Appendix 2: Zircon sample preparation, analyses and data reduction methods

Sample treatment to extract and mount zircons

Samples were trimmed into fist-sized pieces using the diamond saw at MRT Core Library. These smaller portions were then crushed in a hydraulic press at ES/CODES University of Tasmania into chips < 3 cm long. Milling to powder was undertaken in a chrome-steel mill for 4 seconds, cleaning between each sample with river sand to prevent contamination.

Powder was washed and panned to remove light grains with heavy minerals collected and dried. Heavier grains were further washed in a watch glass to separate zircons from other minerals. The wet watch glass was subsequently put in the oven at low temperatures (40 degrees C) until dry. Magnetic grains were removed using a REE magnet and kept in a separate fraction. Some euhedral zircons from the non-magnetic fraction were hand-picked and moved onto surfaces for mounting and some of the non-magnetic fraction was poured onto the surface. Epoxy resin was poured onto the zircons to make the mounts.

Once the epoxy mounts had hardened the zircon surfaces were polished. Cathodoluminescence was undertaken at the Central Science Laboratory at the university of Tasmania (CSL) using the Scanning Electron microscope of up to 200 grains. Some of the resulting images are presented in Appendix 3.

Obtaining zircon geochemistry

The analyses were performed with an ASI RESOLution S-155 ablation system with Coherent Compex Pro 110 Ar-F excimer laser operating at a 193nm wavelength and a pulse width of 20ns. The laser system was coupled to an Agilent 7900 quadrupole ICP-MS. Other analytical conditions are described in Thompson et al. (2018)[1]. All instrumentation is housed at the CODES Analytical Laboratory at the University of Tasmania.

For the detrital zircon the following standards used were

- 91500 (Wiedenbeck et al., 1995, 2005) was used as the primary calibration standard for the Pb/U and Pb/Th ratios
- NIST610 (reference values from http://georem.mpch-mainz.gwdg.de) was used to calibrate the 207Pb/206 Pb ratios and to calibrate the trace element concentrations using the method outlined by Kosler (2001) using Zr as the internal standard element, assuming stoichiometric proportions.
- Plesovice and Temora (Sláma et al., 2008; Black et al., 2003) were used as check standards to ensure that the ages calculated were accurate.
- GSD-IG and BCR-2G were analysed as check standards for the trace elements and for the Pb isotope ratios (reference values from http://georem.mpch-mainz.gwdg.de)
- These were analysed before and after every 23 sample spots.

Analyses were targeting rims of the grains unless noted otherwise. Each analysis on the zircons began with a 30 second blank gas measurement followed by a further 30 seconds of analysis time when the laser was switched on. Zircons were analysed using a 20 and 30 micron laser beam size, firing frequency at 5 Hz and a laser beam energy density of ~ 2 J/cm2. All zircon analyses have a pre-ablation of 5 laser shots to remove any surface contamination. A flow of He carrier gas at a rate of

[1] Thompson J. M., Meffre, S. and Danyushevsky, L. 2018. Impact of laser pulse width and fluence on U-Pb dating of zircons by LA-ICPMS, Journal of Analytical Atomic Spectroscopy, 33:221-230.

0.35 litres/minute carried particles ablated by the laser out of the chamber to be mixed with Ar gas and carried to the plasma torch. Isotopes measured were 31P, 49Ti, 56Fe, 89Y, 91Zr, 93Nb, 139La, 140Ce, 141Pr, 146Nd, 147Sm, 153Eu, 157Gd, 159Tb, 163Dy, 165Ho, 166Er, 169Tm, 172Yb, 175Lu, 178Hf, 181Ta, 204Pb, 206Pb, 207Pb, 208Pb, 232Th, 235U and 238U with each element being measured every 0.25 seconds with longer counting time on the Pb isotopes compared to the other elements

Data Reduction.
The data reduction used LADR software. This automatically set the best parameters and correction using the standards across the analyses time period.

Once the zircon analyses had been calculated by the LADR software, samples were individually examined, and notes were made on the quality of the crystal. Each crystal was included because it's given age was considered accurate or excluded due to the analysis displaying evidence of disturbance of the U-Pb system either due to lead loss in high uranium zones of skewing the age to younger values or due to common Pb making the analyses discordant. Zircons that were <80% concordant or contained obvious mineral inclusions were also excluded as these can affect the age calculations. In some areas the zircons were small and thin, and the laser drilled through the zircon crystal into the underlying epoxy. These analyses were trimmed to calculate ages for the high zirconium parts of the analysis. Some of the time-resolved analyses showed evidence of that the laser sampled both an older core and young rims within a single analysis. Out of the 217 spots that were performed on the samples which were analyzed, 166 samples were included and 51 were excluded. Raw data is listed in Appendix 4.

Finding the populations of the youngest zircon ages.
Once reliable analyses were identified, a ^{207}Pb corrected $^{206}Pb/^{238}U$ age was calculated for each zircon and the maximum deposition age was calculated by selecting youngest coherent population with overlapping uncertainties (probability of equivalence <0.95). From these a weighted mean maximum deposition age was calculated using the Isoplot Excel add-in (Ludwig, 2012)[2]was used to calculate weighted mean ages, plot probability distribution and plot Tera-Wasserberg Concordia diagrams.

[2] Ludwig, K. (2012). User's manual for Isoplot version 3.75–4.15: A geochronological toolkit for Microsoft Excel. Berkley Geochronological Center Special Publication review 5: 1-75.