Development of a Frit 202 Analytic Standard for the Defense Waste Processing Facility

by

R. F. Schumacher

Westinghouse Savannah River Company

Savannah River Site

Aiken, South Carolina 29808

B. J. Hardy

J. F. Sproull

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DEVELOPMENT OF A FRIT 202 ANALYTIC
STANDARD FOR THE DEFENSE WASTE
PROCESSING FACILITY - (U)

R.F. SCHUMACHER, B.J. HARDY &
J.F. SPROULL

Westinghouse Savannah River Co.
Savannah River Site
Aiken, SC 29808

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Approvals & Technical Review

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R.F. Schumacher 5/7/97
R.F. Schumacher (Principal Author)

B.J. Hardy / RFS 5/7/97
B.J. Hardy, Co-Author

J.F. Sproull / RFS 5/7/97
James F. Sproull, Co-Author

John R. Harbour 5/7/97
John R. Harbour, Technical Review

David A. Crowley 5/8/97
D.A. Crowley,
Mgr. Glass Formulation & Melter Technology

E.W. Holtzscheiter 6/4/97
E.W. Holtzscheiter,
Mgr. Vitrification Technology Section

E. Holtzscheiter 6/4/97
Authorized Derivative Classifier

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ABSTRACT

During the qualification of Frit 202 samples for the "DWPF Cold Runs", the need for a reliable chemical frit standard became apparent. A standard was prepared by obtaining a quantity of Frit 202 and grinding into a fine powder. This material was homogenized as one slurry material volume, spray dried to prevent segregation, and hydraulically pressed into discs. These discs were fired and packaged into eleven sub-lots containing approximately 2,000 discs per sub-lot. A number of samples were obtained and analyzed by two analytic laboratories. The chemical analyses were carefully reviewed and evaluated by several statistical means. While there were several statistically significant variations between the sub-lots, it is believed that these variations are partially caused by the variability of the analytic method. These discs should provide a reliable standard for future chemical analyses of DWPF Frits similar in composition to Frit 202.

It is recommended that these discs be used as a standard material included with the representative frit sample to the independent chemical analysis laboratory, and the order of use of these standards be from sub-lot eleven to sub-lot four. It is further recommended that the NIST standard material (93a) be employed along with the 202 standard until confidence in the new standard is gained. The NIST standard should also be used when initial use of a new sub-lot is begun. This procedure should continue to the end of the DWPF program or such time as the chemical composition of the frit is extensively modified.

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DEVELOPMENT OF A FRIT 202 ANALYTIC STANDARD FOR THE DEFENSE WASTE PROCESSING FACILITY - (U)

INTRODUCTION

During the chemical evaluation of initial samples of DWPF Frit 202 for the "Cold Runs", it became apparent, that the establishment of a glass "standard" similar to Frit 202 would be an advantage to the DWPF program[1]. During these evaluations, a National Institute of Standards and Technology (NIST) standard borosilicate glass, NIST No. 93a, was employed to validate the chemical analyses of the DWPF frits. The major disadvantage of using NIST No. 93a as a standard is that it has only three of the five primary components of the DWPF frits and the proportions are significantly different from the composition of the DWPF Frit. After discussion with the DWPF technical staff, it was decided to attempt to develop a glass standard similar in composition to DWPF Frit 202.

An acceptable chemical analysis standard should meet the following general criteria: 1) the standard should have a chemical composition similar to the material to be analyzed, 2) the standard should have a similar physical matrix e.g. a glass, ceramic, metal, etc., 3) the standard should be stable with time, and 4) all the pieces in the standard should be of convenient size and essentially identical chemically. Meeting these requirements and proving that the requirements have been met, beyond a reasonable doubt, provides a rather daunting task.

SUMMARY

A small lot of DWPF Frit 202 was obtained from the frit vendor, carefully ground into a fine powder, slurried in one mixer, and spray dried into a spherical feed for pressing into small discs. The discs were then fired on a carefully controlled, continuous belt furnace. As the discs exited the furnace they were sampled and partitioned into eleven sub-lots (ca. 2000 discs/ sub-lot). After extensive chemical analyses by two different chemical laboratories and inspection of the resulting data it was concluded the material produced could be employed as a chemical reference standard. It is suggested that the order of use be from sub-lot 11 to sub-lot 4. The first three sub-lots (1, 2, and 3) are somewhat suspect and they should not be used. It is recommended that the NIST standard material (93a) should be included with the Frit 202 standard until confidence is gained in the new standard. The NIST standard should also be included when new sub-lot standards are employed, i.e. changing from sub-lot 11 to 10. Any person interested in obtaining samples of this analytic standard should contact Ray Schumacher, who will control the dispersion of samples.

EXPERIMENTAL

Obtaining Glass Frit

During the latter part of 1993 (September) approximately 850 pounds of Frit 202 were obtained from the DWPF frit vendor, Cataphote, Inc., (Jackson, MS). This material was left over after supplying the sixth lot of Frit 202 for the "Cold Runs".

The material was obtained from Cataphote in two 51 gallon metal topped fiber drums with lid and ring clamp seals. The drums contained a polyethylene drum liner with four pre-packaged desiccant packs per drum. The drums were shrink wrapped to a wooden pallet and shipped to SRS. This was conducted under Purchase Requisition D-96950 (PO#AA84373H).

While the material was not a part of Lot 6, it was melted during the same campaign and with the same quality assurance controls. Lot 6 (September 1993) of Frit 202 was analyzed by Corning Engineering Laboratory Services (CELS) under LIMS#12354. The analysis was verified with a National Institute of Standards Reference (NIST) Material 93a analyzed under the LIMS#12355. The results of these analyses are presented in Table 1. Review of Table 1, shows that the normalized analyzed composition of this material is extraordinarily close to the DWPF target specification (within 0.02 wt%). The analysis of the standard reference material NIST 93a is also very close to the NIST certified analysis (within 0.2 wt% SiO₂). In addition, inspection of this Table confirms the need for a better reference material for Frit 202. The general composition of the NIST material is somewhat different from Frit 202 and it does not contain lithia or magnesia. The official independent analysis for Lot 6 is presented at the end of this report in Appendix A. While it was a requirement of the purchase order that the composition of the supplied frit material meet the DWPF Frit 202 specification, it was serendipity that the normalized composition came as close to the target composition as it did (± 0.02 wt%).

