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The composition of KLB-1 peridotite

Fred A. Davis,[1,*] Jean A. Tangeman,[2] Travis J. Tenner,[1] and Marc M. Hirschmann[1]

[1]Department of Geology and Geophysics, University of Minnesota, 108 Pillsbury Hall, Minneapolis, Minnesota 55455 U.S.A.
[2]Corporate Research Materials Laboratory, 3M Company, Building 201-4N-01 St. Paul, Minnesota 55144-1000, U.S.A.

Abstract

Electron microprobe analyses of major- and minor-element oxide components for two glassed samples of natural KLB-1 peridotite are presented. One glass was made with the aid of a phosphate flux, and the second glass was made by laser melting of aerodynamically levitated spheroids resulting in homogeneous silicate glass beads. For unknown reasons, the silicate-phosphate glass yields compositions that are incompatible with the composition of KLB-1 peridotite. However, analysis of the glass bead formed by laser synthesis is believed to give an accurate representation of the composition of KLB-1 peridotite, except for minor loss of Na_2O owing to volatilization. The new data resolve conflicting FeO, CaO, and TiO_2 values from two older measurements present in the literature. Mass-balance calculations using the new composition measurement combined with new analyses of the mineral compositions in KLB-1 result in a lower sum of squares of the residuals than those using the older measurements. There are appreciable differences in calculated modes from partial-melting experiments of KLB-1 when calculated using older KLB-1 analyses or our new analysis.

Keywords: Peridotite, KLB-1, microprobe, mantle, levitating/laser fusion

Introduction

Experimental investigation of the petrologic, geochemical, and geophysical properties of the deep Earth requires suitable analog starting compositions. Probably the most widely employed natural analog of the upper mantle is KLB-1, a spinel lherzolite xenolith from the Kilbourne Hole crater in New Mexico. Samples of KLB-1 have been used to represent the bulk mantle in many high-pressure experimental studies (Takahashi 1986; Herzberg et al. 1990; Agee and Walker 1993; Hirose and Kushiro 1993; Takahashi et al. 1993; McFarlane et al. 1994; Zhang and Herzberg 1994; Hirose and Kawamoto 1995; Herzberg and Zhang 1996; Hirose 1997a, 1997b; Konzett and Fei 2000; Wang and Takahashi 2000; Hirose 2002; Hirose and Fei 2002; Matsukage and Kubo 2003; Yoshino et al. 2004). For many other studies, starting materials were synthesized to approximate KLB-1 (Kawamoto et al. 1996; Kawamoto and Holloway 1997; Kogiso et al. 1998; Trønnes and Frost 2002; Ono et al. 2005; Mibe et al. 2006; Dasgupta and Hirschmann 2006, 2007; Dasgupta et al. 2007). Owing to these many studies, the composition of KLB-1 has become quite influential, and many theoretical treatments commonly assume that KLB-1 is an analogue for the composition of the upper mantle (Iwamori et al. 1995; Arndt et al. 1997; Moore et al. 1998, 1999; Ogawa and Nakamura 1998; Katz et al. 2003; Komiya 2004; Dobretsov et al. 2006). However, the composition of KLB-1 is known from only two analyses (Takahashi 1986; Herzberg et al. 1990), and these are not in complete agreement. Consequently, we have undertaken a new study of the composition of KLB-1 peridotite.

Experimental petrologists and others interested in the major-element or mineralogic composition of the upper mantle rely heavily on the KLB-1 bulk composition because it is among very few natural peridotite samples that may approximate the composition of typical mid-ocean ridge basalt (MORB) source regions. In particular, compositions of MORB indicate that the convecting upper mantle is fertile with respect to major elements such as CaO, Al_2O_3, and Na_2O (Salters and Stracke 2004; Workman and Hart 2005), suggesting compositions with appreciable modal clinopyroxene, but depleted with respect to large-ion-lithophile elements such as K_2O. Virtually all natural peridotite samples from xenoliths or from orogenic peridotites are depleted in modal clinopyroxene relative to the MORB source, which has 13–18% cpx (Salters and Stracke 2004; Workman and Hart 2005), and/or have small amounts of metasomatic phases such as amphibole and phlogopite and, consequently, have non-negligible K_2O and H_2O. Thus, plausible natural analogues of the composition of the convecting upper mantle are rare. For this reason, experimental studies employing natural materials generally have used one of a very small number of xenolith compositions: PHN-1611 (Mysen and Kushiro 1977), KR4003 (Walter 1998), Tinaquillo lherzolite (Jaques and Green 1980), or KLB-1. Among these, KLB-1 has the unique quality of simultaneously being relatively high in Na_2O (~0.3 wt%; Takahashi 1986; Herzberg et al. 1990) and low in K_2O (<0.03 wt%), and therefore is possibly the only readily available well-studied natural peridotite that may plausibly have major and minor elements similar to typical source regions of MORB (0.29 wt% Na_2O; 0.007 wt% K_2O; Salters and Stracke 2004; Workman and Hart 2005).

