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Reply to “Oxygen isotope heterogeneity of the mantle beneath the Canary Islands: a discussion of the paper of Gurenko et al.”

Andrey A. Gurenko · Ilya N. Bindeman ·
Marc Chaussidon

Abstract The comment by Day et al. (Contrib Mineral Petrol, 2012) (1) discusses the validity of the previously obtained oxygen isotope data for El Hierro and La Palma (Canary Island) olivines, (2) questions the approach by Gurenko et al. (Contrib Mineral Petrol 162:349–363, 2011) of using weakly correlated variations of $\delta^{18}\text{O}_{\text{olivine}}$ values with X_{px} (proportion of pyroxenite-derived melt in the parental magma), and (3) provides reasons why oxygen isotope data by secondary ion mass spectrometry (SIMS) “offer sensitive means for detecting melt-crust interactions.” We respond these comments and report a new set of oxygen isotope measurements performed by SIMS and single-grain laser fluorination methods. These measurements confirm our previous data and conclusions and demonstrate the ability of the SIMS technique to analyze O isotopes in terrestrial samples with 2-sigma uncertainty better than ± 0.25 ‰.

Keywords Canary Islands · Oxygen isotopes · Olivine · Ion microprobe · Laser fluorination · Mantle heterogeneity

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Introduction

We are pleased that our work (Gurenko et al. 2011) received critical comments. The comment by Day et al. (2012) (1) compares oxygen isotope data obtained by Day et al. (2009, 2010) and Gurenko et al. (2011) for El Hierro and La Palma (Canary Islands) olivines; (2) questions the approach of Gurenko et al. (2011) to define $\delta^{18}\text{O}$ composition of mantle source components by using weakly correlated variations of $\delta^{18}\text{O}_{\text{olivine}}$ obtained by secondary ion mass spectrometry (SIMS) with X_{px} = proportion of pyroxenite-derived melt in the parental magma inferred from olivine elemental composition (e.g., Sobolev et al. 2005, 2007), and (3) provides reasons why oxygen isotope data by SIMS “offer sensitive means for detecting melt-crust interactions,” whereas the laser fluorination (LF) datasets “remain robust” and reflect true O isotope variations in the mantle.

The models proposed by Day et al. (2009, 2010) and Gurenko et al. (2009, 2010, 2011) suggest similar two-component lithology of the Canary mantle plume, but predict different $\delta^{18}\text{O}$ values of the peridotite and pyroxenite mantle endmembers. We think that the primary reason of such discrepancy is a systematic difference in the mean $\delta^{18}\text{O}$ values and their ranges obtained by Day et al. (2009, 2010) and Gurenko et al. (2011), partly because these two studies dealt with the samples coming from different volcanic formations and partly because different analytical approaches were employed. First, the collection by Day et al. (2009, 2010) is broader and includes both primitive and evolved lavas, whereas the Gurenko et al.’s (2010) study is based mostly on primitive lavas representing shield-building stages of the islands. In fact, only recent basanites from El Hierro and Caldera de Taburiente picritic lavas from La Palma represent common objects,

but not the same units, for both studies. Second, Day et al. (2009, 2010) employed multi-grain LF method, while Gurenko et al. (2011) used both single-grain LF and SIMS methods.

Oxygen isotope compositions obtained for 14 lavas from three different locations on El Hierro by Day et al. (2009, 2010) (5.04–5.32 ‰; average, 5.17 ± 0.16 ‰; 2 SD) lie within the range of mantle peridotites and mid-ocean ridge basalts (4.8–5.4 ‰; Matthey et al. 1994; Eiler 2001), whereas 27 La Palma lavas exhibit systematically more ^{18}O -depleted and diverse signatures (4.71–5.35 ‰; average, 4.87 ± 0.36 ‰; 2 SD). The average $\delta^{18}\text{O}_{\text{olivine}}$ value reported by Gurenko et al. (2011) using SIMS for 13 individual olivines from one El Hierro 2900 BP basanite is similar, but the interval is wider (4.82–5.53 ‰; average, 5.21 ± 0.40 ‰; 2 SD) than those by Day et al. (2009, 2010). In contrast, the average $\delta^{18}\text{O}_{\text{olivine}}$ value based on 22 individual olivines from four Caldera de Taburiente lavas (Gurenko et al. 2011) is higher, and the range is about two times wider (4.62–6.13 ‰; average, 5.48 ± 0.71 ‰; 2 SD), as compared with those reported by Day et al. (2009, 2010). Furthermore, the LF $\delta^{18}\text{O}_{\text{olivine}}$ values reported by Gurenko et al. (2011) also correspond to a common “mantle olivine range” of 4.8–5.4 ‰, whereas the SIMS data appear to be systematically higher in $\delta^{18}\text{O}_{\text{olivine}}$ (Fig. 4 in Gurenko et al. 2011). Because all these observations are crucial for further data interpretation, we first re-demonstrate that our single-grain LF and SIMS methods provide firm, complementary results, and the observed high $\delta^{18}\text{O}_{\text{olivine}}$ variations in the SIMS dataset are real and reproducible.

