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Data Repository

Appendix I. Analytical methods.

Table S1. Zircon LA-ICPMS U-Pb data for amphibolites and orthogneisses from the Huangyuan and Maxianshan Groups.

Table S2. Zircon LA-ICPMS trace element analyses (ppm) for amphibolites and orthogneisses from the Huangyuan and Maxianshan Groups.

Table S3. Zircon LA-MC-ICPMS Hf-isotope data for amphibolites and orthogneisses from the Huangyuan and Maxianshan Groups.

Table S4. Whole-rock composition of representative amphibolites and orthogneisses from the Huangyuan and Maxianshan Groups.

Figure S1. Variation of selected trace elements with Zr for amphibolites from the Huangyuan and Maxianshan Groups.

Figure S2. Discrimination diagrams with bearing on crustal contamination and fractional crystallization for protoliths of amphibolites from the Huangyuan and Maxianshan Groups.

Appendix I. Analytical methods

1. Zircon cathode luminescence (CL) imaging

Sample processing for zircon separation involved crushing, initial heavy liquid and subsequent magnetic separation. Representative zircon grains were hand-picked and mounted on adhesive tape, embedded in epoxy resin, polished to about half their size and photographed in reflected and transmitted light. Zircon structures were studied by CL imaging of zircon grains using a Gatan MonoCL4 instrument mounted on an FEI Nova NanoSEM 450 scanning electron microscope at the Key Laboratory of Deep-Earth Dynamics of Ministry of Natural Resources, Institute of Geology, Chinese Academy of Geological Sciences, Beijing.

2. Zircon U-Pb geochronology

Zircon U-Pb dating was carried out at the State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan, using an Agillent 7500a ICP-MS with a GeoLas2005 193 nm ArF-excimer laser-ablation system. Data were collected using a 32µm spot. Detailed operating conditions for the laser ablation system and the ICP-MS instrument and data reduction follow Liu et al. (2008; 2010a; 2010b). Helium was applied as a carrier gas. Argon was used as the make-up gas and mixed with the carrier gas via a T-connector before entering the ICP. Nitrogen was added into the central gas flow (Ar+He) of the Ar plasma to decrease the detection limit and improve precision (Hu et al., 2008a; Liu et al., 2010b). Each analysis incorporated a background acquisition of ~20–30 s (gas blank) followed by 50 s data acquisition from the sample. The Agilent Chemstation was utilized for the acquisition of each individual analysis. Off-line selection and integration of background and analyte signals, and time-drift correction and quantitative calibration for trace element analyses and U-Pb dating were performed by *ICPMSDataCal* (Liu et al., 2008, 2010a).

Zircon 91500 was used as external standard for U-Pb dating, and was analyzed twice every 5 analyses. Time-dependent drift of U-Th-Pb isotopic ratios was corrected using a linear interpolation with time for every five analyses according to the variations of 91500 (i.e., 2 zircon 91500 + 5 samples + 2 zircon 91500; Liu et al., 2010a). Preferred U-Th-Pb isotopic ratios used for 91500 are from Wiedenbeck et al. (1995). Uncertainty of preferred values for the external standard 91500 was propagated to the ultimate results of the samples. Zircon standard GJ-1 was used as second external standard and analyzed as an unknown sample to verify the accuracy of the method. The obtained U-Pb ages for zircon GJ-1 are consistent with the preferred values (Liu et al. 2010b) within ~2% uncertainty (2σ , n = 32) by external calibration against zircon standard 91500. Concordia diagrams and weighted mean calculations were made using Isoplot/Ex_ver3 (Ludwig. 2003). All U-Pb isotopic data are listed in **Table S1**. Trace element data of zircon grains from the samples are listed in **Table S2**.

