Supplementary

Genesis of Early–Middle Jurassic Intrusive Rocks in the Erguna Block (NE China) in Response to the Late-Stage Southward Subduction of the Mongol–Okhotsk Oceanic Plate: Constraints from Geochemistry and Zircon U–Pb Geochronology and Lu–Hf Isotopes

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Analytical techniques

1. Zircon U–Pb Dating and Trace Element Analyses

Zircon crystals from four samples were separated from whole-rock using conventional heavy liquid and magnetic separation techniques, followed by handpicking under a binocular microscope. The handpicked zircons were mounted in epoxy and polished at the Langfang Regional Geological Survey, Langfang, China. To reveal their internal structures, all zircons were examined employing transmitted and reflected light photomicrographs with an optical microscope (Olympus BX51) at Jilin University, Changchun, China, as well as cathodoluminescence (CL) images obtained using a LEO1450VP scanning electron microscope (SEM; 15 kV, 1.1 nA) housed at Beijing Gaonian Pilot Technology Co. Ltd, Beijing, China.

The zircon U–Pb isotope and trace element analyses were performed using a laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) at the Key Laboratory of Mineral Resources Evaluation in Northeast Asia, Ministry of Land and Resources of China, Changchun, China, using an Agilent 7500a ICP-MS, and a GeoLas 200M 193 nm ArF excimer laser ablation system. The sample was stuck onto a slide with two-sided glue tape under binocular and a PVC ring was put on the slide. Then, the mixture of epoxy and hardener was put into a PVC ring. When totally solidified, the ring was peeled off from slide and polished to an even sample surface. Before analysis, the surface was cleaned using 3% (v·v−1) HNO3. ICP-MS measurements were carried out using peak jumping (1 point per peak) mode. Each spot analysis consisted of approximately 30 s of background acquisition and 40 s sample data acquisition. Zircon 91500 was used as external reference material to correct for isotopic fractionation and calculate the isotopic compositions. Zircon GJ-1 was used as a secondary reference material to validate the result of age measurements/calculations. The trace element concentrations were calibrated by using 29Si as internal standard and zircon NIST 610 as external reference material according to Yuan et al. [1]. Five sample analyses were followed by one zircon 91500 measurement, and ten sample analyses were followed by one zircon GJ-1 measurement, one zircon 91500 measurement, and one NIST 610 measurement. Our analyses yielded weighted mean 206Pb/238U ages of reference material GJ-1 of 606 ± 5 Ma (n = 9, MSWD = 0.8, 2s), which are consistent with the recommended values (Horstwood et al. [2]). Isotope ratios were calculated using Glitter v. 4.0 (Macquarie University), and correction for common Pb was made following Anderson [3]. Concordia diagrams and weighted mean calculations were derived using Isoplot v. 3.0 [4]. Uncertainties on individual analyses by LA-ICP-MS are quoted at the 2s level, and errors on pooled ages are quoted at the 95% (2s) confidence level. The detailed metadata of LA-ICP-MS analyses are presented in Supplementary Table 9.

2. Whole-Rock Major and Trace Element Analyses

For whole-rock major and trace element compositions, seven samples were analysed at the ALS Chemex (Guangzhou) Co. Ltd., Guangzhou, China. For major element analyses, a calcined or ignited sample (0.9 g) was added to 9.0 g of lithium borate flux (50% Li2B4O7-LiBO2), mixed well, and then fused in an auto fluxer at temperatures of 1050–1100°C. A flat molten glass disc was produced from the resulting melt, and the disc was then analysed using XRF. For trace element analyses, a prepared sample (0.200 g) was added to a 0.90 g of lithium metaborate flux, mixed well, and then fused in a furnace at 1000°C. The resulting melt was then cooled and dissolved in 100 mL of 4% HNO3. This solution was analysed using an ICP-MS. Two international reference material samples (kinzingite SARM-45 and Canadian diorite gneiss SY-4) were used as the calibration standards (listed in Supplementary Table 5). Details of the analytical procedures are given by Gao et al. [5]. The analytical precision was better than 5% for the major elements and better than 10% for the trace elements. The detailed metadata of ICP-MS analyses are presented in Supplementary Table 10.

3. In situ Zircon Lu–Hf Isotopic Analyses

*In situ* zircon Lu–Hf isotopic analyses were performed at the Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing, China, employing a Thermo Fisher Scientific Neptune MC-ICP-MS equipped with a 193 nm Lambda Physik COMpex ArF excimer laser ablation system. The analyses were undertaken on zircons that had been previously analyzed during U–Pb dating using a single-spot ablation mode with a spot size of 40–60 μm. Details of the analytical procedures are given by Wu et al. [6]. The determined 176Hf/177Hf ratios of the Mud Tank standard zircon was 0.282514 ± 0.000002, the GJ-1 standard zircon was 0.282022 ± 0.000003, that were in good agreement within errors with the recommendedHf values [7]. The detailed metadata of MC-ICP-MS analyses are presented in Supplementary Table 11.

