Reprinted from Smithsonian Contributions to the Earth Sciences,
No. 22, R. F. Fudali, Ed., 1979.

Electron Microprobe Reference Samples for Mineral Analyses

Eugene Jarosewich, Joseph A. Nelen, and Julie A. Norberg

ABSTRACT

A table is presented containing compositional data for 25 minerals, four natural glasses, and one synthetic glass prepared and analyzed for use as microprobe reference samples at the Smithsonian Institution. The table includes new chemical analyses of minerals and some updated analyses of minerals published previously.

Detailed descriptions of sample preparation and evaluation of homogeneity are given.

Introduction

Microprobe analyses are an essential part of present-day mineralogical and petrological studies. It can be said that the application of the microprobe to mineral studies and material sciences in general is one of the most significant advances since the first use of the petrographic microscope in the middle of the last century. The technique is now well established, widely used, and capable of high-quality analyses. As with all comparative instrumental techniques, however, it requires well-characterized reference samples. Prime prerequisites for microprobe reference samples are homogeneity at the micrometer level and availability in reasonable quantities for standard chemical analyses. Either prerequisite is usually easily satisfied by itself but together are difficult to achieve.

One of the problems with some minerals used as microprobe reference samples is a lack of proper documentation. Even if well-described minerals are from the same locality and/or are obtained from a reliable source, they may vary in chemical composition. Therefore, a mineral sample intended as a reference sample should be carefully selected and used only when analytical data on this particular specimen are available. Since natural materials fulfilling all the above requirements are not always available, synthetic minerals and glasses have occasionally been prepared as substitutes. Again, homogeneity of these materials should be checked and chemical analyses performed. The assumption that the precalculated composition is correct is certainly not always valid.

In general, the most reliable microprobe analyses are obtained when a reference sample of composition and structure close to that of the unknown is used because the matrix and possible wavelength shift effects are minimized, and only small corrections are needed. It is generally accepted that, regardless of the type of correction used, results corrected by more than 10 percent should be viewed with caution. Difficulties with correction procedures in the Si-Al-Mg system have been pointed out by Bence and Holzwarth (1977). Similar discrepancies have been observed by other probe users.

All minerals and glasses described here, except one, are of natural origin. Most have been obtained from the Smithsonian collections and were selected either in conjunction with specific projects or for use in silicate analyses in general.

ACKNOWLEDGMENTS.—We wish to acknowledge those curators and others listed in Table 1 who provided us with samples for use as microprobe reference samples. Brian Mason's careful examination and assistance in separation of minerals is also greatly appreciated.

Preparation of Reference Samples

When a sufficient quantity (at least 2 g) of a mineral or glass is available for use as a microprobe

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reference sample, a thin section is prepared for microscopic examination. Next, a microprobe analysis for composition and homogeneity is performed. If preliminary results are favorable, the material is gently crushed, sized usually between 20 and 80 mesh, and further purified using either a heavy liquid separation or a Franz magnetic separator or both. In some instances cleaning with a suitable acid is also useful. As a final step, the material is examined under a low-powered microscope and most remaining foreign grains are removed by hand. The purified grains are again checked by microprobe for homogeneity (sigma ratios) within and among grains (Table 2). Finally, a chemical analysis using classical methods (Peck, 1964; Hillebrand et al., 1953) is performed on the same separate that is to be used as the reference sample.

Discussion

In Table 1 are presented the data for newly analyzed minerals, earlier published analyses, and updated analyses for several minerals that have been in use for some time. Johnstown meteorite hypersthene and Springwater meteorite olivine have been re-analyzed using what we believe to be much cleaner separates. Kakanui hornblende has been re-analyzed for TiO_2.

Even after the most careful preparation of the reference sample, a grain of accessory mineral or matrix may remain in the sample, which, in the course of preparation of the standard discs, could be included with the reference sample. Occasional grains of the standard itself will be "off composition," due to inhomogeneity. These problems can never be totally eliminated. The user should be aware of the possible presence of such "impurities" and make a thorough check for them. For example, occasional grains are found that are lower in sodium and higher in potassium than usual in the reference sample microcline, lower in manganese than usual in Rockport fayalite, and lower in sodium than usual in Lake County plagioclase. Infrequent inclusions in the glasses 72854, 111240/52, 113498/1, and 113716 are also found.

The overall homogeneity of each sample was determined using the criteria given by Boyd et al. (1967) whereby the sample is considered to be homogeneous if the sigma ratio (homogeneity index) of observed standard deviation (sigma) to the standard deviation predicted from counting statistics alone does not exceed 3. The sigma ratios were calculated with reference to ten ten-second counts on each of ten randomly selected grains. Table 2 gives sigma ratios for the ten grains of each reference sample for major and some minor elements. The values in parentheses indicate the worst sigma ratio observed for an element in a single grain. This does not, however, imply a single worst grain, as different grains may exhibit differing degrees of homogeneity for each element present. When the criterion of sigma ratios is used as a measure of homogeneity, all the reference samples prove to be very homogeneous provided a reasonably large number of counts are taken on a reasonably large number of grains. In practice, however, fewer counts and grains are normally used for standardization, and under these circumstances a grain having a slightly different composition may influence the microprobe results adversely. For this reason, grains showing some discrepancy in composition should be avoided. The percentages of these "impurities" in the whole samples are minimal and the effects on the bulk analyses of the samples are negligible.

These samples were prepared in only small quantities, but they can be judiciously made available to microprobe users interested in the analysis of geologic materials. Potential users should remember that the purified samples differ in bulk chemistry from the specimens from which they were separated and should be very specific in their requests—i.e., the requests should be made for *microprobe standard* USNM no. *n* rather than simply material from specimen USNM no. *n*.

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KEY TO TABLE 1
Analysts, Sources, References

Analysts:

1. E. Jarosewich, Dept. of Mineral Sciences, Smithsonian Institution
2. J. Nelen, Dept. of Mineral Sciences, Smithsonian Institution
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5. B. Wiik, Geological Survey, Finland
6. U.S. Geological Survey, Geochemistry and Petrology Branch
7. D. Mills, X-Ray Assay Laboratories, Ontario, Canada; J. Nelen; J. Norberg
8. E. Kiss, Dept. of Geophysics and Geochemistry, Australian National University
9. J. J. Fahey and L. C. Peck, U.S. Geological Survey

Sources:

1. P. Desautels, J. S. White, Jr., and P. J. Dunn, Dept. of Mineral Sciences, Smithsonian Institution
2. B. Mason, Dept. of Mineral Sciences, Smithsonian Institution
3. G. Switzer, Dept. of Mineral Sciences, Smithsonian Institution
4. W. G. Melson, Dept. of Mineral Sciences, Smithsonian Institution
5. T. L. Wright, U.S. Geological Survey
6. H. Staudigel, Massachusetts Institute of Technology
7. R. S. Clarke, Jr., Dept. of Mineral Sciences, Smithsonian Institution
8. J. H. Berg, Northern Illinois University

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TABLE 1.—Chemical analyses of electron microprobe reference samples; analysts, sources, and references identified in "Key to Table 1" on facing page; these purified samples all differ, to greater or lesser degree, from the bulk chemistry of the USNM specimens from which they were separated (see text).

