minerals

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Supplementary document

Analytical Methods

1. LA-ICP-MS zircon U-Pb dating

Zircons were extracted from a big (5 kilograms) granitic specimen (HG6), using a heavy liquid and magnetic technique, followed by handpicking under a binocular microscope. The positions of U-Pb date analyses were determined using a transmitted and reflected light microscope, as well as cathodoluminescence (CL) imaging with a CAMECA SX51 at 50 kV and 15 nA at the Langfang Regional Geological Survey in Hebei Province, China. CL images were utilized to assess interior textures and choose possible locations for U-Pb dating and Lu-Hf isotopic measurement.

The U-Pb zircon dating analyses were performed at the State Key Laboratory of Geological Processes and Mineral Resources of the China University of Geosciences (Wuhan), China, using an Agilent 7700a ICP-MS (inductively coupled plasma mass spectrometer) apparatus with a 193 nm laser (MicroLas, Gottingen, Germany). Nitrogen was added to the Ar plasma's central gas flow (Ar + He) to decrease the detection limit and enhance the precision. For dating, a 32 m diameter laser beam with a frequency of 50 Hz was used on a Geolas 2005 system. During the analysis, the external standard Harvard zircon 91500 was used to calibrate the isotope results, whereas the zircon standard GJ-1 was utilized as a control standard. An external standard (NIST SRM 610) and a stoichiometric Zr concentration in zircons were used as external and internal standards, respectively, to determine the trace element concentrations of the unknown grains. To calculate the isotopic ratios and element concentrations, the ICPMSDataCal9.0 software (version 9.0, China University of Geosciences (Wuhan)) was used. Concordia diagrams were created using Isoplot/Ex ver.4.0 [1]. The operating conditions and procedures of the analytical techniques were fully described by Liu et al. [2].

2. Whole rock geochemical analysis

The concentration of major oxides was assessed using a PAN analytical Axios X-ray fluorescence spectrometer (XRF) with a precision of 2% (ALS Chemex Company Ltd in Guangzhou, China). The measurements were conducted on fused glass discs prepared from 0.9 g of calcined sample powder mixed with 9.0 g of lithium borate flux (50%–50% $Li_2B_4O_7$–$LiBO_2$) and fused in an auto fluxer at 1050–1100 °C. An Elan 9000 inductively coupled plasma-mass spectrometer (ICP-MS) was used to detect the trace element concentrations using 0.2g of the prepared sample combined with 0.9 g of lithium metaborate flux fused in a furnace at 1000 °C. The resulting ignited powder was diluted in 100 mL of a 4% HNO_3 and 2% HCl solution after cooling. The ICP-MS measurement accuracy was better than ±5–10% for the majority of the elements. The major oxides are given in weight percent (wt.%), whereas the trace elements are given in parts per million (ppm).

3. Lu-Hf isotopes

To complete the zircon Lu-Hf isotopic measurements, the Neptune Plus MC-ICP-MS was employed in combination with a Geolas 2005 excimer ArF laser ablation system (Wuhan). Liu et al. [3] explained clearly the operation conditions and analytical procedure. The $^{176}Hf/^{177}Hf$ ratio (0.282013 ± 0.000022 (1σ, n = 276)) of the standard zircon (GJ1) was compatible with the values within 1σ error recommended by Wu et al. [4]. The ICP-MS-DataCal program was utilized to conduct offline signal selection and integration, in addition to isobaric interference and mass fractionation adjustments[3]). The mantle extraction model age (t_{DM}) was calculated using the zircon $^{176}Lu/^{177}Hf$ ratio, which provides a minimum age for the magmatic melt from which the zircon crystallized. As a consequence, we calculated Hf-t_{DM}^{C} "crustal" model ages based on the assumption that the zircon parental magma originated in an average continental crustal source with $^{176}Lu/^{177}Hf$ = 0.015 [5](G et al.2002) and was subsequently derived from a depleted mantle.

References

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