Table 1
Chemical Analyses (CELS) Frit 202-Lot 6 and NIST 93a Standard

Majors	DWPF Specification (Wt%)	Lot 6 CELS #12354	Lot 6 Norm.to 98.8	NIST 93a Standard (Wt%)	NIST 93a CELS #12355
SiO ₂	77.0 $\pm$ 1.0	76.1	77.02	80.8	80.6
Na ₂ O	6.0 $\pm$ 0.5	5.92	5.99	3.98	3.97
Li ₂ O	7.0 $\pm$ 0.5	6.90	6.98		
MgO	2.0 $\pm$ 0.25	1.98	2.00	0.005	
B ₂ O ₃	8.0 $\pm$ 0.5	7.90	8.00	12.56	12.6
Total	100.0	98.8	100.00		
Minors					
Al ₂ O ₃	<1.89	0.34		2.28	2.27
Fe ₂ O ₃	<0.29	0.038		0.028	
MnO	<0.32	<.001			
NiO	<0.25	<.001			
Cr ₂ O ₃	<0.15	<.001			
PbO	<0.11	<.001			
TiO ₂	<0.25	0.012		0.014	
F	<0.05	0.007			
Cl	<0.05	0.014		0.060	
Others	<0.05			0.042	
K ₂ O				0.014	
CaO		0.04		0.01	
BaO		0.067			
Total Oxide		99.33		99.77	99.44
LOD @ 110°C<0.1		0.06			
LOI @ 600°C<0.1		0.22			

The two drums containing the 850 pounds of DWPF Frit 202 were received at SRS prior to being shipped to Ferro Corporation, Cleveland, Ohio for additional processing and fabrication of the discs.

Preparation of Glass Discs

In order to prepare a chemical glass standard, it is necessary to take into consideration several factors. First, the material should be mixed into a single homogeneous lot of material in a manner where any sample obtained for chemical analysis should have the same chemical composition. Second, the standard should have a structural matrix similar to the unknown. Third, the material should be stable such that it is not affected by its environment, e.g. humidity or time. Finally the standard should be of a convenient size and all pieces essentially identical on a chemical basis. Two bids were received to carry out this effort and prepare the reference standard. The first involved further grinding the frit into a homogeneous lot and pressing and firing the material into small discs. The other bid was to re-mix the frit powder, re-melt the powder in small lots and cast into shapes. It was felt that while the two approaches were quite different either process would probably have produced homogeneous standard samples. Procurement selected the initial process involving the manufacture of discs.

A Purchase Requisition (E13352) was prepared and a Purchase Order (AA84377A) was provided to Ferro Corporation to homogenize and fabricate a minimum of 250 lbs. of sintered glass discs.

The 850 lbs. of Frit 202 was received by the Electronic and Specialty Glass Department at Ferro Corporation and ground in four millings to an average 73% minus 325 mesh powder (-325M = 79.5%, 70.3%, 79.4%, and 63.3%). A portion of the 850 pounds was used to initially "clean" the grinding mill and was discarded. The remaining material was ground in four millings in a 52 - gallon high-alumina mill with 75kg of the Frit 202 in each milling. The grinding media was 204 kgs of 13/16 inch high alumina balls. Approximately 75 ml of alcohol was added as a milling aid and milling time was 7 to 8 hours. The product was designated F-202 PRF while at Ferro Corp. The four millings, (300 kgs), were blended by hand prior to shipment to Elan Technology, a Division of Mansol Industries, Inc., for the additional processing.

Upon arrival at Elan Technology, the entire 300 kgs of milled material was slurried in one large mixer to promote homogeneity of all the glass. The homogeneous slurry was then spray dried into small spherules with a 24 - foot spray dryer in about 25 minutes. An organic binder was added at a 4% level to hold the powder together before and after pressing and to prevent any type of particle segregation.

The spray dried powder was pressed into discs 1.13 inch in diameter and the run broken into 11 sub-lots. Pressing of the discs was accomplished with a ten - ton Denison Press. The firing of the discs was also completed at Elan Technology. The discs were fired on ceramic plates to a fired diameter of approximately one inch and a weight of approximately 6 grams. The belt speed in the furnace was 10 inches per minute and the maximum temperature was 1290°F, (699°C). The furnace was divided into eight zones ranging in temperature from 450 to 1290°F. The 11 sub-lots were fired in order and a total of approximately 20,850 discs were prepared by these procedures.

Table 3
Summary of Chemical Analyses from SLI and CELS
Sub Lots 1 to 11

Oxide Sub-lot	MgO wt%	Li2O wt%	B2O3 wt%	Na2O wt%	SiO2 wt%	Total wt%
NSL-1	2.02	6.90	7.65	5.59	77.24	99.40
2	2.02	6.95	7.67	5.67	77.08	99.39
3	2.03	7.07	7.61	5.73	76.96	99.40
4	2.00	7.07	7.66	5.71	76.96	99.40
5	2.01	7.00	7.67	5.74	77.00	99.43
6	1.98	7.01	7.60	5.68	77.18	99.44
7	1.99	7.01	7.61	5.70	77.12	99.43
8	1.99	7.01	7.66	5.76	77.02	99.43
9	2.01	7.04	7.67	5.71	77.01	99.44
10	1.99	7.00	7.71	5.67	77.07	99.44
11	2.00	6.99	7.65	5.68	77.12	99.45
AVG	2.00	7.00	7.65	5.70	77.07	99.42
Continuous	1.99	6.99	7.67	5.68	77.11	99.43
Powder	2.01	7.00	7.66	5.69	77.16	99.52
CELS-3	1.94	6.94	7.81	5.98	76.1	99.37
-5	1.98	6.97	7.77	5.96	76.3	99.60
-7	1.94	6.91	7.71	5.96	75.9	99.02
-9	1.96	6.97	7.73	5.97	76.3	99.59
-11	1.95	6.94	7.73	5.97	77.0	100.21
Avg.	1.95	6.95	7.75	5.97	76.32	99.56
High Value	2.03	7.07	7.81	5.98	77.24	100.21
Low Value	1.94	6.90	7.60	5.59	75.90	99.02
Range/Low	4.64%	2.46%	2.76%	6.98%	1.77%	1.20%

Table 4
CELS Analyses of Fired Discs from Sub-Lots 3,5,7,9, and 11

Notebook No.E57157-46

CELS No.	15052-94-2	-1	-3	-5	-4		
Sub-lot No.	3	5	7	9	11	Avg	Std. Dev.
<u>Oxide Deter.</u>							
Cr2O3	<.001	<.001	<.001	<.001	<.001		
NiO	<.001	<.001	<.001	<.001	<.001		
PbO	<.001	<.001	<.001	<.001	<.001		
K2O	0.05	0.04	0.04	0.04	0.04	0.042	0.004
Li2O	6.94	6.97	6.91	6.97	6.94	6.946	0.022
Na2O	5.98	5.96	5.96	5.97	5.97	5.968	0.007
Al2O3	0.44	0.47	0.46	0.46	0.47	0.46	0.011
B2O3	7.81	7.77	7.71	7.73	7.73	7.75	0.036
CaO	0.041	0.041	0.037	0.04	0.039	0.0396	0.001
Fe2O3	0.031	0.042	0.034	0.1	0.041	0.0496	0.026
MgO	1.94	1.98	1.94	1.96	1.95	1.954	0.015
MnO2	<.001	<.001	<.001	<.001	<.001		
SiO2	76.1	76.3	75.9	76.3	77	76.32	0.371
TiO2	0.011	0.013	0.008	0.011	0.011	0.0108	0.002
F	0.008	0.006	0.006	0.004	0.006	0.006	0.001
Cl	0.017	0.012	0.018	0.006	0.013	0.0132	0.004
Total	99.368	99.604	99.023	99.591	100.21		
Majors	98.77	98.98	98.42	98.93	99.59		
Sub-Total							
	Normalized	Data					
Li2O	7.03%	7.04%	7.02%	7.05%	6.97%		
Na2O	6.05%	6.02%	6.06%	6.03%	5.99%		
B2O3	7.91%	7.85%	7.83%	7.81%	7.76%		
MgO	1.96%	2.00%	1.97%	1.98%	1.96%		
SiO2	77.05%	77.09%	77.12%	77.13%	77.32%		
Total	100.00%	100.00%	100.00%	100.00%	100.00%		

