Conf- 9104256--8.
Pt.4

WSRC-MS--90-338

DE92 009753

CONTROL OF HIGH-LEVEL RADIOACTIVE WASTE-GLASS MELTERS - PART 4: PRELIMINARY ANALYSIS OF DWPF PROCESS LABORATORY CAPABILITIES (U)

by

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A paper proposed for presentation at the
International Symposium on Ceramics in Nuclear Waste Management
Cincinnati, Ohio
April 28, 1991

and for publication in the proceedings

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CONTROL OF HIGH-LEVEL RADIOACTIVE WASTE-GLASS MELTERS - PART 4: PRELIMINARY ANALYSIS OF DWPF PROCESS LABORATORY CAPABILITIES

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INTRODUCTION

The Defense Waste Processing Facility (DWPF) will immobilize Savannah River Site High Level Waste as a durable borosilicate glass for permanent disposal in a repository. The DWPF will be controlled based on glass composition. The waste glass physical and chemical properties, such as viscosity, liquidus, and durability are functions of glass chemistry. A preliminary assessment of the glass composition analysis and property control capabilities has been conducted based upon melter feed sampling, vitrification, and Inductively Coupled Plasma / Atomic Absorption spectroscopy. The sources and consequences of variations are discussed.

The following discussion is a preliminary analysis of the capability of the laboratory methods that can be used to control the glass composition, and the relationships between glass durability and glass properties important to glass melting. The glass durability and processing properties will be controlled by controlling the chemical composition of the glass. The glass composition will be controlled by control of the melter feed transferred from the Slurry Mix Evaporator (SME) to the Melter Feed Tank (MFT). During cold runs, tests will be conducted to demonstrate the chemical equivalence of glass sampled from the pour stream and glass removed from cooled canisters. In similar tests, the compositions of glass produced from slurries sampled from the SME and MFT will be compared to final product glass to determine the statistical relationships between melter feed and glass product. The total error is the combination of those associated with homogeneity in the SME or MFT, sampling, preparation of samples for analysis, instrument calibration, analysis, and the composition/property model. This study investigated the sensitivity of estimation of property data to the combination of variations from sampling through analysis. In this or a similar manner, the need for routine glass product sampling will be minimized, and glass product characteristics will be assured before the melter feed is committed to the melter.

The precision and accuracy of glass analysis and composition prediction is determined by the abilities to sample, to prepare for analysis and to analyze. DWPF glass composition analysis development has had as its goal that: The results obtained for any key analysis on any single sample that is critical for process control or accountability must be within at least +/- 10% at the 95% confidence level. Results for alkali metals should be within +/- 5% of the true value at the 95% confidence level. The emphasis on having a statistically justifiable value recognized the dual requirements of meeting repository criteria (accountability, durability), and of meeting DWPF operability requirements (production rate goals, continuity of melter operations).

The general validity of these criteria was later substantiated for waste acceptance, but indicated that total control in glass composition must have a narrower absolute range to avoid operating difficulties [1]. The analysis used preliminary predictive equations for glass properties to determine the sensitivity of critical glass properties to variations in the actual relative amounts of the three major streams blended to form waste glass: frit, sludge, and precipitate hydrolysis product. The Slurry Mix Evaporator (SME) to Melter Feed Tank (MFT) transfer was established as the final and most important hold point since it is the last point at which adjustments can be made to the feed prior to its introduction to the melter. The analysis used the three glass property criteria of [2]:

1) Glass durability: Free Energy of Hydration > -7.0 kcal/mole, based on a Free Energy of Hydration model.
2) Liquidus: < 1050 °C, based on a glass composition model.
3) Viscosity: 20 to 100 poise at 1150°C, based on a glass composition model.

The evaluation concluded that control to a total variation of composition of +/-5% should be typical to meet the conflicting constraints of glass durability and processability, with 10% total error on an absolute basis being the maximum which can be tolerated.

DISCUSSION

Preliminary glass property models used in Reference [2] are being updated into final models. As an example for waste glass durability the summation of the products of the mole fractions of the i^{th} compound and the partial molar Free Energies results in the "Glass Free Energy of Hydration", which is what has been typically reported as a glass property, and has a lower limit of about -7 kcal/mole to meet durability requirements. For SRL 165 Average glass the corresponding value is -4.48 kcal/mole, while for SRL 131 Average glass it is -6.19 kcal/mole . The difference between these "target" values and the durability criteria is an estimate of the total range of permissible deviation.

The most current revisions of the glass property equations were used to estimate the major physical properties, which are compared to the operating limits in Table I. *The difference between the operating limits and the expected value of the properties* *constitutes the preliminary operating margin, which consists of the total range that can be attributed to batching variations, sampling errors, analytical variations, and the margin required to establish confidence that the actual value of the limit is met.*

PRELIMINARY POOLED STANDARD DEVIATIONS FOR CALCULATED PHYSICAL PROPERTIES

Since the models of glass durability and glass properties are being developed in terms of the glass composition, it is possible to determine the sensitivity of each property to the variation in analytical precision, and pool the variations in such a way as to weigh both the amount of variation in determination of the elements, and their relative importance on the property. Given the property model equation U:

$$U = U(x_1, x_2, \ldots, x_n)$$

where x_i is the i^{th} chemical element in the glass,

$$\sigma_U^2 = \Sigma_i (\delta U/\delta x_i)^2 \sigma_{xi}^2 \pm (2\Sigma_i \Sigma_{j \neq i} \text{ cv } (x_i, x_j) [\delta U/\delta x_i] [\delta U/\delta x_j]) \tag{1}$$

Where σ_U is an estimate of the pooled standard deviation, which can be compared to the operating margin in the previous section. The second term depends upon the covariance of the elements in the glass (cv). This covariance may be the result of the tendency for sampling to preferentially separate some elements (e.g. those in solution vs. those primarily in coarse particles), in the sample preparation or analysis (e.g. spectral interferences), or in the property modelling (e.g. substitution effects of transition metals in glass devitrification). The estimation of the effect of covariance is beyond the scope of this report, and is the subject of ongoing statistically rigorous analyses (See Postles and Brown, these proceedings). This analysis is intended only as a first order approximation to estimate in a timely manner the production laboratory's capabilities based on methods demonstrated in a different laboratory. Therefore, the second term is not included in the following calculations. Other sites have elected to fit empirical models to their compositional variations [4-7]: in those cases the second term would also be required for a more complete analysis.

The main value of the pooled standard deviation approach is that it weights each element by the its contribution to the property being considered. The calculation produces a single value which can be compared to the operating margin discussed in the introduction, as a means of estimating how many samples are required to provide the necessary confidence that property requirements are being met. Thus, the pooled standard deviation can be used to characterize the statistical spread of properties predicted from compositional analysis, and to determine which variations in elemental analyses are most heavily weighted in the estimation of properties.

The standard deviation of the measurement of the chemical elements (σ_{xi}) in waste glass has been determined by several laboratories [4, 8-10], including round robin testing of identical glass samples. In two cases this has been further broken down into causes of the variations in analysis [9, 11], with the conclusion that the variation resulting from long term drift in the ICP measurements (i.e. calibration variations, and drift with time) is about equal to the other sources of variation (e.g. sampling, sample preparation and analysis). For the following analysis the combination of sampling, glass preparation and analytical variation was demonstrated on a full scale SME with prototypical sampler and sample recirculation loop.

