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EVALUATION OF GLASS DENSITY TO SUPPORT THE ESTIMATION OF FISSILE MASS LOADINGS FROM IRON CONCENTRATIONS IN SB8 GLASSES

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LIST OF ACRONYMS

ARP	Actinide Removal Process
ASTM	American Society for Testing and Materials
CUA	The Catholic University of America
DOE-SR	Department of Energy – Savannah River
DWPF	Defense Waste Processing Facility
EVs	Extreme Vertices
HLW	High Level Waste
NIST	National Institute of Standards and Technology
RMSE	Root Mean Square Error
SB5	Sludge Batch 5
SB6	Sludge Batch 6
SB7a	Sludge Batch 7a
SB7b	Sludge Batch 7b
SB8	Sludge Batch 8
SME	Slurry Mix Evaporator
SO	Sludge Only
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
UTL	Upper Tolerance Limit
VS	Variability Study
VSL	Vitreous State Laboratory
WAPS	Waste Acceptance Product Specifications
WL	Waste Loading

EXECUTIVE SUMMARY

The Department of Energy – Savannah River (DOE-SR) has provided direction to Savannah River Remediation (SRR) to maintain fissile concentration in glass below 897 g/m^3. In support of that guidance, the Savannah River National Laboratory (SRNL) provided a technical basis and a supporting Microsoft® Excel® spreadsheet for the evaluation of fissile loading in Sludge Batch 5 (SB5), Sludge Batch 6 (SB6), Sludge Batch 7a (SB7a), and Sludge Batch 7b (SB7b) glass based on the iron (Fe) concentration in glass as determined by the measurements from the Slurry Mix Evaporator (SME) acceptability analysis. SRR has since requested that the necessary density information be provided to allow SRR to update the Excel® spreadsheet so that it may be used to maintain fissile concentration in glass below 897 g/m^3 during the processing of Sludge Batch 8 (SB8).

One of the primary inputs into the fissile loading spreadsheet includes an upper bound for the density of SB8-based glasses. Based on the measured density data of select SB8 variability study glasses, it is recommended that SRR utilize the 99%/99% Upper Tolerance Limit (UTL) density values given below as upper bounds for the densities of the SB8 glass system at the waste loadings (WLs) as indicated. Thus, these bounding density values are to be used to assess the fissile concentration in this glass system. It should be noted that no changes are needed to the underlying structure of the Excel®-based spreadsheet to support fissile assessments for SB8. However, SRR should update the other key inputs to the spreadsheet that are based on fissile and Fe concentrations reported from the SB8 Waste Acceptance Product Specification (WAPS) sample.

WL	99%/99% UTL for Glass Density (g/cm^3)
32	2.779
34	2.777
36	2.786
38	2.810
40	2.843

1.0 INTRODUCTION

The Department of Energy – Savannah River (DOE-SR) previously provided direction to Savannah River Remediation (SRR) to maintain fissile concentrations below 897 g/m^3 in the high level waste (HLW) glass produced by the Defense Waste Processing Facility (DWPF).[1] To support DWPF in meeting that directive starting with the processing of Sludge Batch 5 (SB5), the Savannah River National Laboratory (SRNL) developed a technical basis and an associated Microsoft® Excel® spreadsheet that facilitates the evaluation of fissile loading of a glass product based on the iron (Fe) concentration in the glass as determined by the measurements from the Slurry Mix Evaporator (SME) acceptability analysis.[2] SRNL provided the necessary information to allow SRR to update the Excel® spreadsheet so that it may be used to maintain fissile concentration in glass below 897 g/m^3 during the processing of Sludge Batch 6 (SB6)[3], Sludge Batch 7a (SB7a)[4], and Sludge Batch 7b (SB7b)[5]. SRR has since requested that information on glass densities be provided to support fissile assessments for the glass system associated with the processing of Sludge Batch 8 (SB8).[6]

Insight into the density of SB8 glasses is to be based on the glasses resulting from the SB8 variability study (VS)[7-9]. Twenty two glasses were selected for the SB8 VS. Twelve of the SB8 VS glasses were determined by combining an optimally selected set of 12 Extreme Vertices (EVs) from the sludge composition region of interest (with each of these sludge compositions being combined with Frit 803 at 36% waste loading (WL)). Five glasses were defined by combining the nominal sludge only (SO) composition with Frit 803 at WLs of 32, 34, 36, 38, and 40% WL. The remaining five SB8 VS glasses were based on the combination of the nominal coupled operations composition, reflecting an Actinide Removal Process (ARP) stream of 1050 gallons, with Frit 803 at WLs of 32, 34, 36, 38, and 40% WL. These glasses were fabricated and densities measured by the Vitreous State Laboratory (VSL) of The Catholic University of America (CUA) in accordance with the test plan supporting this work.[10]