Mineral	SiO_2	Al_2O_3	Fe_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO	Cr_2O_3	H_2O	Total	Analyst	Source	Reference
Anorthite, Great Sitkin Island, AL USNM 137041	44.00	36.03		0.62	<0.02	19.09	0.53	0.03	0.03					100.33	1	1	
Anorthoclase, Kakanui, New Zealand USNM 133868	66.44	20.12		0.20		0.87	9.31	2.35					<0.05	99.29	3	2	
Apatite (Fluorapatite), Durango, Mexico[1] USNM 104021	0.34	0.07	0.06	0.00	0.01	54.02	0.23	0.01		40.78	0.01		0.01	99.94	4	1	2
Augite, Kakanui, New Zealand USNM 122142	50.73	7.86	3.69	3.45	16.65	15.82	1.27	0.00	0.74		0.13		0.04	100.38	5	2	3
Benitoite, San Benito County, CA[2] USNM 86539	43.75								19.35					100.15	2	1	
Chromite, Tiebaghi Mine, New Caledonia[3] USNM 117075		9.92		13.04	15.20	0.12					0.11	60.5		98.89	2	1	
Corundum, synthetic[4] USNM 6578		99.99												99.99	1	1	
Diopside, Natural Bridge, NY USNM 117733	54.87	0.11		0.24	18.30	25.63	0.34				0.04			99.53	2	1	
Fayalite, Rockport, MA USNM 85276	29.22		1.32	66.36					0.04		2.14		0.1	99.18	2	1	
Garnet, Roberts Victor Mine, South Africa USNM 87375	39.47	22.27	2.77	13.76	6.55	14.39			0.39		0.59		<0.01	100.19	1	3	4
Garnet, Roberts Victor Mine, South Africa USNM 110752	40.16	22.70	2.17	9.36	7.17	18.12			0.35		0.19		<0.01	100.22	1	3	4
Glass, Basaltic, Juan de Fuca Ridge USNM 111240/52 VG-2	50.81	14.06	2.23	9.83	6.71	11.12	2.62	0.19	1.85	0.20	0.22		0.02	99.86	1	4	5
Glass, Basaltic, Makaopuhi Lava Lake, HI USNM 113498/1 VG-A99	50.94	12.49	1.87	11.62	5.08	9.30	2.66	0.82	4.06	0.38	0.15		0.02	99.39	3	5	
Glass, Basaltic, Indian Ocean[5] USNM 113716	51.52	15.39	1.12	8.12	8.21	11.31	2.48	0.09	1.30	0.12	0.17		0.18	100.07	3	6	
Glass, Rhyolitic, Yellowstone Nat. Pk., WY[6] USNM 72854 VG-568	76.71	12.06	0.48	0.80	<0.1	0.50	3.75	4.89	0.12	<0.01	0.03		0.12	99.56	3	4	
Glass, Tektite, synthetic[7] USNM 2213	75.75	11.34	0.64	4.32	1.51	2.66	1.06	1.88	0.50	0.00	0.11		0.10	99.88	6	7	
Hornblende, Arenal Volcano, Costa Rica USNM 111356	41.46	15.47	5.60	6.43	14.24	11.55	1.91	0.21	1.41	<0.01	0.15		1.21	99.64	1	4	5
Hornblende, Kakanui, New Zealand[8] USNM 143965	40.37	14.90	3.30	7.95	12.80	10.30	2.60	2.05	4.72	0.00	0.09		0.94	100.02	1	2	4
Hypersthene, Johnstown meteorite USNM 746	54.09	1.23		15.22	26.79	1.52	<0.05	<0.05	0.16		0.49	0.75	0.00	100.25	3	7	
Ilmenite, Ilmen Mtns., Miask, USSR[9] USNM 96189			11.6	36.1	0.31				45.7		4.77			99.40	7	1	
Magnetite, Minas Gerais, Brazil[10] USNM 114887			67.5	30.2										98.16	3	1	
Microcline, location unknown USNM 143966	64.24	18.30	0.00	0.04	0.03	0.02	1.30	15.14	0.01		0.04			99.12	8	3	
Olivine (Fo_{89}), San Carlos, Gila Co., AZ[11] USNM 111312/444	40.81			9.55	49.42	<0.05				0.00	0.14			100.29	1	2	
Olivine (Fo_{83}), Springwater meteorite USNM 2566	38.95			16.62	43.58						0.30	0.02	<0.05	99.47	3	7	
Omphacite, Roberts Victor Mine, So. Africa USNM 110607	55.42	8.89	1.35	3.41	11.57	13.75	5.00	0.15	0.37		0.10		0.02	100.03	1	3	4
Osumilite, Nain, Labrador USNM 143967	60.20	22.60		6.38	5.83	<0.03	0.39	4.00	0.18				0.02	99.60	1	8	
Plagioclase (Labradorite), Lake County, OR USNM 115900	51.25	30.91	0.34	0.15	0.14	13.64	3.45	0.18	0.05		0.01		0.05	100.17	9	1	1
Pyrope, Kakanui, New Zealand USNM 143968	41.46	23.73		10.68	18.51	5.17			0.47		0.28		<0.01	100.30	1	2	4
Quartz, Hot Springs, AR[12] USNM R17701	99.99													99.99	1	1	
Scapolite (Meionite), Brazil[13] USNM R6600-1	49.78	25.05		0.17		13.58	5.20	0.94					0.21	99.86	2	1	

[1] SrO 0.07; RE_2O_3 1.43; ThO_2 0.02; As_2O_3 0.09; V_2O_5 0.01; CO_2 0.05; SO_3 0.37; F 3.53; Cl 0.41; sub-total: 101.52; O equivalent to Cl, F = 1.58; final total: 99.94.

[2] BaO 37.05.

[3] Total Fe reported as FeO.

[4] Emission spectrometric analysis: Si 0.03; Fe 0.003; Mg 0.007; Ca 0.003; Na 0.005; K 0.005.

[5] S 0.12; sub-total: 100.13; O equivalent to S = 0.06; final total: 100.07.

[6] Cl 0.13; sub-total: 99.59; O equivalent to Cl = 0.03; final total: 99.56.

[7] CO_2 not determined (insufficient sample); Cl 0.00; F 0.01. Synthetic glass prepared by Corning Glass Company.

[8] New TiO_2 value: 4.72.

[9] Nb_2O_5 0.92.

[10] Preliminary values: MgO 0.05; TiO_2 0.16; MnO <0.01; Cr_2O_3 0.25.

[11] NiO 0.37.

[12] Emission spectrometric analysis: Al 0.0005; Fe 0.01; Mg 0.005; Ca 0.001; Na 0.001; K 0.0003.

[13] CO_2 2.5; SO_3 1.32; Cl 1.43; sub-total: 100.18; O equivalent to Cl = 0.32; final total: 99.86.

TABLE 2.—Sigma ratios (homogeneity indices) for all analyzed grains of each reference sample (sigma ratio for n grains = $\frac{\text{observed sigma for } n \text{ grains}}{\text{sigma predicted from counting statistics}}$; least homogeneous grain in parentheses; dashes = not evaluated)

Mineral	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO	Cr_2O_3
Anorthite	0.96 (1.51)	0.81 (1.26)			0.92 (1.23)						
Anorthoclase	1.09 (1.60)	0.79 (1.38)				1.11 (1.57)					
Apatite (Fluorapatite)					1.02 (1.51)				0.97 (1.51)		
Augite	0.99 (1.37)	0.97 (1.66)	0.84 (1.26)	0.94 (1.23)	1.00 (1.25)						
Benitoite	---										
Chromite		1.00 (1.47)	1.01 (1.66)	1.11 (1.50)							**1.12 (1.49)**
Corundum	---										
Diopside	1.07 (1.37)			0.97 (1.50)	0.95 (1.50)						
Fayalite	0.95 (1.46)		1.14 (2.32)							1.03 (1.58)	
Garnet, 87375	0.89 (1.26)	1.01 (1.42)	1.06 (1.41)	1.01 (1.49)	0.86 (1.11)						
Garnet, 110752	0.88 (1.32)	0.94 (1.28)	0.90 (1.20)	1.00 (1.34)	0.87 (1.47)						
Glass, 111240/52 VG-2	0.94 (1.10)	0.89 (1.11)	0.86 (1.13)	0.96 (1.61)	1.00 (1.27)	1.05 (1.31)					
Glass, 113498/1 VG-A99	0.94 (1.32)	1.10 (1.46)	1.07 (1.38)	0.92 (1.38)	0.93 (1.34)	1.15 (2.10)		0.97 (1.44)			
Glass, 113716	1.12 (1.42)	1.00 (1.30)	0.94 (1.34)	1.01 (1.36)	0.83 (1.19)	1.25 (2.59)					
Glass, 72854 VG-568	0.97 (1.61)	1.00 (1.47)				2.31 (3.45)	0.98 (1.36)				
Glass, 2213	1.05 (1.72)	0.87 (1.24)	1.01 (1.34)		1.05 (1.61)						
Hornblende, Arenal	1.07 (1.67)	0.97 (1.66)	1.12 (1.36)	1.11 (1.67)	1.01 (1.27)	0.98 (1.32)					
Hornblende, Kakanui	1.01 (1.38)	1.00 (1.24)	1.30 (1.67)	1.16 (2.38)	1.10 (1.73)	1.15 (2.15)	0.90 (1.29)	1.01 (1.49)			
Hypersthene	1.07 (1.55)		1.10 (1.37)	0.93 (1.27)							
Ilmenite			1.72 (3.60)					1.34 (1.98)		1.21 (1.53)	
Magnetite			0.84 (1.16)								
Microcline	0.94 (1.13)	1.04 (1.52)					1.09 (1.59)				
Olivine (Fo_{90}), San Carlos	0.81 (1.13)		0.90 (1.29)	1.00 (1.64)							
Olivine (Fo_{83}), Springwater	0.96 (1.42)		1.06 (1.51)	0.99 (1.12)							
Omphacite	0.89 (1.23)	0.95 (1.64)	0.96 (1.87)	0.91 (1.30)	1.02 (1.51)	0.99 (1.31)					
Osumilite	0.96 (1.90)	1.27 (1.89)	1.20 (2.19)	1.00 (1.70)			1.13 (1.64)				
Plagioclase (Labradorite)	1.09 (1.49)	0.95 (1.40)			1.04 (1.65)	0.91 (1.33)					
Pyrope	1.08 (1.46)	0.95 (1.20)	1.09 (1.59)	0.98 (1.21)	0.97 (1.18)						
Quartz	---										
Scapolite (Meionite)	0.99 (1.29)	0.95 (1.41)			0.91 (1.16)	0.96 (1.41)					

Sigma ratio for 10 grains = $\frac{\text{observed sigma for all grains}}{\text{sigma predicted from counting statistics}}$

Sigma ratio for least homogeneous grain (in parentheses) = $\frac{\text{observed sigma for this particular grain}}{\text{sigma predicted from counting statistics}}$

Reprinted from Smithsonian Contributions to the Earth Sciences,
No. 22, R. F. Fudali, Ed., 1979.

