

**GEOLOGICAL
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**DEPARTMENT OF ENERGY,
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PAPER 70-16

**A BUFFERING AND STANDARD ADDITION TECHNIQUE
AS AN AID IN THE COMPREHENSIVE ANALYSIS OF
SILICATES BY ATOMIC ABSORPTION SPECTROSCOPY**

(Report, 2 figures and 7 tables)

A. E. Foscolos and R. R. Barefoot



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ABSTRACT

Silicate standards were fused with boric acid-lithium carbonate flux and then dissolved in dilute nitric acid. The chemical and/or ionization interferences of calcium, magnesium and potassium which occur in atomic absorption spectroscopy are overcome by the combined use of buffering and standard addition (spiking). The method provides an exact measure of the reduced interferences and minimizes the use of expensive lanthanum compounds. The results compare favourably with those obtained on the same samples by analysts using other techniques.

A BUFFERING AND STANDARD ADDITION TECHNIQUE AS AN AID IN THE COMPREHENSIVE ANALYSIS OF SILICATES BY ATOMIC ABSORPTION SPECTROSCOPY

INTRODUCTION

Since the introduction of atomic absorption spectroscopy in analytical chemistry by Walsh (1955), the method has become a rapid and precise technique for the elemental analysis of silicates.

Initially, the difficult decomposition of silicates was accomplished by acid digestions or alkali fusions (Katz, 1968). More recently, the simpler lithium metaborate fusion technique introduced by Ingamells (1966) has been expounded and improved by Shapiro (1967), Van Loon & Parrissis (1968), Medlin & Suhr (1969), Omang (1969), and Boar & Ingram (1970). Once the sample is in solution, atomic absorption spectroscopy can be used to determine the elemental composition.

However, as with all other methods of analysis, there are problems which must be overcome; two of the most troublesome are the chemical and ionization interferences of some elements (Dickson, R.E., and Johnson, C.M., 1966). Lanthanum nitrate added to the solution can be used to counter chemical interferences and potassium chloride can be used to counter ionization interferences (Perkin-Elmer Corp., 1968). Also, the temperature of the flame can be increased to overcome chemical interferences, but this tends to increase ionization interferences.

The degree of success of these countermeasures should be checked by comparing the results with those obtained by the use of other methods. To compare the results with those obtained by other analysts using the same method, results only in duplication of possible errors. If no results are available for comparison, as is the case for many analyses, the assumption used is that the countermeasures were 100% effective; however this is not always the case.

An alternative procedure is the use of standard addition (spiking) to measure total interference from which accurate results can be calculated. It is advantageous to reduce chemical interferences before spiking the solution to obtain more precise results. This can be accomplished by adding a buffer to the solution. It is conventional to make the solution 1.0% lanthanum (Perkin-Elmer Corp., 1968), but this results in a marked change in the flame which must be countered by using standards also containing 1.0% lanthanum. Such large usage of costly lanthanum makes the analytical method commercially unattractive. The same results can be achieved by diluting the sample to one-tenth the concentration before making the solution up to 0.1% lanthanum. Thus the lanthanum-to-element ratio is the same as in the conventional method, with only one-tenth the amount of lanthanum used, and no apparent changes in the flame. The dilution also reduces ionization interferences. This combination of buffering and standard addition provides the resultant of the reduced chemical and ionization interferences from which the true elemental composition can easily be calculated.

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EXPERIMENTAL

APPARATUS. A Perkin-Elmer Model 303 atomic absorption spectrophotometer with a nitrous-oxide burner, a Boling burner and a digital concentration readout accessory (D.C.R. - 1) was used in this investigation. Perkin-Elmer hollow cathode tubes were employed, as were nitrous oxide - acetylene and air-acetylene flames. The burner heads were modified by cutting out the metal between the notches to the same height as the notches over the 90 degree span, thus enabling the burner head to be set at any angle within the 90° span. Instrument parameters are given in Table 1.

