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# Mineral standards for electron microprobe analysis of oxygen

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## ABSTRACT

Modern electron microprobe instrumentation makes possible the precise analysis of O. However, the lack of standards for which O contents are measured has hindered accurate calibration of O on the microprobe and prohibited rigorous evaluation of the accuracy of O analyses. Samples of albite, almandine, biotite, corundum, dolomite, enstatite, forsterite, gahnite, hematite, kaersutite, muscovite, quartz, and sillimanite have been characterized for this purpose, including measurement of O content by fast neutron activation analysis. These standards are now available for use in calibrating the electron microprobe for analysis of O.

## INTRODUCTION

O is the most abundant element on our planet, yet it is one of the most difficult elements for geochemists and material scientists to measure. In recent years there has been great interest in the ability of the electron microprobe to obtain quantitative O analyses (e.g., Armstrong, 1988; Bastin and Heijligers, 1989; Goldstein et al., 1991). The development and application of the multilayer W-Si crystal (commonly known as LDE1) makes routine and precise microprobe analysis of O possible. These crystals offer high count rates and high peak-to-background ratios that yield excellent statistical precision in analysis of O and other light elements.

Accuracy, however, is not so much a function of the counting apparatus as it is strongly dependent on matrix correction procedures and the quality of the calibration. Traditional matrix corrections (BA and ZAF) are inaccurate for light elements such as O (Armstrong, 1988). A number of authors have considered these problems and are working on improvement of correction factors for O (Bastin and Heijligers, 1989).

A large number of standards are available to electron microscopists, yet few standards have been analyzed for O. To date studies of quantitative O analysis have used mineral standards whose O content is calculated assuming fixed stoichiometry, an assumption that may not always be valid. Cation nonstoichiometry in minerals has long been documented. Recent fast neutron activation data (Dyar et al., 1991) confirm the existence of O nonstoichiometry in a suite of metamorphic biotite from northwestern Maine. Stoichiometric biotite possesses 24 O atoms per formula unit (pfu), but the Maine biotite shows a range of nonstoichiometric O contents from over 24 to less than 22 O pfu. As additional studies of O stoichiometry are made, it might be expected that similar variation in O contents of other commonly occurring silicate minerals will be confirmed.

This project was initiated in an attempt to address the need for mineral standards with measured O contents. Fast neutron activation analysis (FNAA) was used to measure O contents precisely and accurately in a selected group of mineral standards. This technique has a number of advantages: samples may be powders or size-sorted mineral grains; as little as 30 mg of sample are required; the technique is nondestructive; and unlike conventional INAA techniques, the sample is not radioactive for long time periods after analysis and so may be used for further study. Complete compositional characterization of 13 mineral standards included O analysis by FNAA, compositional analysis by electron microprobe and X-ray fluorescence spectroscopy (XRF), analysis of $Fe^{3+}/\Sigma Fe$ by Mössbauer spectroscopy, and analysis of H by U extraction process for hydrous samples.

## SAMPLE SELECTION

Several criteria were used to select mineral standards for this project. All are naturally occurring minerals, and most have compositions that are representative of common rock-forming minerals. A range of compositions, O contents, and mineral structural types (oxides, carbonates, orthosilicates, tectosilicates, etc.) was chosen. To be suitable as standards, samples needed to be homogeneous, free from inclusions, and sufficiently abundant to be distributed. Most of the samples selected have been chemically characterized in previous studies, and some are already in use as electron microprobe standards. Elev-