Microprobe Analyses of Four Natural Glasses and One Mineral: An Interlaboratory Study of Precision and Accuracy

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ABSTRACT

An interlaboratory study for precision and accuracy of electron microprobe analyses of four natural glasses and one mineral is reported in this compilation. The results obtained by the three participating laboratories are in good agreement with chemical analysis values (where available) for SiO_2, Al_2O_3, FeO, CaO, and K_2O ($\geq$ 2 wt %). One laboratory shows a considerable bias for Na_2O and a slight bias for MgO. Some results for MnO, K_2O ($\sim$0.2 wt %), and P_2O_5, considering their low concentrations, are in reasonably good agreement; others show scatter. In general, considering that different reference samples and operating parameters were used to obtain these analyses, the correlation of results is encouraging. On the basis of the available data, however, it is evident that the precision and accuracy of these results cannot be improved much without more elaborate data acquisition techniques.

Introduction

The need for accurate microprobe analyses of natural glasses is of great importance in the study of volcanic and deep sea rocks. Melson et al. (1977) briefly summarized this need, referring specifically to the analyses of glasses from the Deep Sea Drilling Project. Since a standardized method for analysis of such glasses is not available, each laboratory has developed its own techniques and has been using its own preferred reference samples for these analyses. It is rather remarkable that many of the results obtained on the same or similar samples, using different techniques and reference samples, are generally in good agreement. Occasionally, however, some of these results exceed accepted analytical errors. Since more than one institution may participate in the study of volcanic or deep sea rocks and the analytical data may be used interchangeably for petrologic studies, it is of utmost importance that the precision and accuracy of these analyses be determined. As a start in obtaining this type of data, a set of the same samples has been analyzed by three laboratories, each using its own standard operating parameters; these results are compared for precision and accuracy. The participating laboratories were: Massachusetts Institute of Technology (MIT), Smithsonian Institution (SI), and U.S. Geological Survey (USGS).

The data presented in this paper are meant to serve primarily as a general guide for the expected accuracy and precision of microprobe analyses of glasses; however, they can serve as a guide for such analyses of minerals as well.

ACKNOWLEDGMENTS.—We would like to acknowledge Drs. W. G. Melson, G. R. Byerly and T. L. Wright for their suggestions pertaining to this work and Dr. J. S. Huebner for revising the data. We would also like to express our appreciation to Ms. J. Norberg of the Smithsonian Institution for compilation and arrangement of the data.

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Experimental Procedure

One disc was prepared containing only one grain of Kakanui hornblende and only one grain of each

of the following natural glasses:

VG–2 (USNM 111240–52). Basalt with a few rare microphenocrysts of olivine and plagioclase in section, less than 1%. Chemical analysis, Table 4.

VG–A99 (USNM 113498–1). Basalt from Makaopuhi Lava Lake, Hawaii (Wright and Okamura, 1977). Microphenocrysts of plagioclase in section, about 1% or less. Chemical analysis, Table 4.

VG–999 (USNM 113155–614). High-titanium ferro-basalt from DeSteiguer dredge D6, Galapagos Spreading Center (Byerly et al., 1976). Phenocrysts of plagioclase, augite, and olivine. Microphenocrysts of plagioclase and augite, about 10%–15% in section.

VG–D08 (USNM 113154–557). Basalt from DeSteiguer dredge D5, Galapagos Spreading Center (Byerly et al., 1976). Phenocrysts of olivine, plagioclase, and minor spinel. No microphenocrysts in section.

This disc was carbon coated and sent to the three laboratories for microprobe analysis.

These four glasses were selected because they represent the approximate elemental ranges encountered in the study of natural basaltic glasses. Classical chemical analyses are available for Kakanui hornblende, VG-2, and VG-A99 for comparison with the microprobe results (see Table 1, Jarosewich, Nelen, and Norberg, this volume). Although no chemical analyses are available for VG-999 and VG-D08, these two samples were included in this study to obtain additional data on the precision among the microprobe analyses themselves. The same grain was analyzed by each laboratory to eliminate the variable of possible inhomogeneities between different grains of the same sample.

Since the primary purpose of this work was to obtain analytical values for the samples as they are obtained under the laboratories' normal operating conditions, the operating parameters (kv, μA, beam size, etc.) were not specified. The samples were analyzed employing the standard operating parameters and reference samples used in each laboratory (Tables 1, 2).

The precision was obtained by analyzing all samples using Kakanui hornblende as the reference sample. In this manner discrepancies due to the use of different reference samples were eliminated and any deviations could be ascribed to the instrumental parameters. Since Kakanui hornblende is not an ideal reference sample for obtaining accurate analyses of these glasses, the accuracy was determined by analyzing these samples using each laboratory's preferred reference samples and comparing results with the chemical analyses of these glasses. The analytical results were compiled in the following manner. (1) Each sample was analyzed several times using Kakanui hornblende as the reference sample; the number of individual analyses and the averages are given in Tables 5–7. (2) The samples were analyzed several times using each laboratory's preferred reference samples; the number of individual analyses, the averages, and the type of reference samples used in these analyses are also given in Table 5–7. (3) The uncorrected averages obtained using Kakanui hornblende as the reference sample are summarized in Table 3 for comparisons of precision; these results indicate the variation of the data due only to the instrumental variation. (4) Table 4 summarizes the corrected results obtained using each laboratory's preferred reference samples; comparisons of

TABLE 1.—Instrumental parameters used by participating laboratories

Laboratory	Instrument	Accelerating Potential (kV)	Sample Current (μA)	Beam Size (μm)	Counting Time
MIT	ETEC MAC 5	15	0.03	4	30 sec or 60,000 counts
SI	ARL-SEMQ 9 spectrometers	15	0.05	2, 30	Seven 10 sec counts for each analysis
USGS	ARL-EXM-SM	15	0.05	20	20 sec or 20,000 counts for each element

TABLE 2.—Analyses of reference samples used by participating laboratories

Reference sample	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
					MIT					
DJ35	56.88	8.82		12.10	16.83	5.36				
P-140	40.86		7.23	51.63						
AN-60	53.05	30.01			12.38	4.56				
MAC AP	0.05		0.03	0.01	38.82	0.18			17.86	
Orthoclase	64.39	18.58				1.14	14.92			
Di2Ti	54.36			18.26	25.38			2.00		
MnIlm			45.41	0.25				51.61		1.50
Coss	40.93		40.99			6.93		8.71		
					SI					
Kak hornblende	40.37	14.90	10.92	12.80	10.30	2.60	2.05	4.36		0.09
VG-2	50.81	14.06	11.83	6.71	11.12	2.62	0.19	1.85	0.20	0.22
Apatite	0.34	0.07	0.05		54.02	0.23			40.78	
Fayalite	29.22		67.54					0.04		2.14
					USGS					
Rhodonite	46.76	0.96	12.49	0.42	5.62					33.34
Garnet-Pyrope	41.45	23.50	10.76	18.80	5.09			0.51		0.33
Orthoclase	63.42	19.24	0.10		0.08	0.36	15.34		0.49	
Di2Ti	54.39			18.26	25.38			2.00		
Diopside-Jadeite	56.10	3.78		15.84	22.02	2.31				

these results with the chemical values indicate the accuracy of the microprobe results.

Discussion

Comparisons for precision of the results obtained using Kakanui hornblende as the reference sample (Table 3) indicate that careful microprobe analyses for all major elements, titanium, and high potassium ($\geq$2 wt %) produce relatively precise results. The results also indicate that minor elements and sodium vary considerably and more careful work must be done if the results for these elements are to be used for exacting petrological work. There is no pronounced bias in the results by any of the three laboratories for the major elements except for a slight bias in the magnesium results. There is a bias in the results for sodium, low potassium (~0.2 wt %), and phosphorus. The sodium and phosphorus results analyzed by laboratory 1 run consistently high. The potassium results by laboratory 2 run slightly high and by laboratory 3, low. The discrepancy for these three elements is significant because all elements were determined using the same reference sample, indicating that the techniques for acquiring the data should be examined.

