979, std dev = .1006

$$\text{which leaves } \frac{(.5979 \pm .1006) - (.555 \pm .0212)}{(.5979 \pm .1006)}$$

assume numerator errors not correlated so numerator = $.0429 \pm \sqrt{(.1006)^2 + (.0212)^2}$

BEST AVAILABLE COPY = $.0429 \pm .1028$

Title Duratek SO_2 Calculation

Project LLW V:t

Prepared by:

Greg Whyatt

Date

10/3/95

Reviewed by:

M. B. Towell

Date

10/4/95

	SO3 feed	SiO2 glass	SiO2 feed				numerator	denominator	
	X1	X2	X3				X1	X2	
avg	0.447	42.4	31.7			avg	0.0429	0.5979	
std dev	0.0751	0.141	0.308			std dev	0.1028	0.1006	
		Y value	delta	gradient				Y value	delta
x1+3S	0.6723	0.8992278	0.301348			x1+3S	0.3513	0.587556	0.515805
X1	0.447	0.5978801		1.337539		X1	0.0429	0.071751	1.67252
X1-3S	0.2217	0.2965325	0.301348			X1-3S	-0.2655	-0.44405	0.515805
X2+3S	42.823	0.6038448	0.005965			X2+3S	0.8997	0.047683	-0.02407
X2	42.4	0.5978801		0.014101		X2	0.5979	0.071751	-0.16104
X2-3S	41.977	0.5919154	0.005965			X2-3S	0.2961	0.144883	-0.07313
X3+3S	32.624	0.5809465	-0.01693						
X3	31.7	0.5978801		-0.01888		propagated std dev x1/x2			
X3-3S	30.776	0.6158305	-0.01795			0.100637			
propagated std dev x1*x2/x3						check linearity of x2 over 2S			
0.100637						X2+3S	0.7991	0.053685	-0.01807
						X2	0.5979	0.071751	-0.13533
						X2-3S	0.3967	0.108142	-0.03639

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Reviewed by:
MR Powell
10/1/95

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$$\text{or } \% \text{ loss} = \frac{.0429 \pm .1028}{.5979 \pm .1006}$$

See attached spreadsheet. Due to non-linearity, only linearize over a 2 σ range.

$$\text{Std dev} = \sigma_1^2 \bar{r}_1^2 + \sigma_2^2 \bar{r}_2^2 = \left[(1.6725)^2 (.1028)^2 + (-.1353)^2 (.1006)^2 \right]^{1/2}$$

$$= (2.75 \times 10^{-2} + 1.853 \times 10^{-4})^{1/2} = .173$$

$\therefore$ Std dev on calculated result is 17.3 %

or 7.2 % $\pm$ 17.3 % indicating significant uncertainty

Compare SO₂ emissions to Source

SO₂ CEM data from Environmental Services, Table 6.3 @ HEPA Exit

Run #1 = 243 ppm

Run #2 = 245 ppm

Flow at HEPA exit table 6.2 (Environmental Services)

Run #1 593 dscfm

Run #2 565 dscfm assume std = 60°F

Target SO₂ (from Table 1 of Duratek review)

$$\text{SO}_2 = 8.11 \text{ kg} \quad \text{Total glass} = 3838 \text{ kg} \quad \frac{8.11}{3838} = 2.113 \times 10^{-3} \frac{\text{kg SO}_2}{\text{kg glass}}$$

Total glass production = 1800 kg/day

$$\text{Target Source } \left(1800 \frac{\text{kg}}{\text{d}} \right) \left(2.113 \times 10^{-3} \right) \left(\frac{1000 \text{ g}}{\text{kg}} \right) \left(\frac{\text{mol}}{32.06 + 3(16)} \right) \left(\frac{1}{24 \text{ h}} \right) \left(\frac{1}{60 \text{ min}} \right) = 3.30 \times 10^{-2} \frac{\text{mol}}{\text{min}} \text{ SO}_2$$

$$\text{offgas flow } 1 \text{ ft}^3 = (1 \text{ ft}^3) \left(\frac{28.317 \text{ L}}{\text{ft}^3} \right) \left(\frac{273.2}{266.8} \right) \left(\frac{\text{mol}}{22.4 \text{ L}} \right) = 1.196 \frac{\text{mol}}{\text{min}}$$

$$\text{SO}_2 \text{ run \#1} = \left(593 \frac{\text{dscf}}{\text{min}} \right) \left(\frac{1.196 \text{ mol}}{\text{ft}^3} \right) \left(\frac{243 \text{ SO}_2}{10^6} \right) = .1723 \frac{\text{mol}}{\text{min}}$$

$$\text{SO}_2 \text{ @ Run \#2} = \left(565 \frac{\text{dscf}}{\text{min}} \right) \left(\frac{1.196 \text{ mol}}{\text{ft}^3} \right) \left(\frac{245 \text{ SO}_2}{10^6} \right) = .1656 \frac{\text{mol}}{\text{min}}$$

$$\text{Avg} = .169 \frac{\text{mol}}{\text{min}}$$

$\therefore$ detected SO₂ > feed target quantity

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Title Duratek SO₂ Calculation

Project LLW V:t

Prepared by:

Greg Whyatt Greg Whyatt

Date

10/3/95

Reviewed by:

W R Powell W R Powell

Date

10/4/95

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CALCULATION WORKSHEET

Calc. No. 1440

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Duratek Method 5 Results (VSL) Table 5.7e

B	(1.38×10^{-2})	$(16/hr)$	3.283	$=$	$.420 \%$	Volatility (Adjust for carryover based on Sr) ~ 0 (negative)
Cr	8.87×10^{-4}		2.50×10^{-2}	$=$	1.12%	
Cs	1.16×10^{-2}		$.2126$	$=$	5.46%	$.556$
Mo	1.30×10^{-3}		$.1551$	$=$	$.838 \%$	4.90
Sr	9.07×10^{-4}		$.1607$	$=$	$.564 \%$	$.27$
K	3.12×10^{-2}		4.766	$=$	$.655 \%$	0
Na	5.79×10^{-2}		21.67	$=$	$.267 \%$	$.091$
						~ 0 (negative)

Gross Rates based on USGS analytical, Table 1 of review (Appendix F)

$$B \quad \frac{245.40 \text{ kg } B_2O_3}{3838 \text{ kg}} \left(\frac{10.81 + 2}{48 + 10.81 \times 2} \right) \left(\frac{1800 \text{ kg}}{1} \right) \left(\frac{1}{24h} \right) \left(\frac{2.20 \times 10^{-1} \text{ lb}}{\text{kg}} \right) = 3.283 \frac{\text{lb}}{\text{h}} \quad B$$

$$Cr \quad \frac{2.68 \text{ Cr}_2O_3}{3838} \left(\frac{2(52)}{2(52) + 48} \right) \left(\frac{165.4 \text{ lb}}{\text{h}} \right) = 7.90 \times 10^{-2} \frac{\text{lb}}{\text{h}} \quad Cr$$

$$Cs \quad \frac{5.23 \text{ Cs}_2O}{3838} \left(\frac{2(132.9)}{2(132.9) + 16} \right) 165.4 = 2.126 \times 10^{-1} \frac{\text{lb}}{\text{h}} \quad Cs$$

$$Mo_3 \quad \frac{5.40}{3838} \left(\frac{95.94}{95.94 + 48} \right) 165.4 = .1551 \frac{\text{lb}}{\text{h}} \quad Mo$$

$$SrO \quad \frac{4.41}{3838} \left(\frac{87.62}{87.62 + 16} \right) 165.4 = .1607 \frac{\text{lb}}{\text{h}} \quad Sr$$

$$K_2O \quad \frac{133.22}{3838} \left(\frac{2(39.10)}{2(39.10) + 16} \right) 165.4 = 4.766 \frac{\text{lb}}{\text{h}} \quad K$$

$$Na_2O \quad \frac{677.22}{3838} \left(\frac{2(22.99)}{2(22.99) + 16} \right) 165.4 = 21.67 \frac{\text{lb}}{\text{h}} \quad Na$$

Note: Cr_2O_3 result disagrees with table 5.8h of Duratek report. This probably relates to the discrepancy between analytical and target Cr_2O_3 contents of the feed.

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Title Duratek Method 5 Results

Project LLW vit

Prepared by:

Gray Whynett Gray Whynett

Date

10/3/95

Reviewed by:

MR Powell Mr Powell

Date

10/4/95

APPENDIX G

REVIEW OF ENVITCO TEST REPORT

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**Envitco, Inc. LLW Melter Demonstration
Entrainment and Volatility Estimates (MR Powell 9/1/95)
(Modified GA Whyatt 3/21/96)**

The Envitco melter run analytical data were examined and estimates made of the relative volatility and entrainment for each oxide analyte. These analyses and the results are described below. Volatility estimates were made both by comparing the melter feed and glass product compositions and by examining the aerosol collection rates and compositions. Assuming uniform entrainment of feed and zero volatility for a known low volatility component, the fractional volatility of each glass oxide species can be estimated from feed and glass compositions. The entrainment of melter feed was estimated based on the observed average aerosol collection rate on isokinetically-gathered filter samples. Errors were noted in the vendor-provided analysis of the off-gas data and these are described below.

Entrainment

Feed entrainment between 11:25 and 22:25 on 10/12/94 is estimated to have been on the order of 0.05% to 0.10% wt of the feed oxides. During this period of relatively steady operation, the wetted feed pellets were fed to the melter at a rate of approximately 50 lbs/h. Other periods of testing utilized either dried feed pellets or slurry feed, but insufficient off-gas characterization data are available to evaluate the entrainment of these feed types. The wetted feed pellets were made by adding roughly 7%wt water to the dried feed pellets. The water was added to suppress dust generation that was noted when the dried feed pellets were fed to the melter. Entrainment rates for the dried feed pellets and the slurry feed would likely be higher than that observed for the wetted feed pellets.

The 0.05-0.10% entrainment estimate was developed as follows. Two aerosol samples were collected during the 11-h mass balance period. These samples indicate that the total particulate flow rate was about 0.0345 lbs/h. Over 11 hours, this amounts to about 0.38 lbs of total particulate. During this same period, enough feed was delivered to the melter to produce about 468 lbs of glass assuming no entrainment and zero volatility (p.6-5 Envitco final report). If it is assumed that all of the particulate flow was due to entrainment (i.e., no volatility losses), then the rate of feed entrainment is estimated to be $(0.38/468) \times 100\% = 0.08 \text{ wt\%}$. If there were significant volatility losses of feed oxides the actual entrainment rate would be lower. An entrainment rate of 0.05 wt% is used for estimating fractional volatilities from aerosol composition data below. Comparing the measured aluminum aerosol flow rate with the aluminum feed rate yields an estimated 0.044% entrainment rate. These calculations are provided on an attached calculation worksheet.

This low entrainment rate is consistent with the overall mass balance given by Envitco in the final report. A total of 598 lbs of feed was delivered to the melter and a total of 466 lbs of glass was collected during the mass balance run. The feed mass includes 7 wt% water which was added to reduce dusting. The mass loss on ignition (LOI) for the spray-dried feed was measured

at 19.1 wt% on sample E1F3-006U. Envitco chose to average this result with a LOI data point for a feed sample prior to spray drying (E1F2-002U, 24.2%) to arrive at a LOI for the spray dried feed of 21.7 wt%. Laboratory samples are dried prior to measurement of LOI. However, in the spray dry operation some reaction apparently occurred which destroyed 20-30% of the nitrate/nitrite content of the feed. Although this destruction is much less than hoped for, this destruction is sufficient to make it inappropriate to apply pre-spray dry LOI results to post spray dry material. Thus, a LOI of 19.1 wt% is believed a superior choice. Adjusting for loss of water and LOI, the expected glass mass is $598(1-0.07)(1-0.191)=450$ lbs. This is in excellent agreement with the mass of glass collected (466 lbs). The error could be explained by only a 0.5 inch change in melt level. It is not certain what magnitude of melt level change could be detected.

Volatility

Fractional volatility estimates were generated for each of the glass oxide analytes. To make these calculations, it is necessary to select a component which is assumed to have zero volatility. Experience has shown that SiO_2 , SrO , CaO , Fe_2O_3 and Al_2O_3 are potentially good low volatility tie-components. In this case, Fe_2O_3 is not a good tie component because the Fe_2O_3 concentration in the glass was systematically increasing during the 11-h mass balance run presumably in response to the change from UN57 batch feed to UN6 batch feed which contained added iron. The CaO was not selected as a tie component because of the relatively low CaO concentration in the feed. The results were calculated for the remaining tie components SiO_2 , SrO and Al_2O_3 and then averaged (see attached spreadsheet). The volatility calculations were made using the average feed and glass composition data after normalization to 100% on an oxide basis. The variabilities of the feed and glass analyses were used to estimate the 95% confidence intervals for each oxide concentration. Systematic analytical errors are not included in these uncertainty estimates. The results are given in the attached spreadsheets and summarized in the table below. The NiO concentrations for both glass and feed samples were below the analytical detection limit so no results are presented for NiO .