STANDARD SAMPLES. The following samples were obtained from F.J. Flanagan of the U.S. Geological Survey, Washington, D.C.: GSP-1 (granodiorite), AGV-1 (andesite), DTS-1 (dunite), PCC-1 (peridotite), BCR-1 (basalt) and G-2 (granite). The biotite standard was obtained from T. Hugi of the Mineralogisch-Petrographisches Institute, Universität Bern, Bern, Switzerland.

REAGENTS. The flux material was prepared by thoroughly mixing two moles of boric acid with one mole of lithium carbonate. The mixture was allowed to stand for a few hours and then placed in an oven at 110° C until dry and crumbly. The mixture was then crushed, blended and stored in a brown capped bottle.

The standards were prepared in the same manner as those of Boar & Ingram (1970), except that neither their flux mixture nor tartaric acid were used by the present authors.

Buffer solution A which contains 2,000 µg of La/ml or 0.2% La was prepared by dissolving 6.2346 grams of pure La (NO₃)₃ · 6H₂O in a 1,000 ml volumetric flask containing some double-distilled water, and diluting to volume.

Buffer solution B, which contains 2,000 µg of La/ml + 2 µg of K/ml + 2 µg of Ca/ml + 0.2 µg of Mg/ml, was prepared in the same way as buffer solution A, except that 2 ml of 1,000 µg of K/ml, 2 ml of 1,000 µg of Ca/ml, and 0.2 ml of 1,000 µg of Mg/ml were added before diluting to volume.

PROCEDURE. Exactly 0.2000 grams of each sample was weighed into a 50 ml platinum crucible, and one gram of the prepared flux was added. The crucible was then placed in a muffle furnace which was preheated to 1,000° C. After 15 minutes the crucible was removed and immediately swirled so that the smelt covered the sides of the crucible. The crucible was replaced in the furnace for five minutes, removed, swirled and plunged sideways into a 400 ml beaker containing 75 ml of 3% nitric acid, causing the smelt to shatter. The beaker was covered with a watchglass and placed on a shaker; it was then shaken so that the solution slapped the inside of the crucible. The watchglass and crucible were removed and washed thoroughly, collecting the washings in the beaker. The solution was then transferred quantitatively to a 200 ml volumetric flask and diluted to volume with double-distilled water. Thus the solution contained 1,000 µg of sample per ml.

The sample solution was then diluted using one part by volume of sample solution to three parts by volume of double-distilled water (4x dilution). Then, with this dilution and the original solution, silica, aluminum, iron, manganese and sodium were determined by atomic absorption spectroscopy (see Tables 1, 2, 3 and 7).

Buffering and standard addition was accomplished by adding buffer solution A to an equal volume of sample solution, and the same volume of buffer solution B to an equal volume of sample solution. Thus each of the two resulting solutions was diluted in a 1:1 ratio (2x dilution). When further dilutions were required, the sample was diluted before the buffering and standard addition technique was employed. Thus a dilution of 1:4 (5x dilution) which was then buffered and spiked became 1:9 (10x dilution), and similarly a dilution 1:49 (50x dilution) when buffered and spiked became 1:99 (100x dilution). The time in which the buffering and standard addition procedures could be completed was greatly reduced by the use of automatic pipettes. Calcium, magnesium and potassium were then determined from these solutions (see Table 1, 4, 5, 6 and 7).

The adsorbed water (H_2O^-) was obtained by heating one gram of the sample in an oven at 105°C until a constant weight was obtained. The loss in weight was attributed to adsorbed water (Maxwell, J.A., 1968).

The loss on ignition (L.O.I.) was obtained by heating the oven-dry sample in a muffle furnace at $1,000^\circ\text{C}$ until a constant weight was obtained. The weight loss from 105°C to $1,000^\circ\text{C}$ was the loss on ignition.

