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BLAST FURNACE SLAG SL-1: ITS PREPARATION FOR USE AS A CERTIFIED REFERENCE MATERIAL

G.L. Mason and W.S. Bowman



Laboratoire d' / Elliot Lake / Laboratory
Mines Branch - Direction des mines

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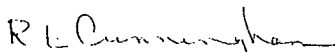
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FOREWORD

The work described in this report contributes to the Canadian Certified Reference Materials Project (CCRMP). The CCRMP in turn contributes to the Utilization Activity (Quality Control sub-Activity) of CANMET's Minerals Research Program by producing mineralogical and metallurgical reference materials (RM's) for use in industrial, commercial and government laboratories in Canada.

The CCRMP was initiated in the early seventies in response to a demand from such laboratories for RM's that were not previously available. Many laboratories now work on their own behalf by willingly contributing analytical information which is ultimately used in the CCRMP to certify RM's.

Now that a relatively large number of reference ores and related materials have been made available, they are being used in a "feed-back" fashion to critically assess analytical methods that are essential for quality-control and research in Canadian enterprises.

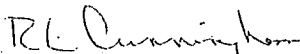

R.L. Cunningham,
Chief

AVANT-PROPOS

Le travail qui est décrit dans le présent rapport apporte une contribution au Programme canadien des matériaux de référence certifiés (CCRMP). De son côté, le CCRMP collabore aux travaux de l'Activité d'utilisation (la sous-activité de surveillance de la qualité) du Programme de recherche sur les minéraux de CANMET en normalisant des matériaux minéralogiques et métallurgiques pour les différents laboratoires industriels, commerciaux et gouvernementaux au Canada.

Le CCRMP a été créé au début des années '70 pour répondre à la demande formulée par les différents laboratoires qui voulaient de tels matériaux de référence qui n'étaient pas disponibles auparavant. Ainsi, plusieurs laboratoires effectuent maintenant des travaux analytiques et par la suite lèguent volontairement les informations nécessaires au CCRMP pour certifier des matériaux de référence.

Maintenant qu'une quantité relativement abondante de minerais de référence et apparentés sont disponibles, on les utilise rétro-activement afin d'évaluer les méthodes analytiques employées par les compagnies canadiennes pour contrôler la qualité et faire de la recherche.


R.L. Cunningham,
Chef

BLAST FURNACE SLAG SL-1: ITS PREPARATION
FOR USE AS A CERTIFIED REFERENCE MATERIAL

by

G.L. Mason and W.S. Bowman

SYNOPSIS

As a facet of the Canadian Certified Reference Materials Project, a Canadian blast furnace slag has been prepared as a compositional reference material.

Twenty-one laboratories participated in the interlaboratory certification program by providing analytical results for up to thirteen constituents on each of two bottles of the slag. A statistical treatment of the data yielded recommended values for six of these constituents: CaO - 37.5%, SiO₂ - 35.7%, MgO - 12.3%, Al₂O₃ - 9.6%, Fe (expressed as FeO) - 0.92%, and S - 1.26%. Four others were assigned provisional values: TiO₂ - 0.38%, MnO - 0.86%, Na₂O - 0.39%, and K₂O - 0.51%. The remainder are given for information only: P₂O₅ - 0.019%, V₂O₅ - 0.004%, and Cr₂O₃ - 0.009%.

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INTRODUCTION

This report describes the preparation and certification of a blast furnace slag, SL-1, for use as a certified reference material. The work is another facet of the Canadian Certified Reference Materials Project (CCRMP) to certify materials that have potential value in conventional analytical or earth sciences laboratories. Certified reference materials (ores and related materials, commercial purity copper and copper-base alloys) issued previously by the CCRMP are described in a catalogue¹ available from the Canada Centre for Mineral and Energy Technology, Department of Energy, Mines and Resources, Ottawa, Canada. SL-1 was chosen because there is a need for such a reference material in laboratories associated with the iron and steel industry.

Initially, fourteen laboratories provided results for two or more of the constituents CaO, SiO₂, MgO, Al₂O₃, S, Fe (expressed as FeO), MnO, TiO₂, Na₂O, K₂O, P₂O₅, V₂O₅ and Cr₂O₃. At a later stage, after a preliminary assessment of the results had been obtained, a further seven laboratories were recruited to supply additional results of the constituents CaO and MgO. From the overall assessment, it was considered that the results are sufficiently satisfactory, in a statistical sense, to warrant the assignment of recommended values for six of the constituents investigated and provisional values for four others. The mean values for the three remaining constituents are presented for information only.

ORIGIN AND PREPARATION OF SL-1

The material for SL-1 was donated to the CCRMP in mid 1974 and is from the Blast Furnace Plant of the Hamilton Works of the Steel Company of Canada Ltd. A total weight of approximately 940 lbs of the material was received in a coarsely-ground state (approx. 1/4"). After drying at 110°C on the drying bed, it was dry-ground in batches by ball-milling until it passed a 74µm screen. The entire material was then tumbled for 8 hours in a 570-ℓ conical blender, immediately after which it was sampled systematically. By means of x-ray fluorescence analysis of the samples for calcium, iron, titanium and manganese, SL-1 was found to be sufficiently homogeneous to be bottled in 200-g units and to be distributed in the interlaboratory certification program.

INTERLABORATORY CERTIFICATION PROGRAM

The laboratories that participated in the program to certify SL-1 are listed herewith alphabetically. Each was assigned a code number so that analytical results could be recorded while preserving the anonymity of the laboratory. The code number bears no relationship to the alphabetical order of the laboratories.

Participating Laboratories

Amsted Research Laboratories, Bensenville, Illinois, U.S.A.

British Steel Corporation, Swinden Research Laboratories, Rotherham, England

British Steel Corporation, Teesside Research Laboratories, Teesside, England

British Steel Corporation, Sheffield Works, Rotherham, England

Canada Centre for Mineral and Energy Technology, Mineral Sciences Laboratories, Ottawa, Ontario

Cominco Ltd., Trail, British Columbia

Geological Survey of Canada, Analytical Chemistry Section, Ottawa, Ontario

Hudson Bay Mining and Smelting Co., Ltd., Flin Flon, Manitoba

Inland Steel Company, East Chicago, Indiana, U.S.A.

Interlake Inc., Beverley, Ohio, U.S.A.

Ministry of Natural Resources, Mineral Research Branch, Toronto, Ontario.

Sydney Steel Corporation, Sydney, Nova Scotia

The Steel Company of Canada Ltd., Hamilton, Ontario

United States Steel Corporation, Duquesne, Pennsylvania, U.S.A.

* Armco Steel Corporation, Middletown, Ohio, U.S.A.

* Bethlehem Steel Corporation, Homer Research Laboratories, Bethlehem, Pennsylvania, U.S.A.

* Falconbridge Nickel Mines Ltd., Metallurgical Laboratories, Thornhill, Ontario

* Inco, Copper Cliff, Ontario

* The Algoma Steel Corporation Ltd., Sault Sainte Marie, Ontario

* United States Steel Corporation, Research Laboratories, Monroeville, Pennsylvania, U.S.A.

* Wheeling Pittsburgh Steel Corporation, Steubenville, Ohio, U.S.A.

Each participating laboratory received two randomly-selected bottles of SL-1 and was asked to determine in triplicate as many as possible of the constituents CaO, MgO, Al₂O₃, SiO₂, Fe (expressed as FeO), S, P₂O₅, MnO, TiO₂, Na₂O, K₂O, V₂O₅ and Cr₂O₃ in

* This group of seven laboratories participated at a later stage to provide additional results on CaO and MgO.

each bottle, by methods of their choice. Of the total results obtained, approximately 50% were by conventional chemical methods (gravimetric, volumetric, compleximetric) and the remainder by instrumental methods (atomic absorption spectrophotometry, flame photometry, x-ray fluorescence spectrometry and optical emission spectrochemistry).

A number of laboratories voluntarily submitted results by more than one analytical method (Table 1). Each method was considered to be independent of the others for statistical purposes, i.e., each set of results was treated as if it originated in a separate laboratory.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

All the analytical results obtained for SL-1 are presented in Table 1. Certain sets of results appeared to deviate grossly from the consensus values and were subjectively rejected from further consideration. The remaining sets were subjected to the widely-used two-standard deviation criterion and certain of these were identified as possible outliers. Both types of outliers are noted in Table 1.

