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Accurate and efficient SIMS oxygen isotope analysis of composition-variable minerals: On-line matrix effect calibration for dolomite - *IAG Young Scientist Medal Lecture*

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Oxygen isotopes are powerful tracers and thermometers. Composition-variable O-bearing minerals are widespread, and their oxygen isotopes play an irreplaceable role in improving our understanding of different geological processes. Secondary ion mass spectrometry (SIMS) offers a new dimension to allowing in-situ isotopic records of O-bearing minerals to be investigated on the micrometer and sub-permil scales, but high-quality oxygen isotope analysis of composition-variable minerals using SIMS is highly challenging. The conventional off-line procedure for SIMS oxygen isotope analysis of composition-variable minerals is inherently inaccurate and analytically inefficient because it requires extra electron probe micro-analyzer (EPMA) chemical compositions to calibrate the instrumental mass fractionation (IMF). The regions analyzed by the EPMA and the SIMS are difficult to be perfectly matched (in terms of spot size, depth, shape, and position). Evaluating and improving conventional off-line methods is crucial for accurate and efficient SIMS oxygen isotope analysis of composition-variable minerals.

We present the first accurate and paired SIMS analysis of $\delta^{18}O$ and Fe# [molar Fe/(Mg+Fe)] in dolomite [1]. Using five newly developed dolomite O-isotopic standards with Fe# ranging from 0.01−0.35, a new accurate and efficient on-line matrix effect calibration method was developed. The method used concurrent SIMS measurements of $^{18}O^{-}$–$^{16}O^{-}$–$^{56}Fe^{16}O^{-}$–$^{24}Mg^{16}O^{-}$ without additional chemical compositions obtained by EPMA. A logistic equation was found to be the best fit for representing the $\delta^{18}O$ matrix effect based on $^{56}Fe^{16}O^{-}/^{24}Mg^{16}O^{-}$ ratios. We also find that a carbonatitic dolomite sample with a large variation of raw SIMS $\delta^{18}O$ measured value has homogeneous oxygen isotope compositions; while a low-temperature original dolomite sample with homogeneous raw SIMS $\delta^{18}O$ values does not represent its true range of variation. The new "online" method can be used as a new strategy for the homogeneity test of in-situ isotope standards in the future. Samples with a large variation in chemical composition but a homogeneous oxygen isotopic composition can be good standards for on-line SIMS analysis. The new on-line method is the best strategy for performing $\delta^{18}O$ analysis of samples with heterogeneous compositions. This method may have further applications in the analysis of other stable isotopes for composition-variable materials.

[1] Xu et al. (2022) *AC*, 22, 7944-7951