Assessment of data quality

We present here 117 new oxygen isotope analyses by SIMS performed on 39 olivine grains from the collection analyzed by Gurenko et al. (2011) using the CAMECA IMS 1270 instrument (Table 1). Of these grains, 13 are analyzed by single-grain LF method (precision ± 0.1 – 0.2 ‰, 2 SD; Bindeman 2008) but not measured by SIMS (Gurenko et al. 2011). The rest of 26 grains are also olivines from Gurenko et al. (2011), which were re-analyzed by SIMS now. We used the same technique of sample preparation, instrument setup, and calibration routine described in Gurenko et al. (2011). Each analysis is an average of three spots on the same olivine grain. The 2 SDOM (standard deviation of mean) uncertainty of a single measurement based on the instrument counting statistics and precision on instrumental mass fractionation is 0.15–0.29 ‰, with a mean of 0.21 ‰. The variability within individual olivine grains (2 SD) is 0.02–0.67 ‰, with a mean of 0.25 ‰, which is either similar or up to ~ 2.3 times higher than the uncertainty of

individual measurements. The last can be due to intra-crystalline oxygen isotope variability found in a few olivine grains, as also reported for zoned Icelandic olivines (Bindeman et al. 2008). The uncertainty on $\delta^{18}\text{O}$ of the unknown olivines was defined as either the uncertainty of single measurement or 2 SD intra-grain reproducibility, whichever is larger, multiplied by $1/\sqrt{N}$ (Table 1). It is worth noting that only six of 39 grains exhibit >2 -fold larger intra-crystal variability, as compared with the uncertainty of a single measurement. This implies that most of the analyzed individual olivine grains appear to be homogeneous in oxygen isotopes, but showing a wide (>1 ‰) inter-crystalline $\delta^{18}\text{O}_{\text{olivine}}$ range.

The 13 new and nine replicated olivine crystals analyzed by LF and SIMS methods fit the 1:1 line and demonstrate the agreement between the two methods (Fig. 1a). Our new measurements range from 4.75 to 5.93 ‰ and show overall reproducibility within ± 0.25 ‰ uncertainty, although eight of 39 analyses plot outside the $\pm 2\sigma$ array (Fig. 1b). After excluding eight outliers and applying Student’s t test for paired populations, a small but statistically significant offset of -0.13 ‰ in $\delta^{18}\text{O}_{\text{olivine}}$ values was found between the new dataset and Gurenko et al.’s (2011) data. However, because it does not exceed the overall ± 2 SDOM analytical precision of SIMS method, we reiterate the significance of our previously reported $\delta^{18}\text{O}_{\text{olivine}}$ range of 4.93–6.09 ‰.

Gurenko et al. (2011) have demonstrated that whole grains and >250 μm fragments of visibly fresh and inclusion-free olivines pre-treated in 30 % HF or HFB₄ solutions before LF analysis may give poor reproducibility and systematically lower $\delta^{18}\text{O}$ values compared with those obtained by SIMS. They ascribed this effect to remnant alteration as some olivine fragments, even after the pre-treatment in HF and HFB₄, looked dull under the binocular microscope. Crushing crystals to smaller size (100–150 μm fraction) and overnight leaching in HFB₄ solution gave better reproducibility of the replicate measurements, and consistent results were obtained by single-grain LF and in situ SIMS methods (Table 1; Fig. 1a).