Comparisons for precision of the results of microprobe analyses in which each laboratory's preferred reference samples were used (Table 4) also check favorably for the major elements, titanium, and potassium ($\geq$ 2 wt %); more important, these results are in excellent agreement with the values for the three samples analyzed by classical chemical methods, thus showing a high level of accuracy. The results for sodium show a much larger discrepancy than those given in Table 3; only the lowest values of the microprobe analyses check well with the chemically analyzed sodium values. Sodium frequently presents a problem in microprobe analysis and to demonstrate this problem, laboratory 2 analyzed sodium using a 2 μm beam and a 30 μm beam; the former gives somewhat lower values for some samples (Table 6, results in brackets), the latter gives acceptable values. Kakanui hornblende sodium results are not affected by the beam size. One of the possible explanations for the higher results of laboratory 1 and lower results of laboratory 2 with the small beam is the unique behavior of sodium in the electron beam for both the reference samples and the unknown samples. If for example a reference sample in which sodium is easily "volatilized" (accepted description of decreasing inten-

TABLE 3.—Summary of uncorrected data from microprobe analyses of the 5 samples, using Kakanui hornblende as the reference sample, for interlaboratory comparison of precision (figure in parentheses indicates number of analyses averaged: each analysis represents up to 10 individual point analyses; laboratory 1 = MIT, 2 = SI, 3 = USGS; data based on Tables 5–7)

Constituent	Lab	KH	VG-2	VG-A99	VG-999	VG-D08
SiO_2	1	40.45 (2)	51.07 (2)	52.49 (1)	52.27 (2)	50.17 (1)
	2	40.58 (4)	51.62 (4)	52.45 (4)	52.21 (4)	50.56 (4)
	3	40.31 (3)	51.05 (4)	52.54 (2)	51.88 (1)	50.36 (2)
Al_2O_3	1	14.88 (2)	14.69 (2)	13.23 (1)	13.61 (2)	16.60 (1)
	2	14.97 (4)	15.06 (4)	13.54 (4)	14.12 (4)	17.27 (4)
	3	14.98 (3)	14.85 (4)	13.13 (2)	14.41 (1)	16.71 (2)
FeO	1	10.85 (2)	11.96 (2)	13.57 (1)	13.79 (2)	9.00 (1)
	2	10.91 (4)	11.84 (4)	13.60 (4)	13.71 (4)	8.97 (4)
	3	10.81 (3)	11.78 (4)	13.46 (2)	13.66 (1)	8.88 (2)
MgO	1	12.83 (2)	7.02 (2)	5.29 (1)	5.98 (2)	9.36 (1)
	2	12.67 (4)	6.90 (4)	4.98 (4)	5.81 (4)	8.71 (4)
	3	12.92 (3)	6.87 (4)	4.99 (2)	5.57 (1)	8.70 (2)
CaO	1	10.20 (2)	11.12 (2)	9.38 (1)	10.39 (2)	12.48 (1)
	2	10.39 (4)	11.16 (4)	9.29 (4)	10.40 (4)	12.39 (4)
	3	10.24 (3)	11.01 (4)	8.80 (2)	10.26 (1)	12.13 (2)
Na_2O	1	2.74 (2)	2.90 (2)	2.91 (1)	2.89 (2)	2.77 (1)
	2	2.59 (2)	2.65 (2)	2.71 (2)	2.60 (2)	2.38 (2)
	3	2.67 (3)	2.78 (4)	2.71 (2)	2.66 (1)	2.40 (2)
K_2O	1	2.02 (2)	0.18 (2)	0.77 (1)	0.14 (2)	0.06 (1)
	2	2.05 (4)	0.20 (4)	0.83 (4)	0.17 (4)	0.08 (4)
	3	1.96 (3)	0.06 (4)	0.72 (2)	0.03 (1)	0.00 (2)
TiO_2	1	4.36 (2)	1.66 (2)	3.65 (1)	1.72 (2)	0.98 (1)
	2	4.38 (4)	1.67 (4)	3.70 (4)	1.74 (4)	1.01 (4)
	3	4.27 (3)	1.64 (4)	3.77 (2)	1.66 (1)	0.92 (2)
P_2O_5	1	0.09 (2)	0.24 (2)	0.44 (1)	0.22 (2)	0.16 (1)
	2	0.06 (2)	0.20 (2)	0.39 (2)	0.19 (2)	0.12 (2)
	3	0.08 (3)	0.20 (4)	0.42 (2)	0.17 (1)	0.12 (2)
MnO	1	0.07 (2)	0.16 (2)	0.16 (1)	0.17 (2)	0.16 (1)
	2	0.09 (2)	0.18 (2)	0.18 (2)	0.20 (2)	0.14 (2)
	3	0.19 (3)	0.25 (4)	0.22 (2)	0.13 (1)	0.16 (2)

sity) is used for standardization to analyze an unknown sample which does not show this volatilization effect, the sodium results for the unknown sample will be high. If on the other hand the sodium in the reference sample is "stable" and sodium in the unknown sample is easily volatilized, low results will be obtained. The use of a larger beam size may diminish this discrepancy.

Low potassium, manganese, and phosphorus results vary just as much as those in Table 3. It should be noted that phosphorus corrects up by 15%–20% in most cases, giving much higher values than those of the chemical analyses. These higher phosphorus corrections are evident with both the up-dated Bence-Albee (Albee and Ray, 1970) and Magic IV correction procedures. Both correction procedures performed by one of us (A. P.) give excellent agreement with each other for all elements (Table 5).

Since for this study some grains were analyzed up

TABLE 4.—Summary of corrected data from microprobe analyses of the 5 samples using each laboratory's preferred reference samples, along with chemical analyses of 3 of the samples, for interlaboratory comparison of precision and accuracy (figures in parentheses indicates number of analyses averaged; each analysis represents up to 10 individual point analyses; laboratory 1 = MIT, 2 = SI, 3 = USGS; Chem. = chemical analysis from Table 1, Jarosewich, Nelen, and Norberg, this volume; microprobe data based on Tables 5–7)

Constituent	Lab	KH	VG-2	VG-A99	VG-999	VG-D08
SiO_2	1	40.65 (5)	50.85 (1)	51.05 (4)	51.16 (1)	50.29 (1)
	2	40.72 (4)	50.72 (4)	51.22 (4)	51.41 (4)	50.18 (4)
	3	40.42 (2)	50.75 (1)	50.80 (1)	50.70 (1)	49.77 (1)
	(Chem.)	40.37	50.81	50.90		
Al_2O_3	1	14.61 (5)	13.81 (1)	12.59 (4)	13.06 (1)	15.80 (1)
	2	14.95 (4)	14.15 (4)	12.66 (4)	13.36 (4)	16.17 (4)
	3	14.76 (2)	13.98 (1)	12.80 (1)	13.54 (1)	15.44 (1)
	(Chem.)	14.90	14.06	12.97		
FeO	1	10.45 (5)	11.26 (1)	13.24 (4)	12.94 (1)	8.67 (1)
	2	10.93 (4)	11.79 (4)	13.47 (4)	13.68 (4)	8.96 (4)
	3	10.54 (2)	11.79 (1)	13.41 (1)	13.30 (1)	8.78 (1)
	(Chem.)	10.92	11.83	13.18		
MgO	1	12.95 (5)	7.01 (1)	5.24 (4)	5.99 (1)	8.88 (1)
	2	12.70 (4)	6.78 (4)	4.95 (4)	5.77 (4)	8.46 (4)
	3	13.03 (2)	7.02 (1)	5.16 (1)	6.13 (1)	8.78 (1)
	(Chem.)	12.80	6.71	5.18		
CaO	1	9.99 (5)	10.85 (1)	9.08 (4)	10.12 (1)	12.23 (1)
	2	10.47 (4)	11.14 (4)	9.28 (4)	10.36 (4)	12.41 (4)
	3	10.12 (2)	10.72 (1)	8.97 (1)	10.18 (1)	12.05 (1)
	(Chem.)	10.30	11.12	9.38		
Na_2O	1	2.80 (5)	3.17 (1)	2.81 (4)	3.06 (1)	2.79 (1)
	2	2.60 (2)	2.66 (2)	2.70 (2)	2.61 (2)	2.25 (2)
	3	2.72 (2)	2.75 (1)	2.73 (1)	2.57 (1)	2.39 (1)
	(Chem.)	2.60	2.62	2.73		
K_2O	1	2.10 (5)	0.20 (1)	0.82 (4)	0.14 (1)	0.07 (1)
	2	2.06 (4)	0.21 (4)	0.90 (4)	0.19 (4)	0.09 (4)
	3	1.94 (2)	0.18 (1)	0.76 (1)	0.04 (1)	0.09 (1)
	(Chem.)	2.05	0.19	0.80		
TiO_2	1	4.87 (5)	1.86 (1)	4.04 (4)	1.93 (1)	1.09 (1)
	2	4.40 (4)	1.91 (4)	4.05 (4)	1.96 (4)	1.14 (4)
	3	4.65 (2)	1.86 (1)	3.77 (1)	1.80 (1)	1.03 (1)
	(Chem.)	4.36	1.85	4.06		
P_2O_5	1	0.14 (5)	0.32 (1)	0.54 (4)	0.29 (1)	0.23 (1)
	2	0.07 (2)	0.23 (2)	0.46 (2)	0.22 (2)	0.15 (2)
	3	0.04 (2)	0.19 (1)	0.31 (1)	0.15 (1)	0.08 (1)
	(Chem.)	0.00	0.20	0.41		
MnO	1	0.10 (5)	0.22 (1)	0.19 (4)	0.29 (1)	0.18 (1)
	2	0.11 (2)	0.22 (2)	0.22 (2)	0.25 (2)	0.17 (2)
	3	0.06 (2)	0.23 (1)	0.19 (1)	0.20 (1)	0.13 (1)
	(Chem.)	0.09	0.22	0.19		