Previous bulk analyses of KLB-1 are an analysis published originally by Takahashi (1986) and a reanalysis by Herzberg et al. (1990). In the former case, the analytical technique was not specified, although Herzberg (pers. comm.) suggested that X-ray fluorescence methods were employed. In the latter case, the analysis was by direct-current plasma analysis (DCP). Although these analyses are in close agreement for SiO_2, Al_2O_3, MgO,

* E-mail: davis957@umn.edu

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Na_2O, and K_2O, they have significantly different concentrations of FeO*, CaO, and TiO_2 (Table 1). Whereas Takahashi (1986) reported 8.1% FeO and an Mg no. of 89.6 [=(molar Mg)/(Mg + Fe)], there is 8.59 wt% FeO* in the Herzberg et al. (1990) analysis and consequently an Mg no. of 89.1. Takahashi (1986) found 3.44 wt% CaO, whereas Herzberg et al. (1990) reported 3.03 wt% CaO. This is a difference of 14% (relative), which has a noticeable effect on the calculated mode of clinopyroxene. Also, there is 0.16 wt% TiO_2 in the analysis of Takahashi (1986), whereas Herzberg et al. (1990) found 0.12 wt%. Although these values are quite similar in absolute terms, this 33% relative difference can have considerable influence in interpretation of small melt fraction experiments, in which TiO_2 can be highly enriched in the melt. Therefore, it is beneficial to resolve this conflict to better understand the many experimental studies performed with KLB-1 and to better guide the synthesis of KLB-1 imitations.

The preferred method for accurate major- and minor-element analyses of peridotites or other whole rocks remains wet chemical analysis, such as those performed by Kuno and Aoki (1970). Unfortunately, wet chemical analysis is labor intensive and a dying art—there remain few laboratories with the required facilities or the skilled practitioners. Rapid and precise measurement can be achieved by modern electron-microprobe analyses, but whole-rock analyses by electron microprobe require fusion to a homogeneous glass and, unfortunately, ultramafic compositions do not quench to a glass under normal conditions. They instead form a heterogeneous microcrystalline matrix that is unsuitable for accurate microbeam analysis.

One method to determine the composition of peridotites is to manufacture a glass with the aid of a flux. For example, Robinson et al. (1998) determined the composition of their synthetic peridotite starting material by microprobe analysis of a glass synthesized with the aid of $LiBO_2$ flux. However, this causes some difficulty because the electron microprobe cannot analyze Li and cannot easily give quantitative analyses of B. Therefore, Robinson et al. (1998) did not take into account the influence of Li and B on the ZAF corrections for analyses of other elements in their glass, and subsequently Robinson and Wood (1998) decided that the estimates of these bulk compositions based on the weighed proportions of reagents rendered a superior estimate of their starting composition (J. Blundy, writ. comm.). Kogiso and Hirschmann (2001) also used the $LiBO_2$ flux method to determine the compositions of their starting materials for clinopyroxenite and olivine websteritic partial-melting experiments. They accounted for the Li and B in their ZAF correction based on the weighed proportions of silicate starting material and flux, but the method still has uncertainties associated with possible weighing errors and may be compromised in the absence of $LiBO_2$-rich silicate glasses for microprobe calibration standards.

An alternative approach, first described by Odling (1995), is to produce an ultramafic glass using a phosphate flux. This has the advantage that the proportion of flux can be analyzed directly with the microprobe. However, as detailed below, our attempts to analyze KLB-1 with this method yielded unsatisfactory results. Subsequently, we were able to obtain a flux-free homogeneous glass of KLB-1 through aerodynamic levitation combined with laser melting (Tangeman et al. 2001), and this procedure facilitated accurate and precise microprobe analysis of this key peridotite composition.