Day et al. (2012) disagree with Gurenko et al. (2011) that oxygen isotope data by Day et al. (2009, 2010) may be influenced by remaining alteration left after cleaning hand-picked “visibly fresh, inclusion-free olivine” separates “using an ultrasonic bath and ultra-pure MQ-H₂O and methanol reagents” (Day et al. 2009, GSA Data Repository DR2009128). Day et al. (2012) provide four indicators that such pre-treatment was sufficient to avoid any possible bias on the measured $\delta^{18}\text{O}_{\text{olivine}}$ values. These are (a) analysis of mineral grains free from visible inclusions or alteration, (b) no systematic relationships between $\delta^{18}\text{O}_{\text{olivine}}$ values and the degree of visible alteration of the host rocks, (c) existence of oxygen isotopic equilibrium between olivine and clinopyroxene phenocrysts, and (d) demonstration

Table 1 Oxygen isotope compositions of the Canary olivines analyzed by SIMS and single-grain LF methods

| Sample N Note | N | $\delta^{18}\text{O}$ SIMS This study | 2 SDOM Single | 2 SD Repr. | 2 SDOM Uncert. | N | $\delta^{18}\text{O}$ SIMS Gurenko et al. (2011) | 2 SDOM Uncert. | N | $\delta^{18}\text{O}$ LF | 2 SDOM Repr. | Fo |
|---------------------|-----|---|------------------|---------------|-------------------|-----|--|-------------------|-----|-----------------------------|-----------------|------|
| <i>Gran Canaria</i> | | | | | | | | | | | | |
| G1265-O11 | 3 | 5.23 | 0.16 | 0.22 | 0.12 | 3 | 4.94 | 0.16 | 2 | 4.91 | 0.07 | 86.6 |
| GC59-O124 | 3 | 5.32 | 0.26 | 0.20 | 0.15 | – | – | – | 1 | 5.34 | – | 89.4 |
| <i>Tenerife</i> | | | | | | | | | | | | |
| TF04-O123 | 3 | 5.07 | 0.27 | 0.37 | 0.22 | – | – | – | 1 | 5.30 | – | 80.2 |
| TF04-O124 | 3 | 5.21 | 0.26 | 0.35 | 0.20 | – | – | – | 1 | 5.16 | – | 83.2 |
| TF04-O125 | 3 | 5.46 | 0.24 | 0.25 | 0.14 | – | – | – | 1 | 5.31 | – | 82.1 |
| TF04-O126 | 3 | 5.14 | 0.19 | 0.30 | 0.17 | – | – | – | 1 | 5.25 | – | 82.3 |
| TF04-O130 | 3 | 5.40 | 0.21 | 0.28 | 0.16 | 3 | 5.60 | 0.20 | – | – | – | 84.6 |
| TF04-O145 | 3 | 5.04 | 0.22 | 0.13 | 0.13 | 3 | 5.87 | 0.25 | – | – | – | 84.7 |
| TF04-O149 | 3 | 5.16 | 0.21 | 0.10 | 0.12 | 3 | 5.97 | 0.19 | – | – | – | 84.2 |
| TF07-O12 | 3 | 5.56 | 0.21 | 0.47 | 0.27 | 3 | 5.81 | 0.19 | – | – | – | 82.9 |
| TF07-O14 | 3 | 5.71 | 0.22 | 0.07 | 0.13 | 3 | 5.85 | 0.29 | – | – | – | 82.8 |
