

Chemical Compositions and Critical Evaluation of Microprobe
Standards Available in the Reston Microprobe Facility

J. Stephen Huebner and Mary E. Woodruff
U. S. Geological Survey
Reston, Virginia 22092

U. S. Geological Survey Open File Report 85-718

This report is preliminary and has not been reviewed for conformity with U.S. Geological Survey editorial standards. Any use of trade names is for descriptive purposes only and does not imply endorsement by the USGS.

CONTENTS

Introduction -----	p.	2
Microprobe Analysis -----	p.	2
Nomenclature -----	p.	3
Mineral Formulas -----	p.	3
Evaluation of Analyses -----	p.	5
Numerical Ratings -----	p.	9
Choice of Standards -----	p.	9
Distribution of Standards -----	p.	9
Future Developments -----	p.	10
References -----	p.	11
Appendix I: List of Standards and Potential Standards -----	p.	12
Appendix II. Summary of Standards in Polished Mounts -----	p.	16
Appendix III: Numerical Evaluations of Standards -----	p.	20
Appendix IV: Summary of Documentation of Standards -----	p.	26

Introduction

Recent research in mineralogy and the emerging specialty of mineral physics has posed questions concerning chemical composition that cannot be answered with certainty using the laboratory reference materials generally available today. Mineralogist, minerals-physicists, and materials scientists want to measure the stoichiometry of the olivine, pyroxene, and oxide minerals because deviations from full site-occupancy of cations and anions or presence of interstitial ions are clues to the presence of point defects, on which the transport properties of these phases depend. Analytical chemists are striving to improve the mathematical algorithms which account for the effect of a mineral's matrix (the chemical and structural environment surrounding an atomic-species being analyzed) on the non-destructive X-ray analysis of a species. Also, petrologists and mineralogists are interested in the occurrence and distributions of minor and trace elements in minerals. When the mineral to be analyzed is present in minute quantity or is intermixed with other phases, the electron microprobe method is commonly the only practical method for the analysis. Many of the standards described in this report will be useful for this method.

Microprobe Analysis

Chemical analysis using the electron microprobe is a non-destructive x-ray spectrographic technique for determining the concentrations of elements in minute volumes (several cubic micrometers) of specimens of interest. With this technique, a finely focussed electron beam strikes the polished surface of the substance to be analyzed (the "unknown") and penetrates several micrometers in depth. Part of the energy of the impinging electrons is emitted as characteristic X-ray spectra of particular elements composing the mineral. The microprobe is equipped with spectrometers that permit examination of narrow regions of the X-ray spectrum, each characteristic of an element being analyzed.

Microprobe analysis is a relative technique in the sense that two sets of data are compared. The microprobe analysis procedure involves measuring the count rates per concentration unit of elements in standards (materials whose compositions are well known) and measuring count rates from the unknown samples. The concentration of an element in an unknown is proportional to the number of X-rays emitted by that element. Similarly, X-ray count rates are proportional to the concentrations of elements in standards. Elemental concentrations in unknowns are calculated using the count rates per concentration unit determined during standardization. Thus the analyses obtained can be no more accurate than the compositions of the standards.

Microprobe operators need a realistic assessment of the quality of the chemical analyses of the standards that they use. Previous lists of standards have included only the chemistry and a homogeneity index (Jarosewich et al., 1979). This report summarizes the information currently available to evaluate, for use as standards, materials available in the Reston microprobe laboratory and includes, wherever feasible, a critical evaluation of each standard using elementary principles of crystal chemistry that must

be met if the chemical analysis is to agree with what is currently known about the limits of composition and site occupancies of the analyzed phases.

Quantitative chemical analysis techniques, based largely on energy-dispersive x-ray spectrographic analysis, have recently become available for use with scanning and transmission electron microscopes. After proper sample preparation, most of the materials described in this report that qualify as microprobe standards will also serve as chemical analytical standards for electron microscopy. However, few microprobe standards have been examined with the fine spatial resolution available with electron microscopes. It is possible that some of the standards which appear homogeneous in the electron microprobe (resolution 2 to 3 micrometers) will prove to be heterogeneous when examined by electron microscopy (300-1000 angstrom resolution for chemical analysis).

Nomenclature

The standards described in this report have 4-letter mnemonic names that can be included in the computer code used to operate microprobes and incorporated in the compact format used to print the analytical results. The first one or two letters designate a mineral group or species:

AM amphibole	OX oxide
AP apatite	PX pyroxene
C carbonate	S sulfide
FS feldspar	SC scapolite
G glass	SP sphene
GT garnet	ST staurolite
M mica	ZO zoisite
OL olivine	

The remaining letters describe a particular standard. Most mnemonic codes are derived from the name that was commonly used before the 4-letter mnemonic scheme was introduced. For example, AMKH stands for the amphibole "Kakanui hornblende" and OLST designates the olivine "synthetic tephroite". Several standards are not members of large mineral groups; in such cases the first two letters refer to the mineral species (STBM is the staurolite from Berkshire, Massachusetts). The mnemonic codes are listed in Appendix I.

Mineral Formulas

Complete chemical analyses have been recalculated to mineral formula units following the standard scheme for such calculations (see Deer et al.,

1966, p. 515-518). All analyses were recalculated using the FORTRAN program MINCLC (Freeborn et al., 1985). MINCLC is a general-purpose program that permits the operator to specify any number of anions and cations as the basis of the formula unit. The routine will attempt to recalculate the analysis to the desired anion to cation ratio by adjusting the proportions of the reduced or oxidized states of the following multivalent elements:

MN (Mn ⁺²)	MC (Mn ⁺³)
FE (Fe ⁺²)	FC (Fe ⁺³)
CS (Cr ⁺²)	CR (Cr ⁺³)
TS (Ti ⁺³)	TI (Ti ⁺⁴)

As a general rule, if both ferric and ferrous iron were reported by the analyst, we present an unadjusted formula unit. If the analyst determined only FeO or Fe₂O₃, we list the adjusted formula unit. Some judgement is necessary in permitting the computer program to force the analysis to fit a preconceived formula stoichiometry by adjusting the oxidation states of analyzed elements. In favor of permitting adjustments is the fact that analyses of the concentrations of the oxidized and reduced species of an element are difficult and may be in error. On the other hand, minor adjustments, such as those that might be brought about by ordinary levels of error in chemical analyses, are trivial and not considered in this report. Adjustments that result in unreasonable site occupancies (for example, significant manganic ions in olivine) or in an incompatible oxidation state and geologic environment (for example, Ti⁺³ in a mineral from the earth's crust) are unreasonable.

Analysis of structural water (and fluorine and chlorine) in minerals is difficult and subject to large uncertainties. Where appropriate, an analysis was recalculated on both a hydrous basis (includes water and halogens) and an anhydrous basis (excludes water and halogens and assumes that all anions are oxygen), using the following formula units:

<u>Mineral Group</u>	<u>Formula Basis</u>	<u>Cations</u>	<u>Anions</u>
amphibole	hydrous	15-16	24
"	anhydrous	15-16	23
dioctahedral mica	hydrous	14	24
"	anhydrous	14	22
trioctahedral mica	hydrous	16	24
"	anhydrous	16	22
pyrophyllite	hydrous	12	24
"	anhydrous	12	23
zoisite	hydrous	8	13
"	anhydrous	8	12.5

Some mica and amphibole formulas depart markedly from the expected stoichiometry when recalculated on a hydrous basis, yet yield stoichiometric formulas when recalculated without water and halogens. In these cases, the weight percent values of water and halogens may simply be incorrect. If use of the anhydrous formula unit results in a superior formula unit, the analytical values for H₂O, F, Cl, and the redox species should be viewed with suspicion, but such suspicion does not necessarily prejudice the rest of the analysis. Many other analyses will yield the desired stoichiometry after conversion to an anhydrous formula unit and adjustment of the proportions of the oxidized and reduced ionic species of the multivalent elements. Evaluation of these cases is more difficult because the analytical error may be either an inaccurate determination of the oxidation state of a polyvalent element or an error in the concentration value of another element. Many micas and amphiboles cannot be recalculated to a formula with a simple cation:anion ratio and thus appear to have nonstoichiometric formulas. In amphiboles, specifically hornblende, the nonstoichiometry is likely to be caused by a partially filled A site in the structure. Non-stoichiometry in micas can have several different causes: solid solution between di-octahedral and tri-octahedral components, leading to excess cations (or vacancies) in the octahedral sites (Foster, 1960); substitution of oxy-components by a mechanism in which one highly charged cation substitutes for 1.5 or 2.0 less highly charged cations, leading to octahedral vacancies (or loss of hydrogen); and intimate inter-growths (interlayers) of alkali-poor compositions leading to unfilled alkali sites (e.g. chlorite-biotite).

The formal assignment of cations to structural sites of minerals that have more than one cation site was guided by the results of experimental determinations of site occupancies in crystals. These results have been summarized for amphiboles (Hawthorne, 1981), feldspars (Ribbe, 1983), garnets (Meagher, 1980), micas (Bailey, 1984; Deer et al., 1962), oxides (Rumble, 1976), sphene (Ribbe, 1980), and pyroxenes (Cameron and Papike, 1980).

Evaluation of analyses

The evaluations in this report focussed on our knowledge of the properties of materials rather than on effects brought about by microprobe operating procedures. We sought to identify chemical constituents that might be reported in error, major and some minor components that might not have been analyzed (data reduction schemes that incorporate a matrix correction require knowledge of the bulk composition of the standard), and heterogenous distributions of components within a material. Five criteria were used to evaluate the suitability of materials for use as standards: (1) the uncertainties inherent in the method used to obtain the analysis, (2) the chemical analysis itself, (3) the formula unit calculated from the analysis, (4) the homogeneity of the material, and (5) the existence of special problems such as very fine grain-size or presence of additional phases included within the grains. One could also have used as a sixth criterion the successful use of the mineral as a standard. We did not use this sixth criterion because success of a standard depends upon additional factors (instrument performance, perfection of polished surface, data reduction scheme, and operator technique) that vary from laboratory to

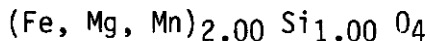
laboratory. Thus it is quite possible that a superbly analyzed and perfectly homogenous material could fail to give reproducible analytical results some of the time (instrumental instability, poor judgement by operator) or all of the time (data reduction scheme insufficient for the particular compositions in question). Conversely, it is possible for an operator to adjust the instrumental operating conditions or to modify the accepted chemical analysis of the standard so that the microprobe delivers the desired chemistry. (This practice leads to satisfying results because it compensates for possible absolute errors in the chemical analyses of standards and for the failures of existing correction schemes to account perfectly for matrix effects. Such satisfying results may be valid in a relative sense, but they cannot have absolute veracity unless tested by the analysis of independently analyzed materials, the subject of this report.)

(1) If the method of analysis is known, it may be possible to estimate the uncertainty associated with the reported values. For instance, mineral analyses performed by conventional rock and mineral analysis methods at the U.S. Geological Survey are reported to the nearest 0.01% and are commonly regarded as being accurate to 0.2% absolute if the constituent exceeds 30%, 0.1% absolute for constituents in the range 10-30%, and 0.05% absolute for constituents below 10% (Clark, 1974, p. 33). Routine rapid-rock-analysis methods are reported to the nearest 0.1% and should be accurate to the nearest 1% absolute for constituents present at greater than 30%, 2% relative for the range 10-30%, 0.1% absolute for the range 1-10%, and 0.02% absolute for constituents present at less than the 1% level (Clark, 1974, p. 35). Some purported standards have been analyzed only by microprobe methods; the uncertainty of these analyses relative to the standards used can be expected to be similar to the uncertainties encountered with the rapid-rock analysis methods. Surprisingly, a brief description of the method used to obtain an analysis is not always included as a part of the documentation provided us with the standard material.

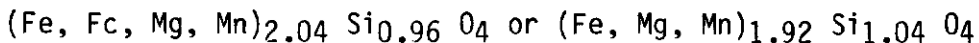
(2) The sum of a complete chemical analysis is ideally 100 percent, but significant departures from that value do not necessarily indicate a serious analytical error. For instance, not all instrumental methods can distinguish the oxidized and reduced states of an element. Reporting an oxidized state as the reduced state results in a low summation because the oxygen, which forms 50-67 atomic percent of common silicates and oxides, is almost never analyzed directly. Thus, pure Fe_2O_3 reported as FeO has a weight percent sum of 89.9 percent. Summations exceeding 100 percent occur when halogens or sulfur are present as anions, yet all cations are reported as oxides. In such cases, an amount of oxygen equivalent to the halogen or sulfur is subtracted from the analysis. For example, in the case of synthetic fluor-phlogopite (MFPH), $\text{KMg}_3\text{AlSi}_3\text{O}_{10}\text{F}_2$, the elements K, Mg, Al, and Si are all given as oxides leading, in the presence of 9.0% F, to a summation of 103.8% by weight. An amount of oxygen equivalent (on the basis of charge compensation) to the fluorine must be subtracted from the summation. In this case, in which oxygen has twice the charge but only 84.2% the atomic weight of fluorine, 3.8% by weight oxygen is subtracted, giving the phlogopite a revised total of 100.0% by weight.

Confirmation of analytical results by replication, preferably using a different analytical method, is the most informative line of evidence to be used in evaluating a standard. Unfortunately, many standards do not have a single complete chemical analysis, much less two complete analyses. In those few cases where two analyses are available, and they agree, one can be quite confident that the bulk compositions are well known. In a case where the replicate analyses disagree, one must try to decide objectively which analysis to use, in the process rejecting the other analysis(es). When an objective decision cannot be reached, the range of possible values must include both analyses. One is left with the distressing (and ironic) situation in which a standard for which there is no confirming analysis will appear to have less uncertainty than a standard for which there are two equally good (but different) values.

(3) It should be possible to calculate a reasonable formula unit from the chemical analysis. A reasonable formula has an appropriate cation:anion ratio and has ions distributed among unlike structural sites to give a plausible (formal) site occupancy. For example, current knowledge of crystal chemistry suggests that Si^{+4} is tetrahedrally coordinated in rock-forming silicates, and the tetrahedral site cannot accept large monovalent or divalent cations such as Na^+ , K^+ , Ca^{++} , Mg^{++} , Fe^{++} , or Mn^{++} , which commonly occur in octahedral or 8-fold coordination. Following this line of reasoning, we would conclude that



would be a reasonable olivine formula unit but that analyses leading to either



must include either components determined in error or an admixed phase.

(4) A good microprobe standard is chemically uniform at the scale of the volume excited by the beam. The homogeneity of each material was measured using operating conditions under which that material might be expected to be used as a standard, most commonly 15KV accelerating potential, 100nA beam current (approximately 10nA specimen current), and using a focussed beam spot (1-2 micrometers in diameter, leading to an activation volume with a diameter of 2 to 3 micrometers). No special care was taken to polish the materials; the homogeneities reported are for materials prepared as conventional microprobe standards - sets of many different standards in a polished brass block or simple polished grain mounts on a glass slides (see Appendix II). The degree of homogeneity is reported as the sigma ratio, the ratio of the observed degree of count rate variation to that predicted, on the basis of counting statistics, from the mean count rate. Counts obtained in 10 or 20 seconds were accumulated from each of 20 points that were distributed among as many grains as possible. In most cases, three elements were measured simultaneously. Elements that were measured together are reported on the same 3 lines in the data sheets. A background value, determined by averaging five 10-20 second counts on a pure oxide such as TiO_2 or SiO_2 , was subtracted

from each measurement. A perfectly homogenous material should give a sigma ratio of unity. We consider materials with sigma ratio values of 2.0 or less to be sufficiently homogenous for routine use as standards, materials with values of 2.0 to 3.0 to be slightly heterogenous and usable only with caution; and materials with values exceeding 3.0 to be unsuitable for use as standards. In terms of microprobe operating procedures, if the sigma ratio is close to unity, the operator need only measure and average count rates on 3 to 4 points during standardization; if the ratio is 2.0, counts from 5 points should be averaged; and if the value is 3.0, it would be advisable to measure and average at least 10 points, a lengthy procedure, or to choose a different standard.

In some cases, we report sigma ratios for fewer than 20 points because we rejected the counts from one or more points and recalculated the sigma ratios. For each rejected point, we obtained anomalous count rates from each of the elements being detected simultaneously. Anomalous points can come about from analysis of included phases, imperfections in the polished surfaces of the materials, or from analysis of a polished surface that is not located at the focal point of the X-ray and light optical systems. We do not disqualify a material from usage as a standard because one or more measured points were anomalous, but the subsequent operator who chooses that as a standard should bear in mind that special care may need to be taken to avoid anomalous points.

It is important to realize that our method for calculating sigma ratios differs from that of Jarosewich et al. (1979). They measured 10 points on each of 10 grains, averaged the 10 counts for each grain, then calculated the sigma ratio on the basis of the averaged counts for the 10 grains. This procedure will give a good estimate of the inter-grain compositional variation that is of concern when splitting crushed samples for bulk chemical analysis, but that procedure obscures the smaller scale, intra-granular spatial dependence of the composition that is of interest to microprobe operators. Our procedure is designed to reveal compositional heterogeneities on the scale of the X-ray activation volume of the electron beam and, as might be expected, our sigma ratios are commonly somewhat larger than those measured by Jarosewich et al. (1979) on splits of the same material.

(5) Some special problems adversely affect the performance of a material as a microprobe standard. One difficulty that plagues many synthetic materials is a grain size that is so small that the operator has difficulty locating the grains or that the excitation volume exceeds that of the grain. The standards OLSF, OLSST, and PXSE have this problem. Another problem pertains to the ability of the standard to take a polish in either a mount of many standards (with unlike polishing properties) or a solitary mount (where all grains have the same properties). Small, very hard grains such as magnetite (OXSM) develop such great topographic relief, relative to the surrounding epoxy medium, that there is only a small amount of flat surface area oriented perpendicular to the microprobe electron beam. The synthetic corundum OXSC, while coarser, is so tough that it tends to be plucked from the mount during polishing. Very soft materials such as fluor-phlogopite (MFPH) and pyrophyllite (PYNC) tend to smear rather than polish. Proper preparation of a well-polished mount containing such unlike phases is almost impossible. A final special problem concerns admixed phases. The glass and two oxides admixed with the synthetic cobalt

olivine, OLSC, present only a mild inconvenience for the operator because the olivine can be distinguished optically. But the distinction of the pyrox-mangite from contaminating olivine in mounts of PXHI, however, can be done only by monitoring the count-rate data, presenting a more serious problem for the operator because the erroneous data that have been collected must be recognized and then rejected.

Numerical Ratings

We assign a numerical score (Appendix III) to each standard and potential standard listed in Appendix I. The scheme for assigning positive and negative point values is given in Appendix IIIA. Although the relative importance assigned to various elements of the scheme is subjective, the decisions about individual criteria can be made objectively. Thus different individuals should be able to arrive at the same point values. It is important to avoid penalizing a standard more than once for each shortcoming. Thus, if an element was not determined and the summation is low, the numerical score should be reduced only once. Similarly, if the apparent poor homogeneity results from a poor polish, the standard should be penalized for one or the other, but not both. The more positive the score, the more dependable is the material for use as a microprobe standard. Standards with scores of 10 or greater qualify as "known unknowns" that can be used to check instrument standardization and operation. Such materials should be candidates for interlaboratory calibrations. Use of materials with scores less than 4 should be avoided. In some cases, low scores result from a lack of documentation and will improve as we learn more about the material.

Choice of Standards

The suitability of a substance for use as a microprobe standard depends to some extent on the purpose for which it will be used. When selecting standards, the microprobe operator must consider the uncertainty in the chemistry of the standard because the microprobe analysis can be no better than the analysis of each standard used to calibrate the microprobe. As a general rule, Reston microprobe operators want to obtain analyses of known-unknowns that reproduce the independently obtained analyses to within 1% absolute for oxides present at the 50% level, 0.2% for oxides present at the 5% level, and 0.1% absolute for oxides present at a level of 1% or less. We have designated standards with chemical analyses judged better than these limits for all major elements as standards suitable for use as known-unknowns; they have numerical ratings > 10 (Appendix III). Other materials are suitable for use only as standards (4-9), are unsuitable (< 3), or are potential standards whose eventual evaluations await further information (< 3).

Distribution of Standards

Communication among the users of standards is important if the documentation supporting a standard is to be updated to include the knowledge of the users. Thus, the documentation includes the names and addresses of individuals

and laboratories to which material has been sent, and incorporates the comments we receive from individuals who have used these materials (Appendix IV). Standards which originate within the Reston Microprobe Laboratory are available for distribution providing sufficient material exists; these standards are indicated with an asterisk in the list of standards (Appendix I). Standards which originate elsewhere may be obtained from the original source, listed on the data sheets. These materials will not be distributed by Reston staff because up-to-date documentation cannot be maintained.

Future Developments

The evaluations presented in this report may bring about changes in microprobe operating procedures. In the near term, the groups of standards used to obtain a multi-element analysis can be improved by making sure that they include only superior materials. By excluding materials of doubtful chemistry and homogeneity, we hope to achieve a reduction in the plethora of standard groups in common use in Reston. These efforts should also lead to the development of new standard blocks which, because they exclude inferior materials, can be made smaller and will surely be less misleading to operators.

Over the longer term, it may be possible to investigate systematically the effectiveness of data reduction schemes (matrix corrections) and the internal consistency of the various standards chosen to form a set of standards for a multi-element mineral analysis. One approach involves asking microprobe operators to check periodically their standardization by analyzing a "known-unknown" (a well characterized material); building a data file that includes analyses of "known-unknowns" and identifies the standards used, by using option 8 of program RDARL4 (Huebner, 1983); then applying multivariate statistical techniques to identify the strengths and weaknesses of individual standards and sets of standards.

References

- Bailey, S.W. (1984) Crystal chemistry of the true micas. In S.W. Bailey, Editor, *Reviews in Mineralogy*, Vol. 13, Micas, p. 13-60. Mineralogical Society of America, Washington, D.C.
- Cameron, Maryellen, and Papike, J.J. (1980) Crystal chemistry of silicate pyroxenes. In C.T. Prewitt, Editor, *Reviews in Mineralogy*, Vol. 7, Pyroxenes, p. 5-92. Mineralogical Society of America, Washington, D.C.
- Clark, J.R. (1974) *Manual of laboratory services*. U.S. Geological Survey, Office of Geochemistry and Geophysics, Geologic Division.
- Deer, W.A., Howie, R.A. and Zussman, Jack (1962) *Rock-Forming Minerals*, Vols. 1-5, Wiley, New York.
- Deer, W.A., Howie, R.A., and Zussman, J. (1966) An Introduction to the Rock Forming Minerals. New York: John Wiley and Sons. 528 pages.
- Foster, M.D. (1960) Interpretation of the composition of trioctahedral micas. U.S. Geological Survey Professional Paper 354-B, 49 pages.
- Freeborn, W.P., McGee, E.S., and Huebner, J.S. (1985) MINCLC: A FORTRAN program for recalculating mineral analyses. U.S. Geological Survey Open File Report 85-257, 45 pages.
- Hawthorne, F.C. (1981) Crystal chemistry of the amphiboles. In D.R. Veblen, Editor, *Reviews in Mineralogy*, Vol. 9A, Amphiboles, p. 1-102. Mineralogical Society of America, Washington, D.C.
- Huebner, J.S. (1983) RDARL4, a FORTRAN interface for transferring chemical data from an Applied Research Laboratories electron microprobe to a PDP-11 computing system. U.S. Geological Survey Open File Report 83-713, 40 pages.
- Jarosewich, E., Nelen, J.A., and Norberg, J.A. (1979) Electron microprobe reference samples for mineral analysis. Fudali, R.F., ed., *Smithsonian Contributions to the Earth Sciences*, No. 22, p. 68-72.
- Meagher, E.P. (1980) Silicate garnets. In P.H. Ribbe, Editor, *Reviews in Mineralogy*, Vol. 5, Orthosilicates, p. 25-66. Mineralogical Society of America, Washington, D.C.
- Ribbe, P.H. (1980) Titanite. In Ribbe, P.H., Editor. *Reviews in Mineralogy*, Volume 5, Orthosilicates, p. 137-154. Mineralogical Society of America, Washington, D.C.
- Ribbe, P.H. (1983) The chemistry, structure, and nomenclature of feldspars. In P.H. Ribbe, Editor, *Reviews in Mineralogy*, Vol. 2, Feldspar Mineralogy, p. 1-20. Mineralogical Society of America, Washington, D.C.
- Rumble, Douglas, III, Editor (1976) *Reviews in Mineralogy*, Vol. 3, Oxides. Mineralogical Society of America, Washington, D.C.

Appendix I
List of Standards & Potential Standards in Appendix IV

Amphibole

AMCM Cummingtonite, Mikoni River, NZ
AMEN Engel's Amphibole
AMKF Potassic Fluor-richterite, Synthetic
AMKH Kakanui Hornblende, NZ
AMMN Minnesota Hornblende, Fremont County, CO
AMSF Sodic Fluor-richterite, Synthetic

Apatite

APCL Chlorapatite, Synthetic
APFD Fluor-apatite, Durango, NM
APRE REE-apatite, Synthetic
APSF Fluorapatite, Synthetic

Carbonate

*CCHM Calcite, Harvard Museum
CCNM Calcite, National Museum
*CDAS Dolomite, Austria
*CDBS Dolomite, Binnetal, Switzerland
CDOS Dolomite, Oberdorf, Austria
*CRAP Rhodochrosite, Alma Park, NM
CSBH Siderite, Broken Hill, NSW
CSIG Siderite, Ivigtut, Greenland
CSTR Strontianite

Feldspar

*FSB0 Benson Orthoclase
*FSLC Plagioclase, Lake County, Oregon
*FSNA Nunivak Anorthoclase, AK
FSTA Tiburon Albite, CA

Glass

GD85 Di₈₅-Jd₁₅ Glass, Synthetic
*GFAB Albite Glass, Synthetic
*GFAN Anorthite Glass, Synthetic
*GFOR Orthoclase Glass, Synthetic
GLBA Barium Glass, Synthetic
*GLDI Diopside Glass, Synthetic
GLJF Basaltic Glass, Juan de Fuca
*GLL1 Lunar Glass (61156), Synthetic
*GLL7 Lunar Glass (77135), Synthetic
*GLL8 Lunar Glass (68415,85), Synthetic

Appendix I (continued)

Glass (continued)

GLMP Basaltic Glass, Makaopuhi, HW
*GLSI Silica Glass
GRE1 REE 1
GRE2 REE 2
GRE3 REE 3
GRE4 REE 4
*GRLS Rhyolite Glass, El Chichón, Mexico
GSDI Diopside Glass, Synthetic
GSEN Enstatite Glass, Synthetic
GSWO Wollastonite Glass, Synthetic
*GWOL Wollastonite Glass, Synthetic

Garnet

GTAL Garnet 12442
GTKN Kakanui Pyrope, NZ
GTRV Garnet, Roberts Victor Mine, S. Africa
GTSP Spessartite Garnet, Brazil

Mica

*MBLM Biotite, Lemhi, ID
MBPS Biotite, PSU, Libby, MT
MBST Biotite, Stillwater, MT
MFPH Fluor-phlogopite, Synthetic
*MMMT Muscovite, Methuen Township, Ontario
*MPAV Paragonite, Venezuela
*MPBO Phlogopite, Burgess, Ontario
*MSFP Fluor-phlogopite, Synthetic

Olivine

OLCO Cobalt Olivine - USNM
OLCR Cobalt Olivine - Robie
OLMJ Olivine, Marjahlatti, Finland
OLNI Nickel Olivine, Synthetic, USNM
OLRF Fayalite, Rockport, MA
OLSC Olivine, San Carlos, AZ
*OLSF Fayalite, Synthetic
OLSM Olivine, Susimaki Meteorite
*OLST Tephroite, Synthetic
OLSW Olivine, Springwater Meteorite

Appendix I (continued)

Oxide

OXAL Corundum, Synthetic
*OXBU Chromite, Bushveld Complex, S. Africa
OXCO Corundum
*OXGH Gahnite, Brazil
*OXHA Hausmannite, Synthetic
OXIL Ilmenite, Ilmen, USSR
OXMN Manganosite, Synthetic
OXMT Magnetite, Brazil
OXNC Nickel Oxide, Single Crystal
*OXPA Partridgeite, Synthetic
OXPE Periclase
*OXQZ Quartz, Brazil
OXRU Rutile, Synthetic
OXR1 Rare Earth Oxide, Synthetic
OXR2 Rare Earth Oxide, Synthetic
OXR3 Rare Earth Oxide, Synthetic
OXR4 Rare Earth Oxide, Synthetic
OXSB Synthetic Bunsenite, Polycrystalline
OXSC Synthetic Corundum, USNM
*OXSM Synthetic Magnetite
OXSP Spinel, Synthetic
*OXSZ Synthetic Zincite
*OXTB Tiebaghi Chromite, New Caledonia
OXUB Chromite, Union Bay, AK
OXVA Vanadium Oxide (V_2O_3), Synthetic
OX51 Chromite 55G-4, Stillwater Complex, MT
OX52 Chromite 55G-15AB, Stillwater Complex, MT

Pyroxene

PXAC Acmite, Synthetic
PXAD Adirondack Diopside, NY
*PXA6 Augite DL6, CA
*PXAG Aegirine, Bear Paw Mts., MT
*PXBH Rhodonite, Broken Hill, NSW
*PXBK Rhodonite, Bald Knob, NC
PXEN Enstatite, Synthetic
PXHD Hedenbergite, MN
*PXHI Pyroxmangite, Homedale, ID
PXHY Hypersthene R2467
PXJD Jadeite, New Idria, CA
PXJT Hypersthene, Johnstown, Meteorite
PXXA Augite, Kakanui, NZ
PXP1 Chrome Augite
PXPS Diopside, Pennsylvania State University
*PXSD Synthetic Diopside
PXSE Synthetic Enstatite
PXSW Enstatite, Shallowater, Meteorite
*PXWO Wollastonite, Mono Co., CA

Appendix I (continued)

Sulfide

- *SAS2 As₂S₃, Synthetic
- *SCDS CdS, Synthetic
- *SSB2 Sb₂S₃, Synthetic
- *SSNS SnS₂, Synthetic
- *SZNS ZnS, Synthetic Sphalerite

Other

- *ANDB Andalusite, Espirito Santo, Brazil
- *KYM Kyanite, Minas Gerais, Brazil
- KYPS Kyanite, Pennsylvania State University
- *NEPH Nepheline, Bear Paw Mts., MT
- *PYNC Pyrophyllite, Staley, NC
- SCMB Scapolite, Brazil
- *SPHC Sphene, Hemet Quadrangle, CA
- *STBM Staurolite, Berkshire, MA
- *TPTM Fluro-topaz, Topaz Mountain, UT
- TSLP Tourmaline, Mexico
- *ZOPC Zoisite, Puerto Cabello, Venezuela

*Available for distribution from Reston

Appendix II. Summary of Positions of Documented Materials in Standard Blocks and Existence of Polished Grain Mounts.

Code	Standard Blocks:					JSH	BKG	Mn	37A	37B	15A	C	Gls	Ox	RE1	RE2	Mica	BS	Polished Mount
	#1	#2	#3	#4	#5														
AMCM	14																		
AMEN										7									
AMKF				31															
AMKH	3							2	4										
AMMN																			*
AMSF				30					10										
APCL													15	7					
APFD														10	12		15		
APRE														9	8				
APSC															14				
APSF														8	13				
CCHM																			*
CCNM								4											*
CDAS											9								*
CDBS																			*
CDOS								5											*
CRAP								6				1							
CSBH												10							
CSIG								7											*
CSTR								8											*
FSB0				47				9	13	7									
FSLC				45				10	14										
FSNA									12										
FSTA				46				11	15	6									
GD85		20				7													
GFAB												6							
GFAN										8		8							
GFOR												7							
GLBA								12		9									
GLDI			49			5		30											
GLJF									33				1						
GLL1									31										
GLL7									30	25									
GLL8									32										

Appendix II (continued)

Code	Standard Blocks:					JSH	BKG	Mn	37A	37B	15A	C	Gls	Ox	RE1	RE2	Mica	BS	Polished Mount
	#1	#2	#3	#4	#5														
GLMP												2							
GLSI						17	1	13											
GRE1																1			
GRE2																2			
GRE3																3			
GRE4																			*
GRLS																			*
GSDI		16	16																
GSEN		17	17			16													
GSWO						8													
GWOL												9							
GTAL																			*
GTKN	1			1				8	28								1		*
GTRV																			*
GTSP																			*
MBLM								15											
MBPS										6						x			*
MBST																x			*
MFPH								16											
MMMT									11	23									*
MPAV												4							*
MPBO												5							*
MSFP																	??		*
OLCO								17											
OLCR										14									
OLMJ	8			8		1			6										
OLNI								18				12						6	
OLRF								19	19	14									*
OLSC												10				15			
OLSF				33		2		20						14					
OLSM				10															
OLST						11		21		18				13					
OLSW	9								7										

Appendix II (continued)

Code	Standard Blocks:					JSH	BKG	Mn	37A	37B	15A	C	Gls	Ox	RE1	RE2	Mica	BS	Polished Mount
	#1	#2	#3	#4	#5														
OXAL							10												*
OXBU																			*
OXCO								22						5					
OXGH								23									8		
OXHA								24											
OXIL								25						2	10		4		
OXMN																			
OXMT								26					12						
OXNC							8												
OXNI															4				
OXPA					36			27											
OXPE							17	28											
OXQZ						12			16										
OXRU							3												
OXSB							7												
OXSC																			*
OXSM					66		12		18										
OXSP													1						
OXSZ							11												*
OXTB								29		9							14		
OXUB												13							
OXVA													7	6					
OX51					51	6													
OX52					52			29											
OXR1														11	4				
OXR2														14	5				
OXR3														13	6				
OXR4														12	7				
PXAC																			*
PXAD	5		5	5		4		9											
PXA6								28											
PXAG																			*
PXBH				40		9		21											
PXBK								20	29										
PXEN									15								10		
PXHD						19B			31										
PXHI								17											
PXHY						20			30										
PXJD				44		13													
PXJT								24				15							
PXKA	2					19A		27											
PXP1									2								13		
PXPS									10										
PXSD			48			3		30	17										
PXSE						15													
PXSW			6																
PXWO								31	27										

Appendix II (continued)

Code	Standard Blocks:					JSH	BKG	Mn	37A	37B	15A	C	Gls	Ox	RE1	RE2	Mica	BS	Polished Mount	
	#1	#2	#3	#4	#5															
SAS2																			*	
SCDS																				*
SSB2																				*
SSNS																				*
SZNS																				*
ANDB												3								
KYMG												2								
KYPS																		2		
NEPH																				*
PYNC										32										*
SCMB								32							1	11				
SPHC				29		10			3											
STBM										33										*
TPTM																				*
TSLP	15								5											
ZOPC									35											

APPENDIX III. NUMERICAL EVALUATION OF MICROPROBE STANDARDS
 IIIA. Criteria

A	The analysis. $(A1 + A2 + A3) \geq 0$	
A1	Method of analysis:	
	classical wet chemistry or equivalent	+4
	rapid rock, XRF, or equivalent	+2
	quant spec, minors	+2
	microprobe	+1
	semiquant spec, minors	+1
	method unknown	0
	theoretical formula	
	synthesis under conditions know to preserve	
	composition of starting material	+2
	ditto with optical or X-ray characterization	+3
	ditto with optical and X-ray characterization	+4
	synthesis not documented	0
A2	Incomplete analysis $(A1 + A2) > 0$	
	each missing nonvolatile major oxide anticipated	-1
	each missing volatile likely to be present	-0.5
A3	Agreement between independent analyses	
	analyses disagree:	
	1 or 2 elements except redox, H ₂ O	-2
	3 or more elements	-4
	analyses agree:	
	same method	+1 to +2
	different methods	+3 to +4
B	Summation	
	excellent summation, sum within 0.2% of 100.00%	+2
	adequate summation	0
	poor summation, not within 0.5%	-2
C	Formula Unit	
	excellent formula unit of 4 or more cation sites	+4
	excellent formula unit of 3 cation sites	+3
	excellent formula unit of 2 cation sites	+2
	reasonable formula unit of 3 or more sites	+2
	reasonable formula unit of 2 cation sites	+1
	formula unit not definitive	0
	impossible short formula unit	-4
	impossible long formula unit	-2
D	Homogeneity of major elements $(D \geq -6)$	
	All S.R. values ≤ 1.5	+4
	most S.R. values ≤ 1.5 , rest ≤ 2.0	+2
	Any S.R. values 2.0 to 3.0	0
	Each S.R. value exceeding 3.0	-2
	Most S.R. values exceed 3.0	-6

Appendix III (continued)

E	Special Problems:	
	Poor polish in multi-mount block (inconvenience)	-1
	Poor polish in individual mount	-2
	Grain size small (<15 micrometers)	-2
	Admixed phases not easily distinguished	-2
	Decomposition under focused beam (2 μ)	-2
	Decomposition under defocused beam (15 μ)	-4

IIIB. NUMERICAL EVALUATIONS

NAME	A1	A2	A3	B	C	D	E	SUM	Date
AMCM	+4	-1	-	0	+2	-6	-	- 1	08/06/85
AMEN	+4	-	-	+2	+2	-4	-	4	08/06/85
AMKF	+4	-	-	-	-	-4	-	0	08/06/85
AMKH	+4	-	-	+2	+3	+2	-	11	08/06/85
AMMN	+2	-1	-	-2	+4	-4	-	- 1	08/06/85
AMSF	+4	-	-	-	-	-2	-	2	08/07/85
APCL	0	-	-	-	-	0	-2	- 2	08/07/85
APFD	+4	-	+2	+2	+2	0	-	10	08/08/85
APRE	0	-	-	-	-	0	-	0	08/07/85
APSF	0	-	-	-	-	0	-	0	08/07/85
CCHM	+4	-1	-	-	-	+1	-2	2	08/07/85
CCNM	+4	-	-	+2	+2	+4	-2	10	10/25/85
CDAS	+2	-	-	-2	+3	+2	-	5	08/08/85
CDBS	+4	-	-	+2	+3	+2	-	11	08/07/85
CDOS	+4	-	-	0	+2	+2	-	8	10/25/85
CRAP	+4	-	-	+2	+2	+2	-	10	08/07/85
CSBH	+4	-	+1	-2	+2	-6	-	- 1	08/07/85
CSIG	+4	-	-	+2	+2	+2	-	10	10/25/85
CSTR	+4	-3	-	-	+1	-2	-	0	08/07/85
FSBO	+4	-	+3	0	-2	+4	-	9	08/07/85
FSLC	+4	-	+4	0	+2	+2	-	12	08/07/85
FSNA	+4	-	+3	+2	-4	-4	-	1	08/07/85
FSTA	+1	-1	+3	-	-	+4	-	7	08/07/85
GD85	0	-	-	-	-	+4	-	4	08/07/85
GFAB	+4	-	-	+2	-	-2	-2	2	08/07/85
GFAN	+2	-	-	-	-	+2	-	4	08/07/85
GFOR	+4	-	-	+2	-	0	-2	4	08/07/85
GLBA	0	-	-	-	-	+4	-	4	08/07/85
GLDI	+3	-	-	-	-	+1	-	4	08/07/85
GLJF	+4	-	+4	+2	-	+2	-	12	08/07/85
GLL1	+4	-	+4	+2	-	-4	-	6	08/07/85
GLL7	+4	-	+3	0	-	-6	-	1	08/07/85
GLL8	+4	-	+3	0	-	0	-	7	08/07/85
GLMP	+4	0	+2	+4	-	+4	-	14	08/07/85
GLSI	+2	-	-	-	-	+4	-	6	08/07/85
GRE1	+3	-	-	-	-	+4	-	7	08/07/85
GRE2	+4	-	-	-	-	+2	-	6	08/07/85
GRE3	+4	-	-	-	-	+4	-	8	08/07/85
GRE4	+3	-	-	-	-	+4	-	7	08/07/85

IIIB. NUMERICAL EVALUATIONS (continued)

NAME	A1	A2	A3	B	C	D	E	SUM	Date
GRLS	+4	-	+4	+2	-	-2	-	8	10/17/85
GSDI	0	-	-	-	-	+4	-	4	08/07/85
GSEN	0	-	-	-	-	+4	-	4	08/07/85
GSWO	0	-	-	-	-	+4	-	4	08/07/85
GWOL	+2	-	-	-	-	+4	-	6	08/07/85
GTAL	+4	-	+1	-1	+3	-2	-	5	08/07/85
GTKN	+4	-	+2	0	+3	0	-	9	08/07/85
GTRV	+2	-	-	0	0	0	-	2	08/07/85
GTSP	+3	-	-	-2	-2	0	-	- 1	08/07/85
MBLM	+4	-	+2	+2	+2	0	-	10	08/07/85
MBPS	+4	-	+2	+2	+2	-6	-2	2	08/07/85
MBST	+4	-	-	+2	0	-6	-	0	08/07/85
MFPH	0	-	+1	-	-	-2	-	- 1	08/07/85
MMMT	+2	-	2	0	0	-6	-	- 2	08/07/85
MPAV	+2	-	0	-2	0	+2	-	2	08/07/85
MPBO	+4	-	-	-2	+2	0	-	4	08/07/85
MSFP	0	-	-	-	-	-2	-	- 2	08/07/85
OLCO	0	-	-	-	-	+4	-	4	08/07/85
OLCR	+1	-	+1	-2	+1	0	-	1	08/07/85
OLMJ	4	-	+2	0	+2	+2	-	10	08/07/85
OLNI	+2	-	-	-	-	+2	-	4	08/07/85
OLRF	+4	-	-	-2	+1	+2	-	5	08/07/85
OLSC	+4	-	-2	0	+1	+4	-	7	08/07/85
OLSF	+2	-	-	-	-	0	-	2	08/07/85
OLSM	+4	-	-	-1	+2	-6	-	- 1	08/07/85
OLST	+4	-	-	-	-	-2	-	2	08/07/85
OLSW	+4	-	-	-2	0	+2	-	4	10/25/85
OXAL	+3	-	-	-	-	-6	-	- 3	08/07/85
OXBU	+4	-	+2	+2	+2	+2	-	12	08/07/85
OXCO	0	-	-	-	-	+4	-	4	08/07/85
OXGH	+4	-	-	+1	+1	0	-	6	08/07/85
OXHA	+4	-	-	-	-	-	-2	2	08/07/85
OXIL	+4	-	+3	-2	+2	0	-	7	08/07/85
OXMN	+2	-	-	-	-	-	-2	0	08/07/85
OXMT	+4	-	-	-2	+2	+3	-	7	08/07/85
OXNC	0	-	-	-	-	+4	-	4	08/07/85
OXPA	+4	-	-	-	-	0	-	4	08/07/85
OXPE	0	-	-	-	-	0	-	0	08/07/85
OXQZ	+1	-	-	-	-	+4	-	5	08/07/85
OXRU	0	-	-	-	-	2	-	2	08/07/85

IIIB. NUMERICAL EVALUATIONS (continued)

NAME	A1	A2	A3	B	C	D	E	SUM	Date
OXSB	+2					+2		4	08/07/85
OXSC	+4	-	-	+2	-	0		6	08/07/85
OXSM	+2	-	-	-	-	+2	-1	3	08/07/85
OXSP	+2	-	-	-	-	+2	-	4	08/07/85
OXSZ	+3	-	-	-	-	-4	-	- 1	08/07/85
OXTB	+4	-	+1	0	+2	0	-	7	08/07/85
OXUB	+4	-	-	+2	+2	-2	0	6	08/07/85
OXVA	+1	-	-	-	-	+4	-	5	08/07/85
OX51	+2	-	-	-	+2	-4	-	0	08/07/85
OX52	+2	-	-	-	+2	0	-	4	08/07/85
OXR1	0	-	-	-	-	+4	-	4	08/07/85
OXR2	0	-	-	-	-	0	-	0	08/07/85
OXR3	0	-	-	-	-	+4	-	4	08/07/85
OXR4	0	-	-	-	-	+4	-	4	08/07/85
PXAC	+4	-	-	-	-	+4	-	8	09/12/85
PXAD	+4	-	+2	0	-4	+2	-	4	08/27/85
PXA6	+4	-	0	0	+2	+2	-	8	08/07/85
PXAG	+4	-	-	-	0	-2	-	2	08/07/85
PXBH	+4	-	-2	0	+1	0	-	3	08/07/85
PXBK	+4	0	+2	-2	-4	0	-	0	08/07/85
PXEN	+4	-	-	-	-	+2	-	6	10/11/85
PXHD	+4	-	+3	0	+3	0	-	10	08/07/85
PXHI	+4	-2	+1	0	-4	-6	-2	- 9	08/07/85
PXHY	+2	-	-	+2	-4	0	-	0	08/08/85
PXJD	+4	-	-	+2	+2	0	-2	6	08/08/85
PXJT	+4	-1	+2	0	+2	0	-	7	08/08/85
PXKA	+4	0	+3	-2	+2	+4	-	11	10/25/85
PXP1	+4	-	+1	+2	+3	-4	-	6	08/08/85
PXPS	+4	-	-	+2	+2	+4	-	12	08/08/85
PXSD	+4	-	-	-	-	-6	-	- 2	08/08/85
PXSE	+4	-	-	-	-	0	-2	2	10/08/85
PXSW	0	-	-	+2	+2	+4	-	8	10/11/85
PXWO	+4	-	-	+2	+2	+4	-2	10	08/08/85
SAS2	+3	-	-	-	-	0	-	3	08/08/85
SCDS	+3	-	-	-	-	0	-	3	08/08/85
SSB2	+3	-	-	-	-	+4	-	7	08/08/85
SSNS	+2	-	-	-	-	+1	-	3	08/08/85
SZNS	+3	-	-	-	-	0	-	3	08/08/85
ANDB	+2	-2	-	-	-	+2	-	2	08/08/85
KYMG	+2	-1	-	-	-	+4	-	5	08/08/85

IIIB. NUMERICAL EVALUATIONS (continued)

NAME	A1	A2	A3	B	C	D	E	SUM	Date
KYPS	+2	-	-	+2	+2	+4	-	10	08/08/85
NEPH	+4	-	-	0	0	-2	-	2	08/08/85
PYNC	+4	-	+4	+2	+3	0	-2	11	08/08/85
SCMB	+4	-	-	+2	+2	+4	-	12	08/08/85
SPHC	+4	-	-	0	+2	0	-	6	08/08/85
STBM	+4	-	+1	0	0	+4	-2	7	08/08/85
TPTM	+4	-	+4	+1	-	+2	-	11	08/08/85
TSLP	+4	-	-	+2	-2	0	-	4	08/08/85
ZOPC	+4	-	+1	0	+2	0	-2	5	08/08/85

APPENDIX IV. Documentation for materials.