Confirmation of Homogeneity Using Interlaboratory Results

Table 2 gives the means, coefficients of variation and summary of the t-tests between bottles at the 5% significance level for all sets of results for SL-1, gross outliers excluded. There is a relatively high proportion of sets of results for which the null hypothesis of no difference between bottles was rejected. A two-way ANOVA with nested design² was performed on the data which conformed to this design (much of the data

could not be used). This analysis confirmed the between-bottle difference except for Al_2O_3 and S. The magnitude of this inhomogeneity, however, was found to be extremely small when compared to the between-set (between-laboratory) component of variance. For this reason, SL-1 should be sufficiently homogeneous for most applications as a reference material.

Estimation of Consensus Values and 95% Confidence Limits

A one-way ANOVA technique² was used to calculate the consensus values and to estimate the variance of the means and hence the 95% confidence limits. Both gross and possible outliers were excluded from these computations in order to avoid the possible introduction of bias to the estimates. The data are assumed to fit the following model:

$$x_{ij} = \mu + y_i + e_{ij}$$

where:

x_{ij} = the j^{th} result reported in set i ;

μ = the true value that is estimated by the overall mean $\bar{x}_{..}$;

y_i = the discrepancy between the mean of the results from set i and the true value; and

e_{ij} = the discrepancy of x_{ij} from the means of the results from set i .

It is assumed in this analysis that both y_i and e_{ij} are normally distributed with means of zero and variances of ω^2 and σ^2 , respectively. The significance of ω^2 can be detected by comparing the ratio of "between-set" mean squares to "within-set" mean squares with the F statistic at the 95% confidence level and with the appropriate degrees of freedom. The magnitude of ω^2 and σ^2 can be estimated from the ANOVA table.

Analysis of variance and expected mean squares for the one-way classification

Source of variance	Sums of squares	Degrees of freedom	Mean squares	E [Mean squares]
Between-sets	$\sum_i^k n_i (\bar{x}_{i.} - \bar{x}_{..})^2$	$k-1$	s_2^2	$\sigma^2 + \frac{1}{k-1} \left(\sum_i^k n_i - \frac{\sum_i^k n_i^2}{k} \right) \omega^2$
Within-sets	$\sum_i^k \sum_j^{n_i} (x_{ij} - \bar{x}_{i.})^2$	$\sum_i^k n_i - k$	s_1^2	σ^2
Total	$\sum_i^k \sum_j^{n_i} (x_{ij} - \bar{x}_{..})^2$	$\sum_i^k n_i - 1$		

The consensus value, in the above model, can be estimated by the overall mean $\bar{x}_{..}$, thus:

$$\bar{x}_{..} = \frac{\sum_{i=1}^k \sum_{j=1}^{n_i} x_{ij}}{\sum_{i=1}^k n_i}$$

with the variance of the overall mean being given by:

$$V[\bar{x}_{..}] = \frac{\sum_{i=1}^k n_i^2}{\left(\sum_{i=1}^k n_i\right)^2} \omega^2 + \frac{1}{k} \sigma^2$$

The 95% confidence limits for the overall mean are then given by:

$$\bar{x}_{..} \pm \left[t_{0.975(k-1)} \sqrt{V[\bar{x}_{..}]} \right]$$

where:

n_i = the number of results reported in set i ;

k = the number of sets.

The above values and other statistics computed from the one-way ANOVA are presented in Table 3.

Certification factor

The certification factor³ is a measure for evaluating the quality of reference materials issued by the CCRMP. It is computed from the following expression:

$$CF = 200 \left[t_{0.975(k-1)} \sqrt{V[\bar{x}_{..}]} / \bar{x}_{..} / \bar{cv} \right]$$

where \bar{cv} is the average of the within-set coefficients of variation and is given by:

$$\bar{cv} = \sum_{i=1}^k cv_i / k$$

The critical value of CF is 4. If a selected constituent has a CF greater than 4, the reference material is considered to be of unacceptable quality with respect to that constituent. Table 3 illustrates that the respective certification factors for the constituents SiO_2 , CaO , MgO , Al_2O_3 , total Fe (expressed as FeO), and S are less than the critical value. The consensus values for these six constituents of SL-1 are thus accepted as recommended values and are presented in Table 4.

Provisional Values for TiO_2 , MnO , Na_2O , and K_2O

The certification factors for TiO_2 and MnO , respectively 4.4 and 4.3, are slightly higher than the critical value of 4. In this case the use of the factor illustrates how an objective decision can be made where it is doubtful whether to proceed with the certification of a particular element or constituent. Thus, provisional values only are given for TiO_2 and MnO . (Table 5).

With regard to Na_2O and K_2O , their respective certification factors fall significantly beyond the critical limit, to the point where a decision was required whether to list their mean concentrations as "provisional", or merely label them "for information only." It is well known that the determination of these constituents in metallurgical slags is particularly troublesome and the development of analytical methodology in this respect still moves slowly. Taking into account the expected use of this reference material, the present state-of-the-art for Na_2O and K_2O , and the fair within-laboratory and between-bottle precision of the 72 observations provided, it is considered justifiable to list the values for these constituents (as given in Table 5) as provisional. However, it is intended that further analyses be carried out on SL-1 to certify it ultimately for Na_2O and K_2O .

Values for P_2O_5 , V_2O_5 and Cr_2O_3

The constituents P_2O_5 , V_2O_5 and Cr_2O_3 are not generally determined in blast-furnace iron-making practice which utilises iron ore in which they are present in amounts as low as 0.10% or less. No doubt, this is the reason why only some 4 to 9 sets of results were obtained from the participating laboratories. In view of the high average cv values and the considerable between-laboratory differences for all three constituents, the associated data is listed for interest only.

REFERENCES

1. FAYE, G.H., Certified and Provisional Reference Materials Available from the Canada Centre for Mineral and Energy Technology (CANMET) as of 1976. CANMET Internal Report No. MRP/MSL 76-3.
2. BROWNLEE, K.A., Statistical Theory and Methodology in Sciences. John Wiley and Sons, Inc., New York (1961).
3. SUTARNO, R., and FAYE, G.H., A measure for assessing certified reference ores and related materials, *Talanta*, 22, 676-681 (1975).

TABLE 1
RESULTS OF ANALYSIS OF SL-1

SILICA (WEIGHT PERCENT)

LAR- 2	35.89	35.94	36.00	35.94	36.20	35.89	36.01	36.00		
* LAR- 3 (A.A.)	34.60	34.71								
LAR- 4	35.80	35.68	35.98	35.80	35.76	35.78				
LAR- 5 (GRAV.)	35.92	35.49	36.02	35.90	35.88	35.90				
LAR- 6	35.12	35.24	35.18	35.56	35.56	35.57				
LAR- 7	35.85	36.04	35.89	36.03	35.99	36.00				
LAR- 9 (GRAV.)	35.76	35.68	35.68	35.66	35.66	35.72				
LAR-10 (GRAV.)	36.10	36.00	36.10	36.00	36.10	36.10				
LAR-11 (GRAV.)	35.60	35.60	35.70	35.60	35.70	35.70	35.80	35.80		
LAR-12 (GRAV.)	35.61	35.70	35.63	35.62	35.63	35.74				
LAR-13 (GRAV.)	35.92	35.88	35.86	35.88	35.90	35.86				
LAR-14 (GRAV.)	35.47	35.50	35.62	35.50	35.55	35.63				
LAR-16 (GRAV.)	35.45	35.53	35.55	35.55	35.58	35.53				
LAR-16 (A.A.)	35.50	35.80	35.50	35.50	35.50	35.30				
LAR-17 (XRF)	35.70	35.70	34.90	35.20						
LAR-17 (GRAV.)	36.20	36.24								
LAR-21 (XRF)	35.85	35.97	35.99	35.84	35.83	35.94	36.15	36.12	35.90	36.19
LAR-21 (A.A.)	35.20	35.75	35.56	35.62						
LAR-21 (GRAV.)	35.30	35.24	35.48	35.30						

A.A. = Atomic absorption; GRAV. = gravimetric; XRF = X-ray fluorescence; VOL. = volumetric
COMPL. = Compleximetric; COLOR = colorimetric; SPECTR. = spectrographic; POTEN. = potentiometric;
COMB. = Combustion; FLAME = emission flame photometry

* gross outlier

TABLE 1 (continued)

LIME (WEIGHT PERCENT)

