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Middle Pleistocene age of the fossiliferous sedimentary sequence from Tarija, Bolivia

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Supplementary Materials

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Appendix--Geochronological Methods

U-Th/He zircon analysis

Aliquots of 2–3 grains were loaded into Nb tubelets and loaded into an all-metal extraction system that was baked overnight. Samples were dropped into a double-vacuum furnace and heated to 1350^{o} C for 60 minutes; each sample was also reheated to verify that complete gas extraction had occurred. Purified gas was then analyzed in a quadrupole mass spectrometer, and ^{4}He amounts were determined using a manometric calibration against numerous interspersed calibration standards. Long-term use of this calibration gives 31.85 ± 0.24 Ma (2σ S.E.) for Durango apatite (age of 31.44 ± 0.18 Ma (2σ), McDowell et al., 2005) and 28.10 ± 0.74 Ma (2σ S.E.) for Fish Canyon zircon (U-Pb age of 28.48 ± 0.06 Ma (2σ), Schmitz and Bowring, 2001; Ar-Ar sanidine age of 28.02 ± 0.28 Ma (2σ)). After unloading from the vacuum system, samples were sent to the University of Arizona for U and Th analysis by isotope-dilution analysis in the laboratory of Dr. Peter Reiners. Before analysis grains were photographed in two orientations in order to determine all major dimensions, and for pyramidal grains, the tip lengths were also measured. Alpha-loss corrections were determined using a Monte Carlo simulation code that determined F_t using 10^7 simulated ejections from a tetragonal prism with bipyramidal terminations.

U-Th/Pb zircon analysis

Zircons for U-Pb analysis were hand picked, embedded into a 25 mm diameter epoxy resin disc and polished and dated using a laser-ablation multicollector inductively-coupled plasma mass spectrometry (LA-MC-ICPMS) system housed at the University of California, Santa Barbara (UCSB). Instrumentation consists of a Nu Plasma MC-ICPMS

(Nu Instruments, Wrexham, UK) and a 193 nm ArF laser ablation system equipped with a two-volume 'HelEx' ablation cell that facilitates rapid transfer and washout of ablated material (Photon Machines, San Diego, USA). Analytical protocol is similar to that described by Cottle et al. (2009, 2011).

U-Th/Pb analyses were conducted for 25 seconds each using a spot diameter of 31 μm, a frequency of 4 Hz and 1.5 J/cm^2 fluence (equating to crater depths of approximately 9 μm). A primary reference material, `91500' zircon (1065.4 ± 0.3 Ma $^{207}$Pb/$^{206}$Pb ID-TIMS age and 1062.4 ±0.4 Ma $^{206}$Pb/$^{238}$U ID-TIMS age, Wiedenbeck et al., 1995) was employed to monitor and correct for mass bias as well as Pb/U fractionation. To monitor data accuracy, a secondary reference zircon `GJ-1' (608.5 ± 0.4 Ma ^{207}Pb/^{206}Pb ID-TIMS age, Jackson et al., 2004) and 601.7 ± 1.3 Ma ^{206}Pb/^{238}U ID-TIMS age) was analyzed concurrently (once every 7 unknowns) and mass bias- and fractionation-corrected based on measured isotopic ratios of the primary reference material. Repeat analyses of the GJ-1 secondary reference zircon during the analytical period yield a weighted mean ^{206}Pb/^{238}U age of 604.1 ± 2.3 Ma (0.4% 2σ), MSWD = 1.8, n = 8. Data reduction, including corrections for baseline, instrumental drift, mass bias, down-hole fractionation and uncorrected age calculations were carried out using Iolite version 2.2. Full details of the data reduction methodology can be found in Paton et al. (2010). All uncertainties are quoted at the 95% confidence or 2σ level and include contributions from the external reproducibility of the primary reference material for the ^{207}Pb/^{206}Pb and ^{206}Pb/^{238}U ratios.

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Supplementary Table 1

Summary of zircon ages produced from the San Blas ash (unit 20), Tolomosa Formation, Tarija Basin, Bolivia. U-Th/He samples correspond to those in Supplementary Table 2. U-Th/Pb samples correspond to those in Supplementary Table 3 (with prefix Zeitler_).

Sample/ Aliquot	**Age (Ma)**	**± 2σ**	**Method**
Used in age estimate:			
1	0.75	0.26	U-Th/He
3	0.68	0.18	U-Th/He
4	0.75	0.16	U-Th/He
SB	0.7	0.2	Fission-Track*
002	0.82	0.05	U-Th/Pb
003	0.78	0.03	U-Th/Pb
004	0.73	0.03	U-Th/Pb
005	0.81	0.04	U-Th/Pb

006	0.77	0.02	U-Th/Pb
008	0.77	0.03	U-Th/Pb
009	0.73	0.03	U-Th/Pb
013	0.74	0.02	U-Th/Pb
*Rejected:***			
2	6.49	0.36	U-Th/He
001	1.25	0.04	U-Th/Pb
005	1.16	0.04	U-Th/Pb
010	1.06	0.05	U-Th/Pb
014	1.11	0.04	U-Th/Pb

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*San Blas (SB) ash, from MacFadden et al (1983).

**Rejected grains were those clearly in a statistically older grouping. For the U-Th/He analysis, one grain in aliquot might have been detrital. For the U-Pb analyses, the older ages are inferred to contain an inherited component.

Supplementary Table 2

Zircon U-Th/He data from the San Blas ash (unit 20), Tolomosa Formation, Tarija Basin, Bolivia.