RESULTS AND DISCUSSION

The per cent composition of the major constituents of G.S.P.-1, A.G.V.-1, D.T.S.-1, P.C.C.-1, B.C.R.-1, G-2, and Biotite, fused with H_3BO_3 : Li_2CO_3 , are shown on Table 7. The data compare favourably with the mean average results obtained by twenty-six other analysts on the same rocks (Flanagan, F.J., 1969). Also, on Table 7 the loss on ignition is presented as a part of the per cent elemental composition to counterbalance the error resulting from the determination of per cent FeO and per cent Fe_2O_3 as total per cent Fe_2O_3 . This adds extra weight on the total per cent composition owing to extra oxygen obtained from the air to convert 2FeO to Fe_2O_3 . The increase in weight is offset by the smaller loss on ignition. Loss on ignition is the net result of two opposing reactions; loss of weight because of H_2O , SO_2 and CO_2 evolution, and gain of weight as a result of oxidation of ferrous to ferric.

With only one dilution, 4X, and the original sample solutions, silica, aluminum and titanium can be analyzed using a nitrous oxide-acetylene flame. With the same solutions, iron, manganese and sodium can be analyzed using an air-acetylene flame. Using the same flame, calcium, magnesium and potassium can be analyzed on sample solutions that are buffered and spiked.

No difficulties were encountered in the determination of silica, aluminum, titanium, iron, manganese and sodium. The expected ionization interference of titanium did not occur with the 4X diluted samples. The expected ionization interference of sodium also did not occur, probably as a result of the high potassium content of the samples.

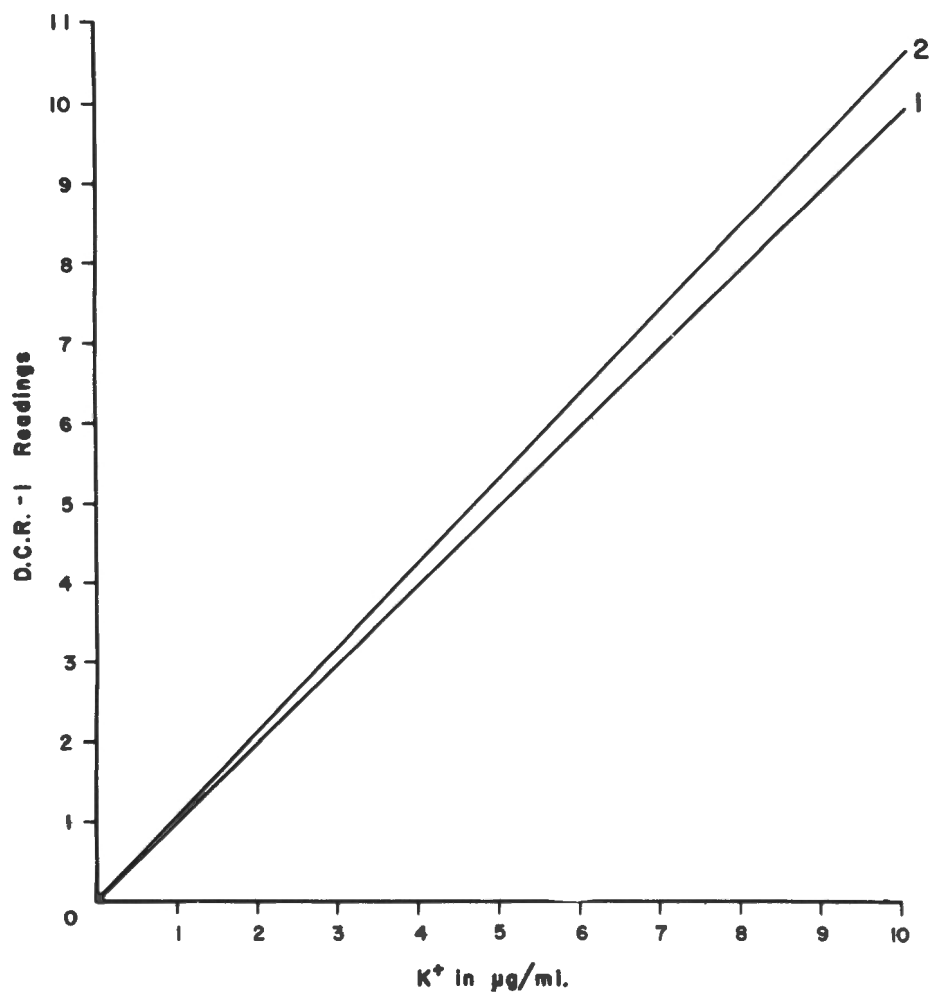


Figure 1. Effect of ionization interference on the standard line. Line 1 - standard line. Line 2 - standard line affected by ionization enhancement.

