**Methods for U-Pb LA-ICP-MS Analysis**

Calcite U-Pb dating was performed by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) on polished thick sections. The analyses were conducted at ETH Zürich using an ASI RESOlution laser ablation system with a 193 nm excimer (ArF) laser source and a two-volume Laurin Technic S-155 ablation cell coupled to a Thermo Element XR sector-field ICP-MS. The analytical and data reduction protocols follow Roberts et al. (2017) using NIST 614 and WC-1 primary reference materials and Guillong et al. (2020) using spot sizes of 110 and 163 m with a matched ablation crater aspect ratio for the primary reference materials and unknowns. U-Pb ages were calculated from Tera-Wasserburg concordia lower intercepts using the IsoplotR software package (Vermeesch. 2018). All uncertainties are reported at the 95% confidence level. A long-term excess variance of 2% relative was propagated by quadratic addition to the uncertainty of the individual lower intercept dates (Guillong et al. 2020). Prior to and after the LA-ICP-MS measurements, cathodoluminescence microscopy was conducted in order to identify potential growth zonings and to identify misplaced or defective ablation spots. In addition to the samples, the two secondary reference materials ASH15D (Nuriel et al. 2021) and JT (Guillong et al. 2020) were analyzed in all sessions for validation and are reported in the Data Supplement. Correction for matrix effects with WC-1 was done with anchoring to 0.85 common-lead while samples and secondary reference materials were not anchored. No disequilibrium correction was applied. The table below shows the metadata of U-Pb analyses and data processing with reporting standards after Horstwood et al. (2016).

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| **Laboratory and sample preparation** | |
| Laboratory name | Department of Earth Sciences. ETH Zürich |
| Sample type/mineral | Calcite |
| Sample preparation | Polished thick sections |
| Imaging | CL8200 Mk5-2 Optical Cathodoluminescence System |
| **Laser ablation system** |  |
| Make. model. and type | ASI (Resonetics) RESOlution S155 |
| Ablation cell and volume | Laurin Technic, two-volume cell, effective volume approx. 1 cm3 |
| Laser wavelength | 193 nm |
| Pulse width | 25 ns |
| Fluence | ~1.8 J cm-2 |
| Repetition rate (Hz) | 5.0 or 7.4. See data for actual value |
| Ablation duration | 40 s |
| Ablation pit depth / ablation rate | Variable. equivalent to 0.09-0.15 mm/pulse |
| Spot diameter (mm) nominal/actual | 110 or 163m. See data for actual value |
| Sampling mode / pattern | Static spot ablation |
| Carrier gas | 100% He in the cell, Ar make-up gas combined in cell above ablation in funnel |
| Cell carrier gas flow | 0.5 l min-1 |
| **ICP-MS instrument** |  |
| Make. model and type | Thermo Element XR, sector-field single collector ICP-MS with high-capacity interface pump |
| Sample introduction | Ablated aerosol direct |
| RF power | 1350-1550 W (optimized daily) |
| Make-up gas flow | 0.90-1.05 l min-1 Ar (optimized daily), 2 ml min-1 N2 |
| Detection system | Triple (pulse counting, analog, Faraday), cross-calibrated daily with 238U, fixed analogue counting factor (ACF) value, all isotopes usually in pulse-counting only (<5 Mcps) |
| Masses measured (amu) | 202, 204, 206, 207, 208, 232, 235, 238 |
| Integration time per peak/dwell times | 11 ms (all masses) except 206, 207 (50 ms) |
| Total integration time per output data point | 0.174 s |
| Sensitvity / efficiency | ~ 1 % U |
| Dead time | 25 ns |
| Typical oxide rate (ThO/Th) | 0.18% |
| Typical doubly charged rate (Ba++/Ba+) | 3.50% |
| **Data processing** |  |
| Gas blank | 20 s |
| Calibration strategy | NIST614 glass standard as primary reference material for drift and 207Pb/206Pb ratios (see Roberts et al. 2017); WC-1 carbonate reference material for matrix matching of 206Pb/238U (anchored to initial Pb composition of 0.85, see Roberts et al. 2017); ASH-15D (Nuriel et al. 2021) and JT (Guillong et al. 2020) calcite validation reference materials for assessing accuracy and repeatability. |
| Reference material info | NIST614 (concentration data Jochum et al. 2011, Pb isotopes Baker et al. 2004); WC-1 (Roberts et al. 2017); ASH-15D (Nuriel et al. 2021); JT (Guillong et al. 2020) |
| Data processing package used | Iolite 2.5, VisualAge for integration, interval selection, and gas blank correction only. In-house spreadsheet data processing. IsoplotR (Vermeesch 2018) for isochrons, intercept ages, and initial Pb compositions |
| Correction for LIEF | No separate LIEF correction. 238U/206Pb corrected to primary RM assumed to have similar LIEF as samples. |
| Mass discrimination | Normalized to reference material (sample-standard bracketing) |
| Common-Pb correction. composition and uncertainty | Not applied. Ages calculated by linear regressions in Tera-Wasserburg concordia plots using the IsoplotR software package (Vermeesch. 2018) |
| Uncertainty level and propagation | Lower intercept ages are quoted at 2 absolute, propagation is by quadratic addition. Counting statistics uncertainties are propagated to the 207Pb/206Pb ratios, together with the uncertainty of the primary RM value and the uncertainty of repeated measurements. The uncertainty value for lower intercept isochron ages includes uncertainties from the primary RM and unsystematic uncertainties, estimated in this work to be 2 %. Decay constant uncertainties are neglected. |
| Quality control / Validation | ASH-15-D:  Weighted mean of 206Pb/238U intercept ages: 2.769 ± 0.144 Ma (2, MSWD = 0.97, n = 4, including weighted average uncertainty (internal) and 2% total external uncertainty)   Pooled measurements for 4 sessions: 2.894 ± 0.092 Ma (2, MSWD = 2.2, n = 107, including internal and 2% total external uncertainty) |
|  | JT:  Weighted mean of 206Pb/238U intercept ages: 13.340 ± 0.680 Ma (2, MSWD = 2.31, n = 4, including weighted average uncertainty (internal) and 2% total external uncertainty)   Pooled measurements for 4 sessions: 13.385 ± 0.367 Ma (2, MSWD = 2.2, n = 112, including internal and 2% total external uncertainty) |
|  | Systematic uncertainty for propagation is 2% (2) |

Metadata of U-Pb analyses and data processing. Reporting standards after Horstwood et al. (2016).

**References**

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