Data Repository

Tables DR1–DR6, including zircon U-Pb, $\delta^{13}C_{carb}$, $\delta^{18}O_{carb}$, $\delta^{13}C_{org}$, TOC (total organic carbon), and $\delta^{18}O_{apatite}$ data.

METHODS

1. U-Pb geochronology

Samples of tuff and tuffaceous sandstone collected for zircon geochronology were on the order of 4 to 6 kg in weight and were carefully excavated along narrow, bedding-parallel strips no wider than 10 cm to avoid mixing between tuffaceous layers of different age to the extent possible. The majority of samples were clay-rich tuffs (bentonitic) and were processed in the lab by soaking in water for 48 hours, followed by complete liquefaction in a blender and gradual clay disintegration in a sonic dismembrator device (Hoke et al. 2014). Heavy-mineral concentrates were obtained using magnetic as well as high-density liquid separation. A few clay-poor samples (tuffaceous sandstones) were processed in the lab by manual sledging and pulverization in a Shatterbox®, before heavy mineral concentration. Final zircon selection was carried out by hand picking under a binocular microscope.

Samples contained to varying degrees mixed populations of zircon characterized by a variety of grain sizes and morphologies. These ranged from equant and sub-equant grains to prismatic and acicular zircon with high aspect ratios and up to 500 μm in length. Preference in zircons selection was given to populations of sharply faceted, prismatic/acicular zircon that contained elongate glass (melt) inclusions parallel to their long axis (Fig. DR1). Our past experience has demonstrated that these grains typically yield the youngest dates in samples characterized by mixed zircon populations and this has proven an efficient screening technique for xenocrystic and/or far transported detrital zircon grains (Ramezani et al., 2011). The oldest measured zircon date in this study was 252.62 ± 0.33 Ma (Table DR1).

Zircons selected for U-Pb analysis were pre-treated by a chemical abrasion technique modified after Mattinson (2005), which involved thermal annealing in a 900 °C furnace for 60 hours, followed by partial dissolution (leaching) in concentrated hydrofluoric acid (HF) in order to mitigate the effects of Pb-loss in zircon that often result in anomalously young dates. For leaching, annealed zircons were loaded with ~75 µl of 29 M HF into 200 µl FEP Teflon® microcapsules, placed within a high-pressure Parr® vessel and left in a 210 °C oven for 12-13 hours. The leached grains were transferred into 3 ml Savillex® FEP beakers and fluxed in

successive steps in a dilute HNO_3 solution and in 6N HCl over a hot plate (1 hour per step), with each step followed by agitation in an ultrasonic bath (for 1 hour) and rinsing with several milliliters of ultra-pure water to remove the leachate. This leach schedule is considered aggressive for zircons of pre-Cenozoic age and/or normal to high U contents, as it increases the risk of total grain disintegration during the leach step. But, it has proven an effective remedy for persistent Pb loss compared to studies that employ a lower temperature chemical abrasion on similar zircons (see Baresel et al., 2017). Thoroughly rinsed zircon grains were loaded back into their microcapsules, spiked with the EARTHTIME ET2535 mixed ^{202}Pb-^{205}Pb-^{233}U-^{235}U isotopic tracer (Condon et al., 2015; McLean et al., 2015) and dissolved completely in 29M HF at 210 °C for 48 hours.

Dissolved Pb and U were chemically separated using a miniaturized HCl-based ion-exchange chemical procedure modified after Krogh (1973), using 50 µl columns of AG1x8 anion-exchange resin. Purified Pb and U were loaded with a silica gel – H_3PO_4 emitter solution (Gerstenberger and Haase, 1997) onto single degassed Re filaments and their isotopic ratios were measured on the Isotopx X62 multi-collector thermal ionization mass spectrometer equipped with a Daly photomultiplier ion counting system at MIT. Pb isotopic measurements were made in a peak-hopping mode on the ion counter, whereas U isotopes were measured as UO_2^+ in a static mode on three Faraday detectors simultaneously.