LAR- 2	36.94	37.02	37.16	37.06	36.98	36.93	36.96	36.95		
LAR- 3 (A.A.)	38.20	38.06								
LAR- 4	38.50	38.50	38.50	38.50	38.00	38.50				
LAR- 5 (VOL.)	37.50	37.50	37.68	37.50	37.61	37.31				
LAR- 6	37.56	37.64	37.48	37.48	37.50	37.46				
LAR- 7	37.21	37.16	37.17	37.28	37.27	37.30				
* LAR- 9 (A.A.)	34.70	34.70	34.70	34.70	35.10	34.70				
LAR-10 (VOL.)	37.40	37.40	37.30	37.40	37.40	37.30				
LAR-10 (A.A.)	37.60	37.60	37.10	37.60	37.60	37.60				
LAR-11 (COMPL.)	37.21	37.26	37.47	36.99	37.47	37.47	36.99	37.47	37.03	
LAR-11	37.14	37.10	37.10	37.12	37.08	37.10	37.10	37.14	37.14	37.12
LAR-12 (GRAV.)	37.49	37.91	37.65	37.70	37.72	37.69				
LAR-13 (VOL.)	37.80	37.80	37.90	37.70	37.80	37.80				
LAR-14 (VOL.)	38.30	38.30	38.30	38.30	38.20	38.20				
LAR-16 (GRAV.)	37.45	37.51	37.60	37.34	37.42	37.40				
LAR-16 (A.A.)	37.40	37.40	37.40	37.20	37.40	37.40				
** LAR-17 (XRF)	36.10	36.00	35.80	36.10						
LAR-17 (A.A.)	36.50	36.60								
LAR-18	36.73	37.00	36.72	36.94	36.87	36.83	36.95	37.02	36.89	37.06
	36.99	36.97								
LAR-20 (A.A.)	37.60	37.40	37.40	37.50	37.50	37.70	37.60	37.40	37.40	37.40
LAR-21 (XRF)	37.19	37.64	37.21	37.56	37.05	36.55	36.84	37.42	36.92	37.01
LAR-21 (GRAV.)	36.98	36.92	37.00	36.95						
LAR-22 (XRF)	37.61	37.48	37.55	37.62	37.78	37.87	37.77	37.96	37.88	37.62
LAR-22 (VOL.)	37.85	37.45	37.66	37.80	37.71	37.70	37.82	37.78	37.62	37.82
LAR-23 (VOL.)	37.67	37.68	37.60	37.58	37.66	37.48	37.57	37.53	37.59	37.58
LAR-24 (GRAV.)	38.24	38.20	37.68	38.00	38.56	37.84	37.62	37.56	38.14	38.14
LAR-25 (GRAV.)	37.41	37.41	37.40	37.38	37.32	37.40	37.37	37.44	37.44	37.34

* gross outlier

** possible outlier

TABLE 1 (continued)

MAGNESIA (WEIGHT PERCENT)

LAB- 2	12.40	12.37	12.35	12.45	12.52	12.29	12.49	12.50		
LAB- 3 (A.A.)	12.20	12.26								
LAB- 4	11.88	11.88	11.88	11.88	11.88	11.88				
LAB- 5 (GRAV.)	11.78	12.15	11.95	12.00	12.02	12.00				
LAB- 6	11.78	11.93	12.09	11.58	11.64	11.66				
LAB- 7	11.84	11.85	11.90	11.91	11.86	11.88				
LAB- 9 (A.A.)	12.20	12.30	12.20	12.20	12.40	12.20				
LAB-10 (GRAV.)	12.60	12.60	12.60	12.60	12.70	12.60				
LAB-10 (A.A.)	12.40	12.40	12.40	12.40	12.40	12.40				
LAB-11 (COMPL.)	12.96	12.90	12.73	12.88	12.96	12.81	13.25	13.27	12.78	12.96
LAB-11	12.88	12.81								
LAB-12 (GRAV.)	12.60	12.64	12.56	12.65	12.57	12.56	12.59	12.52	12.54	12.57
LAB-12	12.76	12.72	12.75	12.71	12.54	12.75				
LAB-13 (A.A.)	12.27	12.27	11.94	11.94	11.94	12.27				
LAB-14 (GRAV.)	12.38	12.38	12.43	12.46	12.43	12.40				
LAB-16 (GRAV.)	12.21	12.13	12.10	12.20	12.23	12.23				
LAB-16 (A.A.)	12.10	12.10	12.10	12.20	12.20	12.20				
** LAB-17 (XRF)	13.20	13.10	13.30	13.00						
LAB-17 (A.A.)	11.70	11.68								
LAB-18	11.81	12.20	11.88	12.09	12.00	11.98	11.74	12.02	11.80	12.16
	11.88	11.98								
LAB-20 (A.A.)	12.20	12.10	12.10	12.10	12.20	12.20	12.10	12.10	12.20	12.20
LAB-21 (XRF)	12.05	12.33	12.16	11.89	12.17	12.20	12.15	12.18	12.35	12.08
LAB-21 (A.A.)	12.35	12.19	12.19	12.27						
LAB-21 (GRAV.)	12.37	12.38	12.41	12.42						
LAB-22 (XRF)	11.75	11.77	11.55	11.86	11.57	12.09	12.13	12.02	12.28	11.96
LAB-22 (VOL.)	11.61	11.69	11.49	11.88	11.72	11.98	12.02	12.20	12.12	12.00
LAB-23 (VOL.)	12.60	12.57	12.57	12.63	12.69	12.63	12.52	12.52	12.53	12.51
LAB-24 (GRAV.)	13.06	12.62	12.46	12.96	13.30	13.01	12.52	12.50	13.22	13.01
LAB-25 (GRAV.)	11.97	12.25	12.12	12.12	12.14	12.06	12.07	11.92	11.98	12.10

** possible outlier

TABLE 1 (continued)

ALUMINA (WEIGHT PERCENT)

LAR- 2	9.99	9.99	9.92	9.89	9.92	10.07	9.77	10.22		
LAR- 3 (A.A.)	9.47	9.34								
LAR- 4	9.70	9.70	8.96	9.40	9.84	9.40				
LAR- 5 (GRAV.)	9.60	9.48	9.51	9.49	9.63	9.53				
* LAR- 6	11.02	10.68	11.00	11.24	11.14	11.20				
LAR- 7	9.67	9.64	9.65	9.64	9.65	9.68				
** LAR- 9 (A.A.)	9.00	8.90	9.00	9.00	9.20	9.00				
LAR-10 (GRAV.)	9.60	9.60	9.60	9.60	9.60	9.60				
LAR-10 (A.A.)	9.60	9.60	9.60	9.60	9.60	9.60				
LAR-11 (COMPL.)	9.77	9.79	9.83	9.77	9.79	9.77	9.77	9.83		
LAR-12 (GRAV.)	9.65	9.59	9.60	9.56	9.68	9.59				
LAR-13 (A.A.)	9.30	9.40	9.50	9.30	9.19	9.50				
LAR-14 (VOL.)	9.70	9.50	9.65	9.50	9.50	9.50				
LAR-16 (A.A.)	9.61	9.60	9.69	9.61	9.69	9.61				
LAR-17 (XRF)	9.61	10.10	9.64	9.25						
LAR-17 (A.A.)	9.52	9.52								
LAR-21 (XRF)	9.52	9.63	9.68	9.64	9.61	9.51	9.68	9.82	9.69	9.75
LAR-21 (GRAV.)	9.68	9.50	9.58	9.54						
LAR-22 (XRF)	9.66	9.72	9.74	9.67	9.59	9.69	9.62	9.62	9.74	9.84

* gross outlier

** possible outlier

TABLE 1 (continued)

TITANIA (WEIGHT PERCENT)

LAR- 2	.37	.38	.37	.37	.37	.37	.39	.36				
LAR- 3 (A.A.)	.42	.41										
* LAR- 4 (A.A.)	.19	.19	.19	.19	.19	.19						
LAR- 5 (COLOR.)	.36	.37	.36	.36	.36	.36						
* LAR- 6	.10	.11	.10	.13	.12	.13						
LAR- 7	.33	.33	.33	.33								
LAR-10 (COLOR.)	.36	.36	.36	.36	.36	.36						
LAR-11 (COLOR.)	.40	.40	.40	.42	.42	.42	.42	.42	.42	.42	.42	
	.42	.42										
LAR-12 (COLOR.)	.41	.42	.40	.41	.41	.41						
LAR-13 (COLOR.)	.38	.37	.36	.38	.35	.37						
LAR-14 (COLOR.)	.37	.38	.37	.37	.37	.37						
LAR-16 (COLOR.)	.35	.36	.36	.36	.37	.36						
LAR-16 (A.A.)	.38	.38	.38	.38	.38	.38						
** LAR-17 (XRF)	.23	.27	.27	.27								
LAR-17 (A.A.)	.34	.33										
LAR-17 (SPECTR.1)	.37	.37	.37	.40	.42	.39						
LAR-17 (SPECTR.2)	.43	.42	.42	.42	.42	.40						
LAR-21 (XRF)	.37	.38	.37	.38	.37	.36	.37	.38	.37	.37	.37	
LAR-21 (COLOR.)	.33	.33	.34	.32	.34	.34	.36	.32				