The purpose of this technical report is to present the density measurements that were conducted for the SB8 VS glasses and to perform a statistical evaluation of these measurements to provide a bounding density value that may be used as input to the Excel® spreadsheet to be employed by SRR to maintain the fissile concentration in its SB8 glass below 897 g/m^3. It should be noted that no changes are needed to the underlying structure of the Excel®-based spreadsheet to support fissile assessments for SB8. JMP Version 9.0.0 was used to support this analysis.[11]

2.0 EXPERIMENTAL: SB8 GLASS DENSITY DETERMINATION

All of the density measurements for this study were performed using the VSL procedure "Test Method for Density of Glass by Buoyancy at Room Temperature,"[12] which is based on the American Society for Testing and Materials (ASTM) method C693-93 "Standard Test Method for Density of Glass by Buoyancy." Duplicate density measurements were performed on each glass. Table 1 provides, in the order recorded, the individual and average density measurements for each of the SB8 VS glasses. Also included in Table 1 are the density measurements of the SRM1827a and SRM1826b glasses, which are National Institute of Standards and Technology (NIST) traceable standard reference glasses.[13,14] The SRM1827a density results indicate no significant issues with the measurement technique. More specifically, the reported density of the SRM1827a glass (from NIST) is 3.593014 ± 0.000025 g/cm^3. The average measured density in this testing for this standard glass was 3.59267 g/cm^3, a difference of ~0.0003 g/cm^3 from the reference value for the standard. In addition, the SRM1827b density results indicate no significant issues with the measurement technique. More specifically, the reported density of the SRM1827b glass (from NIST) is 2.548668 ± 0.000032 g/cm^3. The average measured density in this testing for this standard glass was 2.54933 g/cm^3, a difference of ~0.0007 g/cm^3 from the reference value for the standard.

Table 1. Individual and Average Density Measurements for the SB8 VS Glasses and for the SRM1827a and SRM1826b NIST Standard Glasses.

		1st Read	2nd Read	Average
ES/VSL Glass ID	Description	Density (g/cm^3)	Density (g/cm^3)	Density (g/cm^3)
SRM 1826b	Standard	2.550	-	2.550
SRM1827a	Standard	3.592	-	3.592
VSL-SB8-20	D-Opt EV, 36% WL	2.723	2.722	2.723
VSL-SB8-04	1050 gallons ARP, 34% WL	2.686	2.688	2.687
VSL-SB8-09	Sludge-only, 40% WL	2.739	2.741	2.740
VSL-SB8-03	Sludge-only, 34% WL	2.693	2.691	2.692
VSL-SB8-07	Sludge-only, 38% WL	2.721	2.723	2.722
VSL-SB8-15	D-Opt EV, 36% WL	2.681	2.680	2.681
SRM 1826b	Standard	2.547	-	2.547
SRM 1827a	Standard	3.596	-	3.596
VSL-SB8-11	D-Opt EV, 36% WL	2.704	2.703	2.704
VSL-SB8-22	D-Opt EV, 36% WL	2.698	2.701	2.700
VSL-SB8-19	D-Opt EV, 36% WL	2.703	2.702	2.703
VSL-SB8-16	D-Opt EV, 36% WL	2.707	2.707	2.707
VSL-SB8-21	D-Opt EV, 36% WL	2.708	2.710	2.709
SRM 1826b	Standard	2.549	-	2.549
SRM 1827a	Standard	3.591	-	3.591
VSL-SB8-05	Sludge-only, 36% WL	2.707	2.705	2.706
VSL-SB8-14	D-Opt EV, 36% WL	2.711	2.710	2.711
VSL-SB8-13	D-Opt EV, 36% WL	2.698	2.700	2.699
VSL-SB8-10	1050 gallons ARP, 40% WL	2.734	2.734	2.734
VSL-SB8-02	1050 gallons ARP, 32% WL	2.673	2.673	2.673
VSL-SB8-17	D-Opt EV, 36% WL	2.702	2.701	2.702
SRM 1826b	Standard	2.550	-	2.550
SRM 1827a	Standard	3.592	-	3.592
VSL-SB8-08	1050 gallons ARP, 38% WL	2.718	2.719	2.719
VSL-SB8-18	D-Opt EV, 36% WL	2.700	2.701	2.701
VSL-SB8-01	Sludge-only, 32% WL	2.674	2.675	2.675
VSL-SB8-12	D-Opt EV, 36% WL	2.721	2.718	2.720
VSL-SB8-06	1050 gallons ARP, 36% WL	2.704	2.705	2.705
SRM 1826b	Standard	2.550	-	2.550
SRM 1827a	Standard	3.592	-	3.592
SRM 1826b	Standard	2.550	-	2.550
SRM 1827a	Standard	3.593	-	3.593

