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METHODOLOGIES

Zircons and titanites were mounted in 25 mm diameter epoxy resin mounts

The mounts were coated with a ca. 15 nm-thick layer of carbon.

Zircons were imaged by cathodoluminescence (CL) using a KE Developments Centaurus system attached to a Tescan Mira XMU Field Emission Scanning Electron Microscope (SEM) at the Centre for Microscopy and Analysis (CMA) at Trinity College, Dublin. An accelerating voltage of 10 kV and working distance of ca. 11.5 mm were employed.

Titanites were imaged by Backscattered-Electrons (BSE) using a KE Developments Centaurus system attached to a Tescan Mira XMU Field Emission Scanning Electron Microscope (SEM) at the Centre for Microscopy and Analysis (CMA) at Trinity College, Dublin. An accelerating voltage of 20 kV and working distance of ca. 18.5 mm were employed.

Petrology

The petrological study of the continental sandstones from the North Minwun Basin revealed 25-65% quartz, with monocrystalline more abundant than polycrystalline quartz, and 5-10% feldspar. Plagioclase and orthoclase are common, while microcline is rare. In some thin sections, sericite has replaced feldspar and calcite and clay are observed. Lithic fragments range from 10-30%, and include slate, phyllite, schist, siltstone, dolomite and limestone. A mixture of silt, hematite/magnetite, chlorite and clays form cement, which constitutes 15-30% of the rock. Heavy minerals (HM) such as zircons, titanites, rutiles, apatites comprise less than 1%.

Zircons:

LA-ICPMS elemental and U–Pb age from zircons were acquired using a Photon machines G2 193 nm ArF excimer laser-ablation system with a Helex 2-volume ablation cell coupled to a Thermo Scientific iCAP Qc, at the Department of Geology, Trinity College Dublin. The ICPMS instrument was tuned using NIST612 standard glass to yield Th/U ratios of unity and low oxide production rates (ThO⁺/Th⁺ typically <0.10%).

An ARIS variable-volume signal-smoothing device was used to mix the He aerosol with 0.5 l(m)-1 Ar make-up gas and a small volume of N₂ (ca. 6 ml/m) to enhance signal sensitivity and reduce oxide formation (Table 1).

The Analyte Excite – iCAP Qc setup yields a washout with 90% signal reduction in less than 1.5 s. Seven isotopes (⁹⁰Zr or ⁹¹Zr, ²⁰⁴Pb, ²⁰⁶Pb, ²⁰⁷Pb, ²⁰⁸Pb, ²³²Th and ²³⁸U) were acquired using fluence of ca. 1.4 J·cm² with a total duty cycle of 25 secs.

All experiments employed a 30x30 μm square spot (Table 1).

Table 1

Laser parameters	
Instrument	Photon Machines Analyte Excite ArF Excimer 193 nm
Washout and background	10 and 15 s
Laser repetition rate	10Hz
Spot size	30x30 μm
Energy	1.4 Jcm ²
Laser carrier gas	0.65 L/min He
Mass spectrometer parameters	
Instrument	Thermo Scientific iCAP Qc

Plasma RF power	1550 W
Plasma carrier gas flow	0.5 L/min
Isotopes measured	
with dwell times [ms] listed	⁹¹ Zr [2], ²⁰⁴ Pb [10], ²⁰⁶ Pb [30], ²⁰⁷ Pb [75], ²⁰⁸ Pb [10], ²³² Th [10], ²³⁸ U [30]
Total duty cycle	25s acquisition 12s washout

Data reduction of the raw U-Pb isotope data was performed through the “VizualAge” data reduction scheme (Petrus and Kamber, 2012) in the freeware IOLITE package (Paton et al., 2011). Sample-standard bracketing was applied after the correction of downhole fractionation to account for long-term drift in isotopic or elemental ratios by normalizing all ratios to those of the U-Pb reference standards. Final age calculations and KDE plots were made using the Isoplot add-in for Excel (Ludwig, 2012) and Density Plotter 8.4 (Vermeesch, 2012). 91500 zircon (²⁰⁶Pb/²³⁸U TIMS age of 1065.4 ± 0.6 Ma) (Wiedenbeck et al., 1995) was used as the primary U-Pb calibration standard. The secondary standards

- Temora-2 zircon (²⁰⁶Pb/²³⁸U TIMS age of 416.8 ± 1.3 Ma; Black et al., 2003)
- Fish Canyon zircon (²⁰⁶Pb/²³⁸U TIMS age of 28.478 ± 0.024 Ma; Schmitz and Bowring, 2001)
- WRS 1348 zircon (²⁰⁶Pb/²³⁸U TIMS age of 526.26 ± 0.70; Pointon et al., 2012)

yielded LA-ICPMS ²⁰⁶Pb/²³⁸U ages of 416.2 ± 2.4 Ma, 28.10 ± 0.34 Ma and 527.1 ± 2.4 Ma respectively. In this study, the ²⁰⁶Pb/²³⁸U age was used for zircons younger than 1.2 Ga and the ²⁰⁷Pb/²⁰⁶Pb age for zircons older than 1.2 Ga following Gehrels et al. (2006) and Gehrels (2014).