TABLE 5.—MIT laboratory individual analyses of Kakanui hornblende and 4 natural glasses using Kakanui hornblende as reference sample and also the laboratory's preferred reference samples

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
						Kakanui hornblende					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	40.39	14.69	10.99	12.57	10.13	2.71	2.03	4.34	0.10	0.04
	B-A Corr.	40.35	14.68	10.99	12.59	10.13	2.71	2.03	4.34	0.12	0.04
	Magic IV Corr.	40.35	14.68	10.99	12.59	10.13	2.71	2.04	4.35	0.12	0.05
	σ (5)	0.17	0.11	0.15	0.13	0.15	0.12	0.02	0.03	0.00	0.01
2	Uncorrected	40.51	15.07	10.70	13.09	10.27	2.77	2.00	4.37	0.08	0.10
	B-A Corr.	40.55	15.07	10.71	13.08	10.28	2.76	2.00	4.38	0.10	0.10
	Magic IV Corr.	40.56	15.08	10.70	13.07	10.28	2.75	2.00	4.37	0.12	0.10
	σ (6)	0.53	0.08	0.22	0.22	0.07	0.12	0.04	0.03	0.00	0.03
	Standards	DJ35	AN-60	P-140	P-140	DJ35	DJ35	Ortho	Di2Ti	MAC AP	MnIlm
4	Uncorrected	38.21	12.61	10.48	11.68	10.11	2.55	2.10	5.13	0.10	0.10
	B-A Corr.	40.29	14.45	10.37	12.98	9.92	2.87	2.05	4.94	0.12	0.11
	Magic IV Corr.	40.36	14.54	10.43	13.04	9.96	2.90	2.05	4.94	0.12	0.11
	σ (10)	0.34	0.08	0.27	0.26	0.14	0.09	0.02	0.08	0.00	0.03
5a	Uncorrected	38.92	12.66	10.34	11.58	10.20	2.55	2.16	5.05	0.11	0.12
	B-A Corr.	40.97	14.48	10.23	12.86	10.02	2.86	2.11	4.87	0.13	0.13
	Magic IV Corr.	41.07	14.57	10.29	12.88	10.05	2.89	2.10	4.87	0.15	0.12
	σ (5)	0.21	0.08	0.12	0.13	0.18	0.11	0.04	0.05	0.00	0.01
5b	Uncorrected	38.71	12.68	10.29	11.51	10.18	2.59	2.15	5.11	0.11	0.11
	B-A Corr.	40.75	14.50	10.18	12.78	9.99	2.91	2.10	4.92	0.15	0.12
	Magic IV Corr.	40.85	14.59	10.24	12.82	10.03	2.93	2.09	4.93	0.15	0.12
	σ (5)	0.32	0.08	0.13	0.17	0.13	0.08	0.04	0.05	0.00	0.03
	Standards	DJ35	AN-60	Coss.	DJ35	DJ35	DJ35	Ortho	Di2Ti	MAC AP	MnIlm
6	Uncorrected	38.32	12.83	10.26	12.38	10.18	2.49	2.16	5.03	0.11	0.04
	B-A Corr.	40.43	14.71	10.68	13.10	9.98	2.80	2.11	4.84	0.15	0.04
	Magic IV Corr.	40.52	14.80	10.60	13.18	10.02	2.83	2.10	4.85	0.15	0.04
	σ (10)	0.30	0.09	0.12	0.10	0.07	0.11	0.05	0.05	0.02	0.04
	Standards	DJ35	AN-60	Coss.	DJ35	DJ35	DJ35	Ortho	MnIlm	MAC AP	MnIlm
8	Uncorrected	38.71	13.00	10.35	12.34	10.23	2.29	2.16	4.20	0.13	0.11
	B-A Corr.	40.82	14.89	10.77	13.05	10.04	2.57	2.11	4.79	0.16	0.11
	Magic IV Corr.	40.92	14.97	10.70	13.14	10.09	2.60	2.10	4.73	0.15	0.11
	σ (10)	0.19	0.09	0.18	0.15	0.10	0.07	0.05	0.05	0.00	0.03
						VG-2					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	51.36	14.51	12.15	6.83	11.03	2.82	0.19	1.67	0.23	0.14
	B-A Corr.	49.98	14.03	12.15	6.89	11.03	2.84	0.20	1.67	0.30	0.14
	Magic IV Corr.	50.08	14.03	12.15	6.88	11.04	2.84	0.19	1.67	0.30	0.14
	σ (10)	0.51	0.13	0.14	0.08	0.08	0.11	0.00	0.02	0.02	0.04

Table 5.—Continued

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
2	Uncorrected	50.77	14.87	11.76	7.21	11.20	2.97	0.17	1.65	0.24	0.18
	B-A Corr.	49.52	14.39	11.77	7.26	11.20	2.99	0.17	1.66	0.31	0.18
	Magic IV Corr.	49.61	14.39	11.76	7.25	11.21	2.99	0.17	1.65	0.33	0.18
	σ (10)	0.41	0.19	0.18	0.10	0.07	0.12	0.01	0.03	0.00	0.04
	Standards	DJ35	AN-60	P-140	P-140	DJ35	DJ35	Ortho	Di2Ti	MAC	AP MnIlm
5	Uncorrected	49.61	12.49	11.39	6.27	11.05	2.80	0.20	1.93	0.25	0.20
	B-A Corr.	50.85	13.81	11.26	7.01	10.85	3.17	0.20	1.86	0.32	0.22
	Magic IV Corr.	51.05	13.88	11.33	7.02	10.91	3.19	0.20	1.86	0.33	0.22
	σ (6)	0.83	0.13	0.14	0.06	0.08	0.11	0.00	0.07	0.02	0.03
						VG-A99					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
2	Uncorrected	52.49	13.23	13.57	5.29	9.38	2.91	0.77	3.65	0.44	0.16
	B-A Corr.	50.68	12.78	13.53	5.42	9.36	2.99	0.77	3.64	0.56	0.16
	Magic IV Corr.	50.84	12.78	13.55	5.41	9.37	2.99	0.77	3.64	0.56	0.15
	σ (10)	0.43	0.15	0.09	0.08	0.10	0.08	0.02	0.03	0.02	0.03
	Standards	DJ35	AN-60	P-140	P-140	DJ35	DJ35	Ortho	Di2Ti	MAC	AP MnIlm
4	Uncorrected	50.22	11.18	13.25	4.61	9.11	2.63	0.84	4.30	0.42	0.19
	B-A Corr.	51.03	12.36	13.06	5.24	8.91	3.05	0.82	4.12	0.54	0.20
	Magic IV Corr.	51.30	12.44	13.16	5.26	8.96	3.08	0.82	4.12	0.53	0.20
	σ (10)	0.28	0.09	0.19	0.10	0.06	0.08	0.01	0.05	0.02	0.03
5	Uncorrected	50.25	11.20	12.85	4.54	9.24	2.55	0.84	4.28	0.41	0.17
	B-A Corr.	51.00	12.34	12.68	5.16	9.04	2.95	0.83	4.10	0.53	0.18
	Magic IV Corr.	51.42	12.44	12.77	5.17	9.10	2.98	0.82	4.11	0.53	0.18
	σ (10)	0.49	0.08	0.18	0.07	0.08	0.15	0.02	0.05	0.02	0.03
	Standards	DJ35	AN-60	Coss.	DJ35	DJ35	DJ35	Ortho	Di2Ti	MAC	AP MnIlm
6	Uncorrected	50.32	11.50	13.11	4.88	9.29	2.24	0.83	4.27	0.43	0.18
	B-A Corr.	51.17	12.71	13.61	5.27	9.08	2.61	0.81	4.08	0.55	0.20
	Magic IV Corr.	51.43	12.78	13.53	5.31	9.14	2.63	0.81	4.09	0.56	0.19
	σ (5)	0.32	0.06	0.19	0.03	0.01	0.11	0.02	0.05	0.00	0.01
	Standards	DJ35	AN-60	Coss.	DJ35	DJ35	DJ35	Ortho	MnIlm	MAC	AP MnIlm
8	Uncorrected	50.09	11.73	13.09	4.88	9.51	2.24	0.85	3.41	0.42	0.16
	B-A Corr.	50.99	12.95	13.59	5.27	9.30	2.61	0.83	3.87	0.54	0.18
	Magic IV Corr.	51.26	13.03	13.50	5.30	9.36	2.63	0.83	3.83	0.53	0.18
	σ (10)	0.53	0.11	0.14	0.10	0.11	0.12	0.02	0.07	0.02	0.03