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**TABLE 1.** Compositions of mineral standards

| | Quartz 122838 | Sillimanite 131013 | Kaersutite 131928 | Albite 131705 | Olivine 131929 | Gahnite 111989 | Corundum 126097 | Dolomite 105064 | Hematite 92649 |
|---|---|---|---|---|---|---|---|---|---|
| Si | 46.74 | 17.34 | 18.51 | 31.82 | 19.58 | | | | |
| Al | | 33.29 | 7.82 | 10.47 | | 29.28 | 52.92 | | |
| $Fe^{2+}$ | | | 0.56 | | 6.86 | 1.53 | | 0.06 | 2.03 |
| $Fe^{3+}$ | | | 8.63 | 0.06 | | | | | 67.03 |
| Mg | | | 7.49 | | 29.76 | | | 13.13 | |
| Mn | | | 0.09 | | 0.10 | 0.29 | | 0.02 | 0.01 |
| Ti | | | 3.11 | | | | | | 1.25 |
| Cr | | | 0.00 | | | | | | 0.02 |
| Ca | | | 7.45 | 0.17 | 0.02 | | | 21.75 | 0.04 |
| Na | | | 2.09 | 8.49 | 0.01 | | | | |
| K | | | 1.19 | 0.22 | | | | | |
| Ni | | | | | 0.34 | | | | |
| Zn | | | | | | 34.14 | | | |
| C | | | | | | | | 12.93 | |
| H | | | 0.01 | | | | | | |
| O (FNAA) | 53.55(0.07) | 49.75(0.35) | 42.87(0.83) | 49.83(0.87) | 44.80(0.30) | 35.20(0.57) | 47.10(0.69) | 51.85(0.49) | 30.20(0.29) |
| Total | 100.29 | 100.38 | 99.82 | 101.06 | 101.47 | 100.44 | 100.02 | 99.74 | 100.58 |
| O (calc) | 53.25 | 49.37 | 43.05 | 48.65 | 43.98 | 34.92 | 47.08 | 51.79 | 30.25 |
| Mössbauer $Fe^{3+}/Fe_{tot}$ | | | 0.93 | | | | | | 0.97 |

en of the 13 samples were provided by the Harvard Mineralogical Museum. The amphibole and olivine were provided by AVM from a suite of mantle xenoliths collected by R. G. Coleman and now deposited in the Department of Mineral Sciences, National Museum of Natural History (Smithsonian Institution). These 13 samples are described in Appendix 1.

## ANALYTICAL METHODS

Polished thin sections of each sample were made using cold-set epoxy, taking care to avoid heating during the mounting process. This was done to avoid any possibility of O diffusion in the samples. Samples were first examined using the JEOL 8600 Superprobe at the University of Houston. This microprobe is equipped with four spectrometers, one fitted with an Ovionics multilayer W-Si crystal (LDE1) for light element analysis and a Tracor Northern automation and matrix correction package. Analyses were conducted at 15 kV accelerating voltage, 20 nA beam current (Faraday cup), and focused beam, except that for albite a 10-$\mu$m diameter defocused beam was used to minimize Na diffusion. The ZAF matrix correction procedure was used for all quantitative analyses.

Homogeneity was tested by obtaining a series of line scans measuring peak counts for all elements, including O, with concentrations greater than 0.5 wt%. Two line scans of 30 points each were obtained on different areas of the sample. Each element was counted on two spectrometers simultaneously to allow detection of instrumental variations vs. sample heterogeneities. The exception to this was O, which was counted once on the one available LDE1 crystal. Ag coating (rather than C) was used on the dolomite, which allowed for high count rate and precision in the analysis of O. No degradation of the Ag-coated carbonate sample under a 20-nA beam current was observed. Samples were considered to be homogeneous if the standard deviation of the peak counts for each element was less than 1% of the average peak count value. As a further check on homogeneity, 20–30 quantitative analyses were performed on each sample using normal analytical procedures (60-s count times, ZAF matrix correction, O by difference). WDS scans were obtained to check for the presence of F, but none was detected in any sample. F interferes with the FNAA O analysis, and if present, its concentration must be measured in order to correct the measured FNAA O value.