* gross outlier

** possible outlier

1
∞
1

TABLE 1 (continued)

TOTAL IRON EXPRESSED AS FeO (WEIGHT PERCENT)

LAB- 2	1.150	1.080	1.150	1.070	1.100	1.090	1.080	1.070		
** LAB- 3 (A.A.)	1.240	1.250								
LAB- 4	.970	.970	.970	1.030	1.030	.970				
LAB- 5 (VOL.)	.939	.823	.901	.901	.862	.901				
LAB- 6	1.140	1.140	1.140	.920	.920	.910				
LAB- 7	.875	.875	.875	.875						
** LAB- 9 (VOL.)	1.286	1.158	1.286	1.158	1.286	1.286				
** LAB- 9 (A.A.)	1.222	1.222	1.248	1.274	1.235	1.248				
LAB-10 (A.A.)	.900	.900	.890	.890	.890	.900				
LAB-11 (COLOR.)	.890	.910	.890	.900	.900	.900	.900	.900	.900	.900
	.910	.900								
LAB-12 (VOL.)	.880	.900	.880	.880	.880	.860				
LAB-12 (A.A.)	.890	.880	.880	.870	.860	.860				
LAB-13 (A.A.)	.913	.926	.939	.926	.952	.939				
LAB-14 (VOL.)	.901	.901	.939	.901	.901	.875				
LAB-16 (A.A.)	.936	.936	.936	.936	.900	.900				
LAB-17 (XRF)	.774	.792	.792	.801						
LAB-17 (POTEN.)	.837	.900								
LAB-17 (SPECTR.1)	.870	.800	.910	.950	1.000	.950				
LAB-17 (SPECTR.2)	.910	.870	.800	.780	.720	.890				
LAB-21 (XRF)	.980	1.000	.970	.960	.970	.850	.870	.940	.870	1.010
LAB-21 (A.A.)	.960	.960	.940	.940	.960	.960	.930	.930		

** possible outlier

TABLE 1 (continued)

MnO (WEIGHT PERCENT)

	1.080	1.070	1.080	1.060	1.080	1.070	1.080	1.070		
** LAB- 2	.960	.950								
LAB- 3 (A.A.)	.760	.760	.770	.760	.770	.770				
LAB- 4	.930	.830	.810	.800	.800	.820				
LAB- 5 (COLOR.)	.950	.970	.930	.950	.880	.880				
LAB- 6	.823	.831	.825	.822	.826	.828				
LAB- 7	.850	.850	.850	.850	.850	.850				
LAB- 9 (A.A.)	.840	.840	.850	.840	.850	.840				
LAB-10 (COLOR.)	.850	.850	.850	.850	.880	.850				
LAB-10 (A.A.)	.900	.920	.920	.940	.900	.880	.900	.930		
LAB-11 (COLOR.)	.850	.840	.840	.860	.840	.840				
LAB-12 (COLOR.)	.880	.860	.880	.860	.870	.870				
LAB-13 (A.A.)	.820	.770	.770	.790	.790	.820				
LAB-14 (VOL.)	.878	.878	.881	.873	.878	.873	.891	.884	.872	.884
LAB-16 (A.A.)	.872	.878								
** LAB-17 (XRF)	.710	.690	.720	.700						
LAB-17 (A.A.)	.830	.830								
LAB-17 (SPECTR.1)	.880	.870	.920	.890	.940	.960				
LAB-17 (SPECTR.2)	.800	.800	.770	.760	.750	.800				
LAB-21 (XRF)	.920	.940	.940	.930	.930	.910	.920	.930	.920	.940
LAB-21 (A.A.)	.860	.860	.850	.850	.850	.860	.850	.850		

** possible outlier

TABLE 1 (continued)

SULPHUR (WEIGHT PERCENT)

LAB- 2	1.31	1.30	1.31	1.32	1.33	1.30	1.36	1.34		
LAB- 4	1.28	1.32	1.36	1.32	1.27	1.24				
LAB- 5 (GRAV.)	1.23	1.25	1.22	1.23	1.24	1.25				
LAB- 6	1.19	1.14	1.17	1.17	1.17	1.18				
LAB- 7	1.25	1.27	1.26	1.25	1.25	1.26				
LAB- 9 (COMB.)	1.23	1.21	1.22	1.16	1.18	1.16				
LAB-10 (GRAV.)	1.28	1.29	1.29	1.29	1.30	1.29				
LAB-10 (COMB.)	1.30	1.20	1.30	1.28	1.30	1.28				
LAB-11 (GRAV.)	1.26	1.31	1.26	1.28	1.27	1.31	1.25	1.29	1.24	1.28
	1.26									
LAB-12 (COMB.)	1.25	1.21	1.25	1.26	1.24	1.26				
LAB-12 (GRAV.)	1.15	1.20	1.17	1.18	1.16	1.19				
LAB-13 (COMB.)	1.25	1.26	1.26	1.26	1.25	1.25				
LAB-14 (GRAV.)	1.27	1.25	1.28	1.27	1.26	1.27				
LAB-16 (COMB.)	1.29	1.29	1.28	1.28	1.29	1.28				
* LAB-17 (XRF)	.74	.83	.70	.83						
LAB-17 (COMB.)	1.16	1.16								
LAB-21 (GRAV.)	1.21	1.26	1.22	1.28						
LAB-21 (COMB.)	1.32	1.32	1.34	1.34						

* gross outlier

TABLE 1 (continued)

P₂O₅ (WEIGHT PERCENT)

LAB- 2	.0060	.0080	.0070	.0070	.0050	.0060	.0060	.0070
LAB- 4	.0290	.0340	.0260	.0460	.0340	.0460		
LAB- 7	.0060	.0050	.0050	.0060				
LAB-10 (COLOR.)	.0011	.0011	.0009	.0011	.0011	.0010	.0009	
LAB-12 (COLOR.)	.0320	.0270	.0320	.0270	.0320	.0320	.0009	.0007
LAB-13 (VOL.)	.0180	.0180	.0190	.0190	.0200	.0180		
LAB-14 (VOL.)	.0300	.0200	.0300	.0300	.0400	.0400		
LAB-16 (COLOR.)	.0200	.0200	.0200	.0200	.0200	.0200		
LAB-17 (XRF)	.0500	.0300	.0400	.0300				

TABLE 1 (continued)

V205 (WEIGHT PERCENT)

** LAR- 2	.0480	.0470	.0490	.0480	.0480	.0500	.0490	.0500		
** LAR- 3 (A.A.)	.0420	.0460								
* LAR- 7	.1200	.1200								
LAR-10 (COLOR.)	.0030	.0040	.0030	.0030	.0040	.0030				
LAR-11 (COLOR.)	.0070	.0040	.0050	.0040						
LAR-12 (COLOR.)	.0030	.0032	.0032	.0032	.0032	.0034	.0030	.0032	.0034	.0030
LAR-13 (A.A.)	.0030	.0030								
LAR-17 (SPECTR.1)	.0050	.0050	.0050	.0050	.0050	.0050				
LAR-17 (SPECTR.2)	.0060	.0060	.0060	.0070	.0080	.0070				
	.0050	.0040	.0040	.0030	.0040	.0040				

* gross outlier

** possible outlier

TABLE 1 (continued)

Na₂O (WEIGHT PERCENT)

LAB- 2	.310	.300	.320	.310	.310	.320	.310	.310
LAB- 3 (A.A.)	.430	.420						
LAB- 4 (A.A.)	.338	.359	.337	.344	.329	.340		
LAB- 5 (A.A.)	.380	.400	.400	.400	.380	.410		
LAB- 9 (A.A.)	.320	.320	.320	.320	.320	.320		
LAB-10 (A.A.)	.490	.490	.490	.490	.490	.490		
LAB-11 (FLAME)	.315	.320	.320	.315	.310	.315	.315	.320
LAB-12 (A.A.)	.460	.420	.430	.460	.420	.410		
LAB-13 (FLAME)	.460	.460	.460	.460	.460	.460		
LAB-14 (FLAME)	.390	.420	.400	.390	.390	.400		
* LAB-17 (XRF)	.700	.600	.800	.500				
LAB-17 (A.A.)	.380	.320						
LAB-17 (SPECTR.1)	.460	.440	.450	.420	.450	.440		