A total of 80 single zircons were analyzed from 7 tuff and tuffaceous sandstone beds from the upper Permian and lower Triassic strata of the Penglaitan section. Complete Pb and U isotopic data are given in Table DR1. Data reduction, as well as calculation of dates and propagation of uncertainties were accomplished using the Tripoli and U-Pb_Redux applications (Bowring et al., 2011; McLean et al., 2011). Measured isotopic ratios were corrected for mass-dependent isotope fractionation in the mass spectrometer using the tracer $^{202}Pb/^{205}Pb$ and $^{233}U/^{235}U$ isotopic ratios, as well as for U and Pb contributions from the spike and laboratory blanks. Common Pb in the analyses averaged 0.34 pg, all of which was considered laboratory blank, and its isotopic composition was determined from long-term measurements of the total procedural blank in the lab (see Table DR1 footnotes). Pb/U ratios were also corrected for initial ^{230}Th disequilibrium in magma using a magma initial Th/U model ratio of 2.8 ± 1.0 (2σ). This range of Th/U ratios encompasses all likely compositions of the magma source of an intermediate to felsic tuff (Machlus et al., 2015). Pb isotopic ratios were corrected for isobaric interferences from Tl and $BaPO_2$ on mass 205 by monitoring masses 203 and 201, respectively, and using natural isotopic abundances of ^{138}Ba and ^{205}Tl. Measured U isotopic ratios were also corrected for isobaric interference of $^{233}U^{18}O^{16}O$ on $^{235}U^{16}O^{16}O$ using an $^{18}O/^{16}O$ ratio of 0.00205 ± 0.00004 (2σ), which has been determined by long-term measurements of 272/270 ratio from large U loads. The present-day natural U isotopic composition of 137.818 ± 0.044 (2σ) was used in data reduction following Heiss et al. (2012).

In general, $^{206}Pb/^{238}U$ dates are considered the most precise and accurate in high-precision U-Pb geochronology as they are independent of suspected inaccuracies in the ^{235}U decay constant (Mattinson, 2010; Schoene et al., 2006), that potentially compromises the accuracy of $^{207}Pb/^{235}U$ or $^{207}Pb/^{206}Pb$ dates. Since the presence of xenocrystic/antecrystic or detrital (reworked) zircon in our tuffaceous samples cannot be ruled out, our sample ages are derived

from the weighted mean ^{206}Pb/^{238}U date of a statistically coherent cluster of the youngest zircon analyses in each sample, after excluding older analyses that are considered xenocrystic or detrital. The youngest cluster comprises between 39% (sample PL13-01) to 100% (samples LP02-13 and PL15-03) of the total analyses from each sample (Table DR1), rendering some calculated dates more subjective than others. No analysis was excluded from age calculation for being interpreted as a 'young outlier'. Uncertainties in calculated ^{206}Pb/^{238}U dates are reported at 95% confidence level (Table 1 and Fig. 5) and in the ± X/Y/Z Ma format, where X is the internal (analytical) uncertainty in the absence of all external errors, Y incorporates the U-Pb tracer calibration errors and Z includes the latter as well as the decay constant errors of Jaffey et al. (1971). The external uncertainties must be taken into account if the results are to be compared with U-Pb dates obtained in other laboratories with different tracers, with different techniques, or ones derived from other isotopic chronometers (e.g., ^{40}Ar/^{39}Ar). However, for establishing a chronology based on the results of this study alone, or for making comparisons to those from other studies that use the same U-Pb isotopic tracer, only the analytical uncertainties (X) need to be considered.

The three dated samples that encompass the uppermost Permian at Penglaitan (PL13-04, LP02-09 and PL13-01) all overlap in their weighted mean ^{206}Pb/^{238}U dates within 2σ analytical uncertainties and are thus statistically indistinguishable (Fig. 2). In addition, an objective age for sample PL13-01 (Bed 141) that lies at the extinction horizon is difficult to calculate due to the abundance of older zircon (see above). Therefore, a more feasible age constraint for the extinction horizon (0 m) can be obtained by linear extrapolation between sample PL13-04 (Bed 130) at -27.2 m and sample PL15-03 (Bed 142) at +0.20 m, which is 251.939 ± 0.031 Ma. This yields a duration of 31 ± 31 k.y. for the end-Permian mass extinction.