Samples that proved to be homogeneous were crushed, sieved, and hand-picked to produce pure mineral separates. Mössbauer spectra were obtained on aliquots of Fe-bearing samples to determine their $Fe^{3+}/\Sigma Fe$ ratios. The Mössbauer spectra were measured in the Mineral Spectroscopy Laboratory at the University of Oregon using procedures described in Dyar et al. (1989) and McGuire et al. (1989). Relative errors on the determined $Fe^{3+}/\Sigma Fe$ ratios are about ±3% of total Fe.

H contents were measured on the amphibole, biotite, and muscovite samples using a procedure for volumetric measurement of $H_2O$ vapor extracted from silicates by a U reduction furnace. Measurements were made at the facilities at Southern Methodist University using procedures described in Holdaway et al. (1986) and Dyar et al. (1991). Errors are estimated to be less than 0.1 wt%, based on replicate analyses.

Cation contents of ten samples were analyzed by XRF at the GeoAnalytical Laboratory at Washington State University (analyst Diane Johnson). Quartz, gahnite, and dolomite had high-quality published analyses and were not analyzed by XRF. Samples were powdered in an aluminum oxide mortar and pestle. Because of the small quantity of material that could be spared for destructive analytical work, a quartz-dilution technique was used for the XRF analysis. Each of the samples was mixed in a 1:5 ratio with pure Brazilian quartz. These samples were then analyzed using an automated Rigaku 3370 spectrometer and conventional techniques. The results were then corrected for the quartz dilution. This correction

**Table 1**—*Continued*

| Almandine 112140 | Muscovite 112791 | Biotite 131627 | Enstatite 131709 |
|---|---|---|---|
| 17.95 | 20.90 | 16.05 | 27.06 |
| 11.91 | 19.05 | 7.85 | 0.98 |
| 19.78 | 1.35 | 15.28 | 0.10 |
| 0.83 | | 5.09 | |
| 5.03 | 0.26 | 3.68 | 24.12 |
| 1.69 | 0.04 | 1.00 | |
| 0.03 | 0.09 | 1.61 | |
| 0.03 | | | |
| 1.24 | | | 0.08 |
| 0.09 | 0.37 | 0.07 | |
| | 8.97 | 7.78 | |
| | | | |
| | | | |
| | | | |
| | 0.43 | 0.31 | |
| 41.94(0.39) | 50.15(0.17) | 42.37(0.76) | 49.33(0.21) |
| 100.52 | 101.61 | 101.09 | 101.67 |
| 41.87 | 47.78 | 39.54 | 47.64 |
| | | | |
| 0.04 | | 0.25 | |

procedure consisted of first recalculating Fe to FeO and $Fe_2O_3$ using the $Fe^{3+}/\Sigma Fe$ ratios obtained by Mössbauer spectroscopy. Analyses were then normalized to their anhydrous totals (100% minus analyzed $H_2O$) and corrected for the 1:5 quartz dilution using an analysis of the quartz obtained with the sample analyses. Most of the results were in good agreement (within 5%) with the microprobe analyses. In some cases, the dilution-correction calculation appears to have produced problems such as negative corrected $SiO_2$ contents for both hematite (−2.11 wt%) and corundum (−1.91 wt%) and $SiO_2$ contents for muscovite and biotite about 2 wt% higher than those obtained by the microprobe and predicted by mineral stoichiometry.

O contents were determined by FNAA at the Radioanalytical Services Laboratory at the University of Kentucky (analyst Bang-Fa Ni). Two to six 200-mg aliquots of each sample were submitted for FNAA analysis, depending on the amount of material available. Less than 2 h before analysis, samples were packed in polyethylene vials under dry $N_2$ gas. Samples were irradiated in a Kaman Nuclear A-711 neutron generator. An automated transfer system allowed extremely rapid transport of the samples from the neutron flux source to a pair of NaI(Tl) scintillation detectors. Each sample was measured three times to check for any increase in O content resulting from absorption of $H_2O$ through the polyethylene vial wall. None of our mineral standards was hygroscopic, and the three replicate analyses were averaged to yield the reported O contents. The standard deviations of the average analyses range from 0.1 to 0.9 wt% O. Detailed descriptions of this analytical technique have been published by Vogt et al. (1965) and Ehmann and Morgan (1970).