DISCUSSION OF RESULTS

Inspection of the data indicates that the Spectrochemical Lab analyses of the 78 fired disc samples are very similar and the comparison to the unfired powder is usually within one standard deviation. This was interpreted to mean that the firing process did not have a noticeable influence on the composition of the discs. This would be expected at the low firing temperature of 1290°F (699°C). Comparing the discs obtained from each sub-lot to the discs obtained consecutively, indicated that the standard deviation of the consecutive samples was approximately one half the values for the discs from the sub-lots. Assuming that the consecutive discs had essentially the same composition, then approximately half of the standard deviation observed was due to the variability of the chemical analyses. See Table 5. While the variability of the sub lot data is slightly greater than the samples obtained consecutively, the data is very consistent. There appears to be no significant difference in the analyses of the sub-lots compared to the consecutively gathered samples.

Table 5
Comparison of Average and Standard Deviations for Sub Lot
Samples, Powder Samples, and Consecutive Samples

Sub Lot No		MgO Wt%	Li2O Wt%	B2O3 Wt%	Na2O Wt%	SiO2 Wt%	Total Wt%
#1	Avg.	2.02	6.90	7.65	5.59	77.24	99.40
	S.D.	0.03	0.07	0.08	0.07	0.21	
#2	Avg.	2.02	6.95	7.67	5.67	77.08	99.39
	S.D.	0.03	0.07	0.08	0.06	0.14	
#3	Avg.	2.03	7.07	7.61	5.73	76.96	99.40
	S.D.	0.06	0.07	0.05	0.13	0.22	
#4	Avg.	2.00	7.07	7.66	5.71	76.96	99.40
	S.D.	0.02	0.05	0.08	0.05	0.11	
#5	Avg.	1.98	7.01	7.60	5.68	77.18	99.44
	S.D.	0.03	0.04	0.08	0.04	0.08	
#6	Avg.	1.99	7.01	7.61	5.70	77.12	99.43
	S.D.	0.02	0.06	0.04	0.02	0.08	
#7	Avg.	1.99	7.01	7.61	5.70	77.12	99.43
	S.D.	0.02	0.06	0.04	0.02	0.08	
#8	Avg.	1.99	7.01	7.66	5.76	77.02	99.43
	S.D.	0.01	0.05	0.11	0.04	0.11	
#9	Avg.	2.01	7.04	7.67	5.71	77.01	99.44
	S.D.	0.01	0.03	0.05	0.03	0.08	
#10	Avg.	1.99	7.00	7.71	5.67	77.07	99.44
	S.D.	0.02	0.05	0.08	0.05	0.14	
#11	Avg.	2.00	6.99	7.65	5.68	77.12	99.45
	S.D.	0.02	0.08	0.08	0.03	0.16	
Powder Samples	Avg.	2.01	7.00	7.66	5.69	77.16	
	S.D.	0.02	0.06	0.03	0.05	0.11	
Consecutive Samples	Avg.	1.99	6.99	7.67	5.68	77.11	99.43
	S.D.	0.02	0.04	0.04	0.04	0.08	

A statistical analysis of the data was conducted by B.J. Hardy using the JMP software. The data obtained has been included in this report as appendices D-1 to D-3. Appendix D-1 plots show the estimators for the lot means, the 95% confidence levels for the lot means and the ratio of the between and within lot variances for the 11 lots. The tables give the F ratio for the between and within lot variance, the corresponding probability for F values greater than those obtained are shown. If the probability that F exceeds the calculated F ratio is less than 0.05 there is likely there is likely a significant difference in the compositions between lots. All components other than B₂O₃ show a significant variation between the lots.

Appendix D-2 presents similar plots and tables, except that lots 1, 2, and 3 have been removed. Using the same criterion only the Na₂O and SiO₂ show significant variations between the lots. It should be noted these differences are very small, about 0.1 wt% for SiO₂ and 0.05 wt% for Na₂O.

This is within the limits of the chemical analysis. Another way to state this is that a major portion of the variability in the analysis was due to variability in the chemical analysis compared to the variability between the sub-lots.

The final plots in Appendix D-3 show the correlation between the different components. High degrees of correlation are indicated in the plots whose 95% density ellipses are stretched in a diagonal. Uncorrelated variables possess nearly round density ellipses. Quantitatively the correlations which approach 1.0 are correlated and those which approach 0.0 are uncorrelated.

It should be noted that x-ray diffraction analysis indicated the presence of a small amount of lithium silicate, (Li_2SiO_5) and probably β -Quartz. This may be cause for some concern about the dissolution of the materials prior to the chemical analyses. However, the CELS analyses of the discs from the odd sub-lots did not show any evidence of any problems.

Acknowledgments

The authors wish to acknowledge the following people for their assistance on this project. Lee Young of Ferro Corporation who oversaw the manufacture and sampling of the discs.

References

1. R.F. Schumacher, "Review of DWPF Frit Procurement and Justification of Second Frit Vendor-(U)," WSRC-TR-93-069 Rev.0, February 1, 1993.

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Appendix A

Glass Sample: DWPF LIMS # 12354 - Lot 6, Frit 202 7/2/93
Lab I.D. No.: 15760-4, 1993

Oxide	Read1 Wt.%	Read2 Wt.%	Read3 Wt.%	Average Wt.%	Std.Dev. (n-1)	Method
Al ₂ O ₃	0.35	0.33		0.34	0.014	ICP
B ₂ O ₃	7.87	7.92		7.90	0.035	ICP
CaO	0.071	0.062		0.067	0.006	ICP
Cl-	0.013	0.015	0.014	0.014	0.001	ICP
Cr ₂ O ₃	<0.001	<0.001		<0.001	0.000	AA
F-	0.007	0.008	0.006	0.007	0.001	ICP
Fe ₂ O ₃	0.038	0.037		0.038	0.001	ICP
K ₂ O	0.044	0.043		0.04	0.001	ICP
Li ₂ O	6.91	6.89		6.90	0.014	ICP
MgO	1.98	1.97		1.98	0.007	ICP
MnO ₂	<0.001	<0.001		<0.001	0.000	ICP
Na ₂ O	5.92	5.92		5.92	0.000	ICP
NiO	<0.001	<0.001		<0.001	0.000	AA
PbO	<0.001	<0.001		<0.001	0.000	AA
SiO ₂	76.2	76.0		76.1	0.141	ICP
TiO ₂	0.012	0.012		0.012	0.000	ICP
LOI @600°C	0.20	0.17	0.30	0.22	0.021	LOI
Total				99.53		
LOD@ 110°C	0.05	0.02	0.11	0.06	0.021	LOD
COD	<580ug/g			0.022%		COF

ELECTRONIC & SPECIALTY GLASS
FRIT DIVISION
FERRO CORPORATION

4150 EAST 58TH STREET
P.O. BOX 6550
CLEVELAND, OHIO 44101
TELEPHONE (216) 641-8580
FACSIMILE (216) 641-8867

April 28, 1994

Ray Schumacher
Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29801-0001

Dear Ray:

Approximately 20800 pieces were made, weighing roughly 293 pounds. The product was divided into eleven sublots. The shipping date was May 1, 1994 and the carrier was Carolina Motor Freight

Copies of the actual analysis are included.