#	Age (Ma)	± (1σ Ma)	^{4}He* (mol)	± (mol)	^{238}U (mol)	± (mol)	^{232}Th (mol)	± (mol)	Ft (mol)	F_t error (%)
1	0.75	0.13	1.159E-14	1.978E-15	1.221E-11	1.742E-13	1.639E-11	2.358E-13	0.7496	1.0
2	6.49	0.18	1.041E-13	2.267E-15	1.481E-11	2.114E-13	8.261E-12	1.180E-13	0.7430	1.0
3	0.68	0.09	1.378E-14	1.812E-15	1.794E-11	2.570E-13	1.048E-11	1.494E-13	0.7709	1.0
4	0.75	0.08	1.741E-14	1.791E-15	2.078E-11	2.976E-13	1.317E-11	1.886E-13	0.7539	1.0

Sample age (aliquots #1, 3, 4): 0.72 ± 0.12 Ma (2 σ)

Notes:

1. Uncertainties are one sigma

2. Uncertainty in ages include estimated 50% uncertainty in blank correction for ^{4}He and uncertainty in ^{4}He calibration

3. Sample age is weighted average based on aliquot uncertainties

4. Aliquot 2 rejected as an outlier (detrital grain, or loss of sample from packet before U-Th analysis).

Supplementary Table 3

U-Th/Pb analytical data from the San Blas ash (unit 20), Tolomosa Formation, Tarija Basin, Bolivia.

	Concentrations (ppm) [a]				Measured Isotope Ratios [b]						
Sample name	Pb	U	Th	Th/U	$^{207}Pb/^{206}Pb$	2σ %	$^{207}Pb/^{235}U$	2σ %	$^{206}Pb/^{238}U$	2σ %	Rho [c]
Zeitler_002	0.3930	436	242	0.54	0.2050	17.56	0.00485	31.75	0.0001617	9.15	0.96
Zeitler_003	0.1014	400	228	0.56	0.0619	21.00	0.00103	21.36	0.0001214	4.28	0.75
Zeitler_004	0.4410	439	752	1.71	0.1653	10.28	0.00300	10.67	0.0001333	3.75	0.42
Zeitler_005	0.2784	623	418	0.66	0.0963	12.67	0.00182	13.19	0.0001359	4.71	0.20
Zeitler_006	1.1190	2118	2973	1.41	0.0538	8.55	0.00089	8.35	0.0001204	1.99	0.04
Zeitler_008	0.1212	545	272	0.49	0.0595	18.82	0.00101	19.05	0.0001218	2.79	0.15
Zeitler_009	0.1157	484	267	0.55	0.0645	22.33	0.00101	21.78	0.0001153	3.30	0.05
Zeitler_013	0.6530	861	1525	1.77	0.0909	12.98	0.00151	13.25	0.0001228	2.77	0.44
Zeitler_001	0.3840	848	559	0.66	0.0640	12.19	0.00174	11.41	0.0001997	2.60	0.06
Zeitler_005b	0.3400	584	488	0.83	0.0778	12.60	0.00199	13.07	0.0001884	3.08	0.22
Zeitler_010	0.3200	583	398	0.68	0.1052	14.26	0.00254	14.17	0.0001783	3.37	0.26
Zeitler_014	0.1300	553	147	0.26	0.0715	19.30	0.00174	18.39	0.0001786	3.58	0.53

	Measured Age (Ma) [d]		Common-Lead-Corrected Isotope Ratios [e]							Corrected Age (Ma) [d,e,f]	
Sample name	$^{206}Pb/^{238}U$	2σ	$^{207}Pb/^{206}Pb$	2σ %	$^{207}Pb/^{235}U$	2σ %	$^{206}Pb/^{238}U$	2σ %	Rho [c]	$^{206}Pb/^{238}U$	2σ
Zeitler_002	1.04	0.05	0.04632	2.16	0.000798	7.02	0.0001264	6.17	0.35	0.82	0.05
Zeitler_003	0.78	0.02	0.04671	2.83	0.000759	4.48	0.0001202	4.16	0.78	0.78	0.03
Zeitler_004	0.86	0.02	0.04606	1.35	0.000717	4.46	0.0001130	4.42	0.85	0.73	0.03
Zeitler_005	0.88	0.02	0.04606	1.74	0.000796	5.28	0.0001253	5.11	1.00	0.81	0.04
Zeitler_006	0.78	0.01	0.04771	2.81	0.000759	2.21	0.0001188	2.19	0.91	0.77	0.02
Zeitler_008	0.79	0.01	0.04640	1.47	0.000757	3.43	0.0001194	3.35	0.95	0.77	0.03
Zeitler_009	0.74	0.01	0.04606	1.22	0.000720	3.89	0.0001131	3.89	0.99	0.73	0.03
Zeitler_013	0.79	0.01	0.04606	1.22	0.000728	3.02	0.0001148	3.14	0.99	0.74	0.02

Zeitler_001	1.29	0.02	0.04655	2.15	0.001238	2.91	0.0001945	2.88	0.96	1.25	0.04
Zeitler_005b	1.21	0.02	0.04629	1.34	0.001142	3.15	0.0001799	3.22	0.99	1.16	0.04
Zeitler_010	1.15	0.02	0.04607	1.56	0.001040	4.42	0.0001638	4.40	0.78	1.06	0.05
Zeitler_014	1.15	0.02	0.04624	0.99	0.001090	4.22	0.0001724	4.06	0.98	1.11	0.04

Notes:

[a]Concentration data are normalized to the primary reference material and are accurate to ~5%.

[b]$^{207}Pb/^{235}U$ is calculated assuming a natural $^{238}U/^{235}U$ ratio of 137.88.

[c]Rho value is calculated following the method outlined in Paton et al. (2010).

[d]Age calculations are based on the U decay constants of Jaffey et al. (1971).

[e]Common-lead correction utilizes the method of Andersen (2002).

[f]Correction for excess ^{230}Th follows method of Crowley et al. (2007), assuming a Th/U[magma] = 3.