

An evaluation of precision and accuracy of SIMS oxygen isotope analysis

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Received 23 March 2018; revised 5 May 2018; accepted 8 May 2018

Available online ■■■

Abstract

In this study, a high-precision secondary ion mass spectrometry (SIMS) zircon oxygen isotope ratio analytical protocol is described. The analysis is conducted using a Cameca IMS 1280-HR at the SIMS Laboratory of Guangzhou Institute of Geochemistry, Chinese Academy of Sciences (GIGCAS). Four reference zircons (91,500, Qinghu, TEMORA 2 and Penglai) have been chosen to evaluate the reproducibility (precision) and accuracy. An identical protocol is used for the analyses, with unchanged instrumental parameters and with ion beam intensities kept as identical as possible. The Penglai zircon is used as an external reference material to calibrate the instrumental mass fractionation. The 91,500, Qinghu and TEMORA 2 zircons are used as the unknown samples, which yielded $10.15 \pm 0.26\text{‰}$ (2SD), $5.46 \pm 0.24\text{‰}$ (2SD) and $8.33 \pm 0.29\text{‰}$ (2SD), respectively, all consistent with the recommended values within error (2SD). A half-year long-term precision of 0.44‰ (2SD) is achieved using the Qinghu zircon as a monitoring sample for the routine measurement of zircon $^{18}\text{O}/^{16}\text{O}$ ratio, which demonstrates the reliability of this analytical protocol.

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Keywords: SIMS; Oxygen isotope; Precision and accuracy; Zircon; Long-term precision

1. Introduction

Oxygen is the most abundant element on Earth and a major component of many rocks and minerals. Oxygen isotope compositions of rocks and minerals is a powerful tool to constrain petrogenetic conditions/mechanisms, magma sources and any possible fluid-wall rock interactions (Taylor, 1968; Li, 2013; Yan et al., 2014; Rehman et al., 2014; France et al., 2016; Riishuus et al., 2015). Zircon is a common accessory mineral in wide range of rocks. It has a stable crystal structure and very high closure temperatures, which make it an ideal target for oxygen isotope research (Li, 2013; Cherniak and Watson, 2003). There are three major methods for measuring zircon oxygen isotopes: (1) the conventional BrF_5 method (Clayton and Mayeda, 1963; Swann and Leng, 2009; Wan and Li, 2006), (2) the laser probe BrF_5 method (laser fluorination

analyses, Sharp, 1990, Elsenheimer and Valley, 1992), and (3) secondary ion mass spectrometry (SIMS, Shimizu et al., 1978, Valley and Graham, 1991, Ireland, 1995, Valley et al., 1998, Tang et al., 2015, Kita et al., 2009, Ickert and Stern, 2013). The conventional BrF_5 method was applied to early oxygen isotope research, which mainly focused on whole-rock or rock-forming minerals (Clayton and Mayeda, 1963). However, hydrothermal alteration of rock-forming minerals would also alter the whole-rock oxygen isotope compositions (Xiao et al., 1998). In addition, this method requires a large amount of sample but would only give an average value of the bulk sample, which conceals any intra-sample differences. In recent years, the laser probe BrF_5 method yields high precision data for a single mineral (Sharp, 1990; Elsenheimer and Valley, 1992). However, this method is less effective for in-situ micro-analyses, which is essential for samples such as multi-stage generated zircons or minerals with inclusions, cracks and/or impurities. Since the 1980s, the SIMS (also called ion microprobe) technique have been increasingly adopted

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Peer review under responsibility of Guangzhou Institute of Geochemistry.

<https://doi.org/10.1016/j.sesci.2018.05.001>

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Please cite this article in press as: Yang, Q., et al., An evaluation of precision and accuracy of SIMS oxygen isotope analysis, Solid Earth Sciences (2018), <https://doi.org/10.1016/j.sesci.2018.05.001>

(Shimizu et al., 1978; Valley and Graham, 1991; Ireland, 1995; Valley et al., 1998; Tang et al., 2015; Kita et al., 2009; Ickert and Stern, 2013), and is powerful for in-situ oxygen isotope microanalysis. Currently, this method is capable of zircon oxygen isotope analyses in a 3D cylinder-shaped sampling space with a 5–20 μm diameter and 1–2 μm depth (approximately 0.2–3 ng of zircon), leaving the sample basically undamaged (Kita et al., 2009). Besides, other information such as U–Pb age and Hf isotope can also be obtained from the same zircon grain (Tang et al., 2015; McKeegan et al., 1998; Ushikubo et al., 2008; Yang et al., 2016; Chen et al., 2016; Wang et al., 2016; Borisova et al., 2016). Recent studies have found that small differences in sample and standard surface topography can cause deflections of the secondary beam, and result in low precision and accuracy (Tang et al., 2015; Kita et al., 2009; Ickert and Stern, 2013; Treble et al., 2007; Whitehouse and Nemchin, 2009). High quality of sample mount preparation is thus essential to obtain good analytical results. In this study, we described a high-precision oxygen isotope analytical procedure employed at the SIMS Laboratory of the Guangzhou Institute of Geochemistry, Chinese Academy of Sciences (GIGCAS), and evaluated its precision and accuracy based on our data obtained for four zircons reference samples. We also addressed a half-year long-term reproducibility of the analytical results for the Qinghu zircon, which is used as a secondary reference sample for routine analyses.