The following is a technical summary describing the process used to manufacture the discs.

1.) Milling

- performed at E&SG Dept., Frit Div., Ferro Corp.
- four mill turns
- 52 gallon unibody high alumina mill
- 75 kg of product (F 202) each turn
- 204 kg media load, high alumina 13/16" media
- 75ml of methanol alcohol added as a milling aid
- mill time was between 7 and 8 hours
- milling target was 68-74% minus 325 mesh
- actually the mills were 79.5%, 70.3%, 79.4% and 63.3%--for an average of 73.1%
- mill turns were blended by hand prior to shipping
- the product was designated F 202 PRF in E&SG records

2.) Spray Drying

- performed at Elan Technology a Division of Mansol Industries, Inc.
- the entire 300 Kgs of milled material were slurried together in one mixer, an excellent method of homogenizing
- 24 ft diameter spray dryer was used, spray drying time was about 25 minutes
- organic binder was added at 4% level

3.) Pressing

- performed at Elan Technology
- the green size was 1.13 inches in diameter
- the pressing was done on a ten ton Denison Press
- pressing force is not controlled, but green weight and the dimension parallel with the force applied are metered
- the ten continuous samples were taken roughly half way through the pressing campaign, two sets of samples were taken
- at this point the production run was broken into eleven sublots

4.) Firing

- performed at Elan Technology
- the firing schedule is included on figure 1
- the discs were fired on ceramic plates, discs were laid flat on these plates
- the fired/final size was nominally one inch
- each disc was roughly 6 grams
- the continuous samples were fired in the proper sequence
- the sublots were fired in order
- a total of 20850 discs were made

5.) Sampling

- a. powder samples
 - 1 Kg of powder was taken prior to spray drying and after milling - eight 40 gram samples were taken and sent for analysis
- b. regular production samples
 - taken after firing
 - seven or eight samples sent out for analysis
 - four of the seven samples were taken by Elan personnel from the beginning of the subplot as the subplot emerged from the furnace
 - the remainder of the sample set was taken randomly from the subplot by Ferro personnel
- c. continuous samples
 - taken prior to firing but after pressing
 - two sets of ten samples were taken directly as they came off the press
 - samples taken about half way through pressing run and were fired at the corresponding time

6.) Analysis

- Spectrochemical Laboratories, Inc. performed all

APP. B

- of the analyses
- all analysis were run consistently and were reported in order of completion
 - the powder samples were suppose to be run throughout the entire 'analysis run', but instead were run with the first eight sublots
 - all the analyses were done by plasma jet ICP
 - the actual analyses are included
 - table 1 is an initial analysis done by the author
 - the order of analysis can be found on the analysis, on the top right corner under "Our Lab#"

Table 1 is broken down by subplot and type of sample. Averages and standard deviations were calculated. Also a simple statistical tool, the chi squared test, was employed to determine if there were significant differences between subplot averages. Also, the same tool was used to determine if there were significant differences between the individual measurements. In both cases, no significant differences were found.

Thank you for your business and if you need any further information or have any questions, please do not hesitate to call me at (216) 641-8585 x6568.

Sincerely,

Lee J. Young

cc:\ L Dunning, T Whang, W. Cunningham, E. Stadnicar

TABLE 1

Sublot	Weight (Kgs)	Count	MgO (%)	Li2O (%)	B2O3 (%)	Na2O (%)	SiO2 (%)
1 AVG	11.6	1800	2.02	6.90	7.64	5.59	77.24
STD			0.03	0.07	0.06	0.07	0.19
2 AVG	11.9	1850	2.02	6.95	7.67	5.67	77.08
STD			0.03	0.07	0.08	0.06	0.13
3 AVG	12.5	1950	2.03	7.07	7.61	5.73	76.96
STD			0.05	0.07	0.05	0.12	0.2
4 AVG	10.7	1650	2.00	7.07	7.66	5.71	76.96
STD			0.02	0.04	0.07	0.05	0.11
5 AVG	8.7	1350	2.01	7.00	7.67	5.74	77.00
STD			0.03	0.03	0.06	0.07	0.11
6 AVG	12.3	1900	1.98	7.01	7.60	5.68	77.18
STD			0.02	0.04	0.07	0.04	0.07
7 AVG	10.1	1600	1.99	7.01	7.61	5.70	77.12
STD			0.01	0.06	0.04	0.02	0.07
8 AVG	17.5	2750	1.99	7.01	7.66	5.76	77.02
STD			0.01	0.05	0.1	0.03	0.1
9 AVG	12.4	1950	2.01	7.04	7.67	5.71	77.10
STD			0.01	0.03	0.04	0.03	0.25
10 AVG	12	1900	1.99	7.00	7.71	5.67	77.07
STD			0.02	0.04	0.08	0.05	0.13
11 AVG	13.5	2150	2.00	6.99	7.65	5.68	77.12
STD			0.02	0.08	0.08	0.03	0.14
continuous AVG		10	1.99	6.99	7.67	5.68	77.11
STD			0.02	0.04	0.04	0.03	0.07
powder AVG			2.02	7.00	7.67	5.69	77.14
STD			0.03	0.06	0.07	0.07	0.13
production AVG			2.00	7.00	7.65	5.70	77.08
STD			0.03	0.07	0.08	0.07	0.17
chi squared on avgs			0.001	0.003	0.001	0.011	0.001
chi squared on individ			0.037	0.059	0.06	0.075	0.029
results of chi squared tests			NS	NS	NS	NS	NS
TOTAL	133.2	20850					

NS = Not Significant

FIGURE 1

APP. B

To: Ferro
Attn: Lee J. Young
From: Walter B. Reed
Fax: 216-641-8857
Date: 5/3/94
Pages: 1

Ref: Our phone conversation yesterday.

Below is the profile for firing part #310004.

Furnace #3

Zone 1	450°F
Zone 2	550°F
Zone 3	650°F
Zone 4	800°F
Zone 5	1000°F
Zone 6	1150°F
Zone 7	1270°F
Zone 8	1290°F

Belt speed 10 inches per minute
Raw pressing 0.260 to 0.265

Best Regards,

A handwritten signature in dark ink, appearing to read 'W.B. Reed'.