3.0 RESULTS AND DISCUSSION

One of the primary factors affecting the density of the glass waste-form for a sludge-frit system is its WL. In addition, there are two other sources of variation in the density measurements of the study glasses that are of interest: (1) the repeatability of the measurement process utilized for assessing glass density (i.e., how repeatable is the density measurement for a specific glass) and (2) differences in density from one glass to another (both representing the same WL) due to compositional differences in sludge (e.g., with and without including the ARP stream).

The sources of variation are investigated in Figure 1, which plots the measured density values for each study glass with the glasses grouped as described above for the SB8 VS.[7,8] Specifically, three groupings (i.e., EVs, ARP, and SO) are shown.

With respect to the issue of repeatability of the measurements for a given glass, the overlap (or small variation) of the replicate data for each glass demonstrates that the density measurements are very reproducible. Since the density of interest is the true density of the glass produced by DWPF, the replication error (the variation due to the measurement process) for a single glass may be considered as a nuisance factor while the differences in the densities of the glass from one SME batch to the glass from another SME batch must be understood.

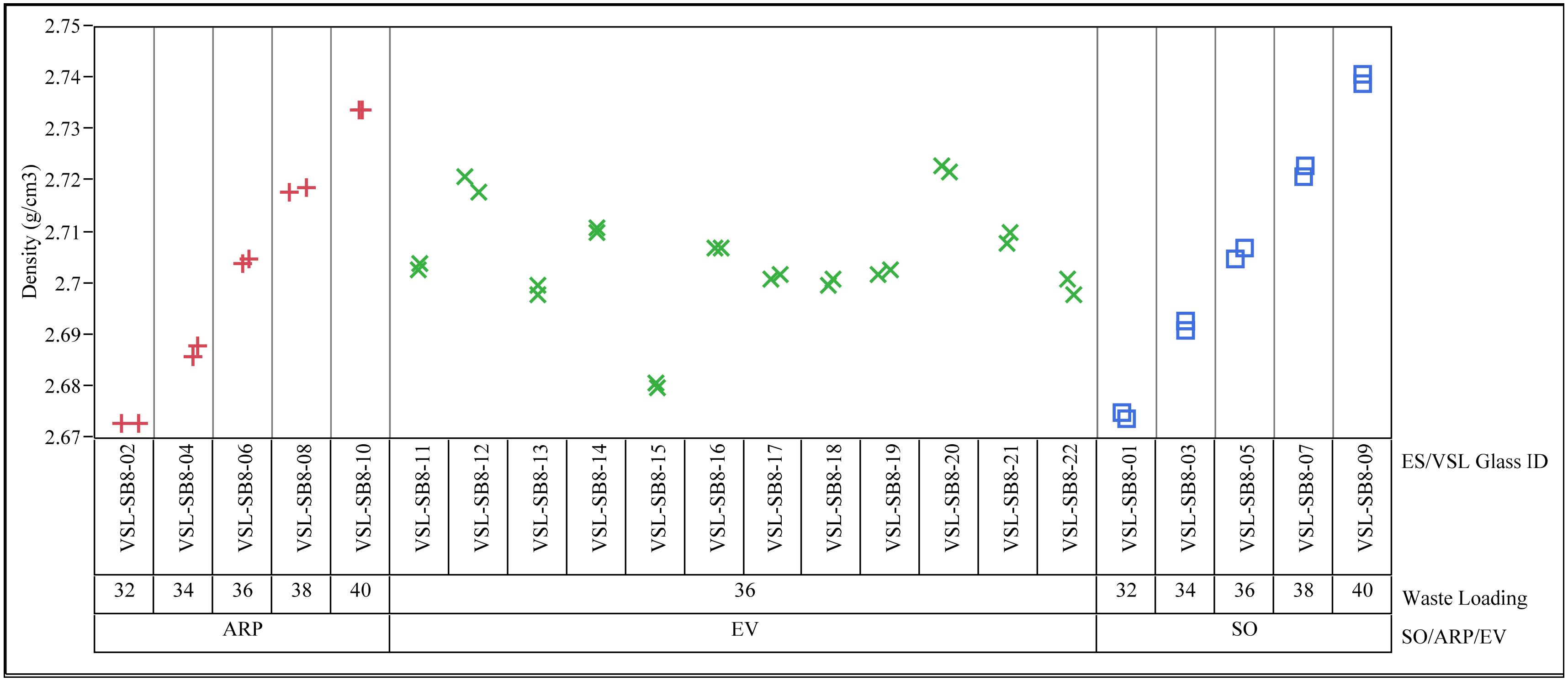


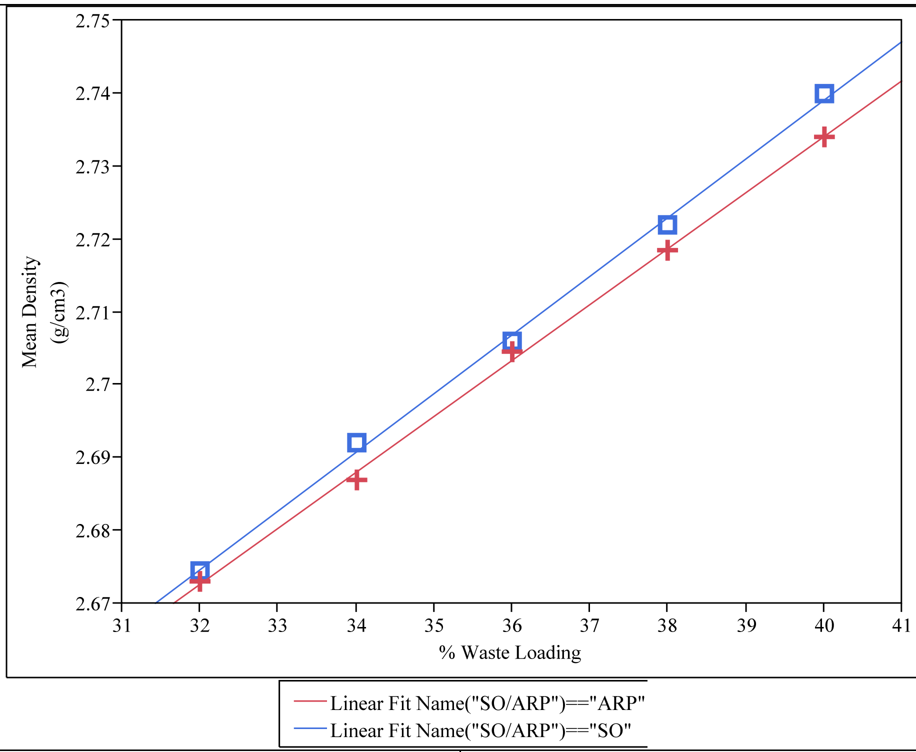
Figure 1. Measured Densities by Glass ID Grouped by Description/Type of Composition and %WL.