Appendix IV should be periodically updated as new information supporting the materials becomes available or as new materials are added to the collection. To avoid the necessity of renumbering the entire Appendix IV following these anticipated revisions, the pages are not numbered. Instead, the documentation is arranged alphabetically, according to the mnemonic codes listed in Appendix I.

Standard: Cummingtonite
 Locality: Mikonui River, New Zealand
 Donor: Wm. Melson to B.A. Morgan
 References: M.B. Baller, analyst

Oxide Wt. %

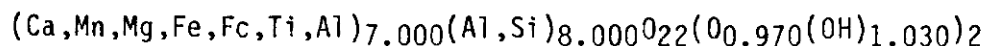
MgO	13.71
Al ₂ O ₃	2.37
SiO ₂	52.9
CaO	0.55
TiO ₂	0.06
MnO	0.97
FeO	27.98
H ₂ O	1.04
Total	<u>99.58</u>

Mg	3.088	3.026
Al	0.422	0.414
Si	7.995	7.833
Ca	0.089	0.087
Ti	0.007	0.007
Mn	0.124	0.122
Fe	3.537	3.465
sum cations	<u>15.263</u>	<u>14.953</u>
sum anions	24.0	23.0

OH 1.05

Element	Si	Fe	Mg
S.R.	2.3	3.7	6.3
#Pts.	20	20	20
#Grains	8	8	8

Evaluation: The wet chemical analysis of the cummingtonite from Mikonui River, N.Z., has a low sum, but does not include alkali and ferric iron. Recalculation of the analysis to an amphibole formula unit suggests further problems. On an anhydrous basis, a 15 cation to 23 oxygen formula unit cannot be achieved, even when all polyvalent species are reduced. The unadjusted hydrous formula unit has 15.263 cations per 24.000 anions, requiring divalent cations in the A-site. Adjustment by oxidizing about 25% of the FeO results in a reasonable amphibole formula unit with an "oxy" component:



The amphibole is heterogeneous; variations in Fe and Mg x-ray count rates are inversely coupled, indicating Fe/Mg zoning. On the basis of the incomplete chemical analysis, low summation, and Fe/Mg zoning, AMCM cannot be recommended for use as a standard.

Standard: Engel's Amphibole
 Locality:
 Donor: C.O. Ingamells to Czamanske, U.S. Geological Survey, Menlo Park, CA
 Reference:

oxide wt.%

K ₂ O	0.91
Na ₂ O	1.63
CaO	11.56
MnO	0.63
MgO	8.67
FeO	13.48
Fe ₂ O ₃	6.19
TiO ₂	0.94
Al ₂ O ₃	12.09
SiO ₂	42.14
H ₂ O	1.66
Total	<u>99.90</u>

K	0.169	0.169
Na	0.459	0.459
Ca	1.800	1.800
Mn ²⁺	0.000	0.078
Mg	1.878	1.878
Fe ²⁺	0.000	0.216
Fe ³⁺	2.316	2.100
Ti	0.103	0.103
Al	2.071	2.071
Si	6.126	6.126
Mn ³⁺	0.078	-
sum cations	<u>15.000</u>	<u>15.000</u>
sum anions	23.093	23.000

OH 1.610

Element	Si	Fe	Mg
S.R.	1.1	3.2	3.6
#Pts.	20	20	20
#Grains	11	11	11

Al	Ca	Na
2.0	1.4	1.5
20	20	20
15	15	15
1.9	1.2	1.6
19	19	19
14	14	14

Mineral: Engel's amphibole

Microprobe analysis: U.S.G.S. ARL-SEMQ microprobe. Bence-Albee method.
03-MAR-82. J. Hammarstrom, analyst. Average of 5 pts each.

	oxide wt% $\pm 1\sigma$	oxide wt% $\pm 1\sigma$
SiO ₂	42.41 \pm 0.39	43.20 \pm 0.39
Al ₂ O ₃	12.74 \pm 0.15	12.79 \pm 0.22
FeO	19.01 \pm 0.08	19.99 \pm 0.34
MgO	8.65 \pm 0.14	8.69 \pm 0.11
CaO	11.72 \pm 0.14	11.77 \pm 0.08
Na ₂ O	1.53 \pm 0.03	1.60 \pm 0.06
K ₂ O	0.98 \pm 0.03	0.94 \pm 0.05
TiO ₂	0.92 \pm 0.02	1.00 \pm 0.13
MnO	0.64 \pm 0.04	0.65 \pm 0.03
Cr ₂ O ₃	-	-
F	-	-
Cl	-	-
Total	98.58 \pm 0.327	100.63 \pm 0.464

Standards:

AMKH - Si, Al, mg, Ca, Na, Ti

AMEN - Fe

MFPH - K

OLST - Mn

Standards:

MBST - Si, Al, Ti

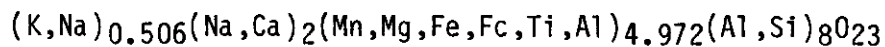
MBLM - Fe

MFPH - Mg, K

AMEN - Ca, Na

OLST - Mn

Evaluation: The sum of the wet chemical analysis is excellent, 99.90 weight percent. The anhydrous formula unit,



is that of a hornblende with a partially occupied A site. Subsequent microprobe analyses, using a variety of standards, reproduce adequately the wet chemical values for K₂O, Na₂O, CaO, MnO, MgO, and TiO₂. The sigma ratios for Na, Ca, Al, and Si are good to excellent. The count rates for Mg and Fe vary inversely, indicating some variation of Fe/Mg between points. In the split examined for homogeneity one erratic grain was found. In a memorandum dated May 4, 1972 to G. Czamanske, analyst C.O. Ingamels reports that splits are homogeneous with respect to potassium. AMEN should be a good standard for all elements but Mg and Fe where an iron-rich hornblende is needed. Its use as a Mg and Fe standard would require standardization on at least 10 grains.

Standard: Potassic Fluor-Richterite
 Locality: Synthetic
 Donor: J.S. Huebner
 Reference: Huebner, J.S., and J.J. Papike (1970) Synthesis and crystal chemistry of sodium-potassium richterite $(\text{Na,K})\text{NaCaMg}_5\text{Si}_8\text{O}_{22}(\text{OH,F})_2$: A model for amphiboles. *Am. Mineral.* 55, 1973-1992.

Oxide wt.% (theoretical)

F	4.53
Na ₂ O	3.70
MgO	24.04
SiO ₂	57.34
K ₂ O	5.62
CaO	6.69
sub-Total	<u>101.91</u>
-O=F	<u>1.91</u>
Total	<u>100.00</u>

Element	Si	Mg	Ca	Na	K	F
S.R.			0.9	5.3		
#Pts.			18	18		
#Grains			7	7		
			1.0		4.8	2.4
			16		16	16
			9		9	9
			1.1			1.1
			18			18
			12			12
	1.3	1.9				
	19	19				
	6	6				
				5.0	10.3	
				19	19	
				9	9	

Evaluation: The x-ray diffraction study of Huebner and Papike (1970) indicates that the amphibole is a richterite. Additional phases (probably forsterite, diopside, and glass) are present. Even if the richterite grains are identified in a mount, AMKF will not serve as a reliable standard: the presence of a highly potassic phase leaves unresolved the possibility that the richterite phase has a composition between potassic richterite, $\text{KNaCaMg}_5\text{Si}_8\text{O}_{22}\text{F}_2$, and sodic richterite, $\text{NaNaCaMg}_5\text{Si}_8\text{O}_{22}\text{F}_2$ (see AMSF). The synthetic potassic fluor-richterite is heterogeneous, with respect to K and Na, confirming this suggestion. Although AMKF might be used as a standard for other elements, a better choice would be AMSF.

Distribution:

Date	From	To	Address
19-NOV-76	Huebner	Arden Albee	Division of Geological and Planetary Sciences California Institute of Technology Pasadena, California 91125
08-MAY-72	Huebner	Bill Bonnicksen	Department of Geological Sciences Kimbell Hall - Cornell University Ithaca, N.Y. 14858
06-JUN-77	Huebner	Eric J. Essene	Dept. Geology and Mineralogy University of Michigan Ann Arbor, MI 48104
11-DEC-74	Huebner	Edward Ghent	Faculty of Arts and Science Department of Geology The University of Calgary Calgary, Alberta, Canada T2N 1N4
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
11-DEC-74	Huebner	Ian Ridley	Lamont-Doherty Observatory Columbia University Palisades, NY 10964
09-SEP-78	Huebner	V.J. Wall	Department of Earth Sciences Monash University Clayton, Victoria Australia 3168
17-FEB-69	Huebner	Paul Weiblen	Dept. of Geology Univ. of Minnesota Minneapolis, Minnesota 55455

Standard: Hornblende

Locality: Kakanui, New Zealand

Donor: B. Mason, USNM 143965

Reference: (1) Mason, B. (1966) Pyrope, augite, and hornblende from Kakanui, New Zealand, N.Z. Jour. Geol. Geophys., 9, p. 476. Wiik, analyst, classical method. Possibly impure separates (2) Same as analysis #1, but with revised Al₂O₃ and TiO₂ by Mason, circa 1969. (3) Mason, B., and R.O. Allen (1973) Minor and trace elements in augite, hornblende, and pyrope megacrysts from Kakanui, New Zealand. New Zealand Journal of Geology and Geophysics, 16, 935-947. (4) Same as analysis (3) but with revised value for TiO₂. From Jarosewich, E., et al. (1979) Smithsonian Contrib. Earth Sciences, no. 22.

	Oxide wt. %			
	(1)	(2)	(3)	(4)
SiO ₂	40.42	40.42	40.37	40.37
Al ₂ O ₃	16.01	13.90	14.90	14.90
TiO ₂	2.55	4.43	4.38	4.72
Fe ₂ O ₃	4.84	4.84	3.30	3.30
FeO	6.85	6.85	7.95	7.95
MgO	12.95	12.95	12.80	12.80
MnO	0.10	0.10	0.09	0.09
CaO	10.28	10.28	10.30	10.30
Na ₂ O	3.04	3.04	2.60	2.60
K ₂ O	2.05	2.05	2.05	2.05
H ₂ O ⁺	0.96	0.96	0.90	0.90
H ₂ O ⁻	0.00	0.00	0.04	0.04
F ⁻	0.15	0.15	-	-
subtotal	<u>100.20</u>	<u>99.97</u>		
less O for F	0.06	0.06		
Total	<u>100.14</u>	<u>99.91</u>	99.68	100.02
Si				5.992 5.855
Al				2.606 2.547
Ti				0.527 0.515
Fe ⁺³				0.369 0.360
Fe ⁺²				0.987 0.964
Mg				2.832 2.767
Mn ⁺²				0.011 0.011
Ca				1.638 1.601
Na				0.748 0.731
K				0.388 0.379
sum cations				<u>16.097</u> <u>15.731</u>
sum anions				24.000 23.000
OH				0.931

Element	Al	Ca	Na	Si	Fe	Mg	Ti
S.R.	1.5	1.1	1.7				
#Pts.	20	20	20				
#Grains	9	9	9				
				1.3	1.5	1.8	
				20	20	20	
				16	16	16	
	1.1		1.8				0.8
	20		20				20
	13		13				13

Mineral: Hornblende

Mineral analysis: (1) L.B. Wiggins, U.S.G.S., ARL-EMX microprobe, 1979;
 (2a,b) J. McGee, U.S.G.S., ARL-SEM-Q microprobe, 05-FEB-82. Bence-Albee;
 (2c) Magic

	oxide wt% $\pm 1\sigma$ (1) (2 grains)	oxide wt% $\pm 1\sigma$ (2a) (10 grains)	oxide wt% $\pm 1\sigma$ (2b) (6 grains)	oxide wt% $\pm 1\sigma$ (2c) (10 grains)
SiO ₂	40.94 \pm 0.25	40.22 \pm 0.11	41.08 \pm 0.20	40.50 \pm 0.25
Al ₂ O ₃	14.66 \pm 0.11	14.91 \pm 0.04	14.88 \pm 0.05	15.07 \pm 0.18
TiO ₂	4.55 \pm 0.05	4.68 \pm 0.12	4.43 \pm 0.09	4.86 \pm 0.11
Cr ₂ O ₃	0.00 \pm 0.00	0.00 \pm 0.00	0.00 \pm 0.00	0.01 \pm 0.01
FeO	10.71 \pm 0.09	10.66 \pm 0.12	10.46 \pm 0.11	11.37 \pm 0.22
MgO	12.14 \pm 0.12	13.10 \pm 0.11	13.10 \pm 0.10	13.10 \pm 0.24
MnO	0.09 \pm 0.02	0.14 \pm 0.01	0.14 \pm 0.00	0.10 \pm 0.01
CaO	10.02 \pm 0.13	10.14 \pm 0.29	9.99 \pm 0.24	10.62 \pm 0.07
Na ₂ O	2.77 \pm 0.03	2.57 \pm 0.04	2.58 \pm 0.02	2.67 \pm 0.05
K ₂ O	2.08 \pm 0.06	2.76 \pm 0.07	2.54 \pm 0.04	2.10 \pm 0.04
P ₂ O ₅	-	0.04 \pm 0.04	0.03 \pm 0.04	-
Total	97.96	99.21 \pm 0.36	99.22 \pm 0.48	100.40 \pm 0.32

Si	5.711
Al	2.410
Ti	0.477
Cr	0.000
Fe ²⁺	0.000
Fe ³⁺	1.249
Mg	2.524
Mn ³⁺	0.011
Ca	1.498
Na	0.749
K	0.370
cations	15.000
anions	22.464

(1)
 Standards:
 FSBO - K
 FSTA - Al, Na
 OLMJ - Mg, Si, Fe
 OLST - Mn
 PXWO - Ca
 OXTB - Cr

(2a,b)
 Standards:
 AMKH - Ca, Mg, K,
 Ti, Na, Fe, Al, Si
 OLST - Mn
 OXTB - Cr
 APFD - P

(2c)
 Standards:
 AMKH - Ca, Si, Al, Fe, Mg
 FSTA - Na
 OLST - Mn
 Orthoclase Or-1 - K
 OXTB - Cr
 OXIL - Ti

Mineral: Kakanui Hornblende

Microprobe analysis: U.S.G.S. ARL-SEM; Bence-Albee reduction; 03-MAR-83.

(1a) aver. of 6 pts (1b) aver. of 5 pts (1c) aver. of 5 pts; J. Hammarstrom, analyst (2) aver. of 8 pts on 3 grains; J. Stormer, analyst, 20-OCT-82.

	wt.% +1σ (1a)	wt.% +1σ (1b)	wt.% +1σ (1c)	wt.% +1σ (2)
SiO ₂	39.68+0.34	41.44+0.28	40.68+0.32	40.63+0.33
Al ₂ O ₃	14.42+0.07	15.00+0.10	14.90+0.13	14.58+0.20
FeO	10.58+0.32	11.07+0.11	10.64+0.14	10.73+0.12
MgO	12.96+0.23	12.58+0.18	12.51+0.20	12.91+0.14
CaO	9.88+0.18	10.43+0.07	10.44+0.11	10.05+0.08
Na ₂ O	2.68+0.07	2.73+0.02	2.59+0.05	2.69+0.01
K ₂ O	2.07+0.03	2.07+0.02	2.08+0.05	2.09+0.36
TiO ₂	4.70+0.20	5.22+0.12	4.69+0.17	4.78+0.11
MnO	0.05+0.01	0.04+0.01	0.06+0.01	0.06+0.01
Cr ₂ O ₃	-	-	-	-
F	-	-	-	0.197+0.05
Cl	-	-	-	0.031+0.02
Ba	-	-	-	0.087+0.05
Total	97.02+0.42	100.58+0.24	98.58+0.36	98.84

Si	5.571	5.641	5.638
Al	2.386	2.406	2.434
Fe ³⁺	1.242	1.260	1.233
Mg	2.712	2.552	2.584
Ca	1.486	1.521	1.550
Na	0.730	0.720	0.696
K	0.371	0.359	0.368
Ti	0.496	0.534	0.489
Mn ³⁺	0.006	0.005	0.007
cations	15.000	15.000	15.000
anions	22.334	22.471	22.432

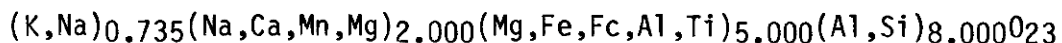
Standards
 PXAD - Si, Ca
 MBLM - Fe
 OLSF - Fe
 OLMJ - Mg
 PSU orthoclase - K
 OXRU - Ti
 OLST - Mn

Standards
 MBST - Si, Al, Ti
 MBLM - Fe
 MFPH - Mg, K
 AMEN - Ca, Na
 OLST - Mn

Standards
 AMKH - Si, Al, Mg,
 Ca, Na, Ti
 AMEN - Fe
 MFPH - K
 OLST - Mn

Standards
 MFPH - Si,
 Mg, K
 GTKN - Al, Fe
 AMSF - Ca, Na
 OXRU - Ti
 PXBH - Mn

Evaluation: Analysis #4 of Jarosewich et al can be recalculated, on an anhydrous basis without adjustment of the redox state, as a hornblende which has its A-site 74% occupied with Na and K:



Recalculation to a hydrous formula unit before adjustment of the ferrous/ferric ratio results in too many cations. Taken together, these two recalculations suggest an inconsistency in either the water analysis or the proportions of ferrous and ferric iron.

The homogeneity of the Kakanui hornblende is very good. Sigma ratio values for Al, Si, Ca, and Ti are excellent. Values for Na, Mg, and Fe are good. The Fe/Mg is more homogeneous than in AMEN or AMMN. The elemental composition of the original AMKH analysis has been confirmed by numerous subsequent microprobe analyses. AMKH is recommended for use both as a standard for amphiboles and micas and use as a "known-unknown" to check a microprobe standardization.

Standard: Hornblende
Locality: McClure Mountain Complex; Fremont County, Colorado
Donor: E.C. Alexander, Jr.; University of Minnesota
Reference: Alexander, E.C., Jr., (1978) in Short Papers of the Fourth International Conference, Geochronology, Cosmochronology Isotope Geology. Geol. Surv. Open-File Report 78-701. Conference held August 20-25, 1978 in Snowmass-at-Aspen, Colorado. XRF: 5 analyses from 3 splits. Analyst, P. Hearn

Oxide wt.% $\pm 1 \sigma$

SiO ₂	37.16±0.44
FeO	20.56±0.26
Al ₂ O ₃	12.92±0.31
CaO	10.18±0.16
MgO	6.42±0.11
TiO ₂	3.68±0.08
Na ₂ O	2.88±0.28
K ₂ O	1.90±0.07
MnO	0.75±0.01
P ₂ O ₅	0.05±0.02
Total	<u>96.50</u>

	adjusted
Si	5.857
Fe ²⁺	2.455
Fe ³⁺	0.255
Al	2.400
Ca	1.719
Mg	1.508
Ti	0.436
Na	0.880
K	0.382
P	0.007
Mn ²⁺	0.100
sum cations	<u>16.000</u>
sum anions	23.000

Element	Si	Fe	Mg		
S.R.	2.2	7.1	10.2		
#Pts.	20	20	20		
#Grains	20	20	20		
			9.6	Al	Ca
			20	2.1	1.9
			20	20	20
			20	20	20

Mineral: Minnesota Hornblende

Microprobe analysis: U.S.G.S. ARL-SEMQ microprobe. Bence-Albee reduction.
04-MAR-82. J. Hammarstrom, analyst.

oxide wt.% 1σ
average of 10 points

SiO ₂	39.46+0.38
Al ₂ O ₃	12.36+0.71
FeO	20.84+0.93
MgO	6.82+0.67
CaO	10.84+0.13
Na ₂ O	2.96+0.10
K ₂ O	1.86+0.08
TiO ₂	3.58+0.60
MnO	0.72+0.05
Total	99.45+0.44

Si	6.033
Al	2.227
Fe ²⁺	2.541
Fe ³⁺	0.123
Mg	1.554
Ca	1.776
Na	0.877
K	0.363
Ti	0.412
Mn ²⁺	0.093
cations	16.000
anions	23.000

Standards:

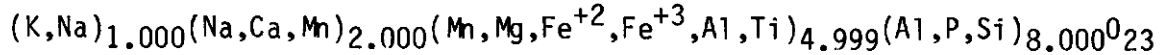
AMKH - Si, Al, Mg, Ca, Na, Ti

AMEN - Fe

MFPH - K

OLST - Mn

Evaluation: The original XRF analysis did not include volatiles (~2%) but even so its total (96.50%) appears to be low. On the anhydrous basis, the original analysis with only ferrous iron calculates to a formula unit with too many cations. The analysis can be recalculated to yield an amphibole formula:



by converting 0.255 cations ferrous iron to the ferric state. Creation of more ferric iron leaves the A site only partially filled, which is quite reasonable. Analysis of 10 consecutive points reveals large standard deviations for Al, Fe, Mg, Ti of 6%, 4%, 10%, and 17%, respectively, of the amounts present. (Based on count rate alone, the percent deviations would be 1%, 1%, 1%, and 3%, respectively.) Subsequent measurement of the sigma ratios reveals that Al, Ca, and Si are sufficiently homogeneous to use AMMN as a standard, but that Mg and Fe are very heterogeneously distributed. The count rates for Mg and Fe vary inversely, indicating that the nature of the inhomogeneity is Fe/Mg variation from point to point. A preferable titaniferous amphibole standard would be AMKH; a preferable iron-rich amphibole would be AMEN. (AMEN is not homogeneous with respect to Fe/Mg, but the degree of inhomogeneity in AMEN is much less serious than in AMMN.)

Standard: Sodic Fluor-richterite #44
 Locality: Synthetic (Na)NaCaMg₅Si₈O₂₂F₂
 Donor: J.S. Huebner
 Reference: Huebner, J.S. and J.J. Papike (1970) Synthesis and crystal chemistry of sodium-potassium richterite (Na,K)NaCaMg₅Si₈O₂₂(OH,F)₂: A model for amphiboles. *Am. Mineral.* 55, 1973-1992.

Oxide wt.% (theoretical)

F	4.62
Na ₂ O	7.54
MgO	24.51
SiO ₂	58.46
CaO	6.82
sub-Total	<u>101.94</u>
-O=F	1.94
Total	<u>100.01</u>

Element	F	Na	Mg	Si	Ca
S.R.		1.4			1.6
#Pts.		20			20
#Grains		4			4
			1.3	1.4	
			20	20	
			4	4	
	1.6				4.1
	17				17
	6				6

 Microprobe analysis: U.S.G.S. ARL-SEMQ microprobe. J. Stormer, 10/20/82;
 10 points on 2 grains averaged.

	wt.% <u>+1 σ</u>
F	4.65+0.17
Na	7.61±0.13
Mg	25.12±0.18
Si	58.89±0.39
Ca	6.85±0.05
Total	<u>103.12</u>

Standards
 F - MFPH
 Na - AMSF
 Mg - MFPH
 Si - MFPH
 Ca - AMSF

Evaluation: The x-ray diffraction study of Huebner and Papike (1970) suggests that AMSF is "on composition". The presence of minor diopside, forsterite, and glass among the run products is not considered detrimental because of the limited possibility for deviations from the ideal formula in the absence of trivalent elements (Fe^{+3}, Al^{+3}). When AMSF is analyzed using a synthetic fluorphlogopite as a standard, J.R. Stormer found good agreement for fluorine but slightly high values for MgO and SiO_2 . The sodic richterite is homogeneous for all elements with the possible exception of calcium, but microprobe operators must be aware that not all grains are the fluor-rich richterite. This material provides a possible alternative to Tiburon albite (FSTA) for Na.

Distribution:

Date	From	To	Address
19-NOV-76	Huebner	Arden Albee	Division of Geological and Planetary Sciences California Institute of Technology Pasadena, California 91125
08-MAY-72	Huebner	Bill Bonnicksen	Department of Geological Sciences Kimbell Hall - Cornell University Ithaca, N.Y. 14858
11-MAR-77	Huebner	Eric J. Essene	Dept. Geology and Mineralogy University of Michigan Ann Arbor, MI 48104
11-DEC-74	Huebner	Edward Ghent	Faculty of Arts and Science Department of Geology The University of Calgary Calgary, Alberta, Canada T2N 1N4
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
11-DEC-74	Huebner	Ian Ridley	Lamont-Doherty Observatory Columbia University Palisades, NY 10964
09-SEP-78	Huebner	V.J. Wall	Department of Earth Sciences Monash University Clayton Victoria Australia 3168
17-FEB-69	Huebner	Paul Weiblen	Dept. of Geology Univ. of Minnesota Minneapolis, Minnesota 55455

Standard: Chlorapatite
 Locality: Synthetic - $\text{Ca}_5(\text{PO}_4)_3\text{Cl}$
 Donor: U.S. National Museum Division of Mineralogy
 Reference: U.S. National Museum #173;"X-rayed" by Jerome Prener (2/72) -
 General Electric Corp. - Schenectady, N.Y. - Luminescence Branch
 (Physics)

Oxide wt.% (theoretical)

CaO	53.84
P ₂ O ₅	40.88
Cl	6.81
sub-Total	<u>101.54</u>
-O=Cl	1.54
Total	<u>100.00</u>

Element	Ca		
S.R.	1.3		
#Pts.	20	Cl	
#Grains	4	2.4	
		20	P
		3	1.3
			20
			4
	1.4		1.3
	20		20
	4		4

Mineral: Chlorapatite

Microprobe analysis: J. Stormer, ARL-SEMQ, 20-OCT-82, 15 kV, .100 μ amp

counts/20 seconds

4278 \pm 1.5%	average of 6 areas, rastered, 20x20
3432	sequence of 20 second cts on single 1 μ m spot
2770	
2249	
2921	
2043	
1509	
1339	

Evaluation: The synthetic chlorapatite is presumed to be stoichiometric. Its apparent homogeneity is deceptive. Numbers of Cl x-ray counts for 20 different points, each exposed to the electron beam for 20 seconds, ranged between 2964 and 3559 counts, for a sigma ratio of 2.4. However, J.R. Stormer's work shows that the count rate decreases with time, from 4280 (at time zero) to 1340 at approximately 130 seconds. The simultaneous behavior of Ca and P is not known. APCL is extremely sensitive to exposure to the electron beam and should not be used unless the beam spot is defocussed or scanned (rastered) rapidly across the surface. With care, it has been used successfully to confirm the Cl content of MBLM.

Standard: Fluorapatite
 Locality: Durango, N.M.
 Donor: USNM to Sam Altschuler to B.A. Morgan (pillbox).
 U.S. Nat. Mus. #104021 (via).

Reference: E.J. Young, et al. (1969) Mineralogy and geochemistry of
 fluorapatite from Cerro de Mercado, Durango, Mexico. U.S.G.S. Prof. Pap.
 650-D, 84-93. Elaine Munson, N.M. Conklin, J.S. Wahlberg, J.N. Rosholt,
 I.C. Frost, and C. Huffman, Jr., analysts.

Oxide wt.%

	wet chem	optical spect.	AA	Isot. Dil.		
CaO	54.02				Ca	9.854
P ₂ O ₅	40.78				P	5.878
Na ₂ O	0.23		0.26		Na	0.076
K ₂ O	0.01				K	0.002
MgO*	0.01		0.02		Mg	0.002
FeO	0.00		0.04		Fe ²⁺	0.000
Fe ₂ O ₃ *	0.06				Fe ³⁺	0.008
Al ₂ O ₃ *	0.07				Al	0.014
MnO	0.01	0.02	0.01		Mn ²⁺	0.001
SrO	0.07	0.07	0.05		Sr	0.007
ZrO ₂		0.003			Zr	0.000
Ce ₂ O ₃		0.55			Ce	0.034
La ₂ O ₃		0.49			La	0.031
Nd ₂ O ₃		0.23			Nd	0.014
Sm ₂ O ₃		0.03			Sm	0.002
Y ₂ O ₃		0.096			Y	0.009
Gd ₂ O ₃		0.023			Gd	0.001
Tb ₂ O ₃		0.012			Tb	0.001
Dy ₂ O ₃		0.017			Dy	0.001
Ho ₂ O ₃		0.003			Ho	0.000
Er ₂ O ₃		0.011			Er	0.001
Yb ₂ O ₃		0.006			Yb	0.000
RE ₂ O ₃	1.43				Th	0.001
SiO ₂	0.34	0.34			Si	0.057
As ₂ O ₅	0.09	0.092			As	0.008
V ₂ O ₅	0.01	0.007			V	0.001
ZnO			0.001		C	0.011
ThO ₂	0.02			0.023	S	0.018
CO ₂	0.05				cations	16.032
SO ₃	0.37				anions	26.000
UO ₂				0.001		
F	3.53					
Cl	0.41				OH	0.011
H ₂ O ⁺	0.01				F	1.901
H ₂ O ⁻	0.00				Cl	0.118
sub-total	101.52				O	23.970
-O=F+Cl	1.58					
Total	99.94					

*considered to be an impurity

Element	P	Ca	F	
S.R.		2.4		USNM #104021
#Pts.		20		
#Grains		4		
	1.4	1.5	1.3	
	20	20	20	
	20	20	20	
	1.3			Pillbox
	20			
	4			
	0.9	1.5	2.0	
	20	20	20	
	6	6	6	

Evaluation: The wet chemical and spectrographic analyses by Young et al. (1969) appear superior and, when combined, yield a formula unit that has a stoichiometry very close to that of ideal apatite. However, the relationship between the material analyzed by Young et al., the vial of USNM #104021, and the crystals in the pillbox donated by Altschuler is not certain. All probably came from the same drawer of crystals at the USNM. Because Eugene Jarosewich (personal communication, August 19, 1985) found that the fluorine and total REE contents of #104021 agreed with the analysis reported by Young et al., he adopted that analysis for the material. Because the reported analysis is more likely to represent USNM #104021 than the contents of the pillbox, USNM #104021 is designated APFD.

Both APFD and the contents of the pillbox are adequately homogeneous. APFD has been used regularly as a standard for P in lunar metal particles and as a "known-unknown" for phosphates. APFD has potential as a "known-unknown" for trace elements that might be analyzed with the microprobe. There is uncertainty between the relationship of the analyzed material, the USNM #104021, and the pillbox of yellow crystals in Reston, and in the relationship between any of these and the samples mounted in Reston standard blocks.

Date	From	To	Address
11-NOV-76	Huebner	Prof. Arden Albee	Division of Geological and Planetary Sciences
	(pillbox material)		California Institute of Technology Pasadena, California 91125

Standard: REE-Apatite
 Locality: Synthetic - $\text{CaY}_{2.25}\text{Er}_{1.5}\text{Ho}_{0.1}\text{Tm}_{0.15}(\text{SiO}_4)_3\text{O}$
 Donor: U.S. National Museum - Division of Mineralogy (No. 587)
 References: R. Hopkins, Westinghouse, Pittsburgh, Pa.

Oxide wt.% (theoretical)

CaO	6.80
Y ₂ O ₃	30.79
Er ₂ O ₃	34.77
Ho ₂ O ₃	2.29
Tm ₂ O ₃	3.51
SiO ₂	21.85
Total	<u>100.00</u>

Element	Si	Ho						
S.R.	0.9	1.1						
#Pts.	20	20	Ca					
#Grain	3	3	1.4					
			20		Tm			
			3		1.2			
					20		Y	Er
					3		1.4	2.7
							20	20
							3	3

Evaluation: There is no record of data that would confirm the theoretical composition of APRE. The sigma ratios are excellent except for the value for Er, which is near the limit of acceptability for a major element standard, but is satisfactory if the concentration in the standard is much greater than that in the unknown. Further work is needed before APRE can be used with confidence as a rare earth standard.

Standard: Fluorapatite
Locality: Synthetic - $\text{Ca}_5(\text{PO}_4)_3\text{F}$
Donor: U.S. Nat. Mus. - Div. of Mineralogy No. 172
References: Jerome Prener - General Electric Corp. - Luminescence Branch
(Physics) Schenectady, N.Y. X-rayed (2/72)

Oxide wt.% (theoretical)

CaO	55.60
P ₂ O ₅	42.22
F	3.77
sub-Total	101.59
-O=F	-1.59
Total	100.00

Element	Ca	F
S.R.	1.8	2.2
#Pts.	20	20
#Grains	5	5

P
1.4
20
5

3.5
19
4

Evaluation: There is no information to verify that this material has the assumed composition. No record of trial use of this sample as a microprobe standard is available. The material appears to be homogeneous and should be evaluated further for possible use as a fluorine standard.

Standard: Calcite

Locality:

Donor: Richard A. Robie (original source: Clifford Frondel, Harvard Museum)

References: Jacobs, G.K., Kerrick, D.M., Krupka, K.M., Phys. Chem. Min. v.7, 1981.

Oxide Wt.%

MgO	0.01	
Fe ₂ O ₃	<0.005	
MnO	<0.002	
CaCO ₃	<u>(99.99)</u>	by difference
Total	100.00	

>153 μ m
Element Ca
S.R. 1.9
#Pts. 20
#Grains 20

<153 μ m
S.R. 1.7
#Pts. 20
#Grains 20

Evaluation: Wet chemical analysis by N. Suhr of Pennsylvania State University indicates CCHM is essentially pure CaCO₃ with trace amounts of MgO, Fe₂O₃, and MnO. A microprobe check for homogeneity on two different size fractions reveals acceptable sigma ratio values for Ca only if the beam is defocussed. CCHM should be an excellent choice as a standard for carbonate minerals which are pure Ca or for those which contain Ca and a small amount of Mg, providing a defocussed beam can be used.

Standard: Calcite USNM 136321

Locality:

Donor: E. Jarosewich to Huebner

Reference; Jarosewich, E., and MacIntyre, I.G., 1983, Carbonate reference samples for electron microprobe and scanning electron microscope analyses. J. Sedimentary Petrology, v. 53, no. 2, p. 677-678.

Oxide wt.%

CaO	56.10
CO ₂	<u>44.01</u>
Total	100.11

Ca	1.9995
C	<u>1.0003</u>
sum cations	2.000
sum anions	3.0

Element	Ca
S.R.	0.7
#Pts.	20
#Grains	11

Evaluation: The chemical analysis of calcite CCNM, by the classical methods of Peck, has an excellent sum. Jarosewich (written communication, October 21, 1985) reports that no additional elements, above trace levels, were found by emission spectrography. The material is a stoichiometric carbonate of almost ideal CaCO₃ composition. A focussed microprobe beam (15kV, 100nA) causes decomposition at the beam spot. Homogeneity was evaluated with a defocussed beam (15 micrometer diameter); with respect to circular areas of approximately 175 square micrometers, this material is homogeneous. Calcite CCNM should be an excellent standard for use with a defocussed beam if caution is taken to avoid decomposition under the beam spot.

Standard: Dolomite
 Locality: Austria
 Donor: U.S. Nat. Museum R10057 via Wetlaufer
 References: Reddick, K.L., Symp. anal. calorimetry, Div. Polymer Chemistry, Amer. Chem. Soc., San Francisco, 1968.; metal cation concn. deter. by atomic absorption anal., flame spectrophot. and wet chem., CO₂ by TGA, alkimeter anal., and C/H₂ analyzer; HCl insol. determined gravimetrically.

Oxide wt.%

CaO	29.71
MgO	20.97
FeO	0.66
MnO	0.03
CO ₂	46.64
sub-Total	<u>98.01</u>

HCl soluble	1.71 (by difference)
HCl insol.	0.28
Total	<u>100.00</u>

Ca	1.000
Mg	0.982
Fe	0.017
Mn	0.001
C	2.000
sum cations	<u>4.000</u>
sum anions	6.000

Element	Mg
S.R.	0.9
#Pts.	20
#Grains	10

Ca
1.7
19
10

Evaluation: The analysis of the dolomite from Austria appears to be excellent. Recalculation of the chemical analysis results in a formula that is greater than 99% CaMg(CO₃)₂ and is, within analytical uncertainties, stoichiometric [Ca,Mg,Fe,Mn]_{2.000}[C]_{2.000}O₆. Sigma ratios are 0.9 and 1.7 for Mg and Ca. CDAS should prove to be excellent as a standard for Mg and Ca in carbonate minerals.

Standard: Dolomite
Locality: Binnental, Switzerland
Donor: R.A. Robie, 3/84

References: Stout, W. and Robie, R.A., J. Phys. Chem. 67, 2248, 1963, Cp
and S₂₉₅. Krupka, K.M., 1983, Ph.D. thesis. Penn. State Univ. Cp at high
temperature cell parameters. Analyzed by spectrochemical and gravimetric
methods.

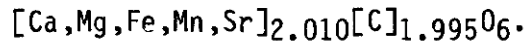
Oxide wt.%

CaO	30.77
MgO	21.54
MnO	0.10
FeO	0.008
SrO	0.017
CO ₂	<u>47.38</u>
Total	<u>99.82</u>

Ca	1.017
Mg	0.990
Mn	0.003
Fe ³⁺	0.0002
Sr	0.0003
C	<u>1.995</u>
sum cations	<u>4.005</u>
sum anions	6.000

Element	Ca	Mg
S.R.	1.4	1.9
#Pts.	20	20
#Grains	9	9

Evaluation: Dolomite from Switzerland has an excellent sum, 99.82 weight percent, and is within analytical uncertainty stoichiometric dolomite:



The sigma ratios for Ca and Mg are good, indicating homogeneity. CDBS is suitable as a standard for Ca or Mg in carbonate minerals.

Standard: Dolomite USNM 10057
 Locality: Oberdorf, Austria
 Donor: E. Jarosewich to Huebner
 Reference: Jarosewich, E. and MacIntyre, I.G., 1983, Carbonate reference samples for electron microprobe and scanning electron microscope analyses. J. Sedimentary Petrology, v. 53, no. 2, p. 677-678.

