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Data Repository

Appendix 1. Analytical_methods

Table DR1. LA-ICP–MS zircon U-Pb isotopic analysis of the granitic batholiths in the WKOB.

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Supplementary File 1 Analytical methods

Zircon U–Pb dating

Zircon U–Pb dating was carried out at the MC–ICPMS (Inductively Coupled Plasma Mass Spectrometry) laboratory of the Institute of Geology and Geophysics, Chinese Academy of Sciences (IGG CAS) in Beijing. Detailed operating conditions for the laser ablation system and the ICP–MS instrument and data reduction were the same as described by Xie et al. (2008). An Agilent 7500a quadruple (Q)–ICPMS and a Neptune multi–collector (MC)–ICPMS with a 193 nm excimer ArF laser–ablation system (GeoLas Plus) attached were used for simultaneous determination of zircon U–Pb ages. Helium carrier gas transported the ablated sample materials from the laser-ablation cell via a mixing chamber to the ICPMS. Every spot analysis consisted of ~30 s background acquisition and 40 s sample data acquisition. $^{207}Pb/^{206}Pb$, $^{206}Pb/^{238}U$, $^{207}U/^{235}U$ ($^{235}U = ^{238}U/137.88$), $^{208}Pb/^{232}Th$ ratios were corrected by using zircon 91500 as external standard. The weighted mean U–Pb ages and concordia plots were processed using ISOPLOT 3.0 (Ludwig, 2003).

Zircon Lu–Hf isotope analyses

In situ Hf isotope measurements were subsequently undertaken using Laser Ablation (LA)–ICPMS with a beam size of 60 μm and laser pulse frequency of 8 Hz with age determinations at the MC–ICPMS laboratory of IGG CAS. Details of instrumental conditions and data acquisition were given in Wu et al. (2006). The isobaric interference of ^{176}Lu on ^{176}Hf is negligible due to the extremely low $^{176}Lu/^{177}Hf$ in zircon (normally < 0.002). During the analyses for this study, GJ–1 as an unknown sample yielded a weighted $^{206}Pb/^{238}U$ age of 609.7 ± 6.3 Ma ($2\sigma_n$, MSWD = 0.97, n = 12) and a weighted $^{176}Hf/^{177}Hf$ ratio of 0.282015 ± 0.000003 ($2\sigma_n$, MSWD = 1.12, n = 94), which is in good agreement with the recommended U–Pb age and Hf isotopic ratio (Black et al., 2003; Wu et al., 2006). During data acquisition of Hf isotopes, $^{176}Hf/^{177}Hf$ ratios of the zircon standard (MUD) were 0.282504 ± 0.000003 ($2\sigma_n$, MSWD = 0.71, n = 82).

Initial $^{176}Hf/^{177}Hf$ ratios and $\varepsilon_{Hf}(t)$ values were calculated with reference to the chondritic reservoir (CHUR) at the time of zircon growth in the magmas. The ^{176}Lu decay constant of 1.867 × 10^{-11} year^{-1} (Söderlund et al., 2004), the chondritic $^{176}Hf/^{177}Hf$ ratio of 0.282785 and $^{176}Lu/^{177}Hf$ ratio of 0.0336 (Bouvier et al., 2008) were adopted. Depleted mantle model ages (T_{DM}) used for mafic to intermediate rocks were calculated with reference to the depleted mantle at a present–day $^{176}Hf/^{177}Hf$ ratio of 0.28325, similar to that of the average MORB (Nowell et al., 1998) and $^{176}Lu/^{177}Hf = 0.0384$ (Griffin et al., 2000).

Zircon oxygen isotope analyses

Measurements of zircon O isotopes were conducted using the Cameca IMS 1280HR large–radius SIMS (Secondary Ion Mass Spectroscopy) at the at State Key Laboratory of Isotope Geochemistry (SKLaBIG), Guangzhou Institute of Geochemistry, Chinese Academy of Sciences (GIG CAS). Analytical procedures are the same as those described by Li et al. (2010a). The Cs^+ primary ion beam was accelerated at 10 kV, with an intensity of ca. 2 nA (Gaussian mode with a primary beam aperture of 200 μm to reduce aberrations) and rastered over a 10 μm area. The spot is ~20 μm in diameter (10 μm beam diameter + 10 μm raster).