| TF07-O111 | 3 | 5.76 | 0.21 | 0.02 | 0.12 | 3 | 5.84 | 0.22 | – | – | – | 82.4 |
| TF23-O11 | 3 | 5.23 | 0.16 | 0.28 | 0.16 | 3 | 4.93 | 0.13 | – | – | – | 84.1 |
| TF23-O12 | 3 | 5.35 | 0.16 | 0.29 | 0.17 | 2 | 5.30 | 0.32 | 1 | 4.91 | – | 82.2 |
| TF23-O121 | 3 | 5.46 | 0.15 | 0.34 | 0.20 | – | – | – | 1 | 5.03 | – | 84.5 |
| TF64-O12 | 3 | 5.51 | 0.16 | 0.18 | 0.10 | – | – | – | 2 | 5.22 | 0.09 | 81.6 |
| <i>La Gomera</i> | | | | | | | | | | | | |
| LG11-O11a | 3 | 4.81 | 0.17 | 0.07 | 0.10 | 2 | 5.28 | 0.32 | 1 | 4.89 | – | 80.3 |
| LG11-O12 | 3 | 4.97 | 0.17 | 0.13 | 0.10 | 5 | 5.15 | 0.14 | 1 | 5.06 | – | 84.2 |
| LG11-O140 | 3 | 5.17 | 0.29 | 0.10 | 0.17 | 3 | 5.29 | 0.22 | – | – | – | 84.5 |
| LG35-O11a | 3 | 5.27 | 0.17 | 0.58 | 0.34 | 2 | 5.04 | 0.31 | – | – | – | 85.3 |
| LG35-O12 | 3 | 5.17 | 0.17 | 0.67 | 0.39 | 3 | 5.26 | 0.19 | 1 | 4.70 | – | 84.4 |
| LG35-O140 | 3 | 5.32 | 0.22 | 0.36 | 0.21 | 3 | 5.66 | 0.22 | – | – | – | 85.1 |
| LG35-O145 | 3 | 5.63 | 0.22 | 0.41 | 0.24 | 3 | 5.79 | 0.22 | – | – | – | 85.3 |
| LG35-O147 | 3 | 5.81 | 0.22 | 0.41 | 0.24 | 3 | 5.91 | 0.22 | – | – | – | 85.5 |
| LG35-O152 | 3 | 5.34 | 0.22 | 0.03 | 0.13 | 4 | 5.52 | 0.19 | – | – | – | 88.0 |
| LG35-O176 | 3 | 5.47 | 0.20 | 0.10 | 0.11 | 3 | 5.67 | 0.22 | – | – | – | 86.1 |
| LG35-O182 | 3 | 5.93 | 0.22 | 0.29 | 0.17 | 4 | 6.09 | 0.22 | – | – | – | 87.4 |
| LG59-O12 | 3 | 5.73 | 0.18 | 0.50 | 0.29 | 3 | 5.91 | 0.25 | 2 | 5.56 | 0.19 | 89.5 |
| LG59-O1107 | 3 | 5.10 | 0.21 | 0.34 | 0.20 | – | – | – | 1 | 5.22 | – | 80.7 |
| LG59-O1108 | 3 | 5.35 | 0.21 | 0.29 | 0.17 | – | – | – | 1 | 5.25 | – | 83.8 |
| LG59-O1110 | 3 | 5.29 | 0.21 | 0.16 | 0.12 | – | – | – | 1 | 5.23 | – | 89.6 |
| <i>La Palma</i> | | | | | | | | | | | | |
| LP1-O11 | 3 | 5.09 | 0.20 | 0.08 | 0.11 | 3 | 5.57 | 0.18 | – | – | – | 85.7 |
| LP1-O12 | 3 | 5.07 | 0.20 | 0.40 | 0.23 | 3 | 5.21 | 0.18 | 1 | 5.15 | – | 84.0 |
| LP13-O12 | 3 | 5.28 | 0.20 | 0.21 | 0.12 | 2 | 5.50 | 0.35 | 2 | 5.07 | 0.07 | 85.8 |
| LP13-O141 | 3 | 5.38 | 0.22 | 0.31 | 0.18 | – | – | – | 1 | 5.28 | – | 85.6 |
| LP13-O142 | 3 | 5.83 | 0.22 | 0.13 | 0.13 | – | – | – | 1 | 5.51 | – | 85.3 |
| LP13-O143 | 3 | 5.20 | 0.22 | 0.06 | 0.13 | – | – | – | 1 | 5.25 | – | 84.2 |
| LP48-O11 | 3 | 4.75 | 0.19 | 0.06 | 0.11 | 3 | 5.38 | 0.18 | – | – | – | 83.6 |
| LP48-O12 | 3 | 5.24 | 0.20 | 0.14 | 0.11 | 3 | 5.40 | 0.18 | 2 | 4.89 | 0.01 | 83.7 |