## RESULTS

Cation and O analyses for the mineral standards are shown in Table 1. Because O was measured, analyses are presented as weight percent of the element, rather than the conventional weight percent oxide. Both calculated and FNAA measured O values are given. Calculated values were derived by assuming perfect charge balance and using the measured H contents and $Fe^{3+}/\Sigma Fe$ values. Standard deviations are given in parentheses next to the FNAA O values.

Accepted analyses for the quartz, dolomite, gahnite, and albite were taken from previously published analyses. Frondel and Hurlbut (1955) used part of this quartz crystal to measure the atomic weight of Si. Their analyses of the quartz showed it to be nearly pure $SiO_2$, and the success of their experiment confirmed that it must be stoichiometric $SiO_2$. Garrels et al. (1960) published an analysis of the dolomite sample. Microprobe analyses agreed with their published composition, and an X-ray diffraction pattern collected on this sample confirmed that it is an end-member dolomite. The gahnite analysis published by Jarosewich et al. (1980) has been widely used, and our microprobe analyses were in excellent agreement with that analysis.

A large number of analyses have been published for Amelia albite. Waldbaum (1966, Table 2-3) provides a summary of the data. The XRF and microprobe analyses obtained in this study exhibit anomalously high $SiO_2$ (probe) or $Na_2O$ (XRF) compositions that rendered these analyses unsuitable for use. An average of the Amelia albite compositions listed by Waldbaum provides a stoichiometric composition that agrees with our XRF and microprobe results (Table 1). The measured O content of 49.83 wt% is slightly higher than the calculated value of 48.65 wt%.

The microprobe analyses, XRF analyses, and agreement between calculated and FNAA measured O contents indicate that the corundum is pure $Al_2O_3$ and the sillimanite is pure $Al_2SiO_5$. Repeated microprobe analysis yielded a stoichiometric Al/Si ratio of 2.0 for the sillimanite. The Al/Si ratio of 2.13 obtained by XRF most likely results from an error in the $SiO_2$ introduced by the quartz-dilution correction. Likewise, negative $SiO_2$ calculated for the corundum XRF analysis must have been caused by problems with the correction procedure.

The enstatite, garnet, and amphibole samples exhibited excellent agreement between XRF and microprobe analyses. For these samples, the XRF analysis is given in Table 1 as the accepted composition. Of these three samples, only the enstatite has a measured O content, 49.33 wt%, that differs from the calculated O value, 47.64 wt%.

Microprobe and XRF analyses were in agreement for the hematite sample, except for the presence of 1.2 wt% $Al_2O_3$ and a $SiO_2$ content of −1.94 wt% in the XRF analysis. Repeated WDS scans were performed with the microprobe to detect $Al_2O_3$ in the hematite, and none was observed. We attribute the XRF $Al_2O_3$ value either to contamination from the mortar during powdering or to error from the dilution correction. The Fe content in the microprobe analysis is too high (analytical total 103%) because of the unavailability of a suitable Fe standard.

