

Supplementary to Analytical Methods

Data reporting Template for LA-ICP-MS U-Pb data:

Laboratory & Sample Preparation	
Laboratory name	Dept of Earth Science, ETH Zurich
Sample type/mineral	Zircon
Sample preparation	Conventional mineral separation, 1 inch resin mount, 1 μm polish to finish
Imaging	AMRAY 1830 SEM, GATAN MiniCL, 3nA, 10kV Dept. of Petrology and Geochemistry, Eötvös University, Budapest
Laser ablation system	
Make, Model & type	Resonetics Resolution 155 (RESolution SE excimer ArF laser)
Ablation cell & volume	Laurin Technics 155, constant geometry, aerosol dispersion volume $<1\text{ cm}^3$
Laser wavelength	193 nm
Pulse width (ns)	25 ns
Fluence ($\text{J}\cdot\text{cm}^{-2}$)	$\sim 2\text{ J}\cdot\text{cm}^{-2}$
Repetition rate (Hz)	5 Hz
Spot size (μm)	30 μm
Ablation rate	$\sim 75\text{ nm pulse}^{-1}$
Sampling mode	Single hole drilling, 5 cleaning pulses
Carrier gas	100% He
Ablation duration	40 s
Cell carrier gas flow	0.7 l/min
ICP-MS Instrument	
Make, Model & type	Thermo Element XR SF-ICP-MS
Sample introduction	Ablation aerosol only, squid aerosol homogenization device
RF power (W)	1500W
Make-up gas flow	$\sim 0.95\text{ l/min}$ Ar (gas mixed to He carrier inside ablation cell funnel)
Detection system	Single detector triple mode SEM, analog, Faraday
Masses measured	202, 204, 206, 207, 208, 232, 235, 238 amu
Integration time per peak	12ms (masses 202, 204), 20ms (masses 208, 232, 235, 238), 40ms (masses 206, 207)
Total integration time per reading	202 ms
Dead time (ns)	8 ns
Typical oxide rate (ThO/Th)	0.18%

Typical doubly charged rate (Ba ⁺⁺ /Ba ⁺)	3.5%					
Data Processing						
Gas blank	10 second prior to each ablation spot					
Calibration strategy	GJ-1 used as primary reference material Secondary reference materials used in sessions: session 140614: Plesovice, 91500, Temora, LG_0302; Plesovice (Slama et al., 2008), 91500 (Wiedenbeck et al., 1995), Temora (Black et al., 2004), LG_0302 (personal communication)					
Reference Material info	GJ-1 ²⁰⁶ Pb/ ²³⁸ U 0.09761±0.0002 (Wtd mean of TIMS analysis, Jackson et al., 2004)					
Data processing package used	IOLITE 2.5, 3.4 (Paton et al. 2010, 2011) with Vizual Age (Petrus and Kamber 2012)					
Mass discrimination	Mass bias correction for all ratios normalized to primary reference material					
Common-Pb correction	No common-Pb correction applied					
Uncertainty level & propagation	Ages are quoted at 2 SE absolute, propagation is by quadratic addition. Reproducibility of reference material uncertainty is propagated.					
Quality control / Validation						
Reference material	Ref. age (Ma)	Weighted mean age (Ma)	2SE	MSWD	No	Average rel. 2SE of spot analyses
Plesovice	337.15	337.7	1	1.4	11	0.8
91500	1063.5	1058.2	3.1	0.88	9	0.9
Temora	416.8	415.9	1.4	1.3	9	0.8
LG 0302	10.67	10.54	0.15	1.8	8	2.9

Laboratory & Sample Preparation	
Laboratory name	Göochron Laboratories, University of Göttingen
Sample type/mineral	Zircon
Sample preparation	Conventional mineral separation, 1 inch resin mount, 3 µm polish to finish
Imaging	AMRAY 1830 SEM, GATAN MiniCL, 3nA, 10kV Dept. of Petrology and Geochemistry, Eötvös University, Budapest
Laser ablation system	
Make, Model & type	Resonetics, Resolution 155
Ablation cell & volume	Laurin Technics 155, constant geometry, aerosol dispersion volume <1 cm ³
Laser wavelength	193 nm
Pulse width	25 ns
Fluence	~2 J.cm ⁻²
Repetition rate	5 Hz
Spot size	33 µm
Ablation rate	~75 nm pulse ⁻¹
Sampling mode	Single hole drilling, 2 cleaning pulses
Carrier gas	100% He
Ablation duration	20 s
Cell carrier gas flow	0.7 l/min
ICP-MS Instrument	
Make, Model & type	Thermo Element 2 SF-ICP-MS
Sample introduction	Ablation aerosol only, squid aerosol homogenization device
RF power	1500 W
Make-up gas flow	~1 l/min Ar (gas mixed to He carrier inside ablation cell funnel)
Detection system	Single detector dual mode SEM, analog
Masses measured	202, 204, 206, 207, 208, 232, 235, 238 amu
Integration time per peak	10 ms (masses 232, 238), 20 ms (masses 202, 204, 235), 30 ms (mass 208), 40 ms (mass 206), 100 ms (mass 207)
Total integration time per reading	250 ms
Dead time	13 ns
Typical oxide rate (UO/U)	0.04 %
Typical doubly charged rate (Ba ⁺⁺ /Ba ⁺)	n.a.