Material synthesis and analytical procedures

An aliquant of powdered KLB-1 was provided to us by Claude Herzberg, Rutgers University. Herzberg (pers. comm.) had obtained a small piece of KLB-1 from Takahaski, and prepared the powder using a WC grinding vessel. Accordingly, the same powder that was analyzed by Herzberg et al. (1990) was analyzed by us in this study.

We first prepared a silicate-phosphate glass from this KLB-1 powder by adapting the methods of Odling (1995). The rock powder with grain size ranging from 10–100 μm was mixed with ammonium-orthophosphate monohydrate [$(NH_4)_2HPO_4$] (98.0%, Alfa Aesar) in proportions to yield 15 wt% P_2O_5, mixed in an agate mortar and pestle under ethanol for 2 h, and heated for 5.5 h at 250 °C to drive off the hydrous flux components. Approximately 2 mg of sample was then loaded into a graphite capsule and fused in a piston-cylinder device at 1650 °C and 1.0 GPa for 13 min using the piston-cylinder assembly and calibration described in Xirouchakis et al. (2001). The target composition had 15 wt% P_2O_5 because the quench products of earlier experiments with 20 wt% nominal P_2O_5 contained phosphate-rich phases that were presumably quenched from immiscible phosphate liquids. As a check on our procedure, we also prepared a silicate-phosphate glass of USGS standard dunite DTS-1 (Gladney et al. 1991) using precisely the same methods.

We also synthesized a glass of KLB-1 without flux by laser melting of an aerodynamically levitated bead (Tangeman et al. 2001). The sample was first spheroidized by laser melting small portions of the homogenized powder on a water-cooled copper

Table 1. Bulk analyses of KLB-1

	Takahashi (1986)	Herzberg et al. (1990)	Silicate-phosphate glass bead	s.d.	Silicate-phosphate glass (normalized P_2O_5-free)	Glass bead by laser fusion	s.d.	Recommended composition
No. analyses			30			18		
SiO_2	44.48	44.30	40.41	0.14	47.61	44.84	0.23	44.84
TiO_2	0.16	0.12	0.09	0.02	0.10	0.11	0.02	0.11
Al_2O_3	3.59	3.54	3.11	0.04	3.67	3.51	0.05	3.51
Cr_2O_3	0.31	NA	0.32	0.03	0.37	0.32	0.04	0.32
FeO	8.10	8.59	6.52	0.07	7.69	8.20	0.12	8.20
MnO	0.12	0.14	0.10	0.02	0.11	0.12	0.03	0.12
MgO	39.22	39.50	31.59	0.13	37.22	39.52	0.36	39.52
CaO	3.44	3.03	2.17	0.04	2.56	3.07	0.06	3.07
Na_2O	0.30	0.30	0.52	0.02	0.62	0.23	0.03	0.30
K_2O	0.02	0.01	0.05	0.01	0.05	0.02	0.01	0.02
P_2O_5	NA	NA	12.72	0.08	0.00	NA		
Total	99.74	99.53	97.59		100.00	99.94		100.01

hearth. The 1.5–2 mm diameter polycrystalline spheroids were then aerodynamically levitated in a stream of pure argon and melted using a continuous-wave CO_2 laser. The molten levitating droplets were cooled by blocking the laser beam, resulting in cooling rates of ~300 K/s. Although the KLB-1 composition is a reluctant glass-former, the absence of nucleation sites due to the container-free, melt-phase processing enabled formation of millimeter-sized glassy black spheroids.

The silicate-phosphate glass was mounted with its graphite capsule in a brass plug using epoxy and polished with alumina-lapping pads to a 1 µm finish. One 1.5 mm glass droplet made by levitation/laser processing was mounted with epoxy on a brass plug and polished with diamond lapping films to a 1 µm finish. SEM imaging and major-element analysis were performed using the JEOL JXA-8900 microprobe at the University of Minnesota. Back-scattered and secondary electron imaging of the silicate-phosphate material revealed a homogeneous glass free of any apparent crystallites down to a resolution of <1 µm. Similarly, the glass droplet appeared free of crystallites, but contained sparse 5 µm bubbles, which are believed to be shrinkage voids (Tangeman et al. 2001).