In an alternative approach, we determined the age and duration of the extinction horizon by constructing a Bayesian age-depth model using the Bchron software package (Haslett and Parnell, 2008; Parnell et al., 2008) and based on the weighted mean dates of all 7 dated samples and their relative stratigraphic positions. The model is constructed similar to that of Baresel et al. (2017) and is shown with its median (solid line) and 95% confidence interval (shaded band) in Fig. 12. It yields an age of 251.940 ± 0.019 Ma and a duration of 19 +29/-19 k.y. for the end-Permian mass extinction. The latter age is indistinguishable from that calculated above using a simple linear extrapolation. However, the underlying Markov chain Monte Carlo rejection algorithm of Bchron generates significantly smaller age uncertainties at the extinction level.

Fig. DR1. Example of zircons selected for analysis from the Penglaitan tuffaceous sample PL13-01 (Bed 141) from 0.25m below the end-Permian mass extinction horizon

2. Carbon isotope and TOC analyses

A total of 321 samples were analyzed for $\delta^{13}C_{carb}$ and $\delta^{18}O_{carb}$, and 77 samples for $\delta^{13}C_{org}$ and TOC (total organic carbon), at the Nanjing Institute of Geology and Palaeontology, Chinese Academy of Sciences (Table DR2). Carbonate carbon isotope analyses were carried out following standard techniques (McCrea, 1950). For each of the 321 samples, ~1 g of powder was collected using a dental drill, with care taken to avoid visible diagenetic features. An aliquot of 80 to100 μg of sample powder was reacted with orthophosphoric acid for 150-200 s at 72 °C in a Kiel IV carbonate device. The evolved CO_2 gas was analyzed for $\delta^{13}C$ and $\delta^{18}O$ using a MAT-253 mass spectrometer. The analytical procedures (Boutton, 1991; Holser and Magaritz, 1992) for organic carbon isotopes were generally followed. A sequential kerogen extraction procedure was applied to all samples from the Penglaitan section. Rock powders (~10 g) were first decarbonated with 4M HCl at 60 ºC for >2 hours. Some samples were further treated with an HCl/HF (1:2 mixture) at 60 ºC for >2 hours to remove silicates. In the final digestion step, the sample was treated with 4M HCl and heated at 60 ºC for 1 h. The organic residue was then washed with deionized water and homogenized after drying in a 50 ºC oven. TOC and organic carbon isotope data were measured on these kerogen concentrates using an Elemental Analyzer (FLASH EA 2000) coupled to Thermo Scientific™ DELTA V isotope ratio mass spectrometer. Carbon ($\delta^{13}C_{carb}$), oxygen ($\delta^{18}O_{carb}$) and $\delta^{13}C_{org}$ isotopic values are reported relative to the Vienna Pee Dee Belemnite (V-PDB) standard, with an analytical precision better than ± 0.04‰ for $\delta^{13}C_{carb}$, ± 0.09‰ for $\delta^{18}O_{carb}$, and ± 0.06‰ for $\delta^{13}C_{org}$ values.

3. In situ oxygen isotope analyses on conodonts

We employed *in situ* secondary ion mass spectrometry (SIMS) to measure the oxygen isotope composition of conodont apatite ($\delta^{18}O_{apatite}$) from the Penglaitan section. Oxygen isotope compositions of conodont apatite were measured using a Cameca IMS-1280 SIMS at the Institute of Geology and Geophysics, Chinese Academy of Sciences, following the standard analytical procedures of the SIMS laboratory as described in Chen et al. (2016), including sample mount preparation, polishing, reflected light imaging, coating, and *in situ* measurement.