Table 2 provides the average density results for each of the SB8 glasses with descriptions that identify the glasses from the EV compositions and those based upon the nominal sludge composition (sludge-only or ARP) combined with Frit 803 at a targeted WL.

Table 2. Average Density Measurement for Each SB8 Glass.

ES/VSL Glass ID	Description	Average Density (g/cm^3)
VSL-SB8-01	Sludge-only, 32% WL	2.675
VSL-SB8-03	Sludge-only, 34% WL	2.692
VSL-SB8-05	Sludge-only, 36% WL	2.706
VSL-SB8-07	Sludge-only, 38% WL	2.722
VSL-SB8-09	Sludge-only, 40% WL	2.740
VSL-SB8-02	1050 gallons ARP, 32% WL	2.673
VSL-SB8-04	1050 gallons ARP, 34% WL	2.687
VSL-SB8-06	1050 gallons ARP, 36% WL	2.705
VSL-SB8-08	1050 gallons ARP, 38% WL	2.719
VSL-SB8-10	1050 gallons ARP, 40% WL	2.734
VSL-SB8-11	EV, 36% WL	2.704
VSL-SB8-12	EV, 36% WL	2.720
VSL-SB8-13	EV, 36% WL	2.699
VSL-SB8-14	EV, 36% WL	2.711
VSL-SB8-15	EV, 36% WL	2.681
VSL-SB8-16	EV, 36% WL	2.707
VSL-SB8-17	EV, 36% WL	2.702
VSL-SB8-18	EV, 36% WL	2.701
VSL-SB8-19	EV, 36% WL	2.703
VSL-SB8-20	EV, 36% WL	2.723
VSL-SB8-21	EV, 36% WL	2.709
VSL-SB8-22	EV, 36% WL	2.700

Figure 2 shows the relationships between density and WL for the nominal SO and ARP VS glasses. As expected, as WL increases, the density of these glasses increases for both sludge compositions with the SO densities being consistently larger than those of the ARP glasses. Since determining an upper bound for the density values at WLs of interest is the objective of this investigation, SO glasses will be used to establish the relationship between density and WL.



