

Analytical Methods

Zircons were separated from the samples utilizing heavy-liquid and magnetic separation techniques, and then hand-picked under a binocular microscope. The crystals were embedded in an epoxy resin disk (25 mm in diameter) and polished to expose the grain centers. Cathodoluminescence (CL) images were taken using a JXA-8100 electron microprobe at the Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing.

Zircon U–Pb isotope analyses were performed at the Beijing Quick-Thermo Science & Technology Co., Ltd. using an ESI New Wave NWR 193UC (TwoVol2) laser ablation system connected to an Agilent 8900 ICP–QQQ, following the analytical procedures described in [1]. The spot size and frequency of the laser were set to 20 μm and 5 Hz, respectively. The individual zircon grains (mounted and polished in epoxy) were ablated in a constant stream of He that was mixed downstream with N_2 and Ar before entering the torch region of the ICP–QQQ. After warmup of the ICP–QQQ and connection with the laser ablation system, the ICPMS was tuned for robust plasma conditions by optimizing the laser and ICP–QQQ settings, monitoring $^{232}\text{Th}^{16}\text{O}^+ / ^{232}\text{Th}^+$ ratios ($\leq 0.2\%$ at all instances) and $^{238}\text{U}^+ / ^{232}\text{Th}^+$ ratios (between 0.95 and 1.05 at all instances) during ablation via the in-line scan mode of NIST SRM 610. The 91500 zircon was used as a primary reference material for all U–Pb age determinations, whereas zircon Plešovice was used as a secondary reference yielded a mean weighted $^{206}\text{Pb} / ^{238}\text{U}$ age at 339 ± 0.4 Ma ($N = 60/60$, $\text{MSWD} = 0.6$). National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 610 reference glass was used to normalize U, Th, Pb, and other trace element contents to allow calibration of the unknowns, with internal standard major element Si. The standards were analyzed two times before and after each analytical session, including 6–8 spots on minerals. The background subtraction and correction for laser downhole elemental fractionation were performed using the Iolite data reduction package within the Wavemetrics Igor Pro data analysis software [2]. In addition, the Concordia plots were processed using IsoplotR [3].

References

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2. Paton, C.; Hellstrom, J.; Paul, B.; Woodhead, J.; Hergt, J. Iolite: Freeware for the Visualisation and Processing of Mass Spectrometric Data. *J. Anal. At. Spectrom.* **2011**, *26*, 2508–2518, doi:10.1039/C1JA10172B.
3. Vermeesch, P. IsoplotR: A Free and Open Toolbox for Geochronology. *Geoscience Frontiers* **2018**, *9*, 1479–1493, doi:10.1016/j.gsf.2018.04.001.