

1. The Analytical Procedures of XRF and ICP-MS for Whole-Rock Major- and Trace Elements

XRF: Prepared samples were fused with lithium metaborate-lithium tetraborate flux which also included lithium nitrate as an oxidizing agent, and then poured into a platinum mold to generate a fused disk for XRF analysis. Loss-on-ignition (LOI) at 1000 °C was measured for each sample, and elemental concentrations were calculated from the results of both analyses.

ICP-MS: Prepared samples were added to lithium metaborate/lithium tetraborate flux, mixed well and fused in a furnace at 1025 °C. The resulting melts were then cooled and dissolved in a mixture of nitric, hydrochloric and hydrofluoric acids prior to analysis.

2. The Processes of Zircon Cathodoluminescent (CL) Images

Zircon CL images were obtained at the Wuhan SampleSolution Analytical Technology Co., Ltd., Wuhan, China, using an Analytical Scanning Electron Microscope (JSM-IT300) connected to a Delmic sparc system. The imaging condition was 0.5–30 kV voltage of electric field and 72 μ A current of tungsten filament.

3. The Analytical Procedures and Data Processing Processes of LA-ICP-MS for Zircon U–Pb and Trace Element Measurements

Laser sampling was performed using an excimer laser ablation system consisting of a GeoLas 2005 with an Agilent 7500a ICPMS used to acquire ion-signal intensities. Laser energy and frequency were 70 mJ and 8 Hz, respectively. The beam diameter is 32 μ m for most zircons, with a 24 μ m used for small grains (< 50 μ m wide). Standards were run at the same spot size as the samples. Helium was used as the carrier gas with argon used as the make-up gas and mixed with the carrier gas via a T-connector before entering the ICP-MS. Nitrogen was added into the central gas flow (Ar+He) of the Ar plasma to decrease the detection limits and improve precision [1]. Each analysis incorporated a background acquisition interval of ~20–30 s (gas blank) followed by a 40 s data acquisition interval for the 32 μ m spots and 30 s for the 24 μ m spots. Zircon 91500 was used as the external standard for U–Pb dating, (preferred U–Th–Pb isotopic ratios from [2], and was analyzed twice for every five unknowns. Time-dependent drift was corrected using a linear interpolation (with time) for every five analyses according to variations of 91500. In addition, zircon standard GJ-1 was analyzed as secondary standard. The weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age for GJ-1 was 598.5 ± 4.35 Ma (2σ , $n = 10$), consistent with the recommended values within uncertainty (599.81 ± 1.7 Ma (2σ)) [3]. Every 10 unknown analysis was followed by one analysis of NIST SRM 610 to correct the time-dependent drift of sensitivity and mass discrimination for the trace-element analysis. Trace-element compositions of zircons were calibrated against multiple reference materials (BCR-2G and BIR-1G), combined with internal standardization (^{29}Si , assumed 32.7 % SiO_2 assumed for zircon unknowns). Detailed analytical conditions and procedures for zircon U–Pb LA-ICPMS dating are described in Liu et al. (2010) [4]. Off-line selection and integration of background and analytical signals, time-drift correction, and quantitative calibration for zircon U–Pb dating and trace-element compositions were performed using ICPMSDataCal 8.3. Common Pb was corrected based on the method proposed by Andersen (2002) [5] with concordia diagrams and weighted mean calculations made using Isoplot 4.5 [6].

References:

1. Hu, Z.C.; Gao, S.; Liu, Y.S.; Hu, S.H.; Chen, H.H.; Yuan, H.L. Signal enhancement in laser ablation ICP-MS by addition of nitrogen in the central channel gas. *Journal of Analytical Atomic Spectrometry* **2008**, *23*, 1093–1101.
2. Wiedenbeck, M.; Hanchar, J.M.; Peck, W.H.; Sylvester, P.; Valley, J.; Whitehouse, M.; Kronz, A.; Morishita, Y.; Nasdala, L.; Fiebig, J.; Franchi, I.; Girard, J.P.; Greenwood, R.C.; Hinton, R.; Kita, N.; Mason, P.R.D.; Norman, M.; Ogasawara, M.; Piccoli, P.M.; Rhede, D.; Satoh, H.; Schulz-Dobrick, B.; Skar, O.; Spicuzza, M.J.; Terada, K.; Tindle,

- A.; Togashi, S.; Vennemann, T.; Xie, Q.; Zheng, Y.F. Further characterisation of the 91500 zircon crystal. *Geostandards and Geoanalytical Research* **2004**, *28*, 9–39.
3. Jackson, S.E.; Pearson, N.J.; Griffin, W.L.; Belousova, E.A. The application of laser ablation-inductively coupled plasma-mass spectrometry to in situ U–Pb zircon geochronology. *Chemical Geology* **2004**, *211*, 47–69.
 4. Liu, Y.S.; Hu, Z.C.; Zong, K.Q.; Gao, C.G.; Gao, S.; Xu, J.; Chen, H.H. Reappraisal and refinement of zircon U–Pb isotope and trace element analyses by LA-ICP-MS. *Chinese Science Bulletin* **2010**, *55*, 1535–1546.
 5. Andersen, T. Correction of common lead in U–Pb analyses that do not report ^{204}Pb . *Chemical Geology* **2002**, *192*, 59–79.
 6. Ludwig, K.R. User's Manual for Isoplot 3.00: A Geochronological Toolkit for Microsoft Excel. *Berkeley Geochronology Center Special Publication* **2003**, No. 4, 70 pp.