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GSA Data Repository Item 2017392

Data Repository Supplement DR1 – Methods

Laser ablation inductively coupled mass spectrometry (LA–ICP–MS) zircon U–Pb geochronology at NGU (sample MO082)

The instrumentation used at NGU consists of a Finnigan MAT Element 1 single-collector high-resolution sector ICP-MS, coupled to a UP193–FX 193 nm short-pulse excimer laser ablation system from New Wave Research. The laser was set to ablate 35 μm spots with a repetition rate of 12 Hz and an energy corresponding to a fluence of 3 J/cm^2. The sample aerosol was transported from the sample chamber in a He gas, and introduced in the ICP-MS instrument as a mixture of He and Ar gas. The data were acquired in a time-resolved counting scanning mode for 60 s. Masses ^{202}Hg, ^{204}(Hg+Pb), ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{232}Th and ^{238}U were measured. Monitoring ^{202}Hg and assuming a ^{202}Hg/^{204}Hg ratio of 4.36 (natural abundance) corrects the interference of ^{204}Hg on ^{204}Pb. A 60 second delay was performed after each zircon analysis, and a 60 second gas blank was acquired at regular intervals. The measured isotope ratios were corrected for element- and mass-bias effects using the Geostandard 91500 reference zircon (1062.4 ± 0.4 Ma; Wiedenbeck et al., 1995), normally based on 15 to 30 analyses during one analytical session. The data reduction was performed using MS Excel spreadsheets with Visual Basic macros developed in-house.

Laser ablation split-stream inductively coupled mass spectrometry (LA–SS–ICP–MS)

The majority of the samples (85XXX) were analysed using a split stream laser ablation system where a portion of the ablated material was split between a quadrupole ICPMS for U–Pb analysis and a multicollector ICPMS for Lu–Hf analysis, meaning that age and compositional information was obtained from the same analytical volume. Zircon was ablated using a Resonetics RESOlution M–50A–LR system, incorporating a COMPex 102 193 nm excimer UV laser. Following two cleaning pulses and a 40 s period of background analysis, samples were spot ablated for 35 s at a 10 Hz repetition rate using a 33 μm beam and laser energy at the sample surface of 2.3 J/cm^2. An additional 40 s of baseline was collected after ablation. The sample cell was flushed with ultrahigh purity He (300 mL/min) and N2 (1.0 mL/min) and high purity Ar was employed as the plasma carrier gas.

For U–Pb analysis isotopic intensities were measured using an Agilent 7700s quadrupole ICP–MS with the following elements monitored for 0.03 seconds each: ^{28}Si, ^{29}Si, ^{204}Pb, ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{232}Th, and ^{238}U. Common lead concentration was monitored throughout the analysis, however, no significant ^{204}Pb was detected and thus no common lead correction has been applied. The primary age standard used in this study was 91500 (1062.4 ± 0.4 Ma; Wiedenbeck et al., 1995) with GJ–1 (608.5 ± 1.5 Ma; Jackson et al., 2004) and R33 (419 ± 0.4 Ma; Black et al., 2004) used as secondary age standards. ^{206}Pb/^{238}U ages calculated for all secondary zircon standards were treated as unknowns and found to be within 3% of the accepted value.

For Lu–Hf analysis on a Nu Plasma II multi-collector inductively coupled plasma mass spectrometer all isotopes (^{180}Hf, ^{179}Hf, ^{178}Hf, ^{177}Hf, ^{176}Hf, ^{175}Lu, ^{174}Hf, ^{173}Yb, ^{172}Yb and ^{171}Yb) were counted on the Faraday collector array. Time resolved data was baseline

subtracted and reduced using Iolite (DRS after Woodhead et al., 2004), where ^{176}Yb and ^{176}Lu were removed from the 176 mass signal using ^{176}Yb/^{173}Yb = 0.7962 and ^{176}Lu/^{175}Lu = 0.02655 with an exponential law mass bias correction assuming ^{172}Yb/^{173}Yb = 1.35274 (Chu et al., 2002). The interference corrected ^{176}Hf/^{177}Hf was normalized to ^{179}Hf/^{177}Hf = 0.7325 (Patchett and Tatsumoto, 1980) for mass bias correction. Zircons from the Mud Tank carbonatite locality were analysed together with the samples in each session to monitor the accuracy of the results. Nineteen analyses of Mud Tank yielded a ^{176}Hf/^{177}Hf value of 0.282507 ± 23 (MSWD = 0.2) identical within uncertainty to the recommended value (0.282522 ± 42; Griffin et al., 2006, 2007). GJ–1 and R33 zircons were also run to verify the method and yielded weighted average ^{176}Hf/^{177}Hf values (GJ-1 = 0.282073 ± 0.000051, MSWD = 1.8, n =18; R33 0.282788 ± 0.000027, MSWD = 1.1, n = 17) within uncertainty of their accepted values (R33 = 0.282764 ± 0.000014, Fisher et al., 2014; GJ–1 = 0.282022 ± 11, Matteini et al., 2010). In addition, the corrected ^{180}Hf/^{177}Hf ratio was calculated to monitor the accuracy of the mass bias correction and yielded an average value of 1.886835, which is within the range of values reported by Thirlwall and Anczkiewicz (2004). Calculation of εHf values employed the decay constant of Scherer et al. (2001) and the Chondritic Uniform Reservior (CHUR) values of Blichert-Toft and Albarède (1997).

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