Supplementary Documents — U-Pb Analytical Methods

   Heavy mineral separates containing populations of moderate to highly elongated, prismatic zircon crystals were separated from hand samples of each bentonite by conventional density and magnetic methods. The bulk zircon separates for each sample were placed in a muffle furnace at 900°C for 60 hours in quartz beakers to anneal minor radiation damage; annealing enhances cathodoluminescence (CL) emission, promotes more reproducible interelement fractionation during laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS), and prepares the crystals for subsequent chemical abrasion (Mattinson, 2005). All zircon U-Pb isotopic analyses were undertaken on crystals previously mounted, polished and imaged by cathodoluminescence (CL), and selected on the basis of zoning patterns (see accompanying image mosaic with selected crystals highlighted). Crystals were plucked from grain mounts, chemically abraded using a single aggressive abrasion step in concentrated HF at 195°C for 12 hours, and the residual crystals processed for isotope dilution thermal ionization mass spectrometry (ID-TIMS). The details of ID-TIMS analysis are described by Davydov et al. (2010) and Schmitz and Davydov (2012). U-Pb dates and uncertainties for each analysis were calculated using the algorithms of Schmitz and Schoene (2007), the U decay constants of Jaffey et al. (1971), and values of $^{235}U/^{205}Pb = 100.2329$ and $^{233}U/^{235}U = 0.99506$ for the ET535. Other details of analytical parameters can be found in the notes to Table DR1. The quoted uncertainties in Table DR1 are based upon non-systematic analytical errors, including counting statistics, instrumental fractionation, tracer subtraction, and blank subtraction. These error estimates should be considered when comparing our $^{206}Pb/^{238}U$ dates with those from other laboratories that used tracer solutions calibrated against the EARTHTIME gravimetric standards. When comparing our dates with those derived from other decay schemes (e.g., $^{40}Ar/^{39}Ar$, ^{187}Re-^{187}Os), the uncertainties in tracer calibration (0.05%; Condon et al., 2007) and U decay constants (0.108%; Jaffey et al., 1971) should be added to the internal error in quadrature. Sample ages are thus reported as ± X (Y) [Z] Ma, where X is the internal error, Y is the internal plus tracer calibration error, and Z is the internal plus tracer plus decay constant uncertainty.

References

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Schmitz, M.D., Schoene, B., 2007, Derivation of isotope ratios, errors and error correlations for U-Pb geochronology using ^{205}Pb-^{235}U-(^{233}U)-spiked isotope dilution thermal ionization mass spectrometric data: *Geochemistry, Geophysics, Geosystems* 8, Q08006, doi:10.1029/2006GC001492.