The tie-component volatilities are within expected uncertainty of zero volatility for most components. The exceptions include Cr_2O_3 , ZrO_2 , SO_3 , and F. The Cr_2O_3 and ZrO_2 sources are attributed to refractory corrosion. Due to analytical difficulties, the target feed concentration (as a ratio to sodium) was used for both chloride and fluoride. The ratio to sodium was considered more accurate than the overall target because the sodium and Cl/F are introduced with the simulant while the feed appeared to be richer in silica than glass target values. The fluoride result indicates a source although no source of fluoride is known in the melter. This result may relate to a deviation from the estimated feed concentration. The final oxide showing a significant loss was SO_3 which was based on only 1 glass sample. Two analytical results in glass were available with the higher value being a less than detection limit result. Therefore, only the lower result was utilized. The second glass sample with a less than detection limit glass result would result in an estimate of >34% loss of SO_3 .

Estimate of Waste Oxide Volatilities Based on Tie Component Calculation. (Average result based on SiO ₂ , SrO and Al ₂ O ₃ tie components.)	
Oxide	Percent Selective Loss (negative indicates gain)
Al ₂ O ₃	-0.5 ± 7.9
B ₂ O ₃	-1.3 ± 18
BaO	-61 ± 62
CaO	14 ± 18
Cr ₂ O ₃	-13 ± 9 (refractory source)
Cs ₂ O	-6.4 ± 8.8
Fe ₂ O ₃	25 ± 65
K ₂ O	-1.3 ± 9.7
Li ₂ O	No Data
MgO	2.9 ± 10.7
MnO ₂	1.3 ± 69
MoO ₃	-1.1 ± 8.9
Na ₂ O	-0.7 ± 9.3
NiO	All feed and glass results below detection limits
P ₂ O ₅	-10.8 ± 18.5
SO ₃	52.7 (1 glass sample, no error estimate possible)
SiO ₂	0.8 ± 7.6
SrO	-0.3 ± 7.8
TiO ₂	-6.9 ± 16
ZrO ₂	-441.6 ± 155 (refractory source)
Cl ⁻	0.2 ± 8.0 (based on feed target)
F ⁻	-9.0 ± 7.3 (based on feed target)

Based on the tie-component method, it cannot be stated whether any of the analytes other than SO_3 exhibited a non-zero volatility during the 11-hour mass balance period. The SO_3 result is consistent with a second less than detection limit glass sample, as well as on-line monitors measuring SO_2 emissions. SO_2 emissions from the melter account for 37% of the feed sulfur content. In addition to SO_2 , there may be additional sulfur as filterable aerosols. Therefore, the result from the tie component calculation is believed consistent with the gas monitoring result.

An alternative method for estimating the volatilities of feed oxides is to examine the composition of the collected aerosol samples. Two filter samples were collected during, the 11-hour mass balance period. The samples were weighed by Trigon Engineering Consultants and provided to Quanterra for analysis to provide an estimate of the flow rate of each analyte to the off-gas system. The average of the two reported emission rates for each analyte was taken to be representative of the entire 11-hour run. Based on the average reported loss rate, the total mass loss of each analyte was computed and then compared to the mass of each analyte fed to the melter. The wt% of each analyte reported by Trigon lost to the off-gas line is shown in the table below. The spreadsheet used to generate these estimates is attached. Also shown in the table is the estimated volatility for each analyte based on an assumed feed entrainment rate of 0.05%wt. This entrainment rate was estimated from the fractional weight loss of alumina (0.044) and ferric oxide (0.055). Data for silica were not reported and the calcium oxide analytical data are suspect, so neither SiO_2 , or CaO can be used to provide entrainment estimates. Sulfur emissions were not quantified as part of these aerosol measurements.

Estimate of Waste Oxide Volatilities Based on Aerosol Composition		
Oxide	Weight % of Total Mass Lost	Estimated volatility (%) (assumes 0.05% wt Entrainment)
Al ₂ O ₃	0.044	0
B ₂ O ₃	0.186	0.14
CaO	17.2*	17.1*
Cr ₂ O ₃	0.022	0
Cs ₂ O	0.641	0.6
Fe ₂ O ₃	0.055	0
K ₂ O	<0.048	0
MnO ₂	0.179	0.13
MoO ₃	0.027	0
Na ₂ O	0.016	0
P ₂ O ₅	<0.056	0
SrO ₂	0.146	0.10
Cl ⁻	1.0 - 13.4	1.0 - 13.4
F ⁻	0.0 - 0.90	0 - 0.85

* This value is probably too high. See discussion in text following table.

Included in the table above are weight loss and volatility estimates for chlorine and fluorine. These values were determined from similar offgas sampling data using Method 26A. The calculations supporting the F and Cl values are given on the attached calculation worksheets. A range of values is given for both F and Cl because some of the analyses were reported as below detection limit. The % weight loss range for F and Cl was determined by assuming all values below detection limit were zero (establishing the lower limit of the range) or assuming they were equal to the detection limit (establishing, the higher limit of each range).

The volatility estimate for CaO is given in the table at 17.1 wt%. This is clearly too high based on previous experience. It has been established that CaO has a very low volatility. The 17.1 wt% estimate is likely the result of a high blank value for CaO, a failure to correct for the Method 29 blank analysis (see discussion under errors), and the fact that the CaO concentration

in the Envitco feed was considerably lower than many of the other vendor tests. Considering the improbability of a 17.1 % CaO volatility, it is recommended that zero volatility be assumed for calcium oxide in the Envitco melter. It should be noted, however, that a non-zero volatility result could result from selective entrainment of CaO. No mechanisms for selective CaO entrainment have been postulated. An extensive review of the aerosol sampling analytical work would be required to resolve whether selective entrainment of CaO is occurring.

Discussion of Errors in Final Envitco Report

As part of the above entrainment and volatility calculations it was necessary to carefully review the aerosol characterization information provided by Trigon in their report which is included as appendix G of the Envitco final report. During this review, several calculation errors and inconsistencies were noted. These are documented below. In view of these errors, it is recommended that the entire Trigon report be carefully reviewed to ensure there are no additional errors.

On page G-9 a summary table is presented giving the "Multiple Metal Sampling" data obtained from each of the 4 isokinetic aerosol samples. The "Metal Emission Rate" data given for the second filter sample are in error. The total "Particulate Emission Rate" is calculated correctly to be 0.043 lbs/hr, but the emission rates for each of the specific metals are incorrect. The error, it seems, resulted from the use of the "Run 1" column of total metal mass data given on page G-28 for the calculation of the respective "lbs/hr" data for both the "Run 1" and "Run 2" columns in the table on page G-29. Only the "Run 2" column is in error. The data columns for runs 1, 3, and 4 appear to be correct. The corrected data for the "Run 2" column on page G-29 (and the same data on page G-9) are given in the table below. This correction also applies to Table 4.6 on page 4-11.

Metal Species	Run 2 Emission Rate (lbs/hr)
Chromium	1.77E-06
Calcium	2.48E-03
Sodium	1.06E-03
Iron	2.93E-05
Boron	2.58E-06
Cesium	9.55E-05
Aluminum	2.71E-04
Potassium	< 2.21E-04
Phosphorus	< 2.21E-05
Manganese	4.28E-06
Molybdenum	1.16E-05
Strontium	5.91E-05

These corrected "Run 2" values were used when generating the volatility estimates given above. However, it appears there may be an additional error concerning this data. In the tables on page G-28 and G-29 a footnote states "Note: Values are not blank corrected". The data is then summarized on page G-9 and 4-11 and reported without performing a blank correction. The results of one method 29 field blank are included on page G-33. It is not possible to tell without original source information which (the footnote or the data treatment) is in error. Since not performing the blank correction results in conservatively high losses to the offgas, calculations were performed without the blank correction. However, the emission values adjusted for the method 29 blank are shown in the table below for the 4 isokinetic offgas samples.

Aerosol Mass Flows of Analytes in lb/hr After Correction for Method 29 Blank Analysis^(a)

Analyte	Sample #1	Sample#2	Sample #3	Sample #4
Al	1.63e-03	6.93E-05	4.77E-05	1.30E-03
B	0	0	0	7.65E-04
Ca	3.03e-3	5.16E-04	0	2.44E-03
Cr	3.77e-5	1.03E-06	7.46E-07	9.14E-06
Cs	9.30e-5	9.55E-05	5.49E-05	3.01E-04
Fe	6.59E-05	1.36E-05	1.36E-05	2.92E-04
K	<2.22E-04	<2.21E-04	3.03E-04	1.34E-03
Mn	0	4.03E-05	0	2.96E-06
Mo	9.61E-06	1.16E-05	2.12E-05	6.54E-05
Na	1.39E-04	4.75E-04	6.79E-04	5.56E-03
P	<2.22E-05	<2.21E-05	<2.24E-05	5.43E-05
Sr	3.92E-05	5.91E-05	1.34E-04	4.77E-04

^(a)Blank analysis results below detection limits are treated as zero results, samples below detection are calculated at the detection level and indicated as < values. Negative results after blank correction are shown as "0" values.

The lack of an expected trend in the data was noted in Table G-9. The table provides values in lb/hr units for individual analyte metals as well as a total particulate emission rate. The sum of the metal analytes do not compare in a predictable manner with the total particulate emissions. The total metal emissions and particulate emissions are reproduced below. Samples 1 and 2 correspond to the mass balance period. The data indicate that the total metals and the mass of aerosol collected are not well correlated (i.e. high masses of particulate collection do not result in high mass of metals collected). This indicates either analytical problems or possibly that the nature of the aerosol changed significantly between different sample times. Analytical difficulties are considered the more likely cause.

	Sample #1	Sample #2	Sample #3	Sample #4
Total Particulate Emission lb/hr	0.026	0.043	0.038	0.114
Total Metal Emission lb/hr	0.00918	0.00659	0.00508	0.0229
% of mass accounted for by metals	35	15	13	20

It is recommended that the analyte mass data given on page G-28 be verified against the aerosol sample chemical analyses. These analyses were apparently performed by Quanterra directly for Trigon and are therefore not included in the compilation of analytical data for the LLW melter tests. It would be useful to have the aerosol sample analytical data included in the Trigon report to maintain traceability of the reported aerosol flow rates.

On page 4-7 of the Envitco report there is a discussion of NO_x generation and conversion rates. The sentence ending "gives an offgas rate of 3.4 g NO_x /min" is incorrect. Multiplying a flow rate of 1.56 m³/min by a NO_x concentration of 5.56 g/m³ gives an offgas rate of 8.7 g NO_x /min. This change results in the "1.31 gN/min" value in the next sentence being changed to 3.20 g N/min. The "indicated nitrate reduction factor" of 76% given in the next paragraph is apparently correct $((13.0-3.2)/13 \times 100\% = 75\%)$. The calculation of 5560 mg/m³ NO_x from 5000 ppm at 130°C and 1 atm should also be checked. Assuming ideal gas behavior and a 50/50 mol% mixture of NO and NO_2 gives 5.745 g/m³ NO_x .

Page References in Attached Calculations

The page references for data in the attached calculations refer to the draft Envitco test report. To adjust the references to the final Envitco report change appendix D to appendix G and add 2 to the page number. For example page D22 becomes G24, D23 becomes G25, etc.

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Estimation of maximum total entrainment - Envtco

The Trigon off-gas report (Appendix E in Envtco report):

See 9055

Envt
Report
Page #

Sample #1: Total particulate catch = 176.5 mg page D-23 G 25
 Gas volume sampled = 41.15 dscf page D-22 G 24
 Total off-gas flow rate = 46.0 dscfm page D-23 G 25

Sample #2: Total particulate catch = 294.6 mg page D-23 G 25
 Gas volume sampled = 40.73 dscf page D-22 G 24
 Total off-gas flow rate = 45.4 dscfm page D-23 G 25

Average particulate flow rate:

$$= \left(\frac{176.5 \text{ mg}}{41.15 \text{ dscf}} + \frac{294.6 \text{ mg}}{40.73 \text{ dscf}} \right) \left(\frac{1}{2} \right) \left[(46.0 \text{ dscfm} + 45.4 \text{ dscfm}) \frac{1}{2} \right]$$

$$= 263.3 \text{ mg particulate/min} = 15.8 \text{ g/h}$$

$$= 0.0348 \text{ lb/h particulate}$$

over 11 hours, total particulate = $(11)(0.0348) = \underline{0.383 \text{ lbs}}$ Total theoretical glass production $\cong 468 \text{ lbs}$ (page X-8 Envtco report)

Assuming the particulate are in the form of glass oxides, the maximum entrainment is:

$$\text{Max Ent} = \frac{0.383 \text{ lbs}}{468 \text{ lbs}} \times 100\% = \underline{0.082\%} \leftarrow$$

Because some of the sample mass is likely due to volatilization, the actual entrainment is probably lower.