140 Little Street, Belleville, NJ 07109-3295 • Tel: (201) 759-8600 • Fax: (201) 751-2506
Division of Mansel Industries, Inc.

Spectrochemical Laboratories, Inc.

APP. B

Telephone: 412-371-2345

FAX: 412-371-0463

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3452

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT RESULTS IN WEIGHT % F202 PRF Sublot 1

OUR LAB	-1	-2	-3	-4	-5
#D3452					
YOUR ID	-A	-B	-C	-D	-E
MgO	1.98	2.02	2.01	2.03	1.99
Li ₂ O	6.81	6.94	6.91	6.97	6.83
B ₂ O ₃	7.52	7.71	7.71	7.62	7.65
Na ₂ O	5.53	5.61	5.56	5.69	5.52
SiO ₂	77.55	77.12	77.19	77.07	77.41

OUR LAB	-6	-7	-8
#D3452			
YOUR ID	-F	-G	Powder Sample 1
MgO	2.07	2.03	1.99
Li ₂ O	6.99	6.85	6.90
B ₂ O ₃	7.67	7.61	7.67
Na ₂ O	5.70	5.55	5.57
SiO ₂	76.96	77.36	77.40

SPECTROCHEMICAL LABORATORIES, Inc.

Steven H. Byers

Spectrochemical Laboratories, Inc.

App. B

Telephone: 412-371-2345

FAX: 412-371-0463

8350 FRANKSTOWN AVENUE

PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3453

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT RESULTS IN WEIGHT % F202 PRF Sublot 2

OUR LAB	-1	-2	-3	-4	-5
#D3453					
YOUR ID	-A	-B	-C	-D	-E
MgO	2.06	1.99	1.98	2.02	2.00
Li ₂ O	7.11	6.93	6.95	6.91	6.91
B ₂ O ₃	7.55	7.77	7.70	7.70	7.61
Na ₂ O	5.79	5.66	5.65	5.68	5.65
SiO ₂	76.85	77.03	77.12	77.08	77.23

OUR LAB	-6	-7	-8
#D3453			
YOUR ID	-F	-G	Powder Sample 2
MgO	2.04	2.05	2.05
Li ₂ O	6.91	6.95	6.96
B ₂ O ₃	7.59	7.75	7.69
Na ₂ O	5.59	5.64	5.66
SiO ₂	77.27	77.01	77.15

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Steven H. Byers
Steven H. Byers

Spectrochemical Laboratories, Inc.

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Telephone: 412-371-2345

FAX: 412-371-0463

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3454

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT RESULTS IN WEIGHT % F202 PRF Sublot 3

OUR LAB #D3454 YOUR ID	-1	-2	-3	-4	-5
	-A	-B	-C	-D	-E
MgO	2.09	2.02	1.97	2.08	2.10
Li ₂ O	7.03	7.05	7.03	7.05	7.23
B ₂ O ₃	7.58	7.68	7.60	7.60	7.55
Na ₂ O	5.83	5.60	5.55	5.71	5.94
SiO ₂	76.84	77.06	77.25	76.96	76.56

OUR LAB #D3454 YOUR ID	-6	-7	-8
	-F	-G	Powder Sample 3
MgO	1.96	2.02	1.99
Li ₂ O	7.02	7.06	7.05
B ₂ O ₃	7.68	7.58	7.72
Na ₂ O	5.69	5.77	5.80
SiO ₂	77.07	76.97	76.95

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Steven H. Byers

Spectrochemical Laboratories, Inc.

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Telephone: 412-371-2345
FAX: 412-371-0463

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3455

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT

RESULTS IN WEIGHT %

F202 PRF Sublot 4

OUR LAB	-1	-2	-3	-4	-5
#D3455					
YOUR ID	-A	-B	-C	-D	-E
MgO	2.02	2.00	2.01	2.04	1.99
Li ₂ O	7.05	7.05	7.03	7.02	7.13
B ₂ O ₃	7.60	7.63	7.72	7.53	7.69
Na ₂ O	5.72	5.79	5.67	5.62	5.72
SiO ₂	77.00	76.93	76.97	77.19	76.88

OUR LAB	-6	-7	-8
#D3455			
YOUR ID	-F	-G	Powder Sample 4
MgO	2.00	1.96	2.04
Li ₂ O	7.13	7.08	7.08
B ₂ O ₃	7.75	7.70	7.61
Na ₂ O	5.70	5.74	5.63
SiO ₂	76.83	76.94	77.16

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Steven H. Byers
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Telephone: 412-371-2345
FAX: 412-371-0463

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab# D3456

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT

RESULTS IN WEIGHT %

F202 PRF Sublot 5

OUR LAB	-1	-2	-3	-4	-5
#D3456					
YOUR ID	-A	-B	-C	-D	-E
MgO	2.01	2.05	1.99	2.01	2.03
Li ₂ O	7.02	7.06	6.97	7.02	6.96
B ₂ O ₃	7.55	7.66	7.69	7.68	7.73
Na ₂ O	5.73	5.85	5.79	5.68	5.65
SiO ₂	77.11	76.80	76.99	77.04	77.06

OUR LAB	-6	-7	-8
#D3456			
YOUR ID	-F	-G	Powder Sample 5
MgO	2.01	1.96	2.06
Li ₂ O	6.97	7.02	6.96
B ₂ O ₃	7.65	7.76	7.75
Na ₂ O	5.66	5.80	5.72
SiO ₂	77.13	76.90	77.03

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FAX: 412-371-0463

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3457

Attn: Mr. Lee Young

ANALYSIS REPORT

RESULTS IN WEIGHT % F202 PRF Sublot 6

OUR LAB	-1	-2	-3	-4	-5
#D3457					
YOUR ID	-A	-B	-C	-D	-E
MgO	2.01	1.96	2.00	1.99	1.94
Li ₂ O	6.95	7.01	7.00	6.99	7.06
B ₂ O ₃	7.71	7.61	7.58	7.50	7.64
Na ₂ O	5.72	5.72	5.65	5.66	5.62
SiO ₂	77.05	77.17	77.22	77.30	77.16

OUR LAB	-6	-7	-8	
#D3457				
YOUR ID	-F	-G		Powder Sample 6
MgO	1.97	2.00	2.00	
Li ₂ O	6.98	7.07	7.09	
B ₂ O ₃	7.65	7.50	7.54	
Na ₂ O	5.69	5.70	5.72	
SiO ₂	77.16	77.17	77.17	

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FAX: 412-371-0463

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3458

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT

RESULTS IN WEIGHT %

F202 PRF Sublot 7

OUR LAB	-1	-2	-3	-4	-5
#D3458					
YOUR ID	-A	-B	-C	-D	-E
MgO	1.97	2.00	1.99	2.00	1.97
Li ₂ O	6.88	7.02	7.04	7.08	6.98
B ₂ O ₃	7.63	7.60	7.68	7.60	7.58
Na ₂ O	5.73	5.71	5.69	5.68	5.74
SiO ₂	77.24	77.07	77.04	77.08	77.17