Table 5.—Continued

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
						VG-999					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	52.20	13.66	14.03	5.85	10.23	2.75	0.15	1.70	0.21	0.13
	B-A Corr.	50.64	13.24	13.99	5.97	10.21	2.82	0.15	1.69	0.27	0.13
	Magic IV Corr.	50.79	13.24	14.01	5.98	10.24	2.82	0.16	1.70	0.27	0.13
	σ (5)	0.34	0.17	0.20	0.05	0.07	0.09	0.00	0.03	0.02	0.01
2	Uncorrected	52.34	13.56	13.55	6.11	10.54	3.02	0.13	1.73	0.23	0.20
	B-A Corr.	50.77	13.15	13.52	6.24	10.52	3.09	0.13	1.73	0.30	0.20
	Magic IV Corr.	50.91	13.16	13.53	6.24	10.54	3.08	0.13	1.73	0.30	0.21
	σ (10)	0.68	0.42	0.17	0.07	0.08	0.09	0.00	0.03	0.00	0.03
	Standards	DJ35	AN-60	P-140	P-140	DJ35	DJ35	Ortho	Di2Ti	MAC	AP MnIlm
5	Uncorrected	50.07	11.80	13.11	5.30	10.33	2.66	0.14	2.01	0.22	0.27
	B-A Corr.	51.16	13.06	12.94	5.99	10.12	3.06	0.14	1.93	0.29	0.29
	Magic IV Corr.	51.42	13.16	13.03	6.02	10.17	3.09	0.14	1.94	0.30	0.29
	σ (10)	0.45	0.08	0.17	0.07	0.06	0.20	0.01	0.03	0.02	0.03
						VG-D08					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
2	Uncorrected	50.17	16.60	9.00	9.36	12.48	2.77	0.06	0.98	0.16	0.16
	B-A Corr.	49.25	16.02	9.04	9.20	12.53	2.70	0.07	0.99	0.21	0.16
	Magic IV Corr.	49.30	16.06	9.04	9.20	12.54	2.70	0.06	0.99	0.21	0.17
	σ (10)	0.43	0.55	0.09	0.18	0.08	0.09	0.01	0.03	0.00	0.03
	Standards	DJ35	AN-60	P-140	P-140	DJ35	DJ35	Ortho	Di2Ti	MAC	AP MnIlm
5	Uncorrected	48.68	14.35	8.73	8.13	12.41	2.55	0.07	1.12	0.18	0.17
	B-A Corr.	50.29	15.80	8.67	8.88	12.23	2.79	0.07	1.09	0.23	0.18
	Magic IV Corr.	50.43	15.86	8.72	8.88	12.29	2.81	0.07	1.08	0.24	0.18
	σ (10)	0.60	0.06	0.10	0.12	0.06	0.12	0.00	0.03	0.02	0.03

to 40 times to obtain good precision, it is reasonable to assume that, on the basis of the data in Table 3, the precision of microprobe analyses of basaltic glasses cannot be much improved without more elaborate data acquisition techniques. The standard deviations for various elements given in Tables 5–7 are of similar magnitudes for the three laboratories and they realistically indicate the expected scatter of analytical results. It is obvious that longer counts would not improve the precision very much and, indeed, extended counting times are impractical in day-to-day operation.

The accuracy, on the other hand, can be in some cases improved by the selection of suitable reference samples for a given unknown sample. From the data in Tables 5–7 it is evident that the microprobe results for some elements are in better agreement with the chemical results when reference samples other than Kakanui hornblende are used. The standard deviation here again is similar to that of the precision and thus could also be used as an indication of the expected scatter of the results. Analysis of elements below 0.1 wt % should be done with extra care and accepted with caution.

TABLE 6.—SI laboratory individual analyses of Kakanui hornblende and 4 natural glasses using Kakanui hornblende as reference sample and also the laboratory's preferred reference samples

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
						Kakanui hornblende					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	41.09	15.05	10.87	12.78	10.70	2.58	*1.98	4.33	0.05	
	B-A Corr.	41.09	15.02	10.88	12.75	10.72	2.58	1.98	4.34	0.07	
	σ (7)	0.49	0.23	0.26	0.43	0.26	0.08	0.08	0.12	0.02	
2	Uncorrected	40.45	14.83	11.11	12.60	10.22	2.60	2.13	4.45	0.06	
	B-A Corr.	40.46	14.82	11.11	12.60	10.24	2.59	2.13	4.45	0.07	
	σ (7)	0.28	0.24	0.27	0.38	0.04	0.07	0.07	0.11	0.02	
3	Uncorrected	40.03	14.95	10.88	12.79	10.22	2.60	2.04	4.43		0.09
	B-A Corr.	40.13	14.98	10.88	12.83	10.23	2.60	2.04	4.43		0.09
	σ (7)	0.38	0.21	0.12	0.26	0.07	0.02	0.05	0.04		0.01
4	Uncorrected	40.76	15.06	10.76	12.51	10.40	2.58	2.05	4.29		0.09
	B-A Corr.	40.76	15.02	10.77	12.49	10.42	2.58	2.05	4.30		0.09
	σ (7)	0.23	0.28	0.08	0.21	0.14	0.01	0.06	0.07		0.01
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	Fay
1	Uncorrected	41.09	15.05	10.87	12.78	10.70	2.58	1.98	4.33	0.05	
	B-A Corr.	41.09	15.02	10.88	12.75	10.72	2.58	1.98	4.34	0.07	
	σ (7)	0.49	0.23	0.26	0.43	0.26	0.08	0.08	0.12	0.02	
2	Uncorrected	40.45	14.83	11.11	12.60	10.22	2.60	2.13	4.45	0.06	
	B-A Corr.	40.46	14.82	11.11	12.60	10.24	2.59	2.13	4.45	0.07	
	σ (7)	0.28	0.24	0.27	0.38	0.09	0.07	0.07	0.11	0.02	
3	Uncorrected	40.44	15.00	10.95	12.77	10.28	2.62	2.03	4.51		0.11
	B-A Corr.	40.51	15.02	10.96	12.82	10.30	2.62	2.03	4.51		0.12
	σ (7)	0.17	0.20	0.14	0.17	0.11	0.04	0.04	0.09		0.03
4	Uncorrected	40.83	14.95	10.76	12.63	10.57	2.58	2.08	4.30		0.10
	B-A Corr.	40.83	14.92	10.76	12.61	10.60	2.57	2.08	4.30		0.10
	σ (7)	0.37	0.22	0.07	0.25	0.14	0.03	0.06	0.06		0.02
						VG-2					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	52.04	15.10	12.01	6.85	11.22	2.53	0.19	1.67	0.20	
	B-A Corr.	51.18	14.55	12.03	6.87	11.32	2.53	0.19	1.68	0.25	
	σ (7)	0.42	0.18	0.10	0.19	0.13	0.11	0.02	0.07	0.03	
2	Uncorrected	51.32	14.90	12.01	7.00	11.19	2.53	0.20	1.71	0.19	
	B-A Corr.	50.49	14.38	12.03	7.03	11.29	2.53	0.20	1.72	0.24	
	σ (7)	0.73	0.16	0.07	0.09	0.14	0.06	0.02	0.01	0.03	
3	Uncorrected	50.89	15.12	11.82	6.91	10.96	2.69	0.21	1.69		0.17
	B-A Corr.	50.19	14.64	11.83	6.98	11.06	2.69	0.21	1.70		0.17
	σ (7)	0.48	0.19	0.11	0.21	0.17	0.03	0.02	0.03		0.02