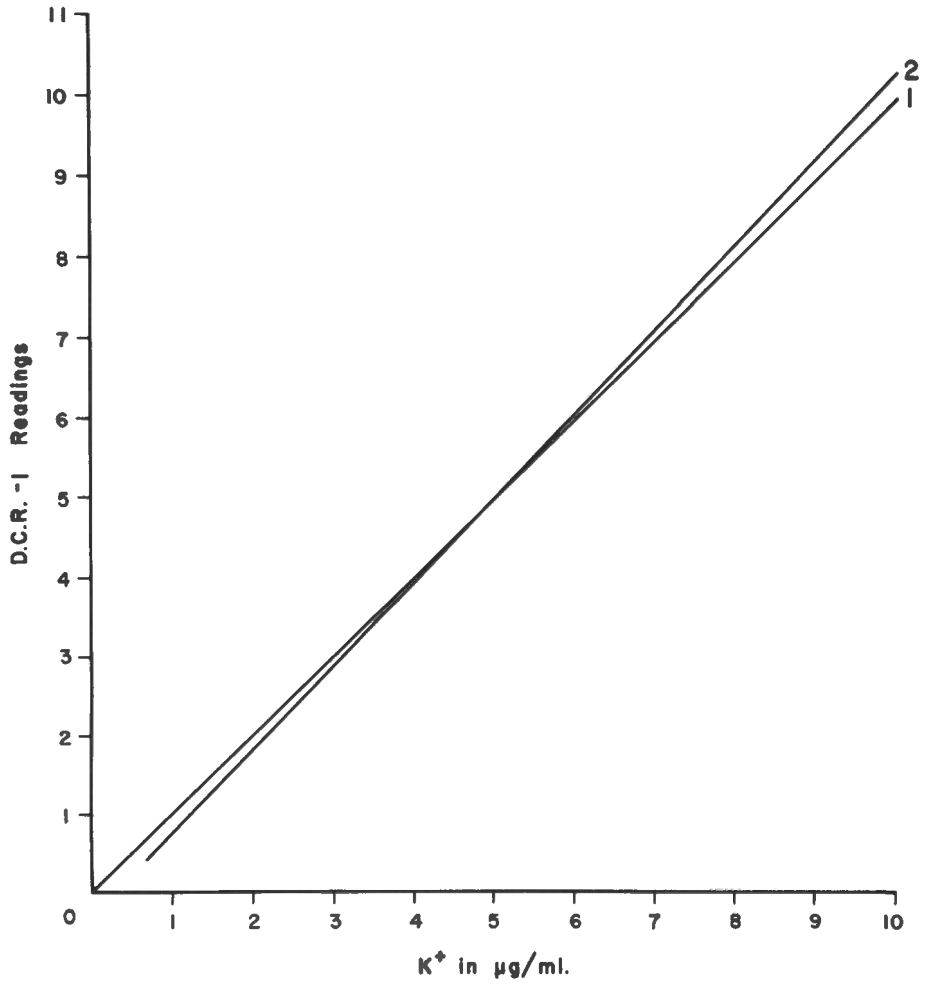


Figure 2. Effect of setting the mid-point of the standard line, affected by ionization enhancement, to read its actual concentration. Line 1 - standard line. Line 2 - standard line affected by ionization enhancement.

Chemical and/or ionization interferences of calcium, magnesium and potassium necessitated the use of the buffering and standard addition technique. The concentration found in the buffered sample solution is compared to the change in concentration found in the buffered and spiked sample solution that is, the change in absorbancy caused by the spike.