Oxide wt.%	
CaO	30.56
MgO	22.04
CO ₂	<u>46.93</u>
Total	99.53

Ca	1.014
Mg	1.017
C	<u>1.984</u>
sum cations	<u>4.016</u>
sum anions	6.0

Element	*Ca	*Mg
S.R.	1.6	1.8
#Pts.	20	20
#Grains	20	20
	*1.8	*1.4
	20	20
	20	20
	**1.5	**2.5
	20	20
	19	19
	1.1	1.6
	20	20
	11	11

*electron beam defocussed to 15μ
 **electron beam defocussed to 30μ

Evaluation: The wet chemical analysis of the USNM dolomite has a slightly low total, but Jarosewich (written communication, October 21, 1985) reports that no additional elements, above trace levels, were found by emission spectrography. The Ca_{1.014}Mg_{1.017}C_{1.984}O₃ formula unit is close to that of stoichiometric dolomite and is acceptable. The material is homogeneous and should prove to be a useful standard for analyses of carbonate.

Standard: Rhodochrosite
 Locality: Alma Park, New Mexico
 Donor: U.S. Nat. Mus. R2478

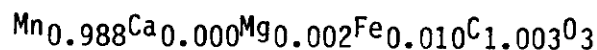
References: (1) Semi-quant. spectrog. analysis 71-WS-SS (1971), analyst,
 J.L. Harris; (2) Wet chemical analysis rep. #68-WO-9 (1968), J.J. Fahey,
 analyst.

	Oxide wt.%	
	(1)	(2)
CO ₂	-	38.50
MnO	>13.	61.11
FeO	0.1	0.30
MgO	0.3	0.04
CaO	0.10	0.00
TiO ₂	0.003	-
V ₂ O ₅	0.018	-
BaO	0.000	-
CuO	0.001	-
Tl ₂ O	0.018	-
SiO ₂	1.	-
Total		<u>99.95</u>

C	1.003
Mn	0.988
Fe ²⁺	0.005
Mg	0.001
Ca	0.000
sum cations	<u>1.997</u>
sum anions	3.000

Element	Mn
S.R.	1.6
#Pts.	20
#Grains	17

Evaluation: Rhodochrosite from Alma Park, New Mexico, appears to be an unusually pure rhodochrosite. The wet chemical analysis has a superior summation, 99.95 weight percent. Recalculation of the chemical analysis results in a formula that is greater than 99% MnCO₃ and is, within analytical uncertainties, stoichiometric with respect to the CO₂ content:



Besides Mn, Ca, Mg, and Fe, semiquantitative spectrographic analysis reveals only 3 elements with concentrations greater than 5 ppm: Ti (0.002%), V (100 ppm), and Tl (150 ppm). By reputation and usage, this standard is assumed to be homogeneous. The sigma ratio for manganese is 1.6. CRAP is an excellent standard for Mn in carbonates.

Distribution:

Date	From	To	Address
01-DEC-69	Huebner	A.E. Bence	Dept. of Earth and Space Sciences State Univ. New York at Stony Brook Stony Brook, New York 11790
30-NOV-73	Huebner	Eric Essene	Dept. of Geology and Mineralogy University of Michigan Ann Arbor, Michigan 48104
01-DEC-69	Huebner	Louis A. Fernandez	Dept. Geology and Geophysics Box 2161, Yale Station New Haven, Connecticut 06520
30-APR-70	Huebner	Bevan French	Planetology Branch-NASA Goddard Spaceflight Center Greenbelt, Maryland 20771
30-APR-70	Huebner	Edward Ghent	Faculty of Arts and Sciences Department of Geology The University of Calgary Calgary 44, Alberta, Canada
10-FEB-70	Huebner	Charles V. Guidotti	The University of Wisconsin Department of Geology and Geophysics Madison, Wisconsin 53706
13-NOV-80	Huebner	Lester Hughes	CONICO, Inc. 244 Research Bldg. P.O. 1267 Ponca City, OK 74601
22-JAN-73	Huebner	Brian Mason	Mineral Sciences Museum of Natural History Smithsonian Institution Washington, D.C. 20560

Distribution:

Date	From	To	Address
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
17-JAN-75	Huebner	Peter Robinson	Department of Geology The University of Massachusetts Amherst, Massachusetts 01002
05-SEP-84	Huebner	Michael Schaffer	Department of Geology University of Oregon Eugene, OR 97403-1272
08-SEP-78	Huebner	V.J. Wall	Department of Earth Sciences Monash University Clayton Victoria Australia 3168
20-FEB-69	Huebner	Paul Weiblen	Dept. of Geology Univ. of Minnesota Minneapolis, Minnesota 55455

Standard: Siderite

Locality: Broken Hill, Australia

Donor: U.S. National Museum 93218 via Wetlaufer

References: (1) U.S.G.S. Anal. Lab. Rep. # W-203839, semiquantitative emission spectrographic analysis (2) U.S.G.S. Anal. Lab. Rep. No. RERR., J. Marinenko and Z. Hamlin, analysts, using wet-chemical and atomic absorption methods. (3) Neutron activation determined by S. Spooner, Asst. Prof. of Met., Ga. Inst. Tech., Engr. Expt. Stat., Atlanta, Ga. (letter to John White, Jr. in Smithsonian files).

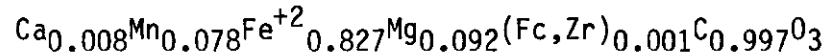
	(1)	oxide wt. % *(2)	(3)
SiO ₂	0.077		Mn = 0.076,
Al ₂ O ₃	0.10		where Mn _x Fe _{1-x} CO ₃ .
Fe ₂ O ₃	>34.	0.4	
FeO	-	52.8	
MgO	3.3	3.3	
CaO	0.49	0.4	
MnO	4.4	4.9	
K ₂ O	<0.082		
Na ₂ O	<0.006		
TiO ₂	<0.011		
ZrO ₂	0.031		
P ₂ O ₅	<0.16		
V ₂ O ₅	0.004		
Y ₂ O ₃	0.002		
PbO	0.003		
Sc ₂ O ₃	0.003		
CO ₂	-	39.0	
Total		100.8	

Fe ²⁺	0.827
Fe ³⁺	0.0006
Mg	0.092
Ca	0.008
Mn	0.078
Zr	0.0003
P	0.0000
V	0.0000
Y	0.0000
Pb	0.0000
Sc	0.0000
C	0.997
sum cations	<u>2.002</u>
sum anions	3.0

Element	Fe	Mg	Mn
S.R.	**2.7	**4.7	
#Pts.	20	20	
#Grains	10	10	
	*4.3	*8.6	
	20	20	
	15	15	
	*3.5	*7.5	
	20	20	
	15	15	
		*5.9	*4.3
		19	19
		19	19

*focussed beam
 **defocussed beam

Evaluation: The emission spec and conventional analyses combined yield a single analysis with a high weight percent total, 100.84%. The combined analysis can be recalculated to yield an almost perfectly stoichiometric carbonate formula,



The material is heterogeneous with respect to the three major elements present, Fe, Mg, and Mn. Use of CSBH as a microprobe standard is not recommended; instead, siderite CSIG should be considered when an iron carbonate standard is needed.

Standard: Siderite USNM R2460
 Locality: Ivigtut, Greenland
 Donor: E. Jarosewich to Huebner
 Reference: Jarosewich, E. and MacIntyre, I.G., 1983, Carbonate reference samples for electron microprobe and scanning electron microprobe analyses. J. Sedimentary Petrology, v. 53, no. 2, p. 677-678.

Oxide wt.%

FeO	59.08
MnO	2.95
CO ₂	<u>37.88</u>
Total	99.91

Fe	1.908
Mn	0.096
C	<u>1.998</u>
sum cations	<u>4.002</u>
sum anions	6.0

Element	*Fe	Mn
S.R.	1.7	
#Pts.	20	
#Grains	20	
		*1.2
		20
		20
	**1.5	
	20	
	20	
	1.8	
	20	
	20	
		1.0
		20
		20

*beam defocussed to ~15μ
 **beam defocussed to ~30μ

Evaluation: The wet chemical analysis has an excellent sum. Jarosewich (written communication, October 21, 1985) reports that no additional elements above trace levels, were found by emission spectrography. The formula unit is that of a stoichiometric carbonate solid solution. Under a defocussed beam (approximately 175 and 700 square micrometers in area) the material is homogeneous. CSIG is also homogeneous under a focussed microprobe beam.

Standard: Strontianite

Locality:

Donor: E. Jarosewich to Huebner

Reference: Jarosewich, E. and MacIntyre, I.G., 1983, Carbonate reference samples for electron microprobe and scanning electron microscope analyses. J. Sedimentary Petrology, v. 53, no. 2, p. 677-678.

	Oxide wt.%	
	(1)	(2)
SrO	68.43	68.43
*CaO	0.84	1.68
CO ₂	<u>30.16</u>	<u>30.16</u>
Total	<u>99.43</u>	<u>100.27</u>

*CaCO₃=1.5-3.0 wt.% (per phone conversation with E. Jarosewich, 1/29/85)

Sr	0.968	0.961
Ca	0.022	0.044
C	<u>1.005</u>	<u>0.998</u>
sum cations	<u>1.995</u>	<u>2.002</u>
sum anions	3.0	3.0

Element	Sr	Ca
S.R.	1.4	7.7
#Pts.	20	20
#Grains	16	16

Evaluation: The weight percent sum of the chemical analysis, presumably by the classical methods of Peck, is excellent. Calculated using the lower range Ca value, 0.84 wt%, the formula unit is (Ca_{0.022}Sr_{0.968})_{0.990}C_{1.005}O₃; cations total 1.995 to 3.0 anions. With the higher Ca value of 1.68 wt%, the formula unit (Ca_{0.043}Sr_{0.961})_{1.004}C_{0.998}O_{3.0} yields a cation/anion ratio of 2.002/3.0. CSTR is homogeneous with respect to Sr but heterogeneous with respect to Ca. It should only be used as a Sr standard.

Standard: Orthoclase

Locality: Benson Mines, St. Lawrence County, New York

Donor: D.B. Stewart

References: (1) Stewart, D.B., and Wright, T.L. (1974) Bull. Soc. Fr. Mineral. Cristallogr., 97, 367. Anal., J.J. Fahey, U.S.G.S. (2) Foland, K.A. (1974) Geochim. Cosmochim. Acta, 38, 151-166. (2a) isotope dilution, (2b) microprobe (2c) flame photometry (2d) atomic absorption. (3) U.S.G.S. analysis by Schnepfe (1979) for Wiggins (Lab. # W-203809); SiO₂ and Al₂O₃ were determined calorimetrically; K₂O, Na₂O, CaO, SrO, and BaO were determined by flame atomic absorption. (4) combined analysis. (5) Czamanske's preferred analysis.

	oxide wt.%		oxide wt.%				oxide wt.%		
	(1)	(2a)	(2b)	(2c)	(2d)	(3)	(4)	(5)	
SiO ₂	63.42		64.56			62.62	63.02	63.42	
TiO ₂	0.00					-	0.00	0.01	
Al ₂ O ₃	19.24		19.57			19.52	19.38	19.10	
Fe ₂ O ₃	0.11					-	0.11	0.05	
FeO	-		0.05						
Cr ₂ O ₃	0.001					-	0.001	0.00	
P ₂ O ₅	0.49					-	0.49	0.49	
PbO	0.003					-	0.003	-	
SrO	0.02	0.04				<0.01	0.03	0.02	
GaO	0.001					-	0.001	-	
BaO	0.62					0.48	0.55	0.62	
MnO	0.001					-	0.001	0.00	
CaO	0.08				0.02	<0.01	0.05	0.00	
MgO	0.00					-	0.00	0.01	
Na ₂ O	0.36		0.76	0.66		0.31	0.34	0.20	
NiO	-	-	-	-	-	-	-	0.00	
CuO	0.002					-	0.002	-	
K ₂ O	15.34		15.64	15.20		15.80	15.57	15.90	
Rb ₂ O	-	0.05				-	0.05	-	
B ₂ O ₃	0.010					-	0.010	-	
H ₂ O*	0.02					-	-	-	
H ₂ O-	0.02					-	-	-	
Total	99.74		100.53			98.73	99.61	99.82	
Si	2.941						2.930	2.944	
Ti	0.000						0.000	0.000	
Al	1.051						1.062	1.045	
Fe ³⁺	0.004						0.004	0.0017	
Cr ³⁺	0.000						0.000	0.000	
P	0.019						0.019	0.019	
Pb	0.000						0.000	-	
Sr	0.000						0.001	0.0005	
Ga	-						0.000	-	
Ba	0.011						0.009	0.011	
Mn	0.000						0.000	0.000	
Ca	0.004						0.0025	0.000	
Mg	0.000						0.000	0.001	
Na	0.032						0.031	0.018	
Ni	-						-	0.000	
Cu	0.000						0.0001	-	
K	0.907						0.924	0.942	
Rb	0.002						0.002	-	
B	0.001						0.0008	-	
sum cations	4.973						4.985	4.983	
sum anions	8.000						8.000	8.000	

Element	Si			
S.R.	1.1			
#Pts.	20	Al		
#Grains	20	1.1		
		20		K
		20		1.4
				20
				20

Mineral: Benson Orthoclase

Microprobe analysis: L.B. Wiggins, U.S.G.S., ARL-EMX, 1979

oxide wt.% +1

SiO ₂	64.82+1.73
Al ₂ O ₃	18.74+0.32
FeO	0.08+0.00
CaO	0.00+0.00
Na ₂ O	0.27+0.04
K ₂ O	15.58+0.43
MgO	0.00+0.00
Total	99.49

Evaluation: Available chemical analyses have adequate weight percent sums and provide a consensus for the K₂O and Al₂O₃ contents, but none of the analyses on page 1 can be recalculated to a stoichiometric formula unit. Each formula has a significant cation deficiency in the A position and a slight excess in the tetrahedral sites. For instance, analysis #4 recalculates to [K,Na,Rb,Ca,Sr,Ba,Cu]_{0.969}[B,P,Fe,Al,Ti,Si]_{4.016}O₈. Nevertheless, the Benson orthoclase is widely used as a K standard and as a known-unknown for Ba in feldspars. Excellent sigma ratio values for Si, Al, and K provide proof of homogeneity. Preferred analyses are #4 (based on conventional chemistry) and #5 (based on conventional chemistry, but adjusted to provide an internally consistent microprobe analysis with respect to other feldspars).

Distribution:

Date	From	To	Address
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
15-JUL-83	Huebner	Robert W. Smith	St. Joe Minerals Company P.O. Box 500 Virburnum, MD 65566

Standard: Plagioclase (Labradorite)

Locality: Lake County, Oregon

Donor: D.B. Stewart, December, 1970

Reference: (1) Emmons, R.C., et al. (1953) Anal. Peck. Geol. Soc. Am. Mem. 52.

(2) Stewart et al. (1966) Anal. Fahey. American Mineralogist 51, 128.

(3) Willis Doehring, USGS - Isotope Geology, Denver, XRF. (4) Wiesman and Shih, NASA - JSC, ID (5) Combined analysis.

	Oxide wt%				
	(1)	(2)	(3)	(4)	(5)
SiO ₂	51.08	51.42			51.42
Al ₂ O ₃	31.05	30.76			30.76
TiO ₂	0.05	0.04		0.038	0.04
Fe ₂ O ₃	0.43	0.24			0.24
FeO	0.12	0.17			0.17
MgO	0.22	0.05		0.136	0.14
MnO	0.01	-			0.01
CaO	13.85	13.42			13.42
Na ₂ O	3.38	3.52			3.52
K ₂ O	0.12	0.23	0.125	0.119	0.12
H ₂ O	0.06	0.04			0.04
BaO	0.011	-		0.007	0.007
Li ₂ O	0.001	-		0.001	0.001
SrO	0.142	-	0.074	0.069	0.07
Total	100.52	99.89			99.96
Si	2.318	2.341			2.338
Al	1.658	1.651			1.648
Ti ⁴⁺	0.0017	0.0014			0.0014
Fe ³⁺	0.0147	0.008			0.008
Fe ²⁺	0.0046	0.0065			0.006
Mg	0.015	0.003			0.010
Mn	0.0004	-			0.0004
Ca	0.673	0.655			0.654
Na	0.297	0.311			0.310
K	0.007	0.013			0.007
Ba	0.0002	-			0.0001
Li	0.0002	-			0.0002
Sr	0.0037	-			0.0018
sum cations	4.996	4.990			4.985
sum anions	8.000	8.000			8.000
OH ⁻					0.012

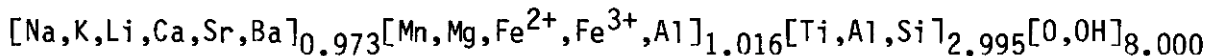
Element Si
S.R 1.4
#Pts. 20
#Grains 4

Al Ca Na
1.1 1.6 1.4
20 20 20
4 4 4

Mineral: Plagioclase (Labradorite)
 Microprobe analysis: Single grain, L.B. Wiggins; U.S.G.S.; 1979; using
 ARL/EMX microprobe

	Oxide wt.% $+1\sigma$
SiO ₂	51.82+0.00
Al ₂ O ₃	30.28+1.64
CaO	13.92+0.25
FeO	0.41+0.04
MgO	0.14+0.00
Na ₂ O	3.66+0.05
Total	100.23

Evaluation: The chemical analysis of Emmons et al. (1953) has a high total of (100.52%); that of Stewart et al. (1966) is excellent, even if the minor elements of Emmons et al. are included (100.05%). The preferred analysis incorporates data from various sources and can be recalculated to a formula unit that is, within the limits of analytical uncertainty, stoichiometric plagioclase:



Sigma ratios for Si, Al, Fe, Mg, Ca, and Na are 1.4, 1.1, 0.8, 1.0, 1.6 and 1.4. FSLC is homogeneous and widely used as a Si, Al, Ca standard for feldspar; as a general purpose Ca standard; and as a "known-unknown."

Date	From	To	Address
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
18-JUL-83	Huebner	Robert W. Smith	St. Joe Minerals Company P.O. Box 500 Viburnum, MD 65566

Standard: Anorthoclase
Locality: Nunivak Island, Alaska
Donor: D.B. Stewart

Reference: 1) D.B. Stewart and T.L. Wright (1974) Bull. Soc. Fr. Mineral. Cristallogr., 97, 356-377. Analyst J.J. Fahey of U.S.G.S. 2) U.S.G.S. Semi-quant Spectrog. Rep. No. 71-WS-133. J.L. Harris, Analyst. 3) G. Czamanske, personal communication, his preferred values by microprobe analysis.

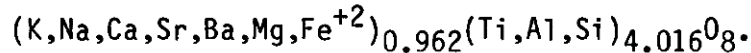
	Oxide wt.% (1)	Oxide wt.% (2)	Oxide wt.% (3)
SiO ₂	66.06	>21.	66.10
Al ₂ O ₃	20.42	19.	20.00
TiO ₂	0.02	0.02	0.03
*Fe ₂ O ₃	0.23	0.21	0.14
MgO	0.02	0.08	0.00
CaO	0.79	0.70	0.90
BaO	0.14	0.11	0.13
Na ₂ O	8.24	13.	8.70
K ₂ O	3.50	4.	3.35
SrO	0.45	0.83	0.61
GaO	-	0.001	-
P ₂ O ₅	-	-	0.21
H ₂ O ⁺	0.02	-	-
H ₂ O ⁻	-	-	-
Total	99.89		100.17

Si	2.937	2.935
Al	1.070	1.047
Ti ⁴⁺	0.0007	0.001
Fe ²⁺	0.008	0.005
Mg	0.0013	0.000
Ca	0.038	0.043
Ba	0.002	0.002
Na	0.710	0.749
K	0.198	0.190
Sr	0.012	0.016
P	-	0.008
sum cations	4.978	4.995
sum anions	8.000	8.000

*Total iron

Element	Si	Al	Ca	Na	K
S.R.		1.0	1.9	2.7	
#Pts.		20	20	20	
#Grains		20	20	20	
			1.3	3.4	4.1
			20	20	20
			20	20	20
	1.2				
	20				
	20				

Evaluation: The chemical analysis reported by Stewart and Wright (1974) appears to be excellent. The cations can be formally assigned to the polyhedral and tetrahedral sites to yield the formula:



Czamanske's preferred values by electron microprobe analysis confirm the original analysis but for the CaO value and the addition of 0.21% P₂O₅. The addition of P₂O₅ to analysis #1 does not improve the formula unit. The homogeneity is excellent for Si and Al, good for Ca, marginal for Na, and unsatisfactory for K. The heterogeneity is not caused by simple K-Na-Ca zoning where high values of one element are associated with low count rates for other elements. FSNA has not been used extensively as a microprobe standard in Reston and, because of both the heterogeneity in K and Na and the poor formula unit, offers little potential as a standard for alkali feldspar.

Date	From	To	Address
06-JUN-77	Huebner	Eric J. Essene	Dept. Geology and Mineralogy University of Michigan Ann Arbor, MI 48104
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
18-JUL-83	Huebner	Robert W. Smith	St. Joe Minerals Company P.O. Box 500 Virburnum, MD 65566

Standard: Albite
 Locality: Tiburon Peninsula, California
 Donor: D.B. Stewart
 References: 1) Maria L. Crawford (1966) Optical properties of metamorphic albite. Am. Mineral. 51, 523-524. Microprobe analysis. 2) theoretical

	oxide wt. % (1)	oxide wt. % (2)
Na ₂ O	-	11.83
Al ₂ O ₃	19.40	19.44
SiO ₂	68.60	68.73
K ₂ O	0.02	0.00
CaO	0.00	0.00
Total	<u>88.02</u>	<u>100.00</u>

Element	Si
S.R.	1.5
#Pts.	20
#Grains	3

Al	Na
1.5	1.0
20	20
4	4
0.6	0.9
18	18
6	6

Evaluation: X-ray powder diffraction and optical properties indicate that the Tiburon albite is pure NaAlSi₃O₈. An incomplete microprobe analysis confirms the theoretical values for SiO₂ and Al₂O₃ and found insignificant K₂O and CaO. Nevertheless, it is disturbing to depend upon a standard that is not supported by a chemical analysis that is both independent and complete. The material is homogeneous. FSTA has been used as a general standard for Na and Al in Reston. Long exposure to the beam could cause loss of alkali.

Standard: Di₈₅Jd₁₅ Glass
 Locality: Synthetic
 Donor: F.R. Boyd (1969) to B.A. Morgan
 References:

	Oxide wt.% (theoretical)
CaO	22.24
MgO	15.98
SiO ₂	56.05
Na ₂ O	2.17
Al ₂ O ₃	3.57
Total	<u>100.00</u>

Element	Si	Ca	Mg	Al	Na
S.R.	1.4	1.5	1.5		
#Pts.	20	20	20		
#Grains	8	8	8		
				1.1	
				20	
				1	
		0.9		1.0	0.9
		20		20	20
		1		1	1

Evaluation: The glass is presumed to have the intended composition. It is very homogeneous and has been used successfully at the Geophysical Laboratory. In Reston, it should prove to be a useful standard for Na₂O and Al₂O₃ in pyroxenes and some volcanic glasses.

Standard: Albite Glass
 Locality: Corning Glass Works
 Donor: D.B. Stewart 6/82

References: 1,2) Replicate analyses of (95GQB³) M13-2435 U.S. Geol. Survey Analytical Laboratories Rep. No. 70-WC-8. J.J. Fahey, analyst. 3) Corning Glass (95GQB³) M13-2435; letter from Corning Glass Works, dated February 19, 1970.

	Oxide wt.%		
	1	2	3
SiO ₂	70.29	70.13	
Al ₂ O ₃	18.17	18.41	
Fe ₂ O ₃	0.00	0.00	
TiO ₂	0.00	0.00	
CaO	0.00	0.00	
MgO	0.00	0.00	
Na ₂ O	11.49	11.43	12.02
K ₂ O	0.00	0.00	0.023
H ₂ O+110°C	0.09	0.09	
Total	100.04	100.06	

Element Si
 S.R. 1.4
 #Pts. 20
 #Grains 6

Al Na
 1.5 4.2
 20 20
 8 8

Evaluation: The albite glass has been examined by a variety of chemical and physical tests. The refractive index is uniform, indicating chemical homogeneity. The unacceptably large sigma ratio for sodium may be related to loss of alkali under the focussed microprobe beam. The USGS analyses appear to be excellent, but there is a significant difference between the USGS and Corning values for Na₂O. This glass will lose alkali when exposed to a focussed electron beam. GFAB should be an adequate standard for defocussed beam analyses of Si and Al in glasses rich in these components.

Standard: Anorthite Glass

Locality: Fusion (at 1577-1602°C in Pt crucible 2 hrs) of CaO, Al₂O₃, and SiO₂ (spectra Sil) in stoichiometric amounts, by James Woodhead (then at Princeton).

Donor: R.A. Robie

References: (1) Ideal formula, CaAl₂Si₂O₈; (2) Robie, R.A., Hemingway, B.S. and Wilson, W.H. (1978) Low-temperature heat capacities and entropies of feldspar glasses and of anorthite. Amer. Mineral. 63, 109-123. Microprobe analysis on USGS ARL-EMX by L.B. Wiggins.

	Oxide wt.%	
	(1)	(2)
SiO ₂	43.19	42.09
Al ₂ O ₃	36.65	37.05
CaO	<u>20.16</u>	<u>20.18</u>
Total	100.00	99.32

Element	Si		
S.R.	1.7		
#Pts.	20		
#Grains	10		
		Al	Ca
		1.3	0.9
		20	20
		12	12

Evaluation: Within analytical uncertainty, electron microprobe analysis confirms the intended values for Ca and Al. The value for Si is low; some Si may have been lost as SiO during fusion at high temperatures. A microprobe check of homogeneity for GFAN reveals a sigma ratio value for Si of 1.7; while those of Al and Ca are 1.3 and 0.9. GFAN should be regarded as a potential standard for Ca and Al.

Standard: Orthoclase Glass
 Locality: Corning Glass Works
 Donor: D.B. Stewart 6/82

References: 1,2) Replicate analyses of Corning Glass (95GQA²) M13-2422;
 U.S. Geol. Survey Analytical Laboratories Report no. 70-WC-8, J.J. Fahey,
 analyst. 3) Corning Glass (95GQA²) M13-2422; letter from Corning Glass
 Works, dated 2/19/70.

	Oxide wt. %		
	1	2	3
SiO ₂	64.71	64.78	
Al ₂ O ₃	18.09	17.97	
Fe ₂ O ₃	0.02	0.02	
TiO ₂	0.00	0.00	
CaO	0.00	0.00	
MgO	0.00	0.00	
Na ₂ O	0.07	0.07	0.024
K ₂ O	16.54	16.49	16.95
H ₂ O-110°C	0.18	0.20	
H ₂ O+110°C	0.58	0.60	
Total	100.19	100.13	

Element	Si		
S.R.	2.6		
#Pts.	20	Al	
#Grains	7	0.9	
		20	K
		6	2.3
			20
			8

Evaluation: The orthoclase glass has been analyzed by several chemical and physical techniques. The complete chemical analyses appear excellent, but the K₂O value is ambiguous in the sense that the Corning K₂O value of 16.95 weight percent is larger than the average USGS value 16.52. The glass had a uniform refractive index when obtained from Corning. GFOR is not stable in the sense that it absorbs moisture from room air (0.5% in one month). Under the focussed electron microprobe beam, the sigma ratio for Al is excellent; values for Si and K are at the margin of acceptability. Perhaps alkali is lost under the focussed microprobe beam. GFOR is a potential standard for Al; if the K₂O value is made certain, GFOR might be a standard for broad beam analyses of K₂O and SiO₂ in "acidic" glasses.

Standard: Barium glass
Locality: Synthetic
Donor: Unknown
Reference:

	Oxide wt.% theoretical
SiO ₂	53.00
BaO	47.00
Total	<u>100.00</u>

Element	Si	Ba
S.R.	1.1	0.8
#Pts.	20	20
#Grains	5	5

Evaluation: The chemistry reported for this glass is, presumably, the intended composition, not a verification that the composition was achieved. The material is very homogenous. For lack of a good alternative, it has been used in Reston as a barium standard. The material is excellent for finding the Ba peak, but if substantive low level Ba analyses are required, FSBO (0.6% BaO) should be used as the Ba standard or as a known-unknown for barium.

Standard: $\text{CaMgSi}_2\text{O}_6$ Glass
Locality: Synthesized by D.B. Stewart
Donor: D.B. Stewart to J.S. Huebner
References: From margins of 25 ml crucible of diopside-composition melt
quenched in water. See PXSD, which is an impurity with the glass.

Oxide Wt. % (theoretical)

SiO ₂	55.49
MgO	18.62
CaO	25.89
Total	<u>100.00</u>

Element	Si	Mg	Ca
S.R.	2.3	1.2	1.2
#Pts	20	20	20
#Grains	1	1	1

Evaluation: Synthetic diopside glass GLDI is homogeneous and appears to be a good standard, despite the lack of an independent chemical analysis. Small amounts of crystalline diopside, PXSD, may be a contaminant, although the composition is similar to GLDI.

Standard: Basaltic glass - VG2
 Locality: Juan de Fuca Ridge
 Donor: W.G. Melson, USNM 111240/52

References: (1) E.J. Jarosewich, J.A. Nelen and J.A. Norbert (1979) Electron microprobe reference samples for mineral analyses. *Smiths. Contrib. Earth Sci.* 22, 68-72. Wet. chem. analysis by Jarosewich. (2) Frey et al. (1974) *J. Geophys. Res.* 79(35), 5507-5528, microprobe analysis of 3 chips. (3) Jarosewich et al. (1979) Microprobe analyses of four natural glasses and one mineral: an interlaboratory study of precision and accuracy. *Smiths. Contrib. Earth Sci.* 22, p. 57. Electron microprobe analyses by A.S. Parker (3a); E. Jarosewich (3b); and L.B. Wiggins (3c), using the preferred standards of each.

	(1)	(2) 1σ	Oxide wt% (3a)	(3b)	(3c)
SiO ₂	50.81	51.2 +0.2	50.85	50.72	50.75
Al ₂ O ₃	14.06	13.6 +0.2	13.81	14.15	13.98
Fe ₂ O ₃	2.23	-	-	-	-
FeO	9.83	11.6 +0.1	11.26	11.79	11.79
MgO	6.71	7.11+0.16	7.01	6.78	7.02
CaO	11.12	10.9 +0.1	10.85	11.14	10.72
Na ₂ O	2.62	2.83+0.1	3.17	2.66	2.75
K ₂ O	0.19	0.20+0.01	0.20	0.21	0.18
TiO ₂	1.85	1.85+0.03	1.86	1.91	1.86
P ₂ O ₅	0.20	-	0.32	0.23	0.19
MnO	0.22	0.19+0.03	0.22	0.22	0.23
H ₂ O	0.02	-	-	-	-
Total	99.86	99.48	99.55	99.81	99.47

Elements	Si	Fe	Mg	Al	Ca	Na
S.R.	0.8	0.9	1.2			
#Pts.	20	20	20			
#Grains	2	2	2			
	0.9	1.6	1.0			
	20	20	20			
	2	2	2			
				1.6	1.6	1.2
				20	20	20
				2	2	2

Mineral: Basaltic glass - VG2

Microprobe analyses: (1) Homogeneity determined from probably 77 five-point counts. Melson, W.G. et al. (1976) The Geophysics of the Pacific Ocean Basin and Its Margin. Geophys. Monogr. 19, p. 352. (2) Microprobe analyses by L.B. Wiggins (Jarosewich et al., 1979, Table 1.). 2a,c,e are averages of 10 points, 2b of 4 points, and 2d of 5 points. Bence Albee correction scheme. Na₂O value of analysis 2b given (presumably incorrect) value of 0.77% in original publication.

	<u>+2σ</u>	<u>2a+1σ</u>	<u>2b+1σ</u>	<u>2c+1σ</u>	<u>2d+1σ</u>	<u>2e+1σ</u>
SiO ₂	+0.35	49.58+0.68	50.25+0.77	49.91+0.48	50.05+0.28	50.75+0.42
Al ₂ O ₃	+0.15	14.46+0.21	14.30+0.43	14.34+0.40	14.61+0.30	13.98+0.19
FeO	+0.17	11.95+0.26	11.83+0.30	11.77+0.19	11.61+0.13	11.79+0.14
MgO	+0.11	6.98+0.10	6.98+0.10	6.91+0.18	6.73+0.03	7.02+0.05
CaO	+0.15	10.91+0.20	10.98+0.28	10.95+0.21	11.24+0.08	10.72+0.06
Na ₂ O	+0.04	2.79+0.11	2.77+0.09	2.86+0.15	2.75+0.05	2.75+0.12
K ₂ O	+0.02	0.02+0.06	0.05+0.07	0.05+0.05	0.10+0.06	0.18+0.06
TiO ₂	+0.06	1.63+0.03	1.68+0.08	1.66+0.07	1.63+0.03	1.86+0.08
P ₂ O ₅	+0.02	0.27+0.05	0.22+0.05	0.28+0.05	0.21+0.07	0.19+0.05
MnO	—	0.23+0.06	0.36+0.03	0.32+0.05	0.08+0.10	0.23+0.05
Total		98.82	97.4	99.05	99.01	99.47

Standards used: (2a,2b,2c,2d)
 KH: Si,Al,Fe,Mg,Ca,Na,K,Ti,Mn
 Ap: P

Standards used:
 Dig₅ Si,Na
 Ortho Al,K,P
 Garnet Fe
 Di2T Mg,Ca,Ti
 Rhod Mn

Evaluation: The wet chemical analysis by Jarosewich et al. (1979a) of basaltic glass VG-2 has an excellent sum. Subsequent microprobe analysis by Frey et al. (1974) yielded values of Al₂O₃, MgO, and Na₂O that differed from the wet chemical analysis by more than 3% of the amount present, but the difference in sums can be attributed solely to the fact that P₂O₅, H₂O, and ferric iron were not determined. Three microprobe laboratories subsequently analyzed GLJF (Jarosewich et al., 1979b); their results bracketed the wet chemical values but for MgO and Na₂O (which were all slightly lower than the wet chemistry) and FeO (all higher). The glass is homogeneous. GLJF has been used as a standard and as a known-unknown for microprobe analyses of basaltic glasses. Despite the inability of the microprobe laboratories to reproduce the MgO and Na₂O values obtained by wet chemical methods, GLJF is one of the best basalt glass standards available.

Standard: Lunar Glass 61156
 Locality: Synthetic CG61156
 Donor: Jean A. Minkin

References: U.S. Geol. Survey Analyt. Lab. Rep. 74DC-1. J.A. Minkin et al. (1976) Three synthetic lunar glasses. *Meteoritics*, 11, 167-171. (1) E.E. Engelman, D.R. Norton, and R.L. Rahill, analysts; conventional rock analysis plus a color. det. of Cr₂O₃. (2) R.F. Christian, anal., x-ray fluorescence.

	Oxide wt.%	
	(1)	(2)
SiO ₂	45.24	45.00
Al ₂ O ₃	23.15	23.06
Cr ₂ O ₃	0.07	-
FeO	7.82	7.71
MgO	9.18	9.05
CaO	13.26	13.55
Na ₂ O	0.17	-
K ₂ O	0.13	0.17
TiO ₂	0.56	0.56
P ₂ O ₅	0.21	-
MnO	0.14	0.14
Total	<u>100.02</u>	<u>99.24</u>

Element	Si	Fe	Mg
S.R.	2.5	1.9	9.5
#Pts.	20	20	20
#Grains	2	2	2

Ca	Na	Al
1.8	1.0	4.6
20	20	20
2	2	2

Microprobe analysis: U.S.G.S. ARL-EMX microprobe. Bence-Albee method.
 J.A. Minkin, analyst. Average of 10 points on each of 4 grains.

Oxide wt.% $\pm 1\sigma$

SiO ₂	45.5 \pm 0.9
Al ₂ O ₃	22.7 \pm 0.5
FeO	7.7 \pm 0.2
MgO	9.2 \pm 0.6
CaO	13.0 \pm 0.3
Na ₂ O	0.29 \pm 0.08
K ₂ O	0.17 \pm 0.08
TiO ₂	0.56 \pm 0.08
MnO	0.12 \pm 0.02
Cr ₂ O ₃	0.04 \pm 0.02
Total	<u>99.28</u>

Evaluation: Synthetic Corning Glass CG61156 has been analyzed by conventional and x-ray fluorescence methods. The sum of the conventional analysis, which is more complete, is excellent. Sigma ratios for Al, and Mg are unacceptably large. The variation of the Mg and Fe count rates is coupled in the sense that points with the highest Mg counts have the lowest Fe counts. Because of its heterogeneity, GLL1 should not be used as a microprobe standard if an alternative standard is available.

Standard: Corning glass (same composition as Lunar 77135)

Locality: Synthetic

Donor: Jean A. Minkin

Reference: U.S. Geol. Survey Anal. Lab. Rep. #74 DC-1; J.A. Minkin et al. (1976) Three synthetic lunar glasses. *Meteoritics* 11, 167-171. (1) E.E. Engleman, D.R. Norton, and R.L. Rahill, analysts; conventional rock anal., plus a colorimetric determ. of Cr₂O₃. (2) X-ray fluor. anal. by R. Christian

	Oxide wt.%	
	(1)	(2)
SiO ₂	46.99	46.79
Al ₂ O ₃	18.11	17.44
FeO	9.18	9.08
MgO	12.19	12.20
CaO	10.87	10.96
Na ₂ O	0.27	-
K ₂ O	0.27	0.29
TiO ₂	1.47	1.44
Cr ₂ O ₃	0.05	-
P ₂ O ₅	0.23	-
MnO	0.14	0.14
Total	99.77	98.34

Element	Si	Fe	Mg
S.R.	2.9	4.9	26.3
#Pts.	20	20	20
#Grains	3	3	3

Al	Ca
3.6	4.2
20	20
3	3

Mineral: Lunar Glass 77135

Microprobe analysis: U.S.G.S. ARL-EMX microprobe. Bence-Albee method.
Minkin et al. (1976). J.A. Minkin, analyst. average of 10 points on
each of 4 grains.

oxide wt. % $\pm 1\sigma$

SiO ₂	46.9 ± 1.4
Al ₂ O ₃	17.8 ± 0.4
FeO	9.1 ± 0.3
MgO	11.4 ± 0.3
CaO	11.0 ± 0.4
Na ₂ O	0.36 ± 0.06
K ₂ O	0.34 ± 0.08
TiO ₂	1.42 ± 0.13
MnO	0.10 ± 0.03
Cr ₂ O ₃	0.03 ± 0.02
Total	98.45

Evaluation: Corning glass 77135, so named because it has the same composition as Lunar basalt 77135, has been analyzed by 3 methods. The conventional rock and X-ray fluorescence methods agreed well, but for the Al₂O₃ values. The electron microprobe analysis, using unknown standards, has a MgO value that is low compared with the other methods; the probe value for Al₂O₃ falls between the values obtained by the other methods. Sigma ratio values are high for all elements analyzed. The covariation of Mg and Fe count rates for individual points is striking: the highest Mg counts are associated with the lowest Fe counts. A. El Goresy (pers. comm. to Minkin et al.) reported a slight difference for some grains between the composition of the outside edge and the core. Minkin obtained a relatively large range of values for SiO₂ by microprobe analysis. GLL7 is not homogeneous and should not be used as a microprobe standard.

Standard: Lunar Glass 68415,85
 Locality: Synthetic (Corning Glass)
 Donor: Jean A. Minkin

References: U.S. Geol. Survey Anal. Lab. Rep. #74 DC-1. J.A. Minkin et al. (1976). Meteoritics, 11, 167-171. (1) E.E. Engleman, D.R. Norton and R.L. Rahill, analysts; conventional rock analysis, plus a colorimetric determination of Cr₂O₃. (2) R.P. Christian, analyst, x-ray fluorescence.

	Oxide wt.%	
	(1)	(2)
SiO ₂	45.10	44.88
Al ₂ O ₃	29.13	29.33
Cr ₂ O ₃	0.11	-
FeO	4.17	4.18
MgO	4.29	4.36
CaO	16.06	16.33
Na ₂ O	0.39	-
K ₂ O	0.12	0.11
TiO ₂	0.26	0.24
P ₂ O ₅	0.07	-
MnO	0.05	0.05
Total	99.75	99.48

Element	Si	Fe	Mg	Al	Ca
S.R.	1.3	0.8	2.2		
#Pts.	19	19	19		
#Grains	3	3	3		
				1.5	1.2
				20	20
				3	3

Mineral: Lunar Glass 68415,85

Microprobe analysis: U.S.G.S. ARL-EMX microprobe. Weight percent values are based on working curves from a variety of standards. J.A. Minkin, analyst. Average of 10 points on each of four grains.

Oxide wt.% $\pm 1\sigma$

SiO ₂	45.5	± 0.9
Al ₂ O ₃	28.9	± 0.4
FeO	4.1	± 0.2
MgO	4.4	± 0.2
CaO	15.9	± 0.4
Na ₂ O	0.53	± 0.07
K ₂ O	0.16	± 0.08
TiO ₂	0.28	± 0.08
MnO	0.10	± 0.02
Cr ₂ O ₃	0.06	± 0.04
Total	99.93	—

Evaluation: Synthetic Corning Glass 86415,85, analysed by conventional rock analysis and X-ray fluorescence in the U.S.G.S. Reston laboratories, has an adequate sum. The three available analyses agree remarkably well. Electron microprobe traverses by J. Minkin and the sigma ratios indicate chemical homogeneity for all elements. It should be noted, though, that we encountered a single anomalous point for which the Mg count rate exceeded the mean by 50% (and which had normal Fe and Si count rates). GLL8 should be a good standard for basaltic glasses rich in anorthite component.

Standard: Basaltic glass - VG-A99 (USNM 113498/1)

Locality: Makaopuhi lava lake, Hawaii

Donor: W.G. Melson

References: (1) Jarosewich et al. (1979) Smithsonian Contr. Earth Sci. 22, 68-72. Wet chemical analysis. (2) T.L. Wright, USGS Prof. Paper 1004, MP-69-1-22. L.C. Peck, Analyst, Wet chemical analysis. 3) R.T. Helz, 29/MAR/82, preferred analysis. (4) Microprobe analyses using preferred standards of each of 3 laboratories. Jarosewich et al., (1979) p. 57. (4a,b) MIT and Smithsonian, average of 4 analyses, <10 pts. each analysis. (4c) U.S.G.S. ARL-EMX microprobe, average of 10 individual points, L.B. Wiggins, analyst.