A total of 381 analyzes were run on 99 specimens (*Clarkina*, n = 71; *Hindeodus*, n = 22; ramiform, n = 6) (Tables DR3, DR4). The reproducibility of analyses for each sample varied between ± 0.08‰ and ± 0.42‰ (1σ) and the average reproducibility was ± 0.29‰. Durango apatite was used as a primary reference material (RM), and Qinghu apatite was measured as a secondary laboratory reference to monitor external precision. The reproducibility of Durango and Qinghu apatite used in the four analytical sessions were in the range of 0.16-0.28‰ and 0.23-0.36‰, respectively (Tables DR5, DR6), which are consistent with the reproducibility of other 29 sessions from May, 2012 to June, 2014 (Table S5 in Chen et al., 2016).

The $\delta^{18}O_{apatite}$ values of random selected Durango apatite crystals have been independently determined at the GeoZentrum Nordbayern, Universität Erlangen-Nürnberg using the laboratory's standard method (Joachimski et al., 2009). Analyses were calibrated by measuring standards TU1 and TU2 as 21.11‰ and 5.45‰, respectively (Vennemann et al., 2002). With this calibration, reference standard NBS 120c was measured as 21.8 ± 0.17‰ (1σ, n = 10), and our Durango apatite gave a mean $\delta^{18}O$ value of 8.8 ± 0.04‰ (1σ, n = 10) (Chen et al., 2016). Strictly speaking, a homogeneity test (Sun et al., 2016) is necessary for every Durango apatite crystal used before they are mounted as RM; however, such procedure is not practical. Moreover, the limited variations in both the SIMS (Table DR5 of GSA Data Repository in this study) and IRMS (Table S6 in Chen et al., 2016) results clearly indicated that our Durango RM does not have the homogeneity problem as suggested by Sun et al. (2016), which showed that the Durango apatite they measured has an inter-crystal $\delta^{18}O$ range of 4.4‰.

Using the SIMS technique to measure oxygen isotope compositions of biogenic apatite is a promising approach for high-resolution ocean paleotemperature reconstructions (Chen et al., 2016; Rigo et al., 2012; Trotter et al., 2008, 2015, 2016; Wheeley et al., 2012). It utilizes Cs+ primary ion beam to bombard the exposed smooth surfaces of untreated biogenic phosphates and releases oxygen from all O-bearing compounds (PO_4^{3-}, CO_3^{2-}, and OH^-), which is different from IRMS technique (PO_4^{3-} only). Therefore, a small offset between the SIMS and IRMS results is expected (Chen et al., 2016), and comparison of results from different approaches should be dealt with caution. Systematic analyses of conodont apatite from the Meishan section by both SIMS and IRMS approaches indicate that the patterns are largely comparable (Vennemann et al., 2002), suggesting both methods are valid. The main difference between the SIMS and IRMS methods lies within the temporal resolution. Our SIMS $\delta^{18}O_{apatite}$ data from Penglaitan (Fig. 12) is generally consistent with previous results from other sections in South China (Chen et al., 2016; Joachimski et al., 2012) and northwest Iran (Schobben et al., 2014), in terms of the timing and magnitude of a sharp decrease in $\delta^{18}O_{apatite}$.

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- **Table DR1**. U-Pb data for analyzed zircon from tuff beds of the Penglaitan section, Laibin, Guangxi Province, South China.
- **Table DR2**. $\delta^{13}C_{carb}$, $\delta^{18}O_{carb}$, $\delta^{13}C_{org}$ and TOC (total organic carbon) data from the Penglaitan section.
- **Table DR3**. *In situ* oxygen isotope compositions of conodont apatite ($\delta^{18}O_{apatite}$) and calculated seawater temperatures from the Penglaitan section.
- **Table DR4**. Raw data of $\delta^{18}O$ values measured on conodont apatite.
- **Table DR5**. Raw data of $\delta^{18}O$ values measured on Durango reference standard.
- **Table DR6**. Raw data of $\delta^{18}O$ values measured on Qinghu reference standard.