Analytical Methods

1. Whole-rock major and trace element analysis

The whole-rock geochemical compositions of the metasedimentary rocks and biotite monzogranite were analyzed at the Analytical Laboratory of the Beijing Research Institute of Uranium Geology (China). Whole-rock major oxides of the samples, including SiO_2, TiO_2, Al_2O_3, TFeO, MnO, MgO, CaO, Na_2O, K_2O, P_2O_5, and loss on ignition (LOI), were measured using an AB104L Axios-mAX X-ray fluorescence spectrometer with >-5% analytical precision after being ground to powder and cleaned. The concentrations of trace elements (including rare earth elements [REEs]) were determined using an inductively coupled plasma mass spectrometry (NexION 300X ICP-MS). The powdered samples (~100 mg) were added to a Teflon bomb containing ultrapure hydrofluoric and nitric acids. These bombs were heated at 180 ℃ on an electric hot plate for approximately 48 h. Subsequently, these solutions were re-dissolved in nitric acid and heated again at 140 ℃ for 6 h. Finally, the digested diluted solution with relative deviations of >5 % for elements was analyzed using an ICP-MS.

2. Zircon U-Pb geochronology and in situ Hf isotopic analyses

In-situ zircon U-Pb dating and Lu-Hf isotopic analyses were conducted using a laser ablation inductively coupled plasma mass spectrometer (LA-ICP-MS) and a laser ablation-multi-collector inductively coupled plasma mass spectrometer (LA-MC-ICP-MS), respectively, at Nanjing Hongchuang Geological Exploration Technical Services Co., Ltd.

The Resolution SE model (Applied Spectra, USA) for testing U-Pb dating used a laser ablation system for this analysis, equipped with an ATLEX 300 excimer laser and a two-volume S155 ablation cell. The laser ablation system was coupled to an Agilent 7900 inductively coupled plasma mass spectrometer (ICP-MS). The detailed tuning parameters were obtained from Thompson JM et al., 2018 [1]. LA-ICP-MS tuning was performed using a 50-μm-diameter line scan at 3 μm/s on NIST 612 at ~3.5 J/cm^2 with a repetition rate of 10 Hz. The gas flow was adjusted to obtain the highest sensitivity (^{238}U~6×10^5 cps) and the lowest oxide ratio (ThO/Th <0.2 %). P/A calibration was conducted on the NIST 610 using a 100 μm diameter line scan.

The Lu-Hf isotopic analysis was conducted using a laser ablation platform that utilizes a Resolution SE 193 nm laser ablation system equipped with an S155 dual-volume sample cell. A Neptune Plus MC-ICP-MS instrument was used, and the analysis process included a signal-smoothing device to enhance the signal stability and isotopic ratio measurement precision.

Helium gas was used as the carrier gas, and a small amount of nitrogen gas was introduced to strengthen the Hf sensitivity. The analysis was conducted in single-point mode with a laser spot size of 50 μm, a laser ablation frequency of 8 Hz, and a laser energy density of 6 J/cm². The accurate measurement of zircon Hf isotopes is challenging because of the interference of ^{176}Yb and ^{176}Lu when measuring ^{176}Hf. Studies have indicated that the mass fractionation coefficient (βYb) for Yb is not a fixed value during long-term testing. The βYb obtained through solution nebulization is unsuitable for interference correction in the laser ablation mode for zircon-Hf isotope analysis. Incorrect estimation of βYb can considerably affect the interference correction of ^{176}Yb on ^{176}Hf, subsequently affecting the accurate determination of ^{176}Hf/^{177}Hf ratios. We obtained real-time βYb values from the zircon samples for interference correction (Gu HO et al., 2019) [2]. Values of ^{179}Hf/^{177}Hf = 0.7325 and ^{173}Yb/^{171}Yb = 1.132685 were used to calculate the mass fractionation coefficients βHf and βYb for Hf and Yb, respectively. The value of ^{176}Yb/^{173}Yb = 0.79639 was used to correct for the isobaric interference of ^{176}Yb on ^{176}Hf. In addition, a value ^{176}Lu/^{175}Lu = 0.02656 was used to correct the relatively minor isobaric interference of ^{176}Lu on ^{176}Hf. Because Yb and Lu share similar physical and chemical properties, the mass fractionation coefficient βYb for Yb was employed to correct the mass fractionation behavior of Lu in this experiment. The analytical data, including sample and blank signal selection and isotopic mass fractionation correction, were processed offline using the Iolite software (Paton C et al., 2011) [3]. To ensure the reliability of the analytical data, three zircon standards, Qinghu (Li XH et al., 2013c) [4] and 91500, were analyzed simultaneously with the samples, and the resulting data were consistent with the reference values within the range of error.

References

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[3] Paton, C., Hellstrom, J., Paul, B., Woodhead, J., Hergt, J. Lolite: Freeware for the visualisation and processing of mass spectrometric data. Journal of analytical atomic spectrometry 2011, 26, 2508-2518.

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