For quantitative analysis, the accelerating voltage was set to 15 kV and the beam current to 10 nA. To avoid migration of alkalis, the beam was defocused to an area of 30 µm for the fluxed glass and 10 µm for the laser-fused glass. Counting times for all oxides except Na_2O were 10 s off peak and 20 s on peak. Na_2O counting times were 5 s off peak and 10 s on, and Na_2O was analyzed on the first pass of the spectrometers. For some analyses, Na_2O counts were monitored as a function of time using the JEOL strip-chart software; no time dependence was detected.

Table 2. List of primary standards used for KLB-1 analyses

Asbestos microcline†	K_2O (all phases)
Black Rock Summit chromium augite‡	SiO_2 (cpx), Al_2O_3 (ol, cpx, opx), FeO* (cpx), MgO (cpx), Ca (ol, cpx, opx, spn)
Corundum	Al_2O_3 (spn)
Johnstown Meteorite hypersthene	SiO_2 (opx), FeO* (opx), MgO (opx)
Kakanui augite	Al_2O_3 (glass), MgO (glass)
Kakanui hornblende	TiO_2 (all phases), Na_2O (glass)
Kakanui pyrope	SiO_2 (spn), FeO* (spn), MgO (spn)
Makaopuhi basaltic glass	CaO (glass)
Mn-hortonolite§	MnO (all phases)
Roberts Victor Mine omphacite	Na_2O (ol, cpx, opx, spn)
San Carlos olivine	SiO_2 (glass, ol), FeO* (glass, ol), MgO (ol)
Tiebaghi Mine chromite	Cr_2O_3 (all phases)

Notes: Samples not otherwise indicated are from Jarosewich et al. (1980). ol = olivine, cpx = clinopyroxene, opx = orthopyroxene, spn = spinel.
† From Smith and Ribbe (1966).
‡ From Jarosewich et al. (1987).
§ Supplied by Ian Steele, University of Chicago.

An array of mineral and glass standards (Table 2) were selected for analysis of the peridotitic glass. To ensure that these standards rendered accurate analyses, we verified that the calibration reproduced accurately the compositions of secondary olivine, hypersthene, and basalt standards (Table 3). The range of concentrations of the secondary standards spans those of KLB-1 for all oxides analyzed.

The mean compositions of the P-fluxed glass and the laser-melted KLB-1 glass were determined by averaging of 30 and 18 points, respectively, taken over a large area of each of the polished samples. Plots of concentrations of different oxides against one another did not reveal any systematic variations in composition, indicating that variations in analyzed concentrations were due chiefly to counting statistics rather than compositional heterogeneities. Analytical uncertainties were estimated from the calculated standard deviation of the analyses (Table 1).

Quantitative analyses of KLB-1 mineral phases were also performed to accompany the peridotite glass analyses. The powdered KLB-1 sample was mounted in epoxy and polished with diamond lapping film and finished with a Syton colloidal silica suspension. Conditions for analysis of olivine, orthopyroxene, clinpyroxene, and spinel were an accelerating voltage of 15 kV and a beam current of 15 nA. The electron beam was focused to a 1 µm spot, and counting times for various oxides were the same as listed above.

Results and discussion

Analyses of phosphate-silicate glasses produced low totals (97%) (Table 1), possibly indicating that heating at 250 °C did not completely dehydrate the starting composition. Furthermore, the analyzed SiO_2, FeO, MgO, CaO, and Na_2O concentrations, when normalized on a P_2O_5-free basis, were markedly different from those expected based on previous analyses (Takahashi 1986; Herzberg et al. 1990). Similar discrepancies were observed between the silicate-phosphate glass fused from standard DTS-1 and the accepted concentrations of that standard (Gladney et al. 1991). The P-fluxed DTS-1 gave high concentrations of SiO_2, Al_2O_3, and Na_2O as well as low concentrations of MgO and FeO. Possible explanations for these discrepancies include impurities from the phosphate reagent, Fe loss during high-temperature fusion in the piston cylinder, and uncertainties in ZAF corrections for phosphate-silicate glasses. However, differences between the analyzed phosphate-silicate glasses and the expected composition

Table 3. Microprobe analyses of secondary standards used to test primary peridotite standards