Table 1. Operating Margins for Initial DWPF Glass

Limit	Nominal Value	Margin	Units
Durability			
> -7.0	-5.12	1.88	kcal/mol
Liquidus			
<1050	998	52	°C
Lower Viscosity			
>20	67	47	poise
Upper Viscosity			
<100	67	33	poise

In this example, $\delta U/\delta x_i$ is just the partial molar Free Energy of Hydration, which is indicated in discussions of the model, as "Component Free Energy" . Thus, if there are no other uncertainties in the Free Energy of Hydration Model, then the pooled standard deviation can be used together with the Student T- test to estimate the number of samples required to guarantee that glass complies with the criterion of $\Delta G > -7$. Based upon this approach, the demonstrated analytical variation is in order of decreasing importance for estimating durability: K, Si, Li, Fe, B, Na, Ti, Mn, and Al. Most of the variations in estimating liquidus and viscosity are attributable to Si.

Sample recovery is assumed to be used in the following analysis to eliminate outlying samples which result from errors in sample transfer and preparation. This method is based upon dissolving a known mass of sample (glass- not slurry) in a known quantity of solvent . The amount of dilution of the sample is also rigorously controlled. The ICP is calibrated in terms of ppm using a matrix matched standard, which permits reporting of the analysis in terms of ppm, rather than as a renormalized analysis. Thus, knowing the respective oxide compounds of the elments it is possible to reconstruct the oxide that was originally dissolved. Experience indicates that the analysis is acceptable when the recovered mass is within +/- 5% of the actual mass dissolved, excluding elements not analyzed (whose content is estimated based on prior knowledge or a statistical method such as the Kalman filter). However, once the analysis is deemed acceptable on this basis, the analysis should be renormalized to 100% to perform any property calculations, since it is the relative amounts of the elements which are important for process control, and since the overall variances are reduced by the renormalization . Advantages of the sample recovery method are 1) unambiguous elimination of faulty analysis, on a technically justifiable basis. 2) An indication of the effectiveness and control of sample dissolution and dilution, with possible clues as to the source of the deviation. 3) Straightforward procedures for including the check as part of the computerized data storage system. Disadvantages of the sample recovery method are 1) increased need to control sample weights, and sample dilutions. 2) disqualification of some marginal analyses that would otherwise be considered acceptable.

CALIBRATION VARIATIONS

ICP calibrations are conducted using a matrix of standard solutions with different concentrations of the elements to be determined, with a solute composition similar to that of actual samples (i.e. matrix matched). Therefore, the precision of such calibrations is subject to to the precision of sample measurements during the calibration, and is subject to the same statistical fluctuations. Once the equipment has been calibrated, any such variation, combined with any drift of the response of the equipment with time, becomes a bias on the subsequent measurements. The initial calibration bias, and drift over time can be estimated by reanalyzing the calibration solution at regular intervals. The precision of the estimation of bias should be somewhat better than that of analyzing replicate glass samples, since sample preparation is not a variable. It does have similar limitations with respect to the number of determinations required to establish confidence. For the sake of the following examples, the value of the variation caused by ICP calibration is assumed to be similar to 60+ analyses of standard solutions over a four month period [11]. Using the above method:

$\sigma\Delta G_{\text{ICP cal, pooled}} = 0.05 \text{ kcal/mol}$

$\sigma\ \text{LIQ}_{\text{ICP cal, pooled}} = 21\ {}^{\circ}\text{C}$

$\sigma\ \text{VIS}_{\text{ICP cal, pooled}} = 0.9 \text{ poise at } 1150^{\circ}\text{C}$

CORRELATION OF SAMPLES

There is a possibility that the results of a sample analysis are influenced by sample carry over from the previous sample. A blank sample is therefore suggested to check for this. The blank sample should consist of the same acid solution used for sample dissolution, and processed through sample digestion, dilution, etc. with the other samples. Instrument flushing should also be the same. The use of the blank also provides a check that the processing equipment and stock solutions have not picked up chemical contamination.

EXPERIMENTAL VARIATIONS AND BIASES RESULTING FROM SAMPLING, SAMPLE PREPARATION, AND ANALYSIS

The statistical study conducted for WVDP indicates that the variations caused by sample prep (sampling, digesting the glass and diluting it to a solution suitable for ICP) is generally small, provided that outliers can be detected and eliminated, which is in general agreement with the analytical results developed in round robin testing.

However, in the DWPF relatively coarse frit is used as a decontamination abrasive, and is then added to the SME as the major source of Si. Simulated melter feed slurry samples were pipetted from a laboratory blender into standard sample vials, and the samples were subsequently transfered into platinum crucibles and vitrified, to evaluate the impact of frit settling, The results of this direct transfer method were not satisfactory, producing a high percentage of outlier results, and wide variations in analysis. Subsequent tests determined that up to 0.2 gram of coarse frit remained in the vial from a 10 cc sample. To overcome this, several transfer, drying, and feed homogenization methods were tried in replicate, and analyzed. Methods which were tried included: 1) a control consisting of slurry directly pipetted from the blender and vitrified, 2) sample vials dumped into a Teflon (TM) coated crucible, the vial flushed with water, the resultant sample dried, mechanically mixed and vitrified, 3) sample vials dumped into a crucible, the vial flushed with water, the resultant sample dried, and the vial roated at an angle during the vitrification, and 4) samples aliquoted from an agitated sample vial. Based upon these tests, the method selected for subsequent SME and SME sampler characterization is a combination of Teflon (TM) coating of the crucible, drying of the slurry, coarse mechanical crushing of the dried cake, followed by 4 hours vitrification at 1150°C in a crucible inclined at a 45° angle and rotated at the rate of 0.2 rpm. A vial flushing device was developed, and a device to automatically perform the crucible roation is being developed. The combined methods are capable of distinguishing the differences between samples containing 5% excess frit. Actual DWPF sample analyses will use somewhat different preparation methods, but results are expected to be similar, based on remote demonstration of most of the methods used in this study.

COMBINED SAMPLING AND ANALYTICAL VARIATIONS

The combination of variations in sampling with those of sample analysis can be performed by taking the square root of the sum of the squares of all the corresponding standard deviations. If the correction for the calibration bias is not necessary, then this results in the pooled standard deviations from sampling and analytical methods presented in Table 2.

EXAMPLE SAMPLE METHOD FOR ACCEPTANCE OF SME PRODUCT

The following sample scheme is an example of what might be routine for analyses of DWPF production SME batches. It is realized that more analyses will be required during cold runs to determine sampling bias, to characterize covariance, and to provide increased confidence that the precision and accuracy of the sampling method, sample preparation, and ICP/AA analyses are known and stable. In this case it is assumed that the precision and accuracy of the samples is comparable to that demonstrated by SRL in sampler testing, using the best available analytical techniques.

Four samples of SME product are taken close to the final wt% solids and are separately vitrified and ground. Two weighed subsamples of each vitrified sample and one sample of a standard glass are weighed and microwave dissolved, after spiking each acid solvent with an internal standard. Any dilution required to reduce the sample concentration is controlled such that the sample recovery method can also be used. Solutions are retained until the batch has been characterized, and accepted. Remaining powdered sample glasses are archived as necessary.