CL Images of Podolia Zircon Grains
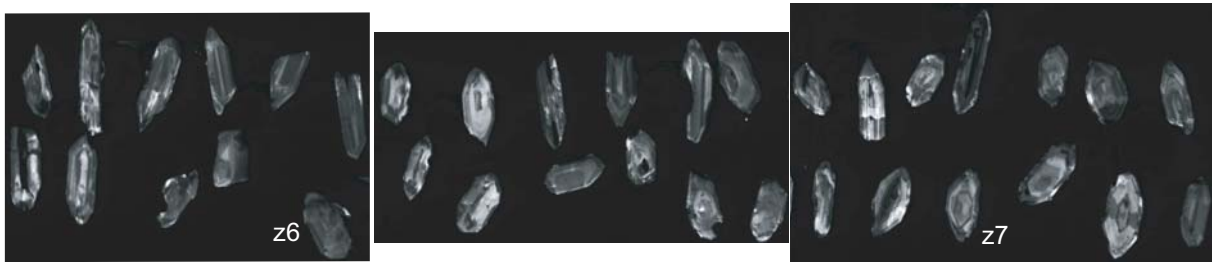
Sample M12
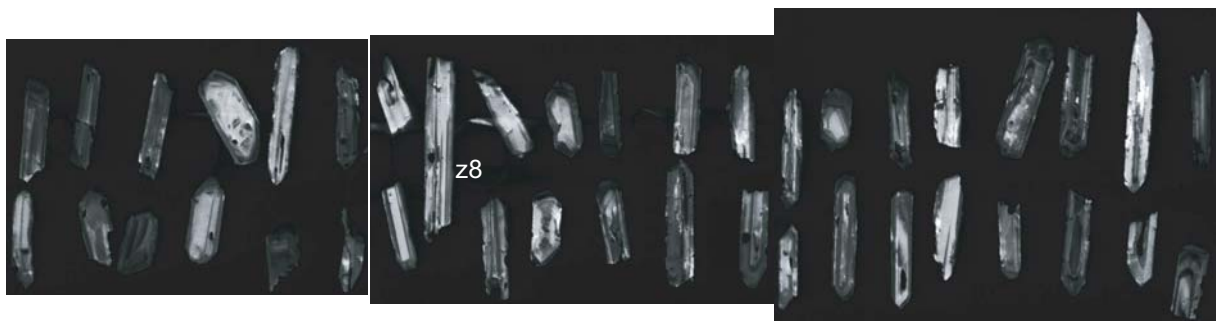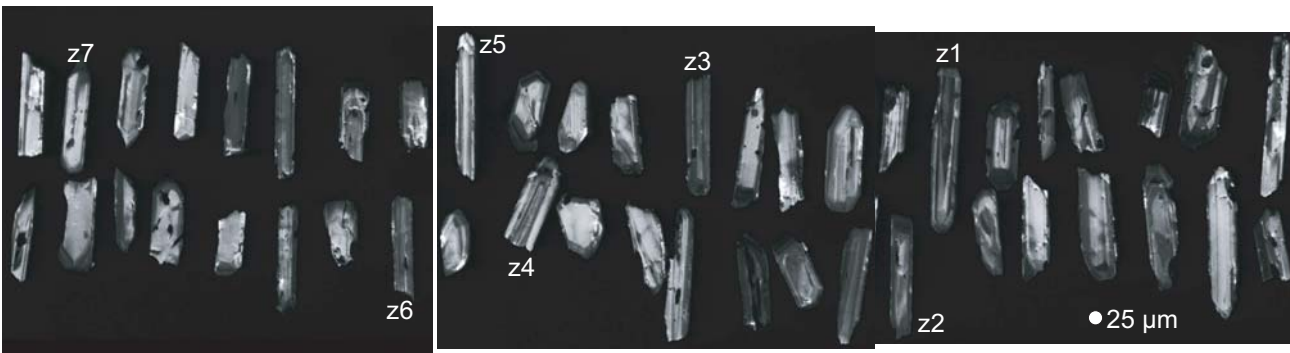
Sample C6