Assuming Aluminum aerosol flow data on page C-27 are accurate, the entrainment can also be estimated:

$$\% \text{ Ent} \cong \frac{\left[(1.83 \times 10^{-3} \text{ lb Al}) + (2.71 \times 10^{-4} \text{ lb Al}) \right] / (11.0 \text{ h}) \left(\frac{1}{2} \right)}{\left(\frac{10.16 \text{ Al}_2\text{O}_3}{100\%} \right) \left(\frac{54 \text{ Al}}{102 \text{ Al}_2\text{O}_3} \right) (468 \text{ lbs theoretical glass})} \times 100\% = \underline{0.044\% \text{ Ent}}$$

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Actual entrainment is probably about 0.04-0.05% wt.

Title Entrainment estimates		Project LLW - Envtco	
Prepared by: M. E. Swell	Date 9/1/95	Reviewed by: [Signature]	Date 10/4/96

Title Calculation of % Chloride lbs		Reviewed by [Signature]		Date 9/1/95	
Project LW Enviro		Prepared by M. J. [Signature]		Date 10/6/95	

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These estimates are within exp. uncertainty of each other.

% CI lost to offgas:

Feed basis: $MM = \frac{0.0146 \text{ lbs Cl}}{0.97 \text{ lbs}} \times 100\% = 1.5\%$

Class basis: $MM = \frac{0.130 \text{ lbs}}{0.97 \text{ lbs}} \times 100\% = 13.4\%$ → MAX.

Class basis: $MM = \frac{0.0146 \text{ lbs Cl}}{0.130 \text{ lbs}} \times 100\% = 1.0\%$ → MIN.

MAX = $\frac{0.130}{0.130} \times 100\% = 1.50$

MIN = $\frac{0.0146}{0.0146} \times 100\% = 8.7\%$

Total CI fed to water:

Feed basis: $\left(\frac{0.0207\% \text{ CI}}{100\%} \right) \left(\frac{78.2 \text{ lbs feed}}{100 \text{ lbs feed}} \right) \left(\frac{100\%}{100\%} \right) = 0.016 \text{ lbs CI} \pm 0.47$

Class product basis: $\left(\frac{0.32\% \text{ CI}}{100\%} \right) \left(\frac{460 \text{ lbs glass theoretical}}{100\%} \right) \left(\frac{100\%}{100\%} \right) = 1.50 \text{ lbs CI} \pm 0.61$

Since CI was only detected in Sample #1, the minimum CI loss rate will be calculated from that value (2.65 x 10⁻² lbs/h).

The Max. loss rate will be calculated assuming all concentrations currently listed as below detection limit are at the detection limit.

Minimum CI flow rate $\approx (2.65 \times 10^{-2} \text{ lbs/h}) \left(\frac{1}{1.33 \times 10^{-2}} \right) = 1.33 \times 10^{-2} \text{ lbs/h}$

Min total CI lost $\approx (1.33 \times 10^{-2} \text{ lbs/h}) \left(\frac{1}{1.33 \times 10^{-2}} \right) = 0.0146 \text{ lbs CI}$

Maximum CI flow rate $\approx \left(\frac{6.44 \times 10^{-2} + 9.99 \times 10^{-2}}{36.5} + \frac{1}{2} \left(\frac{4.11 \times 10^{-2} + 7.14 \times 10^{-2}}{36.5} \right) \right) \left(\frac{1}{1.33 \times 10^{-2}} \right) = 0.0119 \text{ lbs/h CI}$

∴ Max total CI lost $\approx (0.0119)(11 \text{ h}) = 0.130 \text{ lbs CI}$

Calculation of Chlorine flow in offgas (Enviro mass balance)

From the Trigon report (Appendix D in Enviro report, page D-38) (Final Report)

Species	Aerosol sample #1	Sample #2
HCl	$6.49 \times 10^{-2} \text{ lbs/h}$	$9.99 \times 10^{-2} \text{ lbs/h}$
Cl ₂	4.11×10^{-2}	7.14×10^{-2}
CI-	2.65×10^{-2}	3.92×10^{-2}

Calculation of fluoride flow in off-gas (Enviro mass balance).

from the Trench report (Appendix C in Enviro report):

Species	H ₂ F	F ₂
Aerosol Sample #1	$< 3.39 \times 10^{-4}$ lbs/y $< 2.06 \times 10^{-4}$ y $< 7.38 \times 10^{-5}$	
Aerosol Sample #2	$< 5.1 \times 10^{-4}$ y $< 3.67 \times 10^{-4}$ y $< 1.96 \times 10^{-4}$	

Since all fluoride analyses were below detectable limits, the minimum fluoride flow in the off-gas line is taken to be 0 lbs/y.

The maximum fluoride flow rate would be that with all actual concentrations just equal to detection limit. The maximum fluoride flow is thus estimated to be:

$$\text{Total } F^- = \left[(3.25 \times 10^{-4} \text{ y} + 5.1 \times 10^{-4} \text{ y}) \left(\frac{19.4}{20} \right) \right] + (2.06 \times 10^{-4} \text{ y} + 3.67 \times 10^{-4} \text{ y}) \left(\frac{2}{2} \right) + (7.38 \times 10^{-5} \text{ y} + 1.96 \times 10^{-4} \text{ y}) \left(\frac{2}{2} \right)$$

$$\Rightarrow \text{lbs/y } F^- = 8.25 \times 10^{-4} \text{ lbs/y}$$

During the 11-L mass balance run, the total F delivered to the melt is:

$$(598 \text{ lbs}) \left(\frac{78.3 \text{ lbs glass}}{100 \text{ lbs feed}} \right) \left(0.2225 \text{ wt\% } F \right) \div 100\% = 1.04 \text{ lbs } F^-$$

(assuming 598 lbs dry feed) (dry F in feed)

The total % loss of F to the off-gas system is then

$$\% \text{ } F^- \text{ loss} = \frac{(8.25 \times 10^{-4} \text{ lbs}) (11.01)}{0.974 \text{ lbs}} = 0.87 \text{ wt\%}$$

Usual total F found in the glass as a basis (which is probably more accurate given the uncertainty over whether 598 lbs feed is wet or dry => $(418 \text{ lbs glass}) \left(\frac{0.292 \text{ wt\% } F}{100\%} \right) \left(\frac{11.01}{8.25 \times 10^{-4} \text{ lbs}} \right) = 0.66 \text{ wt\%} \sim 0.7 \text{ wt\%}$

Title: Calculation of % fluoride loss
 Project: LLU - Enviro
 Prepared by: M. F. Powell
 Date: 8/31/95
 Reviewed by: [Signature]
 Date: 10/19/95

MR Powell 8/29/95----Revised GA Whyatt 3/21/96

Envitco Data: Estimated Oxide Volatilities based on glass and feed compositions and tie components

Mass Balance run data 10/12/94 at 11:25 to 22:25

	Average		Average		Tie =		Tie =		Tie=		Avg		Propagate
	Glass	95% CI	Feed	95% CI	SiO2	95% CI	Sr	95% CI	Al2O3	95% CI	Sr	SiO2	Error
					Vi		Vi		Vi		in %		95% CI
Al2O3	10.89023	0.150769	10.78554	1.045446	-0.01346	0.136954	-0.00282	0.137294	0	0.138471	-0.54255	7.942874	
B2O3	8.338925	0.643894	8.197217	2.256569	-0.02107	0.307032	-0.01034	0.304616	-0.00751	0.304479	-1.29741	17.63101	
BaO	0.004753	0.000417	0.002937	0.001916	-0.62422	1.07947	-0.60716	1.068715	-0.60265	1.066232	-61.1347	61.86237	
CaO	0.073347	0.015331	0.085184	0.025068	0.135754	0.322176	0.144832	0.319345	0.147233	0.318939	14.26065	18.48426	
Cr2O3	0.045432	0.001671	0.039912	0.003681	-0.14253	0.155546	-0.13053	0.155902	-0.12736	0.157214	-13.3476	9.019495	
Cs2O	0.14068	0.012569	0.131654	0.007877	-0.07253	0.152571	-0.06127	0.152758	-0.05829	0.153903	-6.40301	8.837975	
Fe2O3	0.348989	0.181848	0.464409	0.653595	0.245736	1.134126	0.253659	1.122333	0.255755	1.119288	25.17166	64.96733	
K2O	1.353614	0.031695	1.330708	0.178887	-0.021	0.168677	-0.01027	0.168374	-0.00744	0.169197	-1.2901	9.742789	
Li2O	0	0	0	0									
MgO	0.034396	0.002513	0.035284	0.00521	0.021565	0.18517	0.031843	0.184455	0.034561	0.185025	2.93231	10.67426	
MnO2	0.005917	0.001941	0.005971	0.006963	0.005453	1.208218	0.0159	1.195722	0.018663	1.192539	1.33386	69.21537	
MoO3	0.144554	0.001421	0.142391	0.016924	-0.01897	0.154191	-0.00826	0.15417	-0.00543	0.155146	-1.08865	8.920243	
Na2O	17.88739	0.358856	17.68505	2.227158	-0.0152	0.160313	-0.00454	0.160156	-0.00172	0.161053	-0.7153	9.266923	
NiO													
P2O5	0.220334	0.020194	0.197975	0.050786	-0.11708	0.321594	-0.10534	0.319141	-0.10224	0.319065	-10.8218	18.47147	
SO3	0.102759		0.216141	0.042232	0.522804		0.527816		0.529142		52.65875	0	
SiO2	59.9202	0.907814	60.14299	5.526858	0	0.131714	0.010504	0.13213	0.013282	0.13334	0.792864	7.643918	
SrO	0.0959	0.001634	0.095246	0.008969	-0.01062	0.13495	0	0.135327	0.002808	0.136524	-0.26025	7.829003	
TiO2	0.019252	0.004656	0.017941	0	-0.07709	0.279151	-0.06577	0.277209	-0.06278	0.277309	-8.85479	16.04406	
ZrO2	0.032416	0.006965	0.00596	0.002604	-4.45922	2.706038	-4.40188	2.680242	-4.38671	2.675052	-441.594	155.1424	
Cl*	0.327929	0.032937	0.327173		-0.00604	0.137804	0.004531	0.138098	0.007326	0.139242	0.194063	7.989522	
F*	0.300016	0.019533	0.274118		-0.09855	0.124834	-0.08701	0.125809	-0.08396	0.127455	-8.98369	7.276775	
* due to discrepancy in feed analysis Cl and F are set at feed target based on Na content in feed samples													

* due to discrepancy in feed analysis Cl and F are set at feed target based on Na content in feed samples

Revised by
M. J. Powell
3/27/96

Sheet 1

Analytical Composition Data										Feed Sample Data										Feed sample normalized to 100% oxide										Glass Sample Data									
Glass Sample Data										E155										E155										E155									
-015C1 -017C										-015C1 -017C										-015C1 -017C										-015C1 -017C									
Al2O3	16.975	17.772	25.025	6.66658	6.64641	8.37304	7.43606	10.9692	0.15077	0.7715	8.453	0.0691	8.7325	10.5363	11.1198	9.9654	11.4906	10.7655	10.4545	12																			
SiO2	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	51.51	9																		
CaO	0.045	0.0475	0.0425	0.0645	0.0648	0.04438	0.04325	0.04635	0.03475	0.00442	0.01	0.002	0.003	0.00136	0.00293	0.00381	0.00395	0.00254	0.00192	0.003																			
BaO	0.0685	0.07375	0.061	0.06375	0.07039	0.07564	0.0832	0.09133	0.06868	0.02335	0.076	0.04973	0.07	0.06325	0.1532	0.05936	0.03874	0.0361	0.03691	0.00658	0.14																		
CaF2	0.0455	0.04475	0.044	0.04675	0.04658	0.04307	0.04328	0.04543	0.04343	0.01357	0.0159	0.06975	0.1	0.13069	0.13569	0.12604	0.13165	0.10788	0.1	0.0788	0.05																		
CaO	0.135	0.135	0.13	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.1476	0.003																			
SiO2	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.08355	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.135	0.135	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.13725	0.003																			
SiO2	0.135	0.1

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Revision 0

CALCULATION WORKSHEET

Calc. No. _____

Page _____ of _____

Enviro SO₂ data3 sample periods for SO₂ emission during mass balance period

(pg 4-6)	Time	SO ₂ Rate	feed
10/12/94	11:28 - 12:27	.015 kg/h	wetted un 57
10/12/94	13:05 - 14:04	.014 kg/h	wetted un 57
10/12/94	15:00 - 15:59	.015 kg/h	wetted un 6

Avg glass production rate = 22.7 kg/hr

Glass Target SO₃ content = 0.22 wt%% Sulfur accounted for by SO₂ emissions

$$\frac{\left(\frac{.015 + .014 + .015}{3} \right) \frac{\text{kg SO}_2}{\text{hr}} \left(\frac{32.06 + 3(16)}{32.06 + 2(16)} \right)}{22.7 \frac{\text{kg}}{\text{hr}} \left(\frac{.0022 \text{ kg SO}_3}{\text{kg glass}} \right)} = 0.367 \text{ or } \underline{36.7\%}$$

Note: on pg G-47 SO₃ monitor exceeds instrument scale and was > 99.3 from 15:25 - 15:40. Actual SO₂ emissions may be higher than estimated.