Linear Fit Name("SO/ARP")=="ARP"
Mean(Density (g/cm3)) = 2.4271 + 0.007675*Waste Loading

Summary of Fit

RSquare	0.998951
RSquare Adj	0.998601
Root Mean Square Error	0.000908
Mean of Response	2.7034
Observations (or Sum Wgts)	5

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.00235623	0.002356	2856.030
Error	3	0.00000247	8.25e-7	**Prob > F**
C. Total	4	0.00235870		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	2.4271	0.005186	468.01	<.0001*
Waste Loading	0.007675	0.000144	53.44	<.0001*

Linear Fit Name("SO/ARP")=="SO"
Mean(Density (g/cm3)) = 2.4171 + 0.00805*Waste Loading

Summary of Fit

RSquare	0.998421
RSquare Adj	0.997894
Root Mean Square Error	0.001169
Mean of Response	2.7069
Observations (or Sum Wgts)	5

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.00259210	0.002592	1896.659
Error	3	0.00000410	1.367e-6	**Prob > F**
C. Total	4	0.00259620		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	2.4171	0.006675	362.12	<.0001*
Waste Loading	0.00805	0.000185	43.55	<.0001*

Figure 2. Density as a Function of Waste Loading for the Frit 803 – Nominal SB8 Variability Study Glasses.

It is recognized that the SO linear regression provided in Figure 2 is based on a nominal SB8 compositional projection without accounting for potential waste composition variation. More specifically, the density data and linear fit reported above were based on SB8 SO glasses using a nominal sludge projection that did not account for possible sludge variation at a fixed WL. Thus, the determination of an upper bound for the density of SB8 glasses at a given WL must address two sources of uncertainty: the uncertainty of the linear relationship between glass density and WL in Figure 2, and the effects of sludge variation on glass density.

The uncertainty of the linear fit is addressed using an upper tolerance limit (UTL) approach described by Miller.[15] This approach has been previously used by SRNL to support previous sludge batch density assessments.[2-5] The resulting 100(1–α)%/100(1–α_0)% UTL[a] is for 100(1–α_0)% of all density values of the SB8 glasses at a confidence of 100(1–α)% for each and every WL within the interval from 32 to 40%:

$$\mathrm{UTL}\rho_i = b + m \cdot \mathrm{WL}_i + s\left\{\sqrt{pF_\alpha(p, n-p)}\sqrt{\underline{c}_0\left(\mathbf{X}^T\mathbf{X}\right)^{-1}\underline{c}_0^{\,T}} + z_{1-\alpha_0}\sqrt{\frac{n-p}{\chi^2_{\alpha/2,n-p}}}\right\} \quad (1)$$

where

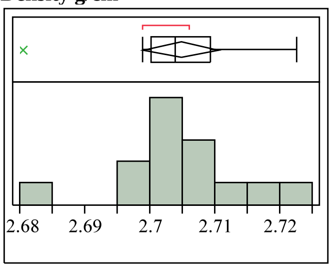
- $\mathrm{UTL}\rho_i$ equals the upper tolerance interval for the glass density at WL_i,
- the estimated slope and intercept of the SO fitted model are m and b, respectively, (where m = 0.00805 and b = 2.4171),
- s is the root mean square error (RMSE) for the fitted model for density as a function of WL (the value is given by 0.001169),
- $F_\alpha(p,n-p)$ is the 100(1-α)% quantile of the F distribution, which depends on n=5 (i.e., the number of data points on which this p-parameter (p=2) model is based), and the desired confidence level for bounding the estimated mean density when the WL is WL_i is represented by 100(1-α)%,
- the inverse product-moment matrix is represented by $(\mathbf{X}^T\mathbf{X})^{-1}$ where the product moment matrix contains information describing the data for the independent variable (i.e., the WLs) used to generate the regression equation (the WL values of this matrix are given as part of the information of Table 2),
- $\underline{c}_0$ is the vector, [1 WL_i], containing the WL_i,
- $z_{1-\alpha_0}$ represents the one-sided 100(1–α_0)% percentile point from the standard normal distribution representing the 1–α_0 fraction of the model predictions to be covered, and
- $\chi^2_{\alpha/2,n-p}$ represents the lower (i.e., 100(α/2)%) percentile point of the χ^2 distribution with (n–p) degrees of freedom, used to establish an upper bound for the variance of the densities around the fitted line.