**Table S9.** Metadata for LA-ICPMS zircon U-Pb and trace element analyses of this study.

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| **Laboratory and Sample Preparation** | |
| Laboratory name | Key Laboratory of Mineral Resources Evaluation in Northeast Asia, Ministry of Land and Resources of China, Changchun, China |
| Sample type/mineral | Igneous zircons |
| Sample preparation | Conventional mineral separation, 1-inch resin mount, 1 lm polish to finish |
| Imaging | CL, LEO1450VP scanning electron microscope, 15 kV, 1.1 nA |
| **Laser Ablation System** | |
| Make, Model and type | GeoLas 200M 193 nm ArF excimer laser ablation system |
| Laser wavelength (nm) | 193 nm |
| Energy | 100 mJ cm-2 |
| Pulse width (ns) | 20 nm |
| Repetition rate (Hz) | 10 Hz |
| Ablation duration (s) | 40 s |
| Ablation pit depth | 16 mm pit depth, measured using an optical system (MicroLas) |
| Spot diameter (μm) | 30 μm |
| Sampling mode/pattern | Static spot ablation |
| Carrier gas | 100% He in the cell |
| Cell carrier gas flow (l min-1) | 0.67 l min-1 |
| **ICP-MS Instrument** | |
| Make, Model and type | Agilent 7500a ICP-MS |
| Data acquisition protocol | Time Resolved Analysis (TRA) |
| RF power (W) | 1320 W |
| Make-up gas flow (l min-1) | 0.85 l min-1 |
| Detection system | Quadrupole |
| Scanning mode | Peak hopping, 1 point per peak |
| Masses measured | 202–207, 235, 238 |
| Dwell time (ms) per isotope  Trace element analysis U-Pb age analysis 29Si 204Pb, 206Pb, 207Pb and 208Pb 232Th 238U | 10 ms  10 ms  62.76 ms (7.31, 21.74, 23.71, 10) 10 ms 10 ms |
| Quadruple settling time (ms) | 1 ms |
| Sweeps | 2 |
| **Data Processing** | |
| Gas blank | 30 s on-peak zero subtracted |
| Calibration strategy | 91500 used as primary reference material, NIST 610 and GJ-1 used as secondaries/validation |
| Reference Material information | 91500 [8] as external reference material (dating) GJ-1 [9] as secondary reference material (dating) 29Si as internal standard (trace element concentration measurement) NIST 610 [10] as external reference material (trace element concentration measurement) |
| Data processing package used/Correction for LIEF | Glitter v. 4.0 (Macquarie University, Australia) program was used for data reduction. LIEF correction assumes reference material and samples behave identically. Isoplot v. 3.0 [4] program was used for concordia diagrams and weighted mean calculations. |
| Mass discrimination | Tl-U tracer solution used for initial mass bias correction with 207Pb/206Pb and 206Pb/238U additionally normalized to reference material. |
| Common-Pb correction, composition and uncertainty | Common-Pb correction applied to the data was made following Anderson [3]. |
| Uncertainty level and propagation | Ages are quoted at 2s absolute, propagation is by quadratic addition. Reproducibility and age uncertainty of reference material and common-Pb composition uncertainty are propagated where appropriate. |
| Quality control/Validation | 91500 – Wtd ave 206Pb/238U age = 1066.0 ± 13 Ma (n = 20, MSWD = 0.072, 2s)  GJ-1 – Wtd ave 206Pb/238U age = 605.5 ± 6.2 Ma (n = 9, MSWD = 0.095, 2s)  Systematic uncertainty for propagation is 2% (2s). |