Also - no data available on aerosol sulfur content so total emissions may be greater by unknown amount.

Title

Enviro SO₂ emissions

Project

LLW Vitrification

Prepared by:

Greg Whyatt Greg Whyatt

Date

3/22/96

Reviewed by:

Mr. [Signature]

Date

3/24/96

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APPENDIX H

MISCELLANEOUS SUPPORTING CALCULATIONS

- H.1 Estimation of NO_x Yields
- H.2 Estimation of Iodine Volatility
- H.3 Total Loss Calculation

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CALCULATION WORKSHEET

Calc. No. H1.1

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B+W NO_x yield based on final report WHC-SD-WM-VI-024 Rev 0.

Method 5 Flow measurements by ETS (pg D1-3)

$$(880 + 840 + 842 + 876 + 839) / 5 = \underline{855 \text{ dscfm}}$$

Note: results in Table 5-1 appear inconsistent with flow measurement above
results in appendix are assumed correct

Final report appears to be void of any NO_x analyzer information.

Data from preliminary report provided the following 5-minute
average data for NO_x at dry scrubber inlet (See Table 7)

(10/13)	830	1082	938	901	
(14/00)	925	985	904		end @ 11/14 11:00
	1021	1012	903		
	1066	989	872		Average = <u>942 ppm NO_x</u>
	963	1034	920		
	999	992	426		
	1041	1044	865		

Feed Source

From notes taken during test, target feed was 100 lb/hr mixed feed
(Also this rate is consistent with preliminary data report)

NO₂, NO₂ content from Sample analysis

	NO ₂ wt%	NO ₂ wt%
B1F7-002C1 (s)	6.82	2.90
B1F7-005C2 (s)	6.56	2.79
B1F7-008C3 (s)	7.58	3.23
B1F7-013C (s)	8.33	3.65

Avg 7.32% 3.14%

$$\text{Source} = 100 \frac{\text{lb}}{\text{h}} \left(\frac{\text{h}}{60 \text{ min}} \right) \left(\frac{453.6 \text{ g}}{16} \right) \left[0.0732 \left(\frac{\text{mol N}}{14 + 48 \text{ g NO}_2} \right) + 0.0314 \left(\frac{\text{mol N}}{14 + 32 \text{ g NO}_2} \right) \right] = 1.41 \frac{\text{mol}}{\text{min}}$$

$$\text{Observed NO}_x = \left(855 \frac{\text{dscf}}{\text{min}} \right) \left(\frac{273.2}{288.8} \right) \left(\frac{28.317 \text{ L}}{\text{ft}^3} \right) \left(\frac{\text{g mol}}{22.4 \text{ L}} \right) \left(\frac{942 \text{ ppm}}{10^6} \right) = .963 \frac{\text{mol}}{\text{min}}$$

$$\text{yield} = .963 / 1.41 = \underline{.68 \%}$$

Note: Results are for no return / No SNCR

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Title

B+W NO_x Yield

Project

LLW Kit

Prepared by:

Greg Whyatt Greg Whyatt

Date

10/2/95

Reviewed by:

MR. Powell M. Tamm

Date

10/3/95

Battelle
Pacific Northwest Laboratories

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Calc. No. H1.2

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WSTC NO_x yield

on-line instrument 2100-2300 ppm independent of feed (pg 7-10)

Caution: Bomb sampler range 5000-50,000, 17,500 ppm Average (pg 7-7)

Flow rate: Argon bomb samples failed, therefore use method 5 flow measurements. Disregard WSTC effort to make orifice calculation across charge door and add back flows. I have much more confidence in pitot measurements.

$$\text{Arg flow} = \frac{(58.78 + 53.15 + 57.70 + 56.40)}{4} = 56.51 \text{ dry std m}^3/\text{min}$$

Source Data

feed rate 3.77 l/min simulant (pg 5-19)

Simulant Sample WISOS-40462(P) $\frac{\text{NO}_2 \text{ mol/L}}{2.56}$ $\frac{\text{NO}_2 \text{ mol/L}}{2.11}$ $\frac{\text{Total N/L}}{4.67 \text{ mol N/L}}$

discrepancies

This is slightly different than report value of pg 5-21 NO₂ = 2.52 mol/L NO₂ = 2.16 mol/L

Also

Note: Lobben indicator simulant at NO₂ = 3.11 NO₂ = 1.67

$$\text{Source } (3.77 \text{ l/min}) (4.67 \frac{\text{mol N}}{\text{L}}) = 17.61 \frac{\text{mol}}{\text{min}}$$

detected on-line, assume: (60°F std temp, line ~ 1 atm)

$$(56.51 \frac{\text{dry m}^3}{\text{min}}) \left(\frac{278.2}{288.8} \right) \left(\frac{1000 \text{ L}}{\text{m}^3} \right) \left(\frac{\text{mol}}{22.4 \text{ L}} \right) = 2387 \times 10^3 \frac{\text{mol}}{\text{min}} \text{ total}$$

$$\left(\frac{2200}{10^6} \text{ NO}_x \right) \left(2387 \times 10^3 \frac{\text{mol}}{\text{min}} \right) = 5.25 \frac{\text{mol}}{\text{min}}$$

$$\text{yield} = \left(\frac{5.25}{17.61} \right) 100 = 29.8 \% \text{ yield}$$

Note Flow indicated is ~ 2000 scfm, which is too high for cupola shift

pg 8-9

$$\text{Air injection } (5.10 + 11.94 + 8.3 \frac{\text{kg}}{\text{min}}) \left(\frac{1000 \text{ g}}{\text{kg}} \right) \left(\frac{\text{mol}}{29.5 \text{ g}} \right) = 620 \frac{\text{mol}}{\text{min}} \text{ total}$$

$$\left[\left(\frac{2200}{10^6} \right) (620) \right] (17.61) \times 100 = 7.75 \% \text{ yield} \leftarrow \text{CEM}$$

$$\text{If bomb sample believed } \left[\left(\frac{17500 \text{ ppm}}{10^6} \right) (620 \frac{\text{mol}}{\text{min}}) \right] / 17.61 \times 100\% = 62 \% \text{ yield} \leftarrow \text{bomb sample}$$

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Title

WSTC NO_x yield

Project

LLW Vit

Prepared by:

Greg Whyatt

Date

10/2/95

Reviewed by:

MR Powell

Date

10/3/95

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CALCULATION WORKSHEET

Calc. No. H13Page 1 of 1USBM NO_x Yield

Data from Harry Smith for Sample USBM-M1-BPW-011P (lab data)

CO ₂	122.1 mmol
H ₂	132 mmol
NO _x	23 mmol
N ₂ O	18.8 mmol
N ₂	78.2 mmol
NH ₃	400 mg ($\frac{\text{mmol}}{17 \text{ mg}}$) = 23.5 mmol

$$\text{feed prep yield} = \frac{23}{(23 + 2(18.8) + 2(78.2) + 23.5)} = \underline{9.56\%}$$

Offgas Data from Meltersverify that furnace is not a significant factor in NO_x yield:

pg 34 final report 3/9 @ ~22:25 feed rate = 750 lb/h

Type A feed, Na analysis results = (15.3 + 15.5 + 17.6)/3 = 16.1 wt% Na

Feed simulant target values Na = 10 M NO₂ = 3.11 M NO₂ = 1.67 MEntropy report pg C48 approx 2.6 ppm NO_x for 641-741 on 3/9Entropy Report pg A14 cyclone inlet flow (unclear if J/n/total) = 223 dscfm
data taken @ 3/9 23:50 only measurement provided. STD = 68°F

$$\left(750 \frac{\text{lb}}{\text{h}} \right) \left(\frac{0.161 \text{ lb Na}}{\text{lb feed}} \right) \left(\frac{453.6 \text{ g}}{\text{lb}} \right) \left(\frac{\text{mol Na}_2\text{O}}{46 \text{ g Na}} \right) \left(\frac{3.11 + 1.67 \text{ mol N}}{10 \text{ mol Na}_2\text{O}} \right) \left(\frac{\text{h}}{60 \text{ min}} \right) = 9.49 \text{ mol/min}$$

$$\left(223 \frac{\text{dscf}}{\text{min}} \right) \left(\frac{35.317 \text{ l}}{\text{ft}^3} \right) \left(\frac{\text{mol}}{22.4 \text{ l}} \right) \left(\frac{273}{293} \right) \left(\frac{2.6 \text{ NO}_x}{10^6 \text{ gas}} \right) = 6.83 \times 10^{-4} \text{ mol/min}$$

$$\frac{6.83 \times 10^{-4}}{9.49} \times 100\% = .007\% \\ \therefore \text{negligible}$$

$$\text{NO}_x \text{ yield} = \underline{9.6\%} \quad \leftarrow$$

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Title

USBM NO_x Yield

Project

LLW vit

Prepared by:

Graig Whyatt *Graig Whyatt*

Date

10/2/95

Reviewed by:

MR Powell *MR Powell*

Date

10/2/95

VECTRA NOx YIELD MT2, Run #102

Omni report:

Run #1 360 ppm NO_x (349 ppm NO) dry basis
Flow 2.95 dscm/min STP 21.1°C 1atm
4/21 23:05 to 4/22 00:05
Filter (70% 5666) = 39.66 mg/dscm NO₂
Filter (0.0126 5666) = .68 mg/dscm NO₃

Run #2 235 ppm NO_x (224 ppm NO) dry basis
Flow 2.81 dscm/min
4/22 11:56 to 4/22 12:56
Filter .718 % (4236) = 30.41 mg/dscm NO₂
1027 % (4236) = 1.14 mg/dscm NO₃

1923 report 865 kg slurry feed added over 14 hrs consisting of:

141 kg batch #1 @ 49.72 wt% simulant

724 kg batch 20, 39 @ 46.38 wt% simulant

Note 57% by weight is glass

Appendix 8.3

Mass balance Sodium conclude 10 M simulant

density @ 10 M Na = 1.42 kg/l NO₂⁻ = 3.11 M
NO₃⁻ = 1.67 M

Source: Lokken + Martin, May 1994, "Makeup Procedures and Characterization Data for Simulated Low-level Wastes"

Potential Source of N

$$\left[141 \text{ Kg } (.4972) \left(\frac{1}{1.42 \text{ kg sim}} \right) \left(3.11 + 1.67 \frac{\text{mol N}}{\text{kg}} \right) + 724 (.4638) \left(\frac{1}{1.42 \text{ kg}} \right) \left(3.11 + 1.67 \frac{\text{mol N}}{\text{kg}} \right) \right] \\ \div (14 \text{ hr} \cdot \frac{60 \text{ min}}{\text{hr}}) = 1.63 \frac{\text{mol}}{\text{min}} \text{ N source}$$

Run #1 Rate

$$\left(2.95 \frac{\text{m}^3}{\text{min}} \left(\frac{1000 \text{ L}}{\text{m}^3} \right) \left(\frac{360 \text{ N}}{10^6 \text{ tot}} \right) \right) = 4.406 \times 10^{-2} \frac{\text{mol N}}{\text{min}} \text{ gas}$$

$$\left[39.66 \frac{\text{mg}}{\text{dscm}} \left(\frac{1}{1000 \text{ mg}} \right) \left(\frac{\text{mol}}{14+32} \right) + (.68 \frac{\text{mg}}{1000 \text{ mg}}) \left(\frac{\text{mol}}{14+48} \right) \right] 2.95 \frac{\text{m}^3}{\text{min}} = 2.58 \times 10^{-3} \frac{\text{mol}}{\text{min}} \text{ particulate}$$

$$\text{total} = 4.66 \times 10^{-2} \frac{\text{mol}}{\text{min}}$$

$$\text{yield} = 4.46 \times 10^{-2} / 1.63 = 2.74 \%$$

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Title <u>Vectra NOx Yield</u>		Project <u>LLW Vit.</u>	
Prepared by: <u>Greg Whyatt</u>	Date <u>10/3/95</u>	Reviewed by: <u>MR Powell</u>	Date <u>10/3/95</u>