OUR LAB	-6	-7	-8
#D3458			
YOUR ID	-F	-G	Powder Sample 7

MgO	2.00	2.01	2.00
Li ₂ O	7.01	7.05	6.99
B ₂ O ₃	7.56	7.62	7.65
Na ₂ O	5.68	5.70	5.66
SiO ₂	77.19	77.05	77.21

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Steven H. Byers
Steven H. Byers

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APP. B

Telephone: 412-371-2345
FAX: 412-371-0463

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3459

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT

RESULTS IN WEIGHT %

F202 PRF Sublot 8

OUR LAB	-1	-2	-3	-4	-5
#D3459					
YOUR ID	-A	-B	-C	-D	-E
MgO	1.97	1.99	2.00	1.98	2.01
Li ₂ O	6.99	7.00	7.01	7.07	7.05
B ₂ O ₃	7.75	7.81	7.50	7.55	7.59
Na ₂ O	5.80	5.76	5.80	5.72	5.71
SiO ₂	76.91	76.86	77.12	77.10	77.08

OUR LAB	-6	-7	-8	-9	
#D3459					
YOUR ID	-F	-G	H		Powder Sample 8
MgO	1.97	1.99	2.00	2.01	
Li ₂ O	6.96	7.05	6.91	6.95	
B ₂ O ₃	7.60	7.75	7.70	7.76	
Na ₂ O	5.78	5.74	5.80	5.77	
SiO ₂	77.12	76.91	77.02	77.05	

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Steven Byers
Steven H. Byers

Spectrochemical Laboratories, Inc.

APP. B

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FAX: 412-371-0463

8350 FRANKSTOWN AVENUE

PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3460

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT RESULTS IN WEIGHT % F202 PRF Sublot 9

OUR LAB	-1	-2	-3	-4	-5
#D3460					
YOUR ID	-A	-B	-C	-D	-E
MgO	2.01	2.01	2.02	2.02	2.00
Li ₂ O	7.08	7.07	7.04	7.05	7.00
B ₂ O ₃	7.71	7.67	7.72	7.62	7.71
Na ₂ O	5.74	5.72	5.73	5.72	5.68
SiO ₂	76.90	76.96	76.93	77.03	77.06

OUR LAB	-6	-7
#D		
YOUR ID	-F	-G
MgO	1.99	2.02
Li ₂ O	7.02	7.03
B ₂ O ₃	7.66	7.60
Na ₂ O	5.70	5.66
SiO ₂	77.08	77.13

SPECTROCHEMICAL LABORATORIES, Inc.

Steven H. Byers
Steven H. Byers

We warrant that the analysis was performed in accordance with the methods and standards of the American Society for Testing and Materials (ASTM) and that the results are accurate and reliable within the limits of the methods used.

Spectrochemical Laboratories, Inc.

App. B

Telephone: 412-371-2345

FAX: 412-371-0463

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab#D3461

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT

RESULTS IN WEIGHT %

F202 PRF Sublot 10

OUR LAB	-1	-2	-3	-4	-5
#D3461					
YOUR ID	-A	-B	-C	-D	-E
MgO	1.98	1.99	1.97	2.01	1.98
Li ₂ O	6.95	6.97	6.95	7.06	7.01
B ₂ O ₃	7.80	7.57	7.70	7.77	7.68
Na ₂ O	5.67	5.72	5.68	5.72	5.56
SiO ₂	77.05	77.20	77.13	76.87	77.21

OUR LAB	-6	-7
#D3461		
YOUR ID	-F	-G
MgO	1.98	2.03
Li ₂ O	7.04	7.05
B ₂ O ₃	7.66	7.78
Na ₂ O	5.67	5.69
SiO ₂	77.10	76.90

SPECTROCHEMICAL LABORATORIES, Inc.

Steven H. Byers
Steven H. Byers

Spectrochemical Laboratories, Inc.

App. B

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FAX: 412-371-0463

8350 FRANKSTOWN AVENUE

PITTSBURGH, PA. 15221

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

April 25, 1994
Sample recd. 4-5-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab #D3462

ANALYSIS REPORT RESULTS IN WEIGHT % F202 PRF Sublot 11

OUR LAB	-1	-2	-3	-4	-5
#D3462					
YOUR ID	-A	-B	-C	-D	-E
MgO	1.95	2.02	2.00	2.01	1.99
Li ₂ O	6.82	6.95	7.02	7.04	7.04
B ₂ O ₃	7.60	7.67	7.71	7.51	7.63
Na ₂ O	5.64	5.69	5.69	5.73	5.68
SiO ₂	77.45	77.11	77.03	77.15	77.11

OUR LAB	-6	-7
#D3462		
YOUR ID	-F	-G
MgO	2.01	1.99
Li ₂ O	7.06	7.01
B ₂ O ₃	7.71	7.75
Na ₂ O	5.66	5.70
SiO ₂	77.00	77.00

SPECTROCHEMICAL LABORATORIES, Inc.

Steven H. Byers

Steven H. Byers

Spectrochemical Laboratories, Inc.

App. B

Telephone: 412-371-2345

FAX: 412-371-0463

8350 FRANKSTOWN AVENUE

PITTSBURGH, PA. 15221

April 25, 1994
Sample recd. 4-8-94
Your Req.# AR 764
Your Charge# EG59273
Our Lab #D3522

Ferro Corporation
4150 East 56th Street
P.O. Box 6550
Cleveland, Ohio 44101

Attn: Mr. Lee Young

ANALYSIS REPORT RESULTS IN WEIGHT %

F202 PRF

OUR LAB	-1	-2	-3	-4	-5
#D3522					
YOUR ID	-A	-B	-C	-D	-E
MgO	1.97	2.01	2.00	1.98	1.98
Li ₂ O	6.96	6.95	6.97	6.96	6.98
B ₂ O ₃	7.66	7.65	7.71	7.64	7.70
Na ₂ O	5.63	5.62	5.66	5.73	5.68
SiO ₂	77.21	77.20	77.09	77.12	77.09
OUR LAB	-6	-7	-8	-9	-10
#D3522					
YOUR ID	-F	-G	-H	-I	-J
MgO	1.98	2.01	1.99	2.00	2.02
Li ₂ O	6.96	7.02	6.97	7.03	7.08
B ₂ O ₃	7.66	7.65	7.63	7.74	7.63
Na ₂ O	5.67	5.73	5.70	5.67	5.69
SiO ₂	77.18	77.03	77.15	77.00	77.02

SPECTROCHEMICAL LABORATORIES, Inc.