The Concordia threshold for the considered ages was placed at 10%. The Concordia values were calculated using the “CONCORDIA” function in ISOPLOT (Ludwig, 2012). This function creates an array function that returns the “Concordia” age for the conventional-Concordia ratios, errors, & error-correlation (see Ludwig, 1998). Input values are ²⁰⁶Pb*/²³⁸U, error, ²⁰⁷Pb*/²³⁵U, error, error-correlation, with LambdaErrs (TRUE to propagate the ²³⁵U and ²³⁸U decay-constant errors), and two optional Boolean variables (PercentErrs and SigmaLevel specifying the type of error input (default is absolute, 2σ). Output is a 2-rows x 4-columns range containing the Concordia Age, 2σ a priori age error, MSWD, and probability-of-concordance. The second row of cells contains captions for the values in the first row.

Titanites:

LA-ICPMS elemental and U–Pb age from titanites were acquired using a Photon machines G2 193 nm ArF excimer laser-ablation system with a Helex 2-volume ablation cell coupled to an Agilent 7900, at the Department of Geology, Trinity College Dublin. The ICPMS instrument was tuned using NIST612 standard glass to yield Th/U ratios of unity and low oxide production rates (ThO⁺/Th⁺ typically <0.12%).

An ARIS variable-volume signal-smoothing device was used to mix the He aerosol with 0.5 l(m)-1 Ar make-up gas and a small volume of N₂ (ca. 6 ml/m) to enhance signal sensitivity and reduce oxide formation (Table 2).

Table 2

Laser parameters	
Instrument	Photon Machines Analyte Excite ArF Excimer 193 nm
Washout and background	10 and 15 s
Laser repetition rate	12Hz
Spot size	47x47 μm

Energy	2.25 Jcm ²
Laser carrier gas	0.5 L/min He
Mass spectrometer parameters	
Instrument	Agilent 7900
Detection system	Dual-mode discrete dynode electron multiplier
Plasma RF power	1550 W
Plasma carrier gas flow	0.65 L/min
Isotopes measured	
Isotopes measured with dwell times [ms] listed	⁹¹ Zr [2], ²⁰⁴ Pb+Hg [10], ²⁰⁶ Pb [30], ²⁰⁷ Pb [75], ²⁰⁸ Pb [10], ²³² Th [10], ²³⁸ U [30], ¹⁷⁷ Hf [30], ⁵⁶ Fe [30], all REE [0.3]
Total duty cycle	25s acquisition 12s washout

The raw isotope data were reduced using a modified version of the “Vizual Age” data reduction scheme (Petrus & Kamber, 2012) of the freeware IOLITE package of Paton et al. (2011). This data reduction scheme (“VizualAge_UcomPbine”) can account for variable common Pb in the titanite standard materials (Chew et al., 2014). User-defined time intervals are established for the baseline correction procedure to calculate session-wide baseline-corrected values for each isotope. The time-resolved fractionation response of individual standard analyses is then characterized using a user-specified down-hole correction model as an exponential curve, a linear fit, or a smoothed cubic spline). The data reduction scheme then fits this appropriate session-wide “model” U—Th—Pb fractionation curve to the time-resolved standard data and the unknowns. Sample-standard bracketing is applied after the correction of down-hole fractionation to account for long-term drift in isotopic or elemental ratios by normalizing all ratios to those of the U—Pb reference standards. Final age calculations and KDE plots were made using the Isoplot add-in for Excel (Ludwig, 2012) and Density Plotter 8.4 (Vermeesch, 2012).

OLT-1 (²⁰⁶Pb/²³⁸U TIMS ages of 1014.8 ± 2.0 Ma; Kennedy et al., 2010) was used as the primary U-Pb calibration standard. The secondary standards

- MKed1 titanite (²⁰⁶Pb/²⁰⁷Pb TIMS age of 1521.02 ± 0.55 Ma; Spandler et al., 2016)
- BLR titanite (²⁰⁶Pb/²⁰⁷Pb TIMS age of 1047.1 ± 0.4 Ma; Aleinikoff et al., 2007)

yielded LA-ICPMS ages of 1057.1 ± 15 Ma and 1535.9 ± 25 Ma respectively. Due to the ²⁰⁷Pb-based correction, no titanite U-Pb age data can be excluded based on discordance criteria. However, the low-U content (sometimes <1 ppm) and consequent near-zero radiogenic Pb content of some titanite grains can result in large analytical uncertainties (or negative ages). We therefore excluded grains with 2σ errors >25%, similar to the approach of Zattin et al. (2012).