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Run #2 Rate

$$2.81 \frac{\text{m}^3}{\text{min}} \cdot \frac{1000 \text{ L}}{\text{m}^3} \cdot \frac{\text{gmol}}{24.1 \text{ L}} \cdot \frac{(235 \text{ N})}{10^6} = 2.74 \times 10^{-2}$$

on Method 5

$$\left[30.41 \frac{\text{mg}}{\text{dscm}} \left(\frac{\text{g}}{1000} \right) \left(\frac{\text{mol}}{14+32} \right) + 1.14 \left(\frac{\text{g}}{1000} \right) \left(\frac{\text{mol}}{14+48} \right) \right] 2.81 \frac{\text{m}^3}{\text{min}} = 1.9 \times 10^{-3} \text{ Particulate emissions (not NO}_x\text{)}$$

$$\text{NO}_x \text{ yield} = \frac{2.74 \times 10^{-2}}{1.63} = 1.68\% \text{ yield}$$

$$\text{Particulate yield} = \frac{1.9 \times 10^{-3} + 2.58 \times 10^{-3}}{1.63} = 0.28\% \text{ Particulate}$$

$$\text{NO}_x \text{ yield Arg} = \frac{2.74 + 1.68}{2} = \underline{\underline{2.21\%}} \leftarrow$$

General check on assumptions based on Na

$$865 \text{ kg feed} \left(0.57 \frac{\text{kg glass}}{\text{kg feed}} \right) = 493 \text{ kg glass}$$

$$\left[(141 \text{ kg}) \left(0.4972 \frac{\text{kg simulant}}{\text{kg feed}} \right) + (724) \left(0.4638 \right) \right] \left(\frac{\text{L}}{1.42 \text{ kg}} \right) \left(\frac{10 \text{ mol Na}}{5} \right) \left(\frac{\text{mol Na}_2\text{O}}{2 \text{ mol Na}} \right) \left(\frac{46+16 \cdot 5}{\text{mol}} \right) \left(\frac{\text{kg}}{1000 \text{ g}} \right) = 88.62 \text{ kg Na}_2\text{O}$$

$$\text{wt \% Na}_2\text{O with no loss} = \frac{88.62}{493} \cdot 100 = 18.0\%$$

Actual glass composition 15.54 %

$$\text{Based on this increase estimate by } 2.21 \left(\frac{18.0}{15.54} \right) = \underline{\underline{2.6\%}} \leftarrow$$

$$\text{particulate emission } .28 \left(\frac{18}{15.54} \right) = 0.32\%$$

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Title

Vectra NO_x Yield

Project

LLW Vit

Prepared by:

Greg Whynatt

Date

10/3/95

Reviewed by:

W.R. Farrell

Date

10/3/95

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Calc. No. HLSPage 1 of 2Duratek NO_x yield

Claim pg 6-6 < 9% emitted past scrubber during steady state period

claim pg 6-7 < 20% with urea

Steady state period 1/19/95 $\rightarrow$ 1/21/95pg 5-2 avg ss feed = 1.55 $\frac{\text{kg}}{\text{min}}$ - but density unreliable

Slurry Analysis

DIF3-111 C(s) $\text{Si} = 22.1$ $\text{NO}_3 = 6.61$ $\text{NO}_2 = 2.63$ DIF3-112 C(s) 24.3 6.42 2.60 DIF3-114 C(s) 23.2 6.60 2.65 Glass Analysis, % SiO_2 41.4 (avg of 8)

Glass Produced 1800 kg/day (pg 5-2)

NO_x Run #1 6445 ppm NO_x
 Run #2 4099
 Run #4 2911

Run #3 likely a problem

Take Flow @ 90 SCFM $\text{std} = 60^\circ\text{F}$

$$1800 \frac{\text{kg}}{\text{day}} \left(\frac{\text{day}}{24 \text{ hr}} \right) \left(\frac{\text{hr}}{60 \text{ min}} \right) \left(\frac{41.4 \text{ \% } \text{SiO}_2}{\text{kg glass}} \right) = .5175 \frac{\text{kg SiO}_2}{\text{min}}$$

slurry samples:

$$111 \text{ C(s)} \left[6.61 \text{ } \text{NO}_3 \left(\frac{\text{mol N}}{14 + 48} \right) + 2.63 \text{ } \text{NO}_2 \left(\frac{\text{mol N}}{14 + 32} \right) \right] / 22.1 \text{ \% } \text{SiO}_2 = 7.41 \times 10^{-3}$$

$$112 \text{ C(s)} \left[6.42 \left(\frac{\text{mol}}{62} \right) + 2.60 \left(\frac{\text{mol}}{46} \right) \right] / 24.3 = 6.59 \times 10^{-3}$$

$$114 \text{ C(s)} \left[6.60 \left(\frac{1}{62} \right) + 2.65 \left(\frac{1}{46} \right) \right] / 23.2 = 7.07 \times 10^{-3}$$

$$\text{Avg} = 7.02 \times 10^{-3} \frac{\text{mol N}}{\text{g SiO}_2}$$

$$\text{or } 7.02 \frac{\text{mol N}}{\text{kg SiO}_2}$$

$$\text{Source rate} = .5175 \frac{\text{kg SiO}_2}{\text{min}} \cdot 7.02 \frac{\text{mol N}}{\text{kg SiO}_2} = 3.64 \frac{\text{mol N}}{\text{min}}$$

$$\text{Offgas} (90 \text{ scfm}) \left(\frac{273.2}{288.8} \right) \left(\frac{26.317}{4.3} \right) \left(\frac{\text{mol}}{22.4 \text{ L}} \right) = 107.6 \frac{\text{mol}}{\text{min}} \text{ total}$$

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Title <u>Duratek NO_x Yield</u>		Project <u>LLW Vit.</u>	
Prepared by: <u>Greg Whyatt</u>	Date <u>10/3/95</u>	Reviewed by: <u>MR Powell</u>	Date <u>10/3/95</u>

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Calc. No. 141.5

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$$\left[107.6 \frac{\text{mol}}{\text{min}} \left(\frac{6445}{10^6} \right) / 3.64 \right] \times 100 = 19.1$$

$$\left[107.6 \frac{\text{mol}}{\text{min}} \left(\frac{4029}{10^6} \right) / 3.64 \right] \times 100 = 12.1$$

$$\left[107.6 \frac{\text{mol}}{\text{min}} \left(\frac{2911}{10^6} \right) / 3.64 \right] \times 100 = 8.6$$

Avg = 13.3 yield

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Title <u>Duratek NO_x yield</u>		Project <u>LLW vit</u>	
Prepared by: <u>Greg Whyatt</u>	Date <u>10/3/85</u>	Reviewed by: <u>MR Powell</u>	Date <u>10/3/85</u>

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Calc. No. H-16Page 1 of 1

Conditions pg VIII-13 *Envitco NO_x yield*
 Average Flow 55-60 CFM @ 125-150°F (assume flow duct in, P_{atm})
 Feed 60-80 lb/h
 offgas NO_x 5000 ppm
 dried feed 4.0 wt% NO₂ pg C-3
 9.8 wt% NO₃ pg C-3
 Remaining NO₂/NO₃ after spray dry = 70-80% of starting pg C-2 sec. 2
 wetted feed material ~ 7 wt% H₂O

assume quoted rate is wetted feed (setting on gravimetric feeder)

$$\left(70 \frac{\text{lb}}{\text{h}}\right) \left(\frac{9.3 \text{ lb dry feed}}{100 \text{ lb wet feed}}\right) \left[0.04 \left(\frac{14}{14+32}\right) + 0.98 \left(\frac{14}{14+48}\right)\right] \left[\frac{453.6 \text{ g}}{16 \text{ N}} \right] \left[\frac{1 \text{ h}}{149 \text{ g N}}\right] \left[\frac{1}{60 \text{ min}}\right] = 1.285 \frac{\text{mol N}}{\text{min}}$$

N of NO₂ N of NO₃

$$57.5 \frac{\text{ft}^3}{\text{min}} \left(\frac{273.2^\circ \text{C}}{273.2 + 58.6^\circ \text{C}}\right) \left(\frac{16 \text{ mol}}{359 \text{ ft}^3}\right) \left(\frac{453.6 \text{ mol}}{16 \text{ mol}}\right) \left(\frac{5000 \text{ g mol N}}{10^6 \text{ gas}}\right) = 0.299 \frac{\text{mol}}{\text{min}}$$

$$\text{NO}_x \text{ yield melter only} = \frac{0.299}{1.285} = 0.25 \quad (75\% \text{ reduction})$$

However, 20-30% may be evolved in spray dry operation
 using 25% evolved in spray dry, overall NO_x yield is

$$\underline{25-50\%} \quad \leftarrow$$

depending on behavior during spray dry

Take value as midpoint of range 25 $\longleftrightarrow$ 25+25

$$\text{or } \underline{37.5\%} \quad \leftarrow$$

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Title *Envitco NO_x yield*Project *LLW vitrification*Prepared by: *Greg Whyatt Greg Whyatt*Date: *9/29/95*

Reviewed by:

*MR Powell Mr Powell*Date *10/3/95*

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USB.M

M18PR-022P

-022 u

-026 u

-043 u

-050 u

wt% NO_2

2.81

1.77

0.51

1.87

2.21

wt% NO_2

0.72

0.36

0.04

0.42

0.35

Arg 1.834 0.378

$$\text{Source to melter} = \left(750 \frac{\text{lb feed}}{\text{h}} \right) \left(\frac{1}{60 \text{ min}} \right) \left(\frac{453.6 \text{ g}}{\text{lb}} \right) \left[.01834 \left(\frac{\text{mol}}{144.1} \right) + .00378 \left(\frac{\text{mol}}{144.32} \right) \right]$$

$$= 2.14 \frac{\text{mol}}{\text{min}}$$

From H1.3 gaseous $\text{NO}_x = 6.83 \times 10^{-4} \frac{\text{mol}}{\text{min}}$, Raw feed = $9.49 \frac{\text{mol}}{\text{min}}$

" Feed reaction destroys $(9.49 - 2.14) / 9.49 = 77.5\%$ of NO_2 / NO_x
 of gases released, 9.6 % is as NO_x

Residue in feed is destroyed with $\frac{2.14 - 6.83 \times 10^{-4}}{2.14} = 99.97\%$ efficiency

Process Overall yield = $(9.6\% \text{ as } \text{NO}_x) \times (.775) = 7.4\%$

EnvitcoSpray dry removes 20-30% of NO_2 , NO_x (assume = 25%)

$$\text{NO}_2 \text{ yield from melter} = \frac{.299 \frac{\text{mol}}{\text{min}}}{1.205 \frac{\text{mol}}{\text{min}} (.75)} = 33\%$$

NO_2 yield from spray dry is unknown so overall process yield
 is $\geq 33\% (.75) = 25\%$ or minimum 25%

If spray dry was all NO_x would be $25 + 25 = 50\%$

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Title

USB.M / Envitco ^{Separate} Melter / Feed Prep NO_x Yields

Project

LLW Vittrification

Prepared by:

Greg Whyatt Greg Whyatt

Date

3/19/96

Reviewed by:

Wm Powell

Date

3/27/96

CALCULATION WORKSHEET

Calc. No. Revision 0

H1.8

Rev.