Steven H. Byers
Steven H. Byers

05 11 94 15 0.40000
Corning Incorporated
CELS—Laboratory Services
Technology Sales & Licensing
HP ME 03 078 G6
Corning, New York 14831
800-235-2357
607-974-6601
fax 607-974-6522

APP. C

Page 1 of 6

August 11, 1994

Mr. James F. Sproull
Westinghouse Savannah River Co.
Building 704-35S
P.O. Box 616
Aiken, SC 29808

CELS Client No.: 11988-048
Date Received : 22-JUL-94
Date Reported : 09-AUG-94
Reviewed &
Approved by : *Linda P. Adams*

CORNING

Copy to: CELS File

Linda P. Adams
Technical Sales
Engineer

SAMPLE IDENTIFICATION:

Sample 1: Frit 202 Disk; 20139; E57157-46-5
Sample 2: Frit 202 Disk; 20140; E57157-46-3
Sample 3: Frit 202 Disk; 20141; E57157-46-7
Sample 4: Frit 202 Disk; 20142; E57157-46-11
Sample 5: Frit 202 Disk; 20143; E57157-46-9
Sample 6: Std.; NIST 93A

ANALYSIS REPORTED:

Exhibit

A Quantitative Chemical Analysis
B Semi-Quantitative Spectrographic Analysis

COMMENTS:

Client Purchase Order No. AA07196N

Direct questions regarding this report to Linda Adams, CELS office.

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LABORATORY ANALYSIS REPORT

CELS Client No.: 11988-048

Exhibit A: Quantitative Chemical AnalysisSample Description:

Sample 1: Frit 202 Disk; 20139; E57157-46-5

Sample 2: Frit 202 Disk; 20140; E57157-46-3

Sample 3: Frit 202 Disk; 20141; E57157-46-7

Lab I.D. No.: 15052-94

<u>Determination</u>	<u>Results (Wt.%)</u>			<u>Test Method</u>
	<u>Sample 1</u>	<u>Sample 2</u>	<u>Sample 3</u>	
Cr ₂ O ₃	<0.001	<0.001	<0.001	AAS
NiO	<0.001	<0.001	<0.001	AAS
PbO	<0.001	<0.001	<0.001	AAS
K ₂ O	0.04	0.05	0.04	FES
Li ₂ O	6.97	6.94	6.91	FES
Na ₂ O	5.96	5.98	5.96	FES
Al ₂ O ₃	0.47	0.44	0.46	PLS
B ₂ O ₃	7.77	7.81	7.71	PLS
CaO	0.041	0.041	0.037	PLS
Fe ₂ O ₃	0.042	0.031	0.034	PLS
MgO	1.98	1.94	1.94	PLS
MnO ₂	<.001	<.001	<.001	PLS
SiO ₂	76.3	76.1	75.9	PLS
TiO ₂	0.013	0.011	0.008	PLS
F	0.006	0.008	0.006	PYRO
Cl-	0.012	0.017	0.018	COLOR
TOTALS				
	99.60	99.37	99.02	

LEGEND:

AAS: Atomic Absorption Spectroscopy
FES: Flame Emission Spectroscopy
PLS: Plasma/Emission Spectroscopy

PYRO: Pyrohydrolysis
COLOR: Colorimetry

APP. C

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LABORATORY ANALYSIS REPORT

CELS Client No.: 11988-048

Exhibit A: Quantitative Chemical AnalysisSample Description:

Sample 4: Frit 202 Disk; 20142; E57157-46-11

Sample 5: Frit 202 Disk; 20143; E57157-46-9

Sample 6: Std.; NIST 93A

Lab I.D. No.: 15052-94

<u>Determination</u>	<u>Results (Wt.%)</u>			<u>Test Method</u>
	<u>Sample 4</u>	<u>Sample 5</u>	<u>Sample 6</u>	
Cr ₂ O ₃	<0.001	<0.001	-	AAS
NiO	<0.001	<0.001	-	AAS
PbO	<0.001	<0.001	-	AAS
K ₂ O	0.04	0.04	-	FES
Li ₂ O	6.94	6.97	-	FES
Na ₂ O	5.97	5.97	4.00	3.98 FES
Al ₂ O ₃	0.47	0.46	2.26	2.28 PLS
B ₂ O ₃	7.73	7.73	12.6	12.56 PLS
CaO	0.039	0.040	-	PLS
Fe ₂ O ₃	0.041	0.10	-	PLS
MgO	1.95	1.96	-	PLS
MnO ₂	<.001	<.001	-	PLS
SiO ₂	77.0	76.3	80.7	80.8 PLS
TiO ₂	0.011	0.011	-	PLS
F	0.006	0.004	-	PYRO
Cl-	0.013	0.006	-	COLOR
TOTALS				
	100.21	99.59	99.56	

LEGEND:

AAS: Atomic Absorption Spectroscopy
 FES: Flame Emission Spectroscopy
 PLS: Plasma/Emission Spectroscopy

PYRO: Pyrohydrolysis
 COLOR: Colorimetry

Wt% MgO By Sublot **Oneway Anova****Summary of Fit**

RSquare 0.256541
 RSquare Adj 0.145577
 Root Mean Square Error 0.028073
 Mean of Response 2.003718
 Observations (or Sum Wgts) 78

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	10	0.01822001	0.001822	2.3119
Error	67	0.05280179	0.000788	Prob>F
C Total	77	0.07102179	0.000922	0.0211

Means for Oneway Anova

Level	Number	Mean	Std Error
1	7	2.01857	0.01061
2	7	2.02000	0.01061
3	7	2.03429	0.01061
4	7	2.00286	0.01061
5	7	2.00857	0.01061
6	7	1.98143	0.01061
7	7	1.99143	0.01061
8	8	1.98875	0.00993
9	7	2.01000	0.01061
10	7	1.99143	0.01061
11	7	1.99571	0.01061

Std Error uses a pooled estimate of error variance

Wt% LiO2 By Sublot

APPENDIX D-1

Oneway Anova

Summary of Fit

RSquare 0.416349
RSquare Adj 0.329236
Root Mean Square Error 0.058507
Mean of Response 7.004744
Observations (or Sum Wgts) 78

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	10	0.16360201	0.016360	4.7795
Error	67	0.22934286	0.003423	Prob>F
C Total	77	0.39294487	0.005103	<.0001

Means for Oneway Anova

Level	Number	Mean	Std Error
1	7	6.90000	0.02211
2	7	6.95286	0.02211
3	7	7.06714	0.02211
4	7	7.07000	0.02211
5	7	7.00286	0.02211
6	7	7.00857	0.02211
7	7	7.00857	0.02211
8	8	7.00500	0.02069
9	7	7.04143	0.02211
10	7	7.00429	0.02211
11	7	6.99143	0.02211

Std Error uses a pooled estimate of error variance

Wt% B2O3 By Sublot

APPENDIX D-1

Oneway Anova

Summary of Fit

RSquare 0.163654
 RSquare Adj 0.038827
 Root Mean Square Error 0.075631
 Mean of Response 7.650897
 Observations (or Sum Wgts) 78

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	10	0.07499254	0.007499	1.3110	
Error	67	0.38324464	0.005720		
C Total	77	0.45823718	0.005951	0.2428	