Thus:

$$\text{Concentration} = \frac{\text{Spike Added X Buffered Reading}}{\text{Buffered and Spiked Reading} - \text{Buffered Reading}}$$

$$\text{Concentration} = \frac{\text{Spike Added X Buffered Reading}}{\text{Spike Reading}}$$

Examples are given in Tables 4, 5 and 6. If the elemental content is high (over 10%) then the addition of a spike must be made on an extremely dilute solution which can cause erratic results since a small change in the DCR-1 reading means a large change in the equivalent per cent concentration of magnesium. In such a case, it would be advisable to take only the reading of the buffered solution (see Table 5).

When plotting the DCR-1 or absorbance values of potassium versus the true concentration values of the potassium standards, a straight line should be obtained, (Fig. 1, line 1). This theoretical line gives a concentration of 1 µg of an element /ml for every DCR-1 reading of 1 increment. However, owing to ionization interference, a line with a greater slope is obtained, (Fig. 1, line 2). Thus for every DCR-1 reading of 1 increment a concentration increment of more than 1 µg / ml is obtained. By setting the mid-point of the enhanced line 2 of Fig. 1 to read the true concentration, the lines 1 and 2 intersect (Fig. 2). As a result, standards with a concentration below the mid-point will read on the DCR-1 lower than their actual concentration, and the standards with a concentration above the mid-point will read on the DCR-1 higher than their actual concentration. To correct the discrepancy, a graph of DCR-1 readings versus actual concentrations must be drawn, similar to line 2, Fig. 2. The analytical results for potassium determination are shown on Table 6.

By buffering solutions with lanthanum, calcium, magnesium and potassium are dissociated from their compounds. If the reaction is complete, increasing the concentration of the solution by 1.0 µg/ml of the element will result in a concentration increase of 1.0 µg/ml in comparison with the buffered solution. However, the results of calcium, magnesium and potassium, Tables 4, 5 and 6 respectively, show that this is not the case. In Table 4, the buffered solution of G.S.P.-1 was found to contain 0.99 µg of Ca/ml. The same solution increased by 1.00 µg of Ca/ml was found to contain only 1.64 µg of Ca/ml, instead of the expected 1.99 µg of Ca/ml, a decrease of 0.35 µg/ml. As a result, spiking was required in addition to buffering because all the calcium apparently was not freed from its complexes.

Alkali metals because of their low ionization potentials, ionize more readily than other metals, even in low temperature flames. Ionization results in a smaller number of ground state atoms which are available for atomic absorption measurements. If another element which can supply free electrons is present in the flame, then there is an increased number of ions returned to the ground state. The result is a positive error or an increase in the absorption because of the increased number of ground state atoms. Although this type of interference is most serious in the determination of alkali metals it occurs also with alkaline earth metals

(Dickson, R.E., and Johnson, C.M., 1966). In Table 6, the buffered G-2 solution was found to contain 3.88 µg/ml K. The same solution increased by 1.00 µg/ml K was found to contain 4.95 µg/ml instead of the expected 4.88 µg/ml which is an apparent increase of 0.07 µg/ml.

Thus, the resultant error caused by chemical and/or ionization interferences can be either negative or positive. Since the error is a precise measure of the interference present, an accurately adjusted analytical result can be calculated from it. Also, with the buffering and spiking technique, the buffer does not have to be 100% effective in eliminating chemical interferences. This is not the case with the straight buffering technique where all of the chemical interferences must be removed. Large amounts of lanthanum are used with this latter technique and adjustments must be made to the sample solutions to attempt to overcome ionization interferences. Therefore, in summary, the technique of buffering and standard addition has the following advantages:

- (i) Overcomes all the effects caused by chemical interferences.
- (ii) Overcomes all the effects caused by ionization interferences.
- (iii) Reduces the amount of lanthanum used to 1/10.