	Oxide wt.%					
	(1)	(2)	(3)	(4a)	(4b)	(4c)
SiO ₂	50.94	50.90	50.90	51.05	51.22	50.80
Al ₂ O ₃	12.49	12.97	12.97	12.59	12.66	12.80
Fe ₂ O ₃	1.87	1.65	1.65	-	-	-
FeO	11.62	11.70	11.70	13.24	13.47	13.41
MgO	5.08	5.18	5.18	5.24	4.95	5.16
CaO	9.30	9.38	9.38	9.08	9.28	8.97
Na ₂ O	2.66	2.73	2.73	2.81	2.70	2.73
K ₂ O	0.82	0.80	0.80	0.82	0.90	0.76
TiO ₂	4.06	3.89	4.06	4.04	4.05	3.77
P ₂ O ₅	0.38	0.41	0.41	0.54	0.46	0.31
MnO	0.15	0.20	0.20	0.19	0.22	0.19
H ₂ O	0.02	0.12	-	-	-	-
CO ₂	-	0.00	-	-	-	-
Cl	-	0.03	-	-	-	-
F	-	0.06	-	-	-	-
Total	<u>99.39</u>	<u>100.02</u>	<u>100.07</u>	<u>99.6</u>	<u>99.6</u>	<u>98.9</u>
-O=F,Cl		0.03	0.03			
Total		<u>99.99</u>	<u>100.04</u>			

Element	Si	Fe	Mg
S.R.	1.0	1.1	0.8
#Pts.	20	20	20
#grains	2	2	2

Al	Ca	Na
0.8	0.8	0.9
20	20	20
2	2	2

Mineral: Basaltic glass - VG-A99 (USNM 113498/1)
 Microprobe analyses: (1) U.S.G.S. ARL-SEMQ microprobe. Bence-Albee reduction 08/FEB/82. J. McGee, analyst. Average of 5 points. (2) ARL-EMX microprobe. Wiggins, analyst, Jarosewich et al. (1979) p. 66. (2a) average of 10 points. (2b) average of 6 points. (2c) average of 10 points. Same as analysis (4c), page 1.

	oxide wt.% $\pm 1 \sigma$	oxide wt.% $\pm 1 \sigma$	oxide wt.% $\pm 1 \sigma$	oxide wt.% $\pm 1 \sigma$
	(1)	(2a)	(2b)	(2c)
SiO ₂	49.64 \pm 0.17	50.80 \pm 0.51	50.69 \pm 0.28	50.80 \pm 0.60
Al ₂ O ₃	12.35 \pm 0.03	12.84 \pm 0.30	12.62 \pm 0.08	12.80 \pm 0.26
Fe ₂ O ₃	-	-	-	-
FeO	13.10 \pm 0.09	13.36 \pm 0.15	13.50 \pm 0.27	13.41 \pm 0.23
MgO	5.10 \pm 0.08	4.99 \pm 0.10	5.18 \pm 0.08	5.16 \pm 0.10
CaO	9.20 \pm 0.08	8.42 \pm 0.48	9.13 \pm 0.18	8.97 \pm 0.17
Na ₂ O	2.37 \pm 0.03	2.88 \pm 0.03	2.69 \pm 0.07	2.73 \pm 0.08
K ₂ O	0.87 \pm 0.03	0.78 \pm 0.11	0.67 \pm 0.08	0.76 \pm 0.07
TiO ₂	3.81 \pm 0.07	3.79 \pm 0.10	3.71 \pm 0.10	3.77 \pm 0.12
P ₂ O ₅	-	0.49 \pm 0.04	0.54 \pm 0.02	0.31 \pm 0.04
MnO	0.22 \pm 0.00	0.27 \pm 0.05	0.16 \pm 0.04	0.19 \pm 0.06
Cr ₂ O ₃	0.01 \pm 0.01	-	-	-
Total	96.68 \pm 0.25	98.62	98.89	98.9

Standards:
 FSTA - Na,Al
 OLST - Mn
 OXIL - Fe,Ti
 OXTB - Cr
 Or-1 Orthoclase - K
 PXAD - Ca,Si

Standards: (2a,2b)
 AMKH - Si,Al,Fe,Mg,
 Ca,Na,K,Ti,Mn
 APFD - P

Standards: (2c)
 Di85 - Si,Na
 Ortho - Al,K,P
 Garnet - Fe
 Di2Ti - Mg,Ca,Ti
 Rhod - Mn

Evaluation: Chemical analysis (2) has a superior sum. Analysis (3), the preferred analysis, is equivalent to analysis (2) but for the use of the earlier TiO₂ value. Three laboratories have carefully reanalyzed GLMP using microprobe methods and each laboratory's preferred standards; their agreement with analysis (2) is generally excellent: The homogeneity of GLMP is also excellent; the sigma ratio values cluster about the ideal value of 1.0. Standard deviations calculated from replicate microprobe analyses reflect the analytical method (fixed versus scanning wavelength spectrometers) rather than any intrinsic heterogeneity of the sample. GLMP should be considered an excellent standard for Na,Al,Mg,Si,Ca,Ti, and Fe in basaltic glasses. It is suitable for use as a known-unknown.

Standard: Silica Glass

Locality: Synthetic

Donor: D.B. Stewart to J.S. Huebner

References: (1) Stewart, 1960, The System $\text{LiAlSiO}_4\text{-NaAlSi}_3\text{O}_8\text{-H}_2\text{O}$ + 2000 bars. Intern. Geol. Congress 21st Session. Ignited and washed Corning silica glass cullet 7940, heated to 1100°C for 3 hrs. Quantitative Spectrographic analysis by H. Bastron. (2) Emission Semiquantitative Spectrographic Analysis Rept. RESC0015; sample W-186838. Norma Rait, analyst.
 oxide wt. %

	(1)		(2)
SiO ₂	100.00 (assumed)	P ₂ O ₅	0.175
FeO	0.0000	MnO	0.0006
Al ₂ O ₃	0.0005		
MgO	0.0007		
CaO	0.0015		
Total	<u>100.00</u>		

Element	Si
S.R.	1.1
#Pts.	20
#Grains	15

Evaluation: The assumed SiO₂ value is 100.0%. The P₂O₅ reported in the semi-quantitative spectrographic analysis is within one standard deviation of the detection limit and can be considered not detectable with reasonable certainty (per Janet Fletcher, March 1, 1985). GLSI should be an excellent standard for Si in silica-rich materials and for background determination of elements other than Si.

Standard: REE 1
 Locality: Synthetic
 Donor: M.J. Drake
 Reference: Drake, M.J. and D.F. Weill (1972) New rare earth element standards for electron microprobe analysis. Chem. Geol. 10, 179-181.
 (1) Theoretical; (2) INAA Oxide wt.% $\pm 2\sigma$

	Oxide wt.% (1)	Oxide wt.% $\pm 2\sigma$ (2)
Al ₂ O ₃	30.52	
SiO ₂	26.96	
CaO	25.16	
EuO	4.20	4.31 \pm 0.57
Gd ₂ O ₃	4.46	
Tb ₂ O ₃	4.35	4.56 \pm 0.58
Tm ₂ O ₃	4.35	
Total	<u>100.00</u>	

Element	Si	Al	Ca	Eu	Gd	Tb	Tm
S.R.	1.1						
#Pts.	20						
#grains	2	0.9	1.1	1.0	1.0	0.9	1.0
		20	20	20	20	20	20
		2	2	2	2	2	2

Evaluation: Independent chemistry is available only for Eu and Tb, for which the INAA results were 3-5% greater than expected. The glass is homogeneous. Drake and Weill (1972) report that "the standard deviation from the mean of a large number of spot analyses is equal to the uncertainty due to counting statistics." (p. 180). We confirmed this conclusion: our sigma ratios cluster close to the ideal value of unity. Glass GRE1 should be a good standard for analyzing small concentrations of Eu and Tb, and because of similar chemical behavior, also Gd and Tm.

Standard: REE Glass 2

Locality: Synthetic

Donor: M. J. Drake

Reference: Drake, M.J. and D.F. Weill (1972) New rare earth element for electron microprobe analysis. Chem. Geol. 10, 179-181. (1) Theoretical; (2) INAA oxide wt.% $\pm 2\sigma$

	oxide wt.% (1)	oxide wt.% $\pm 2\sigma$ (2)
Al ₂ O ₃	30.63	
SiO ₂	27.07	
CaO	25.26	
Nd ₂ O ₃	4.26	4.20 \pm 0.89
Sm ₂ O ₃	4.26	4.32 \pm 0.09
Yb ₂ O ₃	4.26	4.08 \pm 0.10
Lu ₂ O ₃	4.26	4.26 \pm 0.09
Total	<u>100.00</u>	

Element	Si	Al	Ca	Yb	Lu	Nd	Sm
S.R.	1.2						
#Pts.	20	1.0	0.8				
#Grains	4	20	20				
		3	3				
			2.6	2.0			
			20	20			
			4	4			
				1.9	1.0		
				20	20		
				3	3		
						0.9	
						20	
						4	
							0.9
							20
							3

Evaluation: Independent chemistry is available only for the rare earths; the INAA values for Nd, Sm, Yb, and Lu agree to within 4% with the predicted values. REE glass 2 is homogeneous: Drake and Weill (1972) report that "the standard deviation from the mean of a large number of spot analyses is equal to the uncertainty due to counting statistics," and our determination of the sigma ratios tends to agree with this conclusion (although we found the homogeneity of Yb and Ca to be significantly less than that of the other elements). GRE2 should be an excellent standard for analyzing Lu, Nd, and Sm, and an adequate standard for low levels of Yb.

Standard: REE 3
 Locality: Synthetic
 Donor: M.J. Drake
 Reference: Drake, M.J. and D.F. Weill (1972) New rare earth element standards for electron microprobe analysis. Chem. Geol. 10, 179-181. (1) theoretical; (2) INAA

	Oxide wt. %	
	(1)	(2)
Al ₂ O ₃	30.72	
SiO ₂	27.15	
CaO	25.33	
Y ₂ O ₃	4.08	
La ₂ O ₃	4.28	4.59±0.12
Ce ₂ O ₃	4.00	4.30±0.27
Pr ₂ O ₃	4.44	4.60±0.19
Total	100.00	

Element	Si							
S.R.	1.1							
#Pts.	20	Al	Ca					
#Grains	1	1.4	1.0					
		20	20	La				
		1	1	0.9				
				20	Y	Ce		
				1	1.0	1.1		
					20	20		Pr
					1	1		1.1
								20
								1

Evaluation: The only independent chemistry consists of the INAA results for La, Ce, Pr concentrations which are slightly higher than expected. Microprobe analyses by Drake and Weill (1972) show sample REE 3 to be homogeneous with "the standard deviation from the mean of a large number of spot analyses is equal to the uncertainty due to counting statistics." Our determination of the sigma ratios confirms this conclusion. GRE3 should be a good standard for use when analyzing small concentrations of La, Ce, Pr, and (because of similar chemical behavior) Y.

Standard: REE4
 Locality: Synthetic
 Donor: M.J. Drake
 Reference: Drake, M.J. and D.F. Weill (1972) New rare earth element standards for electron microprobe analysis. Chem. Geol. 10, 179-181. (1) REE Glass intended composition. (2) INAA Oxide wt.% $\pm 2\sigma$

	Oxide wt.% (1)	Oxide wt.% (2)
Al ₂ O ₃	32.08	
SiO ₂	28.34	
CaO	26.45	
Dy ₂ O ₃	4.36	
Ho ₂ O ₃	4.41	4.37±0.02
Er ₂ O ₃	4.36	
Total	100.00	

Element	Si	Ca	Al	Er	Ho	Dy
S.R.	1.1	0.9				
#Pts.	20	20				
#Grains	2	2				
			0.9	1.1		
			20	20		
			2	2		
					1.1	
					20	
					2	
						1.3
						20
						2

Evaluation: Independent chemistry is available only for the rare earth, holmium; the INAA value agrees to within 1% of the intended value. REE glass 4 is homogeneous: Drake and Weill (1972) report that the standard deviation from the mean of a large number of spot analyses is equal to the uncertainty due to counting statistics, and our determination of the sigma ratios agrees with this conclusion. GRE4 should be an excellent standard for Ho and probably also for Dy and Er. (It would be reassuring to have a direct confirmation that GRE4 contains close to the intended concentrations of Dy and Er.)

Standard: Rhyolite Glass, RLS 132

Locality: Tulancingo, Mexico

Donor: R.L. Smith via R.A. Bailey

References: (1) University of Reading, England (2) U.S.G.S. analytical chemistry Laboratory. (3) U.S.G.S. Analytical Laboratory sample #W-204910. Be, Li, Mo, Nb, Sn, and W by special methods. Li by AAS; Be and Sn by flameless AAS; Mo, W, and Nb determined spectrophotometrically; Co, Cr, Cs, Fe, Hf, Rb, Sb, Sc, Ta, Th, U, and REE by INAA. (4) University of Lancaster, Reading, England, X-ray fluorescence method.

	Oxide Wt.% (1)	Oxide Wt.% (2)	Oxide Wt. % (3)	Oxide Wt. % (4)
SiO ₂	75.7		BeO	0.000278
Al ₂ O ₃	11.44		Rb ₂ O	0.0208
Fe ₂ O ₃	1.86		Nb ₂ O ₅	0.0119
FeO	0.45		FeO	2.02
MgO	0.05		Cr ₂ O ₃	0.00073
CaO	0.12		Cs ₂ O	0.000435
Na ₂ O	5.25		HfO ₂	0.00288
K ₂ O	4.53		Li ₂ O	0.0153
H ₂ O	0.07		CoO	0.00025
TiO ₂	0.21		Mo ₂ O ₃	0.000825
P ₂ O ₅	0.01		Sb ₂ O ₃	0.000036
MnO	0.15		Sc ₂ O ₃	0.00048
Cl	0.18	0.19	SnO	0.00070
F	0.23	0.19	Ta ₂ O ₅	0.00068
Subtotal	<u>100.25</u>		ThO ₂	0.00205
-O=F,Cl	0.13		UO ₂	0.00073
Total	<u>100.12</u>		WO ₃	0.00020
			La ₂ O ₃	0.00457
			Ce ₂ O ₃	0.0107
			Nd ₂ O ₃	0.00373
			Sm ₂ O ₃	0.00128
			EuO	0.00016
			Gd ₂ O ₃	0.00114
			Tb ₂ O ₃	0.00026
			Tm ₂ O ₃	0.00020
			YbO	0.00136
			Lu ₂ O ₃	0.00020

Element	Si	Fe	Al	Na	K
S.R.	1.2	1.0		2.2	
#Pts.	20	20		20	
#Grains	14	14		14	
		1.1	2.6		0.9
		20	20		20
		13	13		13
		0.6	0.9	3.2	
		20	20	20	
		13	13	13	
		0.9	1.0	3.9	
		20	20	20	
		13	13	13	

Mineral: Rhyolite Glass

Mineral Analysis: SEMQ U.S.G.S., Reston, Microprobe Analyses; 20 points;
J. McGee, analyst; 13-MAR-84; Na₂O may be low due to mobilization.

Oxide Wt. %

SiO ₂	76.00
Al ₂ O ₃	11.50
FeO	2.02
MnO	0.16
MgO	0.08
CaO	0.10
Na ₂ O	4.75
K ₂ O	4.47
TiO ₂	0.19
SrO	0.01
S	0.03
Total	<u>99.30</u>

Standards:

Si - USNM tektite glass	Na - GLMP
Al - GLJF	K - USNM tektite glass
Fe - GLMP	Ti - GLMP
Mn - OLST	Sr - CSTR
Mg - GLMP	S - troilite
Ca - GLJF	

Evaluation: The summation of wet chemical analysis (1) is excellent and the trace element data (3,4) are unusually complete. Independent analyses confirm the values for Cl and F (analysis 2) and 9 major oxides (the microprobe analysis). The homogeneity of the glass is excellent for Si, K, Fe, and probably for Al. The sigma ratio for Na is close to an acceptable value. (The variation in the Na count rates does not appear to be due to an unstable spectrometer with gas flow counter.) With the exception of Na, GRLS should be an excellent standard for siliceous glasses, for halogens, and for exploring the analyses of trace elements with the microprobe.

Standard: $\text{CaMgSi}_2\text{O}_6$ Glass
Locality: Synthetic
Donor: F.R. Boyd
References:

Oxide Wt.% (theoretical)

CaO	25.89
MgO	18.62
SiO ₂	55.49
Total	<u>100.00</u>

Element	Si	Ca	Mg
S.R.	0.9	1.1	1.3
#Pts.	20	20	20
#Grains	2	2	2

Evaluation: Diopside glass GSDI is presumed to have the intended composition, $\text{CaMgSi}_2\text{O}_6$. The material is clear and chemically homogenous. It should be a good standard for Ca, Mg, and Si in pyroxenes and glasses rich in diopside component. Users might first want to compare this material to GLDI, PXSD, PXAD, or PXPS, which are all compositionally similar.

Standard: MgSiO₃ Glass
Locality: Synthetic
Donor: F.R. Boyd
References:

Oxide wt. %

MgO	40.15
SiO ₂	59.85
Total	<u>100.00</u>

Element	Si	Mg
S.R.	0.9	1.2
#Pts.	20	20
#Grains	1	1
	1.0	0.9
	20	20
	1	1
		0.9
		20
		1

Evaluation: The glass of MgSiO₃ composition, GSEN, is clear, very homogenous, and assumed to have the intended composition. Its composition should be verified by comparison with crystalline enstatites PXEN and PXSE.

Standard: CaSiO₃ Glass
Locality: Synthetic
Donor: F.R. Boyd (Geophysical Laboratory) to Huebner
References: E.F. Osborn, Original source (1938)

Oxide wt. %

CaO	48.28
SiO ₂	51.72
Total	<u>100.00</u>

Element	Si	Ca
S.R.	1.0	1.3
#Pts.	20	20
#Grains	9	9

Evaluation: Wollastonite glass GSWO is presumed to have the intended composition, CaSiO₃. The glass is homogeneous. It should be a suitable standard for Ca in Ca-rich glasses, pyroxenes, and pyroxenoids. Before use, its composition should be verified by comparing count rates with the compositionally similar GWOL.

Standard: CaSiO₃ Glass - CaSiO₃

Locality: Synthetic

Donor: T. Haselton

References: fusion of CaO and silica glass (from E.W. Roedder) 1.5 hrs. at
1620°C. Run #P.65

Oxide Wt. %

CaO	48.28
SiO ₂	51.72
Total	<u>100.00</u>

Element	Si	Ca
S.R.	0.9	
#Pts.	20	
#Grains	4	
	0.9	0.8
	20	20
	3	3

Evaluation: Wollastonite glass GWOL is presumed to have the intended composition because it was carefully prepared under conditions that should not permit the bulk composition to change. The material is exceptionally homogenous. GWOL should prove to be a satisfactory standard for Ca-rich glasses and calcic pyroxenes or pyroxenoids. It should also be a good background standard.

Standard: Garnet 12442

Locality:

Donor: J.C. Stormer (originally obtained from Ian Carmichael, Berkeley, CA)

References: (1) wet chemistry, I.S.E. Carmichael, analyst. (2) wet chemistry, gravimetric, colorimetric, atomic absorption, R. Smith, analyst; University of Georgia

	Oxide wt.%	
	(1)	(2)
SiO ₂	39.0	39.82
TiO ₂	0.06	-
Al ₂ O ₃	22.1	-
FeO	21.99	21.76
MnO	0.49	-
MgO	11.53	12.40
CaO	4.20	4.06
Na ₂ O	0.0	-
K ₂ O	0.0	-
H ₂ O ⁺	0.14	-
P ₂ O ₅	0.04	-
V ₂ O ₃	~0.01	-
Cr ₂ O ₃	0.0	-
Sc ₂ O ₃	0.0	-
Total	99.56	

Si	2.954
Ti	0.003
Al	1.973
Fe ²⁺	1.359
Fe ³⁺	0.034
Mn	0.031
Mg	1.302
Ca	0.341
Na	0.00
K	0.00
P	0.003
V	0.00
Cr	0.00
Sc	0.00
sum cations	8.000
sum anions	12.000

OH 0.071

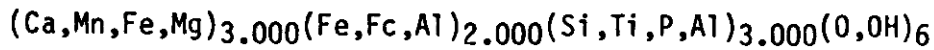
†total iron

*natural homogeneous garnet

**preferred analysis

Element	Si	Fe	Mg	Al	Ca
S.R.	0.9	2.0	2.4		
#Pts.	20	20	20		
#Grains	9	9	9		
			1.5	1.0	3.1
			20	20	20
			10	10	10
			4.0	1.5	3.7
			20	20	20
			10	10	10

Evaluation: The sum of Carmichael's analysis (1) is several tenths of a percent low, whereas adoption of R. Smith's revised values (2) gives an unacceptably high total, 100.9%. In lieu of a ferric iron determination, analysis (1) was recalculated to a formula unit that included structural (OH) and assumed sufficient Fc to achieve perfect stoichiometry:



(The stoichiometric formula corresponds to an analysis with 0.59 percent Fe₂O₃ by weight and totalling 99.61%.) This formula unit is a reasonable garnet formula. Stormer reports that Garnet 12442 is homogeneous at the scale of microprobe analysis, but the sigma ratio for Ca is unacceptably large. GTAL might be considered as a standard for Fe in almandine-rich garnets, providing that the heterogeneity in Ca does not affect the matrix corrections.

Standard: Pyrope Garnet
 Locality: Kakanui, New Zealand
 Donor: B. Mason USNM 143968

Reference: 1. Mason, B., and R.O. Allen (1973) New Zealand Journal of Geology and Geophysics, 16, 935-947. 2. Jarosewich et al. (1976-7) Smithsonian Contributions to the Earth Sciences 22, p. 71.

	Oxide wt. %	
	(1)	(2)
SiO ₂	41.45	41.46
TiO ₂	0.51	0.47
Al ₂ O ₃	23.50	23.73
FeO	10.08	10.68
Fe ₂ O ₃	.76	-
MnO	0.28	0.28
MgO	18.51	18.51
CaO	5.09	5.17
Na ₂ O	0.07	<0.01
K ₂ O	-	-
Total	<u>100.25</u>	<u>100.30</u>

Si	2.963	2.964
Ti	0.027	0.025
Al	1.980	1.999
Fe ²⁺	0.603	0.639
Fe ³⁺	0.041	0.000
Mn	0.017	0.016
Mg	1.972	1.972
Ca	0.390	0.396
Na	0.010	0.000
K	-	0.000
sum cations	<u>8.004</u>	<u>8.011</u>
sum anions	12.000	12.000

Element	Si	Fe	Mg
S.R.	1.6	2.0	3.0
#Pts.	19	20	20
#Grains	4	4	4

Al	Ca
1.4	1.1
20	20
8	8

Mineral: Pyrope-Garnet

Microprobe analysis: 1) and 2) U.S.G.S. EMX microprobe, Wiggins, analyst;
 3) U.S.G.S. ARL-SEMQ microprobe, J. McGee, analyst, 08-FEB-82; 4) U.S.G.S.
 ARL-SEMQ microprobe, J. Stormer, analyst; 100kV [sic], 20-OCT-82

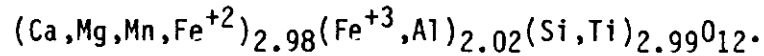
	(1) 1 grain	oxide wt.% (2) 4 grains	(3) 10 points	counts +1σ (4) 10 points
SiO ₂	41.24±0.06	41.64±0.34	41.37±0.27	Al 71992±689
Al ₂ O ₃	23.61±0.32	23.73±0.24	23.50±0.08	Fe 7663±126
CaO	5.21±0.18	5.10±0.08	5.02±0.12	
FeO	10.33±0.33	10.77±0.15	10.37±0.07	
MgO	18.61±0.36	18.82±0.30	18.28±0.18	
Na ₂ O	- -	- -	0.02±0.00	
MnO	- -	- -	0.33±0.01	
TiO ₂	- -	- -	0.41±0.04	
Cr ₂ O ₃	- -	- -	0.07±0.02	
K ₂ O	- -	- -	0.02±0.01	
Total	99.00	100.06	99.39±0.42	

Si	2.972	2.971	2.979
Al	2.005	1.995	1.995
Ca	0.402	0.390	0.387
Fe ²⁺	0.571	0.580	0.622
Fe ³⁺	0.052	0.063	0.003
Mg	1.999	2.001	1.963
Na	-	-	0.003
Mn	-	-	0.020
Ti	-	-	0.022
Cr	-	-	0.004
K	-	-	0.002
cations	8.000	8.000	8.000
anions	12.000	12.000	12.000

Standards:
 FSTA - Na,Al
 OLST - Mn
 OXIL - Ti,Fe
 OXTB - Cr
 Or-1 Orthoclase
 PXAD - Ca,Si,Mg

Standard:
 GTKN - Al,Fe

Evaluation: The original and revised chemical analyses of the Kakanui pyrope appear to be of superior quality. Within limits of analytical uncertainty, each analysis can be recalculated to a stoichiometric garnet formula,



The homogeneity is good for all elements except Mg which has a high sigma ratio of 3.0. There is some covariation between the Mg and Fe count rates, indicating that the cause of the poor Mg heterogeneity is at least in part due to small variations in Fe/Mg. The chemistry has been adequately confirmed by microprobe analysis. GTKN is a good general purpose standard for Al, Si, Ca, and Fe in garnets, pyroxenes, biotites, and hornblendes. It has been widely used as a "known-unknown". It should continue to serve well when at least three, and preferably five, replicate measurements are made during a standardization.

Date	From	To	Address
19-NOV-76	Huebner	Arden Albee	Division of Geological and Planetary Sciences California Institute of Technology Pasadena, California 91125

Standard: Garnet
 Locality: Roberts Victor Mine, S. Africa
 Donor: G. Switzer to B.A. Morgan to J.S. Huebner, 05/1968
 Reference: USNM 110752

	Oxide Wt. %
MgO	7.17
Al ₂ O ₃	22.70
SiO ₂	40.16
CaO	18.12
TiO ₂	0.35
MnO	0.19
Fe ₂ O ₃	2.17
FeO	9.36
H ₂ O	<0.01
Total	<u>100.22</u>

Mg	1.588	1.597	
Al	3.975	3.999	
Si	5.968	6.003	
Ca	2.885	2.902	
Ti	0.039	0.039	
Mn	0.024	0.024	
Fe ³⁺	0.243	-	
Fe ²⁺	1.163	1.414	
sum cations	<u>15.884</u>	<u>15.978</u>	adjusted
sum anions	24.0	24.000	

Element	Si	Fe	Mg	Al	Ca
S.R.	2.6	1.8	2.5		
#Points	20	20	20	0.9	1.5
#Grains	19	19	19	20	20
				19	19

Evaluation: The chemical analysis of the garnet from the Roberts Victor Mine has a good total, 100.2 wt.%. The formula unit, (Ca,Mn,Fe,Mg)_{5.660}(Fc,Al)_{4.218}(Ti,Si)_{6.007}O₂₄, suggests that the garnet is non-stoichiometric or the analysis is in error. Conversion of all ferric iron to ferrous iron improves the stoichiometry, suggesting that the determination of both FeO and Fe₂O₃ is indeed in error. The material is homogeneous and can be considered a good potential standard, pending confirmation of its chemistry.

Standard: ALMAN 1 Spessartine Garnet
 Locality: Minas Gerais, Brazil
 Donor: J. Stormer (originally obtained from Luis Garcia, Minas Gerais, Brazil)
 References: Combination of wet chemical, XRF, and microprobe values.

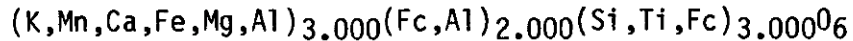
Oxide wt.%

Na ₂ O	0.00
MgO	0.06
Al ₂ O ₃	21.06
SiO ₂	34.77
K ₂ O	0.30
CaO	0.54
TiO ₂	0.08
MnO	20.99
FeO	21.45
Total	<u>99.25</u>

Na	0.000
Mg	0.007
Al	2.059
Si	2.885
K	0.032
Ca	0.048
Ti	0.005
Mn	1.475
Fe ²⁺	1.296
Fe ³⁺	0.192
sum cations	<u>8.000</u>
sum anions	12.000

Element	Si	Fe	Mg	Mn	Al
S.R.	1.8	2.2	1.0		
#Pts.	20	20	20		
#Grains	17	17	19		
			1.2	1.9	1.2
			20	20	20
			19	19	19
					1.3
					20
					20

Evaluation: The spessartine analysis has a low sum (99.3%) and does not include a ferric iron determination. A stoichiometric garnet formula unit:



can be obtained if sufficient FeO is oxidized to form 3.08% by weight Fe₂O₃. However, this adjusted analysis and formula contains octahedrally-coordinated K and tetrahedrally coordinated Fe⁺³, which are unlikely in garnets. The homogeneity of Mn and Fe, the elements likely to be of interest for most microprobe operators, is only fairly good. GTSP does not have the potential to be an excellent standard; if it must be used at all, it should be reanalyzed.

Standard: Lemhi Biotite
 Locality: Lemhi County, Idaho
 Donor: D.E. Lee to David R. Wones (density 3.21); D.E. Lee to J.L. Munoz to
 S. Ludington to S. Huebner
 Reference: Lee, Donald E. (1958) A chlorine-rich biotite from Lemhi County,
 Idaho. Am. Mineral. 43, 107-111. Eileen H. Oslund, analyst.

Oxide wt.%

SiO ₂	33.09
Al ₂ O ₃	17.65
TiO ₂	1.30
Fe ₂ O ₃	2.42
FeO	29.22
MnO	0.04
MgO	2.83
CaO	0.10
Na ₂ O	0.13
Rb ₂ O	0.10
K ₂ O	9.04
BaO	0.09
H ₂ O ⁺	2.92
H ₂ O ⁻	0.04
F ⁻	0.23
Cl	1.11
Total	<u>100.31</u>
Less O=F+Cl	0.34
Total	<u>99.97</u>

Si	5.366	5.320
Al	3.373	3.345
Ti ⁴⁺	0.158	0.157
Fe ³⁺	0.295	0.293
Fe ²⁺	3.963	3.929
Mn ²⁺	0.006	0.005
Mg	0.684	0.678
Ca	0.017	0.017
Na	0.041	0.040
Rb	0.010	0.010
K	1.870	1.854
Ba	0.006	0.006
sum cations	<u>15.789</u>	<u>15.656</u>
sum anions	24.0	22.0

OH	3.202
F	0.118
Cl	0.305

Element	Fe	Al	Mg	Si	Cl	K	Ti
S.R.	3.4		1.0				
#Pts.	20		20				
#Grains	20		20				
	3.1		1.0	1.3			
	20		20	20			
	19		19	19			
	2.8		1.9		1.0		
	20		20		20		
	20		20		20	1.8	1.5
						20	20
	1.8		1.3			20	20
	20		20				
	20		20				
	2.5					1.3	
	20					20	
	20					20	
		1.6	1.2				
		20	20				
		20	20				

Mineral: Lemhi Biotite

Microprobe analysis: (1) U.S.G.S. ARL-SEMQ microprobe. Bence and Albee Method. J. Hammarstrom, analyst, 03-MAR-82. Two sets, average of 5 points each. (2) U.S.G.S. ARL-SEMQ microprobe. J. Stormer, analyst, 20-OCT-82; 15 kV; average of 15 points.

	(1a) wt.% $\pm 1\sigma$	(1b) wt.% $\pm 1\sigma$	(2) wt.% $\pm 1\sigma$
SiO ₂	34.28+0.54	33.73+0.12	33.82+1.28
Al ₂ O ₃	18.23+0.21	18.22+0.12	17.85+2.03
FeO	31.74+0.53	30.35+0.43	31.42+1.37
MgO	2.61+0.26	2.71+0.02	2.87+2.74
CaO	0.01+0.00	0.00+0.00	0.00+0.00
Na ₂ O	0.13+0.02	0.14+0.02	0.12+30.47
K ₂ O	8.95+0.17	8.93+0.05	9.59+8.85
MnO	0.04+0.02	0.04+0.01	0.05+25.00
TiO ₂	1.43+0.09	1.39+0.12	1.36+7.66
BaO	-	-	0.05+100.00
Cl	-	-	0.18+4.89
F	-	-	0.17+32.00
Total	97.407+0.678	95.499+0.708	98.48

Standards
MBST - Si,Al,Ti
MBLM - Fe
MFPH - Mg,K
AMEN - Ca,Na
OLST - Mn

Standards
AMKH - Si,Al,
Mg,Ca,Na,Ti
AMEN - Fe
MFPH - K
OLST - Mn

Standards
MFPH - Si,Mg,F,K
AMSF - Na,Ca
GTKN - Al,Fe
APCL - Cl
OXRU - Ti
PXBH - Mn
BAG - Ba

Evaluation: The sum of the wet chemical analysis is excellent, 99.97 weight percent. The chemistry is unusual in that the biotite contains 1.11 weight percent chlorine. The formula unit,



is reasonable. The mica is approximately 75% trioctahedral, 25% dioctahedral (ferri-muscovite), with 3% vacancies in the A site.

The contents of the two vials appear to be from the same separate but neither vial contains a split of the chemically analyzed material. (The Reston material was separated from the original specimen at a later date. In view of the narrow density range found by Lee (1958) and the good to excellent homogeneity found by measuring the sigma ratios with the microprobe, the Reston material is regarded as equivalent to that originally analyzed by Oslund.) MBLM is homogeneous for Mg, Si, K, Ti, and Cl. The Fe homogeneity is at the margin of the range of acceptability. Independent microprobe analyses give results that agree reasonably well with the wet chemistry. (Note that microprobe analysis 1a used MBLM as the standard for Fe.) Biotite standard MBLM has been used successfully as a standard for biotites and amphiboles in Reston. It should be considered for use as a known-unknown, provided that sufficient points are measured to overcome the heterogeneity in iron.

Standard: Biotite PSU 5-112
 Locality: Rainy Creek igneous complex, near Libby, Montana
 Donor: C.O. Ingamells
 Reference: Boettcher, A.L. (1967) The Rainy Creek alkaline-ultramafic igneous complex near Libby, Montana I: Ultramafic rocks and fenite. Jour. Geol. 75, 526-553. C.O. Ingamells, analyst.

Oxide wt.%

SiO ₂	39.10
Al ₂ O ₃	13.03
TiO ₂	1.21
Cr ₂ O ₃	0.25
Fe ₂ O ₃	2.56
FeO	7.23
NiO	0.02
MnO	0.10
MgO	21.55
CaO	0.12
SrO	<0.005
BaO	0.35
Na ₂ O	0.23
K ₂ O	10.05
Rb ₂ O	0.03
P ₂ O ₅	0.00
H ₂ O ⁺	3.74
H ₂ O ⁻	0.06
F ⁻	0.35
sub-Total	<u>99.98</u>
-O=F	<u>0.18</u>
Total	<u>99.80</u>

Si	5.700	5.681
Al	2.238	2.231
Ti	0.133	0.132
Cr	0.029	0.029
Fe ³⁺	0.281	0.280
Fe ²⁺	0.881	0.878
Ni	0.002	0.002
Mn	0.012	0.012
Mg	4.682	4.467
Ca	0.019	0.019
Ba	0.020	0.020
Na	0.065	0.065
K	1.869	1.863
Rb	0.003	0.003
P ₂ O ₅	0.00	0.000
sum cations	<u>15.934</u>	<u>15.882</u>
sum anions	24.000	22.0

OH	3.695
F	0.161

Element	Si	Fe	Mg	Al	Ti	K
S.R.	13.4	13.4	24.0			
#Pts.	20	20	20			
#Grains	5	5	5			
	26.0	23.8	46.8			
	20	20	20			
	6	6	6			
		12.7		4.6		
		20		20		
		5		5		
		25.0				26.2
		20				20
		5				5
						3.5
						20
						5
				8.1		
				20		
				6		
						16.0
						20
						5
circular glass slide #2						
	4.2	7.0	11.8			
	16	16	16			
	13	13	13			
			6.7	1.4	1.4	
			17	17	17	
			12	12	12	
			7.1		1.9	2.5
			16		16	16
			16		16	16

circular glass slide #2 - 2nd polish

	5.0	8.1	12.0			
	16	16	16			
	16	16	16			
		7.2	11.9	3.7		
		16	16	16		
		16	16	16		
			11.1		2.7	5.8
			16		16	16
			16		16	16

Mineral: Penn State University biotite
Microprobe analysis: U.S.G.S. ARL-SEMQ microprobe. Bence-Albee
method, 3/4/82. J. Hammarstrom, analyst. Average of 5 points

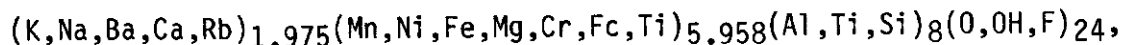
Oxide wt.% +1

SiO ₂	39.11+0.33
Al ₂ O ₃	13.27+0.15
FeO	9.56+1.18
MgO	19.94+1.02
CaO	0.00+0.00
Na ₂ O	0.23+0.08
K ₂ O	9.83+0.08
TiO ₂	1.25+0.19
MnO	0.06+0.04
Total	93.25+0.53

Standards:

AMKH - Si, Al, Mg, Ca, Na, Ti
AMEN - Fe
MFPH - K
OLST - Mn

Evaluation: The conventional analysis of the "biotite" from Montana has an excellent sum, 99.80% by weight. An independent microprobe analysis confirms the values for Na₂O, Al₂O₃, SiO₂, K₂O, TiO₂, and total iron as FeO, but not MgO. The conventional analysis can be recalculated to a formula unit,



that is near to the ideal formula unit for a trioctahedral mica. Ideal stoichiometry can be achieved by reducing some of the Fe₂O₃, in the analysis, to FeO. Calculated on an anhydrous basis, ideal stoichiometry cannot be achieved by reducing all the Fe₂O₃; Cr₂O₃ and TiO₂ must be reduced in addition. Rather than fault the analysis, however, it is probable that MBPS does not have ideal trioctahedral mica stoichiometry; the partially vacant A site supports this suggestion.

The apparent homogeneity of MBPS is very bad; the large values are due to anomalously low count rates for two or three spectrometers, simultaneously. The material is in the form of thin flakes; the low count rates are probably caused by the electron beam overlap onto the epoxy mounting medium. If this overlap can be avoided, MBPS should be an excellent standard for Mg-rich biotites and amphiboles. Conceivably, it could be used as a known-unknown.

Standard: Biotite
 Locality: Stillwater complex
 Donor: Dale Jackson to Gerry Czamanske to Huebner
 Reference: Chemical analysis by Ingamells (1972) for Jackson (U.S.G.S. Lab. No. 64M-1610). V, Ni, and Ba by semi-quantitative spectrographic analysis.

Oxide wt.%

SiO ₂	39.26
Al ₂ O ₃	14.76
Fe ₂ O ₃	0.26
FeO	4.16
MgO	22.02
CaO	0.13
Na ₂ O	0.67
K ₂ O	8.83
TiO ₂	3.86
MnO	0.01
Cr ₂ O ₃	2.15
V ₂ O ₃	0.06
NiO	0.13
BaO	0.13
Li ₂ O	0.02
H ₂ O ⁺	3.42
H ₂ O ⁻	0.06
F ⁻	0.12
Total	<u>100.05</u>
O=F	-0.05
Total	<u>100.00</u>

Si	5.606	5.526
Al	2.484	2.449
Fe ²⁺	0.497	0.490
Fe ⁺³	0.028	0.028
Mg	4.686	4.620
Ca	0.020	0.020
Na	0.186	0.183
K	1.608	1.586
Ti ⁴⁺	0.414	0.409
Mn ²⁺	0.001	0.001
Cr ⁺³	0.243	0.239
V ⁺³	0.006	0.006
Ni	0.015	0.015
Ba	0.007	0.007
Li	0.012	0.011
sum cations	<u>15.812</u>	<u>15.589</u>
sum anions	24.000	22.000

OH 3.315

Element	Ti	K	Al	Cr	Si	Fe	Mg
S.R.	3.4	4.3					
#Pts.	18	18					
#Grains	5	5	1.9	1.0			
			20	20			
			3	3	3.6	5.0	12.3
	2.4	4.8			20	20	20
	19	19	5.7		5	5	5
	5	5	19				
			5		2.9	7.9	9.7
					19	19	19
					4	4	4

Mineral: Stillwater biotite

Microprobe analysis: U.S.G.S. ARL-SEM-Q microprobe, Sept. 3, 1982. Bence and Albee method. J. Hammarstrom, analyst. (1),(2),(4), 5 points each set. (3),(7), 3 points each set. (5),(6), 1 point each.