The resulting samples are analyzed in the sequence:

Cal Std S1 S2 Std SG S3 S4 Std S5 Blank S6 Std S7 S8 Std

Where Cal is ICP calibration using different solutions with multiple concentrations of the elements to be measured, Std is measurement of a stock solution prepared by dissolving a reference glass (containing typical concentrations of the major glass elements and an internal standard) but without any adjustment of the calibration, S1 -S8 are the waste glass samples, and SG is the sample prepared by dissolving the standard glass. Any analyses which do not meet the sample recovery criteria are reanalyzed, and the results checked vs sample recovery criteria. The accepted analyses are normalized and averaged, and reported (together with the number of acceptable samples) as the batch composition for waste quality and operability concerns. If more than 2 of the 8 waste glass samples fail the sample recovery criteria, then additional slurry samples are taken, and the procedure repeated until a minimum of 6 samples have passed the sample recovery criteria. The standard samples may be used to correct for any calibration bias.

Based upon this, using the one sided Student-t statistic, and assuming that 6 sample analyses are considered valid, there remain available 5 degrees of freedom, and for n samples:

$$\text{ERROR}_{\text{total, pooled, conf.\%}} < t_{n-1,\text{conf. \%}} * s_{\text{pooled}} / n^{1/2}$$

$$ERROR_{total,\ pooled,\ 95\%} < 0.90 * s_{pooled}$$

$$ERROR_{total,\ pooled,\ 75\%} < 0.32 * s_{pooled}$$

CONCLUSIONS

The results of full scale SRAT sampling with a prototypic sampler and sample vitrification have been compared to process and product requirements using simplified statistical methods and preliminary correlations of glass composition to property. Based upon this analysis, waste product quality can be assured with a moderate number of replicate samples. An example sample and analysis scheme has been demonstrated. With reduction in the confidence level that is considered acceptable for liquidus, the sample scheme yields:

$ERROR\ \Delta G_{total,\ pooled,\ 95\%}$ < 0.06 kcal/mol
< 5 % of Operating Margin

$ERROR\ LIQ_{total,\ pooled,\ 75\%}$ < 9 °C
< 20 % of Operating Margin

$ERROR\ VIS_{total,\ pooled,\ 95\%}$ < 1.0 poise at 1150°C
< 3 % of Lower Oper. Margin
< 3 % of Upper Oper. Margin

The above confidence levels should be sufficient, since actual batch to batch variations in glass durability, viscosity and liquidus will be damped by mixing with other batches in the melter feed tank, and in the melter itself. Actual values will depend upon demonstrated vatiations and biases under production conditions, and may require more replicate analyses to provide adequate confidence that the process controls are being met. Biases adjustment will probably be required on liquidus estimations.

ACKNOWLEDGEMENTS

This paper was prepared in connection with work done under contract No. DE-AC09-89SR18035 with the U.S. Department of Energy. The authors wish to thank C.L. Fow, and B.A. Pulsipher of PNL, and R.L. Postles, W.J. Jenkins, C.P. Reeve, and C.M. Jantzen of SRL for their advice and comments. Glass samples were prepared and analyzed by H.D. Rhoad, S.F. Nappier, and M.K. Cooley. Teflon is a registered trademark of E.I. du Pont de Nemours & Co. (Inc.).

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Table 2.
Summary of Variations and Biases in Waste Glass Property Prediction

	ΔG hydration kcal/mol	Liquidus °C	Viscosity poise
POOLED STANDARD DEVIATIONS			
ICP Calibration and Drift	0.05	21	0.9
High Solids Slurry (49 wt%)			
Sampler & Analysis	0.03	15	0.6
Sampl., Anal., & Cal.	0.06	26	1.0
Low Solids Slurry (41 wt%)			
Sampler & Analysis	0.03	18	0.7
Sampl., Anal., & Cal.	0.06	28	1.1
BIASES			
High Solids Slurry (49 wt%)			
Sampler (Ref. - Grab)	-0.01	+41	-1.1
Tank Homogeneity (Bottom-Top)	-0.03	-18	+0.4
Low Solids Slurry (41 wt%)			
Sampler (Ref. - Grab)	-0.07	+80	+2.4
Tank Homogeneity (Bottom-Top)	+0.00	-35	+1.0

ADDITIONAL FIGURES FOR PRESENTATION

FIGURE 1A,B,C,D - FRIT ANALYSES :

The purpose of the test was to 1) demonstrate that SRS can check incoming frit to the tight compositional limits required for frit acceptance. 2) Determine the approximate number of samples and standards that are required to accomplish this, with defensible statistical analysis 3) Determine how many standard samples are required for bias correction, and if it is necessary. Glass 202 frit samples from four vendors have been analyzed at SRL by ICP. Fusion is with Na2O2 with nitric acid uptake, paralleled by bomb dissolution in HF-HCl to determine Na2O and ZrO2. Analyses of these results indicate that even when the Frit 165 is used to correct this method, there remains a negative silica bias of about 1%.

K2CO3 fusion with a water uptake was investigated as an alternative, and found acceptable. SRL Frit 165 was used as a standard, together with production frit 202 as a sample. The K2CO3 method has the additional benefit of measuring all the major frit components in one dissolution, unlike the Na2O2 fusion method, which requires an additional HF-HCl dissolution to determine Na.

The alternative ICP preparation method of K2CO3 fusion with water uptake was used to analyze a pattern of 20 samples of one of the frits interspersed with 17 standard Frit 165 samples. Sample recovery by this method is excellent, with only one sample out of 37 falling outside of the bounds of 95 to 105% sample recovery. Essentially all of the variations in Li, Na, and Si analyses are attributable to long term drift in the ICP. The other two major frit components, B and Mg, show similar long term trends but have larger standard deviations which tend to obscure this behavior. The analysis also indicates that the drift can be measured by examining the results for % recovery of the sample, and corrected for by normalizing. Comparison of the average value of the sample frit as measured by Corning, and as normalized K2CO3 fusion data indicates that there is no bias in the method. Bias correction based on the standard was investigated as an alternative to normalizing, but was found to introduce new errors. Comparison of the standard deviations of the unnormalized and normalized K2CO3 data with the permitted range on the glass specifications indicates that either normalization or many duplicate samples will be required to comfirm that samples meet specifications. Thus, the preferred method of frit analysis is K2CO3 fusion with water uptake, with standard samples included, as well as a check on sample recovery. It may be necessary to add a parallel K2CO3 fusion with acid uptake to analyze for minor frit components (transition metals).

STANDARD DEVIATIONS OF ANALYSES OF STANDARD GLASS (FRIT 165)

	B_2O_3	Li_2O	MgO	Na_2O	SiO_2
CORNING GLASS WORKS	0.064	0.045	0.029	0.010	0.017
RAW K_2CO_3 FUSION	0.23	0.13	0.05	0.20	1.35
NORMALIZED K_2CO_3 FUSION	0.10	0.04	0.04	0.07	0.12
SPECIFICATION RANGE	1.0	1.0	0.50	1.0	2.0

Ultrasonic agitation of the sample had no statistical effect on the variation of elements that might be difficult to dissolve in the K_2CO_3 fusion, or precipitate, such as Mg, Ca, and Si. Ultrasonics were examined at the dissolution, dilution, and ICP analysis stage. Ultrasonics might be useful in more complex glasses, but the K_2CO_3 fusion was so effective that there were no significant undissolved materials or precipitates for the ultrasonics to suspend.

SLURRY ANALYSES

FIGURE 2

It was determined that up to 0.2 grams of frit are retained in the bottom of the vial. This is a potential error of about 6% in the frit determination. A bottle washer device was designed and constructed which flushes the vial into the crucible used to perform the fusion.