Data Processing							
Gas blank	9 second prior to each ablation spot						
Calibration strategy	GJ-1 used as primary reference material Secondary reference materials: 91500 (Wiedenbeck et al., 1995), Plesovice (Slama et al., 2008)						
Reference Material info	GJ-1 ²⁰⁶ Pb/ ²³⁸ U: 0.09761±0.00006 (Wtd mean of TIMS analysis, Jackson et al., 2004)						
Data processing package used	UranOS 2.08a (Dunkl et al., 2008) http://www.sediment.uni-goettingen.de/staff/dunkl/software/uranos.html						
Mass discrimination	Mass bias correction for all ratios normalized to primary reference material						
Common-Pb correction	No common-Pb correction applied						
Uncertainty level & propagation	Ages are quoted at 2 s absolute, propagation is by quadratic addition. Reproducibility of reference material uncertainty is propagated.						
Quality control / Validation							
Reference material	Ref. age (Ma)	Weighted age (Ma)	mean	2s	MSWD	No.	Average rel. 2s of spot analyses
Plesovice	337.15	336.9		0.5	0.4	23	1.4
91500	1063.5	1060.6		2.0	0.02	20	1.7

References

- Black, L.P., Kamo, S.L., Allen, C.M., Davis, D.W., Aleinikoff, J.N., Valley, J.W., Mundil, R., Campbell, I.H., Korsch, R.J., Williams, I.S. and Foudoulis, C., 2004. Improved $^{206}\text{Pb}/^{238}\text{U}$ microprobe geochronology by the monitoring of a trace-element-related matrix effect; SHRIMP, ID-TIMS, ELA-ICP-MS and oxygen isotope documentation for a series of zircon standards. *Chemical Geology*, 205(1): 115-140.
- Dunkl I, M.T., Simon K, von Eynatten H 2008. Brief introduction to the Windows program Pepita: data visualization, and reduction, outlier rejection, calculation of trace element ratios and concentrations from LA-ICP-MS data. In: P. Sylvester (Editor), *Laser ablation ICP-MS in the Earth Sciences: Current practices and outstanding issues*. Mineralogical Association of Canada, pp. 334–340.
- Jackson, S.E., Pearson, N.J., Griffin, W.L. and Belousova, E.A., 2004. The application of laser ablation-inductively coupled plasma-mass spectrometry to in situ U–Pb zircon geochronology. *Chemical Geology*, 211(1): 47-69.
- Jaffey, A.H., Flynn, K.F., Glendenin, L.E., Bentley, W.C. and Essling, A.M., 1971. Precision Measurement of Half-Lives and Specific Activities of ^{235}U and ^{238}U . *Physical Review C*, 4(5): 1889-1906.
- Paton, C., Hellstrom, J., Paul, B., Woodhead, J. and Hergt, J., 2011. Iolite: Freeware for the visualisation and processing of mass spectrometric data. *Journal of Analytical Atomic Spectrometry*, 26(12): 2508-2518.
- Paton, C., Woodhead, J.D., Hellstrom, J.C., Hergt, J.M., Greig, A. and Maas, R., 2010. Improved laser ablation U-Pb zircon geochronology through robust downhole fractionation correction. *Geochemistry, Geophysics, Geosystems*, 11(3): n/a-n/a.
- Petrus, J.A. and Kamber, B.S., 2012. VizualAge: A Novel Approach to Laser Ablation ICP-MS U-Pb Geochronology Data Reduction. *Geostandards and Geoanalytical Research*,

36(3): 247-270.

- Sláma, J., Košler, J., Condon, D.J., Crowley, J.L., Gerdes, A., Hanchar, J.M., Horstwood, M.S.A., Morris, G.A., Nasdala, L., Norberg, N., Schaltegger, U., Schoene, B., Tubrett, M.N. and Whitehouse, M.J., 2008. Plešovice zircon — A new natural reference material for U–Pb and Hf isotopic microanalysis. *Chemical Geology*, 249(1): 1-35.
- Wiedenbeck, M., Allé, P., Corfu, F., Griffin, W.L., Meier, M., Oberli, F., Quadt, A.V., Roddick, J.C. and Spiegel, W., 1995. Three natural zircon standards for U-Th-Pb, Lu-Hf, trace element and REE analyses. *Geostandards Newsletter*, 19(1): 1-23.