**Table S10.** Metadata for whole-rock trace element analyses of this study.

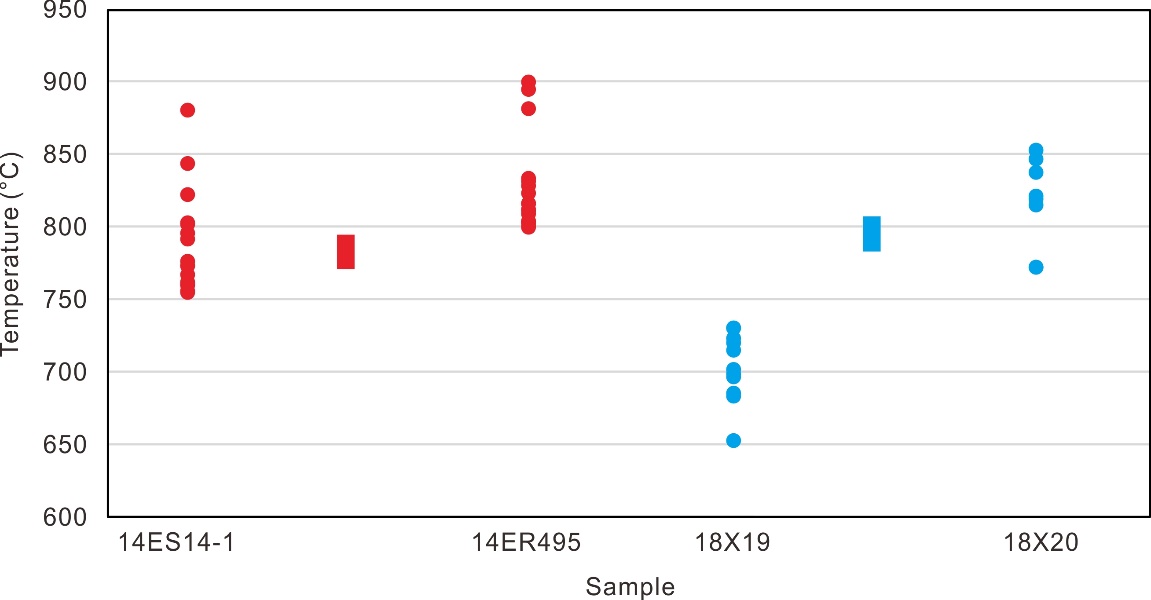
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| **Laboratory and Sample Preparation** | |
| Laboratory name | ALS Chemex (Guangzhou) Co. Ltd., Guangzhou, China |
| Sample type/mineral | Intrusive rocks |
| Sample preparation | Conventional whole-rock solution |
| Imaging | Optical microscope |
| **ICP-MS Instrument** | |
| Make, Model and type | ELAN 6100DRC ICP-MS |
| Nebulizer gas flow | 0.60-0.80 l min-1 |
| Auxiliary gas flow | 0.60-0.90 l min-1 |
| Plasma gas flow | 11-15 l min-1 |
| Lens voltage | 10 V |
| ICP RF power | 1300 W |
| Auto lens | On |
| **Data Processing** | |
| Calibration strategy | SARM-45 and BHVO-2 used as reference materials |
| Reference Material information | Kinzingite SARM-45 [11] Canadian diorite gneiss SY-4 [12] |
| Quality control/Validation | See Supplementary Table 5, better than 5% for the major elements and better than 10% for the trace elements |

**Table S11.** Metadata for zircon Lu–Hf isotopic analyses of this study.

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| --- | --- |
| **Laboratory and Sample Preparation** | |
| Laboratory name | Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing, China |
| Sample type/mineral | Igneous zircons |
| Sample preparation | Conventional mineral separation, 1-inch resin mount, 1 lm polish to finish |
| Imaging | CL, LEO1450VP scanning electron microscope, 15 kV, 1.1 nA |
| **Laser ablation system** | |
| Make, Model and type | Lambda Physik COMpex |
| Laser wavelength (nm) | 193 nm |
| Energy | 100 mJ cm-2 |
| Repetition rate (Hz) | 5–15 Hz |
| Spot diameter (μm) | 40–60 μm |
| Carrier gas | 100% He in the cell |
| Cell carrier gas flow (l min-1) | 0.8–0.9 l min-1 |
| **ICP-MS Instrument** | |
| Make, Model and type | Neptune MC-ICPMS |
| RF forward power | 1303 W |
| RF reflected power | <3 W |
| Cooling gas | 15.2 l min-1 |
| Auxiliary gas | 0.6 l min-1 |
| Sample gas | ∼ 1.2 l min-1 |
| Extraction | -1997 V |
| Focus | -656 V |
| Detection system | Nine Faraday collectors |
| Acceleration voltage | 10 kV |
| Interface cones | Nickel |
| Spray chamber | Glass cyclonic |
| Nebulizer type | Micromist PFA nebulizer |
| Sample uptake rate | 50 μl min-1 |
| Uptake mode | Free aspiration |
| Instrument Resolution | ∼ 400 (low) |
| Mass analyzer pressure | 4–8×10-9 mbar |
| Typical sensitivity on 180Hf | ∼ 16 V/ppm (10-11 Ω resistors) |
| Sampling mode | One run = 9 blocks of 10 cycles for solution  One run = 1 block of 200 cycles for laser |
| Integration time | 4.194 s for solution 0.131 s for laser |
| Baseline/background determination | ca. 1 min on peak in 2% HNO3 |
| **Data Processing** | |
| Calibration strategy | Mud Tank and GJ1 used as reference materials |
| Reference Material information | Mud Tank [13]  GJ-1 (Australian Macquarie University) |
| Quality control/Validation | Mud Tank – 76Hf/177Hf ratios of 0.282514 ± 0.000002  GJ1 – 76Hf/177Hf ratios of 0.282022 ± 0.000003 |

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**Figure 1.** Plot of zircon crystallization temperature and zircon saturation temperature. The solid circles represent crystallization temperatures calculated from single zircon crystals by adopting equation (1); the solid bars indicate the zircon saturation temperatures calculated from whole-rocks by using equation (2).

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