Apatites:

LA-ICPMS elemental and U—Pb age from apatites were acquired using a Photon machines G2 193 nm ArF excimer laser-ablation system with a Helex 2-volume ablation cell coupled to an Agilent 7900, at the Department of Geology, Trinity College Dublin. The ICPMS instrument was tuned using NIST612 standard glass to yield Th/U ratios of unity and low oxide production rates (ThO⁺/Th⁺ typically <0.12%).

An ARIS variable-volume signal-smoothing device was used to mix the He aerosol with 0.4 l(m)-1 Ar make-up gas and a small volume of N2 (ca. 6 ml/m) to enhance signal sensitivity and reduce oxide formation (Table 2).

Table 2

Laser parameters

Instrument	Photon Machines Analyte Excite ArF Excimer 193 nm
Washout and background	10 and 15 s
Laser repetition rate	12Hz
Spot size	47x47 μm
Energy	2.25 Jcm²
Laser carrier gas	0.5 L/min He
Mass spectrometer parameters	
Instrument	Agilent 7900
Detection system	Dual-mode discrete dynode electron multiplier
Plasma RF power	1550 W
Plasma carrier gas flow	0.65 L/min
Isotopes measured	
Isotopes measured with dwell times [ms] listed	³¹ P (1), ⁴³ Ca (1), ⁵¹ V (0.75), ⁵⁵ Mn (0.75), ⁷⁵ As (1.5), ⁸⁸ Sr (0.75), ⁸⁹ Y (0.5), ⁹⁰ Zr (1), ¹³⁹ La (0.5), ¹⁴⁰ Ce (0.5), ¹⁴¹ Pr (0.5), ¹⁴⁶ Nd (0.5), ¹⁴⁷ Sm (0.75), ¹⁵³ Eu (1), ¹⁵⁷ Gd (1), ¹⁵⁹ Tb (1), ¹⁶³ Dy (1), ¹⁶⁵ Ho (1), ¹⁶⁶ Er (1), ¹⁶⁹ Tm (1), ¹⁷² Tb (1), ¹⁷⁵ Lu (1.5), ²⁰² Hg (0.5), ²⁰⁴ Pb+ ²⁰⁴ Hg (0.5), ²⁰⁶ Pb (7.5), ²⁰⁷ Pb (7.5), ²⁰⁸ Pb (0.5), ²³² Th (0.5), ²³⁸ U (7.5)
Total duty cycle	25s acquisition 20s washout

The raw isotope data were reduced using a modified version of the “Vizual Age” data reduction scheme (Petrus & Kamber, 2012) of the freeware IOLITE package of Paton et al. (2011). This data reduction scheme (“VizualAge_UcomPbine”) can account for variable common Pb in the titanite standard materials (Chew et al., 2014). User-defined time intervals are established for the baseline correction procedure to calculate session-wide baseline-corrected values for each isotope. The time-resolved fractionation response of individual standard analyses is then characterized using a user-specified down-hole correction model as an exponential curve, a linear fit, or a smoothed cubic spline). The data reduction scheme then fits this appropriate session-wide “model” U—Th—Pb fractionation curve to the time-resolved standard data and the unknowns. Sample-standard bracketing is applied after the correction of down-hole fractionation to account for long-term drift in isotopic or elemental ratios by normalizing all ratios to those of the U—Pb reference standards. Final age calculations and KDE plots were made using the Isoplot add-in for Excel (Ludwig, 2012) and Density Plotter 8.4 (Vermeesch, 2012).

Madagascar (²⁰⁶Pb/²³⁸U TIMS ages of 473.5 ± 0.7 Ma; Thomson et al., 2012) was used as the primary U-Pb calibration standard. The secondary standards

- Durango titanite (²⁰⁶Pb/²⁰⁷Pb TIMS age of 31.44 ± 0.18 Ma; McDowell et al., 2005)
- McClure titanite (²⁰⁶Pb/²⁰⁷Pb TIMS age of 523.51 ± 0.4 Ma; Schoene & Bowring 2006)

yielded LA-ICPMS ages of 32.58 ± 0.63 Ma and 527.3 ± 6.2 Ma respectively. Due to the ²⁰⁷Pb-based correction, no titanite U-Pb age data can be excluded based on discordance criteria. However, the low-U content (sometimes <1 ppm) and consequent near-zero radiogenic Pb content of some apatite grains can result in large analytical uncertainties (or negative ages). We therefore excluded grains with 2σ errors >25%, similar to the approach of Zattin et al. (2012).

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