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TABLE 1: INSTRUMENT PARAMETERS

Element	Wave-length A	Wave-length Setting	Wave-length Range	* Lamp Current ma	** Slit Opening mm	Burner Setting	Burner Position	Flame ***	Fuel	Air or N ₂ O	Set Concentration	Detection Limit µg/ml	Spike and Buffer	Dilution Sample: Diluent
Si ****	2516	252	U. V.	30	0.3	1 1/2	Parallel	N ₂ O-CHCH	5 1/2	5 1/2	5840	25 - 100	No	1: 3 (4x)
Al	3093	309	U. V.	25	1.0	1 1/2	Parallel	N ₂ O-CHCH	5 1/2	5 1/2	0800	20 - 100	No	1: 3 (4x) None(1x) None(1x)
Ti	3643	364	U. V.	40	0.3	1 1/2	Parallel	N ₂ O-CHCH	5 1/2	5 1/2	3000	2 - 25	No	1: 3 (4x) None(1x) None(1x)
Fe	2483	248	U. V.	30	0.3	1 1/2	Perpendicular	Air-CHCH	9	9	8851	5 - 40	No	1: 3 (4x)
Mn	2795	279	U. V.	30	1.0	1	Parallel	Air-CHCH	9	9	7530	0.5 - 10	No	None(1x)
Na	5890	295	VIS.	15	1.0	1	Perpendicular	Air-CHCH	9	9	1500	3 - 30	No	1: 3 (4x)
Ca	4227	211	VIS.	20	1.0	1.0	Parallel	Air-CHCH	9	7 1/2	4640	0.3 - 20	Yes	1: 1 (2x) 1: 9 (10x) 1:99 (100x) 1: 9 (10x) 1:99 (100x) 1: 1 (2x) 1: 9 (10x) 1:99 (100x)
Mg	2852	285	U. V.	20	3.5	1 1/2	30°	Air-CHCH	9	8	4360	0.3 - 3.0	Yes	1: 1 (2x) 1: 9 (10x) 1:99 (100x) 1: 1 (2x) 1: 9 (10x) 1:99 (100x)
K*****	7665	383	VIS.	25	1.0	1 1/2	Parallel	Air-CHCH	9	9	5542	0.3 - 10	Yes	1: 1 (2x) 1: 9 (10x) 1:99 (100x)

Note:

**Wavelength range - U. V. (Ultraviolet), VIS. (Visible)

**All lamps were hollow cathode

***Boling burner was used for all air acetylene flames

****Detection limit for Si (25 to 200 µg/ml) \approx SiO₂ (50-200 µg/ml)

*****Potassium was run with filter "ir"

TABLE 2
SILICA (SiO₂), ALUMINUM (Al) AND TITANIUM (Ti)
CONTENTS IN SILICATE STANDARD ROCKS AND MINERALS

Sample	Total µg SiO ₂ /ml	%SiO ₂	Total µg Al/ml	% Al	Total µg Ti/ml	% Ti
Blank	0	0.00	0	0.00	0	0.00
GSP - 1	672	67.20	80	8.00	4	0.40
AGV - 1	592	59.20	92	9.20	7	0.70
DTS - 1	408	40.80	2	0.20	0	0.00
PCC - 1	424	42.40	5	0.50	0	0.00
BCR - 1	536	53.60	74	7.40	14	1.40
G - 2	688	68.80	82	8.20	3	0.30
Biotite	368	36.80	96	9.60	18	1.80

TABLE 3
IRON (Fe), MANGANESE (Mn), AND SODIUM (Na)
CONTENTS IN SILICATE STANDARD ROCKS AND MINERALS

Sample	Total µg Fe/ml	% Fe	Total µg Mn/ml	% Mn	Total µg Na/ml	% Na
Blank	0	0.00	0.03	0.00	1.7	0.17
GSP - 1	30.0	3.00	0.22	0.02	21.2	2.12
AGV - 1	48.4	4.84	0.61	0.06	32.4	3.24
DTS - 1	62.8	6.28	0.99	0.10	0.1	0.01
PCC - 1	59.6	5.96	0.94	0.09	0.2	0.02
BCR - 1	95.6	9.56	1.33	0.13	24.4	2.44
G - 2	18.4	1.84	0.18	0.02	30.8	3.08
Biotite	96.8	9.68	0.69	0.07	2.1	0.21

