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Supplemental Material

Text S1: U–Pb, $^{40}\text{Ar}/^{39}\text{Ar}$, and Bayesian modeling analytical methods.

Table S1: Summary of radioisotopic ages from Qingshan Group, Jiaolai Basin, China.

Table S2: Summary of ages determined for Barremian-Aptian boundary and Chron M0r.

Tables S3, S4, and S5: Complete U–Pb CA-IDIRMS, CA-IDTIMS and $^{40}\text{Ar}/^{39}\text{Ar}$ data in three different labs.

Timing of magnetochron M0r, the Barremian-Aptian boundary, and geologic implications: SUPPLEMENTARY MATERIALS

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METHODS

U–Pb ANALYTICAL METHODS

The analytical procedure in the Department of Earth and Planetary Sciences at the University of California, Davis (UCD) is as follows. Zircon grains were firstly annealed at 900° for 48 h to restore the crystallinity of the low to medium radiation damaged parts ([Mattinson, 2005](#)). Then the U-Pb laser-ablation screening analyses were performed to exclude any detrital or inherited zircon grains, and the youngest populations of zircons were selected for the subsequent CA-ID-IRMS analysis. Individual zircon crystals were transferred into 3 ml PFA vials, rinsed in acetone followed by 3 M HNO₃. Individual zircon crystals were loaded into separate microcapsules, and then ~3 µL of 3 M HNO₃ and ~100 µL of concentrated HF were added. The samples were leached in a pressurized vessel at 190° for 15 h ([Huyskens et al., 2016](#)). The residual zircons were then transferred into the corresponding PFA vials and rinsed with MilliQ water, followed by 6 M HCl on a hotplate. Subsequently, the crystals were rinsed with MilliQ water and 3 M HNO₃. For dissolution at 220° for 48 h, the residual grains were placed into the individual microcapsules, and then ~3 µL of 3 M HNO₃ and ~100 µL of concentrated HF were added as well as a mixed ²⁰²Pb-²⁰⁵Pb-²³³U-²³⁶U tracer solution ([Huyskens et al., 2016](#)). After dissolution, the solutions were dried down and redissolved in 2.5 M HCl overnight, and then Pb and U were separated from the matrix elements by standard HCl ion exchange chemistry ([Krogh,](#)

1973). The Pb and U isotopic measurement were performed on the Triton PLUS TIMS and the Neptune Plus Multi-collector inductively-coupled plasma mass spectrometer (MC-ICP-MS) using an ESI APEX introduction system, respectively. U-Pb dates and uncertainties were calculated using the algorithms of Schmitz and Schoene (2007) and the U decay constants of Jaffey et al. (1971). The weighted mean $^{206}\text{Pb}/^{238}\text{U}$ ages were calculated using Isoplot excel macro Ludwig (2012). More details of the analytical methods can be found in Liao et al. (2020). The Temora zircon reference material yielded a $^{206}\text{Pb}/^{238}\text{U}$ age of 417.48 ± 0.11 Ma (MSWD = 1.2, n = 4) during the course of this study.

The analytical procedure in the Department of Geosciences at the Boise State University (BSU) is as follows. Zircon grains were placed in a muffle furnace at 900 °C for 60 h to anneal minor radiation damage. Individual grains were hand-picked, mounted, polished, and imaged by cathodoluminescence (CL) on a JEOL JSM-300 scanning electron microscope and Gatan MiniCL. Based on the compiled images, the sites of spot analyses were selected for laser ablation–inductively coupled plasma–mass spectrometry (LA-ICPMS) using a X-Series II quadrupole ICP-MS and New Wave Research UP-213 Nd:YAG UV (213 nm) laser ablation system following methodology of Macdonald et al. (2018). Based on LA-ICPMS data and CL images, single zircon grains were selected for CA-IDTIMS analysis. Individual zircon grains were chemically abraded in concentrated HF for 12 h at 190 °C to mitigate open system behavior and remove inclusions (Mattinson, 2005). Then the zircon fragments were dissolved in Parr bombs in 29 M HF at 220 ° for 48 h, and spiked with EARTHTIME mixed ^{202}Pb - ^{205}Pb - ^{233}U - ^{235}U (ET2535) isotope dilution tracer. Dissolved zircon solutions were subsequently dried to fluorides, and re-dissolved in 6 M HCL at 180 °C for 12 h. U and Pb were separated and isolated by anion exchange column chromatography using 50 µl columns and AG-1 X8 resin (Krogh,

1973). Isotope ratios were measured using GV instruments IsoProbe-T multicollector thermal ionization mass spectrometer. U-Pb dates and uncertainties were calculated using the algorithms of [Schmitz and Schoene \(2007\)](#) and the U decay constants of [Jaffey et al. \(1971\)](#).

$^{40}\text{Ar}/^{39}\text{Ar}$ ANALYTICAL METHODS

Sanidine crystals (200–350 μm) were wrapped in aluminum foil, alongside the Fish Canyon sanidine (FCs) standard, into 2.4-cm aluminum disks. The disks were irradiated in the Cadmium-Lined-In Core-Irradiation Tube (CLICIT) for 80 hours at the Oregon State University reactor. $^{40}\text{Ar}/^{39}\text{Ar}$ single crystal total fusion analyses were performed using a 60 W CO_2 laser at the University of Wisconsin-Madison WiscAr laboratory. The extracted gases were cleaned with one SAES GP-50 getter for 60 s and a cryotrap at -125°C for 30 s, and then measured using a Nu Instruments Noblesse multi-collector mass spectrometer following procedures of [Jicha et al. \(2016\)](#). Argon beam intensities were corrected for the baseline, blank, radioactive decay, and detector intercalibration, the latter of which was done using an in-house cocktail gas [Jicha et al. \(2016\)](#). The $^{40}\text{Ar}/^{39}\text{Ar}$ weighted mean ages were calculated relative to 28.201 ± 0.046 Ma FCs ([Kuiper et al. 2008](#)) with the ^{40}K decay constant [Min et al. 2000](#). The atmospheric $^{40}\text{Ar}/^{36}\text{Ar}$ value was used from [Lee et al. \(2006\)](#), and the complete $^{40}\text{Ar}/^{39}\text{Ar}$ isotope data are available in [Table S1](#).

BAYESIAN MODELING

Bayesian age-depth model is generated using the modifiedBChron package of [Trayler et al. \(2020\)](#). The positions of four tuffs in the model represent their height above the base of the Houkuang Formation. A 5 m thickness was assumed for each tuff, and age-depth positions were predicted every 5 m. The dates used to determine the weighted mean age in [Figure 2](#) are chosen as inputs to produce the probability distribution for each sample. All $^{206}\text{Pb}/^{238}\text{U}$ and $^{40}\text{Ar}/^{39}\text{Ar}$

dates were integrated into four tuff age likelihood inputs into the Bayesian modeling. The median age-depth model is generated with ten thousand age-depth paths using Markov Chain Monte Carlo technique, and presented as 95% highest density interval (HDI), which is analogous to 2σ uncertainty (Trayler et al., 2020). The posterior distributions superimposed in black on the original likelihoods in color (Figure 2).

METHODS REFERENCES

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