FIGURE 3 A,B

An improved procedure was developed for the vitrification of slurry samples prior to analysis by ICP and AA. Standard glass samples (STD) were interspersed with slurry directly pipetted from a Waring blender (SD), sample vials dumped to a Teflon coated crucible (TC), sample vials dumped to a crucible precessed during vitrification (RC), and samples aliquoted from an agitated sample vial (AM). Where appropriate, the bottle washer device was used to flush the vials.

Four batches of slurry were agitated in a Waring blender and sampled with a 10 ml transfer pipette while the blender was operating. The samples consisted of as received SRAT/SME run #8 product (labeled G), the same material with 100 ml/l of the clear solution replaced with water (labeled H), as received plus 5 % additional Frit 202 (labeled R), and as received plus 10% additional Frit 202 (labeled T). Using the old sample preparation methods, it had been impossible to distinguish between the four slurry types, even with 8 replicate analyses each. In the new method, the crucibles were coated with Teflon (TM) spray and drained before each sample was transfered from its sample vial. After drying, a plastic spatula was used to scrape the residue from the crucible wall, and combine it with the sample in the crucible's bottom. The sample was coarse ground in the crucible, and it was evident that the frit had settled on the crucible bottom, leaving a sludge and salt rich layer above it. The as-dried samples were placed in a furnace mounted on a platform that inclined the furnace at about a 40° angle. The platform was precessed 60° per minute during the vitrification. This stirred the contents of the crucibles, and tended to dissolve any remaining residue on the crucible wall.

The G, H, R, and T samples were analyzed in groups of four, bracketed by standard glass (STD), and show statistically significant differences.

FIGURE 1A

ELEMENTAL FRIT ANALYSES

ALTERNATING GROUPS OF 2 HOMOGENEOUS GLASSES

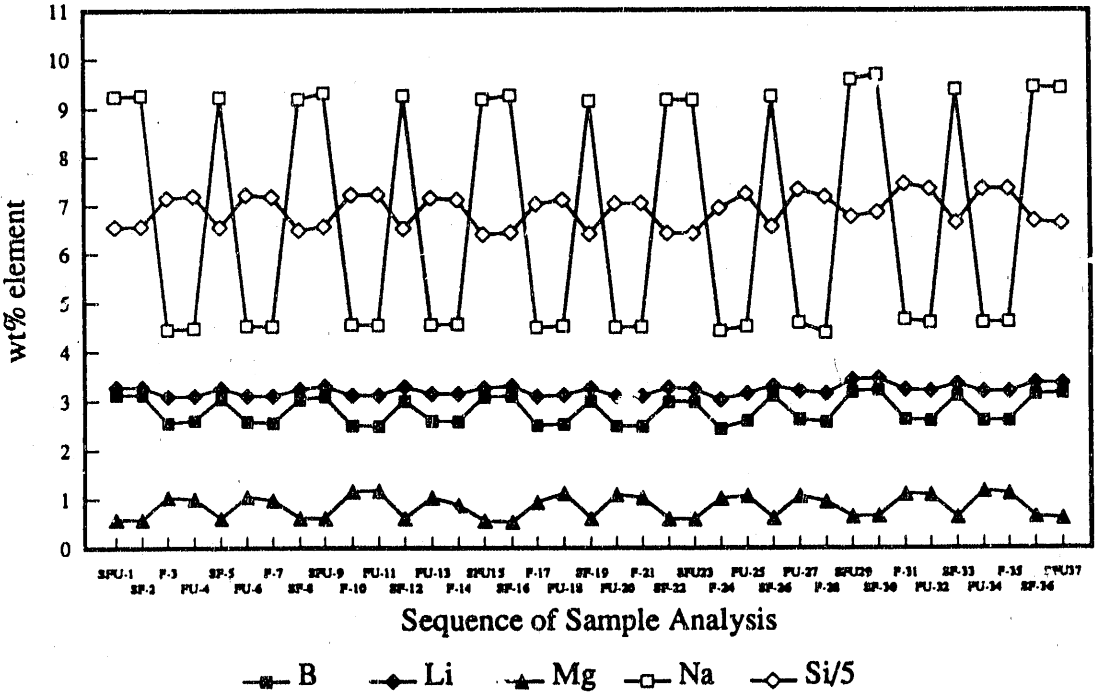


FIGURE 1B

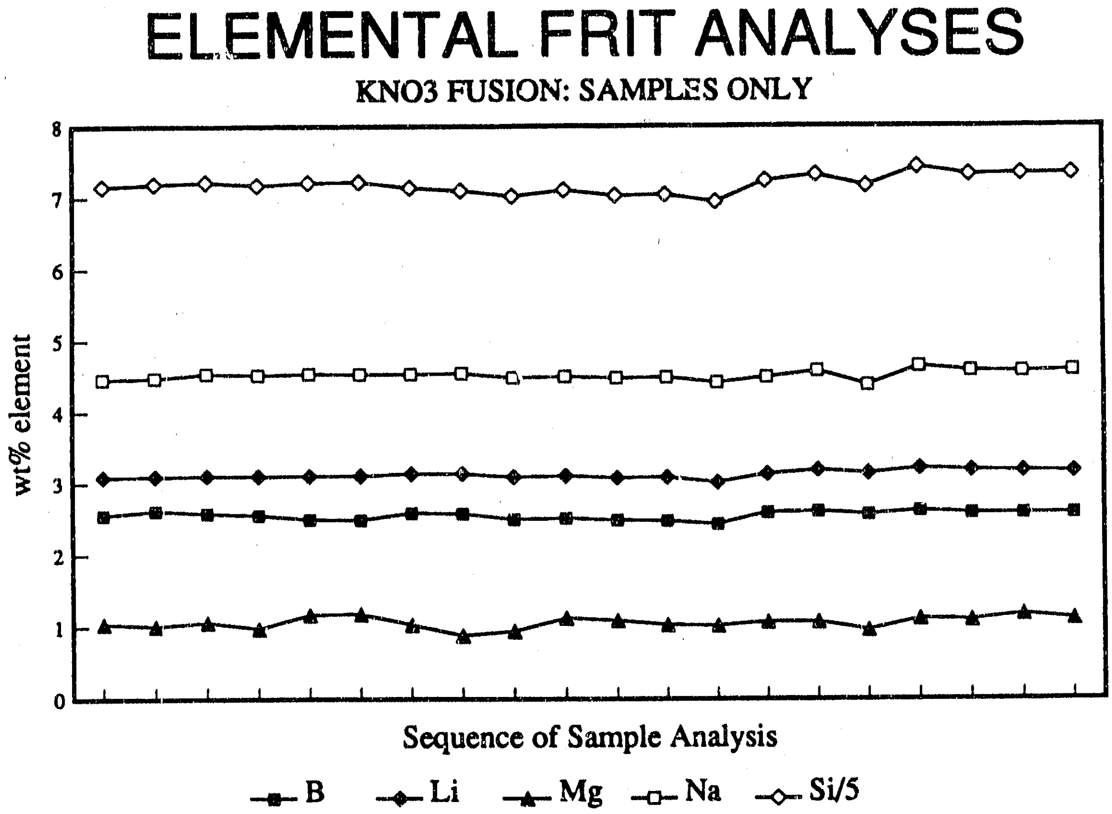

ELEMENTAL FRIT ANALYSES
KNO3 FUSION: SAMPLES ONLY
wt% element
0
1
2
3
4
5
6
7
8
Sequence of Sample Analysis
B
Li
Mg
Na
Si/5