* gross outlier

TABLE 1 (continued)

K₂O (WEIGHT PERCENT)

LAR- 2	.440	.420	.440	.440	.440	.450	.430	.440		
LAR- 3 (A.A.)	.440	.450								
LAR- 4 (A.A.)	.622	.597	.603	.609	.591	.634				
LAR- 5 (A.A.)	.470	.510	.480	.480	.480	.500				
LAR- 7	.530	.590								
** LAR- 9 (A.A.)	.330	.350	.340	.340	.340	.350				
LAR-10 (A.A.)	.510	.510	.510	.510	.510	.520				
LAR-11 (FLAME)	.500	.500	.495	.495	.490	.500	.490	.500		
LAR-12 (A.A.)	.470	.470	.470	.500	.460	.460				
LAR-13 (FLAME)	.520	.520	.520	.520	.520	.520				
LAR-14 (FLAME)	.490	.450	.490	.450	.490	.490				
LAR-17 (XRF)	.590	.640	.640	.620						
LAR-17 (A.A.)	.540	.530								
LAR-22 (XRF)	.500	.510	.510	.520	.500	.500	.490	.490	.480	.500

** possible outlier

TABLE 1 (continued)

 Cr_2O_3 (WEIGHT PERCENT)

** LAB- 3 (A.A.)	.0500	.0600				
LAB- 4 (A.A.)	.0040	.0030				
LAB- 9 (A.A.)	.0300	.0300	.0030	.0070	.0040	.0070
LAB-10 (COLOR.)	.0047	.0048	.0300	.0300	.0300	.0300
LAB-10 (A.A.)	.0053	.0050	.0048	.0048	.0048	.0047
LAB-11 (COMPL.)	.0140	.0130	.0051	.0050	.0050	.0050
LAB-12 (A.A.)	.0060	.0060	.0130	.0130		
LAB-13 (A.A.)	.0080	.0070	.0060	.0060	.0060	.0060
* LAB-16 (A.A.)	.5600	.5700	.0080	.0080	.0070	.0070
LAB-17 (SPECTR.1)	.0020	.0020	.5700	.5300	.5700	.5500
LAB-17 (SPECTR.2)	.0040	.0050	.0020	.0020	.0030	.0030
			.0050	.0060	.0050	.0060

* gross outlier

** possible outlier

TABLE 2

Laboratory means, coefficients of variation and summary of t-tests on results between bottles for SI-1

SILICA (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 2	4	35.9425	.0450	4	36.0250	.1287	A	8	35.9838	.0996	.28
LAB- 4	3	35.8200	.1510	3	35.7800	.0200	A	6	35.8000	.0988	.28
LAB- 5 (GRAV.)	3	35.8100	.2816	3	35.8933	.0115	A	6	35.8517	.1840	.51
LAB- 6	3	35.1800	.0600	3	35.5633	.0058	REJECT	6	35.3717	.2134	.60
LAB- 7	3	35.9267	.1002	3	36.0067	.0208	A	6	35.9667	.0781	.22
LAB- 9 (GRAV.)	3	35.7067	.0462	3	35.6800	.0346	A	6	35.6933	.0393	.11
LAB-10 (GRAV.)	3	36.0667	.0577	3	36.0667	.0577	A	6	36.0667	.0516	.14
LAB-11 (GRAV.)	4	35.6250	.0500	4	35.7500	.0577	REJECT	8	35.6875	.0835	.23
LAB-12 (GRAV.)	3	35.6467	.0473	3	35.6633	.0666	A	6	35.6550	.0524	.15
LAB-13 (GRAV.)	3	35.8867	.0306	3	35.8800	.0200	A	6	35.8833	.0234	.07
LAB-14 (GRAV.)	3	35.5300	.0794	3	35.5600	.0656	A	6	35.5450	.0672	.19
LAB-16 (GRAV.)	3	35.5100	.0529	3	35.5533	.0252	A	6	35.5317	.0440	.12
LAB-16 (A.A.)	3	35.6000	.1732	3	35.4333	.1155	A	6	35.5167	.1602	.45
LAB-17 (XRF)	2	35.7000	0.0000	2	35.0500	.2121	***R**	4	35.3750	.3948	1.12
LAB-17 (GRAV.)	INSUFFICIENT DATA							2	36.2200	.0283	.08
LAB-21 (XRF)	5	35.8960	.0773	5	36.0600	.1310	REJECT	10	35.9780	.1332	.37
LAB-21 (A.A.)	2	35.4750	.3889	2	35.5900	.0424	A	4	35.5325	.2354	.66
LAB-21 (GRAV.)	2	35.2700	.0424	2	35.3900	.1273	A	4	35.3300	.1039	.29
TOTAL								106	35.7342	.2645	.74

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TWO-SIGMA LIMITS 35.205297 AN₀ 36.263194

A = Null hypothesis accepted

REJECT = Null hypothesis rejected

***R** = Null hypothesis rejected due to zero within-bottle variance

TABLE 2 (continued)

LIME (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V. (%)
LAR- 2	4	37.0450	.0915	4	36.9550	.0208	A	8	37.0000	.0780	.21
LAB- 3 (A.A.)	INSUFFICIENT DATA							2	38.1300	.0990	.26
LAB- 4	3	38.5000	0.0000	3	38.3333	.2887	A	6	38.4167	.2041	.53
LAB- 5 (VOL.)	3	37.5600	.1039	3	37.4733	.1518	A	6	37.5167	.1256	.33
LAB- 6	3	37.5600	.0800	3	37.4800	.0200	A	6	37.5200	.0681	.18
LAB- 7	3	37.1800	.0265	3	37.2833	.0153	REJECT	6	37.2317	.0598	.16
LAB-10 (VOL.)	3	37.3667	.0577	3	37.3667	.0577	A	6	37.3667	.0516	.14
LAB-10 (A.A.)	3	37.4333	.2887	3	37.6000	0.0000	A	6	37.5167	.2041	.54
LAB-11 (COMPL.)	5	37.2800	.2010	4	37.2400	.2661	A	9	37.2622	.2172	.58
LAB-11	5	37.1080	.0228	5	37.1200	.0200	A	10	37.1140	.0212	.06
LAB-12 (GRAV.)	3	37.6833	.2120	3	37.7033	.0153	A	6	37.6933	.1349	.36
LAB-13 (VOL.)	3	37.8333	.0577	3	37.7667	.0577	A	6	37.8000	.0641	.17
LAB-14 (VOL.)	3	38.3000	.0000	3	38.2333	.0577	A	6	38.2667	.0516	.13
LAB-16 (GRAV.)	3	37.5200	.0755	3	37.3867	.0416	A	6	37.4533	.0911	.24
LAB-16 (A.A.)	3	37.4000	0.0000	3	37.3333	.1155	A	6	37.3667	.0816	.22
LAB-17 (XRF)	2	36.0500	.0707	2	35.9500	.2121	A	4	36.0000	.1414	.39
LAB-17 (A.A.)	INSUFFICIENT DATA							2	36.5500	.0707	.19
LAB-18	6	36.8483	.1120	6	36.9800	.0587	REJECT	12	36.9142	.1095	.30
LAB-20 (A.A.)	5	37.4800	.0837	5	37.5000	.1414	A	10	37.4900	.1101	.29
LAB-21 (XRF)	5	37.3300	.2556	5	36.9480	.3152	A	10	37.1390	.3373	.91
LAB-21 (GRAV.)	2	36.9500	.0424	2	36.9750	.0354	A	4	36.9625	.0350	.09
LAB-22 (XRF)	5	37.6080	.1112	5	37.8200	.1306	REJECT	10	37.7140	.1599	.42
LAB-22 (VOL.)	5	37.6940	.1553	5	37.7480	.0867	A	10	37.7210	.1220	.32
LAB-23 (VOL.)	5	37.6380	.0449	5	37.5500	.0453	REJECT	10	37.5940	.0629	.17
LAB-24 (GRAV.)	5	38.1360	.3245	5	37.8600	.2760	A	10	37.9980	.3191	.84
LAB-25 (GRAV.)	5	37.3840	.0378	5	37.3980	.0438	A	10	37.3910	.0393	.11
TOTAL								187	37.4441	.4583	1.22

TWO-SIGMA LIMITS 36.527463 AND 38.360665

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAB- 4 MEAN = 38.416667
 LAB-17 (XRF) MEAN = 36.000000

TABLE 2 (continued)