A normal–incidence electron flood gun was used to compensate for sample charging during analysis with homogeneous electron density over a 100 μm oval area. Negative secondary ions were extracted with a −10 kV potential. The field aperture was set to 5000 μm, and the transfer–optics magnification was adjusted to give a field of view of 125 μm (FA = 8000). A 30 eV energy slit width was used, and its mecahnical positions were controlled before starting the analysis (5 eV gap, –500 digits with respect to the maximum). The entrance slit width was ~120 μm, and the exit slit width was 500 microns for multicollector Farady cups (FCs) for ^{16}O and ^{18}O is 500 μm (MRP = 2500). The intensity of ^{16}O was typically 1×10^9 cps. Oxygen isotopes were measured in multi–collector mode using two off–axis Faraday cups. The NMR (Nuclear Magnetic Resonance) probe was used for magnetic field control with stability better than 2.5 ppm over 16 h on mass 17. One analysis takes ~4 min, consisting of pre–sputtering (~120 s), automatic beam centering (~60 s) and integration of oxygen isotopes (10 cycles × 4 s, total 40 s). Uncertainties on individual analyses are reported at a 2σ level. With low noise on the two FC amplifiers, the internal precision of a single analysis is generally better than 0.3‰ (2σ) for $^{18}O/^{16}O$ ratio. Values of $\delta^{18}O$ are standardized to Vienna Standard Mean Ocean Water compositions (VSMOW) and reported in standard per mil notation.

The instrumental mass fractionation factor (IMF) is corrected using the 91500 zircon standard with ($\delta^{18}O$) VSMOW = 9.9 ‰ (Wiedenbeck et al., 2004). Measured $^{18}O/^{16}O$ is normalized using VSMOW, then corrected for the instrumental mass fractionation factor (IMF) as follows:

$(\delta^{18}O)_M = ((^{18}O/^{16}O)_M / 0.0020052 - 1) \times 1000$ (‰)

$IMF = (\delta^{18}O)_M(\text{standard}) - (\delta^{18}O)_{VSMOW}$

$\delta^{18}O\text{sample} = (\delta^{18}O)_M + IMF$

Seventeen measurements of the PengLai zircon standard during the course of this study yielded a weighted mean of $\delta^{18}O = +5.29 \pm 0.07$ ‰, which is identical within errors to the reported value of 5.31 ± 0.10 ‰ (Li et al., 2010b).

Major and trace element analyses

Rock samples examined by optical microscopy and selected whole–rock samples were sawed into small chips and ultrasonically cleaned in distilled water with < 3% HNO_3 and then in distilled water alone and subsequently dried and handpicked to remove visible contamination. The rocks were crushed and ground in a tungsten carbide ring mill, and the resulting powder was used for analyses of major and trace elements, and Sr–Nd isotopes. Major–element oxides were analyzed using a Rigaku RIX 2000 X–ray fluorescence spectrometer at SKLaBIG, GIG CAS on fused glass beads. Calibration lines used in quantification were produced by bivariate regression of data from 36 reference materials encompassing a wide range of silicate compositions (Li et al., 2005), and analytical uncertainties are between 1% and 5%.

Trace element concentrations, including rare earth element (REE) concentrations, were determined with a Perkin–Elmer ELAN–DRC–e inductively–coupled plasma mass spectrometer (ICP-MS) at the State Key Laboratory of Ore Deposit Geochemistry (SKLOG), Institute of Geochemistry, Chinese Academy of Sciences (IGCAS), with analytical uncertainty better than 10%. Samples were digested with 1 mL of HF and 0.5 mL of HNO3 in screw top PTFE-lined stainless steel bombs at 190 °C for 12 h. The analytical precision is generally better than 1% for elements with concentrations >200 ppm, and 1%–3% when less than 200 ppm. The procedure for the trace elements is described in detail by Qi et al. (2000).

Sr–Nd isotope analyses

Sr and Nd isotopic compositions of selected samples were determined using a MC–ICP-MS at SKLaBIG GIG CAS. Analytical procedures are similar to those described in Wei et al. (2002) and Li et al. (2004). The $^{87}Sr/^{86}Sr$ ratio of the NBS987 standard and $^{143}Nd/^{144}Nd$ ratio of the Shin Etsu JNdi–1 standard measured were 0.710285 ± 15 (2σ) and 0.512085 ± 10 (2σ), respectively. All measured $^{143}Nd/^{144}Nd$ and $^{86}Sr/^{88}Sr$ ratios were corrected for fractionation using ratios of $^{146}Nd/^{144}Nd = 0.7219$ and $^{86}Sr/^{88}Sr = 0.1194$, respectively.

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