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P3 1-76 of WHC-SD-WM-VI-031 Rev 0

Fluid Bed Calciner - Vectra Moss Balance Estimation

100% Strach Sugar
500 °C target

Off gas kg "N" as i		Feed kg "N" as i	
N ₂ O	.239	NO ₂	1.749
NO	.176	NO ₃	3.324
NO ₂	.008		
NH ₃	.020		
other, N ₂	balance		5.073 kg "N" feed

$$\begin{aligned} \% N_2O &= .239 / 5.073 = 4.71 \times 10^{-2} & 4.7\% \\ \% NO &= .176 / 5.073 = 3.47 \times 10^{-2} & 3.5\% \\ \% NO_2 &= .008 / 5.073 = 1.58 \times 10^{-3} & .16\% \\ \% NH_3 &= .020 / 5.073 = 3.94 \times 10^{-3} & .39\% \\ \% \text{ other } N_2 &= 100\% - 4.7 - 3.5 - .16 - .39 = 91.3\% \end{aligned}$$

Final Product contains negligible NO₂/NO₃

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Title Vectra Fluid Bed Calciner NO ₂ /NO ₃ product yields		Project LLW Vitrification	
Prepared by: Greg W. Lyart	Date 3/25/96	Reviewed by: W. Paul	Date 3/28/96

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CALCULATION WORKSHEET

Calc. No. H1.9Page 1 of 1Duratel pg 5-41 of Report NH_3 @ 1934 mg/m^2 in 99 dscfm, H1.5; $\text{N}_{\text{source}} = 3.64 \text{ mol/min}$

$$99 \frac{\text{ft}^3}{\text{min}} \left(\frac{26.317 \text{ l}}{\text{ft}^3} \right) \left(\frac{\text{m}^3}{1000 \text{ l}} \right) \left(\frac{1934 \text{ mg}}{\text{m}^3} \right) \left(\frac{1}{1000 \text{ mg}} \right) \left(\frac{\text{mol}}{14.3 \text{ g}} \right) = 0.3189 \text{ mol/min}$$

$$\text{ignore urea} - \text{NH}_3 \text{ yield based on } \text{NO}_2^-/\text{NO}_3^- = \frac{.3189}{3.64} = \underline{8.8\%}$$

USBM see H1.3 feed = 9.49 mol/min , Lub data

$$\text{NH}_3 \text{ yield on feed prep} = \frac{23.5}{(23 + 2(18.8) + 2(78.2) + 23.5)} = \underline{9.8\%}$$

From 2-4 of offgas report: $\dot{V}_2 = .0437 \frac{\text{ft}^3 \text{NH}_3}{\text{hr}}$ $\dot{V}_3 = .0394 \frac{\text{ft}^3}{\text{hr}}$, H1.7 $\text{N}_{\text{source}} = 2.14 \text{ mol/min}$

$$\left(\frac{.0437 + .0394 \frac{\text{ft}^3}{\text{hr}}}{2} \right) \left(\frac{453.6 \text{ g}}{\text{ft}^3} \right) \left(\frac{\text{mol}}{17 \text{ g}} \right) \left(\frac{1}{60 \text{ min}} \right) = 1.85 \times 10^{-2} \text{ mol/min NH}_3$$

$$\text{Melter NH}_3 \text{ yield} \approx \frac{1.85 \times 10^{-2}}{2.14} = \underline{0.87\%}$$

Enviro pg 6-7 emission rate = 0.059 kg/hr - potentially intended to be out of 4 samples shown below from appendix

pg 6-40 15/hr data

.136

.124

.117

.0715

$$\text{Avg} = .1121 \text{ 15/hr} \quad .0509 \text{ kg/hr NH}_3$$

From H1.6 source to melter = 1.205 mol/min

$$\text{Melter NH}_3 \text{ yield} = \frac{(.0509 \text{ kg/hr}) \left(\frac{\text{hr}}{60 \text{ min}} \right) \left(\frac{1000 \text{ g}}{\text{kg}} \right) \left(\frac{\text{mol}}{17 \text{ g}} \right)}{1.205} \times 100\% = \underline{4.14\%}$$

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Title <u>Ammonia Calculations</u>		Project	
Prepared by: <u>Greg Whyatt</u>	Date <u>3/20/96</u>	Reviewed by: <u>Mr. [Signature]</u>	Date <u>3/27/96</u>

Estimate volatility of Iodine based on neutron activation analysis performed by USGS

Data - all sample results as wt % I of once sample ($\pm 10^\circ$)

accuracy @ 1000 ppm = $\pm 2\%$
@ 100 ppm = $\pm 10\%$
40 ppm = detection limit

Bogach & Wilcox

	wt% I	Al ₂ O ₃	SiO ₂	CaO
BIF7 0024	.0904	8.12	43.8	3.85
00542	.1082	7.87	42.5	3.68
00843	.0941	7.92	42.4	3.79
0134	.1082	7.85	42.3	3.74
BIG9 00241	.0156	13.2	59.2	5.35
00542	.0105	12.0	63.2	5.20
0064	.0095	14.5	60.3	5.72
00843	.0025	15.1	64.1	5.57
01345	.0075	13.1	63.0	5.61

-neglect, no corresponding force
Sample to compare

WSTC

	wt% I	Al ₂ O ₃	CaO	Time
WIF12-40744	.0978 ($\frac{A_{90}}{I}$)	14.9	3.76	12/8 15:00
WIG67-42544	<.0025	19.1	4.06	2/8 15:00

Both decontest #4

USGM

	wt% I	SiO ₂	Time
MI APR-0014	.1093	47.1	2/10 8:30
APR-0024	.1206	49.7	2/16 10:00
BPR-0024	.1129	50.1	1/27 8:48
-0264	.1247	51.3	1/27 16:00
0434	.1015	50.3	1/31 11:20
0504	.1117	50.2	2/1 0:15
MIG1-0024	.0118	59.4	3/9 2:15
MIG1-0044	.0081	58.5	3/9 10:30
0054	.0061	58.8	3/9 13:05
0084	.0032	54.6	3/9 18:50
0104	.0076	55.7	3/9 21:15
0114	<.0014	57.2	3/9 22:15
0124	.0053	58.4	3/9 22:35

Correspondence between
feed samples and
melter feed time
not determined.
Use average feed
values.

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Title Selected neutron activation data for iodine		Project LLW Vitrification	
Prepared by: Greg Whyatt	Date 9/29/95	Reviewed by: MR Powell	Date 9/29/95

Battelle Pacific Northwest Laboratories		Revision 0 CALCULATION WORKSHEET
Page 2 of 14 Date: 11/14/14		

Only feed samples from fluid bed (Californ feed so will not trigger release S.O. the only use good sample from M.T. slurry feed, sampled per instruction)

Vector		I wt %	S.O.
VIM2	632 - 012u	.0055	58.9
VIM2	632 - 042u	.0118	58.7
VIM2	632 - 038	.0131	58.5
VIM2	632 - 025	.0118	58.5
Target Ratio in feed		.1055	57.2 (Theoretical)

Durable		I wt %	S.O.
DIF3	-11u	.0869	33.0
DIF3	-112u	.0878	31.8
DIF3	-114u	.0894	31.4
Avg2		}	42.2
D164	-110	.0202	42.3
D164	-115	.0233	42.5
Avg2		}	42.5
Avg2		}	42.5

Envelope		I wt %	S.O.
E1	E1 F2 - 002u	.0357	43.3
E1	E1 F3 - 006u	.0977	51.1
E1	E1 F2 - 057u	.0988	51.1
E1	E1 F2 - 015u	.1074	51.1
Avg2		}	58.6
E165	-009u	.1093	58.6
E165	-019u	.1180	59.7
E165	-028u	.1117	59.0
Avg2		}	11.1

Subject post-spray dry
 Post spray dry
 Subject due to possible losses.

B&W

Greg A Whyatt 9/28/95 6:47 pm IODINE VOLATILITY ESTIMATES													
BABCOCK AND WILCOX													
Feed Information					Glass Information				Al2O3	SiO2	CaO		
(samples B1F7-XXXX)					(samples B1G9-XXXX)				Tie Calc	Tie Calc	Tie Calc		
sample #	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	percent	percent	percent	Average	
B1F7-	I	Al2O3	SiO2	CaO	I	Al2O3	SiO2	CaO	volatile	volatile	volatile	Volatility	
002U1	0.0904	8.12	43.8	3.85	0.0156	13.2	59.2	5.35	89.38455	87.23242	87.58167	88.06622	
005u2	0.1082	7.87	42.5	3.68	0.0105	12	63.2	5.2	93.63563	93.4742	93.13238	93.41407	
008u3	0.0941	7.92	42.4	3.79	0.0025	15.1	64.1	5.57	98.60653	98.24265	98.19227	98.34715	
013U	0.1082	7.85	42.3	3.74	0.0075	13.1	63	5.61	95.84633	95.34592	95.37893	95.52372	
											Overall Average	93.83779	

WESTINGHOUSE SCIENCE AND TECHNOLOGY CENTER IODINE VOLATILITY CALCULATION									
	wt%	wt%	wt%	Al ₂ O ₃ tie	CaO tie	Avg%loss			
feed W1F12-	I	Al ₂ O ₃	CaO	%loss	%loss				
407u4	0.0978	14.9	3.76	98.00587	97.63265	97.81926			
glass W1G07									
425u4	0.0025	19.1	4.06						
note: iodine value is below detection limit in glass									
therefore result is greater than the average% loss shown									

USBM IODINE VOLATILITY CALCULATION				
	wt%	Wt%	Ratio	
feed sample data	I	SiO2	I:SiO2	
M1 APR-001U	0.1093	47.1	0.0023206	
M1 APR-002U	0.1206	49.7	0.0024266	
M1 BPR-022U	0.1139	50.1	0.0022735	
M1 BPR-026U	0.1247	51.3	0.0024308	
M1 BPR-043U	0.1095	50.3	0.0021769	
M1 BPR-050U	0.1117	50.2	0.0022251	
		avg ratio	0.0023089	
	wt%	Wt%		tie Calc
Glass sample data	I	SiO2		Percent loss
M1 G1-002U	0.0118	59.4		91.39622
M1 G1-004U	0.0081	58.5		94.00316
M1 G1-005U	0.0061	58.8		95.5069
M1 G1-008U	0.0032	54.6		97.46165
M1 G1-010U	0.0076	55.7		94.09048
M1 G1-011U	0.0014	57.2	greater than	98.93995
M1 G1-012U	0.0053	58.4		96.06942
avg (info only)	0.006214	57.51429	avg loss	95.35254
result on 011U below detection limit				

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H2

VECTRA IODINE VOLATILITY CALCULATION							
Glass Samples	I wt%	SiO2 wt%	SiO2 tie % loss	SrO glass wt%	SrO tie % loss		
V1M2 G32-012u	0.0085	58.9	93.23543	0.051	86.38571		
V1M2 G32-042u	0.0118	58.7	90.57719	0.059	83.66285		
V1M2 G32-038u	0.0131	58.5	89.50332	0.055	80.54394		
V1M2 G32-025u	0.0118	58.5	90.54498	0.055	82.4747		
		average	90.96523		83.2668		
Feed data was not available for slurry feed							
therefore based on target recipe pg 3-76, 78							
	I	SiO2	SrO				
Target Feed	0.1171	54.89	0.095654				
Note: In this case, SrO tie result is preferred due to possible non-steady state concentrations of SrO and I because these components were not in the startup glass.							

DURATEK IODINE VOLATILITY CALCULATION					
			Ratio		
feed	I wt%	SiO2 wt%	I:SiO2		
D1 F3-111U	0.0869	32	0.002716		
D1 F3-112U	0.0878	31.8	0.002761		
D1 F3-114U	0.0894	31.4	0.002847	SiO2 Tie Calc	
				Percent Loss=	81.5148
		avg	0.002775		
D1 G4-110	0.0202	42.3	0.000478		
D1 G4-115	0.0233	42.5	0.000548		
		avg	0.000513		

ENVITCO IODINE VOLATILITY							
feed sample	I wt%	SiO2 wt%	Al2O3 wt%	I:SiO2 ratio	I:Al2O3 ratio		
E1 F2-002U	0.0957	43.3	8.6	0.00221	0.0111279		
Glass Samples	I wt%	SiO2 wt%	Al2O3 wt%	I:SiO2 ratio	I:Al2O3 ratio	SiO2 Tie % loss	Al2O3 Tie % loss
E1 G5-009u	0.1093	58.6	11.1	0.001865	0.0098468	15.60854	11.51214
E1 G5-019u	0.118	59.7	10.7	0.001977	0.011028	10.56992	0.89747
E1 G5-028u	0.1117	59	10.9	0.001893	0.0102477	14.34019	7.909848
					avg	13.50621	6.773152
				overall average % loss		10.13968	

H-21

U.S. GEOLOGICAL SURVEY
BRANCH OF GEOCHEMISTRY
BOX 25046, DFC, M/S 973
Denver, Colorado 80225-0046

DATE: 5/9/95

TO: ERIC MAST

FAX#: (509) 372-2810

FROM: PAUL LAMOTHE

BGC FAX#: (303)236-3200

COMMENTS:

HERE IS ALL THE IODINE DATA

AVAILABLE TO DATE. IT WAS DETERMINED USING

NEUTRON ACTIVATION ANALYSIS. PLEASE IGNORE THE AL, Ca, & Na

NUMBERS - THEY ARE ONLY APPROXIMATE VALUES.

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Paul

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WHC-SD-WM-ER-517
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JOB NO: WD89

SUBMITTER: LAMOTHE, PAUL

		I (%)	Al (%)	Ca (%)	Na (%)	
D-574345	D1G4-022U2	0.0151	3.60	6.69	13.1	GLASS - DURATEK
D-574347	D1G4-023U3	0.0203	3.54	6.68	12.6	
D-574349	D107-000U	<0.0040	2.60	0.39	3.4	DIATOM. EARTH
D-574351	D107-002U	0.0064	2.35	0.40	4.0	BAGHOUSE

JOB NO: WE35

SUBMITTER: LAMOTHE, P. SHADE, J.