Means for Oneway Anova

Level	Number	Mean	Std Error
1	7	7.65000	0.02859
2	7	7.66714	0.02859
3	7	7.61000	0.02859
4	7	7.66000	0.02859
5	7	7.67429	0.02859
6	7	7.59857	0.02859
7	7	7.61000	0.02859
8	8	7.65625	0.02674
9	7	7.67000	0.02859
10	7	7.70857	0.02859
11	7	7.65429	0.02859

Std Error uses a pooled estimate of error variance

Wt% Na2O By Sublot

APPENDIX D-

Oneway Anova

Summary of Fit

RSquare	0.350285
RSquare Adj	0.253312
Root Mean Square Error	0.062953
Mean of Response	5.695897
Observations (or Sum Wgts)	78

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	10	0.14315682	0.014316	3.6122
Error	67	0.26553036	0.003963	Prob>F
C Total	77	0.40868718	0.005308	0.0007

Means for Oneway Anova

Level	Number	Mean	Std Error
1	7	5.59429	0.02379
2	7	5.66571	0.02379
3	7	5.72714	0.02379
4	7	5.70857	0.02379
5	7	5.73714	0.02379
6	7	5.68000	0.02379
7	7	5.70429	0.02379
8	8	5.76375	0.02226
9	7	5.70714	0.02379
10	7	5.67286	0.02379
11	7	5.68429	0.02379

Std Error uses a pooled estimate of error variance

Wt% SiO2 By Sublot

Oneway Anova

Summary of Fit

RSquare	0.302921
RSquare Adj	0.19888
Root Mean Square Error	0.138148
Mean of Response	77.06821
Observations (or Sum Wgts)	78

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	10	0.5556630	0.055566	2.9115
Error	67	1.2786857	0.019085	Prob>F
C Total	77	1.8343487	0.023823	0.0044

Means for Oneway Anova

Level	Number	Mean	Std Error
1	7	77.2371	0.05222
2	7	77.0843	0.05222
3	7	76.9586	0.05222
4	7	76.9629	0.05222
5	7	77.0043	0.05222
6	7	77.1757	0.05222
7	7	77.1200	0.05222
8	8	77.0150	0.04884
9	7	77.0129	0.05222
10	7	77.0657	0.05222
11	7	77.1214	0.05222

Std Error uses a pooled estimate of error variance

Wt% MgO By Sublot **Oneway Anova****Summary of Fit**

RSquare 0.184386
 RSquare Adj 0.067869
 Root Mean Square Error 0.021259
 Mean of Response 1.99614
 Observations (or Sum Wgts) 57

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	0.00500623	0.000715	1.5825
Error	49	0.02214464	0.000452	Prob>F
C Total	56	0.02715088	0.000485	0.1629

Means for Oneway Anova

Level	Number	Mean	Std Error
4	7	2.00286	0.00804
5	7	2.00857	0.00804
6	7	1.98143	0.00804
7	7	1.99143	0.00804
8	8	1.98875	0.00752
9	7	2.01000	0.00804
10	7	1.99143	0.00804
11	7	1.99571	0.00804

Std Error uses a pooled estimate of error variance

Wt% LiO2 By Sublot **Oneway Anova****Summary of Fit**

RSquare 0.195298
 RSquare Adj 0.080341
 Root Mean Square Error 0.052733
 Mean of Response 7.016316
 Observations (or Sum Wgts) 57

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	0.03306917	0.004724	1.6989
Error	49	0.13625714	0.002781	Prob>F
C Total	56	0.16932632	0.003024	0.1313

Means for Oneway Anova

Level	Number	Mean	Std Error
4	7	7.07000	0.01993
5	7	7.00286	0.01993
6	7	7.00857	0.01993
7	7	7.00857	0.01993
8	8	7.00500	0.01864
9	7	7.04143	0.01993
10	7	7.00429	0.01993
11	7	6.99143	0.01993

Std Error uses a pooled estimate of error variance

Wt% B2O3 By Sublot

APPENDIX D-2

Oneway Anova

Summary of Fit

RSquare	0.174727
RSquare Adj	0.056831
Root Mean Square Error	0.076599
Mean of Response	7.654035
Observations (or Sum Wgts)	57

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	0.06087014	0.008696	1.4820
Error	49	0.28750179	0.005867	Prob>F
C Total	56	0.34837193	0.006221	0.1957

Means for Oneway Anova

Level	Number	Mean	Std Error
4	7	7.66000	0.02895
5	7	7.67429	0.02895
6	7	7.59857	0.02895
7	7	7.61000	0.02895
8	8	7.65625	0.02708
9	7	7.67000	0.02895
10	7	7.70857	0.02895
11	7	7.65429	0.02895

Std Error uses a pooled estimate of error variance

Wt% Na2O By Sublot

APPENDIX D-2

Oneway Anova

Summary of Fit

RSquare	0.323034
RSquare Adj	0.226325
Root Mean Square Error	0.045769
Mean of Response	5.708246
Observations (or Sum Wgts)	57

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	0.04897992	0.006997	3.3403
Error	49	0.10264464	0.002095	Prob>F
C Total	56	0.15162456	0.002708	0.0055

Means for Oneway Anova

Level	Number	Mean	Std Error
4	7	5.70857	0.01730
5	7	5.73714	0.01730
6	7	5.68000	0.01730
7	7	5.70429	0.01730
8	8	5.76375	0.01618
9	7	5.70714	0.01730
10	7	5.67286	0.01730
11	7	5.68429	0.01730

Std Error uses a pooled estimate of error variance

Wt% SiO2 By Sublot

Oneway Anova

Summary of Fit

RSquare	0.30149
RSquare Adj	0.201703
Root Mean Square Error	0.111948
Mean of Response	77.05895
Observations (or Sum Wgts)	57

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	0.26505113	0.037864	3.0213
Error	49	0.61408571	0.012532	Prob>F
C Total	56	0.87913684	0.015699	0.0101

Means for Oneway Anova

Level	Number	Mean	Std Error
4	7	76.9629	0.04231
5	7	77.0043	0.04231
6	7	77.1757	0.04231
7	7	77.1200	0.04231
8	8	77.0150	0.03958
9	7	77.0129	0.04231
10	7	77.0657	0.04231
11	7	77.1214	0.04231

Std Error uses a pooled estimate of error variance

Correlations

Variable	Wt% MgO	Wt% LiO2	Wt% B2O3	Wt% Na2O	Wt% SiO2
Wt% MgO	1.0000	0.2528	-0.1406	0.2247	-0.4277
Wt% LiO2	0.2528	1.0000	-0.0625	0.5221	-0.7308
Wt% B2O3	-0.1406	-0.0625	1.0000	-0.0453	-0.3994
Wt% Na2O	0.2247	0.5221	-0.0453	1.0000	-0.7373
Wt% SiO2	-0.4277	-0.7308	-0.3994	-0.7373	1.0000

Scatterplot Matrix