The complete elemental olivine analyses are reported in Gurenko et al. 2011. All $\delta^{18}\text{O}$ values of the present study (labeled as *This study*) represent replicate measurements by ion microprobe (*SIMS*) in the olivine crystals previously analyzed by *SIMS* and single-grain *LF* methods and reported by Gurenko et al. (2011). Each analysis represents average of several individual spots or replicate measurements (N). We report the uncertainty of a single measurement (2 *SDOM single*), external reproducibility of N measurements of an individual crystal (2 *SD repr.*), and overall analytical uncertainty (2 *SDOM uncert.*) defined as $1/\sqrt{N} \times 2$ *SDOM single* or $1/\sqrt{N} \times 2$ *SD repr.*, whichever is larger. The $\delta^{18}\text{O}$ values are given in permil units and defined relative to the standard mean ocean water (SMOW, $^{18}\text{O}/^{16}\text{O} = 0.0020052 \pm 0.00000043$; Baertschi 1976) standard, that is, $\delta^{18}\text{O} = [(^{18}\text{O}/^{16}\text{O}_{\text{sample}} - ^{18}\text{O}/^{16}\text{O}_{\text{SMOW}}) / ^{18}\text{O}/^{16}\text{O}_{\text{SMOW}}] \times 1,000, \text{‰}$. Fo = forsterite content of olivine given in mol.%, – = no value

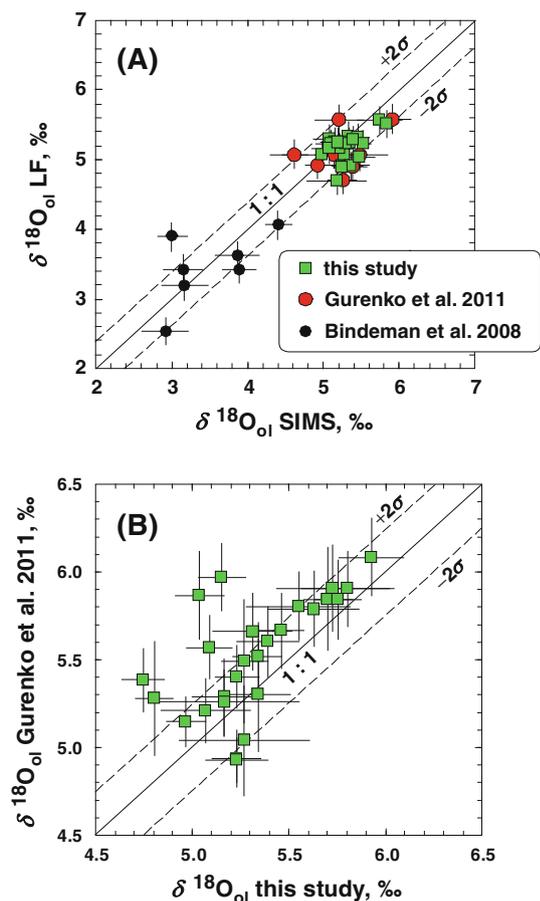


Fig. 1 Oxygen isotope composition of olivine phenocrysts from primitive picrite to basanite lavas representing Gran Canaria, Tenerife, La Gomera, and La Palma, Canary Islands, shield-building stages. **a** The $\delta^{18}\text{O}_{\text{olivine}}$ values obtained by SIMS and single-grain laser fluorination (LF) methods, using different fragments of the same olivine crystals. The $\pm 2\sigma$ array ($\pm 0.4 \text{‰}$) represents the uncertainty calculated from the deviations of individual oxygen isotope measurements from the 1:1 line (Taylor 1982), using the combined Bindeman et al. (2008) and Gurenko et al. (2011) dataset for Icelandic and Canary Island olivines, respectively. **b** Reproducibility of oxygen isotope measurements by SIMS based on a subset of olivines previously analyzed by Gurenko et al. (2011). The $\pm 2\sigma$ array ($\pm 0.25 \text{‰}$) represents average uncertainty of SIMS measurements declared by Gurenko et al. (2011). There is a systematic offset in our new measurements to slightly lower $\delta^{18}\text{O}_{\text{olivine}}$ values (mean of -0.13‰ , excluding eight outliers). The offset is likely caused by particular instrument setting and measurement conditions, which may not correspond exactly to those applied several years ago. As it does not exceed the overall $\pm 2\sigma$ analytical precision by SIMS method, we reiterate the significance of our previously reported $\delta^{18}\text{O}_{\text{olivine}}$ range of 4.93–6.09 ‰

by mass balance calculation that more than 10 % alteration minerals must be present in the crystals to generate the lowest $\delta^{18}\text{O}_{\text{olivine}}$ values (Day et al. 2009, 2010). We consider these arguments reasonable and admit that the oxygen isotope data reported by Day et al. (2009, 2010) represent true values. However, we note that the best way to

demonstrate high data quality and confirm previously reported systematic difference in $\delta^{18}\text{O}_{\text{olivine}}$ between El Hierro and La Palma would be to report an additional subset of olivines treated in accordance with more aggressive sample preparation routine (i.e., crushing crystals to $<250 \mu\text{m}$ fraction, HBF_4 treatment) or, alternatively, to analyze a new set of olivines from these samples by SIMS.