	Johnstown meteorite hypersthene			San Carlos olivine			Indian Ocean basalt glass		
	Accepted value*	Analyzed	s.d.	Accepted value*	Analyzed	s.d.	Accepted value*	Analyzed	s.d.
n		4			4			4	
SiO_2	54.09	53.81	0.21	40.81	40.40	0.28	51.52	51.09	0.09
TiO_2	0.16	0.13	0.03	0.00	–0.02	0.01	1.30	1.28	0.02
Al_2O_3	1.23	1.10	0.02		0.04	0.01	15.39	15.51	0.12
Cr_2O_3	0.75	0.83	0.04		0.03	0.04		0.06	0.03
FeO	15.22	15.30	0.12	9.55	9.48	0.1	9.13	8.80	0.14
MnO	0.49	0.46	0.04	0.14	0.13	0.01	0.17	0.15	0.04
MgO	26.79	26.91	0.05	49.42	50.68	0.07	8.21	8.20	0.10
CaO	1.52	1.45	0.05		0.11	0.01	11.31	11.33	0.05
Na_2O		0.02	0.02		0.01	0.01	2.48	2.61	0.07
K_2O		0.02	0.02		0.01	0.01	0.09	0.08	0.02
Total	100.25	100.03		99.92	100.87		99.60	99.11	

* Jarosewich et al. (1980).

are too great to be attributable to the 2% nominal $(NH_4)_2HPO_4$ flux impurities. Also, short run times and thick graphite containers should minimize the magnitude of Fe loss. Finally, Odling (1995) successfully analyzed peridotite standards with the microprobe using phosphate-silicate glasses. Therefore, we are unsure why this method did not produce reliable results.

Our estimation of the composition of KLB-1 based on microprobe analyses of the quenched silicate glass agrees closely with the two previous analyses where they agree with each other. Our analyses of SiO_2, Al_2O_3, Cr_2O_3, MnO, MgO, and K_2O agree well with both previous measurements (Table 1). For the three other oxides (FeO*, CaO, and TiO_2), concentrations analyzed by microprobe help clarify previous discrepancies between the analyses of Takahashi (1986) and Herzberg et al. (1990). The concentrations of CaO and TiO_2 determined by microprobe analysis closely resemble those determined by Herzberg et al. (1990), but the microprobe value for FeO* is closer to the value given in Takahashi (1986).

The analyzed Na_2O concentration, 0.25 wt%, is lower than the 0.30% value determined by both Takahashi (1986) and Herzberg et al. (1990). We believe that small amounts of Na volatilization during laser fusion compromised our Na_2O analyses and that 0.30 wt% is a more accurate estimate of the Na_2O concentration of KLB-1. Taking the higher value of Na_2O from previous analyses together with the microprobe-determined concentrations of other oxides results in the recommended composition of KLB-1 indicated in Table 1.

The Mg no. of the preferred KLB-1 composition is 89.6. This value is consistent with the compositions of the individual electron microprobe analyses of KLB-1 minerals that we measured (Table 4), which include olivine (89.6), orthopyroxene (90.0), clinopyroxene (89.3), and spinel (76.2). The Mg nos. for our mineral analyses differ from those reported in Hirose and Kushiro (1993) (89.1, 89.7, 89.4, and 78.3, respectively for ol, opx, cpx, and sp), which match more closely the bulk composition reported by Herzberg et al. (1990) bulk KLB-1 Mg no. of 89.1.

Mass-balance calculation by unweighted least-squares fitting using the bulk composition and mineral compositions analyzed in this work indicates that the subsolidus mode of KLB-1 spinel peridotite is 60% olivine, 23% orthopyroxene, 14% clinopyroxene, and 2% spinel, with residual sum of squares of 0.009 (Table 4). The mismatch comes chiefly for FeO*, for which the sum of minerals and the bulk composition differ by 0.07 wt%. Although the mineral modes depart by no more than 2% compared to those reported by Hirose and Kushiro (1993), the mass balance represents a considerable improvement from those derived by the mineral analyses reported by Hirose and Kushiro (1993) and the bulk from Takahashi (1986), which yield a residual sum of squares of 0.230. On the other hand, the combination of the mineral compositions from Hirose and Kushiro (1993) and the bulk composition of Herzberg et al. (1990) gives a much smaller residual sum of squares (0.028). Also, mass-balance calculations using our new mineral analyses and previous bulk compositions published by Takahashi (1986) or Herzberg et al. (1990) have larger residual sums of squares (0.026 and 0.087, respectively, Table 3) than the sum resulting from combination of the new mineral and bulk analyses. Of all of these combinations, mass balance using the combination of our revised mineral and bulk compositions gives the smallest residuals, although some other combinations also may be statistically valid.