		I (%)	Al (%)	Ca (%)	Na (%)	
D-575342	B1G9-002U1	0.0156	6.99	4.29	11.1	GLASS - BABCOCK
D-575344	B1G9-005U2	0.0105	6.88	3.14	10.0	
D-575346	B1G9-006U	0.0095	8.00	3.40	10.0	
D-575348	B1G9-008U3	0.0025	8.38	3.01	8.0	
D-575350	B1G9-013U5	0.0075	7.15	3.87	9.9	

JOB NO: WFO1

SUBMITTER: LAMOTHE, PAUL

		I (%)	Al (%)	Ca (%)	Na (%)	
D-576639	E1G5-009U	0.1093	6.00	0.19	13.0	GLASS - ENVICTO
D-576641	E1G5-019U	0.1180	5.95	0.25	12.6	
D-576643	E1G5-028U	0.1117	5.98	0.24	12.7	

JOB NO: WF42

SUBMITTER: WILSON, STEVE LAMOTHE, PAUL

		I (%)	Al (%)	Ca (%)	Na (%)	
D-577402	B1F7002U	0.0904	4.47	2.73	9.7	FEED - BABCOCK
D-577403	B1F7005U2	0.1082	4.33	2.71	10.3	
D-577404	B1F7008U3	0.0941	4.24	3.00	9.8	
D-577405	B1F7013U	0.1082	4.40	2.89	10.2	
D-577406	E1F2002U	0.0957	4.54	<0.06	9.7	FEED - ENVICTO
D-577407	E1F3006U	0.0977	4.21	<0.04	8.3	

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JOB NO: WF65

SUBMITTER: LAMOTHE, PAUL

		I(%)	Al(%)	Ca(%)	Na(%)	
D-577817	W1G07-425U4	<0.0025	10.4	3.35	15.4	GLASS - WSTC
D-577819	W1A13-406U4	<0.0030	9.69	4.10	0.21	FRIT "A" - WSTC

JOB NO: WH58

SUBMITTER: WILSON, STEVE

		I(%)	Al(%)	Ca(%)	Na(%)	
D-583266	M1APR-001U	0.1093	4.97	3.51	12.4	FEED - BOM (USBR)
D-583268	M1APR-002U	0.1206	5.19	3.16	13.9	
D-583270	M1BPR-022U	0.1139	4.99	3.42	12.6	
D-583272	M1BPR-026U	0.1247	5.21	3.24	13.2	
D-583274	M1BPR-043U	0.1095	5.06	3.42	11.5	
D-583276	M1BPR-050U	0.1117	4.99	3.40	11.8	
D-583278	M1C4-001U	0.7201	0.35	1.10	22.9	CYCLONE - USBM
D-583280	M1B5-001U	1.0350	0.18	0.26	23.2	BAGHOUSE - USBM

JOB NO: WH81

SUBMITTER: SHADE, J. WILSON, STEVE

		I(%)	Al(%)	Ca(%)	Na(%)	
D-583636	V1FP1I21-015	0.0797	4.81	2.30	10.4	FEED - VECTRA
D-583638	V1FP1I21-016	0.0866	5.04	1.98	10.6	

JOB NO: WH99

SUBMITTER: LAMOTHE, PAUL SHADE J.

		I(%)	Al(%)	Ca(%)	Na(%)	
D-583957	V1M2/G32012U	0.0085	5.08	2.17	11.3	GLASS - VECTRA
D-583959	V1M2/G34-016	0.0115	5.39	2.28	11.4	
D-583961	V1M2/G32042U	0.0118	5.37	2.25	11.7	
D-583963	V1M3/G34-072	0.0197	5.31	2.24	11.7	
D-583965	V1M3/G32076U	0.0202	5.35	2.02	12.3	
D-583967	V1M4/G32090U	0.0189	5.32	2.06	12.4	
D-583969	V1M4/G32098U	0.0255	5.32	2.09	12.2	
D-583971	V1M3/G32060U	0.0176	5.54	1.95	12.3	

JOB NO: WJ01

SUBMITTER: LAMOTHE PAUL SHADE, J

		I(%)	Al(%)	Ca(%)	Na(%)	
D-583975	V1M2/G32-038	0.0131	5.05	2.24	11.3	GLASS - VECTRA
D-583977	V1M2/G32-025	0.0118	5.45	2.28	12.2	

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JOB NO: WG05

SUBMITTER: WILSON, S. AND LAMOTHE, P.

		I (%)	Al (%)	Ca (%)	Na (%)
D-578562	W1F12-407U4	0.0960	8.30	3.05	9.74
D-578563	W1F12-407U4	0.0996	8.21	2.52	9.39

FEED - WSTC

JOB NO: WG10

SUBMITTER: LAMOTHE, PAUL

		I (%)	Al (%)	Ca (%)	Na (%)
D-578605	D1G4-110U7	0.0193	3.55	5.59	12.15
D-578606	D1G4-110U7	0.0211	3.57	5.55	12.07
D-578607	D1G4-115U9	0.0224	3.63	5.10	12.17
D-578608	D1G4-115U9	0.0241	3.52	5.55	12.06
D-578609	ARG-1	<0.0032	2.69	0.60	8.08
	LITERATURE	NA	2.47	1.01	8.31
	ICP (WF01)	----	2.4	1.1	----

GLASS - DURATEK

GLASS - DURATEK

JOB NO: WG13

SUBMITTER: WILSON, S. AND SHADE

		I (%)	Al (%)	Ca (%)	Na (%)
D-578651	D1F3-111UA	0.0895	2.65	3.93	9.19
D-578652	D1F3-111UB	0.0843	2.61	4.06	9.20
D-578653	D1F3-112UA	0.0876	2.59	4.38	9.25
D-578654	D1F3-112UB	0.0880	2.61	3.63	8.94
D-578655	D1F3-114UA	0.0895	2.60	3.48	9.13
D-578656	D1F3-114UB	0.0893	2.60	3.73	8.95

FEED - DUPLOK

FEED "

FEED "

JOB NO: WG49

SUBMITTER: LAMOTHE, PAUL

		I (%)	Al (%)	Ca (%)	Na (%)
D-583113	M1G1-002U	0.0118	6.04	3.95	12.1
D-583115	M1G1-004U	0.0081	6.45	4.22	12.1
D-583117	M1G1-005U	0.0061	6.61	4.56	11.8
D-583119	M1G1-008U	0.0032	8.18	7.69	10.3
D-583121	M1G1-010U	0.0076	7.13	5.28	11.4
D-583123	M1G1-011U	<0.0014	6.61	5.49	11.8
D-583125	M1G1-012U	0.0053	6.44	4.45	12.3
D-583127	MIT3-001U	<0.0018	22.2	5.88	17.1

GLASS - BOM (USGM)

GLASS - BOM

GLASS - BOM

GLASS - BOM

GLASS - BOM

GLASS - BOM

GLASS - BOM

GLASS - BOM

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JOB NO: WC55

SUBMITTER: LAMOTHE, PAUL

		I(%)	Al(%)	Ca(%)	Na(%)	
D-583220	E1F3-057U	0.0988	4.87	<0.09	10.3	FEED - ENVICTO
D-583222	E1F3-015U	0.1074	6.08	<0.09	13.3	FEED - ENVICTO

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B&W	I (%)
Feed	0.1002
Glass	0.0091
Duratek 1000	I (%)
Feed	0.0880
Glass	0.0217
Envitco	I (%)
Feed	0.0999
Glass	0.1130
USBM	I (%)
Feed	0.1149
Glass	0.0057
Cyclone	0.7201
Baghouse	1.0350
Vectra	I (%)
Feed	0.0831
Glass	0.0159
WSTC	I (%)
Feed/Frit	-0.0030
Glass	-0.0025

GA Whyatt 3/18/96									
TOTAL LOSS ESTIMATES									
Oxide	B&W	WSTC	USBM-1	USBM-2	USBM-3	Vectra	Duratek	Envitco	
B2O3	69.9	24.1	51.6	23.9	19.0	14.5	0.6	0.19	
Cl	88.1	88.3	82.2	NA	97.0	64.2	48.3	1.05	13.0
Cs2O	84.5	84.4	63.4	NA	39.7	41.4	13.5	0.65	
F	92.7	91.2	91.1	NA	99.7	15.5	53.3	0.90	
I	94.5	98.1	95.1	NA	NA	83.1	82.1	10.05	
K2O	55.3	49.4	35.8	NA	25.9	15.5	0.6	0.05	
LiO2	NA	5.4	NA	NA	NA	NA	NA	NA	
MoO3	63.5	26.1	47.6	50.6	45.7	NA	0.8	0.05	
Na2O	40.7	17.3	21.9	21.0	7.6	13.5	0.6	0.05	
P	46.1	44.5	41.7	NA	54.6	1.7	NA	0.05	
S	55.3	35.8	88.1	NA	94.1	85.1	7.6	53.02	
GROSS ENTRAINMENT ESTIMATES (ALL INPUT DATA)									
	B&W	WSTC	USBM-1	USBM-2	USBM-3	Vectra	Duratek	Envitco	
	8.7	2.7	1.2	1.2	1.2	0.6	0.6	0.05	
for purposes of calculation, the entrainment estimate from USBM-1 was used for USBM-2 and USBM-3 where no entrainment estimate was possible.									
VOLATILITY RESULTS FOR MELTER VENDORS (ALL INPUT DATA)									
Oxide	B&W	WSTC	USBM-1	USBM-2	USBM-3	Vectra	Duratek	Envitco	Envitco
B2O3	67	22	51	23	18	14	0	0.14	
Cl	87	88	82	NA	97	64	48	1	13
Cs2O	83	84	63	NA	39	41	13	0.6	
F	92	91	91	NA	99.7	15	53	0.85	
I	94	98	95	NA	NA	83	82	10	
K2O	51	48	35	SE	25	15	0	0	
LiO2	NA	2.8	NA	NA	NA	NA	NA	NA	
MoO3	60	24	47	50	45	SE	0.2	0	
Na2O	35	15	21	20	6.5	13	0	0	
P	41	43	41	SE	54	1.1	SE	0	
S	51	34	88	SE	94	85	7	53	
WSTC I is >98									
Envitco chloride result of 1 to 13 entered as two cells									
Envitco fluoride result of <0.85 entered as 0.85									
USBM-2 values for K2O -143, P -42, S -303 listed as SE for Source Error									
Duratek P result of -27 listed as SE for Source Error									
Envitco Values for MoO3 -0.01, Na2O -0.01, P -0.1 were set to zero volatility									
Vectra MoO3 -49% entered as source error									

Revised by
M. R. R. 3/28/96

APPENDIX I

REVIEW OF FLUORIDE ANALYTICAL DISCEPANCIES

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**LETTER REPORT: FLUORIDE ANALYTICAL DISCREPANCIES FOR
PHASE-1 LOW-LEVEL WASTE VENDOR TESTING**

C.J. Freeman

June 1995

Prepared for
The U.S. Department of Energy
Under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Richland, Washington 99352

INTRODUCTION

Analyses of Hanford Low-Level Waste (LLW) feed and glass constituents from the various vitrification testing vendors were provided from the U.S. Geological Survey (USGS), Corning Incorporated (CELS Laboratory Services), Quanterra, and Pacific Northwest Laboratory (PNL). The feed materials were composed of a standard high sodium LLW simulant (prepared by Optima Chemicals) mixed with glass forming additives which were specified by the vendor. After the first few samples were analyzed it was apparent that the measured fluoride in the feeds differed from the targets for those simulants. As a result, Westinghouse Hanford Company (WHC) requested that PNL investigate this data to ascertain its validity for subsequent mass balance calculations. Possible explanations for the observed fluoride discrepancies in the waste simulant and feed materials were that the fluoride analytical errors were larger than expected, the fluoride was not fully homogenized in the samples (since a significant fraction of the fluoride is present as solid sodium fluoride in the waste simulant), or the fluoride was not added accurately to the simulant to start with.

FLUORIDE ANALYTICAL TECHNIQUES

From an analytical standpoint fluoride analyses in solid materials can be extremely difficult to obtain. Soluble fluoride can be analyzed quite well with Ion Chromatography (IC) or Specific Ion Electrode (SIE) techniques. The difficulty with measuring fluoride in solid samples arises when the sample is "fused" (for example, a peroxide/acid fusion) and the fluoride is driven off during heating the sample. Additionally, solid samples with an appreciable degree of silica content can produce silica-fluoride complexes (where silica is present in each of the vendor's glass forming additives) after fusing which can not be quantified with IC or SIE instruments. As a result, fluoride values from fused samples are typically not reported by PNL, only "soluble fluoride" values from IC or SIE are reported as accurate. LLW simulant samples from the original batch make-up tanks, prepared by Optima Chemical, were only analyzed by PNL and Quanterra. Like PNL, Quanterra's fluoride analysis capacity is limited by the techniques/sample preparations just described. As a result, both PNL and Quanterra analytical reports showed 57 to 94% lower fluoride than the target values for each of the waste simulant batches produced by Optima.