	(1) wt.% 1σ	(2) wt.% 1σ	(3) wt.% 1σ	(4) wt.% 1σ
SiO ₂	39.08 +0.42	39.29 +0.49	37.22+0.47	37.33+0.94
Al ₂ O ₃	14.89 ±0.44	14.66 ±0.35	15.11±0.34	14.90±0.46
FeO	4.63 ±0.29	4.38 ±0.24	4.39±0.11	4.01±0.09
MgO	19.47 ±0.54	19.65 ±0.56	20.16±0.75	20.62±0.87
CaO	0.00 ±0.00	0.00 ±0.00	0.07±0.03	0.00
Na ₂ O	0.25 ±0.07	0.24 ±0.03	0.42±0.18	0.31±0.17
K ₂ O	9.10 ±0.32	9.37 ±0.06	8.72±0.38	9.14±0.45
TiO ₂	4.11 ±0.32	3.70 ±0.26	3.74±0.51	3.80±0.29
MnO	0.00 ±0.00	0.00 ±0.00	0.00	0.00
Cr ₂ O ₃	-	-	2.07±0.14	2.08±0.11
F	-	-	0.15±0.06	0.15±0.02
Cl	-	-	0.27±0.12	0.17±0.04
Total	91.52 ±1.650	91.29 ±0.477	92.32	92.51

	(5)	(6)	(7)
SiO ₂	38.33	38.94	38.24
Al ₂ O ₃	15.13	14.20	14.79
FeO	4.43	4.44	4.61
MgO	20.44	21.45	19.60
CaO	0.02	0.00	0.00
Na ₂ O	0.65	0.13	0.42
K ₂ O	8.62	8.05	8.92
TiO ₂	4.05	3.33	3.91
MnO	0.04	0.04	0.05
Cr ₂ O ₃	1.99	2.23	2.10
V ₂ O ₃	0.12	0.12	0.09
F	0.31	0.16	0.15
Cl	0.18	0.14	0.20
Total	94.31	93.23	93.10

Standards used (1):

MBST - Si,Al,Ti
 MBLM - Fe
 MSFP - Mg
 AMEN - Ca,Na
 MFPH - K
 OLST - Mn

Standards used (2):

AMKH - Si,Al,Mg,Ca,Na,Ti
 AMEN - Fe
 MFPH - K
 OLST - Mn

Standards used (3):

AMEN - Si,Al,Mg,Ca
 GTKN - Fe
 FSTA - Na
 MFPH - K,F
 RUTILE - Ti
 OLST - Mn
 MBLM - Cl
 MBST - Cr

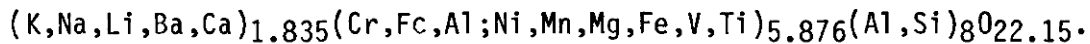
Standards used (4):

PXAD - Si,Mg,Ca
 FSTA - Al,Na
 OXIL - Fe,Mn,Ti
 MFPH - K,F
 APCL - Cl
 OXTB - Cr

Standards used:

(5),(6),(7)
 MBLM- Cl
 MFPH - K,F
 SPHC - Ti
 OXVA - V
 OXTB - Cr
 FSTA - Al,Na
 GTKN - Fe,Si
 PXAD - Ca,Mg
 PXBH - Mn

Evaluation: The chemical analysis of the Stillwater biotite has an excellent sum. The analysis cannot be recalculated to an ideal or stoichiometric biotite. Based on formal site occupancies, this mica is a mixture of trioctahedral and dioctahedral components with a large cation deficiency in the "X" site and a slight deficiency of water, but it is not possible to calculate a unique formula. One scheme is to assign trivalent ions (Cr,Fe,Al) to a dioctahedral component, resulting in an octahedral site with 6.2% dioctahedral component and 0.12 vacancies per 14 cation positions. The formula based on a tetrahedral + octahedral cation sum of 13.88 cations becomes:



This representation is consistent with the low values of (H₂O+F) in the analysis, but the significant deficiency in the alkali (X) site is not explained. There are too many unknowns to support the quality of the chemical analysis by recalculation to a formula unit. Microprobe chemical analyses using a variety of standards give MgO values of 19.5% to 20.6%, significantly less than the 22.0% of the conventional chemical analysis. MBST is homogeneous for Cr alone among the major elements. Although MBST has been widely used both as a Cr standard for silicates and occasionally as a known-unknown for biotites, it is too heterogeneous for this purpose. Its only possible usage is as a chromium standard in silicates, and then only if the effect of the heterogeneous matrix will not be important to the result.

Date	From	To	Address
3/11/77	Huebner	Eric J. Essene	Dept. Geology and Mineralogy University of Michigan Ann Arbor, Michigan 48104

Standard: Fluorphlogopite
 Locality: synthetic
 Donor: E.D. Ghent
 References:

Oxide wt.% (theoretical)

SiO ₂	42.78
Al ₂ O ₃	12.09
F	9.02
MgO	28.71
K ₂ O	11.18
	<u>103.79</u>
O=F	-3.80
Total	<u>99.99</u>

Element	Si	Mg	Al	K	F
S.R.	1.3	3.4	1.2	1.4	
#Pts.	20	20	20	20	
#Grains	7	7	4	6	
	1.6	2.8			
	19	19			
	19	19		1.0	1.8
				17	20
				5	5

Mineral analysis: U.S.G.S. ARL-SEMQ microprobe, J. Stormer, analyst, 15kV, 20-OCT-82; average of 8 analyses.

	counts +1σ
Si	16771±194
F	2391±65
Mg	64495±797

Standard
 MFPH

Evaluation: The fluorphlogopite has not been analyzed directly. However, deviations from stoichiometry are unlikely for this bulk composition and J.C. Stormer has used the fluorphlogopite as a microprobe standard successfully to reproduce the Mg, Si, and F values of AMSF, MBLM, and AMKH. A microprobe homogeneity check of MFPH revealed good sigma ratio values of Si, 1.3; Al, 1.2; and K, 1.4; the Mg value is 3.4. The chief drawback to MFPH is the difficulty of obtaining a suitably flat and polished surface; however, used with care, MFPH is a good standard for Si, Al, K, and F in micas and amphiboles.

19-NOV-76 Huebner Arden Albee

Division of Geological and
 Planetary Sciences
 California Institute of Technology
 Pasadena, California 91125

Standard: Methuen Muscovite
 Locality: Methuen Township, Ontario
 Donor: Richard A. Robie

Reference: 1) Robie, et al. (1976) Jour. Res. U.S.G.S. 4, 631-644.
 (1a) Hurlbut (1956) F.A. Gonyer, anal. (1b) Eugster et al. (1972) J.J. Fahey, analyst. U.S.G.S. (65-WG-27) (1c) Rapid rock analysis by Hezekiah Smith.
 2) Chem. anal., U.S.G.S. Anal. Lab., Menlo Park, Calif. Sarah T. Neil, anal.

	oxide wt.% (1a)	oxide wt.% (1b)	oxide wt.% (1c)	(1c)	oxide wt.% (2)
SiO ₂	45.87	45.20	44.00		44.94
Al ₂ O ₃	38.69	38.46	35.00		35.71
Fe ₂ O ₃	-	0.25	2.20		2.05
FeO	-	-	0.26		0.34
MgO	0.10	0.00	0.57		0.57
CaO	-	-	0.22		0.08
Na ₂ O	0.64	0.59	0.72		0.76
K ₂ O	10.08	10.50	9.60		10.87
H ₂ O ⁺	4.67	4.64	5.60		4.30
H ₂ O ⁻	-	0.08	2.00		0.10
TiO ₂	-	-	0.13		0.16
P ₂ O ₅	-	0.02	0.01		<0.05
MnO	-	-	0.07		0.06
CO ₂	-	-	0.01		0.19
F	-	-	0.29		0.30
			<u>100.68</u>		<u>100.43</u>
-O=F			-0.12		-0.13
Total	100.05	99.74	<u>100.56</u>		<u>100.30</u>

	(1a)	(1a)	(1b) adjusted	(1c)	(1c)	(2)	(2)
Si	6.019	6.031	5.969	5.659	6.020	5.987	5.991
Al	5.983	5.995	5.986	5.306	5.644	5.606	5.611
Fe ³⁺	-	-	0.025	0.213	0.227	0.205	0.206
Fe ²⁺	-	-	-	0.028	0.030	0.038	0.038
Mg	0.020	0.020	-	0.109	0.116	0.113	0.113
Ca	-	-	-	0.030	0.032	0.011	0.011
Na	0.163	0.163	0.151	0.180	0.191	0.196	0.196
K	1.687	1.690	1.769	1.575	1.676	1.847	1.848
Ti ⁴⁺	-	-	-	0.013	0.013	0.016	0.016
P	-	-	0.002	0.001	0.001	-	0.000
Mn ²⁺	-	-	-	0.008	0.008	0.007	0.007
C	-	-	-	0.002	0.002	0.035	0.035
sum cat.	<u>13.871</u>	<u>13.899</u>	<u>13.903</u>	<u>13.123</u>	<u>13.961</u>	<u>14.061</u>	<u>14.073</u>
sum anions	24.000	22.0	24.0	24.0	22.0	24.0	22.0
OH	4.088		4.158	6.521	-	3.909	
F	-		-	0.118	-	0.126	

	Ross's slide (1 grain)			Ross's slide (4 grains)		
Element	Si	Fe	Mg	Si		
S.R.	1.4	1.0	1.3	1.5		
#Points	20	20	20	20		
#Grains	1	1	1	4		
	Al			K		
	1.5	1.1	1.4	1.2		
	20	20	20	20		
	1	1	1	4		
	K			Al	Fe	Mg
	1.4	1.1	1.7	3.2	11.9	10.1
	20	20	20	20	20	20
	1	1	1	4	4	4

	Mica Block			Circular Glass Slide		
Element	Si	Fe	Mg	Si	Fe	Mg
S.R.	4.4	3.0	1.6	2.0	1.8	2.1
#Points	20	20	20	20	20	20
#Grains	4	4	4	12	12	12
	Al			K		
	3.4		1.3	2.1	2.6	
	20		20	20	20	
	10		10	9	9	
	1.9			Al		
	20			2.2	2.2	
	3			20	20	
				8	8	
	K					
	1.4					
	20					
	6					

Mineral: Methuen Muscovite

Microprobe Analysis: 1) G.K. Czamanske, U.S.G.S., Menlo Park, Calif.
 30-OCT-77. Average of 6 points. 2) U.S.G.S. ARL-EMX microprobe.
 Toby Wiggins, Analyst.

	Oxide wt.% $\pm 1\sigma$ (1)	Oxide wt.% (2)
SiO ₂	45.41 \pm 0.38	46.29
Al ₂ O ₃	37.98 \pm 0.64	35.82
FeO	2.16 \pm 0.08	2.27
MgO	0.14 \pm 0.02	0.65
CaO	0.01 \pm 0.00	-
Na ₂ O	0.63 \pm 0.05	0.45
K ₂ O	10.66 \pm 0.13	10.25
TiO ₂	0.17 \pm 0.02	-
MnO	0.08 \pm 0.01	-
Total	97.24 \pm 0.40	95.73

Evaluation: The Methuen muscovite in the Reston microprobe laboratory is from the sample analyzed by Hurlbut (1a), S.T. Neil (2), and Czamanske (2, page 2). Recalculation to a formula unit based on 14 cations indicates a significant deficiency in alkali. This sample of Methuen muscovite may have a hydromuscovite or illite interlayer component but, in view of the range of reported values for Al₂O₃ (35.0 - 37.9%), the substituting component cannot be identified. The Reston sample, MMT, should be examined by high resolution electron microscopy and be reanalyzed for Al₂O₃ in the presence of Fe₂O₃.

Date	From	To	Address
06-JUN-77	Huebner	Eric J. Essene	Dept. Geology and Mineralogy University of Michigan Ann Arbor, MI 48104
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
18-JUL-83	Huebner	Robert W. Smith	St. Joe Minerals Company P.O. Box 500 Viburnum, MD 65566
09-OCT-84	Huebner	Michael Shaffer	Dept. Geology University of Oregon Eugene, OR 97403-1272

Standard: Paragonite
 Locality: Ilas de Margarita, Venezuela
 Donor: R.A. Robie

Reference: (1) Robie, R.A., and Hemingway, B.S. (1984) Am. Mineral. 69,
 p. 859. J. Marinenko, analyst. Rapid-rock method. U.S. Geol. Surv. #W-197310
 (2) L.B. Wiggins, ARL/EMX microprobe, U.S. Geol. Surv., Reston (3),
 (4) Maresch, W.V., Ph.D. thesis, Princeton University, 1972, Microprobe
 analyses Nos. W176-1 and W176-2, respectively.

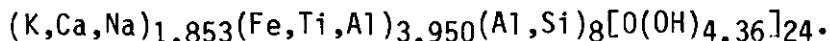
	Oxide wt.%			
	(1)	(2)	(3)	(4)
SiO ₂	46.7	48.02	46.36	46.90
Al ₂ O ₃	40.5	40.13	38.71	38.37
FeO	0.32	0.80	0.77	0.83
TiO ₂	0.46	-	0.05	0.04
MnO	-	-	-	-
MgO	-	0.18	0.10	0.19
CaO	0.40	-	0.22	0.27
BaO	-	-	-	-
Na ₂ O	6.9	6.96	7.79	7.34
K ₂ O	0.73	0.64	0.54	0.51
H ₂ O ⁺	5.2	-	-	-
Total	<u>101.21</u>	<u>96.73</u>	<u>94.54</u>	<u>94.45</u>

	(1)	(2)	(3)	(4)
Si	5.872	5.920	6.034	6.055
Al	6.001	6.051	5.943	5.838
Fe	0.034	0.034	0.084	0.090
Ti	0.044	0.044	-	0.005
Mn	-	-	-	-
Mg	-	-	0.034	0.019
Ca	0.054	0.054	-	0.030
Ba	-	-	-	-
Na	1.682	1.696	1.696	1.953
K ^c	0.117	0.118	0.103	0.089
cations	<u>13.803</u>	<u>13.917</u>	<u>13.893</u>	<u>14.073</u>
anions	24.0	22.0	22.000	22.0

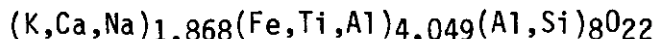
OH 4.361

Element	Si	Al	Na
S.R.	1.8		
#Pts.	19	1.9	3.1
#Grains	6	20	20
		6	6
	1.4		
	20	1.4	1.9
	20	19	19
		19	19
		1.2	1.6
		19	19
		7	7

Evaluation: As is the case with most micas, it is difficult to evaluate the chemical analyses of MPAV. The only complete analysis, No. 1, has an unexpectedly high value for H₂O and a correspondingly high summation. Microprobe analyses Nos. 3 and 4 have large values of "H₂O by difference", suggesting that the sums of the analyzed components are low. The range of analyzed values is disturbingly large: 1.7% in SiO₂, 2.1% in Al₂O₃, and 0.9% in Na₂O. Recalculation to formula units does not resolve the problem. Analysis No. 1 recalculates to



Perfect dioctahedral stoichiometry cannot be achieved by adjustment of the proportions of redox components. On an anhydrous basis,



The cation:anion ratio of the anhydrous formula, 13.917:22, is close to the ideal value for a dioctahedral mica. Again, the ideal stoichiometry of 14 cations per 22 anions cannot be achieved by redox adjustment. The mica also has nearly end member paragonite composition (Na/(Na+K)=0.94). The A-site deficiency indicates the presence of a non-micaceous component in solid solution or intergrowth relationship. The homogeneity of this mica is adequate. Because of its nearly end member composition and stoichiometry, one might be tempted to use MPAV as a standard for Si, Al, and Na in micas and amphiboles. However, the available chemistry does not warrant widespread usage. Use of MPAV should be restricted to analyzing Na in sodium-rich micas.

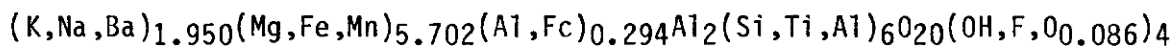
Date	From	To	Address
18-JUL-83	Huebner	Robert W. Smith	St. Joe Minerals P.O. Box 500 Virburnum, MD 65566

Standard: Phlogopite
 Locality: Burgess, Ontario
 Donor: USNM #124158 to R.A. Robie to Huebner
 References: U.S. Nat. Museum 124158, Analyst J. Marinenko, U.S. Geological
 Survey no. W-197307

	oxide wt.%	
SiO ₂	40.3	
Al ₂ O ₃	14.3	
Fe ₂ O ₃	0.63	
FeO	1.11	
TiO ₂	1.32	
MnO	0.03	
MgO	26.4	
CaO	<0.07	
BaO	0.16	
Na ₂ O	0.43	
K ₂ O	10.1	
H ₂ O ⁺	2.63	
H ₂ O ⁻	-	
F ²⁻	3.2	
subtotal	<u>100.61</u>	
O F	1.34	
Total	<u>99.27</u>	
Si	5.702	5.691
Al	2.384	2.380
Fe ³⁺	0.067	0.067
Fe ²⁺	0.131	0.131
Ti ⁴⁺	0.140	0.140
Mn ²⁺	0.004	0.004
Mg	5.567	5.556
Ca	-	-
Ba	0.009	0.009
Na	0.118	0.118
K	1.823	1.819
cations	<u>15.945</u>	<u>15.914</u>
anions	24.000	22.0
OH	2.482	
F	1.432	

Element	Al	Na		Si	Mg
S.R.	1.2	1.1		1.1	2.1
#Pts.	20	20		19	19
#Grains	5	5		4	4
			K		
		1.0	1.8		
		18	18		
		7	7		

Evaluation: The analysis of the Ontario phlogopite has a low sum, 99.3 wt.%.
 A reasonable formula unit can be calculated for phlogopite MPBO:



This formula is approximately 95% trioctahedral component and 5% dioctahedral (ferri-muscovite) component. The formula has a small deficiency in "A" site cations and a small "oxy" component. MPBO is homogeneous and takes a good polish. Used with care, to avoid the few points which give anomalously low X-ray count rates, phlogopite MPBO should be an excellent standard.

Date	From	To	Address
18-JUL-83	Huebner	Robert W. Smith	St. Joe Minerals Company P.O. Box 500 Virburnum, MD 65566

Standard: Fluor-phlogopite
 Locality: Synthetic
 Donor: M. Ross; U.S. Bur. Mines, Norris, Tenn., presumed original source.
 Reference: assumed $\text{KMg}_3\text{AlSi}_3\text{O}_{10}\text{F}_2$

Oxide Wt. %, (theoretical)

K ₂ O	11.18
MgO	28.71
Al ₂ O ₃	12.09
SiO ₂	42.78
F	9.02
Total	<u>103.79</u>
-O=F	3.80
Total	<u>99.99</u>

Elem.	Si	Al	Mg	K	F
S.R.		1.2	3.5		
#Pts.		20	20		
#Grains		2	2	2.3	2.6
				20	20
				2	2
	2.1		5.4		
	19		19		
	6		6	2.2	1.4
		2.2		20	20
		19		6	6
		6			

Evaluation: Neither the chemistry nor the source of the fluor-phlogopite is known with certainty. The large crystals can be polished adequately, but care still must be taken to locate the microprobe beam on a uniform portion of the surface. The MSFP in the collection and on which the homogeneity was determined is probably NOT the same fluorphlogopite that is mounted in various Reston standard blocks. MSFP takes a better polish, is easier to use, and on that basis is the preferred fluorphlogopite for use in Reston.

Standard: Cobalt Olivine
Locality: Synthetic, conditions unknown
Donor: USNM #718
References:

Oxide wt.% (theoretical)

CoO	71.38
SiO ₂	28.62
Total	100.00

Element	Si	Co
S.R.	1.0	1.6
#Pts.	19	19
#Grains	10	10
	1.5	
	20	
	10	

Evaluation: This synthetic cobalt olivine coexists with two more highly reflecting oxides and a darker gray interstitial glass. The olivine is recognized by its deep red (burgundy) internal reflections combined with a tendency to form euhedra. Because of the presence of oxides, the olivine is assumed to be stoichiometric. The olivine grains themselves are homogeneous. Used with care (to avoid other phases), OLC0 can serve as a qualitative analysis standard for Co in silicates.

Standard: Cobalt Olivine

Locality: Synthetic, floating zone method (Takei and Hosoya, 1977, in High-Pressure Research in Geophysics; S. Akimoto, and M.H. Manghnani, eds.)

Donor: R.A. Robie

References: 1) Sumino, Y., 1979, J. Phys. Earth, 27, 209-238. E.P.M.A. analysis by Suzuki. (2) Robie et al., 1982, Amer. Mineral. 67, pp. 470-482. Microprobe analyses, L.B. Wiggins, U.S. Geological Survey, Reston, VA.

	(1)	Oxide wt. %	(2)
SiO ₂	28.3	27.3	27.4
CoO	70.8	71.8	72.1
M ₂ O ₃	-	-	-
Total	<u>99.1</u>	<u>99.1</u>	<u>99.5</u>

Si	0.998	0.974	0.973
Co	2.003	2.053	2.054
sum cations	<u>3.002</u>	<u>3.027</u>	<u>3.027</u>
sum anions	4.0	4.0	4.0

Element	Co
S.R.	2.1
#Pts.	20
#Grains	2

Si
 1.2
 20
 2

Evaluation: Cobalt olivine OLCR is a fragment of a large, synthetic crystal. Microprobe analyses have low totals, yet the formula units, based on four oxygens, have close to ideal stoichiometry. This material is homogeneous and should serve as an excellent standard for cobalt in silicates.

Standard: Olivine
 Locality: Marjalahti, Finland
 Donor: William Melson
 References: (1) Yoder H.S., Jr. and Sahama, T.G. (1957) Olivine x-ray
 determinative curve: Amer. Mineral. 42, 475-491. (2) Melson

	Oxide wt%	
	(1)	(2) Preferred
SiO ₂	40.24	40.22
TiO ₂	0.00	-
Al ₂ O ₃	0.01	-
MgO	48.08	48.08
CaO	0.00	-
FeO	10.92	11.19
Fe ₂ O ₃	0.68	-
Cr ₂ O ₃	0.07	-
Na ₂ O	0.00	-
K ₂ O	0.00	-
MnO	0.28	0.26
H ₂ O ⁺	0.00	-
H ₂ O ⁻	0.04	-
Total	<u>100.32</u>	<u>99.75</u>

Si	0.990	0.993
Al	0.0003	-
Mg	1.764	1.770
Fe ⁺²	0.225	0.218
Fe ⁺³	0.013	0.013
Cr	0.0014	-
Na	0.000	-
K	0.000	-
Mn ⁺²	0.006	0.005
sum cations	3.000 2.999	3.000
sum anions	4.000	4.000

Element	Si	Fe	Mg
S.R.	0.8	1.7	0.9
#Pts.	20	20	20
#Grains	7	7	7
	1.0	1.5	2.1
	20	20	20
	5	5	5

Evaluation: OLMJ from Marjalahti, Finland is supported by a wet chemical analysis with a slightly high total, 100.3% by weight. The corresponding formula unit has a slight deficiency of tetrahedral Si but an almost perfect cation/anion ratio, 2.999/4.000 without redox adjustment. An x-ray determination of the % Fo yields 88.5 mole %, slightly higher than the 87.8% calculated from the analysis. Melson's analysis by unknown methods has a slightly higher FeO value that may be preferable. The presence of Fe⁺³ in the absence of octahedral vacancies is puzzling. OLMJ is homogeneous and, despite the minor uncertainties mentioned above, is an excellent standard.

Standard: Nickel Olivine

Locality: Synthetic - Ni₂SiO₄

Donor: U.S. National Museum #717 via Hammarstrom and Wiggins

References: Synthesized by J. Ito (Robie, R., Hemingway, B., Ito, J., and Krupka, K., Heat capacity and entropy of NiSiO₄-olivine from 5 to 1000K and heat capacity of CoSiO₄ from 360 to 1000K, submitted to American Mineralogist (1984)

Oxide wt.% (presumably theoretical)

SiO ₂	28.68
NiO	71.32
Total	<u>100.00</u>

Element	Si	Ni
S.R.	1.3	
#Pts.	20	
#Grains	7	
		2.0
		20
		12

Evaluation: The chemistry of the nickel olivine is inferred from phase equilibrium stoichiometric considerations. The material contains inclusions of NiO; the most Ni-rich silicate that coexists with NiO is the olivine. Si and Ni are homogeneously distributed in this olivine. OLNI is a useful standard for Ni in olivines and oxides.

Date	From	To	Addresses
13-NOV-80	Huebner	Dan Schulze	Programs in Geosciences Station FO.2.1 The University of Texas at Dallas Box 688 Richardson, TX 75080

Standard: Rockport Fayalite
 Locality: Rockport, Massachusetts
 Donor: E. Jarosewich - USNM#85276
 Reference: Jarosewich, E., Nelen, J.A., and Norberg, J.A., 1977, Smiths. Contr. Earth Sci., No. 22, 53.

Oxide wt.%

SiO ₂	29.22
Fe ₂ O ₃	1.32
FeO	66.36
TiO ₂	0.04
MnO	2.14
H ₂ O	0.1
Total	<u>99.18</u>

Si	0.994	0.996
Fe ³⁺	0.034	0.034
Fe ²⁺	1.887	1.892
Ti	0.001	0.001
Mn	0.062	0.062
sum cations	<u>2.977</u>	<u>2.986</u>
sum anions	4.0	4.0

OH 0.023

Elem.	Si	Fe	Mn
S.R.	1.8	2.6	
Pts.	20	20	
#Grains	8	8	

2.2	1.6
2.0	20
8	8

0.8	1.6
20	20
6	6

Evaluation: The weight percent total of the wet-chemical analysis of OLRF is slightly low, contains appreciable ferric iron, and does not include MgO. The formula unit [(Mn,Fe,Fe)_{1.985}(Fe,Ti,Si)_{1.000}]_{4.000} suggests the presence of octahedral vacancies (ferri-fayalite component). OLRF appears to be homogeneous, but despite the good sigma-ratio value for Mn, E. Jarosewich (oral communication, November 8, 1985) cautions that about 2% of the grains have anomalously low MnO values. The iron content should be confirmed by reference to a synthetic, fayalite such as OLSF. If the wet-chemical iron analysis can be confirmed, OLRF should be considered for wider usage because its coarse grain size makes it easier to use than OLSF.

Standard: San Carlos Olivine
Locality: Gila Co., AZ
Donor: E. Jarosewich - USNM 111312/444
Reference: Jarosewich, E., et al. (1979) Electron microprobe reference samples for mineral analyses. Smithsonian Contributions to the Earth Sciences 22, 68-72.

	Oxide wt.%
SiO ₂	40.81
NiO	0.37
FeO	9.55
MgO	49.42
CaO	<0.05
MnO	0.14
P ₂ O ₅	0.00
Total	<u>100.29</u>

Si	0.996
Ni	0.007
Fe ⁺²	0.188
Fe ³⁺	0.007
Mg	1.798
Mn	0.003
sum cations	<u>3.000</u>
sum anions	4.000

Element	Si	Fe	Mg
S.R.	1.0	1.1	1.0
#Pts.	20	20	20
#Grains	4	4	4

Mineral: San Carlos Olivine
Microprobe analysis: U.S.G.S. ARL-EMX microprobe, 1979;
L.B. Wiggins, analyst; (1) analysis of one grain (2) average of six grains

	oxide wt.% $\pm 1\sigma$	oxide wt.% $\pm 1\sigma$
SiO ₂	40.16 \pm 1.00	40.33 \pm 0.45
FeO	11.58 \pm 0.36	11.54 \pm 0.15
MgO	48.03 \pm 0.88	48.21 \pm 0.45
Total	99.77	100.08

Standards: OLMJ - Mg,Si,Fe

Evaluation: The oxide weight percent sum from the classical chemical analysis is somewhat high, 100.3% by weight. Two microprobe analyses by Wiggins have improved totals but higher FeO/MgO ratios than in the case of the classical analysis. The material is homogeneous; the sigma ratios are very close to the ideal value of 1. Jarosewich (personal communication, October 21, 1985) reports inconsistencies in the Fe content when OLSC is analyzed against other analyzed olivine. This material has generally been considered an excellent standard for olivine for general use, but in view of the discrepancy in FeO/MgO, the classical analysis ought to be verified again by microprobe analysis.

Standard: Fayalite
Locality: Synthetic
Donor: J.S. Huebner

Reference: Fe_2SiO_4 by x-ray. Synthesized from hematite, quartz, and metallic iron at 790°C , 2000 bars total pressure, in $\text{Ag}_{70}\text{Pd}_{30}$ capsule, with Zn in bomb. Brown product. Trace excess silica.

Oxide wt.% (theoretical)

SiO_2	29.49
FeO	<u>70.51</u>
Total	100.00

Element	Si	Fe
S.R.	1.3	2.7
#Pts.	18	18
#Grains	12	12

Evaluation: The composition of synthetic fayalite is inferred from stoichiometric considerations and conditions of synthesis. The brown color suggests that a trace of ferric iron may be present, but the quantity is regarded as being insignificant for microprobe analysis. This material is fine-grained and reportedly contains a small amount of excess silica which fluoresces under the microprobe beam. The poor sigma ratio for iron, 2.7, may be due to the fine grain size of this synthetic product. OLSF has been used as an Fe standard for silicates and as a Si standard in iron-rich metal. Because of the fine grain size, it must be used with care to make sure that the microprobe beam activates only the fayalite.

Date	From	To	Address
19-NOV-76	Huebner	Arden Albee	Division of Geological and Planetary Sciences California Institute of Technology Pasadena, California 91125
08-MAY-72	Huebner	Bill Bonnicksen	Department of Geological Sciences Kimball Hall Cornell University Ithaca, N.Y. 14858
11-DEC-74	Huebner	Edward Ghent	Faculty of Arts and Sciences Department of Geology The University of Calgary Calgary, Alberta, Canada T2N 1N4
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
11-DEC-74	Huebner	Ian Ridley	Lamont-Doherty Observatory Columbia University Palisades, NY 10964
13-APR-75	Huebner	Peter Robinson	Dept. Geology and Geography Morrill Science Center University of Massachusetts Amherst, MA 01002

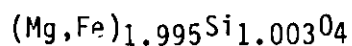
Standard: Olivine
 Locality: Susimaki Meteorite
 Donor: William Melson to B.A. Morgan
 Reference:

	Oxide Wt. %
MgO	25.53
SiO ₂	35.30
FeO	<u>38.47</u>
Total	99.30

Mg	1.081
Si	1.003
Fe	<u>0.914</u>
sum cations	2.997
sum anions	4.000

Element	Si	Fe	Mg
S.R.	1.4	5.0	6.4
#Pts	20	20	20
#Grains	20	20	20
	1.3	2.5	4.0
	20	20	20
	20	20	20

Evaluation: The chemical analysis supplied by Melson has an unacceptably low summation, 99.3% by weight. The formula unit, calculated from the analysis, is close to that of stoichiometric olivine:



This olivine is heterogeneous, but only the most divergent grains show an obvious correlation of high Fe, low Mg counts. Because of the poor summation and the heterogeneity, Susimaki olivine cannot be considered suitable for use as a microprobe standard.

Standard: Tephroite

Locality: Synthetic

Donor: Huebner

Reference: Mn_2SiO_4 by x-ray. Run #Hy-36-synthesized from a reduced mix of synthetic pyrolusite and Lake Toxaway quartz; 641°C, 2000 bars total pressure, 11 days, in unbuffered aqueous fluid phase. Product verified by optical and x-ray powder diffraction methods.

Oxide wt.% (theoretical)

SiO ₂	29.75
MnO	70.25
Total	<u>100.00</u>

Element	Si
S.R.	1.7
#Pts.	20
#Grains	10

Mn
3.1
18
18

3.1
18
18

Evaluation: The chemistry of synthetic tephroite is inferred from stoichiometric constraints and synthesis conditions. The size of individual grains and grain intergrowths is more than adequate for microprobe analysis. Although a separate microprobe test of sigma ratios showed a high value of 3.1 for Mn, low standard deviations and sigma ratios are obtained during standardization; therefore, the material is probably homogeneous for Mn. The material has been used successfully as a Mn standard for phases on the (Mn,Mg)Si₂O₆ join. The deep red fluorescence is a convenient indication of the location of the beam spot. OLST is a good general standard for manganese.

Date	From	To	Address
20-FEB-68	Huebner	Arden L. Albee	Department of the Geological Sciences California Institute of Technology Pasadena, California
01-DEC-69	Huebner	A.E. Bence	Dept. of Earth and Space Sciences State Univ. New York at Stony Brook Stony Brook, N.Y. 11790
30-NOV-73	Huebner	Eric Essene	Dept. of Geology and Mineralogy University of Michigan Ann Arbor, Michigan 48104
01-DEC-69	Huebner	Louis A. Fernandez	Dept. Geology and Geophysics Box 2161, Yale Station New Haven, Connecticut 06520
30-APR-70	Huebner	Bevan French	Planetology Branch-NASA Goddard Spaceflight Center Greenbelt, Maryland 20771
30-APR-70	Huebner	Edward Ghent	Faculty of Arts and Sciences Department of Geology The University of Calgary Calgary 44, Alberta, Canada
10-FEB-70	Huebner	Charles V. Guidotti	The University of Wisconsin Department of Geology and Geophysics Madison, Wisconsin 53706
13-NOV-80	Huebner	Lester Hughes	CONICO, Inc. 244 Research Bldg. P.O. 1267 Ponca City, OK 74601
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Westbrook Place Vancouver, B.C., Canada V6T 1W5
11-MAR-77	Huebner	Ian Ridley	Lamont-Doherty Observatory Columbia University Palisades, NY 10964
24-APR-75	Huebner	Peter Robinson	Dept. Geology and Geography Morrill Science Center University of Massachusetts Amherst, MA 01002
27-FEB-81	McGee	Dan Schulze	Department of Geoscience University of Texas at Dallas Richardson, TX 75080
08-SEP-78	Huebner	V.J. Wall	Department of Earth Sciences Monash University Clayton, Victoria Australia 3168

Standard: Olivine
 Locality: Springwater meteorite
 Donor: E. Jarosewich - USNM 2566
 Reference: Jarosewich, E., et al. (1979) Electron Microprobe Reference Samples
 for Mineral Analyses. Smithsonian Contributions to the Earth Sciences.
 J. Norberg, analyst

Oxide wt.%

SiO ₂	38.95
Cr ₂ O ₃	0.02
FeO	16.62
MgO	43.58
MnO	0.30
H ₂ O	<0.05
Total	99.47

Si	0.992	0.990
Cr ⁺³	0.000	0.000
Fe ⁺²	0.354	0.333
Mg	1.655	1.650
Mn	0.006	0.006
Fe ³⁺	-	0.020
sum cations	<u>3.008</u>	<u>3.000</u> adjusted
sum anions	4.000	4.000

Element	Si	Fe	Mg
S.R.	1.1	1.7	1.6
#Pts.	20	17	20
#Grains	8	8	8
	0.9	1.4	2.6
	20	20	20
	10	10	10
	2.2	1.9	
	20	20	
	14	14	
	1.1		1.7
	20		20
	11		11

Mineral: Olivine

Mineral analysis: U.S.G.S. ARL-EMX Microprobe, 1979, L.B. Wiggins, analyst
(1) analysis of one grain (2) average of three grains

	oxide wt% $\pm 1\sigma$	oxide wt% $\pm 1\sigma$
SiO ₂	39.30 \pm 0.96	39.28 \pm 0.66
FeO	17.02 \pm 0.18	16.94 \pm 0.21
MgO	43.00 \pm 0.46	43.28 \pm 0.55
Total	99.32	99.50

Evaluation: The wet chemical analysis of the olivine from the Springwater meteorite has a low total, 99.5% by weight. The formula unit, (Mn,Mg,Fe)_{2.015}Si_{0.992}O₄, has a small deficiency of tetrahedral, and sufficiency of octahedral, cations. The material is homogeneous with respect to the three major elements, Si, Mg, and Fe. Although OLSW offers a higher FeO content than the Marjalahti olivine, OLMJ, the reported chemistry is uncertain.

Standard: Al₂O₃
 Locality: Synthetic corundum
 Donor: Materials Research Corp.
 References: P.O. #13125 - Lot#3034531-1 2/16/83
 (1) theoretical composition. (2) "typical chemical analysis" by emission spectrographic technique provided by Materials Research Corporation.

	(1)		
	Oxide Wt. %		
	theoretical		
Al ₂ O ₃	100.00	Na	<5 ppm
		Si	<1
		Fe	<3
		Ca	<1
		Zn	<1
		Pb	<1
		Cr	<5
		Ga	<1
		Mg	<1

Element	Al
S.R.	4.5
#Pts.	20
#Grains	7
	12.3
	20
	10
	13.4
	20
	5

Evaluation: The synthetic corundum is assumed to be pure Al₂O₃; there is no evidence that impurities are present. Chunks of OXAL are composed of very fine-grained crystals; polished surfaces of these chunks appear homogeneous, even though pitted. The measured homogeneity is poor, but the cause of the apparent heterogeneity is probably pitting of the surface. The lowest sigma ratios are associated with the best polished mounts. OXAL should not be used as an Al standard but, if adequately polished so that the microprobe beam can be kept out of the pits, it is suitable for use as a background standard, a purpose for which it has been used successfully.

Standard: Bushveld Chromite
 Locality: Bushveld-South Africa
 Donor: B.R. Lipin
 Reference: U.S. Geol. Survey Analytical Laboratory No. W-205250 (1979)
 for Wiggins Neuville and Aruscavage, Analysts.

Oxide wt.%

SiO ₂	0.16
Al ₂ O ₃	14.3
FeO	18.2
MgO	10.7
MnO	0.21
Cr ₂ O ₃	47.44
NiO	0.10
TiO ₂	0.63
Fe ₂ O ₃	8.12
V ₂ O ₅	0.20
Total	<u>100.06</u>

Si	0.005
Al	0.545
Fe ³⁺	0.198
Fe ²⁺	0.492
Mg	0.516
Mn	0.006
Cr	1.213
Ni	0.003
Ti	0.015
V	0.004
sum cations	<u>2.996</u>
sum anions	4.000

Element	Al	Fe	Mg	Cr
S.R.	1.1	1.8	1.2	1.9
#Pts.	20	20	20	20
#Grains	7	12	12	11

Mineral: Bushveld Chromite

Microprobe analysis: (1) U.S.G.S. ARL-SEM; Bence-Albee reduction. Average of ten points. E. McGee, analyst, 3/18/82. (2) University of Oregon. ARL-EMX-SM; Bence-Albee reduction, 5 μ M beam, average of 20 spots on 4 grains. Michael Shaffer, analyst, 2/85.

	wt.% 1σ (1)	wt.% 1σ (2)
SiO ₂	0.00+0.00	
Al ₂ O ₃	14.04+0.08	13.76+0.30
FeO	25.24+0.15	25.64+0.27
MgO	10.97+0.29	10.61+0.23
MnO	0.41+0.01	0.35+0.05
Cr ₂ O ₃	47.48+0.35	47.54+0.34
NiO	0.09+0.03	
TiO ₂	0.56+0.04	0.55+0.05
CaO	0.00+0.00	
Total	98.80+0.59	

Si	0.000
Al	0.537
Fe ²⁺	0.469
Fe ³⁺	0.217
Mg	0.531
Mn	0.011
Cr	1.219
Ni	0.002
Ti	0.014
Ca	0.000
cations	3.000
anions	4.000

Standards

OXIL - Fe, Ti
 OXTB - Cr
 OXGH - Al
 OLNI - Ni
 OLMJ - Mg
 OLST - Mn
 PXWO - Ca, Si

Chromite 52NL-11 - Cr, Al, Fe, Mg
 OXIL - Ti, Mn

Evaluation: The conventional analysis of the Bushveld chromite has an excellent sum; the cation/anion ratio, 2.996/4, does not depart significantly from the stoichiometric ratio, 3.000/4. Independent microprobe analyses reproduce the major elements of the conventional analysis quite well, therefore supporting both the conventional analysis and the standards used for microprobe analysis. The agreement for the Cr_2O_3 value is particularly good; the microprobe standards were OXTB and a Stillwater chromite of M. Beeson. Bushveld chromite appears homogeneous for Al, Fe, Mg, and Cr. OXBU should be an excellent standard for chromium in oxides.

Date	From	To	Address
09-OCT-84	J.S. Huebner	Michael Shaffer	Dept. Geology University of Oregon Eugene, OR 97403-1272
12-MAR-85	J.S. Huebner	Gene Jarosewich	U.S. National Museum Washington, DC 20001

Standard: Corundum
Locality: Synthetic?
Donor:

References: This material may be the corundum OXSC distributed by
E. Jarosewich. It was received by L.B. Wiggins and is now mounted in
the Reston OXIDE standard block.

	Oxide wt.% (theoretical)
Al ₂ O ₃	100.00

Element	Al
S.R.	1.3
#Pts.	20
#Grains	9

Evaluation: Despite its uncertain origin, the material mounted in the oxide standard block is homogeneous Al₂O₃ that has been used successfully as an aluminum standard and as a background standard for elements other than aluminum.

Standard: Gahnite
 Locality: Gahnite, Brazil
 Donor: C.A. Francis (Harvard Museum No. 111989)
 References: (1) U.S.G.S. Analytical Laboratories #W201964. Analyst, Schnepfe;
 Excludes 0.20% SiO₂ present as inclusions; (2) semiquantitative emission
 spec. analysis lab. #W-201964. Dorrzaph/Lancaster, Analysts; for
 L.B. Wiggins, 1978. (3) Combined analysis.

	Oxide wt. %		
	(1)	(2)	(3)
Al ₂ O ₃	55.59	>60.	55.59
SiO ₂	0.58		-
FeO	0.21		0.21
Fe ₂ O ₃	2.34	2.6	2.34
MnO	0.36	0.658	0.36
ZnO	41.22	>1.245	41.22
MgO		0.038	0.038
CaO		<0.021	-
K ₂ O		<0.082	-
TiO ₂		<0.011	-
P ₂ O ₅ *		<0.14	-
BeO		0.004	0.004
CoO		0.001	0.001
Cr ₂ O ₃		0.0005	0.0005
CuO		0.0041	0.0041
GaO		0.024	0.024
NiO		0.001	0.001
V ₂ O ₅		0.000	-
Total	<u>99.72</u>		<u>99.793</u>

Al	1.986
Fe ⁺²	0.005
Fe ⁺³	0.053
Mn	0.009
Zn	0.923
Mg	0.0017
Be	0.0003
Co	0.000
Cr	0.000
Cu	0.0001
Ga	0.0005
Ni	0.0000
sum cations	<u>2.980</u>
sum anions	4.000

Element	Al	Zn
S.R.	1.2	
#Pts.	20	
#Grains	2	
	1.2	2.4
	20	20
	2	2

Evaluation: The combined chemical analysis (analysis #1 plus the additional elements in the semi-quantitative spectrographic analysis #2) has an adequate sum of 99.79%. This combined analysis can be recalculated to a formula unit with 2.980 cations per 4.000 oxygens; the deviation from the ideal 3:4 ratio, 0.02 cations, exceeds that expected of a good microprobe analysis. This degree of nonstoichiometry exceeds the value that can come about through analytical error. It is possible to achieve a 3:4 cation to anion ratio by reducing all iron to the divalent state, but this is not likely to be the case. Perhaps OXGH is a truly non-stoichiometric spinel. The gahnite is unique as a standard for zinc in oxides (Rhodonite PXBK is a possible standard for zinc in silicates.) The gahnite is also used to calibrate the energy dispersive, multi-channel analyzers because the zinc $K\alpha_1$ and $L\alpha_1$ x-ray lines are widely separated (8.631 and 1.009 keV, respectively) and give high count rates.

Date	From	To	Address
06-OCT-83	Huebner	G.K. Czamanske	U.S. Geological Survey Mailstop 910, Bldg. 2 Menlo Park, CA 94025

Standard: Hausmannite

Locality: Synthetic Mn_3O_4

Donor: J.S. Huebner

Reference: Synthesized by firing MnO_2 (see OXPG) in air at $\sim 1000^\circ C$. See
a) Huebner, J.S. (1969) Amer. Mineral. 54, 457-481. b) Huebner, J.S. and
M. Sato (1970) Amer. Mineral. 55, 934-952.