MAGNESIA (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 2	4	12.3925	.0435	4	12.4500	.1074	A	8	12.4213	.0818	.66
LAB- 3 (A.A.)	INSUFFICIENT DATA							2	12.2300	.0424	.35
LAB- 4	3	11.8800	.0000	3	11.8800	.0000	A	6	11.8800	.0000	.00
LAB- 5 (GRAV.)	3	11.9600	.1852	3	12.0067	.0115	A	6	11.9833	.1201	1.00
LAB- 6	3	11.9333	.1550	3	11.6267	.0416	REJECT	6	11.7800	.1963	1.67
LAB- 7	3	11.8633	.0326	3	11.8833	.0252	A	6	11.8733	.0280	.24
LAB- 9 (A.A.)	3	12.2333	.0577	3	12.2667	.1155	A	6	12.2500	.0837	.68
LAB-10 (GRAV.)	3	12.6000	.0000	3	12.6333	.0577	A	6	12.6167	.0408	.32
LAB-10 (A.A.)	3	12.4000	.0000	3	12.4000	.0000	A	6	12.4000	.0000	.00
LAB-11 (COMPL.)	6	12.8733	.0898	6	12.9917	.2170	A	12	12.9325	.1700	1.31
LAB-11	5	12.6040	.0404	5	12.5560	.0270	A	10	12.5800	.0411	.33
LAB-12 (GRAV.)	3	12.7433	.0208	3	12.6667	.1115	A	6	12.7050	.0831	.65
LAB-13 (A.A.)	3	12.1600	.1905	3	12.0500	.1905	A	6	12.1050	.1807	1.49
LAB-14 (GRAV.)	3	12.3967	.0289	3	12.4300	.0300	A	6	12.4133	.0324	.26
LAB-16 (GRAV.)	3	12.1467	.0569	3	12.2200	.0173	A	6	12.1833	.0550	.45
LAB-16 (A.A.)	3	12.1000	.0000	3	12.2000	.0000	***R**	6	12.1500	.0548	.45
LAB-17 (XRF)	2	13.1500	.0707	2	13.1500	.2121	A	4	13.1500	.1291	.98
LAB-17 (A.A.)	INSUFFICIENT DATA							2	11.6900	.0141	.12
LAB-18	6	11.9933	.1405	6	11.9300	.1543	A	12	11.9617	.1445	1.21
LAB-20 (A.A.)	5	12.1400	.0548	5	12.1600	.0548	A	10	12.1500	.0527	.43
LAB-21 (XRF)	5	12.1200	.1628	5	12.1920	.0993	A	10	12.1560	.1327	1.09
LAB-21 (A.A.)	2	12.2700	.1131	2	12.2300	.0566	A	4	12.2500	.0766	.63
LAB-21 (GRAV.)	2	12.3750	.0071	2	12.4150	.0071	REJECT	4	12.3950	.0238	.19
LAB-22 (XRF)	5	11.7000	.1345	5	12.0960	.1218	REJECT	10	11.8980	.2412	2.03
LAB-22 (VOL.)	5	11.6780	.1438	5	12.0640	.0932	REJECT	10	11.8710	.2333	1.97
LAB-23 (VOL.)	5	12.6120	.0502	5	12.5420	.0497	A	10	12.5770	.0598	.48
LAB-24 (GRAV.)	5	12.8800	.3388	5	12.8520	.3238	A	10	12.8660	.3128	2.43
LAB-25 (GRAV.)	5	12.1200	.0997	5	12.0260	.0740	A	10	12.0730	.0965	.80
TOTAL								200	12.2843	.3822	3.11

TWO-SIGMA LIMITS 11.519950 AN_D 13.048750

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAB-17 (XRF) MEAN = 13.150000

TABLE 2 (continued)

ALUMINA (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V. (%)
LAB- 2	4	9.9475	.0506	4	9.9950	.1936	A	8	9.9713	.1335	1.34
LAB- 3 (A.A.)	INSUFFICIENT DATA							2	9.4050	.0919	.98
LAB- 4	3	9.4533	.4272	3	9.5467	.2540	A	6	9.5000	.3185	3.35
LAB- 5 (GRAV.)	3	9.5300	.0624	3	9.5500	.0721	A	6	9.5400	.0613	.64
LAB- 7	3	9.6533	.0153	3	9.6567	.0208	A	6	9.6550	.0164	.17
LAB- 9 (A.A.)	3	8.9667	.0577	3	9.0667	.1155	A	6	9.0167	.0983	1.09
LAB-10 (GRAV.)	3	9.6000	0.0000	3	9.6000	0.0000	***R**	6	9.6000	.0000	.00
LAB-10 (A.A.)	3	9.6000	0.0000	3	9.6000	0.0000	***R**	6	9.6000	.0000	.00
LAB-11 (COMPL.)	4	9.7900	.0283	4	9.7900	.0283	A	8	9.7900	.0262	.27
LAB-12 (GRAV.)	3	9.6133	.0326	3	9.6100	.0624	A	6	9.6117	.0445	.46
LAB-13 (A.A.)	3	9.4000	.1000	3	9.3300	.1572	A	6	9.3650	.1239	1.32
LAB-14 (VOL.)	3	9.6167	.1041	3	9.5000	0.0000	A	6	9.5583	.0917	.96
LAB-16 (A.A.)	3	9.6333	.0493	3	9.6367	.0462	A	6	9.6350	.0428	.44
LAB-17 (XRF)	2	9.8550	.3465	2	9.4450	.2758	A	4	9.6500	.3484	3.61
LAB-17 (A.A.)	INSUFFICIENT DATA							2	9.5200	0.0000	0.00
LAB-21 (XRF)	5	9.6160	.0594	5	9.6900	.1151	A	10	9.6530	.0948	.98
LAB-21 (GRAV.)	2	9.5900	.1273	2	9.5600	.0283	A	4	9.5750	.0772	.81
LAB-22 (XRF)	5	9.6760	.0586	5	9.7020	.0923	A	10	9.6890	.0742	.77
TOTAL								108	9.5995	.2258	2.35

TWO-SIGMA LIMITS 9.147945 AND 10.051129

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAB- 9 (A.A.) MEAN = 9.016667

TABLE 2 (continued)

TITANIA (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAR- 2	4	.3725	.0050	4	.3725	.0126	A	8	.3725	.0089	2.38
LAR- 3 (A.A.)	INSUFFICIENT DATA							2	.4150	.0071	1.70
LAR- 5 (COLOR.)	3	.3633	.0058	3	.3600	0.0000	A	6	.3617	.0041	1.13
LAR- 7	2	.3300	0.0000	2	.3300	0.0000	***R**	4	.3300	.0000	.00
LAR-10 (COLOR.)	3	.3600	0.0000	3	.3600	0.0000	***R**	6	.3600	0.0000	0.00
LAR-11 (COLOR.)	6	.4100	.0110	6	.4200	.0000	***R**	12	.4150	.0090	2.18
LAR-12 (COLOR.)	3	.4100	.0100	3	.4100	.0000	A	6	.4100	.0063	1.54
LAR-13 (COLOR.)	3	.3700	.0100	3	.3667	.0153	A	6	.3683	.0117	3.17
LAR-14 (COLOR.)	3	.3733	.0058	3	.3700	.0000	A	6	.3717	.0041	1.10
LAR-16 (COLOR.)	3	.3567	.0058	3	.3633	.0058	A	6	.3600	.0063	1.76
LAR-16 (A.A.)	3	.3800	.0000	3	.3800	.0000	A	6	.3800	.0000	.00
LAR-17 (XRF)	2	.2750	.0071	2	.2700	0.0000	A	4	.2725	.0050	1.83
LAR-17 (A.A.)	INSUFFICIENT DATA							2	.3350	.0071	2.11
LAR-17 (SPECTR.1)	3	.3700	.0000	3	.4033	.0153	***R**	6	.3867	.0207	5.34
LAR-17 (SPECTR.2)	3	.4233	.0058	3	.4133	.0115	A	6	.4183	.0098	2.35
LAR-21 (XRF)	5	.3740	.0055	5	.3700	.0071	A	10	.3720	.0063	1.70
LAR-21 (COLOR.)	4	.3300	.0082	4	.3400	.0163	A	8	.3350	.0131	3.91
TOTAL								104	.3728	.0340	9.11

TWO-SIGMA LIMITS .304839 AND .440738

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAR-17 (XRF) MEAN = .272500

TABLE 2 (continued)