Both USGS and CELS reported having much more developed techniques for fluoride analysis of solid and slurry samples. The following fluoride analysis description was provided by Paul J. Lamothe of USGS. This procedure was used for all of the LLW samples analyzed by USGS.

Additionally, it is a routine method they have used for the past 15 years for the determination of fluoride in aluminosilicate rocks.

If the material is in a slurry form, 10 grams of the slurry are first added to a 50 mL glass beaker, dried for 24 hours at 110°C, followed by an additional 24 hour drying period at 150°C. The dry residue is then weighed, collected, homogenized, and analyzed for fluoride content using the following method: 200-mg of sample is mixed with 1.2 grams of lithium metaborate (LiBO_2) in a graphite crucible followed by fusing the mixture in a muffle furnace at 900°C for 10 minutes. The molten bead is then poured from the crucible into a polypropylene beaker containing 100 mL of 4% nitric acid. After complete dissolution 100 mL of a buffer solution is added. (The buffer is a solution of diaminocyclohexanetetracetic acid (DCTA) in 40% NaOH, sodium citrate, and sodium chloride, adjusted to a pH of 6.85 with HCl). The fluoride concentration is then measured by fluoride-selective electrode potentiometry. The results are then reported for the sample as received, i.e. corrected for the weight loss that occurred by drying, etc. Two quality control check samples are analyzed along with each batch of unknowns. These check samples are comprised of solid aluminosilicate standard reference materials of known fluoride composition.

Note that this technique is equivalent to a standard glass fusion which, as previously mentioned, is prone to fluoride volatilization during fusing as well as fluoride-silica complexing. These problems presently drive PNL glass analysts to report such fluoride analyses as "indication only," if at all.

CELS reported using two different fluoride analyses for solid and liquid/slurry samples. For solid analysis (with expected levels $>0.01\text{wt}\%$) a pyrohydrolysis technique is used where the material is ground, mixed with U_3O_8 , and heated to 1130°C in the presence of steam. The fluoride is then distilled as HF, scrubbed and titrated with thorium nitrate or directly analyzed by an ion-selective electrode method. The ion-selective electrode measurements were used for nearly all of the LLW glass samples since the resulting fluoride concentrations were too low for the titration approach. For liquid and dilute slurry samples CELS directly measured fluoride with IC. As a result, these analyses only provide soluble fluoride data.

WASTE SIMULANT AND MELTER FEED DISCREPANCIES

Before looking into the various analytical techniques for sources of error it was first decided to inspect the fluoride data from the various melter feeds (Optima waste simulant with glass forming additives) generated by USGS and CELS to see if some of the discrepancies could be attributed to a solid phase separation. If sampling were performed such that the solids were not completely homogeneous, the error in the fluoride data may be directly attributable to this effect.

In order to accurately assess solids inhomogeneity, other known insoluble waste constituents were compared with fluoride. Chromium was chosen as the comparison species because it was present in the original waste simulant and as a precipitation product during simulant make-up. Similar make-up schemes would have resulted in smaller particle sizes compared to many of the bulk glass formers used, such as silica. In order to calculate comparison values of fluoride and chromium, another waste-specific reference ion was selected which was also fairly soluble in the simulant. Using a soluble reference ion enables easy calculations to be made of the fluoride and chromium content without having to rely on the accuracies of each vendor feed target. The reference ion chosen was chloride since chloride analyses are typically quite good and the data from all the waste and feed simulants showed reasonable (close to target) values for this ion.

The calculation made for fluoride, chromium was "percent unaccounted for ion." This calculation was made with the following equation where X refers to either fluoride or chromium:

$$\% \text{ Unaccounted for X} = 100 \left(1 - \frac{\frac{X_{\text{measured}}}{Cl_{\text{measured}}}}{\frac{X_{\text{target}}}{Cl_{\text{target}}}} \right)$$

Table 1. Unaccounted for fluoride and unaccounted for chromium from various analyzed LLW melter feeds

Material:	B&W Feed	B&W Feed	Envitco Feed	WSTC Feed	Duratek 1000 Feed	Duratek 1000 Feed	SSHTM Feed
Analytical Firm:	Corning	USGS	Corning	Corning	Corning	USGS	PNL
Unaccounted for ...							
Fluoride	79%	86%	44%	83%	54%	68%	84%
Standard Dev	15%	25%	15%	2%	11%	17%	38%
Chromium	52%	33%	58%	19%	-12%	1%	-11%
Standard Dev	7.7%	1.6%	17.1%	0.9%	1.0%	0.1%	3.4%

The previous equation assumes that the chloride was added on target and completely accounted for upon analysis. The chloride values for the various feeds analyzed appeared close to their target levels. The following table and plot show the results of applying the previous equation to the Babcox & Wilcox (B&W), Envitco, Westinghouse Science and Technology Center (WSTC), Duratek, and SSHTM (PNL) LLW melter feeds. Each of these feeds used the same waste simulant compositions. Note that the standard deviation values were calculated using data from repeat analyses.

Figure 1. Unaccounted for fluoride versus unaccounted for chromium from various analyzed LLW melter feeds

Figure 1 shows the percent of unaccounted for fluoride plotted against unaccounted for chromium. If the variations in all of these species were

simply due to inhomogeneity of solid constituents, these data would have varied around zero along the 45° line shown. Instead, only the average chromium data varies around zero, indicating inhomogeneity in some of the samples, and the fluoride values are highly skewed (even within the error ranges shown) to a range of 44 to 86% unaccounted for fluoride. Note that the Envitco feed was the only sample in this data set which was calcined. The higher degree of chromium than target from the analyses may indicate that this particular sample was slightly richer in waste simulant solids.

Since the previous calculations indicate that the fluoride discrepancies are not largely due to simple sample inhomogeneity the next possibility examined was whether or not the fluoride was accurately added to the waste simulants, at Optima, in the first place. Although strict quality assurance was maintained during these simulant make-ups the possibility of a mis-marked container, etc. was hypothesized by several WHC staff. Since the only fluoride analysis performed on the waste simulants were made by PNL and Quanterra, who only analyzed for soluble fluoride, the accuracy of these original analytical checks was poor. Therefore, PNL made a few "control" samples of simulant to cross-check the accuracies of the CELS and USGS fluoride data. These control samples consisted of carefully made individual 250 ml batches of LLW simulant. These samples were then sent to both CELS and USGS for analysis. The results from those analyses are shown in Table 2.

The USGS results, shown in Table 2, accounted for only 17.7% of the fluoride in the sample. This discrepancy is mostly likely due to the high temperature fusion step used in sample preparation as well as silica-fluoride complexing in such fusions. The CELS results report two sets of numbers. The first set was generated by a simple IC analysis of the sample supernate. As a result these numbers should reflect only the soluble halides in the sample. Indeed, fluoride values from the IC analyses account for only 41% of that ion. Finally, the last CELS data in Table 2 show the results from their pyrohydrolysis procedure. The fluoride value reported from this procedure accounts for more than the first two, however, it is still low (only 59% accounted for).

Table 2. Fluoride and Chloride Analytical Results for LLW Simulant Control Samples (G1S1-001U & G1S1-001C)

Halide	Target	USGS (Fusion/IC)		CELS (Ion Chrom., solubles)		CELS (Pyrohydrolysis)	
	Mass Content of Halide	Mass Content of Halide	Amount of Halide Missing	Mass Content of Halide	Amount of Halide Missing	Mass Content of Halide	Amount of Halide Missing
Chloride	0.36%	0.27%	25.8%	0.41%	-11.3%	0.33%	9.3%
Fluoride	0.31%	0.05%	82.3%	0.13%	59.0%	0.18%	41.0%

The ranges of unaccounted for fluoride reported in Table 2 are within the ranges observed for the LLW vendor feeds (see Table 1). Since these same ranges of analytical discrepancy were observed for the control samples as were for each of the vendor feed analyses it is apparent that the fluoride was indeed added near target in the original Optima batches. Furthermore, the target feed values for fluoride are expected to be more accurate for subsequent mass balance calculations than the measured values.

FLUORIDE ANALYSES OF GLASS SAMPLES

In addition to investigating waste simulant and melter feed fluoride discrepancies WHC also requested looking at the glass analyses to ascertain the validity of this data. The method chosen to present this data was to use an approach similar to the one shown in the previous section, where a reference ion is used to calculate the unaccounted for fluoride based on its target value (i.e. feed composition targets used, not analyzed values). Two reference ions were chosen for this calculation, manganese and molybdenum. However, manganese was expected to provide a better comparison since molybdenum is known to volatilize from high temperature glasses. Nevertheless, sets of calculations for both of these ions are shown in the following table. Note that the standard deviations shown in this table were not generated from duplicate sample analyses but from all of the glass samples analyzed for each test. As a result, this standard deviation is representative of total sample variation and is not equal to the analytical error. However, these standard deviations do encompass the analytical error.

Table 3. Phase-1 LLW Vendor Testing Glass Fluoride Analyses

Glass Sample:	B&W	B&W	WSTC	WSTC	Duratek	Duratek	Envitco	Envitco	USBM	USBM
Analytical Firm:	Corning	USGS	Corning	USGS	Corning	USGS	Corning	USGS	Corning	USGS
F/MnO2:										
Target (waste)	205.5	205.5	205.5	205.5	205.5	205.5	205.5	205.5	205.5	205.5
Average (glass)	<0.27	0.73	2.62	0.95	1.10	0.74	55.86	>23.81	2.90	2.47
Standard Dev	0.04	0.40	1.79		0.25	0.29	11.02	2.90	0.64	0.45
Unaccounted for F	>99.9%	99.6%	98.7%	99.5%	99.5%	99.6%	72.8%	<88.4%	98.6%	98.8%
Standard Dev	0.0%	0.2%	0.9%		0.1%	0.1%	5.4%	1.4%	0.3%	0.2%
F/MoO2:										
Target (waste)	205.5	205.5	205.5	205.5	205.5	205.5	205.5	205.5	205.5	205.5
Average (glass)	<0.09	0.25	0.20	0.21	1.01	0.86	2.13	2.05	0.33	0.38
Standard Dev	0.05	0.11	0.06		0.22	0.33	0.65	0.29	0.04	0.06
Unaccounted for F	>100%	99.9%	99.9%	99.9%	99.5%	99.6%	99.0%	99.0%	99.8%	99.8%
Standard Dev	0.0%	0.1%	0.0%		0.1%	0.2%	0.3%	0.1%	0.0%	0.0%

Table 3 shows that the unaccounted for fluoride calculated from both the manganese and molybdenum references are between 98 and 100% in all cases but the Envitco glasses calculated with a manganese reference ion. Since the molybdenum reference values were suspect due to potential molybdenum volatilization, the manganese referenced values are expected to be the better of the two data sets. The Envitco glass analyzed by USGS shows a less than sign in front of its unaccounted for fluoride value. This was a result of analytical values being less than their detection limits. Since the Corning analyses of the Envitco glass did not run into detection limit problems, it is expected that this is the more accurate of the two values.

Unfortunately, the glass analytical values can not be used to ascertain the analytical errors of the measurement techniques. The few glass fluoride standards available for doing such a calibration have fluoride contents in the range of a few weight percent, far from the expected LLW glasses contents of less than 0.1 weight percent. As a result the analytical errors quantified from the control waste simulant analyses for fluoride are the best known values to apply to the glass analyses for this range of fluoride content.

CONCLUSIONS

Based on the glass analytical techniques described by both Corning and USGS and the discrepancies found for fluoride in the control samples it is believed that substantial errors could also lie in the glass fluoride analyses provided by each of these firms. It would be recommended that, in addition to using feed target values for fluoride in the mass balance calculations,

fluoride measured in the off-gas samples also be used for those calculations instead of the glass values. This is accomplished by assuming that the portion of the constituent which goes to the glass is equal to feed target amount minus that observed in the off-gas. However, many Phase-1 vendor tests used off-gas sample filters comprised of silica in the form of quartz or glass. As a result, those samples may also be prone to fluoride analytical problems. Still, this data or the data collected from scrubber and quencher samples (in addition to feed target values for fluoride) may have the least amount of error for melter mass balance calculations.

Further quantification of USGS and CELS fluoride analytical error may show the reported values to be reasonable for mass balance calculations, however, neither of those firms could explain the analytical discrepancies for the waste simulant control samples. Consequently, it is recommended that PNL's pyrohydrolysis analytical equipment be used to substantiate this data. Previous pyrohydrolysis data, reported by PNL in 1989 (Goles, 1989), showed good comparability (less than a 20% difference for fluoride) with the known concentrations. Although the differences between this equipment and the set-up used by CELS are small they may be important enough to explain the CELS discrepancies.

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