	Oxide wt.% (theoretical)
MnO	93.006

Element	Mn
S.R.	2.0
#Pts.	20
#Grains	20
	3.5
	18
	18
	3.9
	16
	16

Evaluation: On the basis of examinations by reflected light microscopy, X-ray powder diffraction, and electron microprobe, OXHA is homogeneous, single phase hausmannite. No evidence of γMn_2O_3 , which is isostructural with Mn_3O_4 , was found. A satisfactory value of the sigma ratio is obtained only when great care is taken to avoid the numerous small voids exposed at the polished surface. In theory, OXHA should be a good standard for oxides containing Mn^{+2} and Mn^{+3} ; in practice the nature of the surface makes it difficult to use. OXPA is a preferable standard.

Standard: Ilmenite
Locality: Ilmen Mts., Miask, USSR
Donor: E. Jarosewich (USNM 96189)
Reference: Jarosewich, E., et al. (1979) Electron Microprobe Reference Samples for Mineral Analyses. Smithsonian Contributions to the Earth Sciences 22, 68-72. Chemical analysis using classical methods, plus microprobe analyses.

Oxide wt.%

TiO ₂	45.70
NbO ₅	0.92
Fe ₂ O ₃	11.60
FeO	36.10
MgO	0.31
MnO	4.77
Total	<u>99.40</u>

Ti	0.877
Nb	0.011
Fe ⁺³	0.223
Fe ⁺²	0.770
Mg	0.012
Mn	<u>0.103</u>
sum cations	1.995
sum anions	3.000

Element	Ti
S.R.	1.9
#Pts.	20
#Grains	1

Fe
2.7
20
1

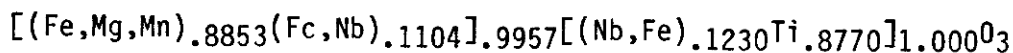
Mineral: Ilmenite
Microprobe analysis: U.S.G.S. ARL/SEM-Q - Bence-Albee reduction. 3/18/82.
E. McGee. Average of 3 points.

	wt.% 1
TiO ₂	45.90±0.60
FeO	46.60±0.23
MgO	0.40±0.01
MnO	4.77±0.09
Cr ₂ O ₃	0.04±0.02
Al ₂ O ₃	0.03±0.01
SiO ₂	0.00±0.00
CaO	0.00±0.00
Total	97.74±0.37

Standards:

OXIL - Fe,Ti
OXTB - Cr
OXGH - Al
OLNI - Ni
OLMJ - Mg
OLST - Mn
PXWO - Si,Ca

Evaluation: Although the summation is significantly lower than the ideal value, the classical wet chemical analysis can be recalculated to an excellent formula unit,



which corresponds to a solid solution between ilmenite (89%) and niobian hematite (11%). An independent microprobe analysis confirms the MgO value. The low summation of the wet chemical analysis could be due to low analytical values for trivalent cations. The sigma ratio for Fe is 2.7, a value which is greater than desirable. It is a general standard for Ti and an Fe and Mn standard for oxides. OXIL also serves as a known-unknown oxide for checking microprobe standardizations, although such usage for Fe must be based on the average of 5 to 10 points.

Distribution:

Date	From	To	Address
2/27/81	McGee	Dan Schulze	Department of Geosciences University of Texas at Dallas Richardson, TX 75080

Standard: Manganosite

Locality: Synthetic

Donor: J.S. Huebner

Reference: Synthesized from MnO_2 (see Pyrolusite, OXPY) in H_2 reduction furnace by Huebner and Sato (1970, The oxygen fugacity-temperature relationships of manganese oxide and nickel oxide buffers. Amer. Mineral. 55. 934-952.)

Oxide wt.% (theoretical)

MnO 100.00.

Evaluation: The manganosite structure, like that of wustite, permits cation vacancies. The composition of OXMN is not known with certainty, but the fact that it was synthesized in hydrogen (very reducing) would suggest that this particular oxide might be stoichiometric MnO. On exposure to air, some manganosites become brown, probably due to oxidation to Mn_3O_4 . OXMN is not recommended as a microprobe standard (use OXPR or OXHA instead) but could find use in a study of manganese X-ray wavelength shift with valence.

Distribution:

Date	From	To	Address
2/20/68	Huebner	Arden L. Albee	Department of Geological Sciences California Institute of Technology Pasadena, California
12/01/69	Huebner	L.A. Fernandez	Dept. Geology and Geophysics Box 2161, Yale Station New Haven, Connecticut 06520
02/10/70	Huebner	Charles V. Guidotti	University of Wisconsin Department of Geology & Geophysics Madison, Wisconsin 53706

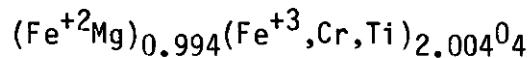
Standard: Magnetite
 Locality: Minas Gerais, Brazil
 Donor: USNM 114887. E. Jarosewich.
 Reference: 1) Jarosewich, E., Nelen, J.A., and Norberg, J.A., 1979, Smiths. Contrib. Earth Sciences no. 22, p. 71, J. Norberg, analyst. (2) same reference as 1), but with iron expressed as FeO.

	Oxide Wt. %	
	(1)	(2)
Fe ₂ O ₃	67.5	-
FeO	30.2	90.94
MgO	0.05	0.05
TiO ₂	0.16	0.16
Cr ₂ O ₃	0.25	0.25
MnO	<0.01	<0.01
Total	<u>98.16</u>	<u>91.40</u>

Fe ³⁺	1.991
Fe ²⁺	0.990
Mg	0.003
Ti	0.005
Cr	0.008
Mn	-
sum cations	<u>2.996</u>
sum anions	4.000

Element	Fe
S.R.	1.6
#Pts.	20
#Grains	19
	1.5
	20
	20

Evaluation: The chemical analysis of the magnetite from Minas Gerais has a low summation, but when recalculated, it yields an almost perfectly stoichiometric formula unit,



The material is homogeneous with respect to the only major element present, iron. The presence of minor Cr₂O₃ and TiO₂ distinguish OXMT from the pure, synthetic magnetite OXSM. The low summation of OXMT is disturbing, but the larger grain size results in a mount that, compared with OXSM, has less polishing relief.

Standard: Bunsenite, NiO
Locality: Synthetic
Donor: J. Castaing to S. Kirby to J.S. Huebner (1984)
Reference: Crushed fragment of single crystal; Arc image <100-1000 ppm
impurities

Oxide Wt.% (theoretical)

NiO 100.00

Element	Ni
S.R.	1.5
#Pts.	20
#Grains	6

Evaluation: OXNC is assumed to be pure NiO. It is homogeneous. The material is an excellent background standard, suggesting that its impurity level is indeed low. OXNC can be used as a background standard with relatively high mean atomic number or as a Ni standard.

Standard: Partridgeite

Locality: Synthetic

Donor: J.S. Huebner

References: Synthetic, prepared by ignition of MnO₂ (XPY) in air at 800°C (Huebner, 1969, Stability relations of rhodochrosite in the system manganese-carbon-oxygen. Amer. Mineral., 54, 457-481.) (1) theoretical weight percent value.

	Oxide wt% (theor.)
	(1)
MnO	89.869

Element	Mn
S.R.	2.9
#Pts.	19
#Grains	19
	3.2
	20
	20

Evaluation: X-ray powder diffraction and reflected light microscopy indicate that OXPA is a single phase, bixbyite, that forms polycrystalline granules. The pyrolusite starting material (XPY) had only 40 ppm impurities detected with semi-quantitative spectrographic analysis. OXPA is presumed to be pure Mn₂O₃. The value of the sigma-ratios are poor; this is believed to reflect the voids which give the surface a pitted appearance. It should be an excellent standard for manganese in oxides, providing that care is taken to avoid the voids in the grains.

Date	From	To	Address
20-FEB-68	Huebner	Arden L. Albee	Department of the Geological Sciences California Institute of Technology Pasadena, California
01-DEC-69	Huebner	Louis A. Fernandez	Dept. Geology and Geophysics Box 2161, Yale Station New Haven, Connecticut 06520

Standard: Periclase
Locality: Synthetic
Donor: Unknown
Reference:

Oxide Wt. % (theoretical)

MgO 100.00

Element	Mg
S.R.	2.1
#Pts.	20
#Grains	1

Evaluation: The chemistry of synthetic periclase, OXPE, is assumed to be pure MgO. The measured value of the sigma ratio is adequate. The material is difficult to polish; perhaps the apparent homogeneity would improve if the polish were better. The Reston collection has had two sources of synthetic periclase. Material from the vial labelled OXPE was definitely used for the Mn standard block. The vial of periclase provided by the University of Calgary is now empty, and the physical appearance of that material is not known. The periclase in the Oxide Block and Block #15A appears similar to OXPE, but the identity has not been verified.

Standard: Quartz
Locality: Brazil
Donor: W. Pecora to E. Roedder
References: Crushed, sized, and washed in HCl by Huebner. U.S.G.S. Semi-Quant.
Spec. Rept. #76RESc0015, Anal. N. Rait, for 68 elements reported only Si.

Oxide wt.% (theor.)

SiO₂ 100.00

Element	Si
S.R.	1.1
#Pts.	20
#Grains	5

Evaluation: Quartz OXQZ is coarsely crystalline, clear, and chemically pure.
The material has an excellent sigma ratio. It is an ideal standard where
high concentrations of SiO₂ are found.

Standard: Rutile
Locality: synthetic
Donor: Bernard Evans (circa 1972) to J.C. Stormer to J.S. Huebner
Reference:

	Oxide wt.% (theoretical)
TiO ₂	100.00

Element	Ti
S.R.	1.9
#Pts.	20
#Grains	2

Evaluation: The synthetic rutile is presumed to be pure TiO₂. It is homogeneous in titanium and has been used as a background standard.

Standard: $Gd_2Mo_3O_{12}$
Locality: Synthetic
Donor: U.S. National Museum Division of Mineralogy
References: U.S. Nat. Museum #A1

Oxide wt.% (theoretical)

Gd_2O_3	45.64
MoO_3	<u>54.36</u>
Total	100.00

Element	Gd
S.R.	1.3
#Pts.	20
#Grains	1

Mo
1.5
19
1

Evaluation: There is no independent chemical analysis or phase characterization to confirm the theoretical chemistry of OXR1. The material is homogeneous. It could probably be used safely as a standard for minor concentrations of Gd and Mo in unknowns, when the desired accuracy is not great.

Standard: GdDyMo₃O₁₂
Locality: Synthetic
Donor: U.S. National Museum Division of Mineralogy
References: U.S. National Museum #A2

Oxide wt.% (theoretical)

Gd ₂ O ₃	22.67
Dy ₂ O ₃	23.32
MoO ₃	<u>54.01</u>
Total	100.00

Element	Gd	Dy
S.R.	1.6	1.9
#Pts.	20	20
#Grains	1	1

Evaluation: There is no chemical analysis or phase characterization that would confirm the theoretical composition of OXR2. The homogeneity of the material is adequate for its use as a standard, but it is inferior to OXR1, OXR3, and OXR4. Use of OXR2 should be restricted to the determination of small concentrations of Gd and Dy in unknowns.

Standard: YNbO_4
Locality: Synthetic
Donor: U.S. National Museum Division of Mineralogy
References: U.S. Nat. Mus. #A4; Dupont 5898-31-1

Oxide wt.% (theoretical)

Y_2O_3	45.93
Nb_2O_5	<u>54.07</u>
Total	100.00

Element
S.R
#Pts.
#Grains

Y
1.2
20
2

Nb
0.7
20
2

Evaluation: There is no chemical analysis or phase characterization of OXR3 that would confirm the theoretical compositions. The homogeneity of Y and Nb, determined by microprobe, is excellent. Without confirmation of composition, use of OXR3 should be restricted to the determination of minor or trace concentrations of Y and Nb in unknowns.

Standard: LaNbO_4
Locality: Synthetic
Donor: U.S. National Museum Division of Mineralogy
References: U.S. Nat. Mus. #C-4 - Dupont 9894-14X

Oxide wt.% (theoretical)

La_2O_3	55.07
Nb_2O_5	44.93
Total	<u>100.00</u>

Element	La
S.R.	1.0
#Pts.	20
#Grains	5

Nb
0.6
20
5

Evaluation: No chemical analysis or phase characterization of OXR4 is available. The sigma ratios were measured with the microprobe and are excellent; the Nb is extraordinarily homogeneous. The lack of independent confirmation of the chemistry prevents LaNbO_4 from being considered a major element standard, but OXR4 is probably dependable for analyzing minor to trace amounts of La and Nb in unknowns (because most microprobe operators do not expect great accuracy when analyzing minor and trace elements).

Standard: NiO
Locality: Synthetic bunsenite
Donor: Materials Research Corp.
References: P.O. #13125 - job 3034531-2 2/16/83, MARZ grade.

Oxide Wt. %	
NiO	100.00

Element	Ni
S.R.	1.6
#Pts.	20
#Grains	6

Evaluation: The purity of the synthetic NiO, OXSB, is not specifically known. The purity of MARZ grade Ni metal, from which OXSB was presumably synthesized, is 99.995%. The material is polycrystalline. If care is taken to assure that the microprobe beam is centered on the small NiO crystals, avoiding the interstitial void, a low sigma ratio results. If used with care, OXSB is suitable as a standard for Ni and as a background standard.

Standard: Corundum
Locality: Synthetic
Donor: E. Jarosewich (USNM) to Huebner, August 1984
References: USNM 657S - Jarosewich et al. (1979) Electron microprobe reference samples for mineral analyses. Smithsonian Contrib. Earth Sciences 22, p. 71. (1) Presumed wet chemical analysis. (2) element emission spec. analysis.

	Oxide Wt.%	
	(1)	(2)
Al ₂ O ₃	99.99	
SiO ₂		0.014
FeO		0.002
MgO		0.004
CaO		0.002
Na ₂ O		0.003
K ₂ O		0.002

Element	Al
S.R.	2.3
#Pts.	20
#Grains	8

Evaluation: The synthetic corundum is virtually pure Al₂O₃, having only very minor impurities. The heterogeneity for Al is somewhat greater than expected, perhaps because of the large degree of polishing relief, relative to the epoxy mounting medium. Another difficulty is that the grains are equant and tend to pluck during polishing. OXSC should be an excellent standard for Al in oxides and a good background standard for all light elements except Al.

Standard: Fe₃O₄
Locality: Synthetic magnetite
Donor: John Haas (USGS)
References: Hydrothermal synthesis

Oxide Wt. %, theoretical

Fe₃O₄ 100.00

theoretical, all iron as FeO

FeO 93.09

Element	Fe
S.R.	1.6
#Pts.	20
#Grains	19

Evaluation: Microprobe mounts of the synthetic magnetite are assumed to be pure Fe₃O₄. The mounted material is homogeneous. (The stock bottle has patches of reddish alteration, perhaps due to oxidation by residual aqueous chloride solution, in which this material was synthesized.) OXSM is homogeneous and, if polished flat (the grains are <100 micrometers across and tend to have topographic relief relative to the epoxy mounting medium), will serve as a good standard for mixed valence iron.

Standard: Spinel - $MgAl_2O_4$

Locality: Single-crystal, synthesized by the Lauda/Airtron Corp.

Donor: A.E. Bence; obtained from Bence by McGee, July, 1978.

Reference: Bence, A.E., and Holzworth, W., 1977, Non-linearities of electron microprobe matrix corrections in the system $MgO-Al_2O_3-SiO_2$. Eighth International Conference on X-ray Optics and Microanalysis and Twelfth Annual Conference of the Microbeam Analysis Society, Boston. p. 38A.

Oxide wt.% (theoretical)

Al_2O_3	71.67
MgO	28.33
Total	100.00

Element	Mg	Al
S.R.	1.7	1.4
#Pts.	20	20
#Grains	1	1

Evaluation: This synthetic spinel is assumed to be stoichiometric $MgAl_2O_4$. It is homogeneous and has been used successfully in Reston as a standard for Mg and Al in oxides.

Standard: ZnO
 Locality: Synthetic zincite
 Donor: Materials Research Corp.
 References: P.O. #13125 - Lot#3034531-3 2/16/83
 (1) Ideal composition (2) "Typical Chemical Analysis" supplied by
 manufacturer, "Mass Spec" method.

	(1) Oxide Wt.% (theoretical)		(2) Wt.%
ZnO	100.00	Cd	0.0001
		Cu	<0.0032
		Fe	<0.0022
		Pb	0.021
		Si	0.0028
		Mn	<0.0016
		Al	0.0008
		Ca	0.0008
		Na	0.0005
		Mg	0.0002

Element	Zn
S.R.	6.1
#Pts.	20
#Grains	5
	3.3
	20
	3

Evaluation: The chemical analysis provided by the manufacturer shows only trace levels of metals other than Pb; thus OXSZ is probably at least 99.9% pure. The synthetic ZnO is an extremely fine-grained, chalky material. The polycrystalline chunks do not take a good polish, and the cores are pitted in one mount and show bubbles under the carbon coat in another mount. The poor surface undoubtedly contributes to the poor sigma ratio values. OXSZ has been used as a background standard, a purpose for which it might be valid, but it should not be used as a standard for zinc.

Standard: Chromite

Locality: Tiebaghi, New Caledonia

Donor: T. Thayer to B. Lipin (conversation with Lipin, March 13, 1985)

References: (1) USGS W-204468, Analysts Neuville and Aruscavage, 5/19/79, wet chem. (2) Relationship with USNM#117075 not known with certainty, but the USNM sample has similar chemistry. Jarosewich et al., 1979, Electron microprobe reference samples for mineral analysis. Smithsonian Contributions to the Earth Sciences 22, 68-72.

	wt. %	
	(1)	(2)
SiO ₂	0.24	-
Al ₂ O ₃	9.6	9.92
FeO	10.0	13.04
Fe ₂ O ₃	3.60	-
MgO	15.6	15.20
MnO	0.13	0.11
Cr ₂ O ₃	60.79	60.5
NiO	0.18	-
TiO ₂	0.20	-
V ₂ O ₅	0.09	-
CaO	-	0.12
Total	<u>100.43</u>	<u>98.89</u>

Si	-
Al	0.361
Fe ²⁺	0.267
Fe ³⁺	0.086
Mg	0.741
Mn	0.004
Cr	1.532
Ni	0.005
Ti	0.005
V	0.002
Ca	-
sum cations	<u>3.003</u>
sum anions	4.000

Element	Al	Fe	Mg	Cr
S.R.	1.2	1.8	2.3	
#Pts	20	20	20	
#Grains	16	16	16	
			3.3	1.3
			20	20
	1.2		4	4
	20			
	16	2.4	2.2	
		19	19	1.8
		12	12	17
				17
		1.8	2.3	
		20	20	
		16	16	

Mineral: Tiebaghi chromite

Microprobe analysis: 1) U.S.G.S. ARL-SEM-Q microprobe. Bence-Albee reduction; J. McGee, analyst, 08-FEB-82; average of 5 pts. 2) U.S.G.S. ARL-SEM-Q. Bence-Albee reduction; E. McGee, analyst, 18-MAR-82; average of 4 pts.

	oxide wt.% $\pm 1\sigma$ (1)	oxide wt.% $\pm 1\sigma$ (2)
SiO ₂	0.02 \pm 0.02	0.00 \pm 0.00
Al ₂ O ₃	9.88 \pm 0.14	9.44 \pm 0.14
NiO	-	0.17 \pm 0.02
FeO	12.92 \pm 0.32	12.87 \pm 0.18
MgO	15.86 \pm 0.27	15.75 \pm 0.40
MnO	0.34 \pm 0.01	0.33 \pm 0.01
Cr ₂ O ₃	62.60 \pm 0.30	61.53 \pm 0.25
TiO ₂	0.12 \pm 0.04	0.12 \pm 0.04
CaO	0.00 \pm 0.00	0.00 \pm 0.00
Na ₂ O	0.00 \pm 0.01	-
K ₂ O	0.03 \pm 0.03	-
Total	101.77 \pm 0.16	100.19 \pm 0.27

(1)
Standards
FSTA - Na,Al
OLST - Mn
OXIL - Ti,Fe
OXTB - Cr
Or-1 Orthoclase - K
PXAD - Ca,Mg,Si

(2)
Standards
OXIL - Fe,Ti
OXTB - Cr
OXGH - Al
OLNI - Ni
OLMJ - Mg
OLST - Mn
PXWO - Si,Ca

Evaluation: The classical analysis of chromite from Tiebaghi has a good sum. The Reston chromite sample originated with T. Thayer; if Thayer procured it from the USNM, the Reston OXTB may be the same as USNM #117075. If the two samples are the same, the USNM analysis supports the validity of the USGS analysis (and vice versa). The USGS analysis has an excellent cation/anion ratio, 3.003/4.000, and can be reconstituted to the reasonable formula unit:



A homogeneity study for OXTB gave good sigma ratios. OXTB is a good standard for Cr, Mg, Al, and Fe in oxides. When OXTB is used as a standard for chromium, microprobe analyses reproduce the chromium value of OXBU.

Date	From	To	Address
22-JUL-81	J. McGee	Dave Clague	MS 99 - U.S. Geological Survey 345 Middlefield Road Menlo Park, CA 94025
09-OCT-84	J.S. Huebner	Michael Shaffer	Dept. Geology University of Oregon Eugene, OR 97403-1272
12-MAR-85	J.S. Huebner	Gene Jarosewich	U.S. National Museum Washington, DC 20001

Standard: Chromite MB-5
Locality: Union Bay, Alaska
Donor: T.N. Irvine

Reference: Can. Jour. Earth Sci. 4, 94. J.L. Bouvier and J.A Maxwell,
analysts. NiO and V₂O determined by X-ray fluorescence by G.R. Lachance
and CaO by emission spectrography.

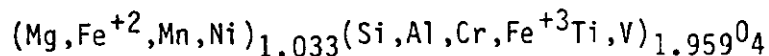
Oxide wt.%

Cr ₂ O ₃	25.2
Al ₂ O ₃	9.6
Fe ₂ O ₃	32.4
FeO	23.1
MgO	7.1
MnO	0.32
CaO	<0.05
TiO ₂	2.0
SiO ₂	0.10
V ₂ O ₃	0.15
NiO	0.15
Total	<u>100.12</u>

Cr	0.680
Al	0.386
Fe ³⁺	0.832
Fe ²⁺	0.659
Mg	0.361
Mn	0.009
Ti	0.051
Si	0.003
V	0.003
Ni	0.004
sum cations	<u>2.991</u>
sum anions	4.000

Element	Fe	Mg		
S.R.	3.1	2.1		
#Pts.	20	20		
#Grains	11	11		
			Al	Cr
			2.6	2.8
			20	20
			14	14
			1.8	2.7
			20	20
			14	14
			2.6	
			20	
			12	

Evaluation: The analysis of chromite MB-5 from Union Bay, Alaska, has an excellent summation. The formula unit



is, within analytical uncertainty, that of a stoichiometric spinel. The homogeneity of the major elements Fe, Al, and Cr is only marginally acceptable. The Bushveld chromite OXBU should be considered for use instead of OXUB.

←

=

Standard: Vanadium Oxide
Locality: Synthetic V_2O_3
Donor: USNM #79
Reference:

Oxide wt.% (theoretical)

V_2O_3 100.00

Element	V
S.R.	1.0
#Points	20
#Grains	2

Evaluation: The single crystal of OXVA is opaque with a bluish, iridescent surface. The black streak and crystal morphology are appropriate for V_2O_3 (V_2O_5 has a yellowish-brown streak). X-ray precession photography by H.T. Evans, Jr., yields the diffraction pattern of a single phase, V_2O_3 . The material mounted in position #7 of the oxide block is homogeneous for V. It is the same material as OXVA. OXVA should be an excellent standard for trivalent vanadium.

Standard: Chromite (55G-4)

Locality: Stillwater Complex, Montana

Donor: Dale Jackson to M. Beeson to B.A. Morgan

References: Analyst, J.I. Dinnin. See J.I. Dinnen (1959) Rapid Analysis of Chrome and Chrome Ore. U.S. Geological Survey Bulletin 1084-B. (1) Original analysis. (2) Corrected with CaO, SiO₂, H₂O, and CO₂ removed and normalized to 100%.

	Oxide wt.%	
	(1)	(2)
Cr ₂ O ₃	36.6	36.83
Al ₂ O ₃	14.4	14.49
Fe ₂ O ₃	16.0	16.14
FeO	24.0	24.05
MgO	7.2	7.02
TiO ₂	0.91	0.925
V ₂ O ₃	0.17	0.176
MnO	0.19	0.178
NiO	0.15	0.150
CaO	<0.01	0.038
CuO	0.008	-
CoO	0.043	-
ZnO	<0.05	-
SiO ₂	0.23	-
H ₂ O	0.12	-
CO ₂	-	-
Total	<u>100.021</u>	<u>100.00</u>

Cr	0.970
Al	0.569
Fe	0.405
Fe	0.670
Mg	0.348
Ti	0.023
V	0.004
Mn	0.005
Ni	0.004
Ca	0.001
sum cations	<u>2.999</u>
sum anions	4.0

Element	Mg	Al	Cr	Fe
S.R.	2.6			
#Pts.	20			
#Grains	13			
		2.5		
		20		
		13		
	5.8			6.5
	20			20
	13			13
			2.1	
			20	
			10	

Evaluation: The chemical analysis of "chromite 51" (so called because it is the fifty-first standard in Ben Morgan's green book) has an excellent summation. The normalized analysis can be recalculated to an almost perfectly stoichiometric spinel formula unit without adjustment of reduced or oxidized species. The homogeneity for Al and Cr are only adequate, and Fe and Mg are very heterogeneously distributed. Better chromite standards, such as OXTB, OXBU, and OX52 are available and preferable to OX51.

Standard: Chromite (55G-15AB)

Locality: Stillwater Complex, Montana

Donor: Dale Jackson to M. Beeson to B.A. Morgan

References: Analyst, J.I. Dinnen. See J.I. Dinnen (1959) Rapid Analysis of Chrome and Chrome Ore. U.S. Geological Survey Bulletin 1084-B. (1) Original analysis. (2) Corrected with CaO, SiO₂, H₂O, and CO₂ removed and normalized to 100%.

	Oxide wt.%	
	(1)	(2)
Cr ₂ O ₃	40.7	40.79
Al ₂ O ₃	12.6	12.62
Fe ₂ O ₃	15.4	15.48
FeO	21.3	21.36
MgO	8.6	8.45
TiO ₂	0.80	0.80
V ₂ O ₃	0.18	0.175
MnO	0.16	0.177
NiO	0.13	0.11
CaO	<0.07	-
CuO	0.0013	-
CoO	0.036	0.038
ZnO	<0.05	-
SiO ₂	0.18	-
H ₂ O	0.06	-
CO ₂	-	-
Total	<u>100.1</u>	<u>100.00</u>

Cr	1.072
Al	0.494
Fe ³⁺	0.387
Fe ²⁺	0.594
Mg	0.418
Ti	0.020
V	0.004
Mn	0.005
Ni	0.003
Co	0.001
sum cations	<u>2.998</u>
sum anions	4.0

Element	Fe	Mg	Cr	Al
S.R.	2.2	2.3		
#Pts.	20	20		
#Grains	14	14		
		2.5	1.7	0.6
		20	20	20
		13	13	13

Evaluation: OX52 is so-named because it is Chromite #52 in Ben Morgan's green book of standards. The original summation is excellent. Analysis #2 was normalized to 100.00% after CaO, SiO₂, H₂O, and CO₂ were removed. The cation to anion ratio calculated from the normalized analysis is 2.998 to 4.0; conversion of 0.24% Fe₂O₃ to FeO results in the ideal stoichiometric spinel ratio. OX52 is homogeneous in Al and Cr but only marginally homogeneous in Mg and Fe. Before OX52 is used as a standard, the compositionally similar OXBU should be considered.

Standard: Acmite
Locality: Synthetic
Donor: D.E. Voigt
Reference: Hydrothermal crystallization, in Ag capsule at 775°C and 15 kbars,
of sintered mix of NaHCO₃, Fe₂O₃, and SiO₂. (2) Iron recalculated as FeO.

	Oxide Wt. % (theoretical)	
	(1)	(2)
Na ₂ O	13.42	13.42
Fe ₂ O ₃	34.56	31.10
SiO ₂	52.02	52.02
Total	<u>100.00</u>	<u>96.54</u>

Element	Si	Fe	Na
S.R.	1.4	1.2	1.1
#Points	20	20	20
#Grains	20	20	20

Standard: Diopside
 Locality: Adirondacks, Natural Bridge, NY
 Donor: William Melson (USNM 117733)
 Reference: Jarosewich, E., et al. (1979) Electron Microprobe Reference Samples
 for Mineral Analysis. Smithsonian Contributions to the Earth Sciences 22,
 68-72.

Oxide wt.%

SiO ₂	54.87
Al ₂ O ₃	0.11
FeO	0.24
MgO	18.30
MnO	0.04
CaO	25.63
Na ₂ O	0.34
Total	<u>99.53</u>

Si	1.991
Al ^{VI}	0.005
Fe ⁺³	0.007
Mg	0.990
Mn	0.001
Ca	0.996
Na	0.024
sum cations	<u>4.014</u>
sum anions	6.000

Element	Si	Mg
S.R.	1.2	1.6
#Pts.	20	20
#Grains	3	3

Ca
 0.9
 20
 5

Mineral: Diopside

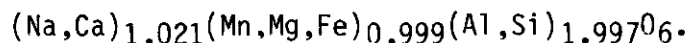
Microprobe analysis: (1) U.S.G.S. ARL-EMX microprobe; 10 points on one grain; L.B. Wiggins, analyst. (2) U.S.G.S. ARL-SEMQ microprobe; MAGIC reduction; average of 10 points; J. McGee, analyst. 18-JAN-82.

	wt% 1σ (1)	wt% 1σ (2)
SiO ₂	55.12±0.28	55.06±0.45
MgO	18.07±0.13	18.25±0.15
FeO	0.26±0.03	0.22±0.05
CaO	26.52±0.35	25.76±0.29
TiO ₂	-	0.03±0.04
MnO	-	0.02±0.01
Na ₂ O	-	0.17±0.07
Al ₂ O ₃	-	0.16±0.13
Total	99.97	99.68

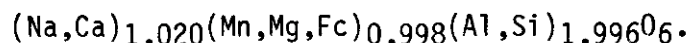
Standards:

OXIL - Fe,Ti,Mn
 FSTA - Na,Al
 PXAD - Ca,Mg,Si

Evaluation: The sum of the conventional analysis is several tenths of a percent low. The formula unit calculated from the original analysis of Jarosewich et al. (1979a) suggests a deficiency of tetrahedrally-coordinated cations and a sufficiency of cations in octahedral coordination:



There is insufficient ferrous iron in the analysis to achieve a reasonable formula unit by assuming all the iron to be ferric:



Jarosewich et al. (1979b) and Reston microprobe operators have found the Adirondack diopside suitably homogeneous for use as a microprobe standard; while a separate test of homogeneity has revealed appropriately low sigma ratio values of 1.2, 0.9, and 1.6 for Si, Ca, and Mg. PXAD has been widely used as a standard for Si in pyroxenes and olivine; as a Mg standard for general use; and as a Ca standard in pyroxenes and for general use. PXAD has served as a known-unknown for pyroxenes. Despite its successful use as a reference material, inspection of the formula suggests that the silica value is too low. However, addition of sufficient silica to achieve an ideal 4 cations per 6 anions would cause the weight percent summation to rise to 101% and create an excess of tetrahedrally coordinated cations, suggesting that silica alone is not the only problem with the analysis. Pending reanalysis, the Adirondack diopside should be used only with caution.

Standard: Augite DL-6
Locality: California
Donor: Ben Morgan from H. Wilshire
References: (1) U.S.G.S. analytical lab. report no. 73-W0-3; J. Fahey, anal. classical wet chem., 1973 (2) U.S.G.S. semiquantitative spectrographic anal. rep. No. 73-WS-71 Norma Rait, analyst; 1973.

	Oxide wt.%		Oxide wt.% (cont'd.)	
	(1)	(2)	(1)	(2)
SiO ₂	46.26	>21.	BaO	- 0.000
Al ₂ O ₃	8.14	9.4	CoO	- 0.002
Fe ₂ O ₃	5.21	-	CuO	- 0.006
FeO	5.60	13.	NiO	- 0.002
TiO ₂	1.42	1.7	PbO	- 0.001
CaO	21.44	>14.	Sc ₂ O ₃	- 0.015
MgO	10.54	16.6	SrO	- 0.018
MnO	0.40	0.3	V ₂ O ₅	- 0.054
Na ₂ O	1.20	0.9	Y ₂ O ₃	- 0.004
K ₂ O	0.02	-	ZrO ₂	- 0.020
Cr ₂ O ₃	-	0.15	GaO	- 0.006
H ₂ O -110°C	0.01		YbO	- 0.0003
H ₂ O +110°C	0.00		Total	<u>100.24</u>

	(1)&(2)
Si	1.735
Al	0.360
Fe ³⁺	0.147
Fe ²⁺	0.176
Ti ²⁺	0.040
Ca	0.862
Mg	0.589
Mn	0.013
Na	0.087
K	0.001
Cr	-
Ba	0.000
Co	0.0001
Cu	0.0002
Ni	0.0001
Pb	0.0000
Sc	0.0005
Sr	0.0004
V	0.0013
Y	0.0001
Zr	0.0004
Ga	0.0001
Yb	-
sum cations	<u>4.013</u>
sum anions	6.000

Element	Si	Fe	Mg			
S.R.	1.3	1.6	1.1			
#Pts.	20	20	20			
#Grains	20	20	20	Al	Ca	Na
				1.1	1.3	0.9
				20	20	20
				20	20	20

Mineral: Augite DL-6

Microprobe analysis: (1) U.S.G.S. ARL-EMX microprobe; L.B. Wiggins, anal.; 1979
 1a) one grain; 1b) av. 3 grns. (2) U.S.G.S. ARL-EMX microprobe; combined EDA &
 WDA; Bence-Albee reduction; C. Thornber, anal. 2a) av. 3 grains; 2b) 5 grains

	oxide wt.% $\pm 1\sigma$ (1a)	oxide wt.% $\pm 1\sigma$ (1b)	oxide wt.% $\pm \sigma$ (2a)	oxide wt.% $\pm \sigma$ (2b)
SiO ₂	46.84 \pm 0.56	46.60 \pm 0.49	46.69 \pm 0.77	46.36 \pm 0.86
Al ₂ O ₃	8.45 \pm 0.51	8.36 \pm 0.17	8.38 \pm 0.24	8.37 \pm 0.17
TiO ₂	1.88 \pm 0.01	1.81 \pm 0.07	1.74 \pm 0.21	1.84 \pm 0.12
FeO	10.21 \pm 0.08	10.19 \pm 0.13	10.12 \pm 0.17	9.72 \pm 0.41
MgO	10.22 \pm 0.41	10.01 \pm 0.22	9.99 \pm 0.20	10.26 \pm 0.19
MnO	0.25 \pm 0.05	0.22 \pm 0.03	0.29 \pm 0.17	0.26 \pm 0.10
CaO	20.62 \pm 0.13	20.48 \pm 0.28	20.53 \pm 0.32	20.52 \pm 0.42
Na ₂ O	1.68 \pm 0.00	1.62 \pm 0.04	0.97 \pm 0.06	1.65 \pm 0.01
K ₂ O	0.06 \pm 0.02	0.05 \pm 0.02	-	-
Cr ₂ O ₃	-	-	0.18 \pm 0.17	0.04 \pm 0.05
Total	100.21	99.34	98.89	99.03

Si	1.749
Al	0.370
Fe ³⁺	0.151
Mn	0.007
Ti	0.051
Fe ²⁺	0.169
Mg	0.560
Ca	0.823
Na	0.118
K	0.002
sum cations	4.000
sum anions	6.000

(1)
Standards
 FSBO - K
 FSTA - Al,Na
 OLMJ - Mg,Si,Fe
 OXRU - Ti
 OLST - Mn
 PXWO - Ca

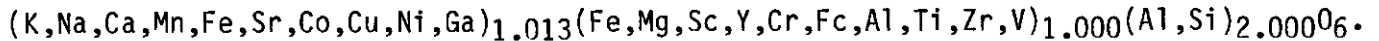
(2)
Standards
 PXAD - Mg,Si
 OXIL - Ti
 FSTA - Al,Na
 OLST - Mn
 PXWO - Ca
 OLSF - Fe

Mineral: Augite DL-6

Microprobe analysis: (2c) average of 3 grains; (3) U.S.G.S. ARL/SEM0 microprobe; Bence and Albee reduction, 02-MAR-82; Kempa, analyst; average of 10 points on 3 grains.

	oxide wt.% $\pm 1\sigma$ (2c)	oxide wt.% $\pm 1\sigma$ (3)
SiO ₂	46.32+0.16	44.91+0.29
Al ₂ O ₃	8.35+0.39	7.15+0.19
TiO ₂	1.84+0.16	1.96+0.10
FeO	9.90+0.18	9.75+0.13
MgO	10.34+0.13	10.21+0.12
MnO	0.27+0.24	0.12+0.02
CaO	20.89+0.32	20.60+0.15
Na ₂ O	1.56+0.01	1.52+0.02
K ₂ O	-	-
Cr ₂ O ₃	0.08+0.05	0.01+0.01
Total	99.54	96.23

Evaluation: The conventional mineral analysis (1) plus spectrographic data (2) has an adequate sum, 100.37%. The chemical analysis can be converted to an adequate pyroxene formula unit:



If 1.2% FeO is allowed to become Fe₂O₃, perfect pyroxene stoichiometry is achieved. Reston microprobe work indicates that DL-6 is homogeneous for all major elements. Augite DL-6 is an excellent standard for major elements in aluminous pyroxenes. It is almost a known-unknown for augites when using Kakanui augite PXKA as the major element standard.

Distribution:

Date	From	To	Address
09-OCT-84	J. S. Huebner	Michael Shaffer	Dept. Geology University of Oregon Eugene, OR 97403-1272

Standard: Aegirine #2 (also labelled BP-Z by Hearn)

Locality: Bearpaw Mountains, Montana

Donor: Carter Hearn (W.T. Pecora, originally)

References: (1) Pecora, W.T., 1942, Nepheline syenite pegmatites, Rocky Boy Stock, Bearpaw Mountains, Montana: Amer. Mineral., v. 27, p. 397-424. (See NEPH). Classical wet chemistry by F.A. Gonyer, Harvard University. (2) K₂O removed as phlogopite, normalized

Oxide Wt. %	(1)	(2)
SiO ₂	51.72	52.17
TiO ₂	1.32	1.36
Al ₂ O ₃	1.56	1.23
Fe ₂ O ₃	26.14	27.05
FeO	2.38	2.46
MnO	0.21	0.22
MgO	1.41	0.56
CaO	2.56	2.65
Na ₂ O	11.28	11.67
K ₂ O	0.34	0.00
ZrO ₂	0.24	0.25
H ₂ O	0.50	0.38
Total	99.66	100.00

Si	1.988	1.999
Ti	0.038	0.039
Al	0.071	0.056
Fe ³⁺	0.756	0.780
Fe ²⁺	0.076	0.079
Mn	0.007	0.007
Mg	0.081	0.032
Ca	0.105	0.109
Na	0.841	0.867
K	0.017	0.000
Zr	0.004	0.005
sum cations	3.984	3.973
sum anions	6.0	6.0

Element	Si	Fe	Na
S.R.	1.4	3.1	2.6
#Pts	20	20	20
#Grains	20	20	20

Evaluation: The summation of the original chemical analysis, 99.7% by weight, is slightly low, but can be recalculated to a pyroxene formula unit with an adequate, but not excellent cation:anion ratio of 3.984:6.000. The K₂O and H₂O values of 0.34% and 0.50% are anomalous for a pyroxene. Removal of all K₂O as phlogopite still leaves 0.38% water and results in a formula unit that has only 3.97 cations per 6 oxygens. Two of the three major components, Fe and Na, are heterogeneously distributed. PXAG is not recommended for a general purpose standard. However, a ferric-iron-bearing pyroxene is necessary to resolve uncertainties in the use of ferrous-iron standards for the analysis of octahedrally coordinated ferric iron in silicates. Until a better ferric-iron silicate standard becomes available, PXAG can be used for peaking of spectrometers and testing for wavelength shifts associated with changes in the oxidation state of iron.

Distribution:

Date	From	To	Address
	Carter Hearn	Alfred Anderson	Dept. Geophysical Sciences University of Chicago Chicago, IL 60637
	Carter Hearn	William Melson	U.S. National Museum Smithsonian Institution Washington, DC 20560
	Carter Hearn	Harry Rose	Branch of Analytical Laboratories U.S. Geological Survey Reston, VA 22092

Standard: Rhodonite

Locality: Broken Hill, N.S.W., Australia

Donor: J.S. Huebner (U.S.N.M. 90102)

Reference: 1) U.S. Nat. Mus. #90102. U.S.G.S. Anal. Lab. Rep. No. 68-WO-9; classical wet chem.; J.J. Fahey, analyst; 1968. (2) U.S.G.S. Semiquant. Spec. Rep. No. 71-WS-55; J.L. Harris, analyst; 1971. (3) composite analysis, Al₂O₃ removed as spessartite. (4) same analysis as (3), including Wiggins' MnO value.