TOTAL IRON EXPRESSED AS FeO (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 2	4	1.1125	.0435	4	1.0850	.0129	A	8	1.0988	.0331	3.02
LAB- 3 (A.A.)	INSUFFICIENT DATA						A	2	1.2450	.0071	.57
LAB- 4	3	.9700	0.0000	3	1.0100	.0346	A	6	.9900	.0310	3.13
LAB- 5 (VOL.)	3	.8877	.0591	3	.8880	.0228	A	6	.8878	.0400	4.51
LAB- 6	3	1.1400	0.0000	3	.9167	.0058	****R**	6	1.0283	.1224	11.90
LAB- 7	2	.8750	0.0000	2	.8750	0.0000	****R**	4	.8750	0.0000	0.00
LAB- 9 (VOL.)	3	1.2433	.0739	3	1.2433	.0739	A	6	1.2433	.0661	5.32
LAB- 9 (A.A.)	3	1.2307	.0150	3	1.2523	.0199	A	6	1.2415	.0197	1.59
LAB-10 (A.A.)	3	.8967	.0058	3	.8933	.0058	A	6	.8950	.0055	.61
LAB-11 (COLOR.)	6	.8983	.0075	6	.9017	.0041	A	12	.9000	.0060	.67
LAB-12 (VOL.)	3	.8867	.0115	3	.8733	.0115	A	6	.8800	.0126	1.44
LAB-12 (A.A.)	3	.8833	.0058	3	.8633	.0058	REJECT	6	.8733	.0121	1.39
LAB-13 (A.A.)	3	.9260	.0130	3	.9390	.0130	A	6	.9325	.0136	1.46
LAB-14 (VOL.)	3	.9137	.0219	3	.8923	.0150	A	6	.9030	.0205	2.27
LAB-16 (A.A.)	3	.9360	.0000	3	.9120	.0208	A	6	.9240	.0186	2.01
LAB-17 (XRF)	2	.7830	.0127	2	.7965	.0064	A	4	.7898	.0115	1.45
LAB-17 (POTEN.)	INSUFFICIENT DATA						A	2	.8685	.0451	5.20
LAB-17 (SPECTR.1)	3	.8600	.0557	3	.9667	.0289	REJECT	6	.9133	.0706	7.73
LAB-17 (SPECTR.2)	3	.8600	.0557	3	.7967	.0862	A	6	.8283	.0736	8.89
LAB-21 (XRF)	5	.9760	.0152	5	.9080	.0665	A	10	.9420	.0579	6.15
LAB-21 (A.A.)	4	.9500	.0115	4	.9450	.0173	A	8	.9475	.0139	1.47
TOTAL								128	.9587	.1260	13.14

TWO-SIGMA LIMITS .706697 AND 1.210788

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAB- 3 (A.A.) MEAN = 1.245000
 LAB- 9 (VOL.) MEAN = 1.243333
 LAB- 9 (A.A.) MEAN = 1.241500

TABLE 2 (continued)

MnO (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V. (%)
LAB- 2	4	1.0725	.0096	4	1.0750	.0058	A	8	1.0738	.0074	.69
LAB- 3 (A.A.)	THERE IS ONLY 1 BOTTLE							2	.9550	.0071	.74
LAB- 4	3	.7633	.0058	3	.7667	.0058	A	6	.7650	.0055	.72
LAB- 5 (COLOR.)	3	.8233	.0115	3	.8067	.0115	A	6	.8150	.0138	1.69
LAB- 6	3	.9500	.0200	3	.9033	.0404	A	6	.9267	.0383	4.13
LAB- 7	3	.8263	.0042	3	.8253	.0031	A	6	.8258	.0033	.40
LAB- 9 (A.A.)	3	.8500	.0000	3	.8500	.0000	A	6	.8500	.0000	.00
LAB-10 (COLOR.)	3	.8433	.0058	3	.8433	.0058	A	6	.8433	.0052	.61
LAB-10 (A.A.)	3	.8500	.0000	3	.8600	.0173	A	6	.8550	.0122	1.43
LAB-11 (COLOR.)	4	.9200	.0163	4	.9025	.0206	A	8	.9112	.0196	2.15
LAB-12 (COLOR.)	3	.8433	.0058	3	.8467	.0115	A	6	.8450	.0084	.99
LAB-13 (A.A.)	3	.8733	.0115	3	.8667	.0058	A	6	.8700	.0089	1.03
LAB-14 (VOL.)	3	.7867	.0289	3	.8000	.0173	A	6	.7933	.0228	2.87
LAB-16 (A.A.)	6	.8768	.0032	6	.8802	.0075	A	12	.8785	.0058	.66
LAB-17 (XRF)	2	.7000	.0141	2	.7100	.0141	A	4	.7050	.0129	1.83
LAB-17 (A.A.)	INSUFFICIENT DATA							2	.8300	0.0000	0.00
LAB-17 (SPECTR.1)	3	.8900	.0265	3	.9300	.0361	A	6	.9100	.0358	3.93
LAB-17 (SPECTR.2)	3	.7900	.0173	3	.7700	.0265	A	6	.7800	.0228	2.92
LAB-21 (XRF)	5	.9320	.0084	5	.9240	.0114	A	10	.9280	.0103	1.11
LAB-21 (A.A.)	4	.8550	.0058	4	.8525	.0050	A	8	.8538	.0052	.61
TOTAL								126	.8682	.0778	8.96

TWO-SIGMA LIMITS .712713 AND 1.023747

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAB- 2 MEAN = 1.073750
LAB-17 (XRF) MEAN = .705000

TABLE 2 (continued)

SULPHUR (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V. (%)
LAB- 2	4	1.3100	.0082	4	1.3325	.0250	A	8	1.3213	.0210	1.59
LAB- 4	3	1.3200	.0400	3	1.2767	.0404	A	6	1.2983	.0431	3.32
LAB- 5 (GRAV.)	3	1.2333	.0153	3	1.2400	.0100	A	6	1.2367	.0121	.98
LAB- 6	3	1.1667	.0252	3	1.1733	.0058	A	6	1.1700	.0167	1.43
LAB- 7	3	1.2600	.0100	3	1.2533	.0058	A	6	1.2567	.0082	.65
LAB- 9 (COMB.)	3	1.2200	.0100	3	1.1667	.0115	REJECT	6	1.1933	.0308	2.58
LAB-10 (GRAV.)	3	1.2867	.0058	3	1.2933	.0058	A	6	1.2900	.0063	.49
LAB-10 (COMB.)	3	1.2667	.0577	3	1.2867	.0115	A	6	1.2767	.0388	3.04
LAB-11 (GRAV.)	6	1.2817	.0232	5	1.2640	.0207	A	11	1.2736	.0229	1.80
LAB-12 (COMB.)	3	1.2367	.0231	3	1.2533	.0115	A	6	1.2450	.0187	1.50
LAB-12 (GRAV.)	3	1.1733	.0252	3	1.1767	.0153	A	6	1.1750	.0187	1.59
LAB-13 (COMB.)	3	1.2567	.0058	3	1.2533	.0058	A	6	1.2550	.0055	.44
LAB-14 (GRAV.)	3	1.2667	.0153	3	1.2667	.0058	A	6	1.2667	.0103	.82
LAB-16 (COMB.)	3	1.2867	.0058	3	1.2833	.0058	A	6	1.2850	.0055	.43
LAB-17 (COMB.)	INSUFFICIENT DATA							2	1.1600	0.0000	0.00
LAB-21 (GRAV.)	2	1.2350	.0354	2	1.2500	.0424	A	4	1.2425	.0330	2.66
LAB-21 (COMB.)	2	1.3200	0.0000	2	1.3400	0.0000	***R**	4	1.3300	.0115	.87
TOTAL								101	1.2562	.0505	4.02

TWO-SIGMA LIMITS 1.155169 AND 1.357306

TABLE 2 (continued)

P₂O₅ (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V. (%)
LAB- 2	4	.0070	.0008	4	.0060	.0008	A	8	.0065	.0009	14.24
LAB- 4	3	.0297	.0040	3	.0420	.0069	A	6	.0358	.0084	23.58
LAB- 7	2	.0055	.0007	2	.0055	.0007	A	4	.0055	.0006	10.50
LAB-10 (COLOR.)	5	.0011	.0001	4	.0009	.0001	REJECT	9	.0010	.0001	14.26
LAB-12 (COLOR.)	3	.0303	.0029	3	.0303	.0029	A	6	.0303	.0026	8.51
LAB-13 (VOL.)	3	.0183	.0006	3	.0190	.0010	A	6	.0187	.0008	4.37
LAB-14 (VOL.)	3	.0267	.0058	3	.0367	.0058	A	6	.0317	.0075	23.77
LAB-16 (COLOR.)	3	.0200	0.0000	3	.0200	0.0000	***R**	6	.0200	0.0000	0.00
LAB-17 (XRF)	2	.0400	.0141	2	.0350	.0071	A	4	.0375	.0096	25.53
							TOTAL	55	.0191	.0141	73.50