2. Analytical protocol

Sample preparation: Four zircon reference samples 91,500 (Wiedenbeck et al., 1995, 2004), TEMORA 2 (Black et al., 2004), Qinghu (Li et al., 2013) and Penglai (Li et al., 2010) with size 50–200 μm (91,500 and Penglai were crushed at first) were embedded into an epoxy resin of 25.4 mm in diameter. All zircon grains were confined within 5 mm radius of the CAMECA's standard mount center to minimize variation in instrumental bias resulted by the X–Y effects (Kita et al., 2009; Peres et al., 2012). The mount was carefully polished to make the surface as smooth as possible. Mount relief of all the sample area, measured by Atomic Force Microscope, was no more than $\sim 2 \mu\text{m}$ (Fig. 1A). Although there may be sharp relief differences of resin at its contact with the sample grains (Fig. 1B), the sample surface is essentially very smooth. The intra-grain relief is no more than 400 nm, while the grain to grain relief is about 1 μm (Fig. 1A). The polished mount is cleaned in high-purity ethanol using ultrasonic bath, and then vacuum-coated with high-purity gold prior to the SIMS analysis.

Instrumentation and analytical settings: A Cameca IMS 1280-HR SIMS installed at the GIGCAS was employed in this study. IMS 1280-HR inherited features from its pioneering IMS 1280 including automatic centering of the secondary beam to correct for small variability in sample geometry and NMR magnet control with long term stability. These features are all important for high precision stable isotope measurements.

The machine is equipped with two primary ion sources: Cs source and Duoplasmatron source. Cs^+ primary ions are used to sputtering oxygen ion from zircon samples. The primary beam is $\sim 10 \mu\text{m}$ in size diameter, and 2–3 nA in intensity. The target area on the sample was pre-sputtered for 35 s using a 20 μm square raster to remove the gold coating. A 10 μm raster was applied during the analyses in order to assure a more uniform primary beam and a flat-bottom sputter crater. The contrast aperture of 400 μm and the field aperture of $5000 \times 5000 \mu\text{m}^2$ are used. The entrance slit is set at $\sim 125 \mu\text{m}$; and the magnification of the transfer system is configured as ~ 100 (equivalent to an 80 μm). The energy slit is 50 eV and shifts 5 V below maximum transmission. A normal-incidence electron gun is used to suppress charge. The nuclear magnetic resonance (NMR) controller is used to stabilize the magnetic field. This instrument is operated in the static multi-collector mode with a mass resolution of ~ 2400 (FWHM) for the multi-collector slit mode is selected 500 μm width slit. The ^{16}O and ^{18}O ions are detected simultaneously by two faraday cups at the L2' and H1 positions, and the currents are amplified by $10^{10} \Omega$ and $10^{11} \Omega$ resistors, respectively. The signal intensity of ^{16}O is $\sim 2.0 \times 10^9$ cps (counts per second) with ~ 2.2 nA primary beam intensity. The details of the analytical conditions are listed in Table 1. A single spot analysis lasted for 3 min, in which 2 min was for pre-sputtering and automatic centering in the secondary optics (centering DTFA and DTCA), and 1 min was for integrating 16 cycles of the oxygen isotope signal. Corrections for instrumental mass fractionation (IMF) were conducted by a “standard-sample-standard” bracketing external standardization method. The Penglai zircon was used as the external standard and the other three reference zircon samples (91,500, Qinghu and Temora 2) were used as unknowns. One Penglai zircon analysis was conducted with every four unknown spots (for all 91,500, Qinghu and Temora 2 zircons) in the whole session. The $^{18}\text{O}/^{16}\text{O}$ ratios were normalized to Vienna Standard Mean Ocean Water (V-SMOW $^{18}\text{O}/^{16}\text{O} = 0.0020052$; Baertschi, 1976) and expressed on the $\delta^{18}\text{O}$ -scale. A recommended value of $\delta^{18}\text{O} = 5.31 \pm 0.10\text{‰}$ (2SD) assigned to the Penglai zircon was used in this study (Li et al., 2010), and then corrected for the IMF as follows:

$$(\delta^{18}\text{O})_{\text{M}} = \left(\frac{(^{18}\text{O}/^{16}\text{O})_{\text{M}}}{0.0020052} - 1 \right) \times 1000(\text{‰}),$$

$$\text{IMF} = (\delta^{18}\text{O})_{\text{M(standard)}} - (\delta^{18}\text{O})_{\text{R(standard)}},$$

$$\delta^{18}\text{O}_{\text{C}} = (\delta^{18}\text{O})_{\text{M}} - \text{IMF}$$

where $(^{18}\text{O}/^{16}\text{O})_{\text{M}}$ indicates the raw value measured by SIMS. $(\delta^{18}\text{O})_{\text{M}}$ is the normalized $(^{18}\text{O}/^{16}\text{O})_{\text{M}}$ by the value of Vienna Standard Mean Ocean Water (VSMOW, $^{18}\text{O}/^{16}\text{O} = 0.0020052$). $(\delta^{18}\text{O})_{\text{M(standard)}}$ represents the measured result of standard sample. $(\delta^{18}\text{O})_{\text{R(standard)}}$ represents the recommended value of standard sample. $\delta^{18}\text{O}_{\text{C}}$ represents the corrected sample value.

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