3. Zircon Lu-Hf isotopic analysis

Experiments were conducted using a Neptune Plus MC-ICP-MS (Thermo Fisher Scientific, Germany) in combination with a Geolas 2005 excimer ArF laser ablation system (Lambda Physik, Göttingen, Germany) that was hosted at the state Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan. The energy density of laser ablation that was used in this study was 5.3 J cm⁻². Helium was used as the carrier gas within the ablation cell and was merged with argon (makeup gas) after the ablation cell. As demonstrated by our previous study, for the 193 nm laser a consistent 2-fold signal enhancement was achieved in helium than in argon gas (Hu et al., 2008b). We used a simple Y junction downstream from the sample cell to add small amounts of nitrogen (4 ml min⁻¹) to the argon makeup gas flow (Hu et al., 2008b). Compared to the standard arrangement, the addition of nitrogen in combination with the use of the newly designed X skimmer cone and Jet sample cone in Neptune Plus improved the signal intensity of Hf, Yb and Lu by a factor of 5.3, 4.0 and 2.4, respectively. All data were acquired on zircon in single spot ablation mode at a spot size of 44um in this study. Each measurement consisted of 20 s of acquisition of the background signal followed by 50 s of ablation signal acquisition. Detailed operating conditions for the laser ablation system and the MC-ICP-MS instrument and analytical method are the same as description by Hu et al. (2012).

The major limitation to accurate in situ zircon Hf isotope determination by LA-MC-ICP-MS is the very large isobaric interference from ¹⁷⁶Yb and, to a much lesser extent ¹⁷⁶Lu on ¹⁷⁶Hf (Woodhead et al., 2004). The under- or over-estimation of the β_{Yb} value would undoubtedly affect the accurate correction of ¹⁷⁶Yb and thus the determined ¹⁷⁶Hf/¹⁷⁷Hf ratio. We applied the directly obtained β_{Yb} value from the zircon sample itself in real-time (Liu et al., 2010a). The 179 Hf/ 177 Hf and 173 Yb/ 171 Yb ratios were used to calculate the mass bias of Hf (β_{Hf}) and Yb (β_{Yb}), which were normalized to 179 Hf/ 177 Hf = 0.7325 and 173 Yb/ 171 Yb = 1.1248 (Blichert-Toft et al., 1997) using an exponential correction for mass bias. Interference of ¹⁷⁶Yb on ¹⁷⁶Hf was corrected by measuring the interference-free ¹⁷³Yb isotope and using 176 Yb/ 173 Yb = 0.7876 (McCulloch et al., 1977) to calculate 176 Yb/ 177 Hf. Similarly, the relatively minor interference of ¹⁷⁶Lu on ¹⁷⁶Hf was corrected by measuring the intensity of the interference-free ¹⁷⁵Lu isotope and using the recommended ${}^{176}Lu/{}^{175}Lu = 0.02656$ (Blichert-Toft et al., 1997) to calculate ${}^{176}Lu/{}^{177}Hf$. We used the mass bias of Yb (β_{Yb}) to calculate the mass fractionation of Lu because of their similar physicochemical properties. Off-line selection and integration of analyte signals, and mass bias calibrations were performed using ICPMSDataCal (Liu et al., 2010a). The Lu-Hf isotopic data are listed in Table S3.

4. Whole-rock geochemistry

Powdered samples were analyzed for major elements by X-ray fluorescence using a Rigaku RIX 2000 spectrometer at the Department of Geosciences, National Taiwan University, Taipei, China. The analytical uncertainties are generally better than 5% for all elements. Trace elements were determined on the Agilent 7500a ICP-MS at the State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan. The powdered samples were digested by HF + HNO₃ in Teflon bombs for analysis. Sample digestion for ICP-MS analysis and analytical precision and accuracy for trace elements follow Liu et al. (2008). Major and trace element compositions of representative amphibolites and orthogneisses from the Huangyuan Group and the Maxianshan Group are listed in **Table S4**.