FIGURE 1C

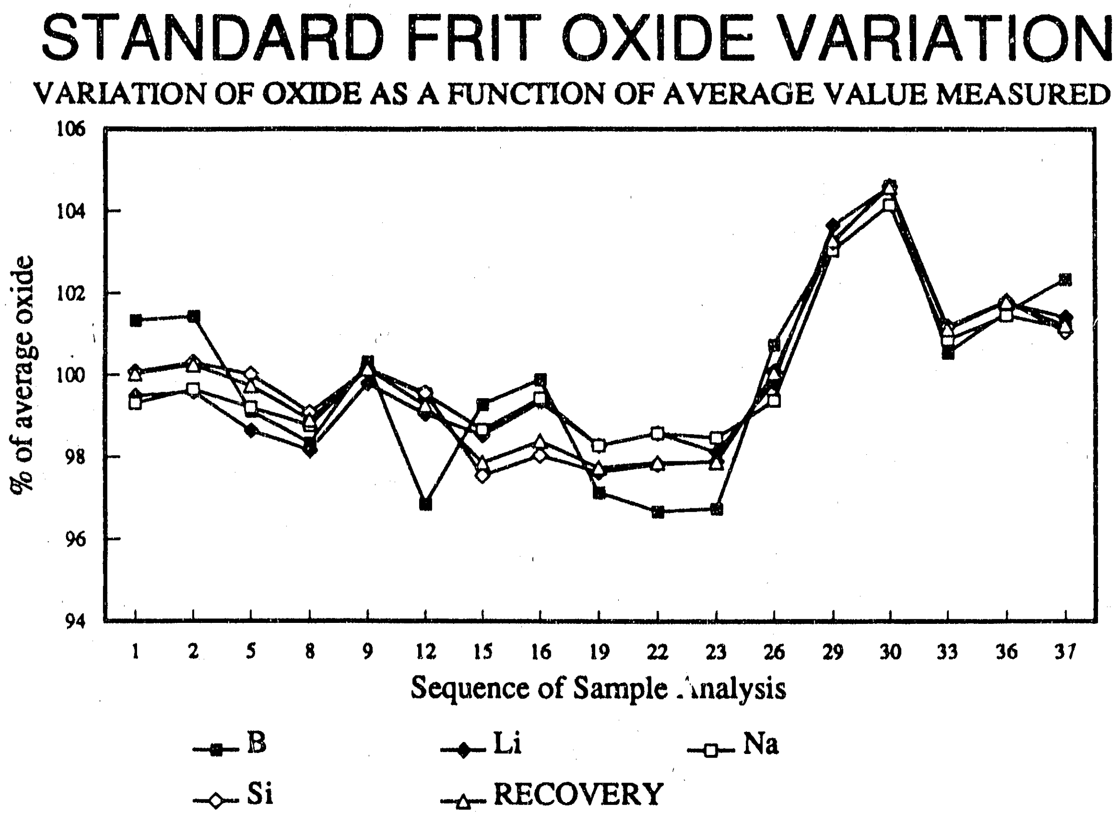

STANDARD FRIT OXIDE VARIATION
VARIATION OF OXIDE AS A FUNCTION OF AVERAGE VALUE MEASURED
% of average oxide
106
104
102
100
98
96
94
1
2
5
8
9
12
15
16
19
22
23
26
29
30
33
36
37
Sequence of Sample Analysis
B
Li
Na
Si
RECOVERY

FIGURE 1A

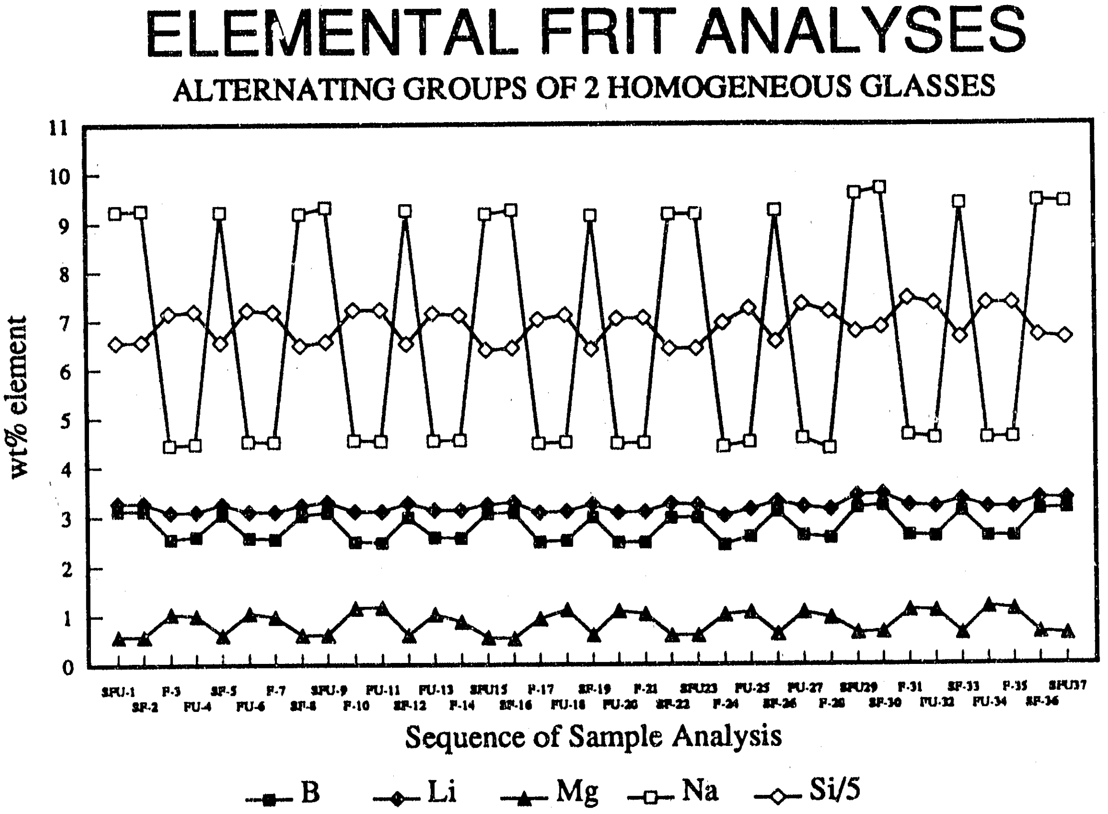

ELEMENTAL FRIT ANALYSES
ALTERNATING GROUPS OF 2 HOMOGENEOUS GLASSES
wt% element
0
1
2
3
4
5
6
7
8
9
10
11
SFU-1
SF-2
F-3
FU-4
SF-5
FU-6
F-7
SF-8
SFU-9
F-10
FU-11
SF-12
FU-13
F-14
SFU15
SF-16
F-17
FU-18
SF-19
FU-20
F-21
SF-22
SFU23
F-24
FU-25
SF-26
FU-27
F-28
SFU29
SF-30
F-31
FU-32
SF-33
FU-34
F-35
SF-36
SFU37
Sequence of Sample Analysis
B
Li
Mg
Na
Si/5