	Oxide wt. %			
	(1)	(2)	(3)	(4)
SiO ₂	46.76	>21.	45.06	45.06
Al ₂ O ₃	0.96*	-	0.00	0.00
MnO	33.34	13.	31.34	35.34
FeO	12.39	13.	12.39	12.39
Fe ₂ O ₃	0.11	-	0.11	0.11
CaO	5.62	7.	5.62	5.62
MgO	0.42	0.2	0.42	0.42
TiO ₂	0.00	-	0.00	0.00
V ₂ O ₅	-	0.012	0.0125	0.0125
Na ₂ O	-	-	-	-
K ₂ O	-	-	-	-
AgO	-	0.0011	0.0011	0.0011
BaO	-	0.0002	0.0002	0.0002
BeO	-	0.0003	0.0003	0.0003
CuO	-	0.0038	0.0038	0.0039
PbO	-	0.0754	0.0754	0.0754
Total	<u>99.60</u>		<u>95.0333</u>	<u>99.033</u>

Si	4.928		5.049	4.925
Al	0.119		0.000	0.000
Mn	3.154		2.975	3.271
Fe ²⁺	1.092		1.161	1.132
Fe ³⁺	0.009		0.000	0.009
Ca	0.634		0.675	0.658
Mg	0.066		0.070	0.068
Na	-		-	-
K	-		-	-
Ti	0.000		0.000	0.000
V	0.0009		0.0009	0.0009
Ag	0.0001		0.0001	0.0001
Ba	0.0000		0.0000	0.0000
Be	0.0001		0.0001	0.0001
Cu	0.0003		0.0004	0.0003
Pb	0.0020		0.0021	0.0021
sum cations	<u>10.005</u>		<u>9.943</u>	<u>10.068</u>
sum anions	15.000			

Element	Si	Mg	Fe	Ca	Mn
S.R.	1.2	1.0	2.4		
#Pts.	20	20	20	1.0	
#Grains	8	8	8	19	2.0
				5	20
				1.0	5
				20	
				5	

Mineral: Rhodonite

Microprobe analysis: (1) U.S.G.S. ARL-EMX microprobe, Reston; 1979;
 L.B. Wiggins, analyst; average of 11 points. (2) same analysis as (1), plus
 spectrographic analyses, from page 1.

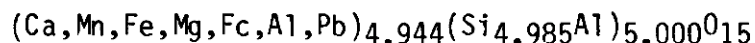
	Oxide wt.%	
	(1)	(2)
SiO ₂	46.46±0.38	46.46
Al ₂ O ₃	0.04±0.02	0.04
MnO	35.34±0.41	35.34
FeO	12.71±0.20	12.71
Fe ₂ O ₃	-	-
CaO	5.32±0.09	5.32
MgO	0.21±0.03	0.21
Ti ₂ O	-	-
V ₂ O ₅	-	0.0125
Na ₂ O	0.00±0.00	-
K	0.07±0.02	-
Ag	-	0.0011
Ba	-	0.0002
Be	-	0.0003
Cu	-	0.0038
Pb	-	0.0754
Total	<u>100.15</u>	<u>100.231</u>

	adjusted	adjusted
Si	4.986	4.985
Al	0.005	0.005
Mn	3.213	3.212
Fe ²⁺	1.109	1.112
Fe ³⁺	0.032	0.029
Ca	0.612	0.612
Mg	0.034	0.034
Na	0.000	-
K	0.010	-
Ti	-	-
V	-	0.0009
Na	-	0.00
K	-	0.010
Ag	-	0.0001
Ba	-	0.0000
Be	-	0.0001
Cu	-	0.0003
Pb	-	0.002
cations	<u>10.000</u>	<u>10.000</u>
anions	15.000	15.000

Standards:

- OLMJ - Si, Fe, Mg
- FSTA - Al, Na
- PXWO - Ca
- FSBO - K
- OLST - Mn

Evaluation: The conventional analysis of the rhodonite from Broken Hill has a sum that is several tenths of a percent low. Wiggins by independent microprobe analysis found similar values for Si, Fe, and Ca, but 35.3% MnO (compared with the wet chemical value of 33.34%) and only 0.04% Al₂O₃ (compared with the wet chemical value of 0.96%, but consistent with the semi-quantitative spectrographic determination). The mineral formula derived from the conventional analysis,



has a small, but significant, deficiency of octahedrally-coordinated cations. Removal of all 0.96% Al₂O₃ as spessartite does not improve the stoichiometry of the wet chemical analysis. In contrast, the Wiggins analysis, plus trace elements, corresponds to a formula with stoichiometry of 10.005 cations per 15 anions; after the redox adjustment to form 0.35% Fe₂O₃, the corresponding formula is



This formula is more reasonable than the formula calculated from the wet chemical analysis.

The Broken Hill rhodonite is very homogeneous for Si, Mg, and Ca; the homogeneity of Fe and Mn is poorer and may reflect a slight Fe/Mn variation in the grains. The microprobe analysis by Wiggins appears to be superior for major elements. PXBH should not be used as a known-unknown until the uncertainty in the chemical analysis is resolved.

Date	From	To	Address
01-DEC-69	Huebner	A.E. Bence	Dept. of Earth and Space Sciences State Univ. New York at Stony Brook Stony Brook, New York 11790
08-MAY-72	Huebner	Bill Bonnicksen	Department of Geological Sciences Kimbell Hall Cornell University Ithaca, N.Y. 14858
30-NOV-73	Huebner	Eric Essene	Dept. of Geology and Mineralogy University of Michigan Ann Arbor, Michigan 48104
30-APR-70	Huebner	Edward Ghent	Faculty of Arts and Science Department of Geology The University of Calgary Calgary 44, Alberta, Calgary
13-NOV-80	Huebner	Lester Hughes	CONICO, Inc. 244 Research Inc. P.O. 1267 Ponka City, OK 74601
22-JAN-73	Huebner	Brian Mason	Mineral Sciences Museum of Natural History Smithsonian Institution Washington, D.C. 20560

11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
11-DEC-74	Huebner	Ian Ridley	Lamont-Doherty Observatory Columbia University Palisades, NY 10964
17-FEB-75	Huebner	Peter Robinson	Department of Geology University of Massachusetts Amherst, MA 01002
13-NOV-80	Huebner	Dan Schulze	Programs in Geosciences - Station FO.2.1 The University of Texas at Dallas
08-SEP-78	Huebner	V.J. Wall	Department of Earth Sciences Monash University Clayton, Victoria Australia 3168
2/20/69	Huebner	Paul Weiblen	Dept. of Geology University of Minnesota Minneapolis, Minnesota 55455

Standard: Bald Knob Rhodonite
 Locality: Sterling Hill, NJ
 Donor: J.S. Huebner

Reference: U.S.G.S. Anal. Lab. Rep. No. 68-WO-9, wet chem., 30-JUN-71,
 J. Fahey, analyst; includes trace elements from U.S.G.S. Anal. Lab. Rep. No.
 71-WS-55, 03-JUN-71, J.L. Harris, analyst. (2) preferred analysis (1) but
 with microprobe values for MnO and ZnO.

	Oxide wt.%	
	(1)	(2)
SiO ₂	46.62	46.62
Al ₂ O ₃	0.28	0.28
Fe ₂ O ₃	0.28	0.28
FeO	1.76	1.76
MgO	1.79	1.79
MnO	31.18	35.0
ZnO	10.28	7.0
CaO	8.22	8.22
TiO ₂	0.0033	0.0033
Na ₂ O	-	-
K ₂ O	-	-
BaO	0.0112	0.0112
BeO	0.0008	0.0008
Cr ₂ O ₃	-	-
CuO	0.0004	0.0004
PbO	0.0032	0.0032
V ₂ O ₅	0.0125	0.0125
Y ₂ O ₃	0.0038	0.0038
ZrO ₂	0.0027	0.0027
YbO	0.00033	0.00033
Total	100.45	100.988

Si	4.958	4.930
Al ^{IV}	0.035	0.035
Fe ⁺³	0.022	0.022
Fe ⁺²	0.156	0.156
Mg	0.284	0.282
Mn	2.809	3.135
Zn	0.807	0.547
Ca	0.937	0.931
Ti	0.0003	0.0003
Na	-	-
K	-	-
Ba	0.0005	0.0005
Be	0.0002	0.0002
Cr	-	-
Cu	0.0000	0.0000
Pb	0.0001	0.0001
V	0.0009	0.0009
Y	0.0002	0.0002
Zr	0.0001	0.0001
Yb	-	-
sum cations	10.011	10.040
sum anions	15.0	15.0

Element	Si	Fe	Mg
S.R.	1.4	2.6	2.7
#Pts.	20	20	20
#Grains.	20	20	20

Ca
1.1
20
20

Mn
2.3
18
18

Zn
2.4
20
20

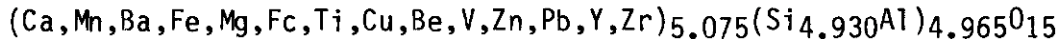
Mineral: Bald Knob Rhodonite

Mineral analysis: (1) Calif. Inst. Tech., 20-APR-71, microprobe analysis, A.L. Albee, analyst (2) Univ. of Wisconsin, 27-AUG-76; av. of 3 microprobe analyses, J.T. Cheney and D.J. Henry, analysts (3) U.S.N.M., microprobe analyses, G. Jarosewich, analyst (4) Univ. Michigan, 14-JUN-77, microprobe analysis, G. Winter, analyst (5) U.S.G.S. microprobe, MAGIC; 20 kV.; Wiggins, analyst.

	Oxide wt.%				
	(1)	(2)	(3)	(4)	(5)
SiO ₂	46.85	47.00	46.88	45.68	45.72±0.90
Al ₂ O ₃	0.00	-	-	-	0.05±0.02
Fe ₂ O ₃	-	-	-	-	-
FeO	1.68	1.71	1.59	1.63	1.69±0.25
MgO	1.37	1.25	1.21	1.22	1.18±0.19
MnO	32.03	34.67	34.70	34.88	35.48±1.65
ZnO	7.74	7.03	6.59	7.20	7.07±0.48
CaO	8.09	7.93	7.78	7.55	8.10±0.10
TiO ₂	0.00	-	-	-	-
Na ₂ O	0.00	-	-	-	-
K ₂ O	0.00	-	0.16	-	-
BaO	0.00	-	-	-	-
BeO	-	-	-	-	-
Cr ₂ O ₃	0.00	-	-	-	-
CuO	-	-	-	-	-
PbO	-	-	-	-	-
V ₂ O ₅	-	-	-	-	-
Y ₂ O ₃	-	-	-	-	-
ZrO ₂	-	-	-	-	-
YbO	-	-	-	-	-
Total	<u>97.76</u>	<u>99.59</u>	<u>98.91</u>	<u>98.16</u>	<u>99.29</u>

Si	5.068	5.023	5.038	4.975	4.921
Al	0.00	-	-	-	0.006
Fe ³⁺	-	-	-	-	0.151
Fe ²⁺	0.152	0.153	0.143	0.049	0.001
Mg	0.221	0.199	0.194	0.099	0.189
Mn	2.935	3.139	3.158	3.218	3.235
Zn	0.618	0.555	0.523	0.579	0.562
Ca	0.938	0.908	0.896	0.881	0.934
Ti	0.00	-	-	-	-
Na	0.00	-	-	-	-
K	0.00	-	-	-	-
Ba	0.00	-	0.022	-	-
Be	-	-	-	-	-
Cr	0.00	-	-	-	-
Cu	-	-	-	-	-
Pb	-	-	-	-	-
V	-	-	-	-	-
Y	-	-	-	-	-
Zr	-	-	-	-	-
Yb	-	-	-	-	-
cations	<u>9.932</u>	<u>9.977</u>	<u>9.974</u>	<u>10.000</u>	<u>10.000</u>
anions	15.000	15.000	15.000	15.000	15.0

Evaluation: The analysis of the rhodonite labelled "Bald Knob, North Carolina" illustrates the difficulty of analyzing for manganese in the presence of zinc. The original wet chemical (gravimetric) analysis reported 39.62% MnO but did not seek ZnO, which apparently co-precipitated with the manganese. Albee (personal communication dated April 20, 1971) first noted ZnO in the sample. Reanalysis by wet chemical methods produced revised values of 10.28% ZnO and 31.18% MnO. The ZnO value is higher, and the MnO value is lower, than in any of the five independent microprobe values. The preferred analysis substitutes average microprobe values, 7.0% and 35.0%, for ZnO and MnO, respectively. The weight percent sum is too high (101%) and the corresponding formula unit is



This pyroxenoid is truly homogeneous only for Si and Ca; other major elements (Mn, Zn, Fe, Mg) are less homogeneously distributed. During measurement of the sigma ratios, significant Na counts were noted; future analyses should include Na₂O. The composition of PXBK is not sufficiently well known for its use as a microprobe standard for all constituents. Nevertheless, this rhodonite is unique as a silicate standard for zinc in minerals such as staurolite. Until a better standard for Zn in silicates becomes available, use of PXBK must suffice.

Note: The original separate is probably mislabelled; a more probable locality is Franklin or Sterling Hill, New Jersey.

Date	From	To	Address
01-DEC-69	Huebner	A.E. Bence	Dept. of Earth and Space Sciences State Univ. New York at Stony Brook Stony Brook, New York 11790
30-NOV-73	Huebner	Eric Essene	Dept. of Geology and Mineralogy University of Michigan Ann Arbor, Michigan 48104
05-FEB-74	Huebner	Thomas Foster	Dept. Earth & Planetary Sciences Latrobe Hall The Johns Hopkins University Baltimore, MD 21204
13-NOV-80	Huebner	Lester Hughes	CONICO, Inc. 244 Research Bldg. P.O. 1267 Ponca City, OK 74601
22-JAN-73	Huebner	Brian Mason	Mineral Sciences Museum of Natural History Smithsonian Institution Washington, D.C. 20560
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5

17-JAN-75	Huebner	Peter Robinson	Department of Geology University of Massachusetts Amherst, Massachusetts 01002
13-NOV-80	Huebner	Dan Schulze	Programs in Geosciences- Station F0.2.1 The University of Texas at Dallas Box 688 Richardson, TX 75080
22-NOV-72	Huebner	W.E. Trzcienski, Jr.	Department of Geology Ecole Polytechnic 2500 Avenue Marie Guyard Montreal 250 Quebec, Canada
08-SEP-78	Huebner	V.J. Wall	Department of Earth Sciences Monash University Clayton, Victoria Australia 3168
20-FEB-69	Huebner	Paul Weiblen	Dept. of Geology Univ. of Minnesota Minneapolis, Minnesota 55455

Standard: Enstatite
Locality: Synthetic MgSiO_3
Donor: J. Ito, University of Chicago
Reference: Ito, J. (1975) High temperature solvent growth of orthoenstatite,
 MgSiO_3 in air. Geophys. Res. Letters 2, 533-536.

Oxide wt.% (theoretical)

SiO ₂	59.85
MgO	40.15
Total	<u>100.00</u>

Element	Si	Mg
S.R.	1.0	1.7
#Pts.	20	20
#Grains	2	2

Evaluation: The synthetic orthoenstatite has not been analyzed chemically, but crystallographically it is Mg orthopyroxene. When annealed at ~1000°C, these crystals develop a very pale blue color, perhaps due to trace amounts of Li, Mo, and/or V from the flux. The Si homogeneity is excellent, and the Mg homogeneity is good. This coarsely crystalline orthopyroxene should be a good standard for Si in pyroxenes and other silicates.

Standard: Hedenbergite (M12330)

Locality: Biwabik Iron Formation, Dunka River area, Minnesota

Donor: Bill Bonnicksen (currently Idaho Geological Survey) to P. Weiblen
 (Univ. Minnesota) to J.S. Huebner

Reference: (1) Bonnicksen, B. (1969) Metamorphic pyroxenes and amphiboles in the
 Biwabik Iron formation, Dunka River area, Minnesota. Mineral. Soc. Amer.
 Spec. Pap. 2, 217-239, sample 330; L.E. Reichen, USGS, analyst. wet chem.
 (2) Analysis #1 with H₂O deleted; elemental C deleted; P₂O₅ removed as
 apatite "(CaO)₅(PO₄)₃"; CO₂ removed as CaCO₃

	Oxide wt.% (1)	Oxide wt.% (2)
SiO ₂	49.20	49.20
Al ₂ O ₃	0.81	0.81
FeO	21.53	21.53
Fe ₂ O ₃	1.50	1.50
MgO	3.50	3.50
MnO	2.30	2.30
CaO	21.02	20.88
Na ₂ O	0.28	0.28
K ₂ O	<0.0005	-
TiO ₂	0.00	-
H ₂ O ⁺	0.25	*
H ₂ O ⁻	0.10	*
P ₂ O ₅	0.06	-
CO ₂	0.05	-
C	0.07	-
S	<0.01	-
Total	<u>100.67</u>	<u>100.00</u>

Si	1.971	1.977
Al	0.038	0.038
Fe ²⁺	0.721	0.723
Fe ³⁺	0.045	0.045
Mg	0.209	0.210
Mn	0.078	0.078
Ca	0.902	0.899
Na	0.022	0.022
P	0.002	-
Ti	0.000	-
C	0.003	-
sum cations	<u>3.992</u>	<u>3.992</u>
sum anions	6.000	6.000

Element	Si	Fe	Mg	Ca	Mn
S.R.	1.2	1.9	1.1		
#Pts.	20	20	20		
#Grains	20	20	20		
				2.2	
				20	
				20	
					2.2
					20
					20

Mineral: Hedenbergite (M-12330)

Microprobe analysis: U.S.G.S. ARL/EMX microprobe; L.B. Wiggins, analyst.

(1) analysis of one grain (2) average of three grains

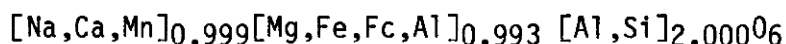
	(1)	oxide wt.% $\pm 1\sigma$	(2)
SiO ₂	49.14 \pm 0.56		49.01 \pm 0.22
Al ₂ O ₃	0.41 \pm 0.01		0.33 \pm 0.08
FeO	22.94 \pm 0.30		22.97 \pm 0.28
MgO	3.72 \pm 0.08		3.49 \pm 0.21
MnO	2.39 \pm 0.05		2.40 \pm 0.03
CaO	20.68 \pm 0.60		20.62 \pm 0.17
Na ₂ O	0.30 \pm 0.02		0.28 \pm 0.02
K ₂ O	0.03 \pm 0.02		- \pm 0.02
	<u>99.61</u>		<u>99.10</u>

Si	1.982	1.991
Al	0.019	0.016
Fe ²⁺	0.733	0.755
Fe ³⁺	0.041	0.025
Mg	0.224	0.211
Mn	0.082	0.083
Ca	0.894	0.897
Na	0.023	0.022
K	0.002	0.000
cations	<u>4.000</u>	<u>4.000</u>
anions	6.000	6.000

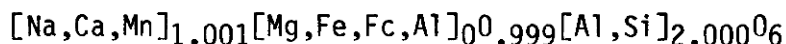
Standards:

- FSBO - K
- FSTA - Al, Na
- OLMJ - Mg, Si, and Fe
- OLST - Mn
- PXWO - Ca

Evaluation: The conventional mineral analysis has a high sum (100.67%) and a good cation/anion ratio that cannot be improved by adjusting the redox states of iron and manganese. Removal of carbon, of CO₂ as CaCO₃, and P₂O₅ as apatite results in the formula:



A slight adjustment of the proportions of ferric iron results in the almost ideal stoichiometric formula:



Independent microprobe analyses yield values of SiO₂, Fe as FeO, MgO, MnO, and Na₂O that are close to the values determined by wet chemistry. The microprobe value for Al₂O₃, however, is half the wet chemical value. No heterogeneities have been noted when using PXHD as a standard or known-unknown. The homogeneity of this phase determined by microprobe gave excellent sigma ratio values for Si and Mg, and adequate values for Fe and Ca. PXHD appears to be a good standard for hedenbergitic pyroxenes.

Date	From	To	Addresses
19-AUG-74	Huebner	Barry B. Hanan	Virginia Polytechnic Inst. & State University Blacksburg, VA

Standard: Pyroxmangite
 Locality: Homedale, Idaho
 Donor: J.S. Huebner

References: (1) U.S.G.S. analytical laboratories report no. 68-WO-9; J. Fahey, analyst. Classical wet chemistry. 1968. (2) U.S.G.S. spectrographic report no. 71-WS-55; J.L. Harris, analyst. 1971.

	Oxide wt. %	
	(1)	(2)
SiO ₂	47.84	>21.
MnO	26.75	>13.
FeO	19.31	>13.
Fe ₂ O ₃	1.31	-
Al ₂ O ₃	1.56	<0.004
CaO	2.06	1.0
MgO	1.42	1.7
TiO ₂	0.00	-
BaO	-	0.0002
BeO	-	0.0014
La ₂ O ₃	-	0.012
Nb ₂ O ₅	-	0.004
NiO	-	0.004
Sc ₂ O ₃	-	0.230
V ₂ O ₅	-	0.009
Y ₂ O ₃	-	0.089
Ce ₂ O ₃	-	0.058
YbO	-	0.008
Total	<u>100.25</u>	

Si	7.002
Mn	3.316
Fe ²⁺	2.364
Fe ³⁺	0.144
Al	0.269
Ca	0.323
Mg	0.310
Ba	0.0000
Be	0.0005
La	0.0007
Nb	0.0003
Ni	0.0005
Sc	0.0298
V	0.0009
Y	0.007
Ce	0.003
sum cations	<u>13.770</u>
sum anions	21.000

Element	Si	Fe	Mg	Mn	Ca
S.R.	0.8	32.0	2.0		1.4
#Pts.	17	17	17		18
#Grains	6	6	6		7
				7.7	
				17	
				7	

Mineral: Pyroxmangite

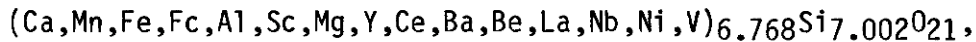
Microprobe analysis: U.S.G.S. ARL/EMX microprobe; L.B. Wiggins, analyst;
 average of 12 points on 4 grains.

Oxide wt.% $\pm 1\sigma$

SiO ₂	46.70+0.92
MnO	28.63+1.16
FeO	20.88+1.79
Al ₂ O ₃	0.07+0.02
CaO	2.09+0.06
MgO	1.89+0.08
TiO ₂	0.03+0.02
Cr ₂ O ₃	0.02+0.01
Total	<u>100.31</u>

Si	1.996
Mn	1.036
Fe ²⁺	0.744
Fe ³⁺	0.002
Al	0.004
Ca	0.096
Mg	0.120
Ti	0.001
Cr	0.001
cations	<u>4.000</u>
anions	6.000

Evaluation: The sum of the combined wet chemical and spectrographic results for the pyroxmangite from Homedale, Idaho, is 100.66%, several tenths of a percent greater than an acceptable value. Wiggins' independent microprobe analysis (standards not known with certainty, but probably the same standards that he used in analyzing PXBH) resulted in slightly higher total iron (as FeO) and MnO, and slightly lower silica. The Al₂O₃ values differed greatly, however; the wet chemical value was 1.56%, whereas the microprobe value was 0.07% and the emission spec showed Al to be present at <0.004 wt.%. The mineral formula corresponding to the wet chemical analysis,



is unacceptable for a pyroxmangite. Pyroxmangite analyses compiled by Deer et al. (1978) contain little or no Al₂O₃. Removal of Al₂O₃ as spessartite (Mn₃Al₂(SiO₄)₃) results in an even less satisfactory cation/anion ratio, 13.752/21.000, and the number of Si cations, 7.140, significantly exceeds the 7 sites available, an impossible situation. Measurement of the homogeneity of PXHI revealed two phases, pyroxenoid and olivine, but no aluminous phase. The pyroxmangite itself is not homogeneous with respect to Mn and Fe. Because of the apparent error in the wet chemical analysis and the heterogeneity, PXHI should not be used as a microprobe standard.

Date	From	To	Address
01-DEC-69	Huebner	A.E. Bence	Dept. of Earth and Space Sciences State Univ. New York at Stony Brook Stony Brook, New York 11790
08-MAY-72	Huebner	Bill Bonnichsen	Department of Geological Sciences Kimbell Hall - Cornell University Ithaca, N.Y. 14858
30-NOV-73	Huebner	Eric Essene	Dept. of Geology and Mineralogy University of Michigan Ann Arbor, Michigan 48104
01-DEC-69	Huebner	Louis A. Fernandez	Dept. Geology and Geophysics Box 2161, Yale Station New Haven, Connecticut 06520
30-APR-70	Huebner	Bevan French	Planetology Branch-NASA Goddard Spaceflight Center Greenbelt, Maryland 20771
30-APR-70	Huebner	Edward Ghent	Faculty of Arts and Science Department of Geology The University of Calgary Calgary 44, Alberta, Canada
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5

22-JAN-73	Huebner	Brian Mason	Mineral Sciences Museum of Natural History Smithsonian Institution Washington, D.C. 20560
13-MAR-72	Huebner	Peter Robinson	Dept. of Geology Univ. of Massachusetts Amherst, Mass. 01002
08-SEP-78	Huebner	V.J. Wall	Department of Earth Sciences Monash University Clayton, Victoria Australia 3168
20-FEB-69	Huebner	Paul Weiblen	Dept. of Geology Univ. of Minnesota Minneapolis, Minnesota 55455

Standard: Hypersthene

Locality:

Donor: Samuel S. Goldich (currently at U.S.G.S., Denver) to Paul Weiblen
(Univ. Minnesota) to Huebner

Reference: Sample identification no. R-2467; Gunderson

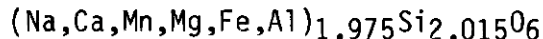
Oxide wt.%

SiO ₂	50.07
Al ₂ O ₃	0.19
FeO	38.17
MgO	10.09
MnO	0.47
CaO	0.89
Na ₂ O	0.28
Total	100.16

Si	2.015
Al	0.009
Fe	1.285
Mg	0.605
Mn	0.016
Ca	0.038
Na	0.022
sum cations	3.991
sum anions	6.000

Element	Si	Fe	Mg
S.R.	1.6	2.2	1.4
#Pts.	20	20	20
#Grains	15	15	15

Evaluation: According to P. Weiblen, this material was analyzed in the rock analysis laboratory of Samuel Goldich, then at the University of Minnesota. The oxide weight percent summation is excellent. Although the cation:anion ratio of 6:3.991 is close to the ideal 6:4 of stoichiometric pyroxene, there is an excess of Si cations in the tetrahedrally-coordinated site:



If this hypersthene is a stoichiometric pyroxene (no octahedral vacancies), the analysis has a SiO₂ value that is too high relative to cations with lesser valence. The problem cannot be the silica value alone because decreasing the silica value from 50.1 to 48.9% to give 2.000 tetrahedral cations per formula unit results in a weight percent sum of 99.1%, unacceptably low for an anhydrous silicate. More recently, Malcolm Ross has observed that the "FeO" value of PXHY is about 5% (relative) low, compared with other standards for FeO. Sigma ratios derived from a microprobe study of PXHY are acceptable: values of 1.6 for Si and 1.4 for Mg, and a slightly high value of 2.2 for Fe. Hypersthene PXHY should not be used as a standard for Si, Fe, or Mg, or as an unknown, until the analytical problem is resolved.

Date	From	To	Addresses
19-AUG-74	Huebner	Barry B. Hanan	Virginia Polytechnic Institute and State University Blacksburg, Virginia

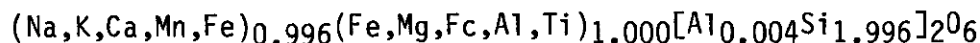
Standard: Jadeite
 Locality: Clear Creek, New Idria District, San Benito County, California.
 Franciscan formation
 Donor: J. Lockwood to B.A. Morgan
 References: R-1854; Coleman, R.G. and Clark, Joan R. (1968) Pyroxenes in the blueschist facies of California. *Am. Jour. Science*, 266, 43-59. Analyst, Eileen H. Oslund, Univ. Minn. Method, probably wet chemistry.

Oxide Wt. %

Na ₂ O	14.95
MgO	0.17
Al ₂ O ₃	24.62
SiO ₂	59.06
K ₂ O	0.01
CaO	0.35
TiO ₂	0.08
MnO	0.03
Fe ₂ O ₃	0.41
FeO	0.18
H ₂ O	0.10
Total	<u>99.96</u>
Na	0.980
Mg	0.009
Al	0.981
Si	1.996
K	0.0004
Ca	0.013
Ti	0.002
Mn	0.0009
Fe ³⁺	0.010
Fe ²⁺	0.005
sum cations	<u>3.996</u>
sum anions	6.0

Element	Si		
S.R.	1.6		
#Pts.	20	Al	Na
#Grains	19	2.3	2.7
		19	19
		19	19

Evaluation: The conventional chemical analyses has a superior sum, 99.96%.
 The formula unit, calculated using formal site occupancies, is reasonable



and has an excellent 3.996 cations per 6.000 anions. Despite the marginally adequate sigma ratios for the major elements, this separate is not homogeneous with respect to minor elements. While measuring the homogeneity index for Si, it was noted that four grains had significant count rates for both Fe and Mg. Similarly, low count rates of both Al and Na are associated with significant count rates for Ca. This behavior could be caused by the presence of augite lamellae. Use of PXJD as a standard should be avoided.

Standard: Hypersthene

Locality: Johnstown meteorite

Donor: E. Jarosewich (USNM #746)

Reference: (1) Mason, B. and E. Jarosewich (1971) The composition of the Johnstown Meteorite. Meteoritics 6, 241-245. H. Haramura, analyst.

(2) Jarosewich, E., et al. (1979) Electron microprobe reference samples for mineral analyses. Smithsonian Contributions to the Earth Sciences, 22, 68-72.

	Oxide wt.%	
	(1)	(2)
SiO ₂	53.63	54.09
Al ₂ O ₃	0.33	1.23
TiO ₂	0.21	0.16
Cr ₂ O ₃	0.81	0.75
FeO	15.66	15.22
MgO	27.23	26.79
MnO	0.50	0.49
CaO	1.39	1.52
Na ₂ O	0.13	<0.05
K ₂ O	-	<0.05
P ₂ O ₅	0.08	-
H ₂ O*	0.41	0.00
Total	<u>100.38</u>	<u>100.25</u>

	adjusted	adjusted
Si	1.937	1.950
Al	0.014	0.052
Ti	0.006	0.004
Cr ³⁺	0.023	0.021
Fe ²⁺	0.394	0.441
Fe ³⁺	0.079	0.018
Mg	1.466	1.440
Mn	0.015	0.015
Ca	0.054	0.059
Na	0.009	0.000
P	0.002	-
sum cations	<u>4.000</u>	<u>4.000</u>
sum anions	6.000	6.000

Element	Si	Fe	Mg
S.R.	1.3	2.4	2.0
#Pts.	20	20	20
#Grains	3	3	3

Mineral: Hypersthene
Microprobe analyses: U.S.G.S. SEMQ
J. McGee (U.S.G.S.) SEMQ, 5 points, 2-8-82

oxide wt.% $\pm 1\sigma$

SiO ₂	53.19 \pm 0.39
Al ₂ O ₃	0.96 \pm 0.09
TiO ₂	0.09 \pm 0.04
Cr ₂ O ₃	0.74 \pm 0.03
FeO	14.72 \pm 0.29
MgO	26.53 \pm 0.36
MnO	0.51 \pm 0.02
CaO	1.28 \pm 0.14
Na ₂ O	0.05 \pm 0.02
K ₂ O	0.03 \pm 0.01
Total	98.10 \pm 0.26

Standards:

FSTA - Na,Al

OLST - Mn

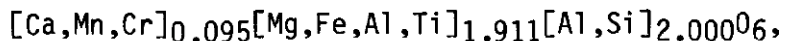
OXIL - Fe,Ti

OXTB - Cr

Or-1 orthoclase - K

PXAD - Mg,Ca,Si

Evaluation: Both conventional analyses of the hypersthene from the Johnstown meteorite have adequate sums, but the more recent analysis (2) has a significantly greater value for Al_2O_3 , 1.23%, than the earlier analysis (1), with only 0.33% Al_2O_3 by weight. Recalculation to a mineral formula provides evidence that the higher Al_2O_3 value (analysis #2) is more plausible than the lower value. The formal site occupancy resulting from analysis #2 has a tetrahedral site that is completely filled with Si+Al:



whereas the tetrahedral site resulting from analysis #1 can only be filled if cations other than Si+Al enter tetrahedral coordination, which in the case of PXJT is not likely. Although ferric iron was not specifically determined, only a minor amount (0.018 cations) of Fe^{3+} need be assumed in the formula recalculated from analysis #2 if that formula is to be made perfectly stoichiometric:



The hypersthene is very homogeneous with respect to Si and only slightly less homogeneous with respect to Mg and Fe. The homogeneity of Cr was not measured in this study nor in that of Jarosewich et al. (1979). Subsequently, Jarosewich (written communication, October 21, 1985) mentions that the Cr is "not very homogeneous." Independent microprobe analysis, using OXTB as a standard, confirms the Cr values of analyses #1 and #2. PXJT should be a good standard for calcium-poor pyroxenes and may serve to check standardizations for Cr_2O_3 in silicates (the Cr_2O_3 value of 0.8% is too small for optimum peak position measurement during standardization), providing a sufficient number of points is collected to overcome any possible Cr heterogeneity.

Date	From	To	Address
27-FEB-81	McGee	Dan Schulze	Department of Geosciences University of Texas at Dallas Richardson, TX 75080

Standard: Kakanui Augite

Locality: Kakanui, New Zealand

Donor: E. Jarosewich - U.S. Nat. Mus. #122142

References: 1) Mason, Brian (1966) N.Z. J. Geol. Geophys. 9, 474-80.

Presumably a wet-chemical method. 2) Trace element analyses by various methods, summarized in Mason, Brian and R.O. Allen (1973) New Zealand Jour. Geophys. 16, 935-947. 3) Jarosewich (personal communication, June 16, 1981). Revision of wet-chemical analysis #1, J. Norberg, analyst. 4) Preferred - incorporates revision of Norberg.

	Oxide wt.%			
	(1a)	(2)	(3)	(4) (Preferred)
SiO ₂	50.73		50.73	50.73
TiO ₂	0.74	0.734	0.74	0.74
Al ₂ O ₃	7.86		8.73	8.73
Fe ₂ O ₃	3.69		1.08	1.08
FeO	3.45		5.37	5.37
MnO	0.13	0.13	0.13	0.13
MgO	16.65		16.65	16.65
CaO	15.82		15.82	15.82
Na ₂ O	1.27		1.27	1.27
K ₂ O	0.00	0.011		
H ₂ O ⁺	0.04			
H ₂ O ⁻	0.00			
Sc ₂ O ₃		0.004		0.00
V ₂ O ₅		0.045		0.04
Cr ₂ O ₃		0.12		0.12
CoO		0.006		0.01
NiO		0.046		0.05
CuO		0.001		0.00
Zn		0.003		0.00
GaO		0.0014		0.00
SrO		0.007		0.01
Y ₂ O ₃		0.001		0.00
Total	<u>100.38</u>	ZrO ₂ 0.004	<u>100.52</u>	<u>100.75</u>
		BaO 0.0001		
Si	1.824	La ₂ O ₃ 0.0002	1.825	1.822
Ti	0.020	Ce ₂ O ₃ 0.0007	0.020	0.020
Al	0.333	Pr ₂ O ₃ 0.0002	0.370	0.370
Fe ²⁺	0.104	Nd ₂ O ₃ 0.0007	0.162	0.161
Fe ³⁺	0.100	Sm ₂ O ₃ 0.0002	0.029	0.029
Mn	0.004	Gd ₂ O ₃ 0.0003	0.004	0.004
Mg	0.892	Dy ₂ O ₃ 0.0003	0.893	0.892
Ca	0.609		0.610	0.609
Na	0.089		0.089	0.088
K	0.0005			0.000
Sc	0.000			0.000
V	0.001			0.009
Cr	0.003			0.003
Co	0.0003			0.0003
Ni	0.0014			0.0014
Cu	0.000			0.000
Zn	0.000			0.000
Ga	0.000			0.000
Sr	0.0002			0.0002
Y	0.000			0.000
sum cations	<u>3.981</u>		<u>4.000</u>	<u>4.000</u>
sum anions	6.000		6.000	6.000

Element	Si	Fe	Mg	Ca	Al	Na
S.R.	1.5	1.4	1.2			
#Pts.	20	20	20			
#Grains	6	6	6			
				1.3	1.4	1.1
				20	20	20
				10	10	10

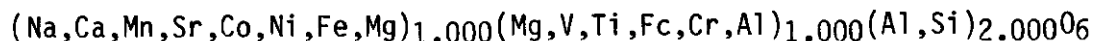
Mineral: Kakanui Augite
 Microprobe analysis: U.S.G.S. ARL-EMX microprobe; Wiggins, analyst; average of 18 points

Oxide wt.% $\pm 1\sigma$

SiO ₂	49.85+0.48
TiO ₂	0.84+0.05
Al ₂ O ₃	8.50+0.18
FeO	6.35+0.12
MnO	0.08+0.02
MgO	16.48+0.47
CaO	16.07+0.32
Na ₂ O	1.45+0.06
K ₂ O	-
Total	99.62

Si	1.805
Ti	0.023
Al	0.363
Fe ²⁺	0.109
Fe ³⁺	0.083
Mn	0.002
Mg	0.889
Ca	0.623
Na	0.102
cations	4.000
anions	6.000

Evaluation: The classical or wet chemical analyses of the Kakanui augite have sums that are slightly high, particularly when the trace elements are included. The preferred analysis (3) incorporates revised values of Al₂O₃, Fe₂O₃, and FeO. The revised Al₂O₃ and total iron are substantiated by Wiggins' microprobe values. The preferred analysis can be recalculated to a perfectly stoichiometric pyroxene without any adjustment of the ferrous/ferric ratio!



The augite from Kakanui is homogeneous with respect to all its major elements. PXXA should be an excellent standard and superior known-unknown for major elements in pyroxenes. It has not been used as much as it deserves.

Standard: Chrome Augite, PSU Px-1

Locality: Libby, Montana

Donor: A.L. Boettcher

References: S.S. Goldich, C.O. Ingamells, N.H. Suhr, and D.H. Anderson (1967)
 Analyses of silicate rock and mineral standards. *Can. J. Earth Sci.* 4, 747-
 756. C.O. Ingamells, analyst.

Oxide wt%

SiO ₂	53.94
Al ₂ O ₃	0.66
TiO ₂	0.26
Cr ₂ O ₃	0.21
Fe ₂ O ₃	1.13
FeO	1.91
MnO	0.07
MgO	16.93
CaO	24.55
SrO	0.035
BaO	0.006
Na ₂ O	0.24
K ₂ O	<0.01
Rb ₂ O	0.00
P ₂ O ₅	0.00
BeO	0.00
H ₂ O ⁺	0.00
H ₂ O ⁻	0.03
F ⁻	0.00
Total	<u>99.97</u>

Si	1.968
Al	0.028
Ti	0.007
Cr	0.006
Fe ³⁺	0.031
Fe ²⁺	0.058
Mn	0.002
Mg	0.921
Ca	0.960
Sr	0.0007
Ba	0.0001
Na	0.017
K	0.000
Rb	0.000
P	0.000
Be	<u>0.000</u>
sum cations	4.000
sum anions	6.000

Element	Si	Fe	Mg	Ca
S.R.	1.2	3.2	3.5	
#Pts.	20	20	20	0.8
#Grains	18	18	18	20
				18
	1.3			0.8
	20			20
	20			20
	0.8	4.0	2.6	
	20	20	20	
	20	20	20	
		5.0	2.6	1.3
		20	20	20
		20	20	20

Mineral: Chrome Augite, PSU Px-1
 Microprobe Analysis: U.S.G.S. ARL-SEMQ microprobe; J. McGee, analyst;
 average of 6 points; 08-FEB-82

oxide wt% $\pm 1 \sigma$

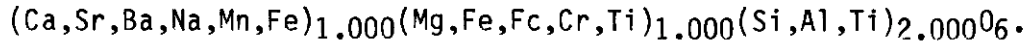
SiO ₂	52.85+0.14
Al ₂ O ₃	0.62+0.04
TiO ₂	0.26+0.04
Cr ₂ O ₃	0.13+0.08
FeO	2.76+0.15
MnO	0.09+0.01
MgO	16.74+0.24
CaO	24.97+0.15
Na ₂ O	0.25+0.06
K ₂ O	0.02+0.02
Total	98.69+0.20

Si	1.949
Al	0.027
Ti	0.007
Cr	0.004
Fe ²⁺	0.009
Fe ³⁺	0.076
Mn ²⁺	0.003
Mg	0.920
Ca	0.986
Na	0.018
K	0.001
cations	4.000
anions	6.000

Standards:

FSTA - Na,Al
 OLST - Mn
 OXIL - Fe,Ti
 OXTB - Cr
 Or-1 Orthoclase - K
 PXAD - Ca,Si,Mg

Evaluation: Ingamells' analysis of the chrome augite has an excellent sum, 99.97%. Using the microprobe and independent standards, J. McGee obtained values for Al, Ti, Fe, Mn, Mg, Cr, and Na that are very similar to Ingamells' values, but Si and Cr by microprobe were low. Omitting water and fluorine, the conventional analysis can be recalculated to a perfectly stoichiometric and reasonable pyroxene formula unit, without any adjustment of the ferrous/ferric ratio):



Homogeneity of the chrome augite is implied by its usage as a standard elsewhere. A microprobe homogeneity check of 20 points yielded excellent sigma ratios for Si and Ca, but unsatisfactory values for Fe and Mg. These large values for the homogeneity index are caused by the 10-30% of the grains measured in each session which, by inspection, have anomalous count rates. Count rates for Mg and Fe show a small tendency to be inversely related, indicating that the heterogeneity is due to a spatially variable Fe/Mg ratio. PXP1 is an excellent Si and Ca standard for diopsidic, aluminum-poor augite. If counts for 10 or more individually measured points are averaged during standardization; PXP1 could also be used as a standard for Mg and Fe.

Standard: Penn State University Diopside

Locality:

Donor: C.O. Ingamells

References: Analyzed by C.O. Ingamells for H.S. Yoder. PSU #63-1827

Oxide wt.%

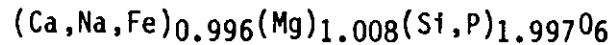
SiO ₂	55.36
Al ₂ O ₃	0.00
Fe ₂ O ₃	0.00
TiO ₂	0.01
FeO	0.09
MnO	0.005
MgO	18.77
CaO	25.70
SrO	0.00
BaO	0.00
Na ₂ O	0.02
K ₂ O	0.00
H ₂ O [±]	0.00
P ₂ O ₅	0.06
Total	<u>100.02</u>

Si	1.995
Al	0.000
Fe ³⁺	0.000
Ti	0.000
Fe ²⁺	0.003
Mn	0.000
Mg	1.008
Ca	0.992
Sr	0.00
Ba	0.00
Na	0.001
K	0.00
P	0.002
sum cations	<u>4.002</u>
sum anions	6.000

Element	Si	Mg
S.R.	1.3	1.1
#Pts.	20	20
#Grains	20	20

Ca
 1.1
 20
 20

Evaluation: The chemical analysis is presumed to be a conventional, wet-chemical mineral analysis because the analyst was Ingamells. The oxide weight-percent sum is excellent, 100.02%. The mineral formula calculated from this analysis,



has a cation:anion ratio of 4.002:6.000, close to the ideal value of 4:6. There is a slight deficiency in the tetrahedral site even if phosphorous is permitted to substitute for silicon, and a sufficiency of octahedral cations. (A similar but more serious discrepancy exists in the case of the diopside from the Adirondacks, PXAD.) However, this problem may be only apparent, caused by the uncertainty in the analytical method.