The new bulk composition also gives improved mass balances for phase compositions measured in high-pressure experiments, and consequently, more accurate estimates of mineral and melt proportions. As an example, in Table 5 we calculate phase proportions from the phase compositions determined from a partial-melting experiment conducted with KLB-1 at 3 GPa and 1510 °C in a piston-cylinder apparatus (Davis and Hirschmann, in prep). Using the revised bulk composition, the calculated sum of squares of the residuals is 0.037, which compares favorably to values of 0.109 and 0.102 that result from calculations when the composition of Takahashi (1986) and Herzberg et al. (1990), respectively, are used (Table 5). Depending on the assumed bulk composition, substantial differences in the calculated modes are evident. Importantly, calculated mineral modes vary considerably depending on the bulk composition employed; proportions of clinopyroxene range from 3% using the Herzberg et al. (1990)

TABLE 4. Phase analysis for powdered KLB-1 grain mount with modes determined from mass-balance calculations for all 3 KLB-1 analyses

	ol	s.d.	opx	s.d.	cpx	s.d.	spn	s.d.
n	6		8		7		8	
SiO_2	40.58	0.36	54.18	0.39	51.33	0.17	0.13	0.07
TiO_2	0.00	0.01	0.10	0.02	0.55	0.02	0.11	0.02
Al_2O_3	0.03	0.02	5.35	0.16	7.83	0.17	59.30	0.25
Cr_2O_3	0.01	0.03	0.35	0.04	0.69	0.03	8.53	0.10
FeO	10.05	0.16	6.35	0.11	3.18	0.06	11.12	0.15
MnO	0.15	0.03	0.13	0.03	0.07	0.01	0.10	0.01
MgO	48.39	0.26	32.08	0.14	14.83	0.16	20.01	0.15
CaO	0.09	0.03	0.82	0.04	19.50	0.15	0.01	0.01
Na_2O	0.02	0.01	0.11	0.03	1.68	0.06	0.01	0.02
K_2O	0.01	0.01	0.03	0.01	0.02	0.01	0.02	0.01
Total	99.32		99.50		99.68		99.34	
Mg no.	89.6		90.0		89.3		76.2	

Bulk composition	Calculated modes				s.s.r.
This study	0.60	0.23	0.14	0.02	0.009
Takahashi	0.61	0.20	0.16	0.02	0.026
Herzberg	0.62	0.22	0.14	0.02	0.087
Modes for mineral compositions of Hirose and Kushiro (1993)					
This study	0.58	0.26	0.14	0.02	0.177
Takahashi	0.59	0.23	0.16	0.02	0.230
Herzberg	0.60	0.25	0.14	0.02	0.028

TABLE 5. Phase analysis for a piston-cylinder experiment performed on KLB-1 at 3 GPa and 1510 °C (Davis and Hirschmann, in prep) with modes determined from mass-balance calculations for all 3 KLB-1 bulk analyses

	opx	cpx	ol	liq
SiO_2	53.88	52.68	41.29	47.10
TiO_2	0.07	0.10	0.01	0.64
Al_2O_3	6.27	6.90	0.19	12.63
Cr_2O_3	0.68	0.83	0.14	0.42
FeO	5.71	5.12	9.60	7.65
MnO	0.11	0.12	0.12	0.15
MgO	31.17	23.35	48.66	17.59
CaO	2.15	10.14	0.29	12.21
Na_2O	0.17	0.59	0.03	1.20
K_2O	0.01	0.02	0.02	0.03
Total	100.22	99.85	100.35	99.62

Bulk composition	Calculated modes				s.s.r.
This study	0.15	0.08	0.63	0.14	0.037
Takahashi	0.10	0.11	0.64	0.15	0.109
Herzberg	0.15	0.03	0.64	0.18	0.102

KLB-1 composition to 11% using the Takahashi (1986) composition. The calculated clinopyroxene mode using the revised KLB-1 composition is 8%.

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