TABLE 4
CALCIUM CONTENT (CaO) IN SILICATE STANDARD ROCKS AND MINERALS

Sample	DCR - 1 Reading µg/ml B* : B+S**	Concen-*** tration µg/ml	Corrected µg/ml (blank)	Dilution Factor	Total µg/ml	% Ca
Blank	0.14 : 0.63	0.286	0.296	2X	0.59	0.06
Blank	0.10 : 0.79	0.145	0.145	10X	1.45	0.15
Blank	0.02 : 0.56	0.037	0.037	100X	3.70	0.37
GSP - 1	0.99 : 1.64	1.523	1.378	10X	13.78	1.38
AGV - 1	0.21 : 0.75	0.389	0.352	100X	35.20	3.52
DTS - 1	0.20 : 0.49	0.690	0.404	2X	0.81	0.08
PCC - 1	0.66 : 0.97	2.129	1.843	2X	3.69	0.37
BCR - 1	0.29 : 0.84	0.527	0.490	100X	49.00	4.90
G - 2	0.94 : 1.56	1.516	1.371	10X	13.71	1.37
Biotite	0.10 : 0.73	0.159	0.014	10X	0.14	0.01

Note

* B = Buffered solution = 2,000 µg/ml La⁺³

** B + S = Buffered + Spike solution = 2,000 µg/ml La⁺³ + 2 µg/ml K⁺ + 2 µg/ml Ca⁺²
+ 0.2 µg/ml Mg⁺²

*** Concentration = Spike Added x Buffered Reading / Spike Reading
ie: AGV - 1 Concentration = $1.0 \times 0.21 / 0.75 - 0.21$
= 0.389 µg/ml

TABLE 5
MAGNESIUM CONTENT (MgO) IN SILICATE STANDARD ROCKS AND MINERALS

Sample	DCR - 1 Reading µg/ml B* : B+S**	**** Concen- tration µg/ml	Corrected µg/ml (blank)	Dilution Factor	Total µg/ml	% Mg
Blank	0.04 : 0.14	0.040	0.040	10X	0.40	0.04
Blank	0.00 : 0.10	0.000	0.000	100X	0.00	0.00
GSP - 1	0.60 : 0.70	0.600	0.560	10X	5.60	0.56
AGV - 1	0.93 : 1.03	0.930	0.890	10X	8.90	0.89
DTS - 1 ***	2.98 :	2.98	2.98	100X	298	29.80
PCC - 1 ***	2.58 :	2.58	2.58	100X	258	25.80
BCR - 1	0.21 : 0.32	0.191	0.191	100X	19.10	1.91
G - 2	0.47 : 0.56	0.522	0.482	10X	4.82	0.48
Biotite	0.98 :	0.98	0.98	100X	98	9.80

Note * B - Buffered solution = 2,000 µg/ml La⁺³

** B + S = Buffered + Spiked solution = 2,000 µg/ml La⁺³ + 2 µg/ml K⁺ + 2 µg/ml Ca⁺²
+ 0.2 µg/ml Mg⁺²

*** DTS - 1, PCC - 1, and Biotite were very high in Mg content and therefore best results are obtained from the buffered solution

**** Concentration = Spike Added x Buffered Reading / Spike Reading
ie: AGV - 1 Concentration = 0.10 x 0.93 / 1.03 - 0.93 = 0.93 µg/ml

TABLE 6
POTASSIUM CONTENT (K_2O) IN SILICATE STANDARD ROCKS AND MINERALS

Sample	DCR - 1 Reading $\mu\text{g/ml}$ $B^* : B+S^{**}$	Corrected $\mu\text{g/ml}$ (graph)	Concen- tration $\mu\text{g/ml}$ ***	Corrected $\mu\text{g/ml}$ (blank)	Dilution Factor	Total $\mu\text{g/ml}$	% K
Blank	0.38 : 1.47	0.51 : 1.58	0.48	0.48	2X	0.96	0.10
Blank	0.05 : 1.06	0.08 : 1.08	0.08	0.08	10X	0.80	0.08
Blank	0.00 : 1.01	0.00 : 1.05	0.00	0.00	100X	0.00	0.00
GST - 1	4.90 : 5.95	4.83 : 5.85	4.73	4.65	10X	46.5	4.65
AGV - 1	2.57 : 3.67	2.62 : 3.68	2.47	2.39	10X	23.9	2.39
DTS - 1	0.58 : 1.58	0.71 : 1.68	0.73	0.25	2X	0.50	0.05
PCC - 1	0.36 : 1.39	0.48 : 1.49	0.48	0.00	2X	0.00	0.00
BCR - 1	1.28 : 2.29	0.38 : 2.35	1.42	1.34	10X	13.4	1.34
G - 2	3.89 : 5.01	3.88 : 4.95	3.63	3.55	10X	35.5	3.55
Biote	7.06 : 8.07	6.91 : 7.85	7.35	7.27	10X	72.7	7.27