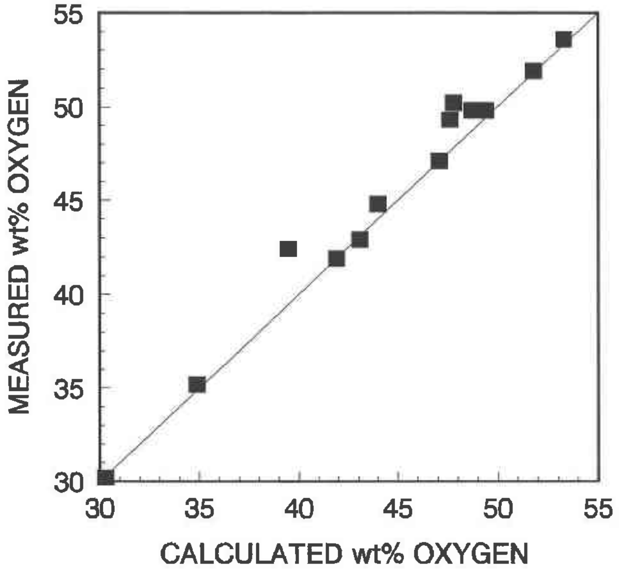

Fig. 1. Comparison of O wt% measured by FNAA vs. calculated O wt%. Only enstatite, muscovite, and biotite fall significantly off the 1:1 line.

Therefore, we give in Table 1 the XRF analysis with $SiO_2$ and $Al_2O_3$ set to 0.0 and the $Fe_2O_3$/FeO ratio calculated from the Mössbauer data. This analysis yields a stoichiometric hematite-ilmenite oxide ($Hem_{96}Ilm_4$) with the required $Fe^{2+}$ to charge balance the $Ti^{4+}$ and a calculated O content identical to that measured.

The remaining samples (olivine, muscovite, biotite) exhibited good agreement between the XRF and microprobe analyses for all cations except Si. The $SiO_2$ values determined by XRF are higher than those obtained by microprobe for muscovite and biotite and are lower for olivine. All three XRF analyses yield significantly nonstoichiometric mineral formulae. This may be caused by errors in the quartz-dilution correction of the XRF analysis, and for this reason, the microprobe analyses (averages of 20–30 points) are given in Table 1 as the accepted compositions of these three samples. The O contents of muscovite and biotite are several weight percent higher than those calculated.

## CONCLUSIONS

The majority of samples studied show stoichiometric O contents (Fig. 1). However, three out of the 13 have higher than expected O contents. This illustrates the danger in assuming that a natural mineral standard will have a stoichiometric O content. This study demonstrates that the FNAA technique can be used to characterize O contents of potential electron microprobe mineral standards. Microprobe analysis of O can, and should, be done using standards whose O contents are measured. If calculated O contents are to be used, it is clear that the $Fe^{3+}/\Sigma Fe$ ratios in Fe-bearing standards and H contents in hydrous mineral standards must also be measured in order to calculate O with any degree of accuracy.

Characterization of standard O contents will not solve all of the problems that plague O microprobe analysis. However, it is hoped that the standards will (1) aid studies of O matrix correction factors by providing materials of known O content with which to work, (2) improve O analyses of geologic materials being obtained by providing standards of similar bulk composition to unknown samples, thus minimizing matrix correction factor problems, and (3) provide some means of testing the accuracy of O analyses made using standards with calculated O contents.

## AVAILABILITY OF STANDARDS

These standards are available to the electron microanalysis community through Carl A. Francis, Curator, Harvard Mineralogical Museum, 24 Oxford Street, Cambridge, Massachusetts 02138, U.S.A.

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## Appendix 1. Sample descriptions

Descriptions of the 13 samples selected for characterization as microprobe standards for O are reported in the following format: species name, ideal formula, locality and geological occurrence, previous characterization, and Harvard Mineralogical Museum catalogue number.

**Albite.** $NaAlSi_3O_8$; white, platy intergrown crystals of the "cleavelandite" variety from the Rutherford no. 2 mine, which exploits a granitic pegmatite near Amelia Court House, Amelia County, Virginia (Sinkankas, 1968). Amelia albite is a widely used microprobe standard. No. 131705.

**Almandine.** $Fe_3Al_2(SiO_4)_3$; dark red crystal fragments, 5–10 mm across, from Green's Creek, Aston Township, Delaware County, Pennsylvania. According to Gordon (1922, p. 185) these occur in the soil above Peter's Mill dam and in the creek bed, presumably eroded from the metamorphic Wissahickon formation (Bascom et al., 1909). No. 112140.