TABLE DR1. U-Th-Pb ISOTOPIC DATA

	Compositional Parameters						Radiogenic Isotope Ratios								Isotopic Ages					
Sample	$\frac{Th}{U}$	$^{206}Pb^*$ x10^{-13} mol	mol % $^{206}Pb^*$	$\frac{Pb^*}{Pb_c}$	Pb_c (pg)	$\frac{^{206}Pb}{^{204}Pb}$	$\frac{^{208}Pb}{^{206}Pb}$	$\frac{^{207}Pb}{^{206}Pb}$	% err	$\frac{^{207}Pb}{^{235}U}$	% err	$\frac{^{206}Pb}{^{238}U}$	% err	corr. coef.	$\frac{^{207}Pb}{^{206}Pb}$	±	$\frac{^{207}Pb}{^{235}U}$	±	$\frac{^{206}Pb}{^{238}U}$	±
(a)	(b)	(c)	(c)	(c)	(c)	(d)	(e)	(e)	(f)	(e)	(f)	(e)	(f)	(f)	(g)	(f)	(g)	(f)	(g)	(f)
C6																				
z1	0.513	0.9366	99.62%	80	0.29	4862	0.161	0.055207	0.186	0.516209	0.202	0.067816	0.038	0.501	420.6	4.1	422.62	0.70	422.99	0.16
z2	0.657	1.6771	99.71%	109	0.40	6334	0.206	0.055343	0.145	0.517430	0.160	0.067809	0.033	0.523	426.1	3.2	423.43	0.55	422.94	0.13
z3	0.795	1.0347	99.59%	80	0.35	4491	0.250	0.055232	0.187	0.516355	0.204	0.067804	0.035	0.552	421.6	4.2	422.71	0.71	422.91	0.14
z4	0.859	0.6094	99.01%	33	0.50	1861	0.270	0.055214	0.460	0.515911	0.494	0.067768	0.058	0.634	420.9	10.3	422.42	1.71	422.69	0.24
z5	0.720	0.4495	98.87%	28	0.42	1622	0.226	0.055405	0.521	0.518044	0.559	0.067813	0.059	0.670	428.6	11.6	423.84	1.94	422.97	0.24
z6	0.836	0.3703	97.66%	14	0.73	785	0.263	0.055126	1.085	0.514858	1.161	0.067738	0.109	0.716	417.3	24.2	421.71	4.01	422.51	0.45
z7	0.818	0.3993	98.54%	22	0.49	1257	0.257	0.055349	0.662	0.517808	0.711	0.067851	0.077	0.675	426.4	14.8	423.69	2.46	423.20	0.32
z8	0.756	0.6114	98.96%	31	0.53	1770	0.237	0.055200	0.466	0.515917	0.500	0.067786	0.052	0.688	420.3	10.4	422.42	1.73	422.81	0.21
M-12																				
z1	1.096	0.1636	97.82%	16	0.30	828	0.344	0.055400	0.514	0.519545	0.574	0.068016	0.132	0.550	428.4	11.5	424.85	1.99	424.19	0.54
z2	0.730	0.3254	98.48%	21	0.42	1188	0.229	0.055381	0.384	0.518886	0.439	0.067954	0.126	0.554	427.6	8.6	424.41	1.52	423.81	0.52
z4	0.731	0.2906	98.70%	24	0.32	1389	0.230	0.055311	0.305	0.518602	0.357	0.068002	0.102	0.618	424.8	6.8	424.22	1.24	424.11	0.42
z5	0.771	0.2644	97.75%	14	0.50	802	0.242	0.055425	0.489	0.519872	0.544	0.068028	0.112	0.574	429.4	10.9	425.07	1.89	424.26	0.46
z6	0.769	0.2782	98.65%	24	0.32	1339	0.242	0.055641	0.360	0.521379	0.425	0.067961	0.149	0.573	438.1	8.0	426.07	1.48	423.86	0.61
z7	0.643	0.2267	96.79%	9	0.62	562	0.202	0.055454	0.682	0.520324	0.752	0.068051	0.145	0.557	430.6	15.2	425.37	2.61	424.40	0.59
z8	0.816	0.1568	90.51%	3	1.37	190	0.256	0.054945	1.642	0.514666	1.762	0.067935	0.186	0.677	410.0	36.7	421.58	6.08	423.70	0.76

(a) z1, z2 etc. are labels for fractions composed of single zircon grains or fragments; all fractions annealed and chemically abraded after Mattinson (2005).

(b) Model Th/U ratio calculated from radiogenic $^{208}Pb/^{206}Pb$ ratio and $^{207}Pb/^{235}U$ age.

(c) Pb* and Pbc represent radiogenic and common Pb, respectively; mol % $^{206}Pb^*$ with respect to radiogenic, blank and initial common Pb.

(d) Measured ratio corrected for spike and fractionation only. Fractionation estimated at 0.15 +/- 0.03 %/a.m.u. for Daly analyses, based on analysis of NBS-981 and NBS-982.

(e) Corrected for fractionation, spike, and common Pb; all common Pb was assumed to be procedural blank: $^{206}Pb/^{204}Pb = 18.04 \pm 0.61\%$; $^{207}Pb/^{204}Pb = 15.54 \pm 0.52\%$; $^{208}Pb/^{204}Pb = 37.69 \pm 0.63\%$ (all uncertainties 1-sigma).

(f) Errors are 2-sigma, propagated using the algorithms of Schmitz and Schoene (2007).

(g) Calculations are based on the decay constants of Jaffey et al. (1971). $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ ages corrected for initial disequilibrium in $^{230}Th/^{238}U$ using Th/U [magma] = 3.

(h) Corrected for fractionation, spike, and blank Pb only.