Note * B - Buffered solution = 2,000 $\mu\text{g/ml}$ La^{+3}

** B+S = Buffered + Spiked solution = 2,000 $\mu\text{g/ml}$ La^{+3} + 2 $\mu\text{g/ml}$ K^+ + 2 $\mu\text{g/ml}$ Ca^{+2}
+ 02 $\mu\text{g/ml}$ Mg^{+2}

*** Concentration = Spike Added x Buffered Reading / Spike Reading
ie: AGV - 1 Concentration = $1.0 \times 4.83 / 5.85 = 4.83$
= 4.73 $\mu\text{g/ml}$

TABLE 7
A COMPARISON BETWEEN THE PER CENT ELEMENTAL COMPOSITION OF SILICATE ROCKS AND MINERALS
OBTAINED BY $H_3BO_3 \cdot 14 CO_2$ FUSION TO THE MEAN AVERAGE PER CENT ELEMENTAL COMPOSITIONAL
OF THE SAME SILICATE ROCKS AND MINERALS OBTAINED BY TWENTY-SIX OTHER ANALYSTS

Sample	% SiO_2	% Al_2O_3	% TiO_2	% Fe_2O_3	% MnO	% Na_2O	% CaO	% MgO	% K_2O	% H_2O^-	*** % L. O. I.	Total %
GSP - 1	67.20	15.12	0.67	4.29	0.03	2.86	1.93	0.93	5.60	0.12	1.26	100.01
*	67.20	15.23	0.65	4.36	0.04	2.88	2.07	0.96	5.55	0.09	1.26	99.69
AGV - 1	59.20	17.38	1.17	6.80	0.08	4.37	4.93	1.48	2.88	0.94	1.20	100.43
*	58.96	17.10	1.05	6.81	0.10	4.33	4.96	1.52	2.92	1.10	1.20	100.05
DTS - 1	40.80	0.38	0.00	8.98	0.13	0.01	0.11	49.41	0.06	0.02	-0.26	99.64
*	40.62	0.42	0.01	8.64	0.13	0.04	0.15	49.76	0.03	0.07	-0.26	99.61
PCC - 1	42.40	0.94	0.00	8.52	0.12	0.03	0.52	42.78	0.00	0.22	4.48	100.01
*	41.94	0.82	0.00	8.28	0.12	0.03	0.49	43.21	0.02	0.44	4.48	99.83
BCR - 1	53.60	13.98	2.34	13.67	0.17	3.29	6.86	3.17	1.61	0.68	0.18	99.55
*	54.23	13.65	2.24	13.57	0.18	3.31	7.01	3.31	1.68	0.86	0.18	100.22
G - 2	68.80	15.48	0.50	2.63	0.02	4.16	1.92	0.80	4.28	0.06	0.65	99.30
*	69.04	15.43	0.50	2.71	0.03	4.15	2.02	0.75	4.54	0.11	0.65	99.93
Biotite	36.80	18.14	3.00	13.84	0.09	0.28	0.02	16.25	8.76	0.47	2.49	100.14
**	37.50	17.8	2.50	13.39	0.10	0.4	0.1	16.5	9.0	3.8% Total H_2O		99.9

Note: * Mean average per cent elemental composition determined by twenty six analysts (Flanagan, 1968).
 ** The biotite values for comparison were those determined by the University of Basle, Switzerland.
 *** L. O. I. is the loss on ignition: Flanagan data do not include L. O. I. Top values were used to determine Total %.