Table 6.—Continued

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
4	Uncorrected	52.21	15.11	11.53	6.83	11.28	2.60	0.20	1.62		0.18
	B-A Corr.	51.32	14.55	11.55	6.85	11.39	2.59	0.20	1.63		0.18
	σ (7)	0.43	0.19	0.09	0.12	0.14	0.03	0.03	0.05		0.02
	Standards	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	AP	Fay
1	Uncorrected	50.56	14.14	11.72	6.57	11.10	2.62	0.22	1.94	0.19	
	B-A Corr.	50.57	14.13	11.73	6.57	11.11	2.62	0.21	1.94	0.24	
	σ (7)	0.48	0.25	0.15	0.11	0.09	0.11	0.02	0.06	0.02	
2	Uncorrected	50.69	14.08	11.54	7.01	11.18	2.47	0.22	1.92	0.18	
	B-A Corr.	50.71	14.08	11.56	6.99	11.19	2.46	0.21	1.93	0.22	
	σ (7)	0.51	0.26	0.09	0.15	0.13	0.21	0.02	0.05	0.03	
3	Uncorrected	50.93	14.15	11.87	6.69	11.13	2.64	0.23	1.92		0.20
	B-A Corr.	50.99	14.16	11.88	6.70	11.13	2.65	0.22	1.92		0.22
	σ (7)	0.59	0.19	0.19	0.11	0.16	0.06	0.07	0.03		0.02
4	Uncorrected	50.52	14.21	11.99	6.83	11.13	2.66	0.22	1.85		0.19
	B-A Corr.	50.60	14.23	12.00	6.84	11.13	2.66	0.21	1.85		0.22
	σ (7)	0.61	0.24	0.11	0.08	0.18	0.05	0.03	0.07		0.02
		VG-A99									
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	52.56	13.72	13.90	4.89	9.30	2.33	0.84	3.66	0.37	
	B-A Corr.	51.39	13.19	13.88	4.97	9.36	2.37	0.84	3.66	0.45	
	σ (7)	0.45	0.12	0.08	0.20	0.08	0.10	0.12	0.06	0.04	
2	Uncorrected	52.76	13.51	13.59	4.98	9.34	2.22	0.82	3.68	0.41	
	B-A Corr.	51.53	12.99	13.57	5.06	9.38	2.26	0.83	3.68	0.51	
	σ (7)	0.29	0.22	0.06	0.11	0.18	0.11	0.04	0.03	0.04	
3	Uncorrected	51.65	13.47	13.62	5.07	9.07	2.72	0.80	3.81		0.18
	B-A Corr.	50.63	13.03	13.59	5.19	9.10	2.75	0.81	3.81		0.18
	σ (7)	0.66	0.27	0.12	0.13	0.17	0.03	0.02	0.10		0.02
4	Uncorrected	52.82	13.46	13.30	4.98	9.46	2.70	0.84	3.65		0.18
	B-A Corr.	51.63	12.96	13.29	5.06	9.50	2.75	0.85	3.66		0.18
	σ (7)	0.58	0.24	0.17	0.10	0.08	0.03	0.02	0.06		0.02
	Standards	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	AP	Fay
1	Uncorrected	51.08	12.68	13.48	4.78	9.18	2.42	0.93	4.05	0.38	
	B-A Corr.	50.78	12.65	13.45	4.83	9.13	2.47	0.90	4.02	0.47	
	σ (7)	0.75	0.23	0.08	0.06	0.04	0.17	0.06	0.09	0.04	
2	Uncorrected	51.71	12.65	13.42	4.95	9.36	2.27	0.91	4.12	0.37	
	B-A Corr.	51.38	12.62	13.39	5.00	9.32	2.32	0.88	4.09	0.45	
	σ (7)	0.28	0.20	0.26	0.44	0.45	0.10	0.04	0.11	0.01	

Table 6.—Continued

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
3	Uncorrected	51.77	12.74	13.39	4.83	9.38	2.64	0.94	4.05		0.19
	B-A Corr.	51.51	12.72	13.36	4.89	9.31	2.70	0.91	4.03		0.21
	σ (7)	0.90	0.22	0.11	0.11	0.12	0.03	0.04	0.17		0.02
4	Uncorrected	51.46	12.64	13.73	5.00	9.41	2.63	0.92	4.08		0.20
	B-A Corr.	51.21	12.65	13.69	5.07	9.35	2.69	0.89	4.05		0.22
	σ (7)	0.62	0.13	0.14	0.14	0.14	0.06	0.04	0.14		0.01
					VG-999						
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	52.46	14.22	13.91	5.71	10.59	2.31	0.16	1.71	0.19	
	B-A Corr.	51.47	13.69	13.89	5.77	10.66	2.35	0.16	1.71	0.23	
	σ (7)	0.70	0.29	0.11	0.11	0.19	0.11	0.03	0.06	0.00	
2	Uncorrected	52.17	14.04	13.76	5.96	10.43	2.32	0.17	1.75	0.18	
	B-A Corr.	51.20	13.54	13.75	6.03	10.50	2.36	0.17	1.75	0.22	
	σ (7)	0.77	0.18	0.13	0.06	0.03	0.07	0.02	0.08	0.02	
3	Uncorrected	51.89	14.01	13.59	5.83	10.10	2.60	0.18	1.75		0.19
	B-A Corr.	51.02	13.55	13.58	5.93	10.17	2.63	0.18	1.76		0.19
	σ (7)	0.61	0.07	0.03	0.15	0.13	0.01	0.03	0.07		0.02
4	Uncorrected	52.31	14.20	13.56	5.72	10.47	2.60	0.16	1.73		0.20
	B-A Corr.	51.35	13.69	13.55	5.80	10.54	2.63	0.16	1.73		0.20
	σ (7)	0.44	0.17	0.13	0.11	0.07	0.05	0.00	0.03		0.02
	Standards	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	AP	Fay
1	Uncorrected	51.64	13.32	13.53	5.57	10.30	2.48	0.21	1.93	0.18	
	B-A Corr.	51.52	13.30	13.51	5.62	10.28	2.52	0.20	1.93	0.22	
	σ (7)	0.58	0.13	0.17	0.10	0.11	0.11	0.02	0.06	0.03	
2	Uncorrected	51.77	13.35	13.59	5.85	10.43	2.38	0.16	2.00	0.18	
	B-A Corr.	51.67	13.34	13.56	5.89	10.42	2.41	0.16	1.99	0.22	
	σ (7)	0.64	0.22	0.20	0.16	0.10	0.17	0.02	0.04	0.03	
3	Uncorrected	51.46	13.37	13.75	5.66	10.33	2.54	0.20	1.97		0.21
	B-A Corr.	51.42	13.37	13.73	5.72	10.29	2.57	0.20	1.96		0.24
	σ (7)	0.38	0.10	0.07	0.17	0.15	0.09	0.03	0.09		0.02
4	Uncorrected	51.06	13.39	13.95	5.80	10.46	2.60	0.18	1.95		0.22
	B-A Corr.	51.04	13.42	13.92	5.86	10.43	2.65	0.18	1.94		0.25
	σ (7)	0.41	0.03	0.09	0.20	0.22	0.06	0.03	0.03		0.02

The data in Tables 5–7 contain a wealth of information for statistical evaluation that is beyond the scope of this paper. One point, however, should be emphasized: there is a general apprehension on the part of the staff responsible for the microprobe analyses regarding use of the observed standard deviation of a single day's analyses, based on one standardization, as a measure of precision. Since most microprobe users complete the analysis of a particular mineral within one day, this is often the

Table 6.—Continued

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
		VG-D08									
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	50.61	17.37	9.06	8.34	12.46	[2.34]	0.07	0.96	0.11	
	B-A Corr.	50.02	16.69	9.11	8.22	12.64	[2.28]	0.07	0.97	0.14	
	σ (7)	0.58	0.19	0.11	0.21	0.15	[0.06]	0.02	0.05	0.02	
2	Uncorrected	50.39	17.25	8.97	8.95	12.45	[2.31]	0.09	1.01	0.12	
	B-A Corr.	49.84	16.62	9.02	8.80	12.63	[2.24]	0.09	1.02	0.15	
	σ (7)	0.73	0.19	0.06	0.11	0.13	[0.03]	0.02	0.05	0.02	
3	Uncorrected	50.42	17.10	8.89	8.79	12.21	2.32	0.08	1.05		0.13
	B-A Corr.	49.90	16.51	8.94	8.68	12.39	2.25	0.08	1.06		0.14
	σ (7)	0.20	0.20	0.23	0.30	0.18	0.09	0.07	0.07		0.02
4	Uncorrected	50.83	17.36	8.97	8.77	12.45	2.44	0.07	1.00		0.15
	B-A Corr.	50.26	16.71	9.02	8.63	12.64	2.37	0.07	1.01		0.15
	σ (7)	0.31	0.13	0.06	0.24	0.04	0.06	0.02	0.03		0.03
	Standards	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	VG-2	AP	Fay
1	Uncorrected	49.37	16.09	8.85	8.36	12.25	[2.38]	0.09	1.13	0.11	
	B-A Corr.	49.63	16.06	8.89	8.20	12.33	[2.31]	0.09	1.14	0.14	
	σ (7)	0.46	0.13	0.12	0.12	0.05	[0.03]	0.02	0.06	0.03	
2	Uncorrected	50.22	16.05	8.89	8.99	12.24	[2.37]	0.09	1.11	0.12	
	B-A Corr.	50.49	16.04	8.93	8.81	12.33	[2.30]	0.09	1.12	0.15	
	σ (7)	0.37	0.20	0.26	0.44	0.45	[0.16]	0.01	0.02	0.01	
3	Uncorrected	50.08	16.30	8.91	8.45	12.39	2.30	0.09	1.14		0.16
	B-A Corr.	50.37	16.25	8.96	8.28	12.46	2.23	0.09	1.15		0.18
	σ (7)	0.56	0.21	0.20	0.11	0.17	0.04	0.02	0.05		0.01
4	Uncorrected	49.93	16.33	9.01	8.73	12.44	2.33	0.10	1.12		0.14
	B-A Corr.	50.22	16.31	9.05	8.56	12.51	2.26	0.09	1.13		0.16
	σ (7)	0.24	0.12	0.05	0.25	0.16	0.01	0.02	0.06		0.01