The homogeneity of PXPS is demonstrated by very low sigma ratio values for Ca, Mg, and Si as well as by its use as a microprobe standard elsewhere. The analysis may be as good as can be obtained by conventional methods. PXPS should be an excellent standard for Ca, Mg, and Si in pyroxenes and should serve as a known-unknown for diopsidic pyroxenes. Use of PXPS is preferable to the similar diopside PXAD.

Standard: Diopside
 Locality: Synthetic, crystallized from Di melt on water quenching of 25 ml
 crucible
 Donor: D.B. Stewart
 Reference:

Oxide wt.% (theoretical)

SiO ₂	55.49
MgO	18.62
CaO	25.89
Total	<u>100.00</u>

Element	Si	Mg	Ca	Ca
S.R.	1.4	4.4		
#Pts.	20	20		
#Grains	5	5	4.4	
			20	
	1.3	13.4	5	
	19	19		
	11	11	8.3	
			20	
	1.2	19.1	6	
	20	20		19.3
	8	8		20
			3.8	10
			20	
			7	5.7
				20
				5
	1.4	32.0	15.6	
	20	20	20	
	12	12	12	

Evaluation: Synthetic diopside PXSD is assumed to be stoichiometric CaMgSi₂O₆. Likewise, the glass remaining after the quench is assumed to have the same composition. Although crystalline material (white) was selected for the microprobe standard, some glass (colorless) is undoubtedly admixed. Homogeneity of Si is excellent, but sigma ratio values for Ca and Mg are unsatisfactory. Points with low count rates for magnesium have high count rates for calcium. (Is wollastonite present?) PXSD should not be used as a standard.

Date	From	To	Address
19-AUG-74	Huebner	B.B. Hanan	Virginia Polytechnic Institute and State University Blacksburg, Virginia
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5

Standard: MgSiO₃
Locality: Synthetic Enstatite
Donor: J.S. Huebner
Reference: Synthesis from oxide mix, run #45, 825°C, 1000 bars H₂O, 4779 hrs, 9/13/'68

Oxide Wt. % (theoretical)

SiO ₂	59.85
MgO	40.15
Total	<u>100.00</u>

Element	Si	Mg
S.R.	2.2	5.6
#Pts.	20	20
#Grains	18	18

Evaluation: PXSE is an orthopyroxene with cell dimensions of pure MgSiO₃ (Huebner, manuscript). Its composition is presumed to be stoichiometric MgSiO₃. The crystals are elongate but narrow (<20µm); care must be taken to be sure that the activation volume remains contained in the pyroxene (the bright cobalt-blue fluorescence is of assistance). The poor sigma ratio for Mg probably reflects the fine grain size, rather than a compositional heterogeneity.

Standard: Shallowater Enstatite
 Locality: Meteorite
 Donor: W. Melson to B.A. Morgan
 Reference:

Oxide Wt. %

MgO ₂	39.96
Al ₂ O ₃	0.13
SiO ₂	59.98
CaO	0.25
Total	<u>100.00</u>

Mg	1.985
Al	0.005
Si	1.999
Ca	0.009
sum cations	<u>3.998</u>
sum anions	6.0

Element	Si	Mg
S.R.	1.2	0.9
#Pts.	20	20
#Grains	12	12
	0.7	1.7
	20	20
	17	17

Evaluation: The analytical method is unknown. The weight percent total is perfect and may have been normalized, but the cation/anion ratio is excellent. Sigma ratios are good to excellent.

Standard: Wollastonite
 Locality: Mammoth Lakes, Mono Co., CA
 Donor: Ben Morgan
 Reference: 1) U.S.G.S. Analytical Laboratory Report No. 72-WO-3. 06-JAN-72;
 J. Fahey, Analyst. 2) U.S.G.S. Spectrographic Lab. Rep. 71-WS-132. 17-DEC-71;
 semi-quant spec.; J.L. Harris, Analyst. (3) composite analysis

	Oxide wt. %		
	(1)	(2)	(3)
SiO ₂	51.57	>21.	51.57
Al ₂ O ₃	0.00	-	0.00
Fe ₂ O ₃	0.09	-	0.09
FeO	0.25	0.3	0.25
MnO	0.04	0.19	0.04
Ti	0.00	-	0.00
CaO	47.29	>13.	47.29
MgO	0.19	0.3	0.19
H ₂ O	0.65	-	0.65
BaO	-	0.000	0.000
CuO	-	0.001	0.001
SrO	-	0.012	0.012
Y ₂ O ₃	-	0.002	0.002
YbO	-	0.000	0.000
Total	<u>100.08</u>		<u>100.095</u>
Si	2.004		2.003
Al	0.000		0.00
Fe ³⁺	0.003		0.003
Fe ²⁺	0.008		0.008
Mn ²⁺	0.001		0.001
Ti	0.000		0.00
Ca	1.968		1.968
Mg	0.011		0.011
Cu	-		0.000
Sr	-		0.0003
Y	-		0.0000
sum cations	<u>3.995</u>		<u>3.995</u>
sum anions	6.000		6.0

Element	Si	Ca
S.R.	1.4	1.8
#Pts.	19	20
#Grains	6	6
		1.3
		19
		8

Mineral: Wollastonite

Microprobe analysis: U.S.G.S. ARL/SEMQ microprobe; Bence and Albee reduction;
12 points on 5 grains; Kempa, analyst; 02-MAR-82.

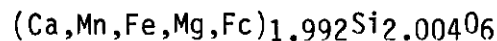
oxide wt.% $\pm 1\sigma$

MgO	0.62+0.02
SiO ₂	50.18+0.28
TiO ₂	0.01+0.02
Cr ₂ O ₃	0.00+0.01
Al ₂ O ₃	0.02+0.03
Na ₂ O	0.00+0.00
MnO	0.00+0.00
CaO	48.96+0.23
FeO	0.01+0.02
Total	99.81+0.40

Standards:

PXAD - Mg,Si
OXIL - Ti
OXTB - Cr
FSTA - Na,Al
OLST - Mn
PXW0 - Ca
OLSF - Fe

Evaluation: The sum of the conventional mineral analysis, 100.08%, is excellent. The analysis can be recalculated to a formula unit that is close to stoichiometric:



Microprobe analyses of 12 points suggests that the wollastonite is very homogeneous with respect to Si and Ca. The sigma ratios for Ca and Si, determined by microprobe, are good, but an exotic grain in the Reston mount must be avoided. PXW0 is a good standard for Ca and Si in oxides and calcic silicates.

Standard: As_2S_3
Locality: Synthetic
Donor: P. Toulmin III
References: Fused 15 min. @300°C in vacuo 15-Sept-1958; starting materials:
reagent elemental As + S. Total batch 13.698 gm.; "apparently homogeneous
deep red glass". Originally prepared as starting material for sulfosalt
synthesis.

Oxide Wt. % (theoretical)

As	60.91
S	39.09
Total	<u>100.00</u>

Element	As	S
S.R.	3.0	1.4
#Points	20	20
#Grains	6	6
	2.0	2.4
	20	20
	6	6

Evaluation: SAS2 is presumed to have the intended composition. When polished carefully, the measured sigma ratios are adequate. SAS2 should prove to be a suitable standard for qualitative measurements of the As levels in silicates and oxides.

Standard: CdS
Locality: Synthetic
Donor: P.M. Bethke
References: Synthesis from elements in evacuated silica tube, February, 1970.

Oxide Wt. % (theoretical)

Cd	77.81
S	22.19
Total	<u>100.00</u>

Element	Cd	S
S.R.	2.7	1.4
#Pts.	20	20
#Grains	20	20

Evaluation: The synthetic CdS appears homogeneous under the microscope. The value of 2.7 for the cadmium sigma ratio is undesirably large, perhaps due to the relatively small grain size of the material, 20-70 micrometers. Nevertheless, SCDS should prove to be a valuable standard, especially in cases where the cadmium content of the unknown is small.

Standard: Sb_2S_3
Locality: Synthetic
Donor: P. Toulmin III
References: synthesized from the elements in an evacuated silica tube, June,
1967.

Oxide Wt. % (theoretical)

S	28.31
Sb	71.69
Total	<u>100.00</u>

Element	S	Sb
S.R.	1.0	1.1
#Pts.	20	20
#Grains	8	8

Evaluation: The antimony sulfide is homogeneous with respect to its optical properties in incident light and the count rates for Sb and S x-rays in the microprobe. There is no reason to suspect that the composition is other than the theoretical composition given above. SSB2 should be an excellent standard.

Standard: SnS₂
Locality: Synthetic
Donor: P.M. Bethke
References: Prepared from stoichiometric mix of elements in evacuated silica glass tube.

Oxide Wt.%

Sn	64.92
S	<u>35.08</u>
Total	100.00

Element	S	Sn
S.R.	2.0	1.8
#Pts.	19	19
#Grains	19	19

Evaluation: No chemical analysis is available to verify that the intended composition was achieved. SSNS can only be presumed to be "on-composition." The sigma-ratio for sulfur is large; this large value may be due to the relatively poor polish achieved by standard techniques. If polished with great care, SSNS might prove to be a good standard for tin in sulfides (and oxides).

Standard: ZnS
Locality: Synthetic sphalerite
Donor: P.M. Bethke
References: original label on bottle indicates synthesis by L.B. Wiggins,
presumably from elements in an evacuated silica tube.

Oxide Wt. % (theoretical)

Zn	67.10
S	32.90
Total	<u>100.00</u>

Element	S	Zn
S.R.	1.9	2.3
#Pts.	20	20
#Grains	20	20
	1.9	2.4
	20	18
	19	19

Evaluation: The synthetic sphalerite is optically homogeneous (with internal reflections). The sigma ratios for Zn and S are not as good as might be expected for a simple, synthetic stoichiometric compound, perhaps due to the small grain size (the largest composite grain is <200 micrometers long). Nevertheless, SZNS should be a good standard for Zn and S.

Standard: Andalusite
 Locality: Espirito Santo, Brazil
 Donor: R.A. Robie 6/82 - Wards Natural Science Establishment
 References: (1) U.S.G.S. analysis W-212963; inductively coupled argon plasma;
 low-temperature heat capacity sample (Robie, pers. comm.). (2) Ideal formula,
 Al_2SiO_5

	Oxide wt.%	
	(1)	(2)
Al_2O_3	-	62.92
SiO_2	-	37.08
Fe_2O_3	0.36±0.05	-
MnO	<0.001	-
Cr_2O_3	<0.001	-
Total		100.00

Element	Si
S.R.	1.3
#Pts.	20
#Grains	5

Al
 1.7
 20
 9

Evaluation: There is no major element analysis of the andalusite from Brazil. Natural andalusites commonly contain several tenths of a percent of Fe_2O_3 , MgO , and CaO . Of these three constituents, only Fe_2O_3 has been determined in ANDB. Pending a direct determination of the MgO (and perhaps the Al_2O_3 and SiO_2), andalusite ANDB can be regarded only as a potential standard for Al and Si in aluminum-rich and silicon-rich minerals. Sigma ratios for Al and Si are acceptable. Each of the 5 grains analyzed is homogeneous with respect to iron, but one of the 5 grains gave higher count rates than the others, resulting in the poor sigma ratio for iron. Used with a kyanite and sillimanite, ANDB might also be of use in investigating the effects of aluminum coordination on the aluminum x-ray spectrum.

Standard: Kyanite

Locality: Wards Natural Science Establishment; Minas Gerais, Brazil

Donor: R.A. Robie, 6/82

References: (1) ICAP; U.S.G.S. analysis W-219962; low-temperature (5-380 K) heat capacity sample (Robie, pers. comm.). (2) Ideal formula, Al_2SiO_5 .

	Oxide wt.%	
	(1)	(2)
Al_2O_3	-	62.92
SiO_2	-	37.08
Fe_2O_3	0.18+0.05	-
Cr_2O_3	0.013	-
MnO	<0.001	-
Total		<u>100.00</u>

Element	Si	Al
S.R.	1.1	1.4

Evaluation: There is no major element analysis of the kyanite from Brazil. Natural kyanite commonly contains several tenths to a percent of Fe_2O_3 and may contain several tenths CaO . Of these, only Fe_2O_3 has been determined in KYMG. Pending a direct determination of CaO (or a demonstration that it is not present), Al_2O_3 , and SiO_2 , kyanite KYMG can only be regarded as a potential standard for Al and Si in aluminum-rich and silicon-rich minerals. KYMG is homogeneous. Used with andalusite and sillimanite, KYMG might also be of use in investigating the effects of aluminum coordination on the aluminum x-ray spectrum.

Standard: Kyanite S-121

Locality:

Donor: Pennsylvania State University

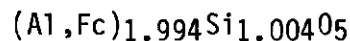
Reference: Analyst unknown. The following elements were sought spectrographically, but not found: Ni, Cu, Zr, Co, Y, Yb, Be, Sn, Zn, Cd, Pb, Sb.

	Oxide wt.%
SiO ₂	37.18
TiO ₂	<0.01
Al ₂ O ₃	62.19
Fe ₂ O ₃	0.67
CaO	<0.01
MgO	<0.01
H ₂ O	0.10
Cr ₂ O ₃	tr.
V ₂ O ₅	tr.
GaO	tr.
Total	<u>100.14</u>

	adjusted
Si	1.005
Al	1.981
Fe ³⁺	0.009
Fe ²⁺	<u>0.005</u>
sum cations	3.000
sum anions	5.000

Element	Si	
S.R.	1.0	
#Pts.	20	Al
#Grains	8	<u>1.2</u>
		20
		11

Evaluation: The chemical analysis of kyanite S-121 appears to be of high quality: the weight percent sum is excellent, the value determined for SiO₂, 37.18%, is very close to the ideal value of 37.08%, and the formula is almost stoichiometric kyanite:



The homogeneity of the kyanite is excellent. Kyanite KYPS is a possible standard for aluminosilicates; it could also be used with andalusite and sillimanite to investigate the relationship between Al coordination and the Al x-ray spectrum.

Standard: Nepheline

Locality: Bearpaw Mountains, Montana

Donor: W.T. Pecora to Carter Hearn to L.B. Wiggins (6/78)

References: Pecora, W.T., 1942, Am. Mineral., v. 27, p. 397-424. Wet chemistry by F.A. Gonyer, Harvard University on material hand-picked from a single crystal. Samples given to Fred Anderson, Bill Melson, and Harry Rose for probe standards; more sample available.

Oxide Wt. %

SiO ₂	41.52
TiO ₂	0.00
Al ₂ O ₃	34.09
Fe ₂ O ₃	0.79
CaO	0.06
Na ₂ O	15.76
K ₂ O	6.85
BaO	0.03
SrO	0.00
H ₂ O	0.55
Total	<u>99.65</u>

Si	4.008	4.053
Ti	0.000	0.000
Al	3.878	3.922
Fe ³⁺	0.057	0.058
Ca	0.006	0.006
Na	2.950	2.982
K	0.844	0.853
Ba	0.001	0.001
Sr	0.000	0.000
sum cations	<u>11.744</u>	<u>11.875</u>
sum anions	16.000	16.000

OH 0.355

Element	Si	Al	Na		
S.R.		1.5	5.2		
#Pts.		20	20	Na	K
#Grains		20	20	4.1	2.2
				20	20
		1.1	1.9	20	20
	3.8	19	19		
	20	19	19		
	20				
				1.7	2.9
				20	20
	1.5		3.2	20	20
	18		18		
	18		18		

Evaluation: The sum of the wet chemical analysis by Gonyer is slightly low, 99.65 weight percent. When compared with the ideal formula unit $(\text{Na,K})_4\text{Al}_4\text{Si}_4\text{O}_{16}$, the formula unit of NEPH clearly shows the substitution of Si for Al and the appearance of vacancies on the alkali sites:



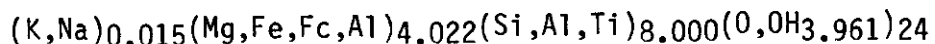
NEPH has 3% normative quartz component. The permissible range of omission solid solution, expressed in terms of normative quartz component in the system $\text{NaAlSi}_3\text{O}_8\text{-KAlSi}_3\text{O}_8\text{-SiO}_2$, is at least 0% to 12%. Thus, the formula unit cannot lend convincing support for the quality of the chemical analysis. The nepheline grains contain numerous small fluid inclusions, an observation that provides an explanation for the 0.55 weight percent H_2O in the analysis and the poor homogeneity. Count rate values, measured during the determination of the sigma ratios, revealed erratic values attributable to inclusions or contaminating grains. Even when the erratic values were removed from the lists of measurements, the resulting sigma ratios were not always acceptable. Because the x-ray count rates are heterogeneous and the analysis has not been verified, NEPH is not recommended as a microprobe standard.

Standard: Pyrophyllite
Locality: Staley, North Carolina
Donor: R.A. Robie

References: Robie, R.A., Hemingway, B.S., and Wilson, W.H. (1976) Jour.
Research U.S. Geol. Survey 4, no. 6, p. 631-644: (1) Report #75LACR0018,
Conventional rock analysis, V.C. Smith, analyst, U.S. Geol. Survey (1975).
(2) (W-179494) Rapid rock analysis by method of Shapiro (1967), Analyst,
H. Smith, U.S. Geol. Survey.

	Oxide wt.%	
	(1)	(2)
SiO ₂	66.32	66.4
Al ₂ O ₃	28.27	28.5
Fe ₂ O ₃	0.32	0.42
FeO	0.03	0.07
MgO	0.07	0.01
CaO	0.00	0.01
Na ₂ O	0.05	0.05
K ₂ O	0.02	0.05
H ₂ O ⁺	4.94	4.7
H ₂ O ⁻	0.00	0.13
TiO ₂	0.01	-
P ₂ O ₅	0.00	0.01
MnO	0.00	-
CO ₂	-	0.01
F	-	0.03
sub-Total	100.03	100.39
less O=F		0.02
Total		100.37
Si	7.972	7.967
Al	4.005	4.030
Fe ³⁺	0.029	0.038
Fe ²⁺	0.000	0.007
Mg	0.003	0.002
Ca	0.000	0.001
Na	0.012	0.012
K	0.003	0.008
Ti	0.001	-
P	-	0.001
C	-	0.002
sum cations	12.037	12.067
sum anions	24.000	24.0
F	-	0.011
OH	3.949	3.866
O	19.979	20.123
Element	Si	Al
S.R.	1.5	2.8
#Pts.	18	18
#Grains	8	8

Evaluation: The conventional mineral analysis (1) of the pyrophyllite from Staley, N.C., has a superior sum, 100.03%. The rapid rock analysis (2) confirms the values for SiO₂, Al₂O₃, and H₂O. The mineral formula calculated from analysis (1):



is nearly the ideal formula unit for pyrophyllite and has 3.961 hydroxyl, close to the ideal value of 4. The substitution of 0.027 aluminum for tetrahedral silicon causes a tetrahedral charge deficiency that must be compensated; this charge compensation is most likely brought about by interlayer Na and K (and perhaps Mg and Fe). Thus, the overall cation sufficiency (12.037 versus the ideal value of 12.000) is probably real. If care is taken to achieve a good polish, adequate sigma ratio values can be obtained for Si and Al. If the pyrophyllite is well-polished, it should be an excellent standard for alumino-silicates.

Date	From	To	Address
7/18/83	Huebner	Robert W. Smith	St. Joe Minerals Company P.O. Box 500 Virburnum, MD 65566

Standard: Scapolite (Meionite)

Locality: Brazil

Donor: U.S. Nat. Mus. R6600-1. Donated by E. Jarosewich.

Reference: Jarosewich, E. et al. (1979) Electron microprobe reference samples for mineral analyses. Smithsonian Contributions to the Earth Sciences, No. 22, 68-72.

Oxide wt.%

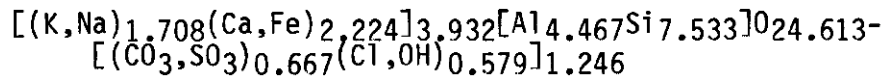
SiO ₂	49.78
Al ₂ O ₃	25.05
FeO	0.17
CaO	13.58
Na ₂ O	5.20
K ₂ O	0.94
H ₂ O	0.21
CO ₂	2.5
SO ₃	1.32
Cl	1.43
	<u>100.18</u>
-O = Cl	0.32
Total	<u>99.86</u>

Si	7.262	7.566
Al	4.307	4.487
Fe ⁺²	0.021	0.000
Fe ⁺³	-	0.022
Ca	2.123	2.211
Na	1.471	1.532
K	0.175	0.182
C	0.498	-
S	0.145	-
sum cations	<u>16.000</u>	<u>16.000</u>
sum anions	25.658	24.963

OH	0.204
Cl	0.354

Element	Si	Al	Na	Ca	Cl	S
S.R.	1.0					
#Pts.	20					
#Grains	14					
		1.0	1.2	1.0		
		20	20	20		
		20	20	20		
				0.9	1.1	
				20	20	
				20	20	
				1.0		0.8
				20		20
				20		20

Evaluation: The summation of the scapolite analysis is excellent. Scapolite has no single or ideal stoichiometry, but all components ideally have (Al+Si) = 12 cations per half unit cell. On this basis, the Brazilian scapolite has the formula



This scapolite composition lies midway between marialite, $(Na,K)_4Al_3Si_9O_{24}Cl$, and meionite, $(Ca,Fe)_4Al_6Si_6O_{24}(CO_3,SO_4)$. The homogeneity of SCMB is excellent. The scapolite SCMB should be a useful standard or known-unknown for chlorine or sulfur in silicates.

Standard: Sphene
 Locality: Hemet Quadrangle, California
 Donor: D. Gottfried
 References: U.S.G.S. Lab report #69-WO-11. Ellen Lillie, Analyst.

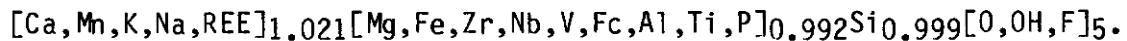
Oxide wt.%		Oxide wt.% (cont'd.)	
SiO ₂	30.54	P ₂ O ₅	0.45
Al ₂ O ₃	1.98	La ₂ O ₃	0.12
Fe ₂ O ₃	0.53	CO ₂	<0.10
TiO ₂	35.72	Nb ₂ O ₅	0.04
CaO	28.71	Ce ₂ O ₃	0.12
MgO	0.04	Nd ₂ O ₃	0.12
MnO	0.09	F	0.30
Na ₂ O	0.10	H ₂ O ⁺	0.21
K ₂ O	0.04	H ₂ O ⁻	0.07
FeO	0.30		<u>99.56</u>
V ₂ O ₅	0.04	-O=F	0.13
ZrO ₂	0.04	Total	<u>99.43</u>

Si	0.999
Al	0.076
Fe ³⁺	0.013
Ti	0.878
Ca	1.006
Mg	0.002
Mn ²⁺	0.0025
Na	0.006
K	0.0017
Fe ²⁺	0.008
V	0.0009
Zr	0.0006
P	0.013
La	0.0014
C	0.000
Nb	0.0006
Ce	0.0015
Nd	0.0014
sum cations	<u>3.012</u>
sum anions	5.000

F	0.0310
OH	0.0611
O	4.9079

Element	Si	Ca	Al	Ti
S.R.	1.3			
#Pts.	20			
#Grains	9			
		1.2	2.6	
		20	20	
		10	10	
				1.4
				20
				11

Evaluation: The chemical analysis of the Hemet sphene is adequate but not superior; the sum is half a weight percent low and the formula unit has a slight excess of cations:



Homogeneity of the major elements is indicated by the low sigma ratios measured by the microprobe. The distribution of the minor element Al is only marginally homogeneous. This sphene has been widely used as a standard for Ti in silicates for which SPHC appears well-suited. Unfortunately, the values for Nb, V, Zr, Ce, La, and Nd were determined by semi-quantitative spectrographic analysis, and D. Gottfried regards the Nb₂O₅ value as being too small. Were they to be redetermined by a quantitative method, SPHC might be a good known-unknown for trace element analyses of these elements by microprobe methods, providing these elements are homogeneously distributed.

Standard: Staurolite 355-1

Locality: Berkshire, MA

Donor: E-an Zen

Reference: 1) Zen, E-an (1981) Geol. Surv. Prof. Pap. 1113. Wet chem. anal.

Ellen Lillie, analyst. 2) same reference as 1; microprobe analysis. 3) same reference as 1; microprobe analysis with calculated value of ferric iron.

4) USGS Semiquantitative Spectrographic Analysis Rept. 63-WS-100. 1963.

H.W. Worthington, analyst.

	Oxide wt. %			
	(1)	(2)	(3)	(4)
SiO ₂	28.55	28.08	28.08	>10.
Al ₂ O ₃	53.54	54.59	54.59	>10.
TiO ₂	0.41	0.62	0.62	0.3
Fe ₂ O ₃	2.54	-	2.61	-
FeO	11.42	14.06	11.71	12.9
MgO	1.06	1.05	1.05	1.2
MnO	0.08	0.05	0.05	0.06
ZnO	0.26	0.35	0.35	0.4
CaO	0.09	0.00	0.00	0.03
K ₂ O	<0.05	-	-	0.0
H ₂ O ⁺	1.61	-	-	-
H ₂ O ⁻	0.02	-	-	-
P ₂ O ₅	0.03	-	-	0.0
CO ₂	<0.02	-	-	-
Total	99.61	98.80	99.06	Na ₂ O 0.01
				Ag ₂ O 0.00001
Si	4.064	4.023		B 0.007
Al ^{VI}	8.982	8.891		BaO 0.0003
Fe ³⁺	0.272	0.269		BeO 0.006
Fe ²⁺	1.359	1.346		CoO 0.013
Mg	0.225	0.223		Cr ₂ O ₃ 0.029
Mn	0.010	0.010		CaO 0.0006
Zn	0.027	0.027		GaO 0.009
Ca	0.014	0.014		Ge 0.003
P	0.004	0.004		Li ₂ O 0.15
Ti ⁴⁺	0.000	0.043		Mb ₂ O ₃ 0.0006
Ti ³⁺	0.044	-		NiO 0.01
sum cations	15.000	14.848		PbO 0.0002
sum anions	24.514	23.5		SrO 0.0004
				V ₂ O ₅ 0.02
OH	1.529			ZrO ₂ 0.03
Element	Si	Fe	Al	
S.R.	1.4	1.2		
#Pts	19	19		
#Grains	19	19	1.6	
			16	
	1.4	1.3	16	
	18	18		
	18	18		
		1.5	1.3	
		17	17	
		17	17	

Mineral: Staurolite

Microprobe analysis: (1) U.S.G.S. ARL/SEMQ microprobe. Bence-Albee reduction method; J. Hammarstrom, analyst; average of ten points; 04-MAR-82. (2) Univ. Oregon, ARL-EMX-SM. Average of 20 spots on 20 grains, Bence-Albee corrected. Michael Shaffer, analyst, 2/85.

	oxide wt.% $\pm 1\sigma$ (1)	oxide wt.% $\pm 1\sigma$ (2)
SiO ₂	27.98 \pm 0.36	28.0 \pm 0.4
CaO	0.01 \pm 0.01	
MgO	1.11 \pm 0.05	1.04 \pm 0.07
FeO	14.30 \pm 0.16	13.9 \pm 0.2
Na ₂ O	0.01 \pm 0.01	
Al ₂ O ₃	54.29 \pm 0.36	54.2 \pm 0.3
K ₂ O	0.02 \pm 0.02	
MnO	0.01 \pm 0.01	
TiO ₂	0.47 \pm 0.11	
Total	98.22 \pm 0.54	

Si	3.960
Al	9.054
Fe ²⁺	0.760
Fe ³⁺	0.933
Mg	0.234
Mn	0.001
Ca	0.002
Ti	0.050
Na	0.003
K	0.004
cations	15.000
anions	24.000

Standards:

PXAD - Si, Ca, Mg

GTKN - Fe

FSTA - Na, Al

PSU Orthoclase - K

OLST - Mn

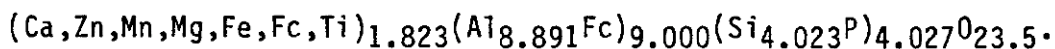
OXRU - Ti

MgAl₂O₄ - Mg

OLSW - Fe

Andalusite - Si, Al

Evaluation: The wet chemical analysis of the staurolite from Berkshire, MA, has a sum that is four tenths of a percent low, 99.61%. The two independent electron microprobe analyses give results that are similar to the wet chemical values, but with several small systematic differences: by microprobe, SiO₂ is 0.5% lower, Al₂O₃ is 0.8% higher, and total iron as FeO is 0.35-0.6% higher. Following Zen (1981), the wet chemical analysis was recalculated to an anhydrous formula unit with 23.5 anions [the equivalent ideal hydrous formula would have 1 hydroxyl and 23 oxygens (Lonker, 1983, Contributions Mineral. Petrol. 84, p. 36-42)]:



The fact that this formula deviates from the assumed ideal formula Fe₂Al₉Si₄O₂₃(OH) is not considered problematic because some staurolites may indeed be non-stoichiometric. The wet chemical value of H₂O, 1.6%, is greater than the expected value of 1.1%, but within the range actually found (Lonker, 1983). Either the staurolite structure accepts more water than is included in the ideal formula, or the analysis is in error (making even worse the low summation).

The staurolite within the separate is homogeneous, but the mineral separate is not, indicating the presence of other minerals. The close agreement between the wet chemical analysis and the microprobe analyses is surprising. Despite the uncertainties in the staurolite analysis and formula, there is no reason to exclude the staurolite from the list of potential standards or even known-unknowns for SiO₂, FeO, and Al₂O₃. STBM should be analyzed against other standards that are used for metamorphic minerals of similar composition in an effort to determine its usefulness as a standard for other elements. The contaminating grains must be avoided.

Date	From	To	Address
06-JUN-77	Huebner	Eric J. Essene	Dept. Geology and Mineralogy University of Michigan Ann Arbor, Mi 48104
11-MAR-77	Huebner	K.C. McTaggart	Dept. of Geological Sciences The University of British Columbia 2075 Wesbrook Place Vancouver, B.C., Canada V6T 1W5
09-OCT-84	Huebner	Michael Shaffer	Dept. Geology University of Oregon Eugene, OR 97403-1272

Standard: Fluor-Topaz

Locality: Topaz Mountain, Utah; D.A. Lindsey (1979) U.S.G.S. O-F Rept. 79-1076

Donor: H.T. Haselton (collected by Mark D. Barton)

References: (1) Barton, M.D., et al. (1982) Amer. Mineral. 67, 350-355.

(2) Penfield, S.L. and Minor, J.C., Jr. (1894) Am. Jour. Sci., 47, 387-396.

F by Berzelius method. (3) Ribbe, P.H., and Rosenberg, P.E. (1971) Amer. Mineral., 56, 1812-1821. Analysis by electron microprobe.

	Oxide wt.% (1)	Oxide wt.% (2)	Oxide wt.% (3)
Al ₂ O ₃	56.08	56.26	
SiO ₂	32.74	31.93	
F	20.3	20.37	20.4
H ₂ O	0.04	0.19	
Fe	-	-	0.04
Cr	-	-	trace
sub-total	<u>109.16</u>	<u>108.75</u>	
O=F	8.55	8.58	
Total	<u>100.61</u>	<u>100.17</u>	

Al	2.015	2.028
Si	<u>0.998</u>	<u>0.977</u>
sum cations	3.012	3.005
sum anions	6.000	6.000
F	1.957	1.970
OH	0.008	0.039

Element	Si
S.R.	1.5
#Pts.	20
#Grains	8

Al
 1.2
 20
 11

F
 2.0
 20
 20

Evaluation: The sums of the fluor-topaz analyses are good (#1) and excellent (#2). Replicate determinations of Al_2O_3 and F agree extremely well. For both analyses, the atomic ratio Al/Si is slightly greater than 2.000, the value expected in the ideal formula unit, $\text{Al}_2\text{SiO}_4(\text{F},\text{OH})_2$. Ideal stoichiometry would be achieved by increasing the analyzed value of SiO_2 , and decreasing the Al, by 0.5 to 1.0% absolute. Topaz TPTM is homogeneous and appears to be a good standard for fluorine. However, if the crystal-chemical environment of the fluorine in the unknown differs markedly from that in topaz, one might want to assess the effect of fluorine coordination on the fluorine X-ray spectrum before committing to this standard.

Standard: Tourmaline

Locality: San Luis Potosi, Mexico

Donor: Wm. Melson (USNM) to B.A. Morgan and J.S. Huebner

Reference: (1) Donnay et al. (1966) Buergerite, a new species of tourmaline.

Amer. Mineral. 51, 198-199. fusion and chemical analytical methods, C.O. Ingamells, analyst. (2) R. Barton, Jr. (1969) Refinement of the crystal structure of buergerite and the absolute orientation of tourmalines. Acta Cryst. (1969) B25, 1524-1533. 0.96% SiO₂ attributed to quartz impurity and removed, giving atomic ratio (Si+B):(O+OH+F)=27:93.

Oxide wt. % (1)		Oxide wt. % (1)		Oxide wt. % (2)	
B ₂ O ₃	10.86	Sr	<0.01	B ₂ O ₃	10.86
Na ₂ O	2.46	Ba	<0.01	Na ₂ O	2.46
MgO	0.13	Cr	<0.01	MgO	0.13
Al ₂ O ₃	30.79	Be	<0.01	Al ₂ O ₃	30.79
SiO ₂	33.86	Ni	<0.01	SiO ₂	32.90
K ₂ O	0.07	Co	<0.01	K ₂ O	0.07
CaO	0.69	V	<0.01	CaO	0.69
TiO ₂	0.55	Zn	<0.01	TiO ₂	0.55
MnO	0.13			MnO	0.13
Fe ₂ O ₃	17.62			Fe ₂ O ₃	17.62
FeO	1.27			FeO	1.27
H ₂ O	0.40			H ₂ O	0.40
F	1.86			F	1.86
	<u>100.69</u>				<u>99.73</u>
-O F	0.78				
Total	<u>99.91</u>				

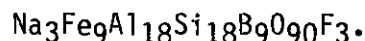
B	9.696	9.772		9.801	9.880
Na	2.467	2.486		2.494	2.514
Mg	0.100	0.101		0.101	0.102
Al	18.770	18.917		18.973	19.126
Si	17.514	17.651		17.201	17.341
K	0.046	0.046		0.047	0.047
Ca	0.382	0.385		0.386	0.390
Ti	0.214	0.216		0.216	0.218
Mn	0.057	0.057		0.058	0.058
Fe ³⁺	6.858	6.912		6.932	6.988
Fe ²⁺	0.549	0.554		0.555	0.560
sum cations	<u>56.655</u>	<u>57.099</u>		<u>56.764</u>	<u>57.224</u>
sum anions	93.0	91.5		93.0	91.5
OH	1.380			1.395	
F	3.043			3.076	

Element	Si	Fe	Na	Al	F
S.R.	2.2	2.5	1.4		
#Pts.	20	20	20		
#Grains	7	7	7		
				1.8	1.2
				20	20
				6	6

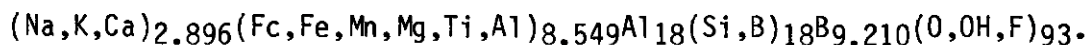
Mineral: San Luis tourmaline
 Microprobe analysis: U.S.G.S. ARL-SEM microprobe, 07-APR-83. J.F. Slack,
 analyst. each data set, average of 3 points.

	(1)	(2)
SiO ₂	34.26	33.31
Al ₂ O ₃	32.31	33.01
FeO	17.99	17.79
MgO	0.41	0.09
CaO	0.40	0.42
Na ₂ O	2.39	2.36
TiO ₂	0.62	0.79
Cr ₂ O ₃	0.07	0.08
MnO	0.22	0.22
Total	<u>88.67</u>	<u>88.06</u>

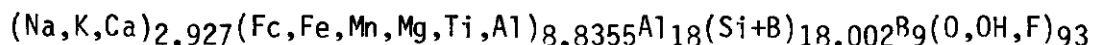
Evaluation: The analysis of the tourmaline by conventional methods has an excellent summation, despite the difficulty of the analytical procedure, which involved fusions. The tourmaline species is buergerite with an end member formula, based on 150 atoms per unit cell, of



The chemical analysis can be recalculated on this basis (Donnay et al., 1969) or, alternatively, on the basis of 93 anions, to give



This formula unit suffers from an excess of (B+Si) which, on crystal-structural grounds, should not exceed 27, even with vacancies present on the other sites. Neither adjustment of the proportion of Fe and Fc (on the assumption that the ferrous and ferric iron determinations were in error) nor recalculation on the basis of an anhydrous formula unit (on the assumption that the H₂O and F determinations might be in error) solves the problem of the excess (Si+B). Barton (1969) reports that the original specimen, analyzed by Ingamells, was intergrown with quartz. Assuming quartz contamination, Barton removed sufficient silica (0.96%) from the analysis to make (Si+B)=27.00 cations per 93 anions. The resulting formula unit,



is structurally reasonable.

The good to adequate homogeneity and large Fc/Fe make TSLP an appealing standard for tourmalines and ferric-iron-bearing silicates. However, the SiO₂ value is uncertain and, considering the difficulty in making the original analysis, other components may contain errors that are larger than expected. On the basis of the structure, Barton's (1969) adjusted analysis (2) is to be preferred.

Standard: Zoisite
 Locality: Puerto Cabello, Venezuela
 Donor: Ben Morgan
 Reference: U.S.G.S. analytical lab. rep. 71-WO-9 (1971); J. Fahey, analyst.
 Classical wet chemistry.

Oxide wt.%

SiO ₂	39.02
Al ₂ O ₃	32.87
Fe ₂ O ₃	1.42
FeO	0.33
TiO ₂	0.08
MnO	0.03
CaO	24.12
SrO	0.24
MgO	0.17
H ₂ O -100°C	0.00
H ₂ O +110°C1	2.09
Total	<u>100.37</u>

Si	2.960	2.967
Al	2.939	2.945
Fe ³⁺	0.081	0.081
Fe ²⁺	0.021	0.021
Ti	0.005	0.005
Mn	0.002	0.002
Ca	1.960	1.965
Sr	0.011	0.011
Mg	0.019	0.019
sum cations	<u>7.997</u>	<u>8.015</u>
sum anions	13.000	12.5

OH 1.058

Element	Si	Al	Ca
S.R.	1.6	1.8	0.9
#Pts.	20	20	20
#Grains	20	20	20
		1.9	3.4
		20	20
		19	19

Mineral: Zoisite

Microprobe analysis: 1) U.S.G.S. ARL/SEM-Q microprobe; Bence-Albee reduction method; average of ten points; 2) Magic reduction; average of six points; 04-MAR-82, J. Hammarstrom, analyst. (3) Univ. Oregon, ARL-EMX-SM, 5 μ M beam spot, average of 30 points on 3 grains. Michael Shaffer, analyst, 2/85.

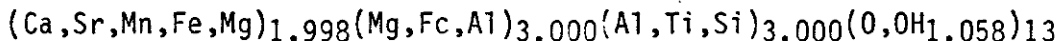
	oxide wt.% 1 σ (1)	oxide wt.% 1 σ (2)	oxide wt.% 1 σ (3)
SiO ₂	40.04 \pm 0.41	38.74 \pm 0.23	39.39 \pm 0.43
CaO	24.28 \pm 0.14	25.64 \pm 0.15	24.04 \pm 0.15
MgO	0.23 \pm 0.03	0.60 \pm 0.03	<0.03
FeO	1.52 \pm 0.45	2.05 \pm 0.39	1.72 \pm 0.36
Na ₂ O	0.01 \pm 0.01	0.02 \pm 0.01	
Al ₂ O ₃	32.24 \pm 0.33	31.73 \pm 0.17	32.52 \pm 0.44
K ₂ O	0.04 \pm 0.01	0.03 \pm 0.02	
MnO	0.00 \pm 0.00	0.03 \pm 0.02	
TiO ₂	0.06 \pm 0.02	0.05 \pm 0.03	
Total	98.43 \pm 0.54	98.88 \pm 0.42	

Si	3.028	2.941
Ca	1.967	2.085
Mg	0.026	0.068
Fe ²⁺	0.028	0.129
Fe ³⁺	0.068	0.130
Na	0.002	0.002
Al	2.874	2.839
K	0.004	0.003
Mn ²⁺	0.000	0.002
Ti	0.003	0.003
cations	8.000	8.073
anions	12.500	12.500

Standards:

PXAD - Si, Ca, Mg
 GTKN - Fe
 FSTA - Na, Al
 PSU Orthoclase - K
 OLST - Mn
 OXRU - Ti

Evaluation: The sum of the conventional analysis is slightly high, 100.4%. The zoisite formula unit calculated from the conventional analysis is almost stoichiometric but has more than one hydroxyl:



Recalculation on the basis of an anhydrous formula unit or downward adjustment of the H₂O value to 1.97% (1.00 ion of OH) results in a slight excess of cations (8.015 per 12.5 anions). There is insufficient FeO in the analysis to achieve stoichiometry by oxidizing FeO to Fe₂O₃. Preliminary microprobe analyses (1) for SiO₂, Al₂O₃, and CaO agree within 2.6%, 1.9%, and 1.5%, respectively, of the amounts present in the conventional analysis. The material is homogeneous with respect to Si, Al, and Ca. Michael Shaffer of the University of Oregon reports (letter of February 26, 1985 to Huebner) that he found slight heterogeneity with respect to Fe, and one spot with anomalously high MgO (0.66 wt%). Perhaps there are inclusions or contaminating grains of clinozoisite in the separate. Overall, the available information for ZOPC is excellent. ZOPC has good potential as a standard for silicates with appreciable Ca and Al, such as epidote group minerals and feldspars.

Date	From	To	Address
09-OCT-84	J.S. Huebner	Michael Shaffer	Dept. Geology University of Oregon Eugene, OR 97403-1272