However, using equation (1) to bound the density for the SB8 glass system based solely on the results from fitting the SO regression line of Figure 2 would be inadequate to capture the impact of the variation in sludge composition on density. To quantify this variation, the densities of all of the EV-based glasses are utilized as well as the SO and ARP glasses at 36% WL. Figure 3 provides a histogram and descriptive statistics for these data. From this figure, the standard deviation of the density values for the EV-based glasses is given by 0.009901 g/cm^3. This standard deviation includes variation in the densities due to differences in sludge composition. Substituting the 0.009901 g/cm^3 value in the computation of equation (1) for the RMSE value instead of the 0.001169 g/cm^3 value from Figure 2 provides a more representative UTL for the relationship between density and WL for

[a] The UTLs were determined using the approach provided on page 124 of Miller. The notation x%/y% UTL, such as 95%/95% UTL, will be used to represent these UTLs. The notation refers to the x% confidence tolerance interval with y% of the densities (in this case) being less than the UTL. The approach is based on a normal distribution.

the SB8 glass system since it accounts for variations in the density values due to variations in the sludge composition. As this substitution is made in equation (1), the number of degrees of freedom, 13, for the 0.009901 value from Figure 3 is also used for the determination of the radical involving the χ^2 distribution as well as the degrees of freedom for the χ^2 distribution itself.

Density g/cm³



Quantiles

100.0%	maximum	2.7225
99.5%		2.7225
97.5%		2.7225
90.0%		2.721
75.0%	quartile	2.70938
50.0%	median	2.704
25.0%	quartile	2.70025
10.0%		2.68975
2.5%		2.6805
0.5%		2.6805
0.0%	minimum	2.6805

Moments

Mean	2.7047143
Std Dev	0.0099009
Std Err Mean	0.0026461
Upper 95% Mean	2.7104309
Lower 95% Mean	2.6989977
N	14

Figure 3. Histogram and Descriptive Statistics for the Densities of SB8 VS Glasses at 36% WL.

Based upon the information in Figure 2 and Figure 3 and utilizing equation (1), a 99% upper tolerance limit (using 0.009901 for the value of s in the equation) was developed to provide an upper bound at 99% confidence for the densities of SB8 glasses at WLs from 32 to 40%. Table 3 summarizes the predicted (mean) density as a function of WL for the linear model, and the 99%/99% UTL.

Table 3. Predicted Densities as a Function of WL for the Linear Fit with 99%/99% UTLs.

WL	Predicted Mean Density (g/cm^3)	99%/99% UTL
32	2.675	2.779
34	2.691	2.777
36	2.707	2.786
38	2.723	2.810
40	2.739	2.843

It is recommended that DWPF utilize the 99%/99% UTL values given in Table 3 as the upper bounds for the densities of SB8 glasses at the WLs indicated to assess the fissile concentration in this glass system.

4.0 SUMMARY

DOE-SR has provided direction to SRR to maintain fissile concentration in glass below 897 g/m^3. In support of that guidance, SRNL provided a technical basis and a supporting Microsoft® Excel® spreadsheet for the evaluation of fissile loading in SB5 glass based on the Fe concentration in glass as determined by the measurements from the SME acceptability analysis. SRR has since requested that the necessary density information be provided to allow SRR to update the inputs to the Excel® spreadsheet so that it may be used to maintain fissile concentration in glass below 897 g/m^3 during the processing of SB8.

One of the primary inputs into the fissile loading spreadsheet includes a bounding density for SB8-based glasses. Based on the measured density data of the SB8 variability study glasses, it is recommended that SRR utilize the 99%/99% UTL density values given below as bounding densities for SB8 glasses to assess the fissile concentration in this glass system at the WLs as indicated. It should be noted that no changes are needed to the underlying structure of the Excel®-based spreadsheet to support fissile assessments for SB8. However, SRR should update the other key inputs to the spreadsheet that are based on fissile and Fe concentrations reported from the SB8 Waste Acceptance Product Specifications (WAPS) sample.

WL	**99%/99% UTL**
32	2.779
34	2.777
36	2.786
38	2.810
40	2.843

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