*Na_2O results in brackets, obtained using a 2μm beam, are not included in compilations of Tables 3 and 4. See explanation in text.

only measure of precision that can be observed. It is clear, however, that the average values of analyses made on two separate days, with separate standardizations, may occasionally be different. For example, in the analysis of Kakanui hornblende, one of us (A. P.) performed a Student's *t*-test for runs 1 and 2 (Table 5). Run 1 gives a mean for MgO of 12.57 with a standard deviation of 0.13 and run 2 gives a mean of 13.09 with a standard deviation of 0.22. The Student's *t*-test indicates that the difference between the two means (i.e., the error) is significant at the 99% confidence level. Of course, this is to be expected occasionally, given the limited stability of the instruments and the vagaries of data acquisition in general. For microprobe users, however, it is difficult to realize, and frequently even more difficult to accept, the fact that their data might not be as good as it appears. Concern about the precision and accuracy of microprobe analyses is ever-present with the discriminating worker. One way to monitor the

TABLE 7.—USGS laboratory individual analyses of Kakanui hornblende and 4 natural glasses using Kakanui hornblende as reference sample and also the laboratory's preferred reference samples

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
						Kakanui hornblende					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	40.43	14.93	10.50	13.02	10.66	2.68	1.89	4.31	0.09	0.08
	B-A Corr.	40.42	14.93	10.51	12.99	10.66	2.68	1.89	4.32	0.11	0.09
	σ (10)	0.28	0.40	0.14	0.28	0.49	0.13	0.12	0.18	0.02	0.12
2	Uncorrected	40.35	15.09	10.86	12.89	9.71	2.76	1.98	4.30	0.09	0.11
	B-A Corr.	40.39	15.10	10.86	12.89	9.72	2.76	1.98	4.30	0.11	0.11
	σ (10)	0.56	0.26	0.03	0.35	0.63	0.12	0.02	0.08	0.02	0.10
3	Uncorrected	40.14	14.91	11.06	12.84	10.34	2.58	2.02	4.20	0.06	0.37
	B-A Corr.	40.15	14.93	11.05	12.86	10.34	2.58	2.02	4.20	0.07	0.37
	σ (10)	0.64	0.45	0.19	0.17	0.08	0.07	0.05	0.13	0.05	0.41
	Standards	Di85	Ortho	Garnet	Di2Ti	Di2Ti	Di85	Ortho	Di2Ti	Ortho	Rhod
4a	Uncorrected	38.61	12.71	10.53	12.34	10.57	2.47	1.95	4.90	0.02	0.07
	B-A Corr.	40.73	14.48	10.50	13.13	10.20	2.68	1.91	4.74	0.02	0.07
	σ (5)	0.24	0.43	0.44	0.58	0.32	0.12	0.04	0.15	0.07	0.04
4b	Uncorrected	37.94	13.21	10.59	12.15	10.40	2.54	2.01	4.70	0.05	0.04
	B-A Corr.	40.11	15.04	10.57	12.93	10.04	2.76	1.97	4.55	0.05	0.04
	σ (10)	0.24	0.42	0.19	0.17	0.11	0.05	0.02	0.10	0.05	0.01
						VG-2					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	50.63	14.87	11.94	6.93	10.90	2.77	0.02	1.63	0.22	0.23
	B-A Corr.	49.58	14.46	11.95	6.98	10.91	2.79	0.02	1.63	0.27	0.23
	σ (10)	0.68	0.21	0.26	0.10	0.20	0.11	0.06	0.03	0.05	0.06
2a	Uncorrected	51.38	14.71	11.82	6.94	10.97	2.75	0.05	1.65	0.17	0.36
	B-A Corr.	50.25	14.30	11.83	6.98	10.98	.77	0.05	1.66	0.22	0.36
	σ (4)	0.77	0.43	0.30	0.10	0.28	0.09	0.07	0.08	0.05	0.03
2b	Uncorrected	51.00	14.75	11.77	6.88	10.94	2.85	0.05	1.66	0.22	0.32
	B-A Corr.	49.91	14.34	11.77	6.91	10.95	2.86	0.05	1.66	0.28	0.32
	σ (10)	0.48	0.40	0.19	0.18	0.21	0.15	0.05	0.07	0.05	0.05
3	Uncorrected	51.18	15.07	11.60	6.71	11.22	2.75	0.10	1.63	0.18	0.08
	B-A Corr.	50.05	14.61	11.61	6.73	11.24	2.75	0.10	1.63	0.21	0.08
	σ (5)	0.28	0.30	0.13	0.03	0.08	0.05	0.06	0.03	0.07	0.10
	Standards	Di85	Ortho	Garnet	Di2Ti	Di2Ti	Di85	Ortho	Di2Ti	Ortho	Rhod
	Uncorrected	49.19	12.65	11.82	6.55	11.11	2.55	0.19	1.92	0.20	0.21
	B-A Corr.	50.75	13.98	11.79	7.02	10.72	2.75	0.18	1.86	0.19	0.23
	σ (10)	0.42	0.19	0.14	0.05	0.06	0.12	0.06	0.08	0.05	0.05

Table 7.—Continued

Run No.	Conditions	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO
						VG-A99					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	52.39	13.25	13.39	4.89	8.44	2.80	0.77	3.81	0.40	0.27
	B-A Corr.	50.80	12.84	13.36	4.99	8.42	2.88	0.78	3.79	0.49	0.27
	σ (10)	0.51	0.30	0.15	0.10	0.48	0.20	0.11	0.10	0.04	0.05
2	Uncorrected	52.69	13.00	13.53	5.08	9.16	2.62	0.67	3.73	0.44	0.16
	B-A Corr.	50.69	12.62	13.50	5.18	9.13	2.69	0.67	3.71	0.54	0.16
	σ (6)	0.28	0.08	0.27	0.08	0.18	0.07	0.08	0.10	0.02	0.04
	Standards	Di85	Ortho	Garnet	Di2Ti	Di2Ti	Di85	Ortho	Di2Ti	Ortho	Rhod
	Uncorrected	49.58	11.61	13.48	4.76	9.33	2.44	0.77	3.91	0.32	0.18
	B-A Corr.	50.80	12.80	13.41	5.16	8.97	2.73	0.76	3.77	0.31	0.19
	σ (10)	0.60	0.26	0.23	0.10	0.17	0.08	0.07	0.12	0.04	0.06
						VG-999					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
3	Uncorrected	51.88	14.41	13.66	5.57	10.26	2.66	0.03	1.66	0.17	0.13
	B-A Corr.	50.62	13.98	13.63	5.65	10.25	2.72	0.03	1.66	0.21	0.13
	σ (10)	0.32	0.40	0.36	0.08	0.14	0.11	0.04	0.08	0.01	0.30
	Standards	Di85	Ortho	Garnet	Di2Ti	Di2Ti	Di85	Ortho	Di2Ti	Ortho	Rhod
	Uncorrected	49.37	14.24	12.75	5.67	10.57	2.30	0.04	1.87	0.15	0.20
	B-A Corr.	50.70	13.54	13.30	6.13	10.18	2.57	0.04	1.80	0.15	0.20
	σ (10)	0.09	0.04	0.21	0.10	0.03	0.03	0.00	0.17	0.11	0.04
						VG-D08					
	Standards	KH	KH	KH	KH	KH	KH	KH	KH	AP	KH
1	Uncorrected	50.30	16.38	8.75	8.86	12.03	2.44	0.00	0.88	0.13	0.18
	B-A Corr.	49.47	15.86	8.79	8.71	12.10	2.39	0.00	0.89	0.16	0.19
	σ (10)	0.73	0.47	0.22	0.18	0.66	0.04	0.00	0.03	0.01	0.01
3	Uncorrected	50.41	17.04	9.01	8.54	12.22	2.36	0.00	0.96	0.10	0.14
	B-A Corr.	49.60	16.46	9.05	8.40	12.29	2.30	0.00	0.97	0.12	0.15
	σ (5)	0.66	0.34	0.19	0.12	0.15	0.08	0.11	0.07	0.02	0.21
	Standards	Di85	Ortho	Garnet	Di2Ti	Di2Ti	Di85	Ortho	Di2Ti	Ortho	Rhod
	Uncorrected	47.97	14.02	8.76	8.36	12.42	2.23	0.09	1.05	0.08	0.12
	B-A Corr.	49.77	15.44	8.78	8.78	12.05	2.39	0.09	1.03	0.08	0.13
	σ (10)	0.24	0.17	0.18	0.07	0.03	0.07	0.06	0.05	0.02	0.01

precision and accuracy is to present data of samples with known composition together with the microprobe analyses of the unknown samples. This suggestion has been made frequently and is followed by many, but it would serve the scientific community much better if practiced even more extensively.

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