FIGURE 1B

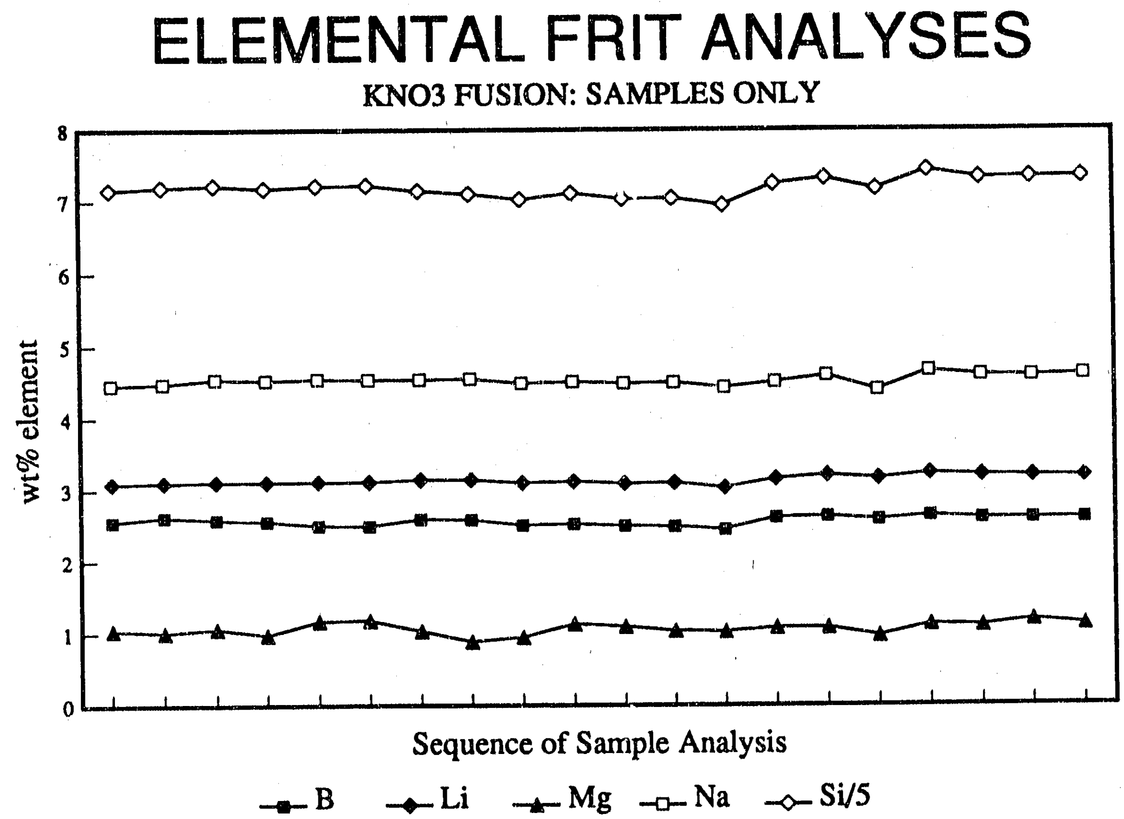

ELEMENTAL FRIT ANALYSES
KNO3 FUSION: SAMPLES ONLY
wt% element
8
7
6
5
4
3
2
1
0
Sequence of Sample Analysis
■ B
◆ Li
▲ Mg
□ Na
◇ Si/5

FIGURE 1C

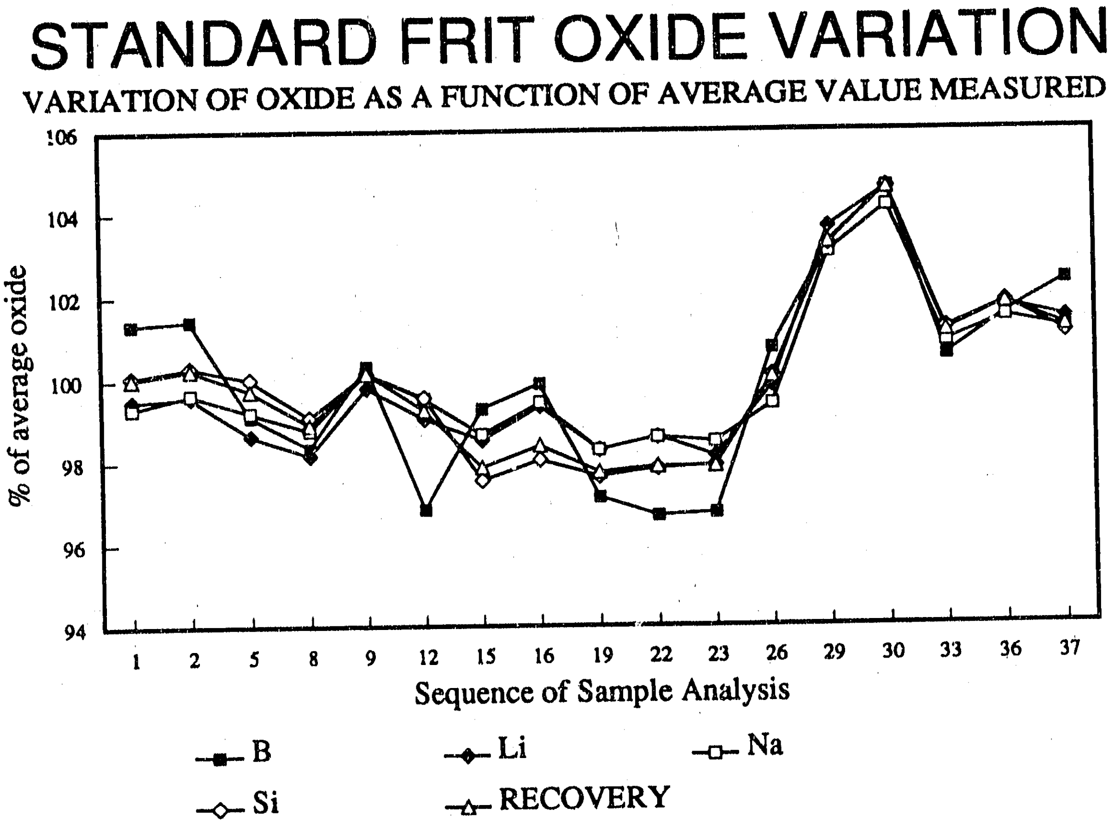

STANDARD FRIT OXIDE VARIATION
VARIATION OF OXIDE AS A FUNCTION OF AVERAGE VALUE MEASURED
106
104
102
100
98
96
94
% of average oxide
1
2
5
8
9
12
15
16
19
22
23
26
29
30
33
36
37
Sequence of Sample Analysis
B
Li
Na
Si
RECOVERY