Do oxygen isotope variations in olivine represent mantle or crustal signature?

Gurenko et al. (2011) argued that the lavas from which primitive olivine phenocrysts have been studied for oxygen isotopes appear to be free of crustal contamination, as evidenced by their mantle-like Sr–Nd–Pb isotopic ratios (Gurenko et al. 2006). Day et al. (2012) dispute this conclusion and suggest that “oxygen isotope compositions obtained by SIMS on intercrystalline variations in phenocrysts from OIB lavas may offer sensitive means for detecting melt–crust interactions.” We are aware that many mafic Canary Island magmas are contaminated due to interaction with Jurassic oceanic crust and marine sediments. These effects have been discussed in our previous publications (Gurenko and Schmincke 2000; Gurenko et al. 2001, 2006, 2009, 2010), suggesting that the lavas particularly studied by Gurenko et al. (2011) are free of crustal contamination. We thus stand by our conclusion that the observed range of $\delta^{18}\text{O}_{\text{olivine}}$ of 4.93–6.09 ‰ (Gurenko et al. 2011) or our presently reproduced range (4.75–5.93 ‰) represent true variations in the mantle-derived magmas, resulting from pre-Mesozoic recycling low- and high- $\delta^{18}\text{O}$ oceanic crust (e.g., Turner et al. 2007) and addition of subcontinental lithosphere (e.g., Gurenko et al. 2010).

Oxygen isotope composition of the peridotite- and pyroxenite-derived melts

Another goal of our investigation was to find correlations between $\delta^{18}\text{O}$ values and the amount of pyroxenite component in the mantle source (e.g., Sobolev et al. 2005, 2007), using O isotopic ratios and Ni and Mn concentrations measured in the same spot by electron and ion microprobes inside *individual* olivine crystals. However, no good correlations between $\delta^{18}\text{O}_{\text{olivine}}$ values and $\text{Ni} \times \text{FeO}/\text{MgO}$ and Mn/FeO ratios were found, except for only a weak correlation ($R = 0.54$, $N = 13$) in one basanitic lava from El Hierro (Gurenko et al. 2011).

Although being weak and only marginally significant (or formally insignificant, as R needs to be >0.55 at 95 % confidence level), the correlation found for the El Hierro

olivines may still help to shed more light on the scale of oxygen isotope heterogeneity in the source of a single OIB lava. We thus used this correlation to crudely estimate oxygen isotopic composition of the melts derived from HIMU-like peridotite ($\delta^{18}\text{O}_{\text{melt}} = 5.3 \pm 0.3 \text{ ‰}$) and MORB-like, depleted pyroxenite ($\delta^{18}\text{O}_{\text{melt}} = 5.9 \pm 0.3 \text{ ‰}$). We note that Bindeman and Kamenetsky (2009) tried to find a worldwide correlation of “pyroxenitic parameters” with $\delta^{18}\text{O}$ in olivines measured by more precise single-grain LF method, but also found poor or no relationships. Day et al. (2012) question this approach by demonstrating that olivines from different lavas provide much wider range of estimated $\delta^{18}\text{O}$ values (4.2–5.2 ‰ for peridotite and 5.5–6.7 ‰ for pyroxenite) that “spans nearly the entire range of O isotope compositions measured by LF for uncontaminated mantle-derived OIB to date.” We agree with Day et al. (2012) that the $\delta^{18}\text{O}_{\text{olivine}}$ versus X_{px} correlation from Gurenko et al.’s (2011) study has to be taken with caution. We also emphasize that our data collectively with Bindeman and Kamenetsky (2009) and Day et al. (2009, 2010, 2012) may suggest that the pyroxenitic component of the mantle appears to be not universally high or low in $\delta^{18}\text{O}$, but may have a wide range of oxygen isotope ratios.

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