**Biotite.** $K(Mg,Fe)_3(Si_3Al)O_{10}(OH,F)_2$; lustrous, black, pseudohexagonal cleavages, 2–3 cm across, in graphic granite from the wall zone of an unnamed granitic pegmatite formerly quarried for feldspar south of the Cathance River in Topsham, Sagadahoc County, Maine (Shanin, 1948). No. 131627.

**Corundum.** $Al_2O_3$; colorless, translucent, abraded crystal from Eheliyagoda, northwest of Ratnapura, Sabaragamuwa Province, Sri Lanka. Recovered from gem gravel derived from the Precambrian Highland Group of granulite grade metasediments (Katz, 1986). No. 126097.

**Dolomite.** $CaMg(CO_3)_2$; colorless, translucent, 3-cm rhombohedral crystal from the magnesite mines at Oberdorf on the Laming River in Styria, Austria. A wet chemical analysis by J. Ito is reported by Garrels et al. (1960), who determined its solubility and calculated its standard free energy of formation. No. 105064 (Harvard microprobe standard no. 259).

**Enstatite.** $MgSiO_3$; colorless crystal fragment, 1–2 cm, from the bank of the Chandrika Wewa reservoir near Embilipitiya, Sabaragamuwa Province, Sri Lanka. Composition and physical properties have been described by Harding et al. (1982), Zoysa (1985), and Zwaan (1986). No. 131709.

**Forsterite.** $Mg_2SiO_4$; green, translucent anhedral crystals from a spinel peridotite mantle xenolith, collected at Harrat al Kishb, Saudi Arabia, and characterized by McGuire (1988). No. 131929, which is part of USNM no. 116507.0063.

**Gahnite.** $ZnAl_2O_4$; dark green, translucent crystal from Brazil, presumably from a granitic pegmatite. Extensively characterized by Dymek et al. (unpublished; Harvard microprobe standard no. 437). Analyzed by Jarosewich et al. (1980) and widely distributed as a microprobe standard. No. 111989, of which USNM no. 145882 is a part.

**Hematite.** $Fe_2O_3$; black, 8 × 3.5 × 0.3-cm crystal from a hydrothermal deposit near Miguel Burnier, presumably akin to the famous precious topaz deposits in the Quadrilatero Ferrifero of Minas Gerais, Brazil (e.g., Olsen, 1971). No. 92649.

**Kaersutite.** $NaCa_2(Mg,Fe)_4Ti(Si_6Al_2)O_{22}(OH)_2$; black, 3 × 2 × 1-cm megacryst in alkali basalt at Harrat Hutaymah, Saudi Arabia. Included in a study of $Fe^{3+}$ and D/H values of mantle amphibole by Dyar et al. (1992; no. H366-92). No. 131928, which is part of USNM no. 116503.0032.

**Muscovite.** $KAl_2(Si_3Al)O_{10}(OH,F)_2$; euhedral crystal from granitic pegmatite at Blue Mountain, Methuen Township, Peterborough County, Ontario. The crystallography, composition, and physical properties have been described by Hurlbut (1956), and additional compositional data are reported by Eugster et al. (1972) and Robie et al. (1976). No. 112791.

**Quartz.** $SiO_2$; colorless crystal from fissure veins in folded Paleozoic sediments in the Ouachita Mountains near Hot Springs, Garland County, Arkansas (Engel, 1951). Physical properties and trace element chemistry meticulously determined by Frondel and Hurlbut (1955; no. C-105). No. 122838 (Harvard microprobe standard no. 409).

**Sillimanite.** $Al_2SiO_5$; pale blue, transparent, etched crystals from Okkampitya, Sabaragamuwa Province, Sri Lanka, from gem gravel derived from granulite grade metasediments. Similar crystals were described by Spencer (1920). Equivalent material supplied to Hemingway et al. (1991), who reported two independent analyses including trace elements. No. 131013.