FIGURE 1D

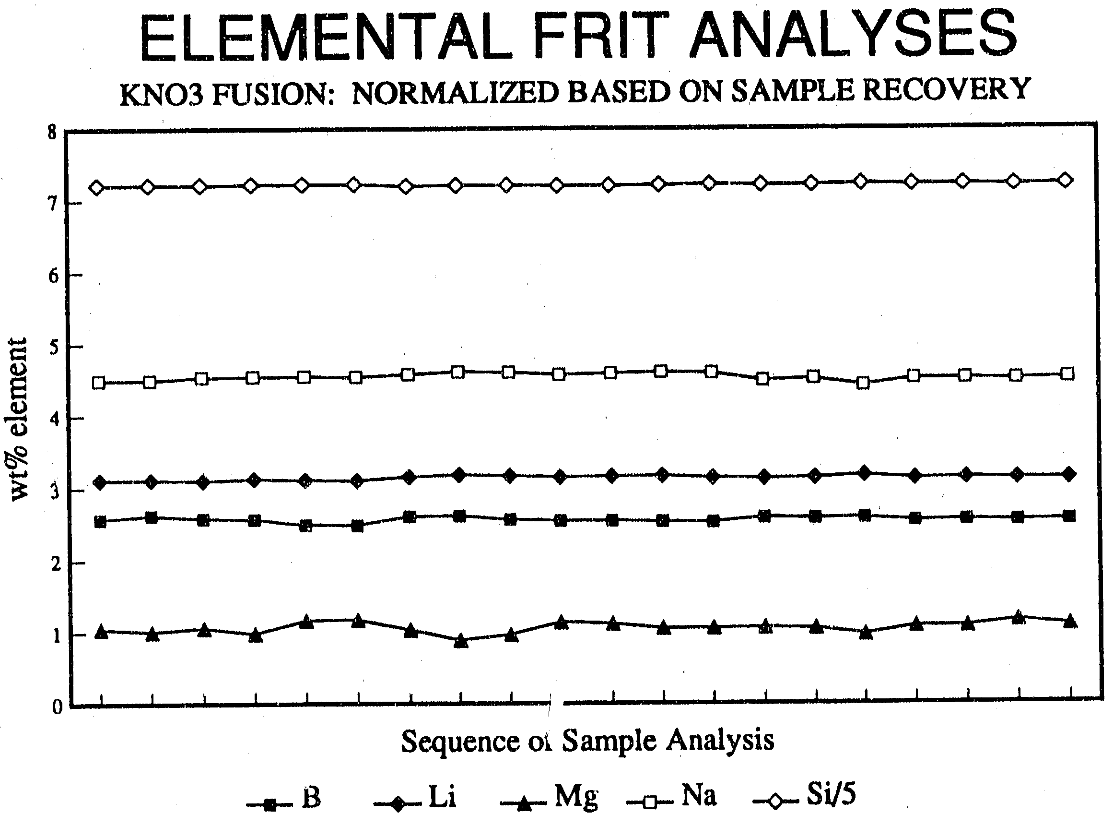

ELEMENTAL FRIT ANALYSES
KNO3 FUSION: NORMALIZED BASED ON SAMPLE RECOVERY
8
7
6
5
4
3
2
1
0
wt% element
Sequence of Sample Analysis
B
Li
Mg
Na
Si/5

FIGURE 2

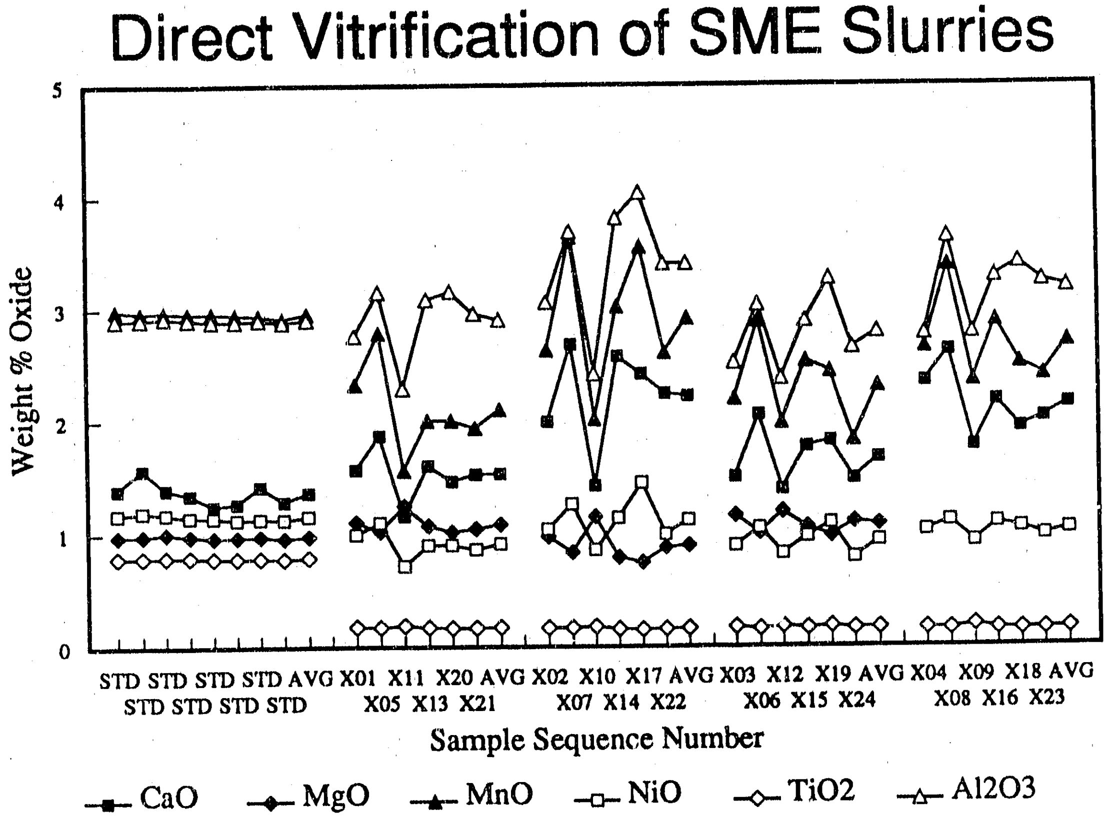

Direct Vitrification of SME Slurries
5
4
3
2
1
0
Weight % Oxide
STD STD STD STD AVG X01 X11 X20 AVG X02 X10 X17 AVG X03 X12 X19 AVG X04 X09 X18 AVG
STD STD STD STD X05 X13 X21 X07 X14 X22 X06 X15 X24 X08 X16 X23
Sample Sequence Number
CaO
MgO
MnO
NiO
TiO2
Al2O3