TWO-SIGMA LIMITS -.008989 AND .047237

TABLE 2 (continued)

V₂O₅ (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 2	4	.0480	.0008	4	.0493	.0010	A	8	.0486	.0011	2.18
LAB- 3 (A.A.)	INSUFFICIENT DATA							2	.0440	.0028	6.43
LAB-10 (COLOR.)	3	.0033	.0006	3	.0033	.0006	A	6	.0033	.0005	15.49
LAB-11 (COLOR.)	2	.0055	.0021	2	.0045	.0007	A	4	.0050	.0014	28.64
LAB-12 (COLOR.)	6	.0032	.0001	6	.0031	.0002	A	12	.0032	.0002	4.79
LAB-13 (A.A.)	3	.0050	0.0000	3	.0050	0.0000	***R**	6	.0050	0.0000	0.00
LAB-17 (SPECTR.1)	3	.0060	0.0000	3	.0073	.0006	***R**	6	.0067	.0008	12.25
LAB-17 (SPECTR.2)	3	.0043	.0006	3	.0037	.0006	A	6	.0040	.0006	15.81
TOTAL								50	.0130	.0176	135.71

TWO-SIGMA LIMITS -.022244 AND .042196

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAB- 2 MEAN = .048625

NOTE: Lab-3 (Mean = .0440) was also considered to be a possible outlier

TABLE 2 (continued)

Na₂O (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V. (%)
LAB- 2	4	.3100	.0082	4	.3125	.0050	A	8	.3113	.0064	2.06
LAB- 3 (A.A.)	INSUFFICIENT DATA							2	.4250	.0071	1.66
LAB- 4 (A.A.)	3	.3447	.0124	3	.3377	.0078	A	6	.3412	.0100	2.94
LAB- 5 (A.A.)	3	.3933	.0115	3	.3967	.0153	A	6	.3950	.0122	3.10
LAB- 9 (A.A.)	3	.3200	0.0000	3	.3200	0.0000	***R**	6	.3200	0.0000	0.00
LAB-10 (A.A.)	3	.4900	.0000	3	.4900	.0000	A	6	.4900	.0000	.00
LAB-11 (FLAME)	4	.3175	.0029	4	.3150	.0041	A	8	.3163	.0035	1.12
LAB-12 (A.A.)	3	.4367	.0208	3	.4300	.0265	A	6	.4333	.0216	4.99
LAB-13 (FLAME)	3	.4600	0.0000	3	.4600	0.0000	***R**	6	.4600	.0000	.00
LAB-14 (FLAME)	3	.4033	.0153	3	.3933	.0058	A	6	.3983	.0117	2.93
LAB-17 (A.A.)	INSUFFICIENT DATA							2	.3500	.0424	12.12
LAB-17 (SPECTR.1)	3	.4500	.0100	3	.4367	.0153	A	6	.4433	.0137	3.08
TOTAL								68	.3861	.0632	16.35

TWO-SIGMA LIMITS .259832 AND .512433

TABLE 2 (continued)

K₂O (WEIGHT PERCENT)

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 2	4	.4350	.0100	4	.4400	.0082	A	8	.4375	.0089	2.03
LAB- 3 (A.A.)	INSUFFICIENT DATA							2	.4450	.0071	1.59
LAB- 4 (A.A.)	3	.6073	.0131	3	.6113	.0216	A	6	.6093	.0161	2.64
LAB- 5 (A.A.)	3	.4867	.0208	3	.4867	.0115	A	6	.4867	.0151	3.09
LAB- 7	INSUFFICIENT DATA							2	.5600	.0424	7.58
LAB- 9 (A.A.)	3	.3400	.0100	3	.3433	.0058	A	6	.3417	.0075	2.20
LAB-10 (A.A.)	3	.5100	.0000	3	.5133	.0058	A	6	.5117	.0041	.80
LAB-11 (FLAME)	4	.4975	.0029	4	.4950	.0058	A	8	.4963	.0044	.89
LAB-12 (A.A.)	3	.4700	.0000	3	.4733	.0231	A	6	.4717	.0147	3.12
LAB-13 (FLAME)	3	.5200	.0000	3	.5200	.0000	A	6	.5200	.0000	.00
LAB-14 (FLAME)	3	.4767	.0231	3	.4767	.0231	A	6	.4767	.0207	4.33
LAB-17 (XRF)	2	.6150	.0354	2	.6300	.0141	A	4	.6225	.0236	3.80
LAB-17 (A.A.)	INSUFFICIENT DATA							2	.5350	.0071	1.32
LAB-22 (XRF)	5	.5080	.0084	5	.4920	.0084	REJECT	10	.5000	.0115	2.31
TOTAL								78	.4942	.0675	13.65

TWO-SIGMA LIMITS .359257 AND .629102

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAB- 9 (A.A.) MEAN = .341667

TABLE 2 (continued)

Cr₂O₃ (WEIGHT PERCENT)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAR- 3	(A.A.)	INSUFFICIENT DATA							2	.0550	.0071	12.86
LAR- 4	(A.A.)	3	.0033	.0006	3	.0060	.0017	A	6	.0047	.0019	39.90
LAR- 9	(A.A.)	3	.0300	.0000	3	.0300	.0000	A	6	.0300	.0000	.00
LAR-10	(COLOP.)	3	.0048	.0001	3	.0048	.0001	A	6	.0048	.0001	1.08
LAR-10	(A.A.)	3	.0051	.0002	3	.0050	0.0000	A	6	.0051	.0001	2.39
LAR-11	(COMPL.)	2	.0135	.0007	2	.0130	0.0000	A	4	.0133	.0005	3.77
LAR-12	(A.A.)	3	.0060	0.0000	3	.0060	0.0000	***R**	6	.0060	0.0000	0.00
LAR-13	(A.A.)	3	.0077	.0006	3	.0073	.0006	A	6	.0075	.0005	7.30
LAR-17	(SPECTR.1)	3	.0020	0.0000	3	.0027	.0006	A	6	.0023	.0005	22.13
LAR-17	(SPECTR.2)	3	.0047	.0006	3	.0057	.0006	A	6	.0052	.0008	14.57
TOTAL									54	.0103	.0120	116.83

TWO-SIGMA LIMITS -.013761 AND .034354

THE FOLLOWING LIE OUTSIDE THESE LIMITS

LAR- 3 (A.A.) MEAN = .055000

TABLE 3

Estimation of statistical parameters for SL-1
(After rejection of outliers)

	SiO ₂	CaO	MgO	Al ₂ O ₃	Total Fe as FeO	S	TiO ₂	MnO	Na ₂ O	K ₂ O	P ₂ O ₅	V ₂ O ₅	Cr ₂ O ₃
No. of Participating Laboratories	14	20	21	14	13	14	12	14	11	12	9	5	5
No. of Sets of Results	18	25	27	17	18	17	16	18	12	13	9	6	9
No. of Observations	106	183	196	102	114	101	100	114	68	72	55	40	52
Median %	35.75	37.47	12.20	9.62	0.90	1.26	0.37	0.85	0.39	0.50	0.020	0.004	0.006
Mean %	35.73	37.48	12.27	9.63	0.92	1.26	0.38	0.86	0.39	0.51	0.019	0.004	0.009
95% Confidence Limits of the Mean, %													
Low	35.61	37.30	12.12	9.56	0.89	1.23	0.36	0.83	0.34	0.47	0.008	0.003	0.002
High	35.86	37.65	12.41	9.71	0.96	1.28	0.39	0.89	0.43	0.54	0.030	0.006	0.015
Certification Factor	2.1	2.9	3.0	1.7	2.2	2.9	4.4	4.3	7.9	5.4	8.5	5.6	15.4

TABLE 4

Recommended values for reference blast-furnace slag, SL-1

	% SiO ₂	% CaO	% MgO	% Al ₂ O ₃	Total Fe as FeO	% S
Recommended Value	35.73	37.48	12.27	9.63	0.92	1.26
95 % Confidence Limits						
	Low	35.61	37.30	12.12	0.89	1.23
	High	35.86	37.65	12.41	0.96	1.28

TABLE 5

Provisional Values for TiO₂ and MnO in SL-1

	TiO ₂	MnO	Na ₂ O	K ₂ O
Provisional Value	0.38	0.86	0.39	0.51