REFERENCES CITED

- Blichert-Toft, J., Chauvel, C., and Albarède, F., 1997, Separation of Hf and Lu for high-precision isotope analysis of rock samples by magnetic sector-multiple collector ICP-MS: Contributions to Mineralogy and Petrology, v. 127, p. 248–260, <u>https://doi.org/10.1007/s004100050278</u>.
- Hu, Z.C., Gao, S., Liu, Y.S., Hu, S.H., Chen, H.H., and Yuan, H.L., 2008a, Signal enhancement in laser ablation ICP-MS by addition of nitrogen in the central channel gas: Journal of Analytical Atomic Spectrometry, v. 23, p. 1093–1101, <u>https://doi.org/10.1039/b804760j</u>.
- Hu, Z.C., Liu, Y.S., Gao, S., Hu, S.H., Dietikerc, R., and Günther, D., 2008b, A local aerosol extraction strategy for the determination of the aerosol composition in laser ablation inductively coupled plasma mass spectrometry: Journal of Analytical Atomic Spectrometry, v. 23, p. 1192–1203, <u>https://doi.org/10.1039/b803934h</u>.
- Hu, Z.C., Liu, Y.S., Gao, S., Liu, W.G., Zhang, W., Tong, X.R., Lin, L., Zong, K.Q., Li, M., Chen, H.H., Zhou, L., and Yang, L., 2012, Improved in situ Hf isotope ratio analysis of zircon using newly designed X skimmer cone and Jet sample cone in combination with the addition of nitrogen by laser ablation multiple collector ICP-MS: Journal of Analytical Atomic Spectrometry, v. 27, p. 1391–1399, <u>https://doi.org/10.1039/c2ja30078h</u>.
- Liu, Y.S., Zong, K.Q., Kelemen, P.B., and Gao, S., 2008, Geochemistry and magmatic history of eclogites and ultramafic rocks from the Chinese continental scientific drill hole: Subduction and ultrahigh-pressure metamorphism of lower crustal cumulates: Chemical Geology, v. 247, p. 133–153, <u>https://doi.org/10.1016/j.chemgeo.2007.10.016</u>.
- Liu, Y.S., Gao, S., Hu, Z.C., Gao, C.G., Zong, K.Q., and Wang, D.B., 2010a, Continental and oceanic crust recycling-induced melt-peridotite interactions in the Trans-North China Orogen: U-Pb dating, Hf isotopes and trace elements in zircons of mantle xenoliths: Journal of Petrology, v. 51, p. 537–571, <u>https://doi.org/10.1093/petrology/egp082</u>.
- Liu, Y.S., Hu, Z.C., Zong, K.Q., Gao, C.G., Gao, S., Xu, J., and Chen, H.H., 2010b, Reappraisement and refinement of zircon U-Pb isotope and trace element analyses by LA-ICP-MS: Chinese Science Bulletin, v. 55, p. 1535–1546, <u>https://doi.org/10.1007/s11434-010-3052-4</u>.

- Ludwig, K.R., 2003. ISOPLOT 3.00: A Geochronological Toolkit for Microsoft Excel: Berkeley Geochronology Center, California, Berkeley, p. 39.
- McCulloch, M.T., Rosman, K.J.R., and De Laeter, J.R., 1977, The isotopic and elemental abundance of ytterbium in meteorites and terrestrial samples: Geochimica et Cosmochimica Acta, v. 41, p. 1703–1707, https://doi.org/10.1016/0016-7037(77)90202-2.
- Wiedenbeck, M., Alle, P., Corfu, F., Griffin, W.L., Meier, M., Oberli, F., Quadt, A.V., Roddick, J.C., and Spiegel, W., 1995, Three natural zircon standards for U-Th-Pb, Lu-Hf, trace element and REE analyses: Geostandards and Geoanalytical Research, v. 19, p. 1–23, <u>https://doi.org/10.1111/j.1751-908X.1995.tb00147.x</u>.
- Woodhead, J., Hergt, J., Shelley, M., Eggins, S., and Kemp, R., 2004, Zircon Hf-isotope analysis with an excimer laser, depth profiling, ablation of complex geometries, and concomitant age estimation: Chemical Geology, v. 209, p. 121–135, <u>https://doi.org/10.1016/j.chemgeo.2004.04.026</u>.