FIGURE 3A

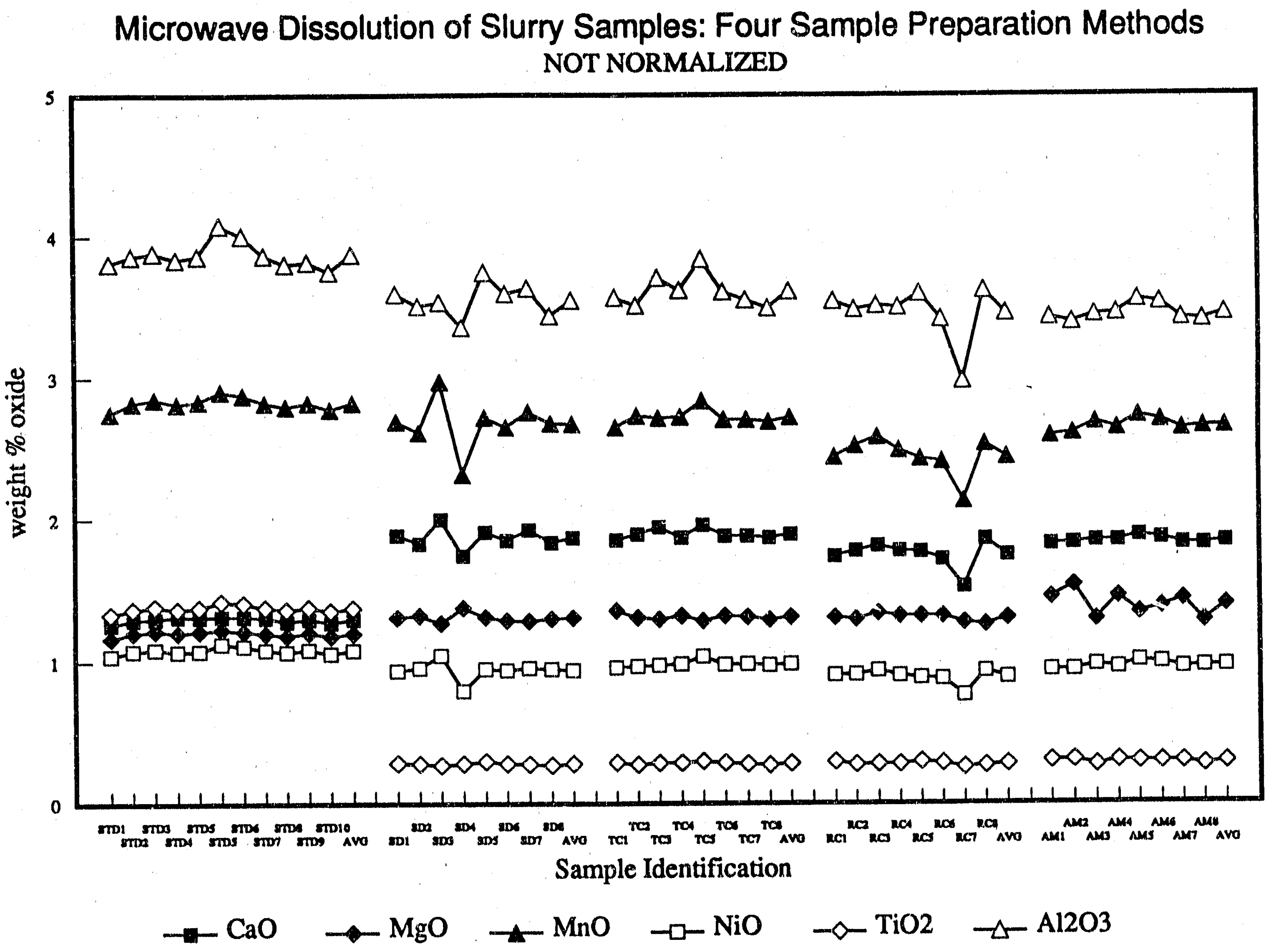
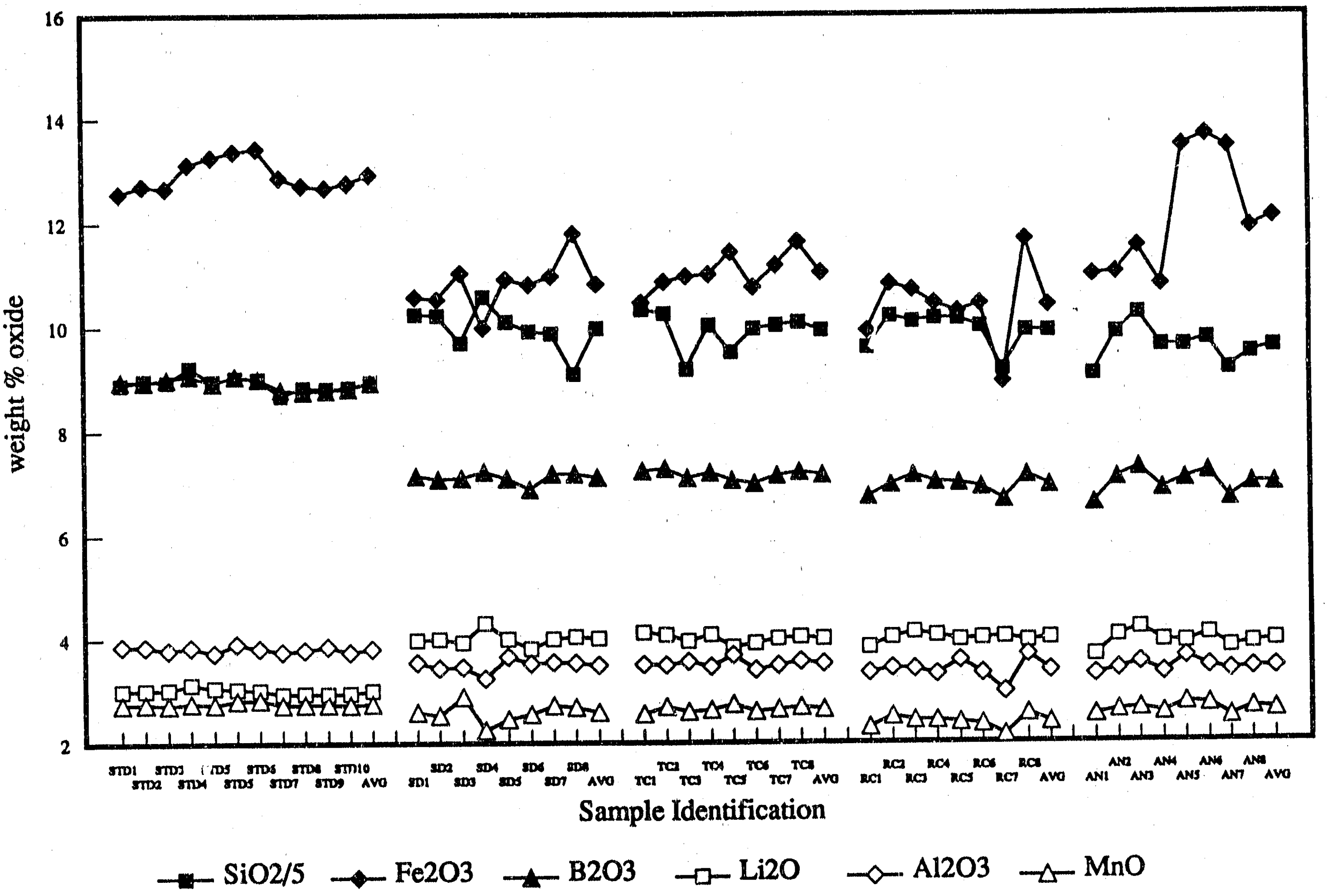

Microwave Dissolution of Slurry Samples: Four Sample Preparation Methods
NOT NORMALIZED
weight % oxide
0
1
2
3
4
5
STD1
STD2
STD3
STD4
STD5
STD5
STD6
STD7
STD8
STD9
STD10
AVG
SD1
SD2
SD3
SD4
SD5
SD6
SD7
SD8
AVG
TC1
TC2
TC3
TC4
TC5
TC6
TC7
TC8
AVG
RC1
RC2
RC3
RC4
RC5
RC6
RC7
RC8
AVG
AM1
AM2
AM3
AM4
AM5
AM6
AM7
AM8
AVG
Sample Identification
CaO
MgO
MnO
NiO
TiO2
Al2O3

FIGURE 3B



Na2O2 Fusion of Slurry Samples: Four Sample Preparation Methods

NOT NORMALIZED

END

DATE
FILMED

4 / 29 / 92